Grain Refinement in a Commercial Al-Mg-Sc-Zr Alloy during Hot ECAP

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Abstract. The microstructure evolution of an as-cast commercial Al-Mg-Sc-Zr alloy during Equal-Channel Angular Pressing (ECAP) at 325°C was investigated. In the early stages of deformation strain induced boundaries were created within the initial coarse grains and constitute the deformation bands. Repeated ECAP led to an increase of the number and misorientation of deformation bands. Further straining up to e~8 resulted in the formation of a new fine-grained structure with an average crystallite size of 1.2 μm. It is concluded that the progressive increase of the misorientation of deformation induced boundaries is the main mechanism of structure formation under high temperature ECAP.

Introduction

Commercial Al alloys with fine- and ultra fine-grained structures are considered as advanced structural and functional materials, since they exhibit unusual mechanical and physical properties. Equal-channel angular pressing (ECAP) is a common technique of severe plastic deformation to produce sub-micron and nano-scale grain structures [1].

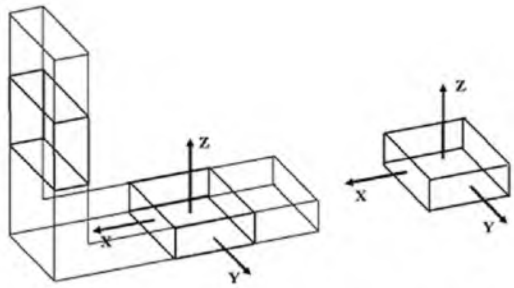

Fig. 1 Used coordinate system of a specimen during and after ECAP.

Numerous reports deal with the investigation of the final microstructure and texture in aluminium alloys after ECAP [2-7]. However, only a few studies have addressed the microstructure evolution that results in grain refinement during intense plastic deformation (IPD), in particular, during high-temperature ECAP [e.g. 2,3,5]. It is established that under dynamic deformation conditions grain refinement occurs by microstructural processes, which are often associated with continuous dynamic recrystallization (CDRX) [2,3,5,10,12]. However, the formation of high angle boundaries (HABs) during CDRX can occur by several mechanisms, for instance, migration of subboundaries or (sub) grain growth, absorption of dislocations into subboundaries, and (sub) grain rotation [10]. The exact mechanism of the formation of new grains during warm-to-hot ECAP is not fully understood. In the current study, microstructure and texture evolution of a commercial Al-Mg-Sc-Zr alloy, hereafter denoted as *1570c Al alloy*, were investigated during high-temperature ECAP. The mechanism of the formation of a new ultra-fine grain and the relation to the microstructure and texture development after various ECAP passes was analysed and will be discussed in detail. Besides, this alloy is an advanced structural material for the automotive and aviation industry and, hence, the evaluation of its potential for grain refinement by means of IPD seems to be very important for commercial application.

Various processing routes including sample rotation between each pass can be used for ECAP. In earlier investigations [8] it was reported that a rotation around the Z axis could be applied to plate-type samples in contrast to conventional ECAP rod samples (Fig.1). This allows two additional processing routes. Route D, where the plate is rotated by 90° around the Z axis after each pass, is a relatively new method used in the current work. In addition, the samples were also rotated by 180° about the extrusion axis (X axis). For this, the top and bottom sides of the plates were repeatedly changed from pass to pass, in order to accomplish a more uniform microstructure formation.

Experimental Procedure

A commercial aluminium alloy with a chemical composition of Al–5%Mg–0.18%Mn–0.2%Sc–0.08%Zr–0.002%Be (in weight %) was used as a starting material. The alloy was cast into a steel mold to form an ingot with the dimensions of 90x220x220 mm^3. The ingot was homogenized in air at 350°C for 6 h. and then sectioned into plates with the dimensions of 125x125x25 mm^3. These plates were subjected to ECAP processing in air at 325°C using a die with a rectangular internal cross-section. The channel had an L-shape configuration with an angle 90° between the two channels. Deformation through this angle produced a strain of about ε~1 during each passage through the die. The processing speed was approximately 6 mm/s. The specimens were deformed to strains of ~1, ~3, ~6, ~8. For The samples for the analyses were machined from the centre parts of the pressed plates. For comparison, the microstructure was also investigated in two mutually perpendicular planes in longitudinal and transverse sections. The (sub) grain boundary misorientation distributions were obtained by EBSD using a LEO1530 scanning electron microscope (SEM) operating at 20kV. X-ray texture measurements were performed with Co Kα radiation in back reflection mode.

Results

The typical original microstructure of the investigated alloy after solution treatment is described in details elsewhere [11]. In the initial state, the alloy was composed of equiaxed grains with an average size ~24 μm. These grains had almost no dislocation substructures (see Fig.3 (a)). The fraction of HABs and the average misorientation angle in this structure were 0.94 and 40.9°, respectively.

The microstructural evolution during ECAP in longitudinal and transverse sections of the deformed plates is presented in Fig. 2. Fig. 3 shows the typical OIM map of the original microstructure (a) and the enlarged views (b,c) of Fig. 1 (a) and (b), respectively. The different greyscale levels indicate the different crystallographic orientations, and the boundaries with misorientation θ>2°, θ>5° and θ>15° are marked by thin, bold white and bold black lines, respectively. The structural changes during ECAP are characterized by a rapid development of a substructure, which takes place within the initial coarse grains during the early stages of deformation. Subboundaries are created already during the first ECAP pass. These deformation-induced boundaries have a low- and medium misorientation, and some of them constitute deformation bands inside the initial grains. At ε~3, the density of deformation bands has increased and occurs in various directions due to the rotation of the sample between ECAP passes. The mutual intersection of the deformation bands leads to a fragmentation of the original coarse grains into small misoriented domains. Some of them are transformed into new fine grains, frequently along the initial high angle boundaries. With further deformation, the fraction of new fine grains rapidly increased. They propagated from grain boundary regions to the coarse grain interiors resulting in a more homogeneous microstructure. However, some parts of the original grains still remained even at high ECAP strains. At ε~8, their fraction was about 0.2.

Fig. 3 shows the typical point-to-point misorientations ($\Delta\theta$) developed along the indicated lines L_1.and L_2. These misorientations define a relative difference of a crystal orientation between two neighbouring scan points with a step size of 0.2 μm. It can be seen (Fig. 3 (a)) that the misorientation generally does not exceed 2°.

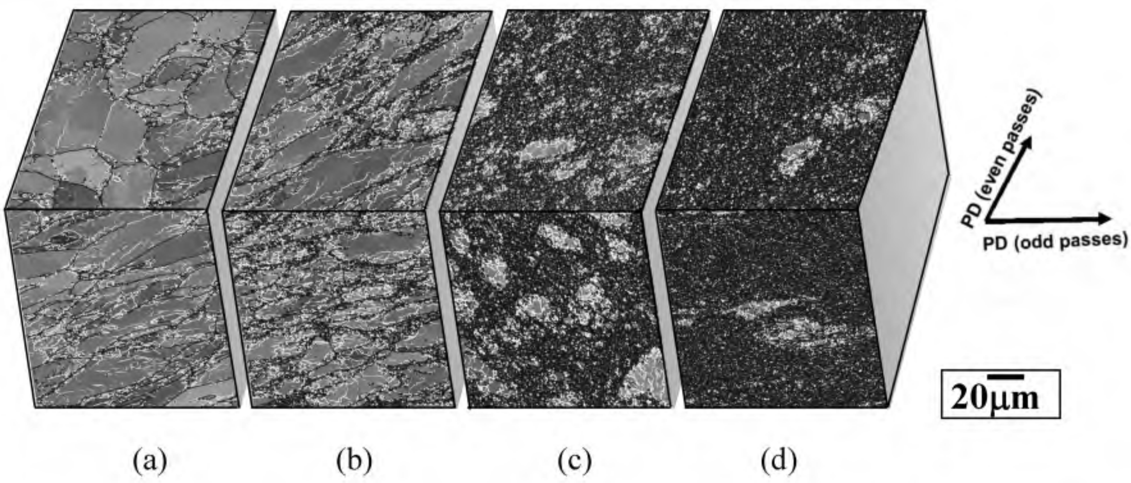

Fig. 2 The typical microstructure developed in the 1570c alloy after ECAP in the different planes: (a) ε~1; (b) ε~3; (c) ε~6; (d) ε~8. PD means the pressing direction.

Besides, subboundaries with medium angle misorientation from 5 to 10° developed in the initial grain interior at a strain of ε~1, which may correspond to the boundaries of deformation bands, mentioned in Fig. 2 [2,3]. With further deformation to ε~3, some of these boundaries convert to large misorientation beyond 10-15° i.e. transform into HABs (Fig. 3 (b)). This suggests that, along with the development of deformation bands surrounded by medium angle boundaries, new fine (sub) grains with high misorientation close to 40° were created by warm-to-hot ECAP.

Misorientation distributions for the samples deformed to different ECAP strains are presented in Fig. 4. At the early stages of deformation, many boundaries with low and medium misorientation were introduced by ECAP deformation of the original as-cast structure. As a result, the fraction of HABs decreased after the first pass, and the misorientation distribution at moderate-to-high strains was bimodal with two peaks corresponding to low-angle boundaries (LABs) and HABs. This is directly connected with the development of deformation bands at the earlier stages of ECAP. With further straining the fraction of LABs decreased, and the average misorientation angle grew due to an increasing fraction of new fine grains caused by the transformation of LABs into HABs.

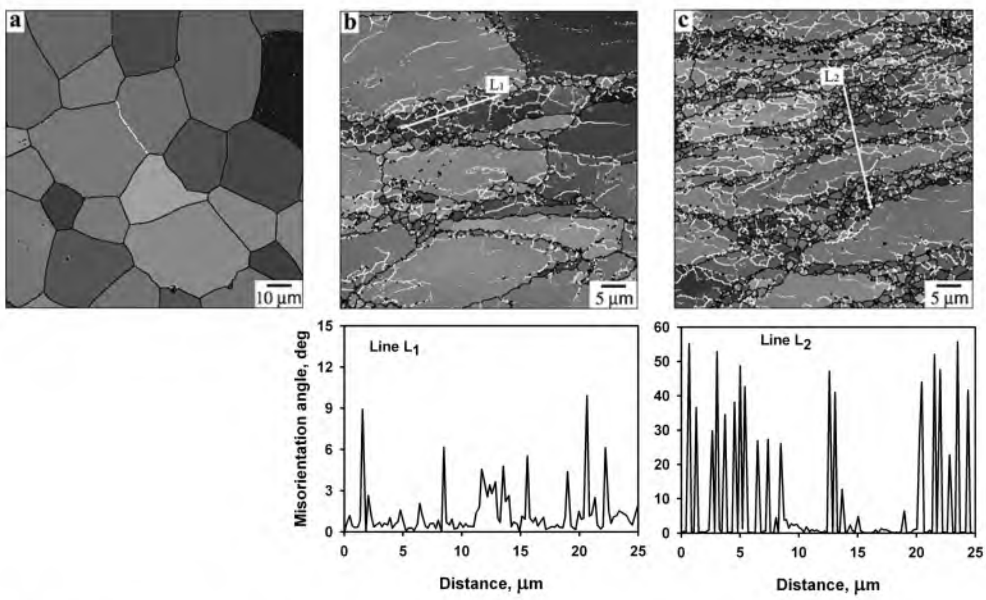

Fig. 3 Typical EBSD maps of the 1570c alloy: (a) initial state; (b) ε~1; (c) ε~3 and point-to-point misorientations measured along the marked lines L_x.

Fig. 5 shows the evolution during ECAP of (a) the mean grain size (d_{rex}), (b) the fraction of HABs (V_{HABs}), and (c) the average misorientation angle (θ_{ave}), derived from the EBSD analysis. The angular characteristics of the new fine-grained structure were measured and analysed separately in the transverse (Z) plane and in the longitudinal (Y) plane that contained "the shear component" of ECAP deformation[1]. According to Fig. 5 (a) the size of new (sub)grains gradually decreased with increasing strain from about 2.2 μm at ε=1 to about 1.2 μm at ε=6 but did not change at strains larger than ε~6. The fraction of HABs and the average misorientation angle did not change significantly during the early stages of deformation but then progressively increased with further ECAP to about 0.86 and 38^o, respectively. It is also interesting to note that, after the first pass through the die, both values, V_{HABs} and θ_{ave}, for the "shear" plane, were remarkably lower then those measured in the Z plane. However no significant difference in these parameters remained at larger strains.

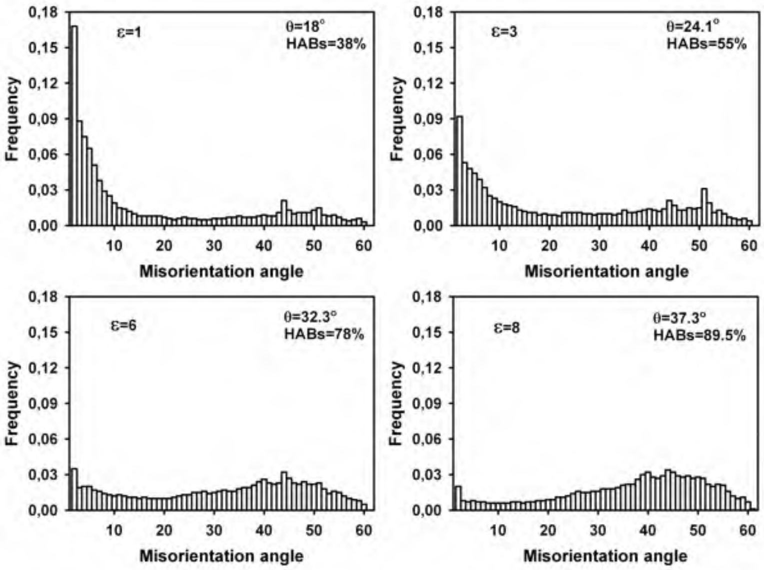

Fig. 4 The change in misorientation distribution with increasing of strain.

The measured {111} pole figures for the deformed specimens are shown in Fig. 6. It is seen that the texture strengthened during the first three ECAP passes. The maximum texture intensity increased from 4.38 for ε~1 to 6.65 for ε~3. However, with further deformation, the texture weakened, and the texture maximum decreased to 1.47 at ε~8.

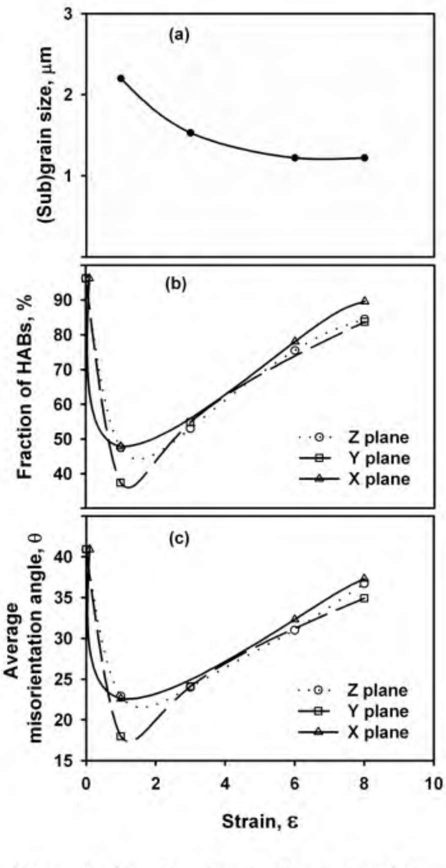

Fig. 5 Strain dependency of a (sub) grain size (a), fraction of HABs (b) and average misorientation angle (c).

Discussion

The observations described above suggest that the formation of a new ultrafine-grained microstructure with average crystallite size about 1.2 μm in Al alloy 1570c is the result of a continuous microstructure evolution during hot ECAP. The inhomogeneous deformation introduced by ECAP results in the formation of deformation bands. The process of grain fragmentation starting at the early stages of deformation leads to the development of a substructure with low-to-medium angle misorientation within the initial coarse grains. Due to the sample rotation between passes in route D, they developed in various directions and intersected each other.

[1] In each ECAP pass the planes X and Y were defined as the transverse and longitudinal ones that were perpendicular and parallel, respectively, to the last pressing direction (see Fig.1).

The intersection of new and prior deformation bands led to the fragmentation of initially coarse grains into small misoriented domains. The boundary misorientation of these domains grew rapidly with increasing strain to eventually create new grains surrounded by HABs.

A similar formation of a new fine-grained structure was recently observed during IPD of Al alloys at different temperatures (0.5-0.6 T_m) [2-5,10,12]. It was reported that, under severe plastic deformation conditions, deformation bands subdivided the original grains and fine crystallites formed at high strains. However, such structural changes during ECAP of Al alloys were mostly investigated at ambient temperature and, less frequently, at moderate temperatures below 250°C. Only few studies addressed high-temperature ECAP. In the following, we will discuss, therefore, some peculiarities of the structure and texture evolution, which take place during ECAP at warm-to-hot deformation conditions, i.e. at 325°C (~0.65T_m). Deformation bands have been reported for cold and warm (0.4 T_m) deformation [3,5]. It is well known that with increasing temperature slip becomes more homogeneous [12]. Strain gradients are likely to disappear due to intensive dynamic recovery. This leads to the development of strain-induced boundaries such as deformation bands in the conventional Al alloys. By contrast, the Al alloy 1570c contains Sc, Zr and Mn additions. The high fraction of nanoscale dispersoids Al_6Mn, Al_3Zr and Al_3Sc stabilizes the dislocation structure and prevents rectystallization. This promotes the formation of subboundaries and their further conversion into HABs and the development of a fine-grained structure even at elevated deformation temperatures. On the other hand, an increasing temperature may be very important for the rise in misorientation of the deformation-induced subboundaries. It has been reported recently [13] that a strong pinning of dislocations by particles in a dispersoid-containing alloy with uniform spatial distribution of fine particles causes a more diffuse dislocation arrangement during ECAP at ambient temperature and suppresses their transformation into HABs. A high deformation temperature allows the lattice dislocations to rearrange within the boundaries of the deformation bands to convert them into stable subboundaries which rising misorientation upon further straining.

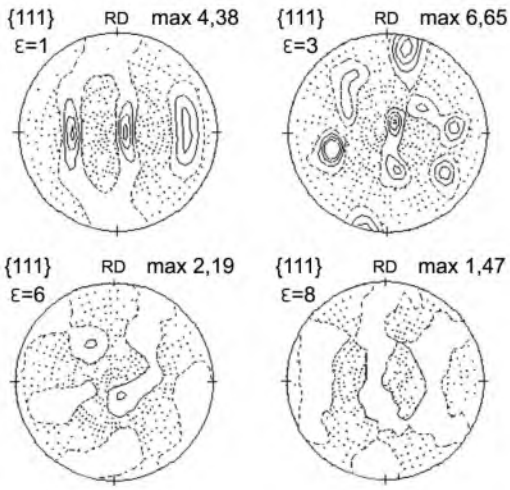

Fig. 6 Measured {111} pole figures for deformed specimens

It is interesting to note in Figs. 4 (b) and (c) that a difference in V_{HABs} and θ_{ave} in the X, Y and Z planes of ECAP appears only after the first ECAP pass and then vanishes during further deformation. By contrast, during room-temperature ECAP such differences may persist through high strains as reported for pure Al subjected to all main ECAP routes, i.e. A, B, and C [14]. This is not surprising, because it is known that more prominent substructures should always be introduced in the shear plane of ECAP. It seems that, except for the influence of route D on texture evolution, when the shear plane is repeatedly changed, the difference of microstructural evolution for cold and hot ECAP may also be attributed to the enhanced thermal activation of the deformation mechanisms at high temperature, e.g. diffusional processes and grain boundary sliding (GBS).

Randomization of the texture in the fine-grained regions during warm-to-hot ECAP (Fig.5) indicated that the transformation of LABs into HABs may be assisted by (sub) grain rotation caused by grain boundary sliding during hot deformation. The reported enhanced superplastic properties of the investigated alloy, achieved in a wide temperature interval at high strain rates [11], support this assumption. It is well known that superplastic deformation randomizes the deformation texture [15]. The subdivision of the original grains may cause the generated domains to rotate to different terminal orientations. As a result, high angle boundaries between adjacent crystallites will be created [9]. A detailed model for the dynamic evolution of a new fine-grained structure by (sub) grain rotation and GBS has been given elsewhere [10]. GBS is likely to occur first near the initial grain boundaries and subsequently, along deformation bands with HABs, leading to progressive (sub) grain rotation and finally to the development of new fine grains at high strains. Thus, several mechanisms, including the rearrangement of lattice dislocations and rotation of (sub) grains need to take place during microstructure evolution to cause the misorientation of deformation-induced boundaries.

Generally, it can be concluded that the formation of new fine grains occurs by a microstructure development similar to CDRX. At the early stages of deformation, the evolution of a new structure occurs by formation and mutual intersection of deformation bands. The created fine crystallites can rotate relative to each other, which promotes the increase of their misorientation. With increasing strain, GBS leads to a further rotation of neighbouring (sub) grains and a rapid conversation of low-angle boundaries into HABs.

Summary

The microstructure evolution of the as-cast commercial Al-Mg-Sc-Zr alloy deformed by ECAP via route D at 325°C up to 8 passes was analyzed. The main results can be summarized as follows:

1. New fine grains with equiaxed shape and an average size of 1.2 μm developed during hot ECAP. These grains occurred first close to the original grain boundaries and propagated into the grain interiors on further straining. A fraction of original coarse grains ~ 20% remained up to a strain of $\varepsilon\sim8$.
2. All experimental data confirm that the evolution of the microstructure is akin to continuous dynamic recrystallization. Under hot deformation conditions, the initial slip deformation appears to be followed by grain boundary sliding (GBS) and respective (sub) grain rotation. This leads to a large mean misorientation and a randomization of texture.
3. The retardation of grain boundary migration by nanoscale dispersoids seems to be instrumental for the development of the observed microstructure evolution.

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References

[1] R. Valiev, R. Islamgaliev, I. Alexandrov: Prog. Mater. Sci. 45 (2000), p. 103
[2] O. Sitdikov, T. Sakai, E. Avtokratova, R. Kaibyshev, Y. Kimura, K. Tsuzaki: Mater. Sci. Eng. Vol. A444 (2007), p. 18
[3] A. Goloborodko, O. Sitdikov, T. Sakai, R. Kaibyshev, H. Miura: Mat. Trans. Vol. 44 (2003), p. 766
[4] O. Mishin, D. Juul Jensen, N. Hansen: Mater. Sci. Eng. Vol. A342 (2003), p. 320
[5] A. Goloborodko, O. Sitdikov, R. Kaibyshev, H. Miura, T. Sakai: Mater. Sci. Eng. Vol. A381 (2004), p. 121
[6] X. Molodova, R. Berghammer, G. Gottstein, R. Hellmig: Int. J. Mat. Res. 98 (2007), p. 158
[7] S. Ferrasse, V. Segal, F. Alford: Mater. Sci. Eng. Vol. A372 (2004), p. 235
[8] S. Ferrasse, V. Segal, S. Kalidindi, F. Alford: Mater. Sci. Eng. Vol. A368 (2004), p. 28
[9] P. Sun, P. Kao, C. Chang: Mater. Sci. Eng. Vol. A283 (2000), p. 82
[10] X. Yang, H. Miura, T. Sakai: Mat. Trans. Vol. 43 (2002), p. 2400
[11] R. Kaybishev, E. Avtokratova, A. Apollonov, R. Davies: Scripta Mater. 54 (2006), p. 2119
[12] R. Kaibyshev, O. Sitdikov, A. Goloborodko, T. Sakai: Mater. Sci. Eng. Vol. A344 (2003), p. 348
[13] P.J. Apps, M. Berta, P.B. Prangnell: Acta Mater. 53 (2005), p. 499
[14] Y. Iwahashi, Z. Horita, M. Nemoto, T.G. Langdon: Acta Mater. Vol. 46, No. 9 (1998), p. 33
[15] J. Hirsch, K.A. Padmanabhan and K. Lücke: Proceedings of the 8th Intern. Conference of Texture of Materials (ICOTOM 8), edited by J.S. Kallend and G. Gottstein (Metallurgical Society of AIME), 1988, p.555