JOURNAL OF MATERIALS SCIENCE 10 (1975) 1651–1654

The structure and properties of a cold-rolled and annealed Al-0.8 wt % Zr alloy

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The effect of annealing at 400° C on the microstructure of a cold-worked Al–0.8 wt % Zr alloy is reported. It is shown that the initially high dislocation density in the cold-rolled material is progressively reduced, although the grains and subgrains were exceptionally resistant to coarsening. Precipitation of the metastable cubic Al_3Zr phase occurred, both discontinuously in the form of fan shaped precipitates and also on the grain boundaries and within the grains as small, nearly spherical particles. The mechanical properties of the alloy at 400° C are consistent with a major dislocation contribution to the overall deformation process, in contrast with most other fine grained materials which are superplastic.

1. Introduction
There have been several reports [1–3] of investigations of precipitation in dilute Al–Zr alloys containing $\gtrsim$ 0.5 wt % Zr. The observed sequence on ageing is the nucleation of cubic Al_3Zr precipitates with the Ll_2 structure, followed at much longer ageing times by the formation of the equilibrium tetragonal Al_3Zr phase, DO_{23} structure. Only a limited amount of work has been reported [4] on the influence of prior cold-work on the aged microstructures, and this aspect is persued further in the present study. Zirconium is known to be an effective grain refining agent in aluminium alloys, and so the present work was undertaken to ascertain whether cold-work and annealing of an Al–0.8 wt % Zr alloy could produce a stable, fine grained structure capable of superplastic deformation.

2. Experimental
An Al–0.8 wt % Zr alloy was prepared from high purity aluminium and 99.8 zirconium sponge. The materials were melted in an alumina crucible and held at 1000° C for 10 min before chill casting into an ingot 12.5 mm diameter. Chemical and spectrographic analysis showed the following composition in wt %, balance Al

Ca	0.004	Cr	0.0005	Cu	0.001	Fe	0.003
Mn	0.0001	Mg	0.01	Si	0.01	Na	0.0001
Zn	0.27	Zr	0.80				

A slice 1 mm thick was cut from the ingot and the remainder was cold-rolled to 1 mm thickness. Specimens 10 mm square were heat-treated at 400° C in a fluidized bed for times up to 200 h. Tensile specimens were cut with the tensile axis parallel to the rolling direction and deformed at 400° C. Samples suitable for transmission electron microscopy were prepared by electropolishing at −20° C in a solution of 5% perchloric acid in methanol using a potential of 20 V. For the transformation studies the window method was used whereas disc specimens were used for deformed material. All structures were observed parallel to the initial rolling plane.

3. Results and discussion
The microstructure of the as-cast material consisted mainly of essentially single phase grains. However some regions were observed containing fan-shaped Al_3Zr precipitates with the Ll_2 structure, (Fig. 1), and their morphology is consistent with growth by a discontinuous reaction, as proposed by Nes and Ryum [3]. Dark-field micrographs taken using aluminium reflections imaged the matrix on both sides of the interface, showing that the misorientation across it was probably less than 1°, i.e. the reaction front is not a high-angle grain boundary as is commonly observed in discontinuous precipitation reactions. Dislocations were observed at the transformation front, (Fig. 1), and because of the low mismatch between matrix and particle [2] it is unlikely that these dislocations are punched out by the precipitates, as

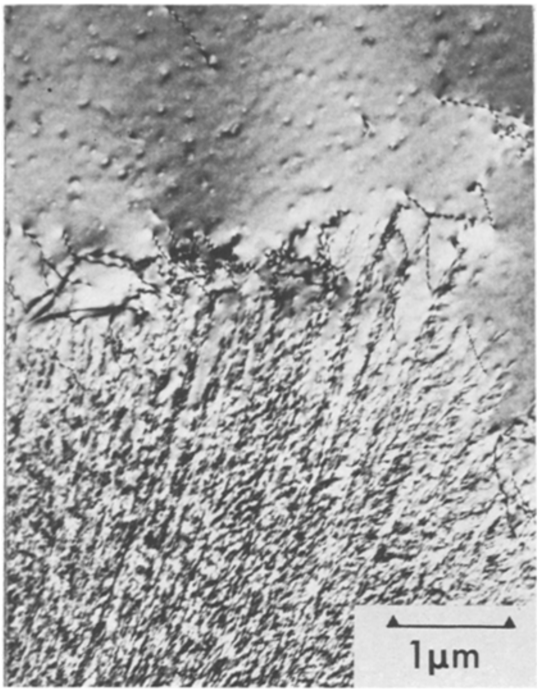

Figure 1 Fan shaped precipitates of Al_3Zr in the as-cast material. Note the accumulation of dislocations at the interface.

has been suggested for the autocatalytic growth of some discontinuous reactions [5, 6]. A more probable explanation is that dislocation debris from within the grains is accumulated at the transformation front. However, in any case the dislocations may act as pipe diffusion paths to aid precipitate growth. The inhomogeneity of the as-cast structure probably reflects segregation in the cast ingot, since it has been shown [1] that fan-shaped precipitates are associated with Zr-rich regions.

After cold-rolling, these precipitates were broken up so that well-defined rods were no longer present, and a high dislocation density was introduced into the precipitate free regions, (Fig. 2). Elongated grains were present, containing a cell structure with dislocation tangles and sub-boundaries separating regions of lower dislocation density.

On ageing cold-worked material at 400° C, there was both a progressive decrease in dislocation density, and concommitant precipitation of the metastable cubic Al_3Zr phase. The dislocation sub-boundaries became more well defined with a lower dislocation density between them, (Fig. 3), and on further ageing many of the dislocation networks annealed out. Thus after 1 h at

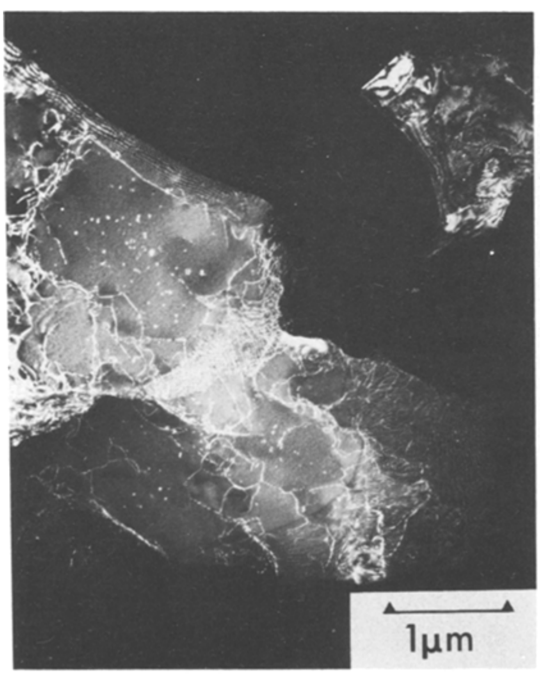

Figure 2 The microstructure of the cold-rolled material. Dark-field electron micrograph taken under weak beam diffraction conditions.

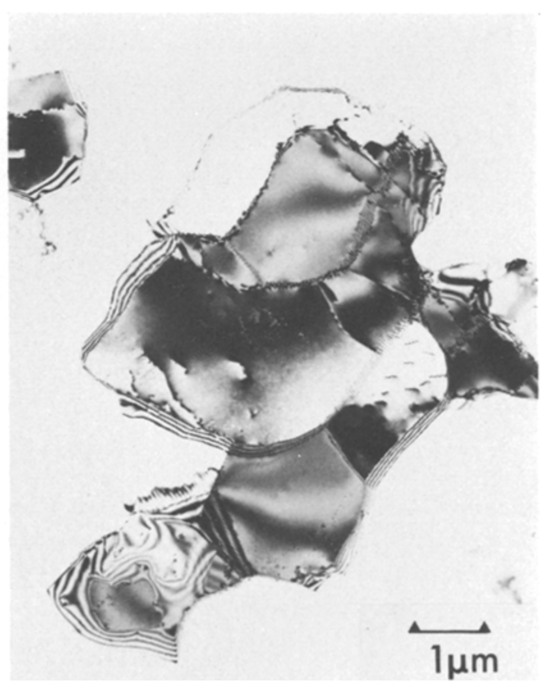

Figure 3 The microstructure produced after cold-rolling and annealing for 30 min at 400° C.

400° C, the high dislocation density present in the cold-worked material is considerably reduced and small, nearly defect-free grains and subgrains are present. Dark-field imaging techniques showed that small specimen tilts $\gtrsim 5°$ brought several adjacent "grains" into a diffracting position, indicating that many of the boundaries present are of low angle. After ageing for 175 h there was no further appreciable change, i.e. the grains and subgrains are exceptionally resistant to coarsening.

Three morphologies of cubic Al_3Zr precipitates were observed. Fan-shaped precipitates, (Fig. 4), were present at very short ageing times, 1 min or less, and then no further significant change occurred within these regions. This transformation proceded very rapidly and was only observed after it had gone to completion. However, as will be discussed later, Al_3Zr precipitates are very effective in pinning the grain boundaries. Consequently, the most probable sequence of events is that the boundary starts to migrate before precipitation occurs, the driving force for this movement being a reduction of dislocation density within the grain. As the boundary moves, accumulated solute leads to Al_3Zr precipitation and continued migration of the boundary enables the precipitates to grow discontinuously. Where precipitation occurs on a static boundary, it is difficult to obtain a driving force to start to move the boundary in one direction [7].

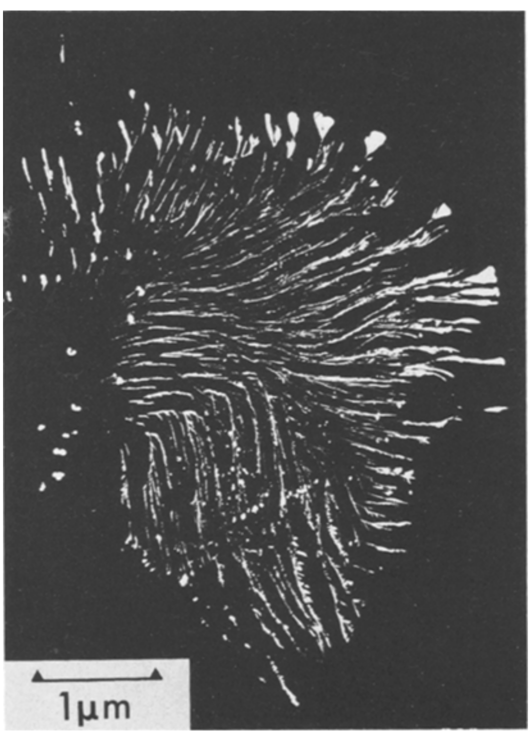

Figure 4 Fan-shaped precipitates of Al_3Zr produced on ageing for 1 min at 400° C. Dark-field micrograph taken using a (1 1 0) superlattice reflection.

Small, nearly spherical particles of cubic Al_3Zr were observed within some of the grains of the as-cast material, and also on the grain boundaries after ageing. On ageing for 175 h at 400° C, rod shaped precipitates of cubic Al_3Zr were observed, and also some particles of tetragonal Al_3Zr. All these precipitate morphologies and distributions are similar to those previously reported by other workers [1, 2, 8].

The mechanical properties of the cold-rolled alloy tested in tension at 400° C after 30 min

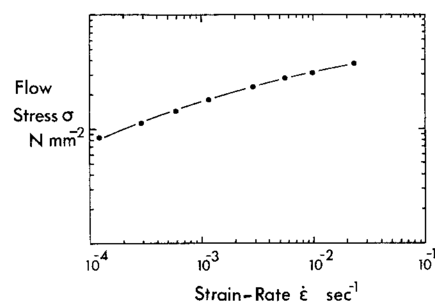

Figure 5 The flow stress (σ)–strain-rate ($\dot{\epsilon}$) relation for material deformed in tension at 400° C.

ageing prior to deformation are illustrated in Fig. 5 in the form of a log flow stress/log strain-rate curve determined incrementally on a single specimen. The strain-rate sensitivity m, defined as $d \log \sigma / d \log \dot{\epsilon}$, is given by the slope of the curve and = 0.2, decreasing slightly at higher strain-rates. Since the datum points for the curve are obtained at low total strains (~ 10% elongation) it is thought that this value is probably characteristic of the fine grained regions, the few large grains acting as non-deforming particles.

Fig. 6 shows the structure within the fine grained regions after 100% deformation at an initial strain rate of 1.2×10^{-3} sec^{-1}, with 30 min age at 400° C before testing at that temperature. Under these conditions, the maximum elongation of 140% was obtained. It can be seen that the dislocation density is much higher than in material annealed for similar total times, with the dislocations in tangles and networks and no well-defined sub-boundaries introduced. Furthermore, considerable grain growth was observed after deformation compared with material annealed for

Figure 6 The microstructure after 100% tensile deformation at 400° C. Note the increased grain size and higher dislocation density within the grains compared to Fig. 3.

the same time. This deformation enhanced grain growth is probably a consequence of the extra driving force provided by the dislocations introduced within the grain.

The value of strain-rate sensitivity and the high dislocation density both indicate that dislocation creep contributes significantly to the deformation process. This is in contrast with most fine grained materials, which deform predominantly by grain boundary sliding and show large elongations characteristic of superplasticity [9]. Although the coarse grained areas containing Al_3Zr precipitates may be responsible for the low ductility of the present alloy, the grain growth and high dislocation density observed within the fine grained regions indicate that even if decomposition during casting were eliminated, low total elongations would still be expected.

Benedek and Doherty [10] showed that boundary misorientation may be important in superplasticity, since small misorientations lead to a decrease in grain-boundary sliding and a decrease in boundary diffusivity. The present work also indicates that the presence of many low-angle boundaries results in an increase in the contribution of slip processes to the overall deformation.

Acknowledgements
This work was carried out at the Department of Metallurgy and Materials Science, University of Cambridge, England and the author is grateful to Professor R. W. K. Honeycombe for the provision of laboratory facilities and to the Science Research Council for financial support.

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Received 21 March and accepted 14 April 1975.