

## Evolution of texture in an ultrafine and nano grained magnesium alloy

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### Abstract

The evolution of texture was discussed during the formation of ultra-fine and nano grains in a magnesium alloy severely deformed through accumulative back extrusion (ABE). The microstructure and texture obtained after applying multiple deformation passes at temperatures of 100 and 250°C were characterized. The results showed that after single ABE pass at 100°C an ultrafine/nano grained microstructure was obtained, while the initial texture was completely replaced by a new fiber basal texture, inclined at 40° to the transverse direction. As the processing temperature increased to 250°C, the obtained texture intensities were strengthened, though the c-axis of crystals gradually rotated towards the transverse direction and a <10-11> fiber texture parallel to normal direction was developed. Moreover, repetitive ABE was associated with the tendency of the basal plane to lie parallel to TD, while the orientation of the prismatic planes showed a random distribution around ND. After eight passes, the most noticeable texture obtained included the fiber basal texture oriented almost parallel to the transverse direction, and <10-10> perpendicular to the ED and <10-11> parallel to the ND. The maximum texture intensity decreased as the number of passes increased, which is attributed to strain path change involved during each consecutive ABE pass, as well as promoted the contribution of non-basal slip systems.

**Keywords:** *magnesium, nano recrystallization, texture.*

### 1. Introduction

Magnesium alloys are expected to be one of the most promising structural materials for application in aerospace and automotive industries because of their low density and high specific strength. However, magnesium alloys exhibit poor formability and limited

ductility at room temperature, ascribed to their hexagonal close-packed (HCP) crystal structure. In order to exploit the benefits of magnesium alloys, there is considerable current interest in processing magnesium samples through procedures involving the

imposition of severe plastic deformation (SPD) [1]. Previous researches have shown that the processing of magnesium alloys by SPD processes is challenging and the related property enhancement is limited by three main factors: 1. the processing of many magnesium alloys are relatively difficult at low temperatures, owing to the low symmetry hexagonal crystal structure, resulting in the development of substantial cracking or segmentation, 2. SPD processing should be carried out at high temperatures to prevent cracking. As a result, the occurrence of dynamic recrystallization and growth occur inevitably and thereby may diminish the grain refinement effect, 3. The post-SPD mechanical properties result from combined effects of grain refinement and crystallographic texture changes. A discussion on SPD textures is complicated, since many factors influence texture development.

These factors can be broadly categorized as those related to i) the deformation condition, ii) material parameters (microstructural mechanisms), or iii) the starting texture. It has been reported that the effect of grain size on the strength and ductility of magnesium is challenging due to changes in the texture [2]. Some processing conditions have been reported to result in lower post-processing strength and/or ductility, as compared to the starting commercial material [2, 3]. Furthermore, it has been shown that the degree of grain refinement by SPD, itself, is strongly coupled to the development of texture and substructural evolutions. It has been proposed that grain refinement is primarily the result of the interaction of shear plane with texture and the crystal structure, with a secondary role coming from the accumulation of redundant shear strain during severe deformation [4]. Therefore, it is indispensable to realize the texture evolution developed during severe deformation to enable the optimization of materials and processing parameters for desired properties. Also, it assists in analyzing the final mechanical properties, e.g. prediction of anisotropic mechanical behavior and in design for better control and energy efficiency during the deformation processes. A new continuous SPD process so-called accumulative back extrusion process (ABE),

which is appropriate for mass production, has been recently introduced by recent authors [5]. It has been shown that ABE is effective in reducing the grain size of AZ31 magnesium alloy [5,6] and pure copper [7] down to submicrometer ranges. In previous studies, the authors dealt with the developed strain patterns, microstructural evolutions and homogeneity during ABE processing. The present work was initiated to determine the deformation texture evolution which is enforced by ABE processing in wrought AZ31 magnesium alloys, subjected to various processing conditions. Moreover, the effect of processing temperature as well as the number of passes has also been evaluated.

## 2. Experimental Procedure

The composition of the commercial magnesium alloy AZ31 used in this study was Mg-2.9Al-0.85Zn-0.7 Mn (wt. %). This alloy was received in the form of hot rolled plates of 22 mm thickness. The initial material showed a mean grain size of 25  $\mu\text{m}$ . The texture of the initial material is illustrated in Figure 1, which shows basal planes aligned parallel to the RD-TD plane with a strong fiber texture. This is further confirmed, considering the high relative intensity of X-ray diffraction for the basal plane. Cylindrical samples for ABE experiments were machined with 18 mm diameter and 8 mm height, the deformation axis was selected to be parallel to the initial rolling. The billets lubricated with MoS<sub>2</sub> paste were inserted into the die, and held for 5 min until they reached the processing temperature. The experiments were conducted at 100 and 250°C up to eight passes at a ram speed of 25 mm/min. According to the relatively low stroke speed, the increase in temperature during extrusion was ignored. The experimental ABE procedure has been previously described in detail elsewhere [5]. Previous work showed that an average equivalent strain of 2 is induced during each step. The specimens for microstructural observations were cut parallel to their longitudinal axis. For all the samples, the center of rectangular cross section was considered for representative metallographic observations. The microstructures of the deformed material were examined through

optical microscopy, as well as field emission scanning electron microscopy. The electron microscopy study was carried out using a Zeiss Ultra Plus microscope operated at 10 kV. The dynamically recrystallized grains could be almost readily distinguished from the pre-existing grains by size and morphology. The size and fraction of recrystallized grains were determined using a powerful image analyzing system.

Crystallographic texture measurements were conducted using X-ray diffraction in the reflection geometry with a four circle goniometer and Cu K $\alpha$  radiation. Experimental data (10-10), (0002), and (10-11), and pole figures were collected on a 5° $\times$ 5° grid for sample tilts,  $\alpha = 0-85^\circ$ , and azimuthal rotations,  $\phi = 0-355^\circ$ . Background and defocusing corrections were made using experimentally determined data from random powder samples. The texture samples were sections from the flow plane (ED-TD plane) and the texture measurements of all the samples were made

parallel to the extrusion axis. The X-ray spot was focused on the more uniformly deformed central portion of the samples.

### 3. Results and Discussion

Figure 2a shows the typical microstructure obtained after the first ABE pass at 100°C, where an inhomogeneous structure with elongated and recrystallized grains was observed. Dynamic recrystallization (DRX) has significantly influenced the grain's morphology, having an area fraction of 70%. The recrystallized area includes ultrafine and nano grains of 80 nm to 1 micron sizes with a mean size of 150 nm (Fig. 2b), the non-recrystallized grains, however, have a size of about 25  $\mu\text{m}$ . As the temperature increased, the mean size and area fraction of recrystallized grains also increased [6]. The effect of temperature on macrostructure evolution has been discussed by the present authors in details in another study [6].

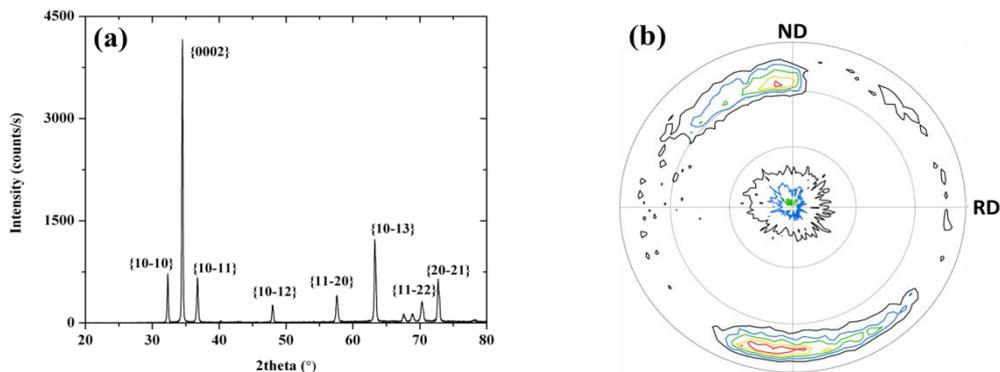


Fig. 1. a) The X-ray diffraction analysis, b) (0002) pole figure of the experimental alloy in the as-received condition.

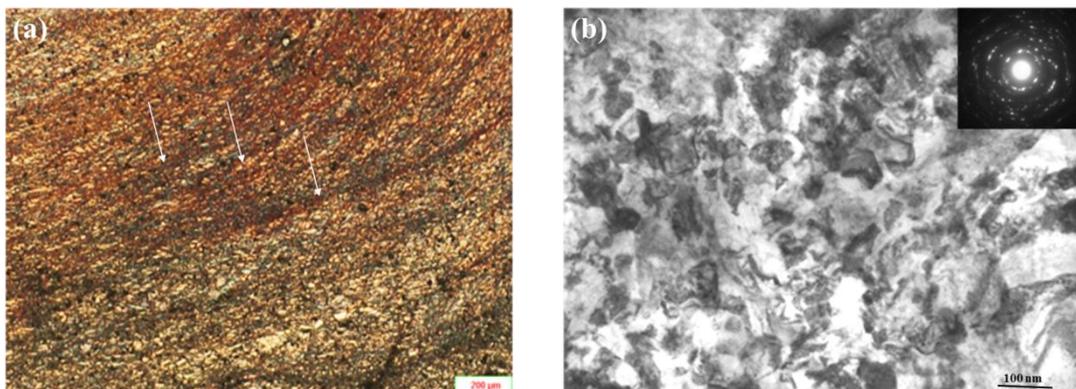
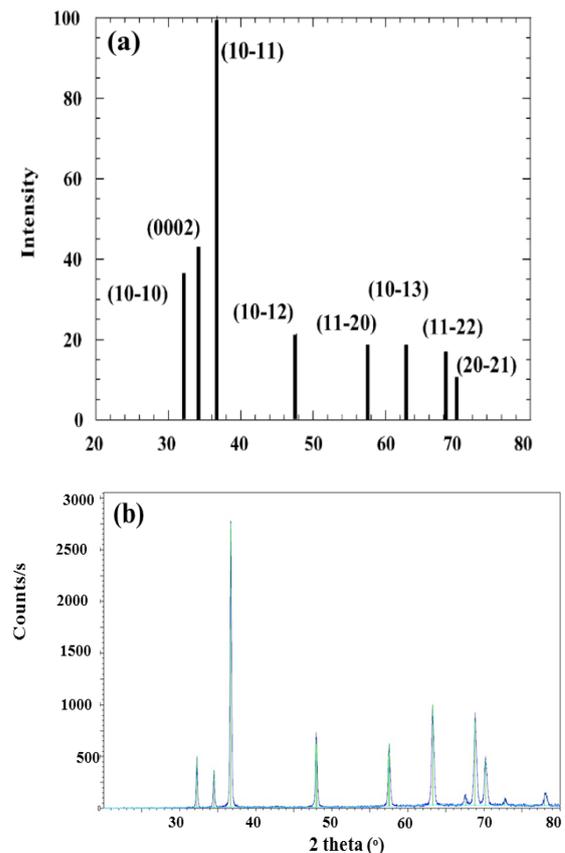


Fig. 2. a) Microstructure of the experimental alloy ABE processed at 100 °C, b) TEM microscopy of recrystallized grain structure.

XRD diffraction pattern of the experimental material after single pass ABE is shown in Figure 3. As shown, 10-11 pyramidal planes introduced high diffraction intensity, denoting that they are mainly aligned parallel to the extrusion plane. It can be realized by comparison of the relative intensities with that of randomly oriented texture in magnesium (Fig. 3a). Figure 4a depicts the basal, prismatic and pyramidal pole figures of the sample deformed by single ABE pass at 100°C. The corresponding maximum intensity was presented in figures as multiples of random density (mrd). As is realized the original texture was completely replaced and a new texture developed during the first pass. It is worth mentioning that ECAP technique hardly could establish the corresponding deformation texture during the single pass processing [2]. This is connected to the high shear strain magnitude imposed during ABE deformation. The obtained ABE texture corresponds to the basal poles lied  $\sim 40^\circ$  away from the transverse direction (TD) in ED-TD plane and  $66^\circ$  from the normal direction (ND) in TD-ND plane, while the maximum of prismatic planes was revealed at  $40\text{-}50^\circ$  in the TD-ED plane and that of pyramidal plane was inclined around  $30^\circ$  from TD. These texture components mostly place the magnesium crystal inclined to the ABE axis. Accordingly, the basal planes are oriented nearly coincident with the predicted morphological texture that is parallel to the “shear plane”, identified by finite element simulation (FEM). The FEM results positioned the shear plane inclined at  $40\text{-}50^\circ$  to the extrusion axis [8].

The changes in crystallographic orientations that take place during deformation are a consequence of the fact that deformation occurs on the most favorably oriented slip or twinning systems. It causes the deformed metal to acquire a preferred orientation or texture. The obtained post-ABE texture is also comparable to ideal orientations for hcp materials in simple shear, mapped systematically in theoretical studies [9], where five fibers were defined in Euler space for an applied shear. The post-ABE texture may be discussed relying on the presence of favorable initial basal texture, where the c-axis is mostly oriented by  $40\text{-}50^\circ$  from shear direction during ABE [8]. Thus, the starting orientation places the crystals in a

favorable orientation for basal slip with respect to the ABE shear plane. This provides a high schmid factor for basal slip systems, promoting the development of basal texture nearly parallel to the shear plane. However, there is a  $\pm 10^\circ$  deviation between the inclination of the fiber basal texture and the FEM-predicted shear plane angle in a previous work [8]. It should be noted that continuum theory calculations employed during FEM simulation are expected to only approximate the distortions of actual grains in a polycrystal. Also, it should be noted that no monoclinic texture symmetry of simple shear deformation [9] could be realized in the present texture results. This may be related to the fact that in reality, the grains do not deform as homogeneous simulation elements but along specific planes and directions governed by their crystal lattice. Moreover, due to material variation, such as initial texture and grain distribution topology, the shapes of the deformed grains will assume different inclinations.



**Fig. 3. XRD diffraction pattern of a) ideally random texture in magnesium [5], b) the ABEed sample after single pass at 100 °C.**

The texture of ABEed materials after single pass deformation at higher temperature of 250°C is shown in Figure 4b. With increasing temperature, one can observe no significant displacement for the maxima positions; however, the poles maxima tend to spread around. Similar results were reported in the ECAP Mg alloy textures studied by Agnew et al. [10]. The inclination angle for the basal poles tends to decrease at higher temperature, so that the c-axis is gradually rotated towards TD. This is associated with the development of ND// <10-11> texture, where the prismatic

plane of most grains tend to lie parallel to ND. It should be noted that the maximum texture intensity increased with increasing processing temperature, from 7.9 (mrd) at 100°C to 1.2 at 250°C. The effect of temperature on the texture obtained after ABE is explained by the increase in the size of the recrystallized grain [6]. The latter is accompanied by a decrease in plastic compatibility stress in the vicinity of grain boundaries, thereby decreasing the share of non-basal slip [10]. This, in turn, strengthens the ABE texture.

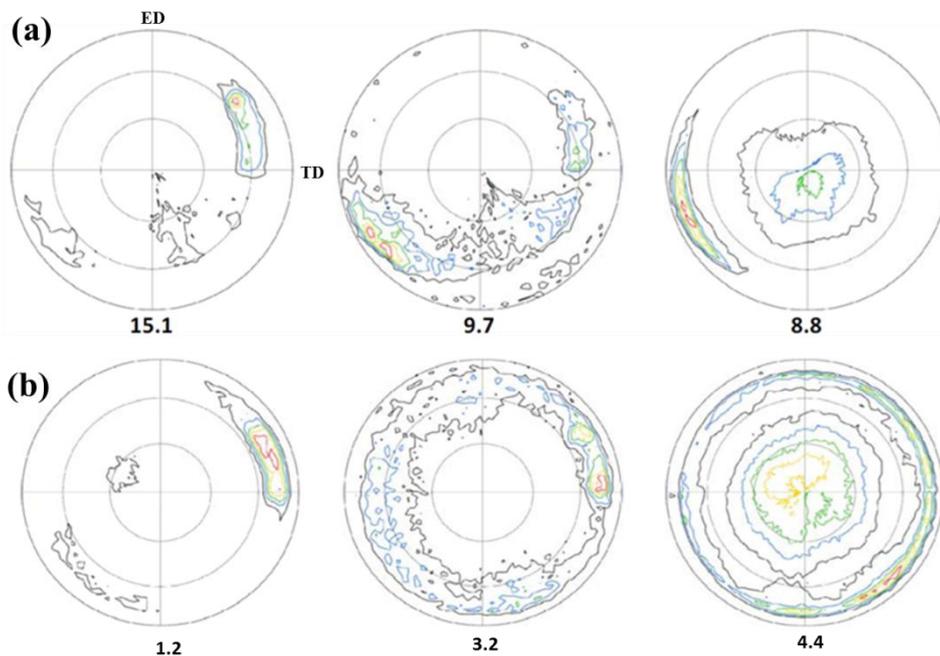


Fig. 4. The effect of processing temperature on the texture component after single ABE pass at a) 100 °C, b) at 250 °C.

The increase in temperature as well, increases the strain rate sensitivity and diminishes the flow localization effect and contribution of shear banding phenomena. Therefore, at low temperature the continuous flow cannot be established and consequently the deformation follows in shear bands of internally localized plastic flow crossing many grains. Shear bands tend to cause rotation of the material about the transverse direction and this may lead to a decrease in intensity of the preferred texture. Quite rare research could be found in the literature dealing with the effect of processing temperature on the post-SPD magnesium texture. Yoshida et al. [11] followed the texture evolution in AZ31 during

one pass ECAP at two different temperatures, 250 and 300°C. The texture at lower temperature had its basal poles aligned with TD at 30° from the ND, whereas the high temperature texture had its basal poles aligned with the ED. They believed that with increasing temperature, the secondary system involved during deformation became altered from a prismatic to a pyramidal system.

However, it was shown that the different non-basal mechanisms occurred only in the early stages of the extrusion and in the remainder, the deformation mechanism transitioned to basal slip [10].

#### 4. Conclusion

An inhomogeneous microstructure was obtained after single pass ABE at 100°C including recrystallized ultrafine/ nano grains. The mean sizes and areas fraction of the recrystallized grain increased with increasing temperature to 250°C. The texture after single pass ABE at 100°C includes the basal poles lied ~40° away from the TD in ED-TD plane and 66° from the ND in the TD-ND plane, while the maxima of the prismatic planes were revealed at 40-50° in the TD-ED plane and that of the pyramidal plane were inclined around 30° from TD. With increasing deformation temperature, the inclination angle for the basal poles tends to decrease, such that the c-axis gradually rotated towards TD. This is associated with the development of ND // <10-11> texture, where the prismatic plane of most grains tend to lie parallel to ND. The diminishing role of shear banding and larger grain size at higher deformation temperatures played a significant role in increasing the texture intensity.

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