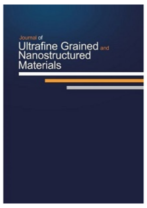

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The effect of twinning on texture evolution during ECAP processing of AM30 magnesium alloy

S. M. Fatemi*[*,1], A. A. Kazemi Asl, A. Abedi

[1]Department of Metallurgy and Materials Engineering, Shahid Rajaee Teacher Training University, Lavizan, Tehran, Iran.

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** Corresponding author email: mfatemi@sru.ac.ir*

ABSTRACT

An AM30 magnesium alloy was processed through ECAP method at 200 °C. Optical and transmission electron microscopy as well as electron back scattered diffraction (EBSD) technique were employed to characterize the deformed microstructure. A partially recrystallized microstructure including ultrafine/nano structures was obtained. The area fraction of 49% was measured for the recrystallized regions, where grains of 95 nm to 1 micron were detected. X-ray diffraction pole figure measurements were conducted to assess the final texture. The processed material exhibited moderate randomized texture including three basal pole peaks denoting the effect of dynamic recrystallization on deformation texture. TEM analysis confirmed the contribution of nano twinning during deformation of AM30 alloy. EBSD characterization implied that extension twin may contribute to the nucleation of new grains and thereby to be related to texture evolution. The apparent crossing pattern of intersected extension twins provided fine domains with high angle grain boundaries, which act as DRX nuclei. The formation of intersection boundaries also plays a critical role in pileup of dislocations and provide a local high stored-energy for dynamic recrystallization nucleation site. The aforementioned recrystallization mechanisms generally adopt orientations which are different from those produced by conventional recrystallization mechanisms, and thus it offers a route to manipulate the texture.

Keywords: *recrystallization; texture; twinning; magnesium.*

1. Introduction

Magnesium alloys as the lightest engineering alloys offer a wide range of opportunities to automobile and aerospace industrie. Nevertheless, as a consequence of their hexagonal close packed (HCP) structure they generally present limited ductility and strength at low temperatures. In order to employ the capability of magnesium alloys, it is important to develop a variety of secondary processing routes to enhance their mechanical properties. It is well established that microstructural grain refinement may assist to overcome the limited mechanical properties magnesium alloys. Accordingly, numerous investigations have been conducted to generate ultrafine grained magnesium alloys by employing severe plastic deformation (SPD) techniques. However, results showed that the combined effects of grain refinement and crystallographic texture changes dictate the post-SPD mechanical properties.

Fundamental investigations illustrated that the deformation texture developed during SPD is usually completely replaced by recrystallization texture dynamically occurred during deformation

[1]. Thus, mechanisms of dynamic recrystallization and respective texture alteration may effectively influence the final texture. In this regards, it has been known that a strong basal texture deteriorates final mechanical properties, particularly formability of magnesium alloys. Accordingly, considerable effort is currently being directed to weaken the basal texture or obtain a random texture in Mg products. Hence, it is desirable to identify the microstructural mechanisms by which the basal texture of the deformed material may be alleviated. To promote the latter mechanisms, one can design the alloying contents (e.g. [2]) and/or deformation parameter [3, 4] to elaborate final texture of SPD-processed materials. Certain recrystallization mechanisms have been known to induce a desired texture changes during deformation of magnesium alloys, like shear band nucleation [5], and particle stimulated nucleation [6].

The literature presents a complex view of the mechanisms by which dynamic recrystallization (DRX) operates in magnesium alloys. Different types of discontinuous [7] and continuous DRX [8, 9] and twin DRX [8] have been reported under different conditions of conventional deformation processes, even for a given alloy. Tan and Tan [10] studied the DRX behavior of AZ31 alloy, and suggested that the grain refinement could be attributed to continuous dynamic recrystallization (CDRX). extension and contraction twins are the most commonly observed twin types in magnesium alloys. Contraction twins are much harder to nucleate due to energetic prospects of atomic shuffling [11]. Twinning reorients the crystal such that a mirror symmetry relative to the parent grain is created. In contrast to slip, twinning provides shear strain via re-orientation of the lattice. Such reorientation in turn drastically modifies the crystallographic texture. Nevertheless, it was illustrated that contraction twins do not cause remarkable texture changes during deformation, due to their low volume fraction [12]. Extension twins were observed to contribute at low strains and grow gradually with increasing cumulative strain, eventually encompassing the whole grain [13]. Recrystallization mechanisms related to various twin types, twin variants, twin-twin and twin-grain boundaries intersections were precisely determined during annealing [14]. Moreover, the results showed that the orientation characteristics in local regions during nucleation of new grains are similar to those of subgrains within compression twins during static annealing [15]. However, in the literature little information can be found on the influence of twins on DRX during hot deformation of magnesium. Al-samman studied the occurrence of twin DRX during conventional compressive deformation of AZ31 alloy [1]. The results implied that the orientation relationship of the recrystallized twins with respect to the parent grains and the neighboring twins is so that *c*-axes of the parent grains were scattered around the compression axis within 30-70°. The mechanism of DRX in twins was found to be of continuous nature, involving the formation of low angle boundaries and their conversion to high angle boundaries forming new fine grains. However, few results were presented in the literature dealing with the possible role of twins during severe plastic deformation of magnesium alloy.

In this research, the contribution of twinning to texture evolution occurred during SPD processing of an AM30 magnesium alloy has been investigated. Toward this end, microstructural observations using electron back scattered diffraction (EBSD) as well as transmission electron microscopy (TEM) were made.

2. Experimental procedure

The initial material was hot-extruded AM30 (Mg-2.85% Al-0.6% Mn, wt.%) magnesium alloy exhibiting ring basal texture. Considering the ED (extrusion direction), ND (normal direction) and TD (transverse direction) axis, samples of square cross section of 12 mm × 12 mm × 100 mm were extracted from the initial material. The samples were deformed in an ECAP die with inter-channel angle 90° without rounding of corners, where an effective strain of 1.17 per pass could be imposed. The samples were well lubricated using MoS_2 spray and then inserted in the ECAP die with the RD in the front and the ND down. ECAP was carried out following the route B_C. Experiments were carried out at 200 °C. The billets were preheated to the experimental ECAP temperature for 10 minutes before entering the first channel of the die through the inlet. As twins would contribute significantly during severe deformation of initial coarse grained material, the alloy processed up to different stages of the first pass mainly studied in the present work. While during the consequent passes, the occurrence of twinning is remarkably limited, due to fine grained microstructure already developed in the microstructure. Microstructure and texture of the ECAPed material were characterized by Transmission and Electron Microscopy (TEM) Electron Back Scattered Diffraction (EBSD) analysis performed on a Field Emission Gun - Scanning Electron Microscope (FEG-SEM). EBSD was performed on samples along the initial ED-ND plane. The samples for EBSD analysis were initially ground using emery papers to #4000, followed by pre-polishing using Al_2O_3 (0.3 mm) suspension, and final polishing on a Struers Rotopol-15 automatic polishing machine using OPS suspension (0.04 mm sized SiO_2 particles). The EBSD data processing was carried out with TSL software. New recrystallized grains were characterized by their size and morphology using optical and SEM micrographs.

3. Results and discussion

To study the development of crystallographic texture for the processed material the basal, prismatic and pyramidal pole figures (PFs) of the sample deformed by single ECAP pass at 200 °C were obtained and presented in Fig. 1. The corresponding maximum intensity was presented in figures as multiples of random density (mrd). It is worth mentioning that the ECAP technique could establish the corresponding deformation texture during the single pass processing [16]. However, moderate texture randomization is realized form the obtained texture results. PFs in Fig. 1 indicates that a three- pole peak configuration was formed for the basal plane normal. Two of the basal pole peaks lies 25° and 40° inclined to the ECAP direction (ED) and the other almost parallel to the transverse direction (TD) in the TD–ND plane, while the prismatic and pyramidal planes were revealed to be randomly distributed. These texture components generally place the magnesium crystal inclined to the ECAP axis. Accordingly, the basal planes shows discrepancies from the predicted morphological texture i.e., parallel to the shear plane [17]. The latter recalls the fact that the deformation texture may be influenced by the recrystallization texture. This texture alteration is closely related to the microstructural mechanisms. In contrast to the present results, Mostaed et. al. [18] observed that basal texture weakens by ECAP deformation on ZK60 magnesium alloys at 250 °C. The presence of Mg-Zn-Zr second phase particles of 1 to 2 μm sizes in the alloy could take a critical role in weakening the texture through inducing particle stimulated nucleation (PSN). The effect of PSN on randomizing the texture was thoroughly discussed by Ref. [19].

Figure 2a shows the typical ultrafine/nano structure obtained after the single ECAP pass.

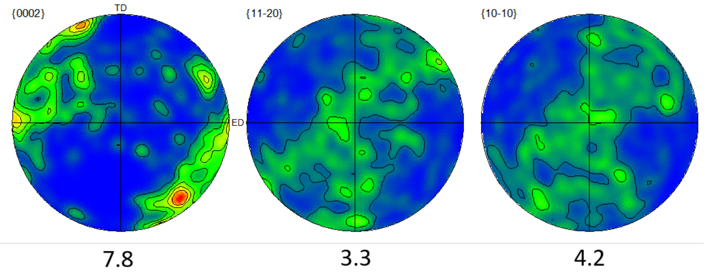

Fig. 1- (0002), (10–10), and (10–11) texture pole figures of the experimental material after one-pass ECAP (The corresponding maximum intensity was presented as mrd).

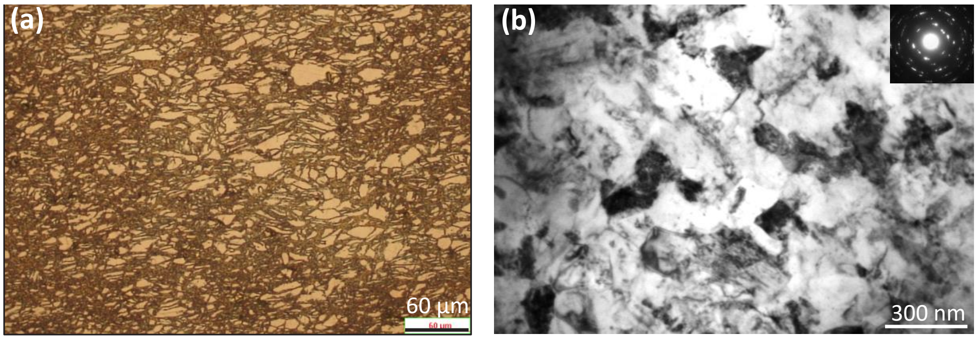

Fig. 2- a) Microstructure of AM30 magnesium alloy, single-pass ECAPed at 200 °C, b) typical nano structure developed during deformation.

Dynamic recrystallization (DRX) has significantly influenced grains morphology, resulting in an area fraction of 49%. The recrystallized area (dark regions in Fig. 2a) includes ultrafine and nano grains of 95 nm to 1 micron sizes with a mean size of 0.2 µm (Fig. 2b).

Optical results implies that twinning contributes to the evolution of microstructure of the AM30 alloy to accommodate severe deformation imposed through ECAP. TEM investigations revealed that nano twinning also plays an important role in the coordination of deformation during ECAP process. Fig. 3 shows a typical nano twin formed in the alloy deformed to one pass ECAP. It can be seen that the dislocation density is extremely high and dislocation tangles are bounded by of twin boundaries. To study the contribution of twinning on texture evolution, the microstructure taken from the material deformed to early stages of ECAP was subjected to EBSD analysis. After first ECAP pass, nearly all the twins activated were extension twins. The presence of extension twins could be attributed to their low critical resolved shear stress (CRSS) in the range of 2–3 MPa [12]. Typical orientation analysis provided in Fig. 4b, present common pole between the twinned area and parent grain is common accompanied with an 86° rotation of the basal pole across the twin boundary. These observation confirmed that the twins formed in the alloy are of extension type. The latter observation is further evaluated considering the misorientation profile taken crossing the twin band. Based on the observation made in previous works [20, 21], it is suggested that the concomitant events of dislocation slip and dynamic recovery within the twinned areas cause most of the extension twins to be consumed by dynamic recrystallization where orientations are much different from the developed parent orientation. The inverse pole figure map revealed that the parent grain with off-basal may provide sufficient non-basal activity in

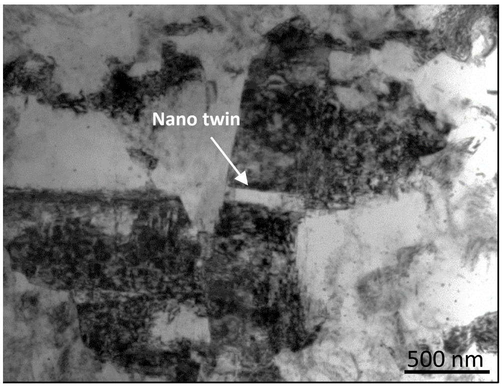

Fig. 3- Formation of nano twins in AM30 magnesium alloy during ECAP.

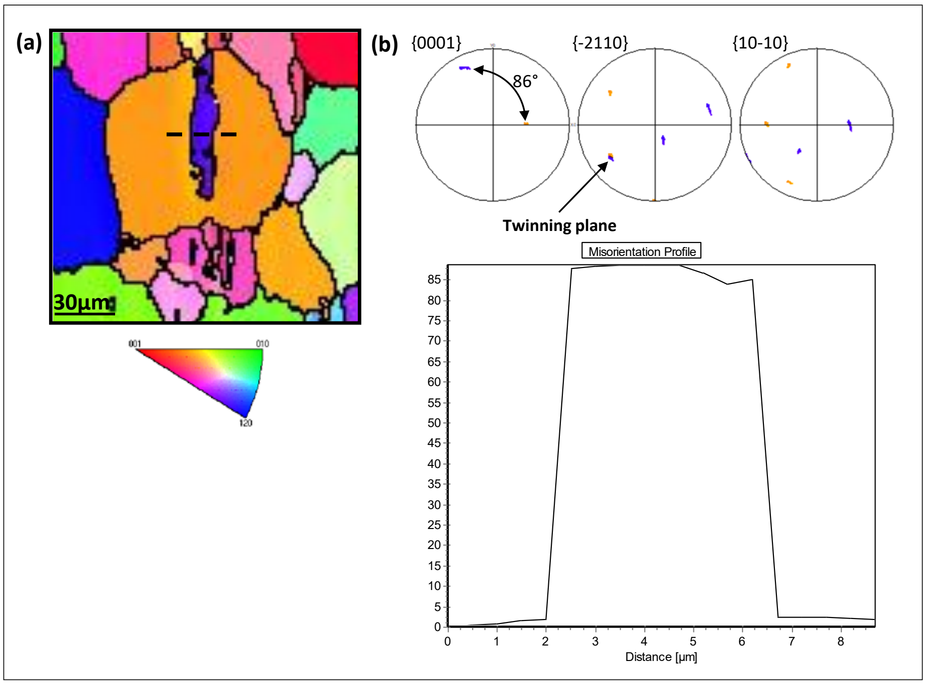

Fig. 4- a) IPF of a region including deformed twin, b) related {10-12} pole figure, and misorientation profile across the twin segment denoted by dashed line in (a).

the twinned area. It is noteworthy that the stacking fault energies (SFEs) of the prismatic/pyramidal planes [22] are high (265 mJ m^{-2} and 344 mJm^{-2} respectively). Therefore, cross-slip might prevail, leading to recovery over recrystallization in these planes. Our analysis is corroborated by a previous work [23], which mentioned that at and above 200 °C, continuous dynamic recrystallization is promoted during ECAE of Mg alloys. Accordingly, subgrains divide twins and secondary twins into small regions, in which recrystallized grains usually nucleate due to high storage energy. Moreover, following deformation can demolish the coherency of twin boundaries and supply a large density of dislocations, thus offer a larger driving force than the parallel twins, suggesting more effective nucleation sites for recrystallization [21, 24].

Microstructural observations made using EBSD technique implied that some twin segments locate in grain interiors, arrowed in Fig. 5a. These segments generates geometrically a new grain nucleus. This type of geometrical type nucleation has been previously recognized near prior grain boundaries and termed as "twin-assisted" recrystallization [21]. For the latter nuclei, as is obvious in the pole figure (Fig. 5b), the twinning plane is again (10-12). The misorientation profile taken across the twin band showed a near 86° rotation in the basal planes of twinned area in comparison to the parent grains. The amount of observed texture rotation is matched to the rotation associated with {1012} extension twinning (86°). This result present an alternative mechanism for the formation of new grain introduced by twinning. An important feature of the grains nucleated from the twin segments is that they generally adopt orientations which are different from those produced by conventional recrystallization mechanisms, and thus it offers a route to manipulate the texture. This is particularly interesting for magnesium alloys to provide texture weakening during thermomechanical process.

Careful observation revealed the intersections of the twins in some particular regions, as typically shown in Fig. 6a. Orientation analysis provides an apparent proof that the twins are extension type. Three different misorientation relationships can be achieved between different variants of extension twins and these are 60° <10-10>, 60:4° <8-1-70> and 7.4° <1-210> [25]. The intersection between different twin variant pairs could retard the twin growth and then trigger new twins near the intersection region. As observed in Fig. 6a. The apparent crossing pattern of intersected twins provide fine domains with high angle grain boundary, which act as DRX nuclei. Some examples are indicated by black arrows in Fig. 6a. The formation of intersection boundaries plays a critical role in pileup of dislocations and could cause a tilt of the crystal in the intersection region. Via TEM analysis, Sun et al. [26] confirmed that such

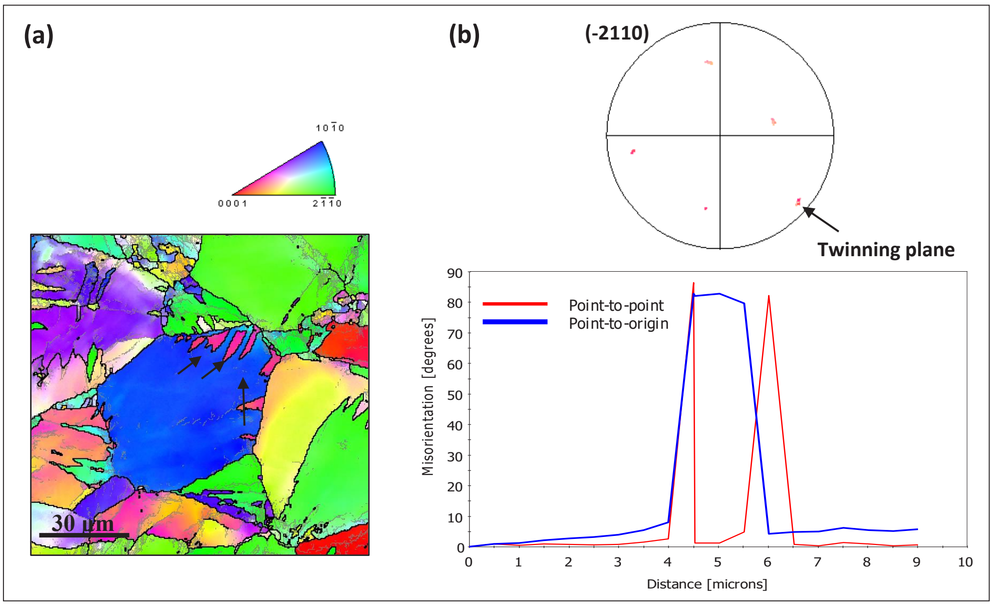

Fig. 5- a) IPF of a magnified view of a region including twin segment, b) related {10-12} pole figure, and misorientation profile across the twin segment denoted by dashed line in (a).

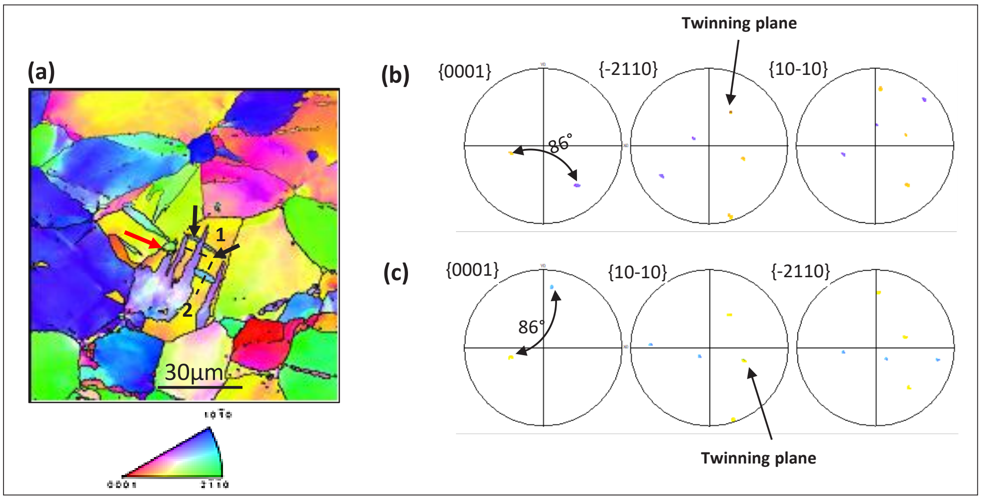

Fig. 6- a) IPF of a region including intersected twin, b and c) related {10-12} pole figure across the twin denoted by dashed line in (a) as (1) and (2), respectively.

intersections make a very large deviation from the original coherent twin boundary. It implies that the twin interaction regions offer a local high stored-energy for DRX nucleation site. An example of the new grains formed through the latter mechanism is arrowed in red in Fig .6a. The rearrangement of dislocations within the twins further subdivided the twin band into non-equilibrium sub-grains, and then converted into the grains with high angle boundaries increasing strain [21].

4. Summary

A wrought AM30 magnesium alloy was processed through ECAP. Ultrafine/nano grains formed in the processed microstructure. Texture analysis showed that basal texture with two or three peak poles was developed during ECAP processing. Contribution of nano twins to the microstructural evolutions was confirmed. Twin segments were traced at initial stages of deformation which are supposed to act then as recrystallization nuclei. Moreover, twin intersections were observed in the deformed microstructure, where new grains develop in the intersected area. These grains adopted orientations different to the parent texture. The twinning contribution during deformation and related following recrystallization may assist randomization of ECAP texture of the AM30 alloy.

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