Fine-Grained Structure Formation in Al- Mg- Sc Alloy during Hot ECAP

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Abstract. Grain refinement taking place during equal channel angular pressing (ECAP) was studied in a commercial Al-6%Mg-0.4%Mn-0.3%Sc alloy at a temperature of 450°C. Inhomogeneous deformation occurring during hot ECAP of the present alloy leads to formation of deformation bands. Repeated ECAP results in mutual crossing and increase in number and misorientation of deformation bands, followed by transformation of boundaries of deformation bands into high angle boundaries. As a result, a new fine-grained microstructure with an average crystallite size of 2.8 μm develops at large strains above 8. It is concluded that grain refinement occurs in accordance with deformation-induced continuous reactions; that is essentially similar to continuous dynamic recrystallization. The mechanisms of new grain evolution, as well as factors promoting grain refinement, are discussed in detail.

Introduction

There have been number of works to date dealt with the studies of evolution of (ultra)fine grain microstructures in Al-based alloys during equal-channel angular pressing (ECAP) [e.g. 1-5]. For cold to warm ECAP, it is widely accepted that highly strained Al alloys contain a major fraction of low-to-large angle subboundaries; the misorientation angle of such strain-induced boundaries can increase with strain increasing [1-3,5]. It was presumed that grain refinement during ECAP can take place by the operation of a form of continuous dynamic recrystallization (cDRX), which generally involves the following processes; i.e. the formation of arrays of low angle boundaries and a gradual increase in the boundary misorientations during deformation, finally leading to new grain development at high strain [6,7]. At the same time, little is known about the grain refinement, which occurs during high-temperature ECAP. While some experimental evidence of new fine grain evolution during warm to hot ECAP has been presented and discussed for aluminiums in the recent publications [2-5], the *exact mechanisms,* by which these grains evolved, have not been, however, developed due to an insufficiency of the related experimental data.

The aim of the present research was to study the microstructural evolution in a commercial coarse-grained Al–6%Mg–0.3%Sc alloy subjected to hot ECAP. The starting material was a warm-extruded rod with elongated grains having a stable (sub)grain structure with low-to-moderate angle misorientation resulted from previous working and annealing operations. These crystallites were expected to play a role in the initiation of new grain formation during subsequent severe deformation [6,7]. Specific attention was paid to elucidate main factors promoting grain refinement in this alloy under ECAP conditions and to discuss the mechanism of grain formation in detail.

Experimental Procedure

The alloy used had the following chemical composition; Al–6%Mg–0.4%Mn-0.3%Sc (in mass pct). It was fabricated by casting into a steel mold at the Kamensk-Uralsk Metallurgical Works (Russia), and then homogenized at 520°C for 48 hrs. Extrusion was performed at 390°C to a strain of about 0.7, followed by annealing at 400°C for 1h. Samples for ECAP were machined parallel to the extrusion axis into rods with a diameter of 20 mm and a length of around 100 mm. ECAP was carried out repeatedly at 450°C by using route A up to a strain of 12 with a strain of about 1 in each passage through the die. The samples pressed were quenched in water after each deformation pass. Deformed microstructures were examined in the central regions of a cross-section parallel to the pressing direction. The metallographic analysis was carried out using a Neophot-32 microscope and an Epiquant automatic structure analyzer. (Sub)grain boundary misorientation distributions were obtained from electron back scattering diffraction pattern (EBSP) by using a LEO1530 scanning electron microscope (SEM). Thin samples for transmission electron microscopy (TEM) were examined with a JEOL-2000FX TEM.

Results

In the initial state, the alloy was composed of coarse elongated grains lying parallel to the extrusion axis. The grain size varies from 150 to 200 μm and from 50 to 100 μm in longitudinal and transverse directions, respectively. A fine grain structure with a volume fraction of around 0.35 and the average grain size of around 4 μm was presented in the mantle regions. On a mesoscale, the alloy contained a high density of dislocation subboundaries with an average misorientation angle of around 5°. The size of the crystallites fragmented by these boundaries was in the range of 3 to 30 μm. Two different types of dispersion particles were identified by TEM as coherent Al_3Sc dispersoids having an average size of about 20 nm, and incoherent Al_6Mn precipitates having a size of about 200 nm.

A series of typical microstructures (a) just before ECAP ($\varepsilon = 0$) and (b) - (d) evolved during ECAP to $\varepsilon = 1$ to 12 is represented in Fig. 1. It is seen that during hot ECAP, original elongated grains presented in initial structure (Fig. 1(a)) are pancaked along the pressing direction (EA). Concurrently, new fine grains are frequently evolved in some parts of grain interiors, which are mainly adjacent to the mantle regions and so, well-defined necklace-like structure composed of portions of original grains and areas of fine grains, can be clearly detected during the earlier stages of deformation (Fig. 1(b)). With further ECAP, areas of such new fine grains progressively propagate from mantle regions to coarse grain interiors, as shown in Figs. 1(b) – (d). At high strains, the regions of fine grains and remnant original grains are distributed more homogeneously. At a strain of 12 (Fig. 1d), initial coarse-grained microstructure is almost fully replaced by new fine-grained one, although relatively coarse fragments of original grains are still noticed. Also, while the microstructure is comprised mainly by fine grains,

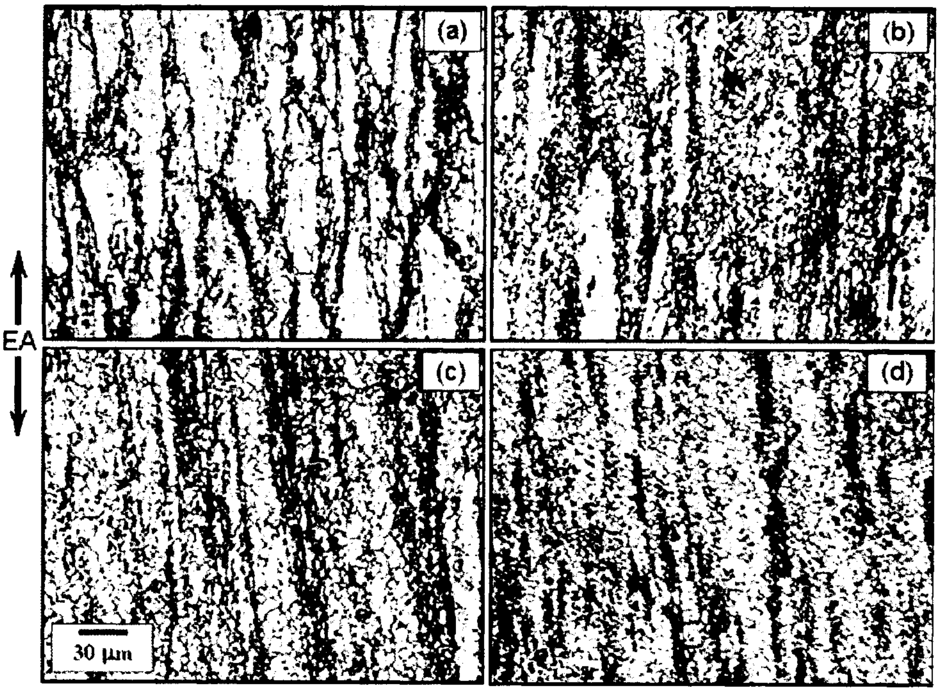

Fig. 1 Typical microstructures evolved during ECAP to various strains: (a) initial; (b)$\varepsilon = 1$; (c) $\varepsilon = 4$; (d) $\varepsilon = 12$. Hereafter, EA is the extrusion axis.

clusters of predominantly large crystallites sometimes persist in the newly evolved regions.

The typical structures observed by orientation imaging microscopy (OIM) at $\varepsilon = 0$ to 2 are represented in Fig. 2. Here the different grayscale levels indicate the different crystallographic orientations and the misorientation angles (Θ) between neighboring grid points, $\Theta > 2^\circ$, $\Theta > 4^\circ$ and $\Theta > 15^\circ$ are marked by the thin white, narrow gray and bold black lines, respectively. It is seen in Fig. 2 that new fine grains and dislocation boundaries with low- to medium angle misorientations are evolved in the mantle regions and in grain interiors, respectively. It is noteworthy that new dislocation boundaries with moderate and even high angle misorientations are developed and intersected by each other after first pass through the die (Fig. 2(b)) frequently in the regions adjacent to fine-grained matrix and occasionally in the middle parts of original grains. The crystal orientation is frequently changed in the regions fragmented by such boundaries. These mutual crossing boundaries can subdivide original grains into small separate misoriented domains, some of which are transformed into new fine grains.

Figs. 3(a)-(c) represent the distribution of typical point-to-point ($\Delta\Theta$) misorientations developed along the lines T_1, T_2 and T_3 indicated in Figs. 2 (a) - (c), respectively. The value of $\Delta\Theta$ defines the relative difference in crystal orientation between two neighboring scan points with step of 0.3 μm. The length of lines T was taken to be commensurable with original grain size. This allows detecting a misorientation distribution in a whole coarse original grain. It can be seen that $\Delta\Theta$ does not exceed generally 2-3° except some spots with $\Delta\Theta \geq 5^\circ$ to 15°, which correspond to deformation-induced new boundaries. This indicates that highly heterogeneous deformation takes place within initial grains, leading to local lattice rotation and then formation of dislocation subboundaries. The latter may correspond to those of deformation bands and/or geometrically necessary boundaries (GNBs), which are able to be introduced into an Al alloy even under hot deformation conditions, as discussed in detail elsewhere [8]. It is important to note that such boundaries located near mantle regions have a relatively large misorientation and their density significantly increases by ECAP processing. Fig. 3(c) also shows the variation of $\Delta\Theta$ developed in a typical grain deformed to $\varepsilon = 2$. It can be seen that the $\Delta\Theta$ for some dislocation subboundaries rises up to values, which correspond to high-angle misorientation, and new GNBs are evolved by repeated ECAP from $\varepsilon = 1$ to 2.

Fig. 4 represents changes in the misorientation distributions of deformation–induced dislocation boundaries derived from OIM analysis. It is seen in Figs. 4 (a) and (b) that most of the boundaries developed in the initial microstructure just before hot ECAP and after the first pass exhibit low-to-medium angle misorientations from 5 to 15°. With further deformation, the fraction of low and moderate angle boundaries rapidly decreases and that of high-angle ones conversely increases in

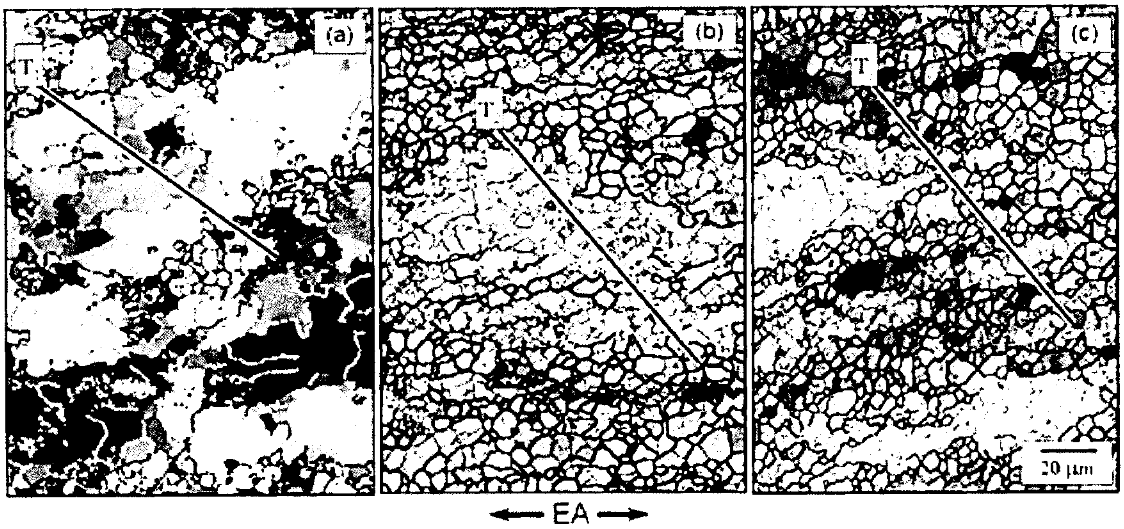

Fig. 2 Typical OIM maps of Al-Mg-Sc alloy: (a) initial; (b) $\varepsilon = 1$; (c) $\varepsilon = 2$.

fine-grained regions (Figs. 4(c)-(d)). Noteworthy that the average misorientation of 27.7° in Fig. 4(d) at $\varepsilon = 12$ is less than that of 40.7°, predicted by Mackenzie for randomly misoriented polycrystalline aggregates of cubic metals [9]. This may be attributed to the strain-induced microstructure evolved through grain fragmentation, in which a fraction of low-to-medium angle boundaries is always present (Fig. 4).

Strain dependencies of (a) the average grain size, d_{rex}, (b) the volume fraction, V_{rex}, and the average misorientation, Θ_{ave}, in the regions of newly developed grains are depicted in Fig. 5. The d_{rex} gradually drops from around 5.5 μm at $\varepsilon = 1$ and approaches a constant value of about 2.8 μm at $\varepsilon \geq 8$. Both Θ_{ave} and V_{rex} values start to increase from 13.7° and 0.35, respectively, just before ECAP, and rise at almost constant rates against strain at $\varepsilon \geq 4$, and approach to around 28° and 0.85 at higher strains.

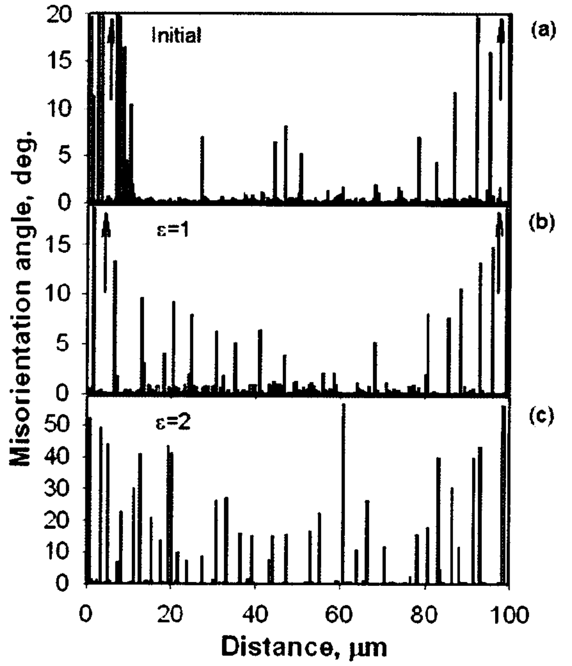

Fig. 3 Point - to - point misorientations, $\Delta\theta$, measured along the lines (a) T_1; (b) T_2 and (c) T_3 marked in Figs. 2(a); 2(b) and 2(c), respectively.

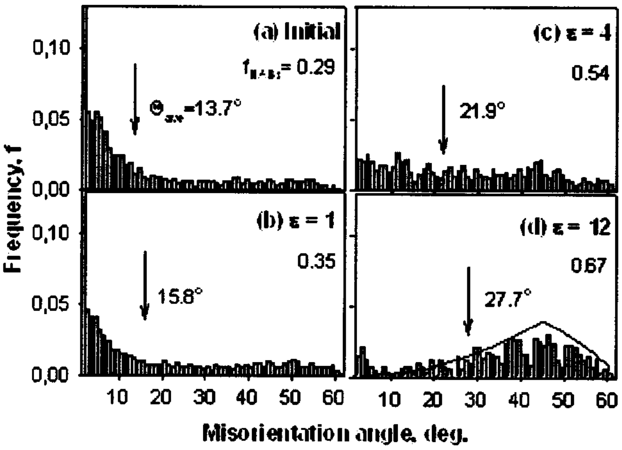

Fig. 4 Changes in misorientation distribution of dislocation and (sub)grain boundaries developed with straining by ECAP. (a) Initial, (b) $\varepsilon = 1$. (c) $\varepsilon = 4$ and (d) $\varepsilon = 12$ show the data for fine-grained structure. The solid line in (d) indicates the random misorientation distribution evaluated by Mackenzie [9].

Discussion

The results mentioned above show that new grains are mainly developed due to grain fragmentation process accompanied by frequent evolution of deformation-induced subboundaries with low to moderate angle misorientation, which can be recognized as the boundaries of deformation bands and/or GNBs [10]. It was recently reported [3,4,8,11,12] that under severe plastic deformation conditions at low to high temperatures, original grains can be subdivided by deformation bands, followed by evolution of fine crystallite components at high strain, and such a process can play an important role in grain refinement. It has been also pointed out in [4,5] that inhomogeneous deformation characteristics of ECAP in itself can lead to formation of deformation bands. They are considered to be developed by relaxation of strain gradients resulting from heterogeneous strains introduced by ECAP. A mechanism of grain refinement can be summarized as follows [4]. Repeated ECAP results in an increase in the number and misorientation of the boundaries of deformation bands. Their mutual crossing leads to continuous fragmentation of coarse grains into misoriented domains.

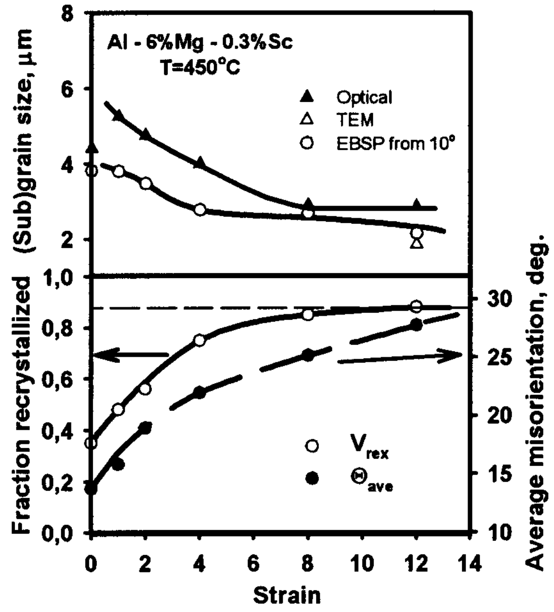

Fig. 5 Effect of hot ECAP on (a) the average (sub)grain size, d_{rex}, and (b) the fraction of fine grains, V_{rex}; and the average misorientation of dislocation/(sub)grain boundaries in fine-grain structure evolved, Θ_{ave}.

The boundary misorientations of these domains grow rapidly with increasing strain, followed by their transformation into new grains surrounded by high-angle boundaries (HABs).

The same discussion can be applied to the structural mechanism, which operates during hot ECAP of the current alloy. It should be noted, however, in this connection, that heterogeneous deformation introduced by ECAP should decrease with increasing temperature [3,5]. Dislocation motion occurs more homogeneously at elevated temperatures and, even if any strain gradients could be formed on a mesoscale, they should rapidly disappear due to frequent operation of some relaxation processes, such as grain boundary sliding (GBS) and/or dynamic recovery [6,8]. As a result, deformation bands following high strain gradients can scarcely develop under hot ECAP conditions of conventional Al alloys with equiaxed grain structures [e.g.3]. It has been shown in [8], however, that highly inhomogeneous deformation could be sometimes introduced in a coarse grain interior of an Al alloy with a non-uniform and/or unsymmetrical grain structure even during hot deformation due to inhomogeneous operation of GBS, so as to collectively maintain the compatibility with surrounding grains. It should be remembered in Figs. 1(a) and 2(a) that the current Al-Mg-Sc alloy has the coarse elongated grains in initial structure with a fraction of fine grains, which are inhomogeneously distributed along the boundaries of the coarse grains. This fine-grained structure can readily support GBS in the present deformation conditions (i.e. especially at T = 450°C), as it was well documented for the same alloy in [13]. At the same time, GBS can hardly operate along the boundaries of coarse and elongated original grains. Plastic flow, therefore, can be localized mainly in fine-grained mantle regions. As a result, high strain gradients should be introduced in the columnar grains, followed by development of deformation bands even at early stages of hot deformation[1]. Repeated ECAP can promote development of such boundaries in various directions [4]. They are continuously formed by strain accumulation and microstructural heterogeneities, which evolve in each pass, accompanying with their intersection in grain interiors. In this way, the original fragmented substructure containing GNBs (Figs. 2(a), 3(a)) is very much suitable for development of new fine grains [7]. It can be seen in Figs. 1 - 5 that the new grain structure in the current alloy starts to rapidly develop just after first pass through the die. In contrast, such strain-induced HABs formation in as-cast and/or well-annealed Al alloys is usually retarded up to $\varepsilon_c \geq 2$ [5]. The medium-to-high angle boundaries in initial structure may be resulted from deformation bands developed by prior-warm extrusion and exist stably during high-temperature exposure just before hot deformation. They can be frequently intersected with deformation bands produced at the earlier stages of ECAP. This results in the rapid evolution of new equiaxed crystallites

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[1] This can be further enhanced by the presence of transition metal alloying elements such as Sc and Mn. The high fractions of nanoscale dispersoids phases Al_6Mn and mostly Al_3Sc formed in the present alloy may be effective in dislocation pinning and so retard or prevent any relaxation of strain gradients. This is considered to be another factor promoting the evolution of deformation bands under high temperature deformation conditions [8].

with a stable grain size and therefore significantly accelerates kinetics of grain refinement [12].

Transformation of low-angle boundaries into large-angle ones also may be attributed to GBS, which can frequently take place in new fine-grained regions [13]. In such regions, GBS occurs first near initial grain boundaries and subsequently along all boundaries of deformation bands. The progressive rotation of the crystallites surrounded by high angle- and low angle- boundaries, caused by GBS, can promote an increase in the misorientation and their rapid conversion into high angle ones [7], resulting in dynamic evolution of fine grain structure. The formation of new grains may occur in the same way as an *in-situ* dynamic recrystallization by progressive lattice rotation [6]. It should also be noted in Figs. 4 and 5 that strain dependencies of microstructural parameters, developed during such grain refinement process can be considered to be a specific feature of strain–induced grain structures, which is typical of cDRX [3-8,11-12]. It can be concluded therefore that new grains evolved during hot ECAP of the present alloy, resulted from a series of strain-induced continuous reactions; this is essentially similar to cDRX. In this connection, it may be interesting to note that in the present alloy, the newly evolved granular structure is developed heterogeneously during cDRX, making up the mantle of fine grains (Figs. 1 and 2). This is because the highest strain gradients can be introduced through GBS in the grain boundary- (and/or near-mantle-) regions compared to those in grain interiors [11]. As the result, deformation bands with large misorientation are readily developed at the earlier stages of deformation in these regions (Fig. 3), leading to more rapid evolution of new grains. Such structural behaviour of cDRX looks similar to that of the "necklace" dynamic recrystallization, which takes place in low stacking fault energy materials during hot working [e.g. 6,11], although their physical mechanisms are completely different.

Summary

Microstructural evolution in a commercial as-extruded Al-6%Mg-0.4%Mn-0.3%Sc alloy subjected to ECAP at T= 450°C was examined in the present work. The main results can be summarized as follows:

(1) ECAP results in a considerable grain refinement. A new fine-grained microstructure with an average crystallite size of about 2.8 μm develops at large strains above 8. The new grains are evolved first in the mantle region and propagated into the original grain interiors with further straining.

(2) Inhomogeneous deformation occurring during hot ECAP of the present alloy leads to formation of deformation bands. Repeated ECAP results in mutual crossing, increase in number and misorientation of deformation bands.

(3) The processes in (2) lead to transformation of boundaries of deformation bands into high angle ones and evolution of new fine grains at high strains. It is concluded that grain refinement takes place by a deformation-induced continuous reaction; that is essentially similar to cDRX.

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