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Microstructural Evolution in Pure Aluminium and a 7034 Alloy Processed by Equal-Channel Angular Pressing

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Abstract

Experiments were conducted to evaluate the microstructural evolution occurring in pure aluminium and a spray-cast Al-7034 alloy after processing through equal-channel angular pressing (ECAP). Electron back-scatter diffraction (EBSD) was used to determine the grain boundary misorientations and the texture after ECAP and differential scanning calorimetry (DSC) was employed to evaluate the nature of thermal effects taking place during heating. The results demonstrate there is an increase in the fraction of boundaries having high angles of misorientation with increasing strain. For pure aluminium, there is an S texture {122} <634> after 4 passes and a brass texture {011} <211> after 8 passes. For the Al-7034 alloy, the textures changes from a fibre $<111>_x$ texture in the as-received condition to a $<101>_y$ after 1 pass, $<111>_y$ after 4 passes and $<100>_z$ after 8 passes. For the Al-7034 alloy, the DSC analysis identifies the occurrence of several thermal effects during heating involving the formation, coarsening, dissolution and melting of the η-phase.

1. Introduction

The reduction of the grain size of polycrystalline metals to the submicrometer or nanometer range is attractive for two reasons. First, it will lead to an increase in the strength and toughness of the material. Second, there is a potential, if the ultrafine grains are reasonably stable at elevated temperatures, for attaining a superplastic forming capability at strain rates that are substantially higher than in conventional superplastic alloys. Several procedures are now available for producing metallic alloys with extremely small grain sizes but many of these procedures, such as inert gas condensation and high-energy ball milling with subsequent consolidation, are not satisfactory for producing large bulk fully-dense materials without any residual porosity. Thus, attention has focussed instead on the alternative procedure of using severe plastic deformation in order to achieve significant grain refinement in bulk materials [1]. The process of Equal-Channel Angular Pressing (ECAP) appears to be especially attractive for producing bulk metallic alloys having grain sizes that lie, typically, in the submicrometer range [2], where ECAP denotes the process of pressing a bulk sample, in the form of a rod or bar, through a die constrained within a channel bent through an abrupt angle. To date, there have been numerous investigations to evaluate the capabilities of the ECAP processing technique [3].

The present investigation was initiated to provide a critical evaluation of the microstructural evolution associated with the ECAP processing of pure aluminum and an Al-7034 alloy using electron back-scatter diffraction (EBSD) and differential scanning calorimetry (DSC).

2. Experimental Materials and Procedures

The experiments were conducted using pure aluminium of 99.99% purity and a spray-cast Al-7034 alloy containing, in wt. %, 11.5% Zn, 2.5% Mg and 0.9% Cu. The processing by ECAP was conducted using a die with an internal angle of 90° so that each pass corresponded to an imposed strain of ~1 [4] and using processing route B_C in which the samples are rotated by 90° in the same sense between each pass [5]. The EBSD analysis used a JEOL JSM-6500 multi-purpose high performance FEG-SEM with a step size of 0.2 μm for pure Al and 0.05 μm for the Al-7034 alloy. Data were analyzed using HKL Channel 5 software. The DSC experiments were performed using a Perkin-Elmer Pyris 1 in a nitrogen atmosphere at a constant heating rate of 10 K/min and with samples in the form of discs with thicknesses and diameters of ~0.9 and ~5 mm, respectively. All runs were corrected by subtracting the DSC baseline obtained from a run with an empty pan [6].

3. Experimental Results and Discussion

3.1 Pure Aluminium

The pure Al had an initial grain size of ~1 mm which was reduced to ~1.1 μm after 4 and 8 passes of ECAP. Typical grain boundary maps and misorientation distributions, obtained by EBSD, are shown in Figs 1 and 2 after ECAP through 4 and 8 passes: the high-angle boundaries are defined specifically as having misorientations >15º.

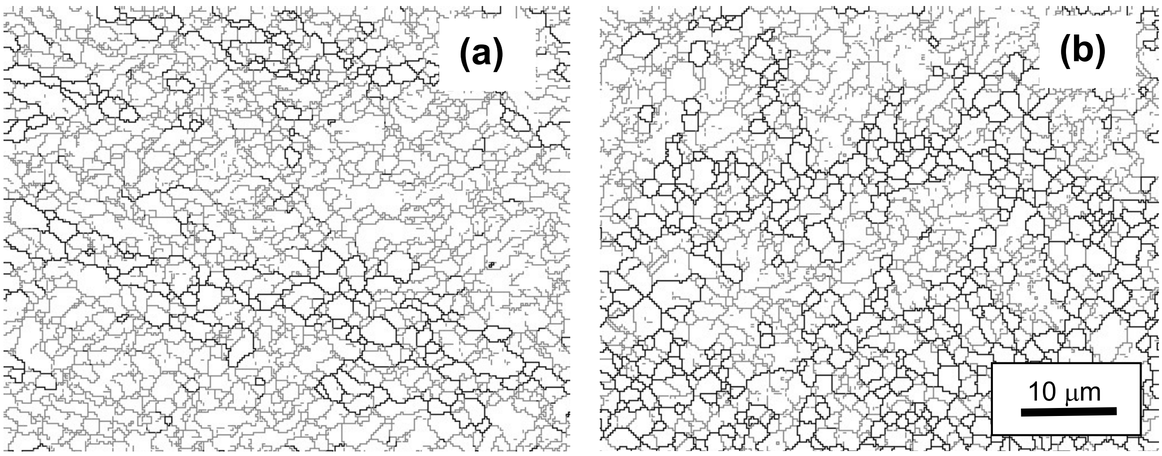

Figure 1: Grain boundary maps for pure aluminium after (a) 4 and (b) 8 passes of ECAP. High angle grain boundaries (misorientation >15º) in black, low angle grain boundaries in grey.

In Figure 1, the low-angle boundaries having misorientations from 2° to 15° are denoted by grey lines and the high-angle boundaries are shown as dark lines. It is apparent that the grain sizes are similar after 4 and 8 passes but there is a higher fraction of high-angle boundaries after 8 passes. The transition to an array of grains having higher angles of misorientation is further confirmed by the distribution histograms shown in Figure 2. Nevertheless, although the array of grains produced by severe plastic deformation after 8 passes has a higher fraction of high-angle boundaries than the sample pressed through only 4 passes, the distribution remains far removed from the theoretical random distribution which is shown by the solid line in Figure 2. Thus, the distribution of misorientation angles after 8 passes shows a bimodal character with peaks at both low (~5º) and high (~55º) angles.

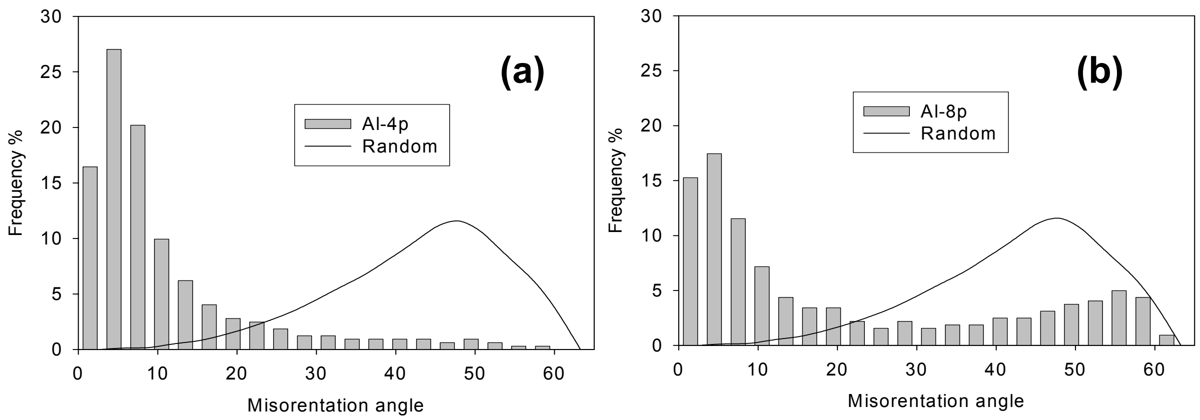

Figure 2: Boundary misorientation distributions of pure Al after (a) 4 and (b) 8 passes of ECAP.

The discrepancy by comparison with the random distribution arises in part because the ECAP process incorporates continuous deformation without any heat treatment so that dislocations are introduced into the material on each separate pass leading to an unusually high fraction of low-angle boundaries [7]. There is experimental evidence showing the distributions approximate more closely to the random distributions using high-pressure torsion [8]. Analysis of the EBSD grain orientation data revealed the presence in pure Al of an S{123}<634> texture after 4 passes and a brass {011}<211> texture after 8 passes.

3.2 Al-7034 Alloy

Typical EBSD grain boundary maps are shown in Figure 3 for the Al-7034 alloy and Table 1 summarizes the change of microstructural parameters after ECAP including the average grain area, the grain diameter, the grain aspect ratio and the number of neighbouring grains after pressing up to 8 passes.

Table 1: Microstructural characteristics of Al-7034 using EBSD analysis

ECAP	Grain area (μm^2)	Grain diameter (μm)	Aspect ratio	No. neighbour grain
As-received	2.79	1.66	1.57	5.67
1 p	0.41	0.60	1.49	4.26
4 p	0.26	0.49	1.63	5.26
8 p	0.30	0.51	1.51	5.18

The average grain size of the alloy was reduced from ~1.7 μm in the as-received condition to ~0.5 μm after 4 and 8 passes. The grain boundary maps from the EBSD analysis shown in Figure 3 demonstrate clearly the change occurring after 4 and 8 passes where there are smaller grains than in the as-received condition and after 1 pass. However, the grain size remains reasonably similar between 4 and 8 passes. The corresponding misorientation distributions are shown in Figure 4 and it is apparent there is a decrease in the population of low-angle boundaries with increasing straining and a corresponding increase in the fraction of high-angle boundaries. Again the distributions of the misorientation angles display a bimodal character with peaks at both low and high angles. Furthermore, the measured distribution contains a significant fraction of low angle boundaries even at the highest strain of ~8 after a total of 8 passes. These results are generally in agreement with the trends reported for pure aluminium and other Al-based alloys [7,9,10] where there appears to be a relatively rapid increase in the fraction of high-angle boundaries during the initial passes and a subsequent more gradual increase in the later stages of ECAP processing. In practice, the situation with the Al-7034 alloy may be more complicated because a detailed analysis has shown that the relatively coarse rod-like η ($MgZn_2$) precipitates present in the as-received alloy with length ~0.5 μm are broken into smaller particles during ECAP at 473 K and there is also a fine distribution of Al_3Zr precipitates [11]. The precise influence of these precipitates requires a more detailed investigation. A pole figures analysis demonstrate clearly that the textures changes from a fibre $<111>$x texture in the as-received condition to a $<101>_y$ texture after 1 pass, a $<111>_y$ texture after 4 passes and a $<100>_z$ texture after 8 passes.

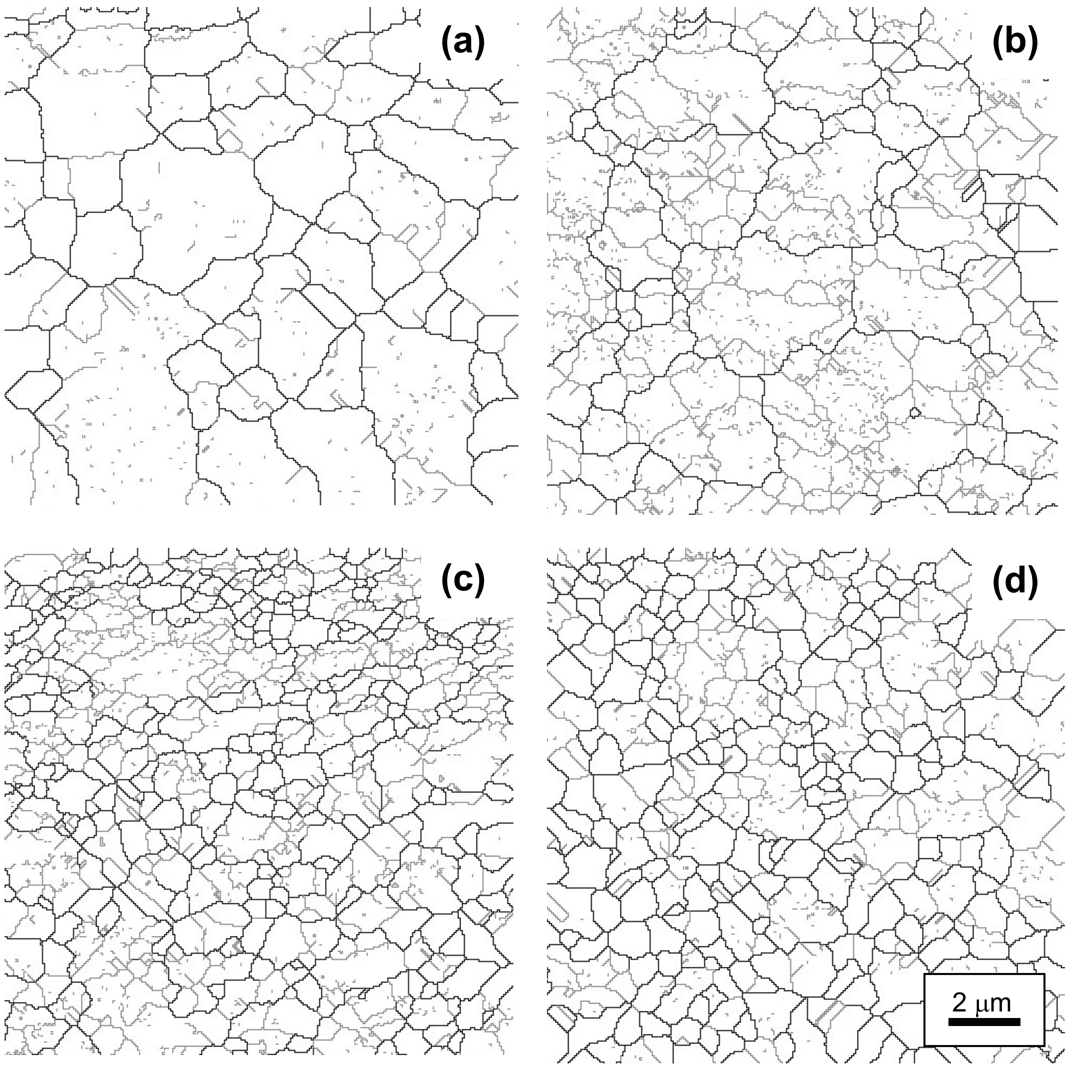

Figure 3: Grain boundary maps of Al-7034 in (a) the as-received condition and after (b) 1, (c) 4 and (d) 8 passes.

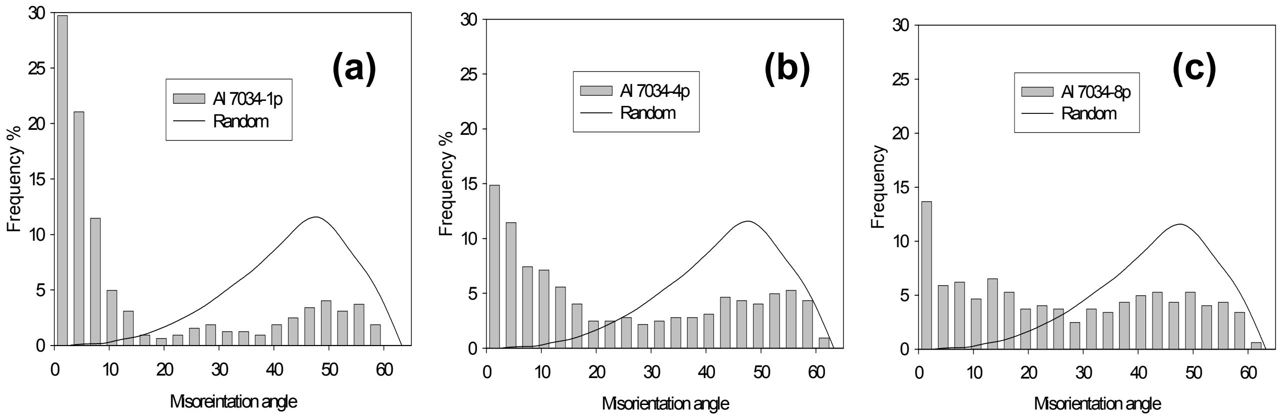

Figure 4: Misorientation distributions for the Al-7034 alloy after (a) 1, (b) 4 and (c) 8 passes of ECAP.

In general, two precipitation sequences can occur in Al-Zn-Mg-Cu alloys with low Cu content [12,13,14]:

$$\alpha_{SS} \rightarrow GP \rightarrow \eta^{'} \rightarrow \eta(Mg(Zn,Al,Cu)_2 \approx MgZn_2)$$
$$\alpha_{SS} \rightarrow S(Al_2CuMg)$$

In these reactions, α_{SS} is the supersaturated solid solution, GP is the Guinier Preston zones, η' is a metastable phase and η and S are the equilibrium phases. Among these phases, η' and η are the primary strengthening precipitates in T6 and T7 7xxx alloys. The DSC curves are shown in Figure 5. Earlier research [12,13] indicates that the main thermal effects (I-V) for the as-received condition are due mainly to GP and/or η' dissolution (I), formation of η strengthening precipitates (II), coarsening of the fine η precipitates (typically 10-50 nm diameter discs [12]) (III), η dissolution (IV) and η melting (V). Note that η precipitates involved in reactions I-III are distinct from the much coarser rod like η particles present in the as-received material. It is apparent from Figure 5 that the ECAP processing at 473 K has caused substantial precipitation leading to the disappearance of effects I and II. Also the small exothermic effect due mainly to coarsening which was observed at about 530 K for a range of peak aged and slightly overaged (T76 and T73 type) 7xxx alloys [12] is absent for the present alloys. This indicates that during ECAP the fine disc shaped η precipitates have substantially coarsened to a state well beyond the state encountered after commercial T7 tempers. Thus ECAP appears to cause a breaking up of the coarse η rods (about 0.5 μm in as received condition) as well as precipitation and coarsening of the finer η precipitates. The curve for 8 passes shows an obvious melting of the η-phase at a temperature of about 753 K but for the as-received condition and the samples taken through 1 and 4 passes it appears that most of the η-phase is dissolved before 753 K. Another interesting feature is that the trough in the η dissolution zone occurs at about 703 K for the as-received samples and after 1 and 4 passes whereas this trough is displaced to 673 K after 8 passes. More experimental work would be needed to explain these differences, but possible sources of these changes could be a contribution of recrystallisation to the heat effects and changes is the distribution and sizes of the rod-like η-phase particles, which would alter their dissolution rates. The similarities in the shapes of the latter stages of the dissolution effects in the DSC curves after 1, 4 and 8 passes demonstrate that the breaking of the rod-like η-phase particles occurs mostly in the first pass of ECAP.

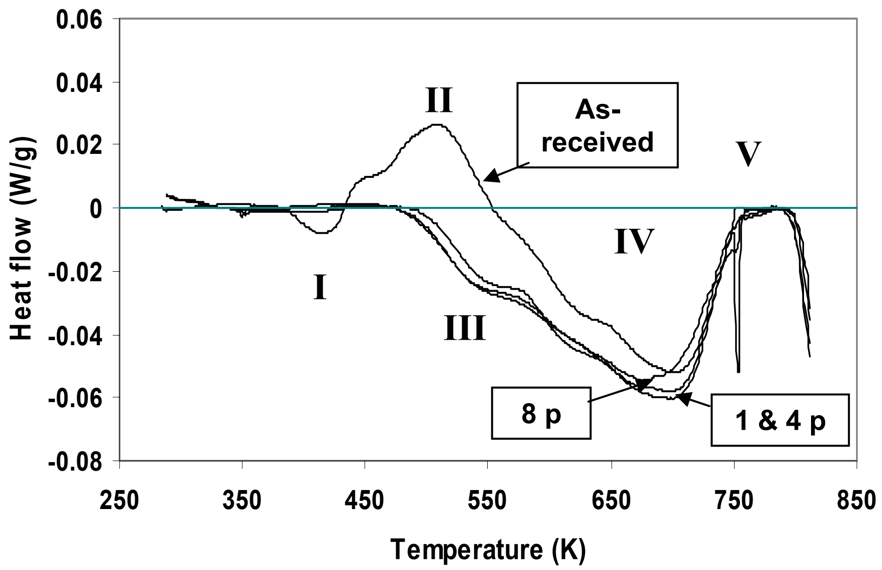

Figure 5: DSC curves for the Al-7034 alloy before and after ECAP processing.

4. Conclusions

The increase of the ECAP passes results in an increase in the fraction of boundaries having high angles of misorientation. For pure aluminium, there is an S texture {122} <634> after 4 passes and a brass texture {011} <211> after 8 passes. For the Al-7034 alloy, the textures changes from a fibre $<111>_x$ texture in the as-received condition to a $<101>_y$ after 1 pass, $<111>_y$ after 4 passes and $<100>_z$ after 8 passes. For the Al-7034 alloy, the DSC analysis identifies the occurrence of several thermal effects during heating involving the formation, coarsening, dissolution and melting of the η-phase.

Acknowledgements

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