A comparative study on the microstructural evolution in AM60 alloy processed by ECAP and MDF

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1. Introduction

Magnesium alloys are the lightest commercially available structural materials, and therefore, they are used in many applications such as aerospace, automotive, transportation and electronic industries [1–3]. The potential of the practical applications of Mg alloys can be further increased by the improvement of their mechanical properties using severe plastic deformation (SPD) processing. SPD techniques applied at room temperature are able to refine the grain size into the ultrafine-grained regime, i.e., below 1 μm, thereby increasing the strength of metallic materials. However, for Mg alloys SPD processing is usually performed at elevated temperatures due to the low ductility of hexagonal materials. SPD carried out at about 200–300 °C can result in a grain refinement to several microns without the failure of the Mg alloy samples [4].

Equal-channel angular pressing (ECAP) and multi-directional forging (MDF) are frequently used SPD methods since they are capable of producing homogeneous fine-grained microstructures in bulk materials with large dimensions. The formation of microstructure is influenced by the parameters of ECAP processing, such as the temperature, the ram speed, the angle between the two channels (ϕ), the outer radius angle (Ψ) and the applied route. The effect of these parameters on the microstructure and the mechanical properties has been investigated for various Mg alloys [5–8]. Gan et al. [9] achieved a reduction in the grain size from 900 to 38 μm in pure magnesium by applying four passes of ECAP. Their results also showed that the ultimate tensile strength increased from 70 to 170 MPa due to ECAP. Horita et al. [10] found that an initial extrusion increased the effectiveness of ECAP in the reduction of grain size. In the recent years, several experiments have been conducted to improve the yield and the ultimate tensile strength of magnesium with the application of ECAP processing [11,12]. However, some studies revealed that despite the strong grain refinement, the yield strength after ECAP might be lower than that in the extruded state before ECAP, due to the change of the crystallographic texture [13,14]. It was also reported that with increasing the passes of ECAP the microstructure and the micro-hardness distribution in Mg alloys became more homogeneous [15].

Besides ECAP, MDF is another attractive method for producing bulk fine-grained Mg alloys since this procedure is very simple and able to process samples with various sizes [16–19]. For example, Jiang et al. [20,21] applied MDF process to AZ61 and Mg–7Al–2Sn alloys, resulting in a small grain size and an improved strength.
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Cai et al. [22] investigated the effect of MDF technique on AZ31B magnesium alloy and reported that both the yield and the ultimate tensile strength values were improved compared to the extruded samples, along with the formation of a homogeneous microstructure. Guo et al. [23] investigated the effect of MDF on AZ31 alloy and reported the formation of a homogeneous microstructure with the grain size of 1.3 μm and a simultaneous improvement of the ultimate tensile strength. In the study carried out by Xia et al. [24] the grain size of Mg-Gd-Y-Nd-Zr alloy decreased from 200 μm to 5.1 μm after six passes of MDF, and a significant improvement was observed in both the yield and ultimate strength values.

AM60 alloy is one of the most widely used magnesium alloys with the main alloying elements of Al (6 wt%) and Mn (0.35 wt%). Therefore, the study of the effect of SPD-processing on the microstructure is important from the point of view of the practical applications of this alloy. ECAP and MDF are frequently used SPD methods as both processing techniques are able to produce fine-grained microstructures in bulk samples with large dimensions. In addition, both methods are usually applied in a similar equivalent strain range of 1–10. At the same time, the loading conditions during the two procedures are considerably different which may yield significant differences in the as-processed microstructures of AM60 alloys even for the same equivalent strains. Therefore, a comparison of the microstructures formed in ECAP- and MDF-processed AM60 alloy samples has a great significance. Although, some studies have already investigated the ECAP-induced changes in the grain size and the mechanical performance of AM60 alloy [12,25], a systematic comparison between the microstructures formed by ECAP and MDF methods has not been given yet. In addition, a comparative study on the development of the dislocation density in ECAP- and MDF-processed AM60 alloys is missing from the literature. The present work fills this gap and provides the evolution of the type and density of dislocations as well as the grain size in AM60 alloy as a function of ECAP and MDF passes. In addition, the change of the secondary phase content as a function of the number of passes was also monitored and the differences observed for ECAP and MDF are discussed. The hardness of the samples was measured and correlated to the microstructure.

2. Material and methods

2.1. Sample processing

High purity (about 99.9 wt %) Mg, Al and Mn were used to prepare AM60 Mg-alloy with the alloying element contents of 6 wt % Al and 0.35 wt% Mn. The elements were melted in a graphite crucible placed in an electrical furnace at 750 °C under Foseco Magrex 36 covering flux in order to protect molten magnesium from oxidation. The melt was held at 750 °C for 30 min and mechanically stirred for 2 min. The melt was then poured into a pre-heated steel die by a tilt-casting technique in order to minimize casting defects and turbulences of the melt. The as-cast cylindrical billets with 44 mm in diameter were extruded to bars with the cross-section of 13 mm × 13 mm at 380 °C and a speed of 2 mm/s.

2.2. ECAP processing

ECAP billets with the dimensions of 13 mm × 13 mm × 100 mm were machined from the extruded bars. All samples were processed at 220 °C using an ECAP die having an internal angle of φ = 90° between the two channels of the die. The outer radius at the intersection of the channels was ψ = 20°. The temperature was continuously monitored and controlled to be within ±5 °C during the experiments. For each pass, first the die was heated to the target temperature and stabilized before the introduction of the sample into the die. Once the ECAP die achieved the desired temperature, a period of 15 min was allowed to elapse before the ECAP processing started. This time was long enough to allow the billet to reach a steady-state temperature (220 °C). The deformation for a single pass of ECAP corresponds to an equivalent strain of about one. The multiple passes of ECAP were performed following route BC because former studies demonstrated that this is the most effective route in the achievement of a fine-grained microstructure. The samples were coated with molybdenum disulphide (MoS2) lubricant and pressed at a speed of 10 mm/min for 2, 4 and 6 passes. Therefore, for one pass of ECAP the sum of the sample heating (15 min) and the deformation (10 min) periods was 25 min.

2.3. Processing of the samples by MDF

The extruded bars were cut into rectangular MDF billets with the dimensions of 13 mm × 13 mm × 20 mm. The samples were subjected to MDF at a temperature of 220 °C using a screw driven MTS material testing system. First, the die was heated to the target temperature and stabilized before the introduction of the sample. Once the MDF die achieved the desired temperature, a period of 15 min was allowed to elapse before the starting of the MDF process. This time was enough for the billet to reach the temperature of 220 °C. Then, the sample was forged to the dimensions of 20 mm × 13 mm × 13 mm. According to the changes of the dimensions of the billets, an equivalent strain of 0.5 was achieved in each pass of MDF. The initial forging direction was parallel to the extrusion axis. Between the consecutive MDF passes, the loading axis orientation changed with an angle of 90°. The samples were coated with a 0.05-mm-thick PTFE film as a lubricant and pressed at a speed of 1 mm/min for 2, 4 and 6 passes. Therefore, for one pass of MDF the sum of the sample heating (15 min) and the deformation (7 min) periods was 22 min which is close to the time needed for one pass of ECAP (25 min).

2.4. Study of the phase composition and the microstructure

For the study of the phase composition and the microstructure in the AM60 alloys, the samples were immediately water quenched after the various passes and sectioned perpendicular to the longitudinal axis of the ECAP and MDF processed samples. Thus, the normal of the studied surface was parallel to the longitudinal axis of the samples obtained by both SPD-processing methods. The phase composition of the samples was studied by X-ray diffraction (XRD) using a Philips Xpert θ–2θ powder diffractometer operating at 40 kV and 30 mA with CuKα radiation (wavelength: λ = 0.15418 nm). The surface studied by XRD was first mechanically polished with 1200, 2500 and 4000 grit SiC abrasive papers and then the polishing was continued with a colloidal silica suspension (OP-S) with a particle size of 40 nm. Finally, the surface was etched in a solution consisting of 75 ml ethylene glycol, 1 ml HNO3 and 24 ml H2O for 10 s.

The grain structure of the Mg matrix was studied by scanning electron microscopy (SEM) using a FEI Quanta 3D electron microscope. Electron back-scattered diffraction (EBSD) was used for the determination of the average grain size. The studied areas were etched by focused ion beam (FIB) of Ga+ ions with an inclination angle of 6° using an accelerating voltage of 30 kV and an ion current of 15 nA. The step size in the EBSD experiments was set at 400 nm for the extruded sample and 100 nm for the SPD-processed specimens. The orientation maps obtained by EBSD were evaluated for the grain size using the OIM software. The grains were considered as the regions in the EBSD images bounded by high-angle grain boundaries (HAGBs) with misorientations higher than 15°.
program determines the size of a grain as the diameter of the equivalent circle with the same area. Then, the grain size distribution was plotted and the area-weighted average grain size was calculated for the studied samples.

The lattice defect structure in the specimens was studied by X-ray line profile analysis (XLPA). The X-ray line profiles were measured on the surface prepared for the XRD study by a high-resolution rotating anode diffractometer (type: RA-MultiMax9, manufacturer: Rigaku) using CuKα (wavelength: $\lambda = 0.15406$ nm) radiation. Two-dimensional imaging plates detected the Debye-Scherrer diffraction rings. The line profiles were determined as the intensity distribution perpendicular to the rings. The peak profiles were evaluated by the convolutional multiple whole profile (CMWP) fitting procedure [26]. In this method, the diffraction pattern is fitted by the sum of a background spline and the convolution of the instrumental pattern and the theoretical line profiles related to the diffraction domain size and dislocations. The instrumental diffraction peaks were measured on a LaB6 standard material. For each reflection, the theoretical line profile was obtained as the convolution of the theoretical size and strain profiles. The first 14 reflections of the Mg-matrix were evaluated. The peaks of the precipitates were included in the background of the patterns. The area-weighted mean diffraction-domain size ($<x^2_{\text{area}}>$) and the dislocation density ($p$) were determined by the CMWP fitting evaluation procedure. The value of $<x^2_{\text{area}}>$ was calculated as $<x^2_{\text{area}}>=m \cdot \exp(2.5\sigma^2)$, where $m$ is the median and $\sigma$ is the square root of the lognormal variance of the diffraction domain size distribution. In addition, parameters denoted as $q_1$ and $q_2$ are obtained from XLPA for the characterization of the active dislocation slip systems.

2.5. Hardness measurements

The Vickers hardness ($HV$) was determined after extrusion, ECAP and MDF processes. The measurements were carried out on mechanically polished surfaces cut perpendicular to the longitudinal direction of the samples using a V-Test hardness tester manufactured by Bareiss (Germany) with a load of 1 kg and a dwell time of 30 s. The hardness of the samples was determined as an average of at least five measurements taken at random places in the central region (6 mm × 6 mm) on the surface of the specimens.

3. Results

3.1. Phase composition of the samples processed by ECAP and MDF

The phase composition for the extruded AM60 sample, and the specimens processed by different passes of ECAP and MDF was studied by XRD. As an example, Fig. 1 shows the X-ray diffraction patterns obtained for the extruded alloy and the sample deformed by 6 passes of ECAP. The extruded sample was almost a single phase hexagonal close packed (hcp) Mg solid solution and only very weak patterns obtained for the extruded alloy and the sample deformed region (6 mm3.1. Phase composition of the samples processed by ECAP and MDF

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3.2. Effect of ECAP and MDF on the grain size

The EBSD orientation map in Fig. 3a shows the microstructure of the extruded sample before SPD-processing. In the vicinity of some grain boundaries, the color changes from pixel to pixel. In these areas, the EBSD image quality index was low, therefore the determination of the crystallographic orientation by the OIM software was uncertain and the neighboring pixels received different colors in the figure. The poor image quality was most probably caused by strong lattice distortions due to dislocations piled up against grain boundaries. These areas were excluded from the evaluation of the grain size owing to the uncertain determination of the crystallographic orientation. The grain size distribution of the extruded sample obtained from the EBSD image is shown in Fig. 3b. The area-weighted average grain size was obtained as ~13 µm. After 2 passes of ECAP and MDF, the grain size was reduced to ~3 µm. Higher numbers of passes for MDF did not result in further grain refinement. As an example, Fig. 3c and d show an EBSD orientation map and the extracted grain size distribution after 6 passes of MDF, respectively. The areas where the color changed from pixel to pixel were excluded from the evaluation of the grain size. ECAP-processing for 6 passes yielded an average grain size of ~2 µm as shown in the EBSD orientation map in Fig. 3e and the corresponding grain size distribution in Fig. 3f.

3.3. Evolution of the type and density of dislocations during ECAP and MDF

The Debye-Scherrer rings detected for the extruded sample contained high intensity spots which were caused by large diffraction domains with very low dislocation density. As an example, Fig. 4a shows the Debye-Scherrer diffraction ring with the indices {110} for the extruded specimen. The spotty rings cannot be
evaluated, as the breadth of the spots corresponds to the instrumental broadening of the applied diffraction system. For the extruded sample, the diffraction domain size was larger while the dislocation density was lower than the detection limits of the present diffraction configuration (~1 \( \mu \text{m} \) and ~10\(^{13} \text{m}^{-2} \), respectively). For the samples processed by ECAP and MDF, the Debye-Scherrer rings were free of spots as shown in Fig. 4b for reflection 110 of the sample processed by 2 passes of ECAP.

The average diffraction domain size and dislocation density in the samples processed by 2, 4 and 6 passes of ECAP and MDF were determined by XLPA. As an example, Fig. 5 shows the CMWP fitting on the X-ray diffraction pattern obtained for the sample deformed by 6 passes of MDF. The dislocation density as a function of the number of passes for ECAP and MDF is plotted in Fig. 6a. The dislocation density for the extruded sample was below the detection limit of the present XLPA method (~10\(^{13} \text{m}^{-2} \)), as discussed above. In order to visualize the trend in the evolution of the dislocation density in Fig. 6a, the value of the detection limit was considered as the dislocation density for the extruded material. The dislocation density significantly increased when the extruded sample was processed for 2 passes of ECAP and MDF. After 2 passes of ECAP, a maximum value of 5.6 ± 0.6 \( \times 10^{14} \text{m}^{-2} \) was obtained for the dislocation density, while between 2 and 4 passes the dislocation density was reduced considerably to 0.7 ± 0.2 \( \times 10^{14} \text{m}^{-2} \). Further ECAP-processing to 6 passes did not cause any significant change in the dislocation density. In the MDF-processed samples, the evolution of the dislocation density was very different from that observed for ECAP. Namely, the dislocation density was saturated at a much smaller value after 2 passes (1.8 ± 0.2 \( \times 10^{14} \text{m}^{-2} \)) than for ECAP and further MDF deformation yielded a lower relative
The dislocation density values after 4–6 passes were practically the same for ECAP and MDF. The difference in the evolution of the dislocation density for ECAP and MDF may be caused by the different equivalent strain values for the same number of passes. Therefore, the dislocation density was also plotted as a function of the equivalent strain in Fig. 6b. This way of plotting did not result in a coincidence between the data obtained for ECAP and MDF, which suggests that besides the applied strain the mode of deformation has also a considerable effect on the defect structure developed during SPD at 220 °C. The possible reasons of this difference will be discussed in section 4.

The $q_1$ and $q_2$ parameters of the dislocation contrast factors obtained by XLPA depend on the character of dislocations and therefore enable the determination of the prevailing dislocation slip systems in the sample. In this analysis, the experimentally determined $q_1$ and $q_2$ values are compared with the theoretical values calculated for the eleven possible slip systems in Mg [27]. The eleven dislocation slip systems are usually classified into three groups based on their Burgers vectors: $b_1 = 1/3 < -2110> \langle <a> \text{-type}\rangle$, $b_2 = <0001> <c\text{-type}>$ and $b_3 = 1/3 < -2113> \langle c< a> \text{-type}\rangle$. In the determination of the slip system population, the evaluation software selected some dislocation systems from each group and averaged their theoretically calculated $q_1$ and $q_2$ values. Then, these values were averaged for the three Burgers vector groups using their fractions as weights. The unknown fractions were determined by making these average theoretical $q_1$ and $q_2$ values equal to the two experimentally determined parameters $q_1$ and $q_2$. A third equation for the fractions of $(a)$, $(c)$ and $(c-a)$ dislocation groups was obtained from the condition that the sum of the three fractions must be 100%. The three equations gave solutions for the fractions of the three dislocation types. A more detailed description of this method can be found in Ref. [28].

The analysis of the dislocation types showed that in the samples deformed for different passes of ECAP and MDF the relative fractions of the $(a)$, $(c)$ and $(c-a)$ dislocations are in the ranges 56–86%, 2–19% and 8–25%, respectively. The abundance of $(a)$-type dislocations can be explained by their small formation energy due to their relatively short Burgers vector compared to $(c)$ and $(c-a)$ dislocations. Fig. 7a and b show the fraction of $(a)$-type dislocations as a function of the number of passes and the equivalent strain, respectively, for both ECAP and MDF. Between 2 and 6 passes of MDF-processing, the fraction of $(a)$-type dislocations changed only slightly, in accordance with the moderate variation of the dislocation density. At the same time, for ECAP the fraction of $(a)$-type dislocations increased significantly between 2 and 4 passes. As the dislocation density decreased considerably between 2 and 4 passes of ECAP, the change in the dislocation fractions was most probably caused by the easier annihilation of $(c)$ and $(c-a)$ dislocations due to their higher energy compared to $(a)$-type dislocations.

The average diffraction domain size values as a function of the number of SPD passes and versus the equivalent strain are plotted in Fig. 8a and b, respectively. The diffraction domain size for the extruded sample was above the detection limit of the present XLPA method (≈1 μm). For this sample, the detection limit was considered as the diffraction domain size in Fig. 8a and b. Both ECAP and MDF-processing resulted in a significant decrease in the mean diffraction domain size. The minimum diffraction domain size was achieved after 2 passes with the values of 110 ± 30 nm and 300 ± 40 nm for ECAP and MDF, respectively. Between 2 and 6 passes of ECAP and MDF, the average diffraction domain size increased to a similar value of about 530 nm. The decrease and increase of the diffraction domain size before and after 2 passes, respectively, occurred simultaneously with the increase and decrease of the dislocation density (compare Figs. 6 and 8). This observation suggests that the refinement of the diffraction domain size was caused by the rearrangement of dislocations into subgrain boundaries. Similar to the dislocation density, the evolution of the diffraction domain size is influenced by both the equivalent strain and the mode of SPD, as shown in Fig. 8b. It is noted that for both
ECAP- and MDF-processed samples the diffraction domain size obtained by XLPA is smaller than the grain size determined by microscopy. This difference can be explained by the fact that the diffraction domain is equivalent to the volume scattering X-rays coherently and dislocation patterns inside the grains may break the coherency of X-rays. Dipolar dislocation walls and low-angle grain boundaries are typical examples which cause fragmented grains. Therefore, the diffraction domain size can be considered as the size of subgrains and/or dislocation cells in the grain interiors. As an example, the inset in Fig. 3c shows low-angle grain boundaries (LAGBs) in the interior of a grain for the sample processed for 6 passes by MDF. The LAGBs were defined as the boundaries with misorientations between 2 and 15°.

Fig. 6. Evolution of the dislocation density as a function of (a) the number of passes and (b) the equivalent strain for ECAP and MDF.

Fig. 7. The dependence of the fraction of $<a>$-type dislocations on the (a) number of passes and (b) the equivalent strain for ECAP and MDF.

Fig. 8. The average diffraction domain size as a function of (a) the number of passes and (b) the equivalent strain for ECAP and MDF.
software is only 2°. Since XLPA is sensitive to much smaller orientation differences, diffraction domain boundaries may also exist in those areas where LAGBs are not indicated by the OIM software.

3.4. The influence of ECAP and MDF on the hardness

Fig. 9a shows the hardness as a function of the number of ECAP and MDF passes. For the ECAP-processed sample, the hardness increased from ~650 to ~990 MPa during the first two passes. Between 2 and 4 passes, the hardness was reduced to ~780 MPa which remained practically unchanged up to 6 passes of ECAP. MDF-processing for 2 passes also resulted in an increase of the hardness to ~800 MPa, however this hardening is much smaller than that for ECAP. Between 2 and 6 passes of MDF, significant changes in the hardness was not observed. The difference in the evolution of hardness for ECAP and MDF remained significant even if it was plotted as a function of the equivalent strain (see Fig. 9b). The correlation between the microstructure and the hardness will be discussed in the next section.

4. Discussion

The difference in the evolution of the microstructure during ECAP and MDF can be understood by analysing the processing conditions of the two methods. One pass of ECAP and MDF took similar times, namely 25 and 22 min, respectively (see sections 2.2 and 2.3). This includes a heating period of 15 min at 220 °C and a deformation period at the same temperature. For both ECAP and MDF, the temperature was monitored and kept at 220 °C during both periods. Therefore, the samples processed for the same number of passes by ECAP and MDF spent nearly the same time at 220 °C. At the same time, the strain imposed during one pass was very different for the two processing ways: about one for ECAP and ~0.5 for MDF. In addition, the strain rate during ECAP (~1.7 × 10⁻³ s⁻¹) was about 40% higher than for MDF processing (~1.2 × 10⁻³ s⁻¹). The average strain rate was estimated as the ratio of the average equivalent strain and the time elapsed during deformation. After 2 passes, the higher dislocation density achieved by ECAP compared to MDF can be explained by the higher imposed strain (as the time elapsed at 220 °C was similar for both processes). The slightly higher strain rate may also have a contribution to the higher maximum dislocation density in the ECAP-processed sample as it impedes the annihilation of dislocations during SPD. Plotting the dislocation density as a function of the equivalent strain, a coincidence between the data determined for ECAP and MDF was not achieved (see Fig. 6b) since for MDF the same equivalent strain was achieved in a much longer period at 220 °C, compared to ECAP. Therefore, the maximum and the subsequent reduction in the dislocation density occurred at smaller strain values for MDF-processing. It is noted that former finite element modeling studies [29–31] revealed that there is a strain heterogeneity in ECAP-processed samples which decreases with (i) decreasing friction between die wall and sample, (ii) increasing alloying element content and (iii) increasing number of passes. After 2 passes of ECAP-processing in a well-lubricated die, the scattering of the strain values in alloys usually is not higher than ±20%. In the present study, for a given number of passes the average equivalent strain is two times larger for ECAP than for MDF. Therefore, the strain inhomogeneity inside the samples was most probably much smaller than the difference between the average strain values obtained for the two processes.

It should also be noted that the two SPD methods have different loading conditions. MDF can be considered as a plane strain compression, while during ECAP shear occurs in the plane of intersection of the two channels. In addition, the ECAP billet was rotated between the consecutive passes in accordance with route Bc applied in this study. This difference between the loading conditions may also contribute to the different densities and types of dislocations observed at the same equivalent strain for MDF and ECAP. Indeed, former studies have proved that the deformation history has a significant effect on the microstructure developed during plastic straining [32–35]. For instance, the deformation history can be characterized by the strain trajectory in the five-dimensional space of the independent deviatoric strain tensor components [33]. Then, the ratio of the length of the strain trajectory and the spacing between the starting and end points of deformation in the five-dimensional space characterizes the non-monotonicity of plastic deformation. A larger value of this ratio corresponds to a higher degree of non-monotonicity of deformation. It has been shown that a stronger non-monotonic character of straining resulted in a faster multiplication of dislocations and grain refinement during deformation [32,34,35]. It has also been revealed that ECAP has a high degree of non-monotonic among plastic deformation techniques [32,33] which most probably contributed to the higher initial increase of the dislocation density compared to MDF-processing.

The formation of Al12Mg17 precipitates during high temperature SPD-processing was faster in the case of ECAP than for MDF either as a function of the number of SPD passes or the equivalent strain (see Fig. 2). This effect can be explained by the promotion of
precipitation by both annealing and plastic deformation. The dislocations formed during SPD facilitate the nucleation and growth of precipitates, as they act as fast diffusion path [36,37]. Therefore, the higher dislocation density formed during the first two passes of ECAP induced a much faster precipitation than in the sample processed by 2 passes of MDF, although the time elapsed at 220 °C was similar for the two samples. The precipitate fraction remained higher for ECAP than for MDF even after 6 passes.

Between 2 and 4 passes of ECAP, the dislocation density decreased considerably from $5.6 \pm 0.6 \times 10^{14} \text{m}^{-2}$ to $0.7 \pm 0.2 \times 10^{14} \text{m}^{-2}$, while the diffraction domain size increased from $110 \pm 30$ to $240 \pm 40$ nm. These changes in the microstructure indicate the occurrence of recovery/recrystallization after 2 passes ECAP. Indeed, former studies [38–40] have already revealed that dynamic recrystallization has a significant role in the grain refinement during SPD-processing at elevated temperatures. The present investigation showed that during the recovery/recrystallization occurred in the ECAP-processed AM60 samples between 2 and 4 passes the annihilation of (c) and (c+a) dislocations was more pronounced than for (a)-type dislocations due to the higher energy of the former dislocations. It is worth to note that although the dislocation density remained unchanged between 4 and 6 passes of ECAP, the diffraction domain size increased significantly. As the diffraction domains usually correspond to the subgrains in SPD-processed materials, the increase in their size without changing the dislocation density suggests the development of a more dense dislocation configuration in the subgrain boundaries between 4 and 6 passes of ECAP. Similar trend can be observed for the MDF-processed samples.

The most significant differences in the evolution of the microstructures for the ECAP and MDF-processed specimens were detected for low numbers of passes (up to 4 passes), while they tend to coincide for large numbers of SPD passes (see Figs. 2a, 6a and 7a and 8a). For 2 passes, ECAP resulted in a much higher dislocation density and precipitate fraction, as well as a smaller diffraction domain size due to the larger imposed strain. Besides the increase of the dislocation density, the grain size was refined to ~3 µm. Between 2 and 4 passes of ECAP, a very large decrease in the dislocation density and an increase in the diffraction domain size were observed which indicate a significant recovery/recrystallization. At the same time, for the MDF-processed sample the changes in the microstructure between 2 and 4 passes were less pronounced. The lower dislocation density after 2 passes yielded a smaller driving force for recrystallization compared to ECAP, therefore only a structural relaxation (e.g., recovery) of the dislocation structure might occur between 2 and 6 passes of MDF. This is confirmed by the unchanged grain size in the MDF-processed sample between 2 and 6 passes. Similar microstructure relaxation has already been observed for other SPD-processed materials [41,42]. At the same time, for the ECAP-processed specimen the grain size was reduced from ~3 µm to ~2 µm between 2 and 6 passes due to recrystallization. The higher precipitate fraction in the ECAP-processed sample might contribute to the smaller grain size as precipitates hinder the motion of grain boundaries, i.e., impede the grain growth during recrystallization.

The comparison of Figs. 6 and 9 reveals that the variation of the hardness is similar to the evolution of the dislocation density for the ECAP- and MDF-processed samples. Indeed, there was a sharp increase in the microhardness for the specimen processed by 2 passes of ECAP. Then, the hardness decreased between 2 and 4 ECAP passes while it remained practically unchanged between 4 and 6 passes, similar to the evolution of the dislocation density. For the specimens deformed by MDF, the hardness reduction between 2 and 4 passes is negligible despite the significant reduction in the dislocation density. This can be explained by the fact that not only the dislocation density but also the arrangement of dislocations influence the strength of materials. For instance, clustering of dislocations into low energy configurations (e.g., into dipolar walls) usually increases their hardening effect [43]. This arrangement of dislocations might compensate the softening caused by the decrease of the dislocation density, thereby resulting in an unchanged hardness between 2 and 4 passes of MDF. The similar tendency in the hardness and the dislocation density suggests that dislocations have a major contribution to the hardness of the samples processed by ECAP and MDF.

5. Summary and conclusions

Experiments were conducted for the comparison of the evolution of the microstructure in AM60 magnesium alloy during processing by ECAP and MDF up to 6 passes at 220 °C. The following conclusions were obtained from the results:

1. In the initial extruded sample, the area-weighted average grain size was determined as ~13 µm, which was refined to ~3 µm after 2 passes for both ECAP and MDF. In addition, the dislocation density increased and the diffraction domain size decreased due to SPD-processing. These changes were more pronounced for the sample processed by ECAP due to the higher strain and different loading conditions. Precipitation of an Al12Mg17 phase was also observed after 2 passes of SPD-processing. A much higher fraction of precipitates was observed for the ECAP-processed sample as the larger dislocation density accelerated the precipitation process.

2. Between 2 and 4 passes of ECAP, the dislocation density considerably decreased while the diffraction domain size increased which indicate recovery/recrystallization of the microstructure. For the MDF-processed specimen, only slight changes in the dislocation structure and the grain size were observed, suggesting that only a microstructure relaxation (e.g., recovery) occurred in this specimen. In the sample processed for 2 passes of MDF, the much smaller dislocation density yielded a lower driving force for recrystallization, compared to the ECAP-processed specimen.

3. After 6 passes of MDF and ECAP, the density and type of dislocations as well as the diffraction domain size became similar. Differences were found only for the grain size and the precipitate fraction. Namely, the amount of precipitates was higher while the grain size was smaller for the ECAP-processed sample. During recrystallization between 2 and 6 passes, the precipitates can hinder the motion of grain boundaries, thereby resulting in a smaller grain size for the ECAP-processed specimen.

4. Coincidence between the evolutions of the microstructure for the ECAP- and MDF-processed samples was not found, either studied as a function of the number of passes or investigated versus the equivalent strain. Therefore, our study demonstrated the significant dependence of the microstructure development in AM60 alloy on the method of SPD-processing, especially for low numbers of passes.

5. The variation of the hardness as a function of the number of passes showed similar trend than that for the evolution of the dislocation density, suggesting that dislocations have a major contribution to the hardness in the samples processed by ECAP and MDF.

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