INFLUENCE OF ROLLING ON THE SUPERPLASTIC BEHAVIOR OF AN Al-Mg-Sc ALLOY AFTER ECAP

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Introduction

It is now well established that the grain size of metallic alloys may be substantially refined, to the submicrometer or even the nanometer range, through the application of severe plastic deformation [1–3]. Several experimental techniques are available for applying the deformation but Equal-Channel Angular Pressing (ECAP) is an especially attractive procedure because it provides an opportunity for producing reasonably large fully-dense bulk samples. It was noted earlier [4] that, since the strain rate associated with optimum superplasticity is displaced to faster strain rates when the grain size is reduced, it may be possible to use ECAP to prepare materials capable of exhibiting superplastic ductilities at rapid strain rates above \( \sim 10^{-2} \text{ s}^{-1} \) in the region generally designated High Strain Rate Superplasticity [5]. Several reports have confirmed this possibility in both commercial and laboratory alloys [6–10]. The introduction of a rapid forming capability through ECAP was also demonstrated recently in an Al-3% Mg-0.2% Sc alloy by cutting disks from the as-pressed rods and forming into domes using a simple biaxial gas-pressure forming facility [11]. It follows from these preliminary results that it may be feasible to use ECAP to overcome the limitations imposed on conventional superplastic forming operations due to the relatively slow forming rates (\( \sim 10^{-3}–10^{-2} \text{ s}^{-1} \)) and the long forming times (\( \sim 20–30 \text{ minutes} \)) required for the production of each component [12,13].

Samples produced by ECAP are generally in the form of rods with either square or circular cross-sections. Although this shape is suitable for use in superplastic forging operations, it is not appropriate for conventional superplastic forming where materials are in the form of thin sheets. To date, there are no results showing whether the superplastic characteristics introduced into a material by ECAP, and revealed in standard tensile testing, are also retained if the as-pressed materials are subsequently rolled into sheets. The present investigation was initiated to evaluate the effect of cold rolling after ECAP.

The Al-3% Mg-0.2% Sc alloy was selected for use in these experiments because it was established earlier that this alloy exhibits exceptionally high tensile ductilities at rapid strain rates following ECAP [11].

Experimental Material and Procedures

The experiments were conducted using an aluminum alloy having a composition, in wt%, of 3% Mg and 0.2% Sc. Full details of the preparation procedure for the alloy were given earlier [11]. Briefly, the
alloy was prepared by arc melting 99.99% purity Al and 99.999% Sc to form an Al-3% Sc alloy and then remelting with additional Al and 99.9% purity Mg to give an Al-3% Mg-0.2% Sc alloy. This alloy was cast, homogenized in air at 753 K for 24 h, cut into bars and swaged into rods with diameters of 10 mm, cut to lengths of ~60 mm for ECAP and then solution treated for 1 h at 883 K. The mean initial grain size prior to ECAP was measured as ~200 μm.

The principles of processing by ECAP are described elsewhere [14–17]. All pressings were conducted at room temperature using a solid die having an angle between the two channels of 90° and an angle of ~45° at the outer arc of curvature at the point of intersection of the two channels. This configuration leads to an imposed strain of ~1 on each passage through the die [18]. Samples were coated in an MoS₂ lubricant and pressed at a speed of ~19 mm s⁻¹ for different numbers of passes through the die. The samples subjected to repetitive pressings were rotated by 90° in the same sense between each pass in the procedure designated route B₀C [19]. This route was selected because it has been shown for pure Al that it leads most expeditiously to a homogeneous microstructure of equiaxed grains separated by boundaries having high angles of misorientation [20].

Following ECAP, the as-pressed rods were circular in cross-section and there was no reduction in their initial diameter of 10 mm. Each as-pressed rod was ground parallel to the z plane and ~1.5 mm was removed from either side to give bars with two flat parallel surfaces, where the z plane is defined as the top plane at the point of exit from the ECAP die [19]. This bar, having a thickness of ~7 mm, was then rolled at room temperature to a final thickness of 2.2 mm, giving a total reduction of ~70% and an equivalent strain of ~1.3 when the strain is estimated from the expression {1.15 ln (t₀/t₁)}, where t₀ and t₁ are the initial and final thicknesses, respectively [21]. The reduction in thickness was adjusted to ~0.2 mm in each rolling pass and 25 passes were used to achieve the final thickness in each sample.

Following ECAP and cold rolling (CR), tensile specimens were machined from the samples with gauge lengths oriented parallel to both the pressing and the rolling direction and with cross-sectional areas of 2 × 3 mm² and gauge lengths of 5 mm. These samples were pulled to failure in air at a temperature of 673 K using a testing machine operating at a constant rate of cross-head displacement and with an initial strain rate of 3.3 × 10⁻² s⁻¹. Each sample was heated to 673 K over a period of ~30 minutes, held at temperature for ~10 minutes and then tested with the temperature maintained constant to within ±1 K. For comparison purposes, some tensile samples were also machined directly from the as-pressed rods without any subsequent CR and tested under identical conditions and, in addition, some samples were also prepared by CR without ECAP. The testing temperature of 673 K and strain rate of 3.3 × 10⁻² s⁻¹ were selected because earlier experiments revealed high superplastic ductilities in this alloy under these conditions [11].

Specimens were prepared for transmission electron microscopy (TEM) using the procedure described earlier [22] and they were examined in an Hitachi H-8100 transmission electron microscope operating at 200 kV. The TEM observations were made on the y plane for the samples after ECAP and on the z plane after ECAP and CR, where the y plane is defined as the plane parallel to the side face of the sample at the point of exit from the ECAP die [19]. Selected area electron diffraction (SAED) patterns were taken for each condition from regions having diameters of 12.3 μm.

Results and Discussion

Figure 1 shows the microstructures, and the associated SAED patterns, for samples subjected to (a) ECAP to a total of 8 passes and (b) ECAP to a total of 8 passes plus CR. Inspection of Fig. 1 leads to three conclusions. First, the grain size is very significantly refined through ECAP with a grain size in Fig. 1(a) of ~0.2 μm; this result is consistent with earlier reports [9,11]. Second, Fig. 1(b) reveals that the grains become elongated into a banded structure during the subsequent CR, these bands are oriented...
essentially parallel to the rolling direction, and many dislocations are introduced within the grains although there is no significant change in the measured grain size. Third, although the overall appearance of the microstructures in Figs 1(a) and (b) is different, both sets of SAED patterns show well-defined ring patterns indicative of the presence of boundaries having high angles of misorientation: this latter trend was noted earlier for samples subjected only to ECAP [9,11] but there have been no similar observations after ECAP plus CR.

Although Fig. 1 refers to the microstructures after 8 passes of ECAP, detailed observations were made also after CR without ECAP and after CR following ECAP through 1, 4 and 6 passes. These observations revealed an SAED net pattern and low angle boundaries in all areas after CR without ECAP and after 1 pass of ECAP plus CR, SAED net and ring patterns in area fractions of ~40% and ~60%, respectively, after 4 passes of ECAP plus CR, and SAED net and ring patterns in area fractions of ~30% and ~70%, respectively, after 6 passes of ECAP plus CR. By contrast, the ring pattern shown in Fig. 1(b) was representative of all areas after 8 passes of ECAP plus CR. These observations reveal the gradual evolution of the microstructure from initial arrays of subgrains after CR or after ECAP for 1 pass plus CR to arrays of high angle boundaries after 8 passes of ECAP plus CR.

Plots of true stress versus elongation are shown in Fig. 2 for the samples subjected to CR after ECAP from 0 (unpressed) to 8 passes, where the number adjacent to each curve denotes the number of passes in ECAP. These curves reveal a transition from very limited ductility after CR following ECAP through 0 or 1 pass to exceptional superplastic behavior after CR following ECAP to a total of 8 passes. It is also apparent that the maximum flow stress decreases with increasing numbers of ECAP passes.

Figure 1. Microstructures and associated SAED patterns after (a) ECAP to 8 passes and (b) ECAP to 8 passes plus CR.
Figure 3 shows the variation of the elongation to failure with the number of ECAP passes for samples after ECAP without CR (open points) and after ECAP plus CR (closed points). It is apparent that the overall trends are very similar in both sets of samples, especially when ECAP is taken through 4 or more passes. Indeed, the close superposition of the two datum points after 8 passes demonstrates unequivocally that the potential for achieving superplastic ductilities is not diminished by the subsequent cold rolling of samples after processing by ECAP. The slightly larger elongations attained in the samples after CR following ECAP through 0, 1 and 2 passes probably reflects additional microstructural evolution introduced during the subsequent rolling. Nevertheless, these elongations remain relatively small because of the preponderance of low-angle boundaries.

The appearance of the samples after testing to failure following ECAP plus CR is shown in Fig. 4 where there is an undeformed sample at the top and the other specimens were subjected to identical CR
after ECAP through 0 to 8 passes, respectively. The very uniform deformation within the gauge length of the lower sample, which pulled out to an elongation of 1860%, confirms the occurrence of true superplasticity in this sample.

These experiments lead to the following conclusion concerning the effect of cold rolling in producing sheet metal following processing by ECAP. Although the ultrafine grains introduced in ECAP become elongated within a banded structure parallel to the rolling direction in CR, and many dislocations are introduced within the grains, CR provides a simple procedure for producing superplastic sheet metals in as-pressed samples without any diminution in the superplastic properties.

**Summary and Conclusions**

1. Equal-channel angular pressing (ECAP) produces an ultrafine grain size and superplastic ductilities at very rapid strain rates in an Al-Mg-Sc alloy.
2. Essentially identical high superplastic elongations are also observed after ECAP plus cold rolling (CR) to form a sheet metal. The elongations remain unchanged because, although the grains become elongated within a banded microstructure in CR and dislocations are introduced within the grains, the boundaries of the ultrafine grains retain their high angles of misorientation.
3. The results confirm that cold rolling after ECAP is an effective procedure for the production of a superplastic sheet metal.

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