Comparison of the microstructure and the mechanical properties of AX41 magnesium alloy processed by EX-ECAP via three different routes A, Bc and C

T Krajňák¹, K Máthis¹, M Janeček¹, J Gubicza²
¹Charles University in Prague, Ke Karlovu 3, 121 16, Prague 2, Czech Republic
²Eötvös Loránd University, Department of Materials Physics, Budapest, Hungary

E-mail: tom.krajnak@gmail.com

Abstract. This work is devoted to the creep-resistant AX41 magnesium alloy (Mg-4 wt.% Al-1 wt.% Ca), processed by extrusion and consecutive Equal Channel Angular Pressing (EX-ECAP) up to total of eight passes, via route A, Bc and C at 220 °C. Beyond the deformation behavior the change of the microstructures was studied by electron back-scattering and X-ray diffraction methods. Significant grain refinement was found for all processing routes (grain sizes decreased below 1 μm after 8 passes). The X-ray line profile analysis performed by Convolutional Multiple Whole Profile (CMWP) fitting method has not revealed differences between the particular dislocation structures. Route A was found to be the most effective processing route from the point of view of grain size refinement and the room temperature strength. The influence of the texture and the dislocation structure on the plastic deformation processes is discussed in detail.

1. Introduction

Magnesium is known as one of the lightest metallic structural material with high specific strength, good castability and recyclability. Consequently, the magnesium alloys have been found profitable for applications in electronics and transport industry. However, there are several drawbacks stemming from the hexagonal closed packed structure of magnesium, which seriously hinder their wider applicability. Particularly the limited formability at ambient temperatures and strong plastic anisotropy belong to the most often referred issues [1,2]. The latter phenomenon causing e.g. the significant difference between the tensile and compressive yield strength is often reported for wrought alloy undergoing various thermo-mechanical processing as rolling or extrusion. Therefore, the current research is focused on the development of processing techniques, which are able to produce magnesium alloys having relatively isotropic plastic properties and good low temperature formability [3].

In the last decades, severe plastic deformation (SPD) techniques resulting in grain size reduction and introduction of large amount lattice defects have been extensively used for improving mechanical properties of wide range of materials. The degree of grain fragmentation during SPD processing was found to be dependent on various experimental parameters such as strain path [4], amount of accumulated strain [5,6], temperature and speed of processing [7,8]. In this study the samples are processed by equal channel angular pressing (ECAP) which is one of the most frequently used SPD technique [9]. Generally in ECAP the sample in the form of rod or bar is pressed through a die, which consists of two channels with intersection angle \( \phi \). Since the ECAP does not alter the dimensions of the sample, the pressing can be repeated many times. The sample rotation around its longitudinal axis between the consecutive passes yields alternation of the shear stress characteristic and activation of different dislocation slip systems. According to the angle and direction of rotation four basic processing routes of ECAP (Routes A, B_A, B_C and C) can be distinguished [10].
common features of the materials processed by ECAP are the small grain size in submicron or nanometer range and the superb mechanical properties [11].

The main aim of the presented paper is to investigate the influence of the different procession routes (A, B, C and D) of ECAP technique on the microstructure, texture and dislocation structure of extruded AX41 magnesium alloy. The obtained results are used for the explanation of the differences in the mechanical properties of the samples processed by different ECAP routes.

2. Experimental procedures

Measurements were conducted on commercial AX41 magnesium alloy with the composition of Mg - 4 wt. % Al - 1 wt. % Ca, severely deformed using combination of hot extrusion (EX) and ECAP. This procedure is referred to as EX-ECAP. Samples were extruded at 350 °C at the speed of 60 mm/min with an extrusion ratio of ER = 19:1. Prior the ECAP, billets with the dimensions of 10 x 10 x 100 mm were machined from the extruded rod. ECAP processing was carried out at temperature of 220 °C for eight passes, following routes A, B, C or D. The samples were pressed with the speed of 20 mm/min through a die consisting of rectangular channel (inner angle $\phi = 90^\circ$, outer curvature $\psi = 0^\circ$) with equal cross section of 10 x 10 mm. The samples are labeled according to the applied ECAP route. For instance the specimen processed by 8 passes of route Bc is labeled as 8xBc.

Microstructure and microtexture of EX-ECAPed samples were examined by electron backscatter diffraction (EBSD). Samples for EBSD measurements were cut from the rod perpendicular to the pressing direction, grinded by 1200, 2400, 4000-grit SiC papers and subsequently polished by diamond suspensions with the particle sizes of 1 $\mu$m and $\frac{1}{4}$ $\mu$m. For final surface treatment ion-polishing was performed on a Gatan PIPS™ ion mill at 2 kV and an incidence angle of 4°. EBSD investigations were taken using Quanta FEG scanning electron microscope operated at 10 kV from the middle part of the billet. The area examined by EBSD was approximately 300 x 250 $\mu$m and 100 x 100 $\mu$m for extruded and EX-ECAPed samples, respectively.

Room temperature mechanical properties were tested in tension at a constant strain rate of 10$^{-4}$ s$^{-1}$ using an Instron 5882 testing machine. Flat specimens with rectangular cross section of 6 mm x 1 mm and gauge length of 13 mm were fabricated with the tensile axis lying parallel to the longitudinal axis of the ECAP-processed billets.

The X-ray diffraction measurements were carried out by a high-resolution rotating anode diffractometer using CuK$_\alpha$ (wavelength: $\lambda = 0.15406$ nm) radiation. The size of the X-ray beam spot was about 2 x 0.2 mm$^2$. The Debye–Scherrer diffraction rings were detected on two-dimensional imaging plates having a linear spatial resolution of 50 $\mu$m. The X-ray line profiles were obtained as a function of the diffraction angle by integrating the two-dimensional intensity distribution along the rings at discrete angle values. The X-ray diffraction patterns were evaluated by Convolutional Multiple Whole Profile (CMWP) fitting method [12, 13]. In this procedure, all the measured peaks are fitted simultaneously by the sum of a background spline and the convolution of the physical and instrumental profiles. The theoretical physical profile functions are calculated on the basis of a model of microstructure, where the crystallites have spherical shape and lognormal size distribution. The lattice strains are assumed to be caused by dislocations. Nineteen peaks of Mg were used in the fitting procedure, which cover an angular range of diffraction of about 30-135°. Peaks at high angle positions are of great importance because the peak broadening is more pronounced at higher diffraction angles.

3. Experimental results

Microstructural observations performed by EBSD method are shown in Fig. 1. The color code of the figures is given by the reference triangle, which is the same for all maps. The microstructure of the extruded sample is bimodal and contains considerable fraction of large grains with the size between 10
- 20 µm. This bimodal character preserves also after processing via route Bc, where the size of the large grains decreased down to 3-5 µm with the fraction of ~25%. The average grain size (determined by the linear intercept method) of 8xBc sample was set as 1 µm. On contrary, homogeneous microstructures consisting of fine and equiaxed grains with the average grain size of 0.8 and 0.9 µm were observed after processing by routes A and C, respectively. Further impact of the different processing routes can be observed in the case of high angle grain boundaries. Whereas the fraction of high angle grain boundaries (HAGBs), (the misorientation is larger than 15°) was 82% for both microstructures in samples 8xA and 8xBc, in the case of specimen 8xC this fraction decreased to 66%, similar to the extruded sample. Because all the EX-ECAPed microstructures contain significant amount of very small and equiaxed grains with the size below 1µm and HAGBs, dynamic recrystallization as the main mechanism of grain refinement cannot be excluded [14].

![Crystal orientation maps for samples after (a) extrusion, (b) 8xBc passes, (c) 8xC passes and (d) 8xA passes. The high angle grain boundaries (> 15°) and low angle grain boundaries (< 15°) are outlined by black and red colors, respectively.](image)

**Figure 1** - Crystal orientation maps for samples after (a) extrusion, (b) 8xBc passes, (c) 8xC passes and (d) 8xA passes. The high angle grain boundaries (> 15°) and low angle grain boundaries (< 15°) are outlined by black and red colors, respectively.

As an example the diffraction pattern for sample 8xC is shown in Fig. 2. The open circles and the red line represent the measured data and the fitted curves, respectively. The inset in the upper right corner provides enlarged view of the quality of fitting. Parameters obtained by Convolutional Multiple Whole Profile (CMWP) fitting method, namely the dislocation density ($\rho$) and the crystallite size
(<x>_area) are summarized in Table 1. The area-weighted mean crystallite size (<x>_area) was calculated according to the formula: <x>_area=m.exp (2.5σ²) [15], where m and σ represents the median and the variance of the log-normal size distribution, respectively. For the extruded state the crystallite size and the dislocation density were larger and smaller, respectively, than the detection limits of the line profile analysis method. Therefore, the values for the as-extruded state in the table represent the limiting values for the parameters of the microstructure.

It has been found that the crystallite size and the dislocation density in the samples after eight passes do not depend significantly on the processing routes. The crystallite size was estimated to be about 250 nm and the dislocation density was approximately 0.7 x 10¹⁴ m⁻². It is noteworthy that the crystallite size determined from X-ray line profiles is equivalent to the size of coherently scattering domains, which corresponds to dislocations cells or subgrains in SPD-processed metallic materials. Therefore crystallite size does not match the grain size determined by EBSD method, which take into account only grains with high angle grain boundaries.

Table 1 - The parameters of the microstructure and the plastic behavior obtained by X-ray line profile analysis, EBSD and tensile tests: the area-weighted mean crystallite size (<x>_area), the dislocation density (ρ), the grain size (d), the fraction of high-angle boundaries (f_HAB), the yield strength (σ_<sub>0.2</sub>) and the ductility (ε).

| Number of EX-ECAP passes | X-ray | EBSD | Tensile tests |
|--------------------------|-------|------|---------------|
|                          | <x>_area [nm] | ρ [10¹⁴ m⁻²] | d [µm] | f_HAB [%] | f_LAB [%] | σ_<sub>0.2</sub> [MPa] | ε [%] |
| Extruded                 | 1µm <     | < 0.1 | 3.8 ± 0.4 | 61      | 9       | 161            | 18 |
| 8xA                      | 217 ± 22  | 0.7 ± 0.1 | 0.8 ± 0.1 | 82      | 10      | 223            | 23 |
| 8xBc                     | 268 ± 27  | 0.7 ± 0.1 | 1.0 ± 0.1 | 82      | 9       | 180            | 23 |
| 8xC                      | 249 ± 25  | 0.8 ± 0.1 | 0.9 ± 0.1 | 66      | 17      | 139            | 29 |

Figure 2 - The CMWP fitting for the sample processed by 8 ECAP passes via route C. The open circles and the red solid line represent the measured data and the fitted curves, respectively. The
intensity is plotted in logarithmic scale. In the inset the intensity is plotted in linear scale and the
difference between the measured and the fitted patterns is shown at the bottom.

The sample coordinate system (x-, y- and z-axes) and the three orthogonal planes X, Y and Z used for
the representation of texture measurements are depicted in Fig. 3. The x-axis is parallel to the pressing
or extrusion direction.

Figure 3 - Schematic representation of the sample coordinate system.
Figure 4 - The EBSD (0001) and (10\bar{1}0) pole figures of (a) the extruded and (b) 8xB, (c) 8xC, (d) 8xA samples.

The (0001) and (10\bar{1}0) pole figures for the cross section (plane X) of the extruded and EX-ECAP states are shown in Fig. 4. A \(<10\bar{1}0>\) fiber texture typical for magnesium alloys after extrusion [16,17] can be observed in Fig. 4a. The one strong maximum observed in the center of (10\bar{1}0) pole means that majority of the prismatic \{10\bar{1}0\} planes lie perpendicular to the x-axis and consequently the (0001) basal planes are oriented parallel to the x-axis. Evenly distributed maximum around the edge of (0001) pole figure reflects that basal planes are randomly rotated around the x-axis.

Fig. 4b represents the texture of 8xB sample. It is apparent from the pole figures (0001) and (10\bar{1}0) that the crystallographic c-axis in the majority of crystallites is lying in plane Z and tilted by approximately 45° to axis x. The prismatic planes are rotated randomly around the crystallographic c-axis in the polycrystalline sample. It is noted that the orientation of the basal planes follows the theoretical shearing plane activated during the ECAP processing [18,19]. The pole figures (0001) and (10\bar{1}0) depicted in Fig. 4c show also a strong texture in sample 8xC, where mostly the crystallographic c-axis is lying in plane Y and tilted by approximately 40-50° to axis x. The prismatic planes are rotated randomly around the crystallographic c-axis in the polycrystalline sample. The pole figures for sample 8xA indicate that the crystallographic c-axis is mainly lying in plane Z parallel to axis y. The prismatic planes are rotated randomly around axis y. The common feature of the textures evolved in the extruded sample and the specimen processed by route A is that the crystallographic c-axis is perpendicular to the longitudinal axis of the billets, i.e. the basal plane is parallel to axis x. The texture analysis clearly shows that the various processing routes results in very different textures.
Nevertheless, further texture analysis on the basis of ideal orientations in simple shear of hexagonal crystal structures is planned in order to get a deeper understanding of texture formation (the details of this procedure can be found in [20]). This investigation has been not completed yet, thus it is not included in this paper.

The processing route dependence of the true stress–true strain curves obtained from room temperature tensile tests is shown in Fig. 5. Values for yield strength ($\sigma_{0.2}$) and ductility ($\varepsilon$) are given in Table 1. The ductility is characterized by the maximum true strain measured up to the necking of the samples during tension. The sample processed by route A exhibits the highest value of yield strength 223 MPa among the EX-ECAPed samples. The strength increment due to ECAP is approximately 30% in comparison with the as-extruded state. Since all the investigated microstructural parameters, namely the area-weighted mean crystallite size ($<x>_{\text{area}}$), the dislocation density ($\rho$), the grain size ($d$), of EX-ECAPed samples were found to be the same within the experimental error, except 8xC sample having slightly lower fraction of high-angle boundaries ($f_{\text{HAB}}$), the difference in mechanical properties can be mainly attributed to the texture variation. The 8xBc sample exhibits better mechanical properties in comparison to the extruded specimen as well, primarily due to the lower grain size, the higher dislocation density and also the higher fraction of high-angle boundaries. This observation deviates from the common finding in the literature [21], where usually the extruded samples possess higher values of yield strength and tensile strength due to the strong $<10\bar{1}0>$ fiber texture. Table 1 shows that ECAP processing has a beneficial effect on the ductility. The observed values are larger than that obtained for either as-extruded or as-cast state [22].

![Figure 5 - Room temperature true stress-true strain curves of extruded and EX-ECAPed samples.](image)

4. Discussion

Based on the investigated microstructures shown in Fig. 1, it can be concluded that the type of processing route strongly affect the fragmentation rate of the original microstructure and the fraction of the high angle boundaries. EX-ECAP processing for 8 passes via route A resulted in formation of very fine, equiaxed grain structure having large fraction (>82%) of high angle grain boundaries. On contrary, a bimodal grain structure, similar to that after extrusion, was observed after route Bc processing. Fragmentation level of the microstructure after processing by route C was similar to that of the route A, but the microstructure has lower fraction of high angle grain boundaries. In summary, route A seems as the most effective in the formation of ultrafine-grained microstructure in AX41 magnesium alloy. It is noted that the dislocation densities and the crystallite sizes determined after 4 passes of EX-ECAP for any route (not included in this paper) agree with the values obtained after 8 passes within the experimental error. Therefore it is reasonable to assume that the microstructure has already reached a saturated level after 4 passes due to the dynamic equilibrium between the formation and annihilation of lattice defects, irrespectively of the applied ECAP route.

EBSD microtexture analysis revealed the presence of strong textures in samples after EX-ECAP processing. Texture of the 8xA sample was similar to the extruded one (the (0001) basal planes are oriented parallel to the x-axis). However, unlike the extruded state where the basal planes are
randomly rotated around the extrusion axis, the basal planes in 8xA sample are lying only parallel to the Y-plane of billet. The textures of samples processed by routes B_c and C has similar character, e.g. the crystallographic c-axis is tilted by approximately 45° to the pressing direction, and the basal planes are tilted roughly by 45° to both y and z-axes. This orientation of the basal planes follows the theoretical shearing plane activated during ECAP processing [19].

The results of tensile tests (Fig. 4) showed that the values of yield strengths varied according to route of processing. The highest yield strength was achieved for the sample processed by route A. Since the dislocation density and the grain size are the same within the experimental error for all the applied routes, the difference between the strength might be attributed to the texture variation. Similar dependence of the yield strength on the route of ECAP processing was found by Kim et al. [23]. In that case, the extruded AZ61 magnesium alloy processed via route A up to eight passes exhibited also higher yield strength in comparison with route B_c, despite the similar mean grain sizes of the two samples. This difference was explained by the different values of Schmid factor for the basal slip system in the dominant texture component (1012)[1210] for route A, and in the two texture components (1011)[0111] and (1012)[1210] for route B_c. According to the zero average Schmid factor for basal slip system in sample 8xA (identical with the as-extruded alloy) and the non zero value for route B_c, the basal planes in sample 8xB_c are more favorably orientated for basal slip than in sample 8xA, which led to a smaller yield strength compared to sample 8xA. In the case of sample 8xC the texture softening overwhelmed the strengthening caused by the increment in dislocation density and the grain refinement, resulting in a smaller yield strength compared to the as-extruded state.

5. Conclusions

The effect of hot EX-ECAP processing conducted via three deformation routes A, B_c and C on the microstructure and mechanical properties of AX41 magnesium alloy was studied. The application of ECAP on the extruded samples leads to further grain refinement. The average grain size decreased from 3.8 µm to approximately 1 µm. Whereas the as-extruded and 8xB_c samples exhibited a bimodal microstructure, the microstructures of specimens 8xA and 8xC were homogenous and consisted of fine and equiaxed grains. The fraction of the high angle grain boundaries for samples 8xA and 8xB_c increased up to 82%. At the same time, in sample 8xC the fraction of high angle grain boundaries was only 66%, similar to the as-extruded sample. The X-ray line profile analysis did not reveal any substantial differences between the dislocation densities and subgrain sized in the EX-ECAPed samples processed by various routes. The crystallite size decreased to about 250 nm while the dislocation density increased to approximately 0.7 x 10^14 m^-2 for all samples. Despite the similar microstructure the texture was very different after the various ECAP routes. For route A the basal planes were orientated parallel to the x-axis of the ECAP-processed sample, similar to the as-extruded state. For routes B_c and C, the inclination angle between the basal planes and the sample axis was about 45°. Therefore, the larger Schmid factor for basal slip in specimen 8xA resulted in the highest yield strength compared to other ECAP routes. Additionally, the softening due to texture change for route C was stronger than the strengthening owing to grain refinement and the increase of the dislocation density, which led to smaller yield strength than in the as-extruded state. Thus, the differences in the plastic behavior can be mainly attributed to the observed texture variation. Route A was found to be the most effective in improving mechanical properties.
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