Effect of grain size on cavitation in superplastic Zn-Al eutectoid

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The effect of initial grain size on cavitation during superplastic deformation in two commercially available Zn–Al eutectoid alloys has been studied using metallography and precision density measurements. Cavitation was found to be minimal for initial grain sizes below about 5 μm. Superplastic deformation caused grain growth in both alloys under all testing conditions, and when the grain size exceeded about 8 μm a significant level of cavitation was produced. The grain size and extent of cavitation increased with increasing strain along the specimen gauge length, with cavities concentrated in regions adjacent to the fracture tip. Although never very large, the cross-sectional area at fracture increased with increasing levels of cavitation. It was concluded that cavitation in Zn–Al eutectoid results from incomplete accommodation of grain-boundary sliding when excessive grain growth leads to restricted grain-boundary diffusion and/or to restricted grain-boundary migration.

1. Introduction

Despite the many studies that have been made of superplastic flow in Zn–Al alloys, only two independent groups of workers have reported the presence of cavities in these materials. The first observations of cavitation were made by Pollard [1, 2] for a range of commercial and laboratory prepared alloys containing between 8 and 12 wt% Al. Metallography revealed substantial levels of cavitation and the growth and interlinkage of cavities led to premature "quasi-brittle" tensile fracture. In the more recent studies by Langdon and his co-workers on Zn–Al eutectoid [3–5] the levels of cavitation observed metallographically were appreciably less than those previously reported, and the cavities appeared to be concentrated near the regions of fracture. They clearly played little or no role in the fracture process since the specimens pulled down to a fine point at failure.

In view of the different cavitation behaviours reported by these two groups of workers, and the fact that observations of cavitation in these extensively studied alloys are so restricted, the present work was carried out in an attempt to define the conditions under which cavitation does occur. Metallographic and density studies have been made on two commercially available superplastic Zn–Al eutectoid alloys. Specimens were annealed to give various initial grain sizes and were then superplastically deformed in tension over a range of strain rates and temperatures.

2. Experimental procedures

2.1. Materials

"Zilôn" was obtained from Vereiningte Zinkwerte of West Germany in the form of sheet of 2.5 mm thickness. Tensile specimens with a 13 mm gauge length and 5 mm gauge width were milled from the sheet. The other commercial alloy "Super-Z 200" was manufactured by the New Jersey Zinc Company USA, and was also obtained in the form of sheet of thickness 2.5 mm. The alloy was kindly supplied by Professor T. G. Langdon from a batch of material studied by Langdon and co-workers [3–5]. Tensile specimens of similar dimensions to those used by the latter workers, i.e. 6.35 mm gauge length and 5 mm gauge width, were milled from this sheet.

The concentration of zinc in the alloys and the

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TABLE I Analysed compositions of Zn–Al alloys

Alloy	Contents			
	Al (wt%)	Fe (ppm)	Cu (ppm)	Mg (ppm)
Zilôn	23.1	77	20	33
Super-Z 200	22.5	70	20	–

levels of the main impurity elements are given in Table I.

2.2. Mechanical tests

Mechanical testing was carried out in air inside a capsule attached to an Instron machine. The capsule was surrounded by a "book" furnace which facilitated rapid heating and cooling of the specimen. A temperature variation of ± 4° C was measured over the central 100 mm of the furnace. A constant strain-rate device was used in conjunction with the Instron for some of the tests. Tensile specimens were pulled to failure, or to preselected elongations, at various constant strain rates or constant cross-head velocities, at 200° C (473 K) or 260° C (533 K).

2.3. Metallographic and density studies

Metallographic features were examined using optical microscopy. Preparation of specimens pulled to failure presented considerable difficulty as a very thin foil of soft, highly strained material had to be polished. This could easily be damaged during mounting, while perforation during polishing would lead to a greatly exaggerated level of observed cavitation. For a specimen which elongated 2400% at fracture, the final thickness would be about 500 μm, assuming uniform thinning.

As specimens gradually developed necks during straining the thickness of the fracture region was always much less than 500 μm and micrometer measurements indicated that this could be as little as 40 μm. An alternate etch and polish procedure was used to remove any distorted surface layers during metallographic preparation. Final skid polishing was carried out using fine γ-alumina. Specimens were etched in 1% nital and then 1% sodium hydroxide to reveal grain and phase boundaries. Grain-size measurements were made before and after superplastic deformation by counting 1000 grain-boundary intercepts. Grain sizes are quoted as $1.74L$, where L is the mean linear intercept. Cavity morphology was observed on polished specimens.

Precision density measurements were made on the gauge lengths of specimens, deformed to preselected extents, by hydrostatic weighing in ethyl iodide, maintained at a constant temperature in a Dewar flask. A Mettler micro-balance capable of weighing to an accuracy of 0.000 01 g was used. This technique has given satisfactory and reproducible results on other alloys [6–8]. The undeformed heads of the tensile specimens were used as density standards since they had received the same thermal treatment as the gauge lengths but had not undergone mechanical deformation.

3. Results

3.1. Mechanical deformation

Zilôn specimens in the as-received condition (average initial grain size, $d_0 \sim 1.2\,\mu$m) were pulled to failure at various constant strain rates at 200° C (473 K), with strain applied in the rolling direction. Specimens showed considerable elongation and drew down to a very fine point at failure (Fig. 1).

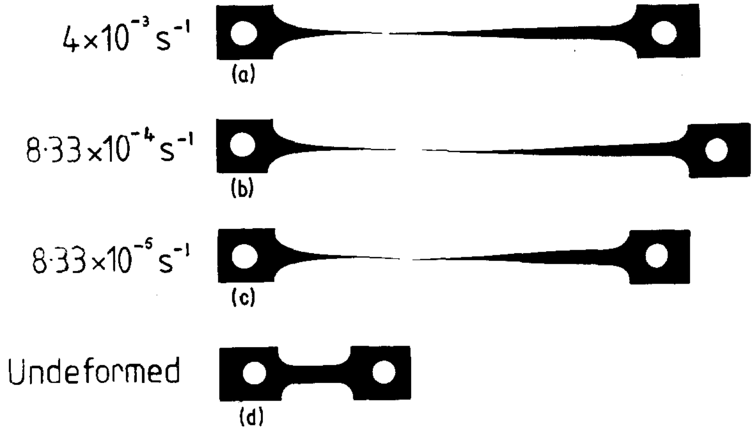

Figure 1 Fracture profiles of specimens of as-received Zilôn pulled to failure at 200° C at constant strain rates of (a) $4 \times 10^{-3}\,sec^{-2}$ (b) $8.33 \times 10^{-4}\,sec^{-1}$ (c) $8.33 \times 10^{-5}\,sec^{-1}$ and (d) an undeformed specimen.

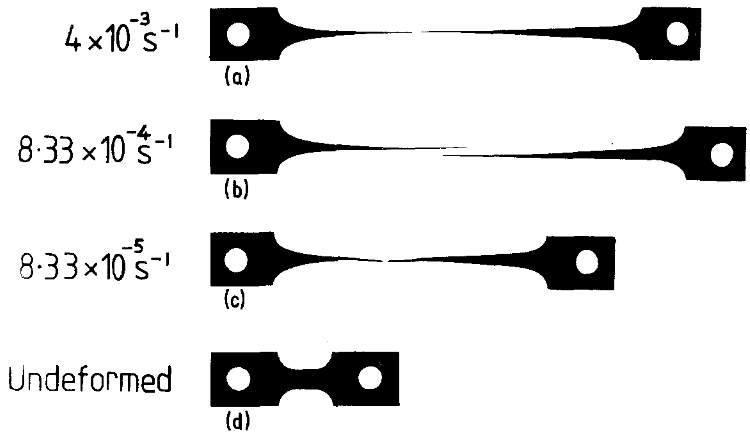

Figure 2 Fracture profiles of specimens of as-received Super-Z 200 pulled to failure at 200° C at constant strain rates of (a) 4×10^{-3} sec^{-1} (b) 8.33×10^{-4} sec^{-1} (c) 8.33×10^{-5} sec^{-1} and (d) undeformed specimen.

Similar tests were performed on as-received specimens of Super-Z 200 ($d_0 \sim 0.9\,\mu$m) with the strain also applied in the rolling direction. Fig. 2 shows that Super-Z 200 gives higher elongations then Zilôn. However, this was thought to be mainly due to the shorter gauge lengths of these specimens, rather then to an inherently greater degree of superplasticity.

Metallographic examination of these specimens failed to reveal significant cavitation. Further tests were performed at constant cross-head velocity on both alloys, in order to simulate more closely the testing conditions under which the Zn–Al eutectoid has reportedly cavitated [3–5]. Prior to testing, specimens were annealed for 0, 1, 20 and 1000 h at 260° C (533 K) to give a range of initial grain sizes (see Fig. 3).

Fig. 4 shows the influence of grain size on the profiles of specimens pulled to failure at 260° C at a cross-head velocity of 8.33×10^{-2} mm sec^{-1}. It is clear that an increasing initial grain size results in an increasing area of cross-section at fracture and also a reduced elongation. The effect on elongation is at least partially due to a reduced strain-rate sensitivity at larger grain sizes.

3.2. Metallography

Metallographic studies were made on alloys after testing under selected conditions. For all specimens deformed in the as-received condition, the

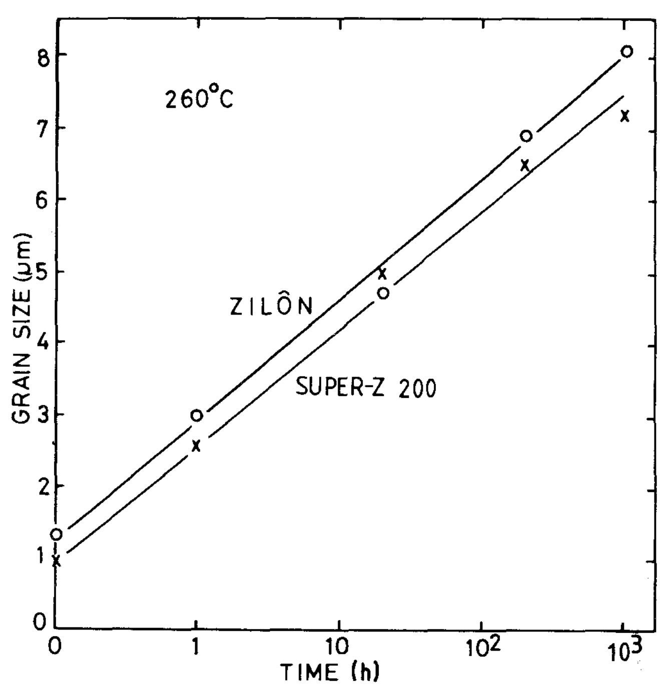

Figure 3 Effect of annealing at 260° C on grain growth in Zilôn and Super-Z 200.

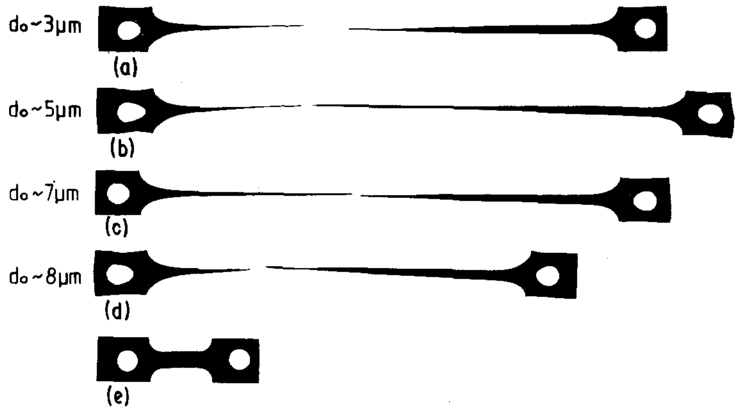

Figure 4 Effect of initial grain size on the fracture profiles of specimens of Zilôn pulled to failure at 260° C at a cross-head velocity of 8.33×10^{-2} mm sec^{-1} (a) $d_0 \sim 3\,\mu$m, (b) $d_0 \sim 5\,\mu$m (c) $d_0 \sim 7\,\mu$m (d) $d_0 \sim 8\,\mu$m and (e) an undeformed specimen.

degree of cavitation observed was negligible (Fig. 5), except in regions near the fracture tip. This was a general observation for all strain rates and temperatures.

Cavities were observed in the gauge lengths of specimens deformed after annealing and the extent of cavitation increased with increasing initial grain size and increasing strain. Fig. 6a and b refer to positions at which similar strains have occurred in two specimens of Super-Z 200 of different initial grain sizes. It can be seen that the specimen for which $d_0 \sim 5\,\mu$m (Fig. 6a) has cavitated much less extensively than that with $d_0 \sim 6.5\,\mu$m (Fig. 6b). Fig. 6c shows a position much closer to the fracture tip for the specimen with $d_0 \sim 6.5\,\mu$m and it is clear that cavities concentrate in areas of high strain. The micrographs also show that cavities tend to lie in rows or "stringers" orientated in the direction of applied strain, which is parallel to the direction of rolling of the sheet, and that they are sometimes elongated in this direction. Smaller cavities are irregular in shape and are situated at interphase boundaries (Fig. 7). Observations of grain elongation were made at positions very close to the point of fracture in several specimens (Fig. 8).

Grain-size measurements revealed that grain growth occurred in both alloys under all testing conditions and for all initial grain sizes (Figs 9, 10 and 11). Since measurements were taken from specimens pulled to failure, it should be noted that no allowance for differences in overall strain has been made. Recent work by Mohamed and Langdon [9] has shown that there is a variation of strain along the gauge length due to specimen necking. Other work, by Ghosh and Hamilton [10] on Ti–6% Al–4% V has shown that grain size increases continuously with increasing strain during superplastic flow. It would therefore be expected that the grain size would increase gradually from the end of the gauge length to the fracture tip. This was indeed found to be so and Figs 9 to 11 show that there is a significant difference between these two positions for specimen pulled to failure.

3.3. Density studies

Precision density measurements were made on

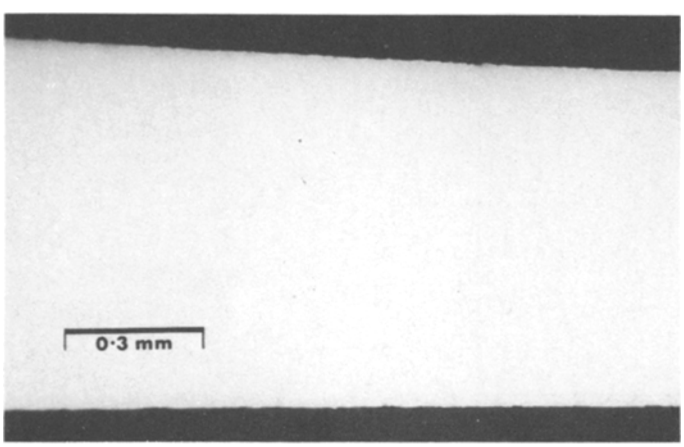

Figure 5 Polished longitudinal section of a specimen of as-received Super-Z 200 pulled to failure at 200° C at a cross-head velocity of 8.33×10^{-4} mm sec^{-1}.

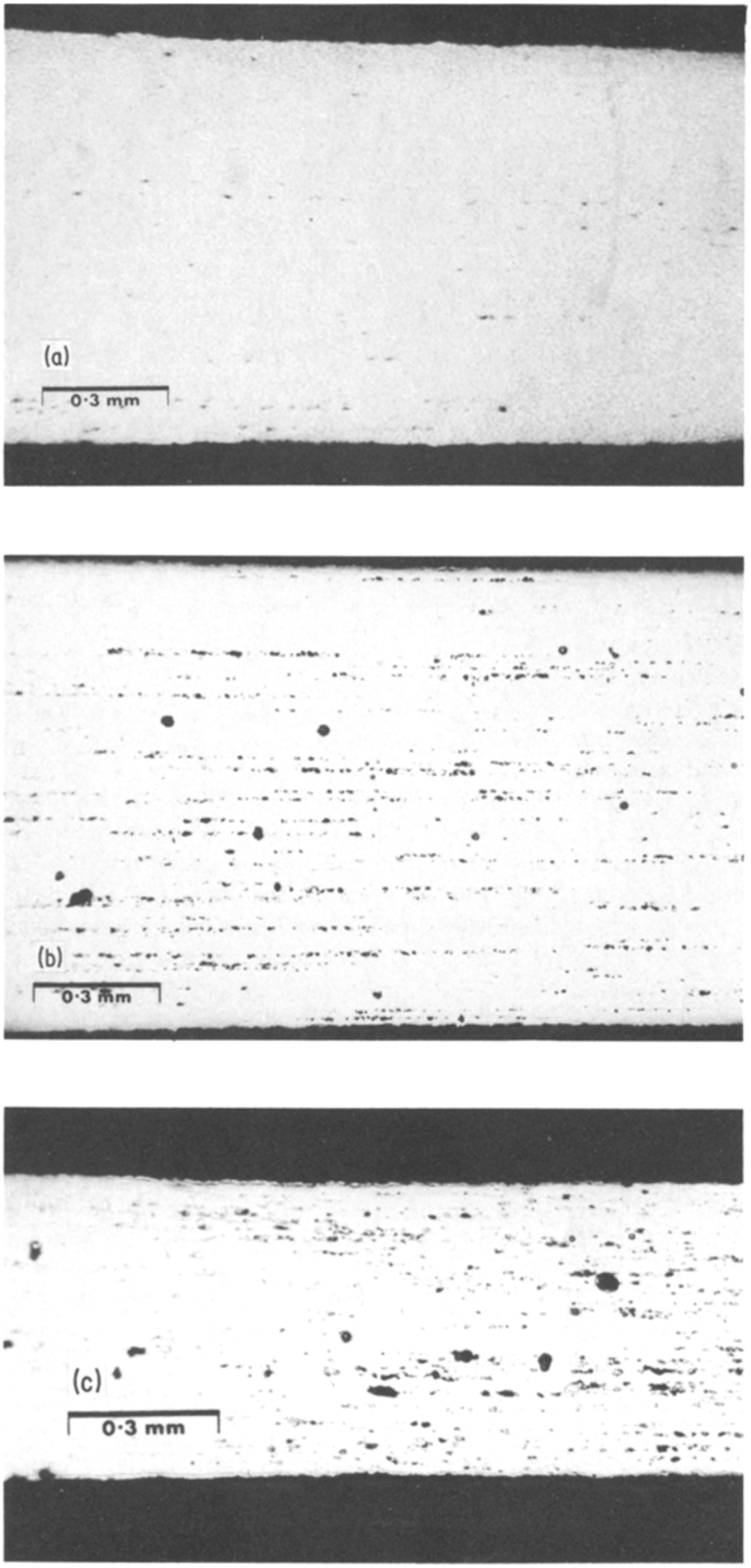

Figure 6 Polished longitudinal section of a specimen of Super-Z 200 pulled to failure at 200° C and at a cross-head velocity of 8.33×10^{-4} mm sec^{-1} (a) $d_0 \sim 5\,\mu m$ (b) $d_0 \sim 6.5\,\mu m$ (c) Grain size as for (b). Shows cavities close to fracture tip.

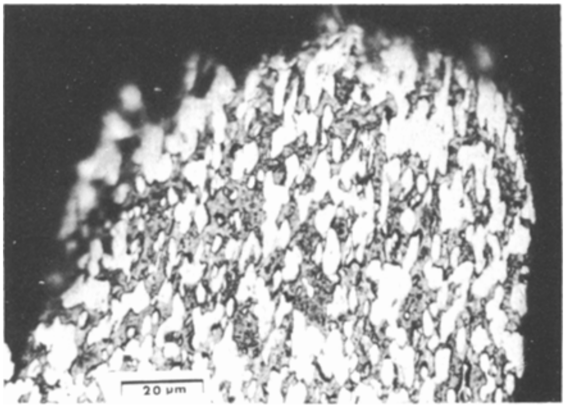

Figure 7 Micrograph of an etched specimen of Super-Z 200 of d_0 ~ 7.5 μm pulled to failure at 260° C at a cross-head velocity of 8.33×10^{-4} mm sec^{-1} showing cavities at interphase boundaries.

specimens of both alloys tested over a range of conditions. In most cases the level of cavitation was minimal, although, as the measurements were for the whole of the gauge length, this did not rule out the possibility of a highly localized concentration of cavities near the fracture tip. The overall level of cavitation increased with increasing initial grain size, and, to a lesser extent, increased with decreasing strain rate (Figs 12, 13 and 14).

The results show that an initial grain size of about 5 μm is necessary for significant cavitation to occur during superplastic flow. Figs 12 to 14 are interrelated to Figs 9 to 11 in terms of initial and final grain size, and an initial grain size of about 5 μm results in a final grain size of about 9 μm and about 11 μm near the fracture tip for

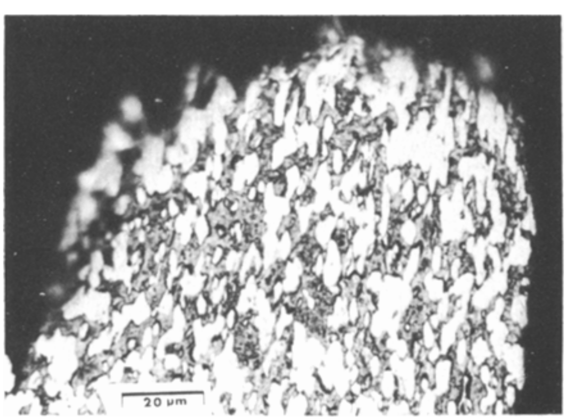

Figure 8 Micrograph of the fracture tip of a specimen of Zilôn with d_0 ~ 3 μm deformed at 200° C at a cross-head velocity of 8.33×10^{-4} mm sec^{-1} showing elongated grains.

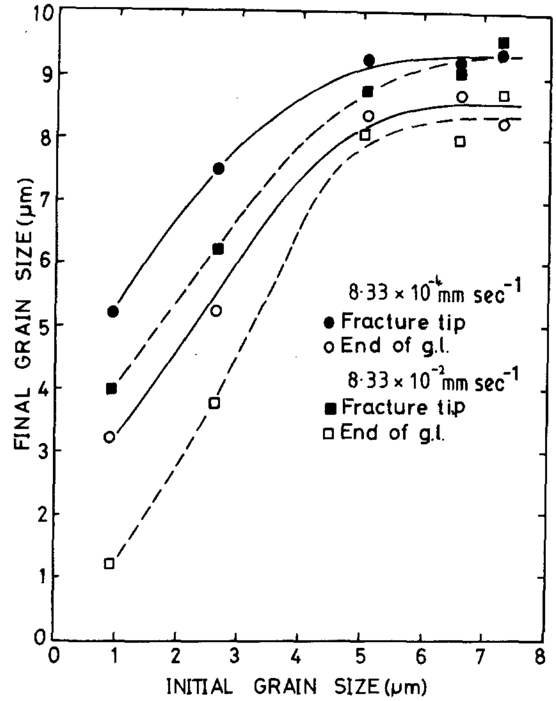

Figure 9 Final grain size against initial grain size for specimens of Super-Z 200 pulled to failure at 200° C at two cross-head velocities.

both materials tested at 200° C (473 K) and 260° C (533 K), respectively. It is suggested that the latter grain sizes are of greater relevance to the eventual level of cavitation. Figs 12 to 14 also show that relatively large volumes of cavities are formed at much lower elongations for specimens with larger initial grain sizes. This would be due to the grain size reaching the critical values of approximately 9 or 11 μm in these specimens at low strains.

4. Discussion

The development during superplastic tensile flow of a significant level of cavitation in the Zn–Al eutectoid has been reported by two independent groups of workers.

Pollard [1, 2] tested a number of alloys: a commercially extruded Zn–14% Al–0.6% Cu–0.01% Mg alloy; laboratory prepared Zn–14% Al alloys, with and without 0.6% Cu and 0.1% Mg; and laboratory prepared alloys containing 8.1 to 21% Al. The deformation temperatures, 250 to 300° C (523 to 573 K), were rather high and there were large variations in both elongation to fracture and degree of cavitation in these alloys. Specimens of Zn–21% Al were tested in both the "as-extruded" and "homogenized and quenched"

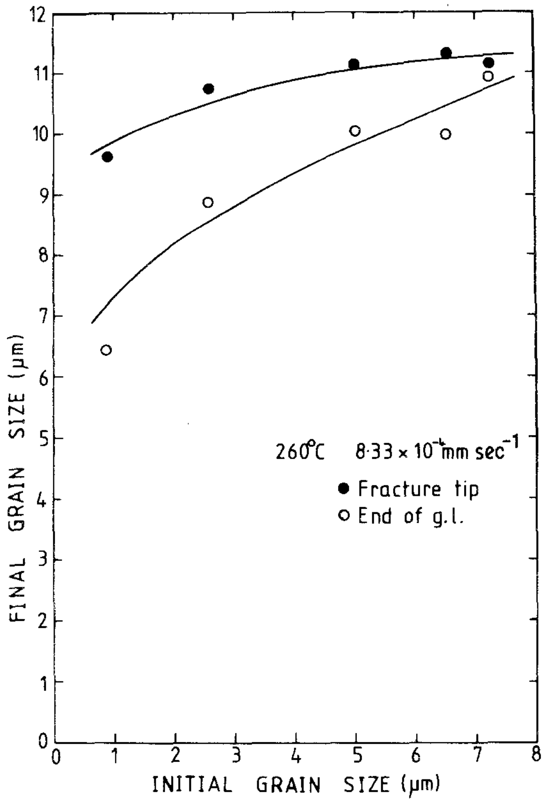

Figure 10 Final grain size against initial grain size for specimens of Super-Z 200 pulled to failure at 260° C at a cross-head velocity of 8.33×10^{-4} mm sec^{-1}.

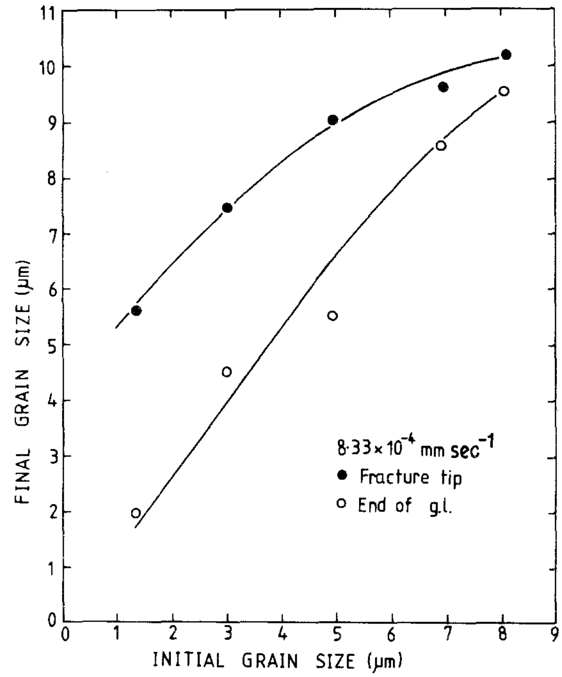

Figure 11 Final grain size against initial grain size for specimens of Zilôn pulled to failure at 200° C at a cross-head velocity of 8.33×10^{-4} mm sec^{-1}.

conditions. Specimens were heat treated at 250° C (523 K) for 1 h before testing at this temperature at an initial strain rate of 3.6×10^{-3} sec^{-1}. The "as-extruded" specimen had moderate to severe cavitation in regions of the gauge length remote from the fracture tip. The heat-treated specimen, however, showed much less cavitation near the fracture surface where the voids lay in stringers parallel to the axis of straining.

Langdon and co-workers [3, 4] have reported cavitation in Super-Z 200. After annealing for 1 h at 260° C (533 K), specimens were deformed at 200° C (473 K) using initial strain rates of 3.33×10^{-1} sec^{-1} to 1.33×10^{-5} sec^{-1}. Cavities were reported to occur at positions remote from the fracture tip, which was effectively a tapered point for strain rates lying within Region II of the logarithmic stress against strain-rate curve. Cavitation was found to increase with decreasing initial strain rate and was absent in specimens pulled at the highest strain rates. A tendency for cavities to form stringers parallel to the tensile axis was also noticed. It was suggested that both observations were consistent with the increasing importance of diffusional growth of cavities at low strain rates [4].

Essentially identical results were obtained by Miller and Langdon [5] for a very high purity Zn–Al eutectoid which eliminated the possibility that impurity particles, other than oxides, were responsible for the nucleation of cavities.

The results of the present work provide an explanation for cavitation observed in Zn–22% Al eutectoid. Even in specimens annealed for 1 000 h at 260° C (533 K), grain growth was observed during deformation, which suggests that complete stabilization is impossible. Another important result is the variation in grain size along the specimen gauge length which was not reported in the previous work.

The increasing level of cavitation with increasing initial grain size and decreasing strain rate suggests that grain size and grain growth are primarily responsible for the cavitation found in the specimens tested in the present work. Other work by Humphries and Ridley [11] on α/β brass and by Ghosh [12] on an aluminium alloy has also shown the importance of grain size on the level of cavitation produced during superplastic flow.

In general, cavity nucleation occurs when the

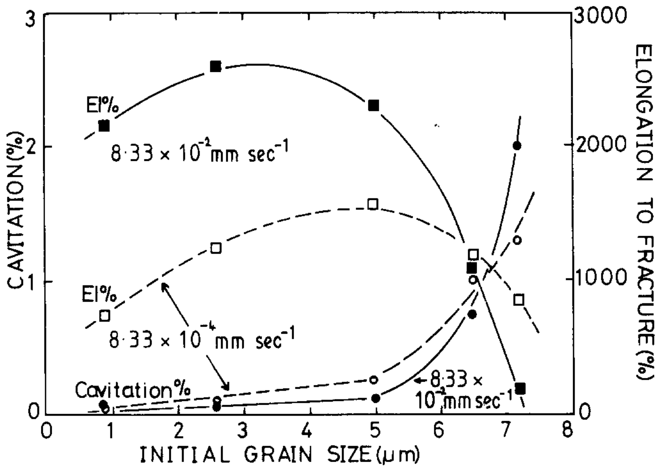

Figure 12 Overall volume per cent of cavities and elongation to fracture against initial grain size for specimens of Super-Z 200 pulled to failure at 200° C at two cross-head velocities.

rate of grain boundary sliding exceeds the rate of accommodation at an irregularity or second-phase particle situated at a grain boundary. There are several possible mechanisms of accommodation including grain growth, which involves grain boundary migration. The cavitation in Zn–Al occurs at high strains when the structure has coarsened and the rate of grain growth has slowed down. Consequently, less accommodation can occur in grain-boundary diffusion or by grain-boundary migration.

Specimens of high intial grain size, and consequently containing higher levels of cavitation, fracture by a combination of necking and cavity interlinkage normal to the direction of applied strain. A degree of tri-axiality develops which accelerates interlinkage in the highly strained and necked region of the specimen. This leads to the small but significant cross-sectional area of the fracture surface observed in Fig. 4. This phenomenon has been observed, but usually to a more marked extent, in other cavitating alloys systems [6, 8, 13].

Figs 12 to 14 show that the sudden increase in the level of cavitation with increasing initial grain size is matched by a sudden decrease in elongation to fracture. In this case the premature fracture is only partially due to the level of cavitation. A larger grain size results in lower strain-rate sensitivity, and an increased rate of necking and cavity interlinkage (material between cavities can be regarded as internal necks). The combination of increased external and internal necking, therefore, leads to premature fracture.

When specimens of Zn–Al are deformed under conditions which do not lead to excessive grain size, the overall level of cavitation is negligible and, as a result, the material is able to pull down to a fine point at fracture. However, the local strain rates in necked regions would be greatly increased prior to fracture and there is some evidence that final failure may occur by a creep

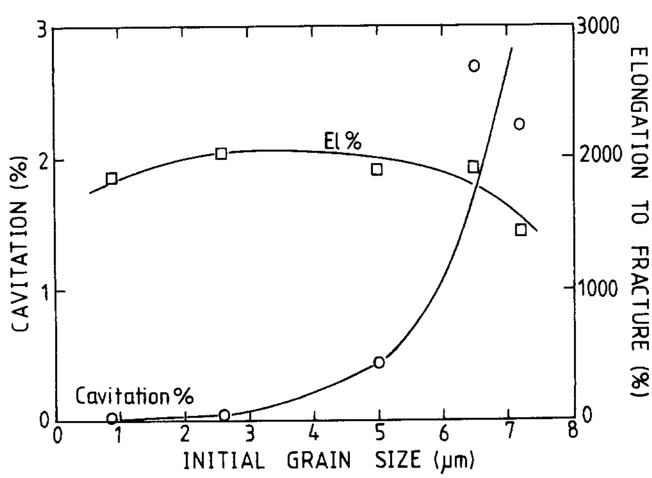

Figure 13 Overall volume per cent of cavities and elongation to fracture against initial grain size for specimens of Super-Z 200 pulled to failure at 260° C at a cross-head velocity of 8.33×10^{-4} mm sec^{-1}.

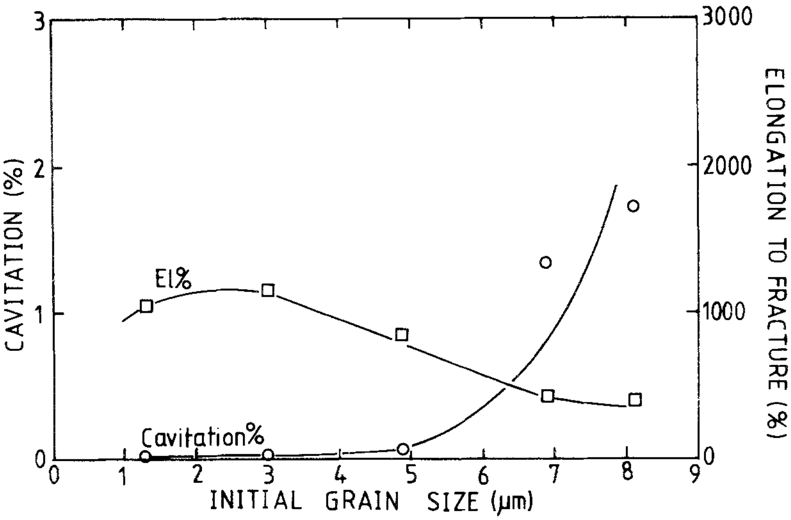

Figure 14 Overall volume per cent of cavities and elongation to fracture against initial grain size for specimens of Zilôn pulled to failure at 200° C at a cross-head velocity of 8.33×10^{-4} mm sec^{-4}.

mechanism involving grain elongation, (Fig. 8).

In the work of Pollard [1, 2] large variations of elongation were observed and this would seem to indicate that some of the alloys tested had remnants of the cast microstructure. This could possibly be the case for the Zn–21% Al alloy which was extruded and showed severe cavitation. A similar alloy produced by rolling, heat treatment and quenching, cavitated much less. Other alloys containing additions of copper could have contained hard intermetallic precipitates. The effect of "second-phase" particles on cavitation in the Zn–Al eutectoid is currently being investigated by the authors [14]. It is clear from the micrographs shown that the cavitation observed by Pollard was much more severe and probably quite different from that observed by Langdon and co-workers, and resulted in fracture faces of large cross-sectional area.

5. Conclusions

Conclusions can be drawn as follows:

(a) The two commercial Zn–Al eutectoid alloys, Super-Z 200 and Zilôn, displayed a very similar susceptibility to cavitation during superplastic tensile flow. The materials only cavitated to a significant extent when the initial grain size was increased by prior heat treatment to above a value of about 5 μm. This resulted in final grain sizes of approximately 9 and 11 μm at 200° C (473 K) and 260° C (533 K), respectively, after superplastic deformation, but even then the overall cavity volume was always < 3%.

(b) Complete stabilization of the grain size of both alloys was impossible and grain growth occurred under all testing conditions. In addition, a variation of grain size along the specimen gauge length was observed and this indicated that grain growth was primarily strain controlled.

(c) Cavities were concentrated heavily in areas of high strain close to the fracture surfaces, which showed an increased cross-sectional area with increasing level of cavitation.

(d) Increasing cavitation with increasing grain size can be explained in terms of a reduced ability to accommodate grain-boundary sliding by grain-boundary diffusion or by grain-boundary migration.

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