Influence of high-pressure torsion on microstructure, hardness and shear strength of AM60 magnesium alloy

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The evolution of the microstructure, crystallographic texture and hardness of an AM60 magnesium alloy was studied in the center and the edge parts of disks processed by high-pressure torsion (HPT) technique at room temperature. In addition, the mechanical properties were also tested by shear punch test (SPT). The number of HPT turns varied between ½ and 10. It was found that the grain size of the initial extruded material (~16 μm) decreased to ~2 and ~0.8 μm at the disk center and edge, respectively, even after ½ turn of HPT. Ten turns of HPT resulted in further grain refinement to ~1 and ~0.23 μm at the disk center and edge, respectively. The dislocation density saturated even after ½ turn with the value of ~11 × 10¹⁴ m⁻². The maximum value of the hardness was ~1300 MPa that was measured at the edge of the disk deformed for ½ turn. Despite the practically unchanged grain size and dislocation density, the hardness decreased between 3 and 10 turns which can be explained by texture softening. A good agreement was observed between the yield strength estimated as one-third of the hardness and the values calculated from the dislocation density, grain size and texture. The difference between the microstructure and hardness obtained for AM60 samples processed by HPT and equal channel angular pressing (ECAP) is discussed.

1. Introduction

Magnesium alloys are frequently used in special applications, e.g., in automobile industry due to their low density and high specific strength. The mechanical strength of Mg alloys can be improved by severe plastic deformation (SPD) since this method introduces a large density of dislocations and also results in a grain refinement [1]. Among SPD methods, equal channel angular pressing (ECAP) [2,3], high-pressure torsion (HPT) [1,4-8] and multi-directional forging (MDF) [9] are the most commonly used procedures.

The microstructural refinement caused by SPD is quite beneficial for improving the mechanical properties of magnesium alloys [2,10,11]. Many previous studies observed remarkable grain refinement in SPD-processed magnesium alloys [1,2,12]. For example, a grain size reduction into the range of a few microns was observed in several magnesium alloys processed by ECAP [2,10,11]. Mabuchi et al. [13] performed ECAP on an extruded AZ91 magnesium alloy at 448 K with a total imposed strain of ~6.3. The results showed that the grain size was reduced to a very small value of ~0.5 μm. An exceptional grain refinement has been observed in AZ91 magnesium alloy processed by HPT at 296 K [14]. After 10 turns of HPT, a very fine-grained microstructure with an average grain size of ~35 nm was formed. It has been concluded that the grain size in magnesium alloys decreased gradually with increasing the degree of deformation until reaching a critical minimum grain size [12,15]. With further increasing the strain, the average grain size remained practically unchanged. Early studies reported that the processing temperature and the strain rate have also a significant effect on the fine-grained microstructures in Mg alloys processed by SPD techniques [2,16,17].

The evolution of the dislocation density in some Mg alloys processed...
by ECAP and MDF techniques was investigated in several previous studies [12,18,19]. It was observed that the dislocation density increased with the imposed strain until reaching a maximum value. After the achievement of this maximum, the dislocation density dramatically decreased due to dynamic recovery and recrystallization during further SPD-processing. It was also found that the maximum value of the dislocation density and the minimum grain size strongly depended not only on the type of Mg alloy but also on the applied method of SPD [20].

The alloying element content in Mg has an effect on the minimum grain size achievable by SPD, thereby influencing the mechanical properties of the as-processed alloy. In Mg alloys containing a high fraction of aluminum (~3 wt%), usually a secondary Al12Mg17 phase forms [12,21–23]. Then, the precipitates of this phase can hinder the motion of grain boundaries, leading to a smaller grain size in the SPD-processed Mg alloys [12,22,24]. Accordingly, the volume fraction of Al12Mg17 particles significantly influences the mechanical properties of the fine-grained Mg alloys processed by SPD [22]. For example, a recent study [22] on ultrafine-grained AZ31, AZ61 and AZ91 Mg alloys showed that AZ91 exhibits the highest tensile strength of ~370 MPa among the studied alloys which observation was attributed to the highest fraction of Al12Mg17 [23,25].

The crystallographic texture developed during SPD has a considerable effect on the mechanical properties of Mg alloys [9,26,27]. Usually, strong texture forms in Mg alloys during plastic deformation which depends on the way of SPD. In addition, the texture of the initial material also influences the microstructure, the texture and the mechanical properties of SPD-processed Mg alloys. It was documented that conventional rolling usually leads to a strong texture, resulting in a limited deformability during further rolling [17,28]. Textural investigations on MDF-processed AM60 alloy revealed that not only the texture intensity increased but also the type of texture changed during deformation [9].

AM60 is one of the most frequently used Mg alloys in practical applications. Akbaripanah et al. investigated the structural changes and the applications of the mechanical properties of this alloy after ECAP processing at 493 K [27,29]. A significant grain refinement from ~19.2 μm to ~2.3 μm was observed in the sample processed by ECAP up to 6 passes. Although the grain size decreased with increasing number of passes, the maximum strength of the ECAP-processed material has already been achieved after the second pass of ECAP. Additional increase of the ECAP passes from 2 to 6 led to a reduction in both the yield strength and the ultimate tensile strength, most probably due to the texture softening effect. A large improvement in the fatigue lifetime was also observed only after the second pass of ECAP. On the other hand, the sample processed for 6 passes of ECAP exhibited higher ductility and lower yield strength compared to the initial extruded specimen. In another study, Hecezel et al. [12] studied the mechanical properties of AM60 alloy processed by ECAP and MDF at 493 K. For both SPD processes, the grain size was refined ~3 μm even after two passes. Further grain refinement was not observed with increasing the number of MDF passes. At the same time, a slight reduction in the grain size to ~2 μm was detected after 6 passes of ECAP processing. The dislocation density significantly increased reaching maximum values of ~5.6 × 1017 m–2 and ~1.8 × 1014 m–2 after 2 passes of ECAP and MDF, respectively. Between 2 and 4 passes of ECAP, the dislocation density remarkably decreased to ~0.7 × 1014 m–2. Between 4 and 6 passes, the same values of the dislocation density were determined for the samples processed by both ECAP and MDF. In addition, the dislocation density in the grain size observed after 6 passes of MDF and ECAP, the amount of Al12Mg17 precipitates was higher in the ECAP-processed specimen. The above-mentioned researches have demonstrated the high potential of SPD for grain refinement and improving the mechanical properties of AM60 alloy. At the same time, to the knowledge of the authors the influence of HPT processing on the microstructure and the mechanical properties of this alloy has not been investigated in the literature yet.

In this paper, the evolution of the microstructure, texture and mechanical properties of an AM60 alloy processed by HPT at RT is investigated. The mechanical properties are studied by hardness measurement and shear punch test (SPT). SPT enables the investigation of the mechanical behavior of samples with limited size such as the HPT processed disks [30,31]. The experimentally measured strength is compared with the values calculated from the parameters of the microstructure. Moreover, the grain size, the dislocation density and the hardness obtained on the HPT-processed AM60 alloy are compared with the values determined previously for the same alloy processed by ECAP.

2. Materials and methods

2.1. Processing of materials

The material used in the present study was a Mg - 6 wt% Al - 0.35 wt% Mn (AM60) alloy. First, the constituent pure metals were melted in a graphite crucible in an electric furnace, and a layer of Foseco Magrex 36 covering flux was used in order to prevent oxidation. The casting method used for this alloy was a tilt-casting where the mold was pre-heated to 200 °C. The initial as-cast AM60 samples were obtained in cylindrical form with a diameter of 44 mm which were then used to create cubical samples with dimensions of 14 mm × 14 mm by extrusion at 380 °C [29]. Then, the wire cutting process was used to produce disk samples with a diameter of 10 mm and a thickness of 0.25 mm for HPT processing [4]. The HPT deformation was carried out at a pressure of 6 GPa and a rotational speed of 1 rpm for ½, 1, 3 and 10 turns at room temperature (RT). No visible damage or cracks were observed on the surface of the samples after HPT processing.

2.2. Characterisation of the microstructure

The microstructure of the HPT processed samples were studied by scanning and transmission electron microscopy techniques (SEM and TEM). SEM ZEISS Auriga Compact equipped with an EDAX electron backscatter diffraction (EBSD) camera was used for the characterization of the influence of HPT processing on the microstructure in the center of the specimens. For the EBSD investigations, the disks were mechanically polished. The polishing was finalized by using a diamond paste with a particle size of 1 μm. Additional ion-polishing was applied using a Leica EM RES102 system prior to the EBSD measurement. The step size of the EBSD scan was 100 nm. Owing to the severely deformed microstructure, the edge parts of the HPT disks were analyzed by Automated Crystal Orientation Mapping TEM (ACOM -TEM). Lamellae oriented perpendicularly to the surface of the disks were cut by a focused ion beam (FIB) technique. The ACOM-TEM was performed by a JEOL FX200 microscope. The step size was 10 nm. Both the raw EBSD and ACOM-TEM data were analyzed using TSL OIM 7.3 software made by EDAX. The normal vector of the ACOM-TEM lamellae was perpendicular to the surface normal of the HPT disk. Since the crystal orientations were intended to study parallel to the disk surface normal, the crystallographic directions of the grains were rotated by 90° in the ACOM-TEM images. Only points having confidence index (CI) > 0.1 were taken into account in the analysis. The grains were defined as the areas bounded by boundaries with the misorientations higher than 15°. Texture measurements were performed in both the center and the edge of the HPT disks using inverse pole figures obtained by EBSD and ACOM-TEM, respectively.

The dislocation density and the area-weighted mean diffraction domain size were determined by X-ray line profile analysis (XLP A) [32]. The X-ray diffraction (XRD) patterns were measured in the center and the edge of the HPT-processed disks by a rotating anode diffractometer (type: MultiMax-9, manufacturer: Rigaku, Japan) using CuKα radiation (wavelength: 0.15406 nm). The beam size had a rectangular shape with...
the dimensions of 0.2 mm × 2 mm. The diffractograms were recorded on imaging plates. The patterns were evaluated by the Convolutional Multiple Whole Profile (CMWP) fitting software [33]. This method is described briefly in the next paragraph.

In the CMWP fitting procedure, the experimentally determined XRD pattern is fitted by a theoretical diffractogram which is the sum of a background spline and the calculated diffraction peaks. For the calculation of the theoretical peak profiles, it was assumed that the peak broadening is caused by the finite size of crystallites (or diffraction domains) and the lattice distortion due to dislocations. Therefore, each calculated diffraction peak profile was obtained as the convolution of the ‘size profile’ and the ‘distortion profile’. In order to accelerate the fitting in the CMWP procedure, instead of the convolution of the different profile components, the intensity profile for each peak was calculated as the inverse Fourier transformation of the product of the Fourier transforms of the theoretical size and distortion profiles. In the case of size broadening, the Fourier transform of the profiles (A(f)) is given in analytical form as [32]:

\[ A(f) = \frac{1}{\sqrt{2\pi}} \exp \left( -\frac{f^2}{2} \right) \]

where \( m \) and \( \sigma \) are the median and variance of the assumed log-normal size distribution of the diffraction domains, respectively, \( L \) is the Fourier variable and \( \text{erfc}(\cdot) \) is the complementary error function given as:

\[ \text{erfc}(x) = \frac{2}{\sqrt{\pi}} \int_x^\infty \exp(-t^2) \, dt. \]

The Fourier transform of the distortion peak profile (\( A^D(f) \)) can be approximated by the following formula [32]:

\[ A^D(g, L) = \exp \left( -2\pi^2 L^2 g^2 \langle \varepsilon^2 \rangle \right), \]

where \( g \) is the length of the hkl diffraction vector (\( g = \frac{h}{\lambda} \) where \( \lambda \) is the Bragg-angle of the diffraction peak and \( \lambda \) is the wavelength of X-rays) and \( \langle \varepsilon^2 \rangle \) is the mean-square strain normal to (hkl) lattice planes which depends on both \( g \) and \( L \). For dislocations, the mean-square strain can be given as:

\[ \langle \varepsilon^2 \rangle = \frac{b^2}{4\pi^2} C f \left( \frac{L}{R_e} \right), \]

where \( b \) and \( R_e \) are the dislocation density and the magnitude of the Burgers vector, respectively, \( C \) is the contrast factor of dislocations and \( R_e \) is the effective outer cut-off radius of dislocations with length dimension. The function \( f \left( \frac{L}{R_e} \right) \) is given in Ref. [32]. Further details of the CMWP method can be found in [33].

2.3. Hardness testing

The hardness distribution along the diameter of the HPT-processed disks was determined by a Zwick Roell ZHU microhardness tester at RT using a Vickers indenter. The load applied for hardness measurement was 500 g while the dwell time was 10 s. In addition, the distribution of the microhardness on the whole surface of the disks was studied by an automatic Vickers microhardness tester Qness Q10a. In order to achieve a fine resolution in the hardness map, the load was reduced to 100 g. The dwell time was 10 s. A mesh having 540 measurement points was defined in advance for determining the hardness maps of the specimens.

The probe points were arranged into concentric circles. The radius of these circles increased with a step size of 0.4 mm. The spacing between the neighboring points along the arc for each circle was 0.4 mm. After the hardness test, the Cartesian coordinates of the measurement points were converted into polar coordinates. Then, a polar contour plot was drawn using Origin® software, where the ‘intensity bar’ indicates the hardness values. The Thin Plate Spline algorithm was used for the interpolation between the probed points in order to obtain a smooth contour plot.

2.4. Shear punch test

SPT test was performed in the center part of the disk at RT and a shear strain rate of \( 1.77 \times 10^{-2} \, \text{s}^{-1} \) with a punch diameter of 2.98 mm. In order to increase the accuracy, each test was repeated at least three times. The details of the SPT procedure can be found in Ref. [34]. The shear stress can be calculated from the punch force \( F \) using the following equation [35]:

\[ \tau = \frac{F}{4d\delta}, \]

where \( d \) is the punch diameter and \( \delta \) is the sample thickness. Using eq. (5), the shear stress was plotted as a function of the normalized displacement \( \delta \). The value of the normalized displacement was calculated as \( \delta = \frac{h}{2} \) where \( h \) is the displacement of the punch during SPT.

3. Experimental results

3.1. Evolution of the microstructure during HPT-processing

The phase composition in the center and the edge of the HPT-processed disks was determined from the XRD patterns measured by the rotating anode diffractometer. As an example, Fig. 1 shows the diffractogram taken on the center of the disk processed by 1 turn of HPT. In addition to the Mg matrix, the peaks of Al12Mg17 secondary phase were also detected. The amount of this phase is characterized by the intensity fraction of its XRD peaks. The intensity fraction was determined as the ratio of the sum of the areas under the Al12Mg17 peaks and the sum of the areas under all peaks appeared on the diffractogram taken in the diffraction angle range between 30 and 150°. These areas were determined after background subtraction. It is noted that the XRD intensity fraction is not equivalent to the volume fraction of the investigated phases as the unit volumes of the crystalline phases with different structures and chemical compositions usually scatter X-rays with different intensities. Nevertheless, the change of the fraction of precipitates during HPT-processing can be monitored using this quantity. The intensity fractions of the Al12Mg17 phase determined for the center and the edge parts of the HPT disks are listed in Table 1. In the center of
the disk processed for ½ turn, the intensity fraction of the secondary phase was ~0.8% which increased to ~1.5% at the edge of this sample. For 1 turn, the fraction of the Al\textsubscript{17}Mg\textsubscript{17} phase saturated at a value of 2.0 ± 0.3% in both the center and the edge of the disk which practically remained unchanged for higher numbers of turns if we take the errors of the intensity fraction values into consideration.

The present study revealed that HPT-processing yielded precipitation of Al\textsubscript{17}Mg\textsubscript{17} phase which indicated that the alloying element (i.e., Al) concentration in the initial, hot extruded Mg material was higher than the equilibrium solubility limit at RT. The precipitation was promoted by the fast diffusion along dislocations and grain boundaries for low number of turns (N = ½) the Al\textsubscript{17}Mg\textsubscript{17} phase fraction in the center part was lower than that at the edge due to the different imposed strains. For higher numbers of turns, the strain was enough even in the center part to achieve the saturation fraction of precipitates. Therefore, significant difference between the fractions measured in the center and the edge parts was not observed. It should be noted that due to the 2 mm height of the XRD beam, the Al\textsubscript{17}Mg\textsubscript{17} phase fraction obtained nominally for the center part must be considered as the average for a region in which the distance from the disk center varies between zero and 1 mm.

The initial extruded AM60 alloy had a uniform and equiaxed grain structure with an area-weighted mean grain size of ~16 μm as shown in Ref. [20]. The grain orientation maps obtained for the center and the edge of the HPT-processed disks by EBSD and ACOM-TEM, respectively, are shown in Figs. 2 and 3, respectively. The area-weighted average grain sizes determined from the images are listed in Table 2. For ½ turn, the grain size values in the center and the edge of the disk were ~2 and ~0.8 μm, respectively. In the center area, the grain size decreased to ~1.7 μm when the number of turns increased to one while at the edge the average grain size was reduced to ~0.31 μm. Further increase in the number of turns resulted in additional grain refinement in the center while at the disk edge only marginal changes were observed. After 10 turns, the grain sizes in the disk center and edge were ~1 and ~0.23 μm, respectively, i.e., homogeneous grain structure along the disk diameter was not achieved.

The high-angle grain boundary (HAGB) fractions for the center and the edge parts of the HPT-processed disks are listed in Table 2. The HAGBs were defined as the boundaries with the misorientations higher than 15°. The lower bound of misorientation for low-angle grain boundaries (LAGBs) was 2°. The fraction of HAGBs was as high as 47% even in the center of the disk processed only by ½ turn. Small differences were observed between the HAGB fractions determined in the center and the edge parts of the disks for all numbers of turns. Moreover, a decrease in the HAGB fraction was detected in the center with increasing the number of turns from 1/2 to 1. The highest value of 47% was achieved for the center of the disk processed by 1/2 turn of HPT (see Table 2).

The texture evolution in the disk center during HPT-processing is illustrated by the inverse pole figures (IPFs) shown in Fig. 4. Similar images are presented for the edge parts of the disks in Fig. 5. These IPFs in Figs. 4 and 5 were created from the EBSD and ACOM-TEM images, respectively, using the OIM software (see section 2). A former study showed that for the initial extruded sample in the main texture component the crystallographic direction 10T0 was parallel to the disk surface normal and the normal vector of the basal plane (crystallographic direction 0001) was lying in the disk plane [20]. In the center of the disk processed for ½ turn, where the strain is the lowest among the studied samples and locations, a remaining 10T0 texture component can be seen. A similar texture was detected in ZK60 Mg alloy after ½ turn of HPT [8]. At the same time, for a considerable fraction of crystallites, the crystallographic orientation differs from 10T0 (see Fig. 4a). With increasing the number of turns to one, the 10T0 texture disappeared, and instead a 0001 texture formed, i.e., the basal planes are aligned parallel to the disk surface in both the center and the edge after 1 turn of HPT (see Figs. 4b and 5b). This basal texture has been already observed in rolled and HPT-processed magnesium alloys [36,37], and it also appeared in Mg samples processed by rotational deformation routes such as friction stir processing [38]. For 3 and 10 revolutions, the 0001 texture gradually disappeared with increasing the number of turns.

The analysis of the XRD peak profiles provided the diffraction domain size and the dislocation density. First, the diffraction line breadth was investigated using the Williamson-Hall method [32]. In this procedure, the full width at half maximum (FWHM) of the peaks in the reciprocal space was plotted as a function of the magnitude of the diffraction vector (g). The FWHM and g values were determined as cosθ/Δ(2θ)/λ and 2sinθ/λ where θ is the Bragg angle, Δ(2θ) is the peak breadth in radians and λ is the wavelength of X-rays in nm unit. Fig. 6 shows the Williamson-Hall plots in both the center and the edge of the HPT disks for all numbers of turns. Considerable difference between the peak breadths of the different samples was not observed, suggesting that the microstructural parameters obtained from XLPA may differ only marginally. Indeed, the area-weighted mean diffraction domain size was found to be 60 ± 10 nm while the dislocation density was obtained as (11 ± 2) × 10\textsuperscript{14} m\textsuperscript{-2} using the CMWF fitting method, irrespectively of the number of turns and the location along the disk radius. As an example, Fig. 7 illustrates the fitting on the XRD pattern taken for the edge part of the disk processed by 3 turns of HPT.

### 3.2. Mechanical properties of the AM60 alloy deformed by HPT

The hardness distributions along the disk diameter for the different numbers of HPT turns are plotted in Fig. 8a. It should be noticed that the hardness of the initial extruded sample was ~650 MPa [12]. This value increased to ~980 MPa in the center of the disk processed for ½ turn. The error of the hardness values was about ±40 MPa. With increasing the distance from the disk center, the hardness increased and its value reached ~1300 MPa at the edge of the sample deformed for ½ turn. Further increase in the number of turns resulted only in moderate changes in the hardness distribution. In the disk center, the hardness increased to 1050–1100 MPa for 1–10 turns while at the edge its value slightly decreased to ~1100 MPa when the number of turns was enhanced to ten. It is worth noting that the hardness distribution curve for 1 turn appears slightly asymmetric about the disk center, however the hardness values measured at the same distance from the center of the disk is only 70 MPa which is within the experimental error range (±40 MPa).

Fig. 8a reveals a hardness enhancement with increasing the distance from the disk center which may be caused by the higher imposed strain. If only the torsional deformation is considered during HPT, the equivalent strain can be expressed as:

\[
\varepsilon_{eq} = \frac{2\pi N r}{h^3/5}
\]

where N is the number of turns, r is the distance from the center and h is the disk thickness. Fig. 8b shows the hardness as a function of the
equivalent strain for all the four numbers of turns. It can be seen that although the hardness monotonously increases with increasing the equivalent strain for each turn as expected, the data obtained for the different turns do not coincide. This means that the equivalent strain solely cannot describe the evolution of the hardness. Nevertheless, it is clear from Fig. 8b that the majority of the hardness increment took place up to the equivalent strain of about 20. Further increase in strain caused only a slight additional hardening for all numbers of turns. These types of microhardness evolution have been previously reported for various Mg alloys [8, 39]. It is obvious that the AM60 alloy exhibits a strain hardening behavior during the hardness evolution towards homogeneity where a constant limit of hardness is ultimately achieved within the disk. This strain hardening behavior was suggested according to a theoretical approach using strain gradient plasticity [40].

In order to assess the microstructural and strain distribution uniformity, Vickers microhardness measurements were also carried out on the total surface of the HPT specimens. The color-coded microhardness distribution maps are shown in Fig. 9. The hardness values in the maps are in good agreement with the microhardness measured along the disk diameters with a higher load (500 g while the maps were taken with 100 g). It is revealed that there is a variation of the hardness determined at the same distance from the center. Nevertheless, it is evident that the hardness distribution in the whole disk became more uniform when the number of turns increased.

The mechanical behavior of the HPT-processed AM60 alloy was also studied by SPT technique. This method has also been applied for other HPT-processed Mg alloys such as ZK60 [31] and Mg–9Gd–4Y–0.4Zr [41]. In the present study, the samples were fabricated from the center part of the HPT disks due to the dimensional requirements of the SPT technique. Fig. 10 shows the shear stress versus the normalized displacement for the different numbers of turns as obtained by SPT. The peak stress was achieved at the normalized strain of about 0.56. The maximum shear stress was ~148 MPa after ½ turn which increased to ~176 MPa when 1 HPT turn was completed (see Fig. 10). This value remained practically unchanged for 3 turns but between 3 and 10 revolutions the maximum shear stress decreased to 155 MPa. This trend resembles the hardness variation in the center part of the disks (see Fig. 8a).

4. Discussion

4.1. Microstructure and texture evolution during HPT-processing

The electron microscopy observations and the X-ray diffraction analysis suggest that an early saturation of the microstructure occurred during HPT-processing. Indeed, the grain size was refined from ~16 μm to ~2 μm and ~0.8 μm, respectively, in the disk center and edge even after ½ turn of HPT. In addition, XLPA revealed that the diffraction domain size decreased to ~60 nm while the dislocation density increased to ~11 × 10^{14} m^{-2} even in the center of the disk processed by ½ revolution. The dislocation density after HPT at RT is at least two orders of magnitude higher than the value in the initial extruded sample, ~10^{13} m^{-2} (c.f. Ref. [20]). The dislocation domain size and the dislocation density are almost constant and evenly distributed for all turns. In contrast, the grain size exhibited further refinement down to ~1 μm and ~0.23 μm in the center and the edge, respectively. It is noted that a similar level of grain refinement was reported earlier for a ZK60 alloy processed by HPT [8]. The very similar microstructural parameters obtained by XLPA in the center and the edge of the disks processed for
low numbers of turns (½ and 1) can be partly explained by the relatively large area studied by XLPA. Indeed, the area illuminated by X-rays was 0.2 × 2 mm², therefore the XLPA results obtained from the center must be considered as the average for a region in which the distance from the disk center varies between zero and 1 mm. Thus, the characterization of the microstructure by the present XLPA is less local than EBSD or ACOM-TEM.

In addition, it seems that the dislocation density saturated earlier than the grain size if the results obtained for the edge parts of the disks processed by different numbers of HPT turns are compared. At the edge, the XLPA microstructural parameters can be considered as very local information since in that case the width of the studied area along the disk radius was only ~0.2 mm. Even if the strain at the edge was very different for ½ and 10 turns, the microstructural parameters obtained by XLPA agreed within the experimental error. It should be noted that the significant difference between the values of the diffraction domain size determined by XLPA and the grain size obtained by EBSD and ACOM-TEM has been already observed for other SPD-processed materials and can be explained by the higher sensitivity of XLPA to misorientations. Therefore, XLPA gives the subgrain size or dislocation cell size while EBSD and ACOM-TEM are usually used for the determination of the dimension of the grains bounded by HAGBs. Thus, it seems that the saturation of the dislocation density occurred earlier with increasing strain during HPT than that for the grain size. Similar behavior has been observed for other SPD-processed materials [42, 43]. The saturation dislocation density (~11×10¹⁴ m⁻²) and diffraction domain size (~60 nm) achieved by HPT at RT for the present AM60 alloy were slightly higher and lower, respectively, than the values obtained for HPT-processed AZ31 magnesium alloy (~8×10¹⁴ m⁻² for the dislocation density and ~70 nm for the diffraction domain size) [44]. This difference can be explained by the higher alloying element content in AM60 alloy, since both solute atoms and precipitates hinder the annihilation of dislocations during SPD-processing [45], thereby resulting in a higher saturation dislocation density.

As it is obvious from Table 2, the HAGB fraction significantly decreased in the center when the number of HPT turns increased to one. This effect can be attributed to the development of LAGBs from the newly formed dislocations. Between 1 and 10 turns, only slight changes in the HAGB fraction was observed in the disk center. At large strains, it is expected that the LAGBs are transformed into HAGBs and there is a recrystallization, resulting in a grain refinement and an increase of the fraction of HAGBs. Indeed, at the edge parts of the disks where the imposed strain is much higher than in the center, the HAGB fraction is higher than in the center. However, it should be noted that these values

<table>
<thead>
<tr>
<th>Number of HPT turns and location along the disk radius</th>
<th>Grain size [μm]</th>
<th>HAGB fraction [%]</th>
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<tr>
<td>1/2 center</td>
<td>2.0 ± 0.1</td>
<td>47</td>
</tr>
<tr>
<td>1/2 edge</td>
<td>0.80 ± 0.05</td>
<td>43</td>
</tr>
<tr>
<td>1 center</td>
<td>1.7 ± 0.1</td>
<td>36</td>
</tr>
<tr>
<td>1 edge</td>
<td>0.31 ± 0.05</td>
<td>40</td>
</tr>
<tr>
<td>3 center</td>
<td>1.0 ± 0.1</td>
<td>36</td>
</tr>
<tr>
<td>3 edge</td>
<td>0.45 ± 0.05</td>
<td>40</td>
</tr>
<tr>
<td>10 center</td>
<td>1.0 ± 0.1</td>
<td>32</td>
</tr>
<tr>
<td>10 edge</td>
<td>0.23 ± 0.05</td>
<td>44</td>
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Fig. 3. Grain orientation maps obtained by ACOM-TEM for the edge parts of the samples processed for (a) ½, (b) 1, (c) 3 and (d) 10 turns.
Fig. 4. Inverse pole figures obtained by EBSD for the center parts of the samples processed for (a) $\frac{1}{2}$, (b) 1, (c) 3 and (d) 10 turns. The vertical color bar gives the intensity in the multiple of random distribution, where the value becomes unity when the grains are oriented randomly.

Fig. 5. Inverse pole figures obtained by ACOM-TEM for the edge parts of the samples processed for (a) $\frac{1}{2}$, (b) 1, (c) 3 and (d) 10 turns. The vertical color bar gives the intensity in the unit of multiple of random distribution (m.r.d.), where the value becomes unity when the grains are oriented randomly.
are lower than the HAGB fractions formerly observed for other Mg alloys processed by ECAP or MDF (e.g., about 80% for AX41 alloy processed by ECAP at 220 °C [46]). This difference can be explained by the hindering effect of the high pressure applied during HPT on the recovery of the microstructure, resulting in a relatively high dislocation density ($\sim 11 \times 10^{14} \text{ m}^{-2}$) and a large fraction of LAGBs. Most probably, the low melting point of the studied material and the very high strain applied during HPT caused a dynamic recrystallization during SPD processing. However, as this recrystallization occurred during severe deformation at RT, many new dislocations and LAGBs formed in the refined grain structure. Therefore, for high numbers of HPT turns the HAGB fraction only slightly increased.

The effect of HPT on AM60 alloy was also monitored by the measurement of the spatial distribution of microhardness. Fig. 8a shows that the hardness considerably increased from 650 MPa to 980 MPa even in the center of the disk processed by $\frac{1}{2}$ turn, although the equivalent strain is zero nominally in that place (see eq. (6)). This apparent contradiction can be explained by (i) the hardening effect of the initial compression applied in the beginning of HPT and (ii) the spread of plastic deformation into the disk center from the surrounding regions where the nominal strain is non-zero. Namely, the dislocations formed at a distance from the center have a long-range stress field, resulting in dislocation motion and multiplication even in the center. Thus, there is a strong decrease in the grain size (from $\sim 16$ to $\sim 2 \mu$m) and an increase in the dislocation density in the center, leading to a significant hardness enhancement. With increasing number of turns, the hardness distribution became more homogeneous since at the edge the microstructure saturation the hardness does not increase anymore while in the center further microstructural development occurs during further deformation. It should be noted that fully homogeneous hardness distribution was not achieved even after 10 turns of HPT as shown in Fig. 8a. This can be explained by the difference between the grain sizes observed in the center and the edge parts of the disks even after 10 turns (see Table 2).

It is noted that perfect hardness homogeneity along the disk diameter was also not observed for other HPT-processed Mg alloys [39,47]. However, the increase of the number of HPT turns can result in homogenization of the hardness along the disk radius, as shown in previous studies [37,44–49]. For room temperature HPT, a lower hardness in the center compared to the edge part has been observed for AZ31 alloy even after 5 turns, and a homogeneous hardness distribution was achieved only after 15 turns of HPT [44]. Another study revealed that the increase of the HPT temperature from room temperature to 200 °C yielded a more homogeneous but lower hardness along the disk diameter in AZ31 alloy processed for 5 turns [48]. For extruded AZ80 and ZK60 alloys, the homogeneity was achieved even after 5 HPT turns [37,47,49]. Most probably, the pressure and the temperature applied during HPT, and the initial state of the material (e.g., extruded or not) influence significantly the evolution of the microstructure and hardness in Mg alloys. In addition, the chemical composition must also have an effect on the homogenization of the microstructure and hardness in HPT-processed Mg alloys. Therefore, the homogeneous hardness distribution along the disk diameter can be achieved at different numbers of
turns, depending on the processing conditions and the material composition.

4.2. Correlation between microstructure, texture and mechanical properties

In order to reveal the effect of strain on hardness evolution, the microhardness versus the equivalent strain was plotted for different HPT turns in Fig. 8b. It can be seen that the saturation hardness for each disk was achieved at an equivalent strain of about 20–40, irrespectively of the number of turns. At the same time, the saturation hardness value decreased with increasing the number of turns which is in an apparent contradiction with the fact that both the grain size and the dislocation density saturated at the disk edge after 1 turn of HPT. This dichotomy may be caused by the effect of texture on the hardness, therefore the texture evolution during HPT is discussed in the next paragraph.

For Mg alloys, the easiest deformation mode at RT is the dislocation glide on the basal plane. Therefore, the crystallographic texture has a significant effect on the measured hardness and mechanical strength of these materials. For instance, if the loading axis of compression is close to the 10\(\overline{1}0\) or the 0001 direction in Mg, the Schmid factor of basal slip is very low, resulting in texture hardening. Although, hardness testing is not a compression along the loading direction, due to the flatness of the Vickers tip the texture effect on the hardness can be understood by considering only a simple compressive stress perpendicular to the surface of the HPT disk. Then, for \(\frac{1}{2}\) turn the weakening of 10\(\overline{1}0\) texture resulted in softening but after 1 turn of HPT the 0001 texture yielded

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Fig. 9. Microhardness maps measured under the load of 100 g for the disks processed for (a) \(\frac{1}{2}\), (b) 1, (c) 3 and (d) 10 turns.

Fig. 10. Shear stress versus the normalized displacement measured for the different numbers of HPT turns.
hardening again. This type of texture can be the result of the dominance of basal slip since its activation at RT is easier than that for non-basal slip systems [39]. For 3 and 10 turns, the 0001 texture gradually weakens, i.e., the change of the crystallographic orientation of grains softens the material. Then, the decrease of the saturation hardness at the disk edge after 1 turn can be attributed to a texture effect since the dislocation density and the grain size at the edge remained practically unchanged between 1 and 10 turns.

The effect of texture is also apparent in the variation of the maximum shear stress in SPT. The shear stress is mostly parallel to the SPT loading direction which is perpendicular to the surface of the HPT disks [50]. Therefore, for 0001 texture when the basal planes in the crystallites are perpendicular to the shear stress, the basal slip is very difficult. Thus, when 0001 texture formed in the center of the disk processed for 1 turn (see Fig. 4), this effect might have contributed to the large increase of the maximum shear stress between 1 and 1 turns as shown in Fig. 10. Between 1 and 3 turns, although the dislocation density remained unchanged and the grain size decreased from ~1.7 to ~1 μm, the maximum shear stress did not increase since the hardening effect caused by the reduction of the grain size was compensated by the softening due to the weakening of the 0001 texture. For 10 turns, the texture 0001 completely disappeared, resulting in softening as compared to the sample processed by 3 turns (see Fig. 10), even if both the grain size and the dislocation density remained unchanged in the disk center between 3 and 10 turns of HPT.

In our recently published paper on ECAP-processed AM60 alloy [20], the strengthening effects of grain size, dislocation density and texture were combined in the following formula:

\[
\sigma_y = \frac{M_T}{M_0} \left( \sigma_0 + kd^{1/2} + a M_b G \rho b^{1/2} \right),
\]

where \(\sigma_y\) is the yield stress calculated from the microstructure, \(\sigma_0\) is the friction stress (~30 MPa [51]), \(d\) is the grain size, \(k\) is Hall-Petch slope (~159 MPa μm\(^{1/2}\) [52]), \(a\) is a parameter depending on the existing dislocation slip systems (0.6 [20,52,53]), \(G\) is the shear modulus (~17 GPa), \(b\) is the magnitude of the Burgers vector of dislocations and \(\rho\) is the dislocation density. In SPD-processed AM60 alloy samples, most of the dislocations have \(\langle a >\) type Burgers vectors [12], therefore \(b\) is taken as 0.32 nm (corresponding to lattice parameter \(a\)). The influence of texture on the yield stress was taken into account by the factor \(M_T/M_0\) where \(M_0\) is the Taylor factor for random crystallographic orientation and \(M_T\) is the Taylor factor calculated on the basis of the texture in the studied specimens. The value of \(M_0\) is 4.5 [52] while \(M_T\) can be estimated as the reciprocal of the Schmid factor of the basal slip since this glide system gives the main contribution to plasticity at RT [53]. The Schmid factor of basal slip was determined from the EBSD and ACOM-TEM images using the OIM software. Fig. 11 compares the calculated yield strength with the values measured for the HPT-processed AM60 samples and determined previously for AM60 alloy deformed by ECAP and MDF [20]. The measured yield strength was obtained as one-third of the experimentally determined microhardness. It is well known that this is only an approximation of the yield strength since one-third of the hardness gives the flow stress corresponding to the plastic strain of 8% [54]. Fig. 11 shows the strength values obtained for both the center and the edge parts of the HPT-processed disks. In the case of the center parts, due to the extension of the X-ray beam the yield strength was calculated from the hardness averaged for the points along the disk diameter which are closer to the disk center than 1 mm (see Fig. 8a). An acceptable correlation between the calculated and the measured yield strength values was obtained.

4.3. Comparison between the HPT- and ECAP-processed AM60 alloys

In our recently published paper [20], the microstructure evolution in AM60 alloy during ECAP processing at 220 °C was studied. The reason of the elevated temperature of ECAP was the low level of formability of Mg and its alloys at RT which may cause early cracking and fracture during SPD, such as ECAP. In the case of HPT, the high applied pressure assures the integrity of the sample even at large strains due to the suppression of crack initiation and propagation. Therefore, HPT can be performed on Mg alloys even at RT without the failure of the specimen as proved in this study. Although the temperature of SPD processing was different for the previous ECAP and the present HPT, it is worth to compare the parameters of the microstructure and the hardness of the two SPD-processed AM60 alloy.

Fig. 12 compares the grain size, the diffraction domain size, the dislocation density, the intensity fraction of the Al\(_{12}\)Mg\(_{17}\) phase and the hardness in the saturation state for AM60 alloy processed by HPT at RT and ECAP at 220 °C. It can be seen that the dislocation density is about two times larger while the diffraction domain size is about half for HPT than for ECAP. This difference in the two microstructures is also revealed if the Williamson-Hall plots for HPT- and ECAP-processed samples are compared in Fig. 6. The larger lattice strain due to the higher dislocation density causes the higher slope of the fitted straight line for the HPT samples as compared to ECAP. The grain size is smaller by a factor of eight for HPT as compared to ECAP. The higher dislocation density and the smaller domain and grain sizes in the HPT-processed sample can be attributed to the lower temperature and the
higher applied pressure during SPD processing. Indeed, the higher deformation temperature facilitates diffusion which is a basic mechanism of climb necessary for annihilation of edge dislocations. In addition, the hydrostatic pressure applied during HPT hinders diffusion since it enhances the vacancy formation and migration enthalpies. Thus, the more difficult diffusion resulted in a higher saturation dislocation density and a smaller minimum achievable grain size. Then, it is not surprising that the hardness of the HPT-processed sample was about 30% higher than that for ECAP. Fig. 12 also shows that the fraction of Al12Mg17 secondary phase was about three times larger for the ECAP sample. This effect can also be explained by the slower diffusion during HPT since in the initial extruded material the intensity fraction of Al12Mg17 phase was only ~1% and this value increased during both HPT and ECAP due to precipitation. The higher temperature and the lower pressure promoted diffusion during ECAP more effectively, resulting in a larger increase of the secondary phase fraction (from ~1% to ~6%) as compared to HPT (from ~1% only to ~2%).

5. Conclusions

HPT was conducted on an extruded AM60 alloy at RT. The evolution of the microstructure, hardness and shear strength was studied as a function of the number of turns. The most important results are the following:

1. HPT at RT resulted in a strong grain refinement. The initial grain size of ~16 μm decreased to ~2 and ~0.8 μm in the disk center and edge, respectively, even after ½ turn of HPT. For the highest applied number of turns (N = 10), the grain size reduced to ~1 and ~0.23 μm in the disk center and edge, respectively, i.e., a fully homogeneous microstructure was not achieved. At the same time, the dislocation density saturated even after ½ turn with the value of ~11 × 10^14 m^-2.

2. The initial 10T0 texture gradually changed to 0001 texture when the number of turns increased to one. Then, the 0001 texture weakened after 3 and 10 turns of HPT. The latter effect can explain the softening observed between 3 and 10 turns by both hardness testing and SPT even if the grain size and the dislocation density did not change.

3. The hardness distribution exhibited also an inhomogeneous character, similar to the microstructure. Although the inhomogeneity decreased with increasing the number of turns, a significant variation in the hardness was observed even after 10 turns of HPT. It was revealed that the hardness versus equivalent strain curves for the different number of turns do not coincide, mainly due to texture softening occurred for high numbers of turns. The maximum achievable hardness was ~1300 MPa that was measured at the edge of the disk processed for ½ turn.

4. The yield strength calculated from the dislocation density, grain size and texture was in a reasonable agreement with the values determined experimentally as one-third of the hardness. The microstructure-strength relationship found formerly for ECAP- and MDF-processed AM60 alloy samples is also valid for the center and edge parts of the HPT disks.

5. The saturation grain size for HPT at RT was about eight times smaller than that for ECAP performed at 220 °C while the maximum achievable dislocation density was two times higher in the HPT-processed material. This difference can be explained by the retarding effect of the lower temperature and the higher pressure on the diffusion during HPT which then hindered the annihilation of dislocations. As a result, the hardness of the HPT-processed sample was about 30% higher than that for ECAP.

Credit author statement

A.A. Khaleghi: Methodology, Data processing, Original draft preparation, Investigation.

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Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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