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Effect of processing route and second phase particles on grain refinement during equal-channel angular extrusion

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Abstract

The effect of coarse and fine second-phase particles on the formation of ultra-fine grained (UFG) structures have been compared during severe deformation by equal-channel angular extrusion using routes A (no rotation) and B_C (+90° rotation). The presence of coarse particles has been found to increase the rate of grain refinement with route A and the homogeneity of the submicron grain structure formed, but appears less effective using route B_C. In contrast, the presence of fine dispersoids inhibits the development of new high-angle grain boundaries and the formation of an UFG structure with both routes. Retardation is far more pronounced with rotation of the sample and the dispersiod-containing alloy processed by route B_C contained mainly subgrains. The mechanisms operating in each case are discussed.

Keywords: ECAE; Severe deformation; Grain refinement; Particles; Dispersiods; Processing route

1. Introduction

Equal channel angular extrusion (ECAE) is now a well-established method of processing metals to ultra-high strains, and has seen considerable research interest over the last decade (e.g. [1–5]). It has been frequently demonstrated that severe deformation by ECAE to strains of the order of 10 can produce submicron grain structures in metallic alloys [1–5]. Although much research has been conducted on the nature of the grain structures of severely deformed alloys [1–5], and more recently on the evolution of the deformation structure to ultra-high strains [2–5], there is still some debate about the effects of the processing route and the mechanisms of grain refinement. For example, it has been found that rotation of the billet by 90° between each extrusion cycle can increase, or reduce, the level of grain refinement, depending on the die angle, die corner profile and material [2,6–8]. To date, the role of the material variables have not been systematically considered in this context. In previous work it has been shown that, during ECAE processing without rotation of the billet, second phase particles can have a very important effect on the rate and level of grain refinement achieved [9,10]. Coarse micrometer-scale particles were found to accelerate the rate of grain refinement by generating new high angle boundaries within their deformation zones and from particle simulated deformation bands [9], whereas nanometer scale dispersiods can retard the rate of grain refinement by homogenising the slip [10]. Methods of reducing the strain required to form an ultra-fine grained (UFG) material, such as exploiting the use of coarse particles, are clearly of industrial importance, while in many cases fine dispersiods are required in an UFG material to stabilise the grain structure for subsequent applications, such as superplastic forming.

In this paper new results are presented on the effect of coarse particles and fine dispersiods on the level of grain refinement and homogeneity of the sub micrometer grain microstructure that can be produced during ECAE processing with billet rotation, using route B_C (+90°). The results are compared to data, obtained using the same simple model alloys without billet rotation (route A).

2. Experimental

Three alloys: 8079 (Al–1.3 wt.% Fe–0.09 wt.% Si), Al–0.2 wt.% Sc and Al–0.1 wt.% Mg, were deformed by ECAE at room temperature by 15 extrusion cycles to a Von Mises strains

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of ∼10. A die angle of 120° was used with a sharp die corner. Processing was carried out without rotation (Route A) and with rotation by 90° (Route B_C) between each extrusion cycle. Prior to deformation, the 8079 and Al–0.2Sc alloys were heat treated to develop a uniform distribution of second phase particles (see references [9,10]). All the materials had a similar initial grain size of ∼300 μm. The 8079 and Al–0.2Sc alloys contained coarse θ ($Al_{13}Fe_4$) particles and fine coherent Al_3Sc dispersiods, with diameters of ∼2 and ∼20 nm and an interparticle spacings of the order of 10 and 100 nm, respectively. The high-purity single-phase Al–0.1% Mg alloy was used as a control material. The material's deformation structures were characterized using samples taken from the centre of the of the ECAE billet in the ND–ED plane, by high-resolution EBSD using a FEGSEM fitted with HKL technology EBSD system. The spatial and angular resolution of the system were >50 nm and ∼1°, respectively. A low misorientation cut-off of 1.5° was used to minimise misorientation noise. Low angle boundaries (LABs) have been defined as having between 1.5° and 15° misorientations and high angle grain boundaries (HAGBs) greater than 15° misorientation (depicted as black and light grey lines).

3. Results and discussion

3.1. Severe deformation by route A

The microstructures of the three materials processed by route A are shown in EBSD maps in Fig. 1. More detailed descriptions can be found in [2–4,9,10]. Here, their main features are summarised after deformation to an effective strain of ∼10 (15 ECAE passes) for comparison purposes with route B_C. After this ultra-high strain deformation all three alloys mainly contained submicron grains structures with a high fraction of HAGBs (Fig. 1a and b). The grain structures tended to be fibrous, elongated in the extrusion direction, and develop from a 'lamellar' HAGB structure seen at lower strains [2–4]. Overall the grain structures appear reasonably similar, but there are significant differences between the three materials.

Statistical analysis of the percentage HAGB area, mean boundary misorientations and grain sizes are shown in Table 1, from over 5000 grains. The grain sizes have also been measured using the mean linear intercept method parallel and perpendicular to the main direction of HAGB alignment and from the equivalent circular diameter (ECD) after grain reconstruction from the EBSD data. The ECD data has also been used to determine the width of the grain size distributions from the standard deviation normalised with respect to the distribution average. It should be noted that the grain length linear intercept (λ_y) is unreliable with aligned SMG microstructures because the retained longer fibrous grains are frequently bent and do not all align perfectly with the reference frame. Nevertheless, the 8079 alloy, which contains coarse second phase particles, had the most uniform and consistently lowest aspect ratio submicrometer grain structure. It also had the lowest level of retained larger fibrous grains and lowest fraction of low angle boundaries of less than 15° in misorientation (%HAGBs ∼85%; Table 1)

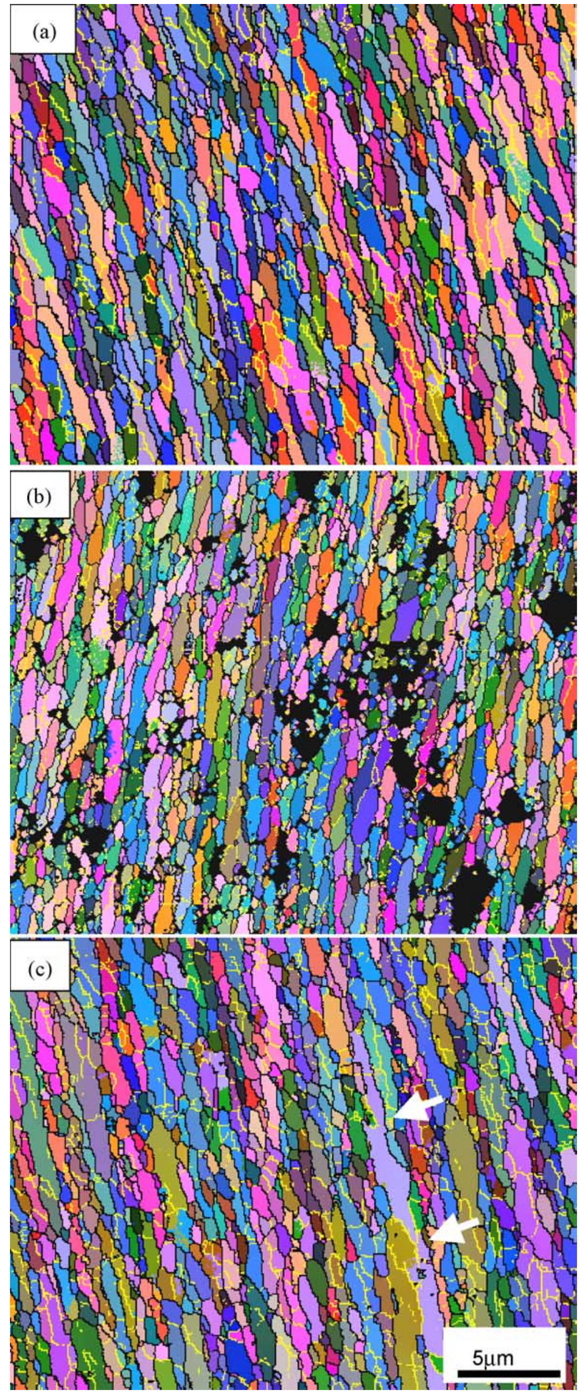

Fig. 1. Example EBSD maps of the three model alloys deformed to an effective strain of 10 by route A: (a) the single phase alloy Al–0.13% Mg, (b) the coarse particle containing 8079 and (c) the Al–0.2% Sc alloy with fine dispersoids.

as well as the highest average boundary misorientation. In comparison, the dispersiod-containing alloy had the most retained fibrous grain fragments (arrowed), lowest fraction of HAGBs (64%; Table 1), and the largest grain size, while the single-phase alloy lay somewhere in between (74% HAGBs). The dispersiod-containing alloy also had the widest grain size distribution, reflecting the higher frequency of larger retained fibrous grain fragments.