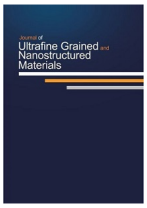


Journal of Ultrafine Grained and Nanostructured Materials
https://jufgnsm.ut.ac.ir
Vol. 52, No.1, June 2019, pp. 18-24
Print ISSN: 2423-6845 Online ISSN: 2423-6837
DOI: 10.22059/JUFGNSM.2019.01.02

# Recrystallization texture during ECAP processing of ultrafine/nano grained magnesium alloy

***Seyed Mahmood Fatemi****

*School of Mechanical Engineering, Shahid Rajaee Teacher Training University, Lavizan, Tehran.*

*Recieved: 04 December 2018; Accepted: 20 January 2019*
** Corresponding author email: mfatemi@ut.ac.ir*

**ABSTRACT**

An ultrafine/nano grained AZ31 magnesium alloy was produced through four-pass ECAP processing. TEM microscopy indicated that recrystallized regions included nano grains of 75 nm. Pole figures showed that a fiber basal texture with two-pole peaks was developed after four passes, where a basal pole peak lies parallel to the extrusion direction (ED) and the other ~20° away from the transverse direction (TD) in the ED–TD plane and 70° from the normal direction (ND ) in the TD–ND plane. The texture of recrystallized grains at the vicinity of grain boundaries was addressed. To investigate the recrystallization texture, the orientation relationships for the recrystallized and parent grains were studied for the material deformed up to first and second passes. The EBSD results implied that the grains recrystallized at the grain boundaries during the first pass, almost follow the orientation of prior deformed grains, while during the second pass the grain boundary recrystallization adopted a texture different from the parent grains. It was rationalized that as the number of passes increased, the grains recrystallized at prior grain boundaries tend to take different orientation to that of deformed grains.

***Keywords:*** *Nonograins; Recrystallization; Texture; Magnesium.*

## 1. Introduction

Despite many fascinating properties, the applications of wrought magnesium alloys are still restricted due to: (i) their hexagonal close-packed crystal structure with an insufficient number of operative slip systems at room temperature, and (ii) the development of strong crystallographic texture during deformation processes. The texture developed after deformation processes is controversial as the activation of different dynamic recrystallization mechanism during deformation may alter the final texture [1]. Severe plastic deformation (SPD) processes are considered to improve room temperature mechanical properties in magnesium alloys, e.g. [2]. During SPD, the initial grain size of the material is significantly reduced by thermally aided plastic deformation through different recrystallization mechanisms [3].

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 a change in the texture [4, 5]. In this regards, some SPD processing conditions have been reported to result in lower post-processing strength and/or ductility as compared to the starting commercial material [6]. Furthermore, it has been demonstrated that the degree of grain refinement by SPD, itself, is strongly coupled to the development of texture and substructural evolutions [7, 8]. 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 [9]. Therefore, it is indispensable to realize the texture evolution developed during severe deformation to enable the optimization of the materials and processing parameters for desired properties.

Due to the prevalence of dynamic recrystallization during SPD of magnesium alloys, the texture evolutions are mainly governed by recrystallization texture. According to the related literature, present understanding of the recrystallization textures in magnesium is little more than qualitative in nature. There are plenty of reported researches dealing with the texture component obtained after SPD processing of magnesium alloys. For example, during extrusion, the basal plane reorients itself in one of two directions, where either the <11-20> axis or the <10-10> axis is parallel to the extrusion direction [10]. Nevertheless, only a few misorientation relationships are established between the recrystallized and the parent grains. According to the literature, recrystallization in magnesium is not usually accompanied by sharp changes in the crystallographic texture. Yi et al. [11] have reported that the intensities in orientation distribution function in AZ31 were similar in the deformed and small dynamically recrystallized (DRX) grains. Such recrystallization appears to promote 30° <0001> rotations that preserve the basal texture or, at the very least, delay its decomposition [12]. For a Mg-Zn alloy, it was found that the texture intensity in DRXed region formed around the second phase at grain boundaries (GBs) was weaker compared with the deformed region [13]. Moreover, simulations showed that GB energy and mobility emerge as the key governing effects in the experimentally-observed preferred orientation selection during nucleation and growth of recrystallized grains [14]. Galiyev et al. [15] reported that continuous DRX is the controlling mechanism during hot compression at intermediate temperatures of 200~250 °C and discontinuous DRX dominates the deformation at high temperatures of 300~450 °C. It should be stressed that the reported results mainly obtained during conventional deformation processes involving low strain magnitudes, while quite rare results and discussions have been given on the crystal orientation during formation of new grains and related texture evolution of SPD processed magnesium alloys [16]. The latter notion is complicated by involving the influences of nucleation sites of various types and the energy and mobility of the boundaries [17]. Biswas et. al. [18] pointed out that continuous dynamic recovery and recrystallization was operative during asymmetric rolling of pure magnesium, where the texture of the partitioned DRX grains was shifted by ~30° along the c-axis from the deformed grains.

Previous results on AZ31 magnesium alloy undergone consecutive SPD passes indicated that DRX may occurred repetitively [19]. The major aim of the present work is to address the texture evolution associated with the DRX occurred at the prior GBs during multiple SPD passes. The obtained results may assist in engineering the final texture developed during large deformation of magnesium alloys. To this end, consecutive passes of equal channel angular pressing (ECAP) were applied to AZ31 magnesium alloy. Moreover, interrupted ECAP deformation followed by TEM and electron back scattered diffraction (EBSD) investigations were employed.

## 2. Experimental procedure

The initial material was hot rolled AZ31 magnesium alloy exhibiting strong basal texture. Considering the RD (rolling 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 $B_C$ up to four passes. Experiments were carried out at 250 °C for the first two passes, after that the remaining ECAP passes was carried out at 200 °C. The billets were preheated to the experimental ECAP temperatures for 10 minutes before entering the first channel of the die through the inlet. Microstructure and texture were characterized by transmission electron microscopy (TMP) Electron back scattered diffraction (EBSD) analysis performed on Field Emission Gun - Scanning Electron Microscope (FEG-SEM). EBSD was performed by cutting the samples along the initial RD-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 SiO2 particles).The EBSD data processing was carried out with TSL software. New recrystallized grains were recognized considering their size and morphology using optical and SEM micrographs.

## 3. Results and discussion

Figure 1 shows the typical ultrafine/nano structure obtained after the forth ECAP pass. Dynamic recrystallization (DRX) has significantly influenced the grain's morphology, resulting in an area fraction of 89%. The recrystallized area includes ultrafine and nano grains of 75 nm to 1 micron sizes with a mean size of 120 nm (Fig. 1b). To study the texture obtained after 4 ECAP passes, inverse pole figures (IPFs) as well as pole figures were provided and presented in Fig. 2. It is evident that a strong preferred crystallographic texture was formed, where a fiber basal texture with two pole peaks was observed. The prismatic and pyramidal plane normals tend to align parallel to TD. According to the final microstructure, it is obvious that the new grains influenced the final texture. IPFs were obtained to study the development of crystallographic texture of new grains. Figure 2a shows the IPF obtained, where grains mainly take similar texture components. PFs in Fig. 2b indicates that a two- pole peak configuration was formed for the basal plane normal. The corresponding

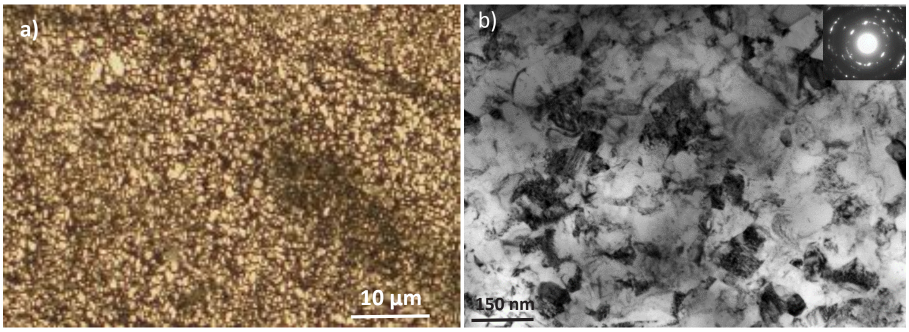


Fig. 1- a) Microstructure of the experimental alloy ECAP processed at 200 °C, b) TEM of recrystallized grain structure.

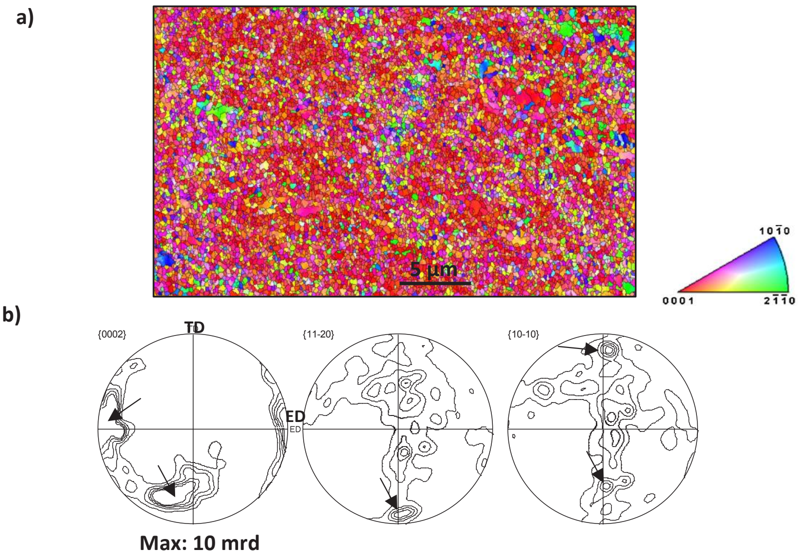


Fig. 2- a) IPF, b) PFs of the experimental alloy deformed up to four ECAP passes.

maximum intensity was presented in figures as multiples of random density (mrd). It is worth mentioning that the ECAP technique could hardly establish the corresponding deformation texture during the single pass processing [20]. One o the basal pole peaks lies parallel to the ECAP direction (ED) and the other ~20° away from the transverse direction (TD) in the ED–TD plane and 70° from the normal direction (ND) in the TD–ND plane, while the maximum of prismatic and pyramidal planes were revealed parallel to the TD in the TD–ED plane. These texture components generally place the magnesium crystal inclined to the ECAP axis. Accordingly, the basal planes are not oriented corresponding to the predicted morphological texture i.e., parallel to the "shear plane", identified by finite element simulation (FEM). The FEM results positioned the shear plane inclined 40–50° to the EACP axis [10]. The latter implied that the deformation texture was effectively influenced by recrystallization texture which dictated by microstructural mechanisms. In contrast to the present results, Wang et. al. [21] observed that basal texture weakens by ECAP deformation on AZ80 magnesium alloys at 250 °C. The presence of second phase particles in the alloy may take a critical role in weakening the texture. The effect of precipitates on randomizing the texture was thoroughly discussed by Ref. [22].

Recrystallization shares several common characteristics as phase transformation in that the replacement of deformed materials by the nucleation and growth of recrystallized grains, both can lead to a drastic changes in texture [17]. However, they exhibit two major differences. First, the nuclei during recrystallization are regions that already exist in the deformed microstructure and second recrystallization does not lead to precise orientation relationship between the deformed and recrystallized materials in contrast to the case of phase transformation. To analyze the relation between recrystallization texture of DRXed grain and the deformation texture EBSD maps were provided of the alloy deformed up to the first pass (Fig. 3.a). The maps of DRX and parent grains were isolated (Fig. 3.b) and related pole figures were obtained separately (Fig. 3.c and d). The deformed parent grains exhibit a basal texture where the c-axis aligned 20-70° to the TD in ED-TD plane and almost random distribution of prismatic and pyramidal planes is observed. It is observed that the grains mainly formed at the vicinity of prior GBs. As can be obviously realized from Fig. 3d, the DRXed grains almost follow the same orientation to the old grains from which they have grown.

The driving force for the nucleation on GBs is usually presumed to arise from a difference in dislocation content on opposite sides of the GB. There is considerable evidence in magnesium alloys that new grains with high angle boundaries

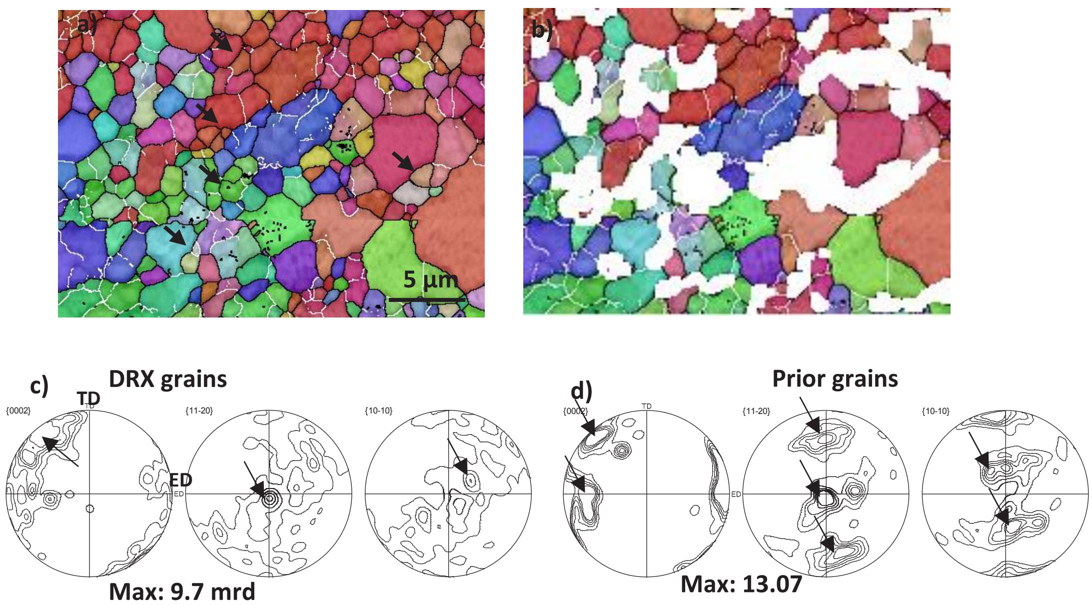


Fig. 3- (a) EBSD image showing the microstructure of AZ31 alloy ECAPed up to one pass, EBSD map with b) excluded parent grains, c) pole figures of DRX grains, d) pole figues of parent grains.

may be formed during straining, by the progressive rotation of subgrains at the vicinity of original GB with little accompanying boundary migration [23]. This is a strain-induced phenomenon which should not be confused with the subgrain rotation which has been postulated to occur during static annealing. Consistent with this theory, it is simply observed (arrowed regions) in Fig.3a that the new grain are fragmented from their parent grains. Formation of low angle boundaries (white color lines) strengthen the possibility of lattice rotation at the vicinity of boundaries, which may be progressively transformed into high angle boundary upon further straining. The mechanisms by which this progressive subgrain rotation occurs, is most frequently found in materials in which dislocation motion is inhibited by either a lack of slip systems, like magnesium alloys, or by solute drag [15].The phenomenon involves the progressive rotation of subgrains adjacent to pre-existing GBs as the material is strained, with the old grains developing a gradient of misorientation from center to edge. In the center of the old grain, subgrains may not be well developed or may have very low misorientations. Towards the GB, the misorientations increase so that at larger strains, high angle boundaries may be developed. Although this phenomenon usually results in a partially recrystallized necklace microstructure, at large strains a completely recrystallized structure may be formed. The point is that this phenomenon may be frequently repeated as the material deformation proceeds and dislocation structure is developed within the DRXed grains.

To further trace the microstructure evolution associated with recrystallized grains, IPF and PFs were provided for the material after the second pass, too. DRX grains and parent grains were isolated (Fig. 4.b) and related pole figures were plotted separately (Fig. 4c and d). The deformed parent grains exhibit a basal texture where the c-axis aligns 25° to the TD in ED-TD plane and almost random distribution of prismatic and pyramidal planes is seen. However, it is obvious in Fig. 3d that the DRXed grains follow completely different orientation to the prior grains. They produced two-peak pole parallel to and 20° inclined to TD, where prismatic and pyramidal planes took preferred orientations. The results implied that the GB recrystallization texture exhibit different relationship to the old grain as the number of passes increased. Similarly, different orientation of DRXed grains compared to their parent grains was reported by Zeng et. al [24] after severe deformation of Mg-0.3Zn-0.1Ca (wt.%). They hypothesized that the alloying elements may segregate strongly to high-energy boundaries of the recrystallized grains that would trigger them to grow preferentially, leading to a more uniform growth of recrystallized grains with randomized orientations.

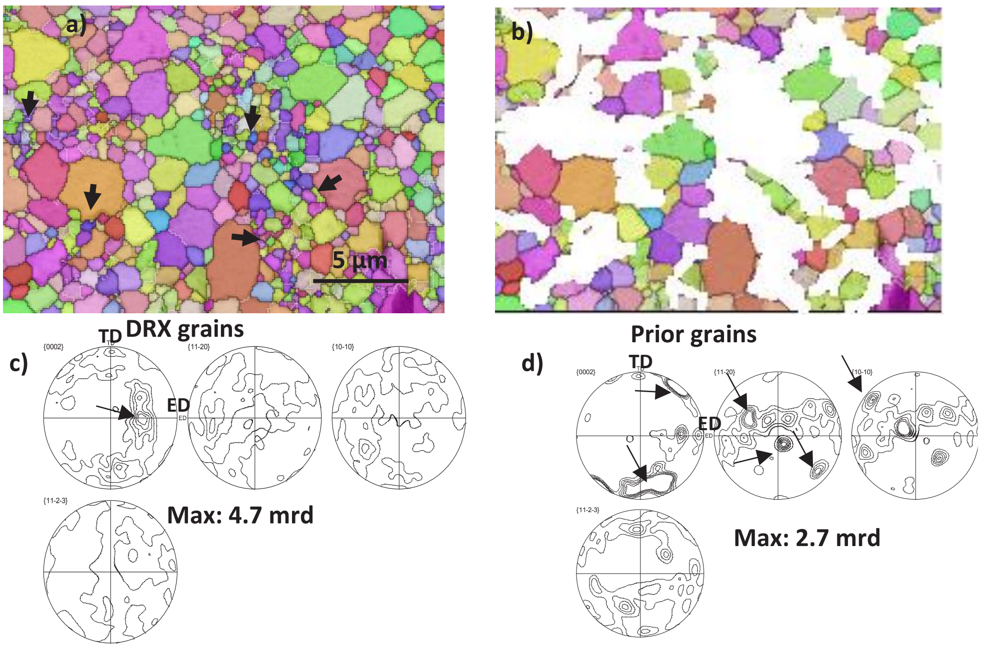


Fig. 4- (a) EBSD image showing the microstructure of AZ31 alloy ECAPed up to two passes, EBSD map with b) excluded parent grains, c) pole figures of DRX grains, d) pole figures of parent grains.

The production of homogeneous three dimensional microstructures in magnesium alloys requires the activation of both non-basal and basal slip. Experimental observations showed that non-basal slip is activated more easily in the interiors of the grains of magnesium alloys when the grain size is reduced [25]. Since the evolved microstructure during the second pass is characterized by finer grain sizes, a higher area fraction of grains interior is influenced by incompatibility stress. The latter results in a higher share of non-basal slip during deformation and thereby a more homogeneous deformation. Accordingly, finer subgrains may be developed around the GB, the coalescence of which require more relative rotation. Thus one may expect that the rotation recrystallization at the prior GBs should be associated with remarkable rotation. This may end up to the formation of new grains characterized with a rotated orientation compared to the parent grains. Therefore, it seems that as the severe deformation passes increases GB recrystallization may adopt a different texture to the old grains. It is a new finding in the field of recrystallization texture of magnesium according to the present literature [14, 26]. However, it should be noted that, according to the previous studies [19], the recrystallization phenomena is governed, at large strains, by continuous recrystallization rather than GB recrystallization.

## 4. Conclusion

An ultrafine/nano grained AZ31 alloy was obtained through four-pass ECAP processing, where a moderate texture strength was obtained. It was showed that rotation recrystallization may be operative during the first pass, the texture of which was relatively similar to their parent matrix. However, as the DRXed grain further evolved through the second pass, the grains recrystallized at the vicinity of prior grain boundaries invoke a significant texture rotation, compared to their parent grains.

## References

1. Yang X-Y, Ji Z-S, Miura H, Sakai T. Dynamic recrystallization and texture development during hot deformation of magnesium alloy AZ31. Transactions of Nonferrous Metals Society of China. 2009;19(1):55-60.
2. Yang H, Widiantara I, Ko Y. Effect of deformation path on texture and tension properties of submicrocrystalline Al-Mg-Si alloy fabricated by differential speed rolling. Materials Letters. 2018;213:54-7.
3. Fatemi-Varzaneh S, Zarei-Hanzaki A, Beladi H. Dynamic recrystallization in AZ31 magnesium alloy. Materials Science and Engineering: A. 2007;456(1-2):52-7.
4. Guo W, Wang Q, Ye B, Zhou H. Microstructure and mechanical properties of AZ31 magnesium alloy processed by cyclic closed-die forging. Journal of Alloys and Compounds. 2013;558:164-71.
5. Zhao Z, Chen Q, Hu C, Shu D. Microstructure and mechanical properties of SPD-processed an as-cast AZ91D+ Y magnesium alloy by equal channel angular extrusion and multi-axial forging. Materials & Design. 2009;30(10):4557-61.
6. Kim W, Park J, Kim W. Effect of differential speed rolling on microstructure and mechanical properties of an AZ91 magnesium alloy. Journal of Alloys and Compounds. 2008;460(1-2):289-93.
7. Del Valle J, Ruano OA. Influence of texture on dynamic recrystallization and deformation mechanisms in rolled or ECAPed AZ31 magnesium alloy. Materials Science and Engineering: A. 2008;487(1-2):473-80.
8. Chen M-S, Yuan W-Q, Li H-B, Zou Z-H. New insights on the relationship between flow stress softening and dynamic recrystallization behavior of magnesium alloy AZ31B. Materials Characterization. 2018.
9. Sun W, Qiao X, Zheng M, He Y, Hu N, Xu C, et al. Exceptional grain refinement in a Mg alloy during high pressure torsion due to rare earth containing nanosized precipitates. Materials Science and Engineering: A. 2018;728:115-23.
10. Mishra RK, Gupta AK, Rao PR, Sachdev AK, Kumar AM, Luo AA. Influence of cerium on texture and ductility of magnesium extrusions. Essential Readings in Magnesium Technology: Springer; 2016. p. 363-8.
11. Yi S, Zaefferer S, Brokmeier H-G. Mechanical behaviour and microstructural evolution of magnesium alloy AZ31 in tension at different temperatures. Materials Science and Engineering: A. 2006;424(1-2):275-81.
12. Beausir B, Biswas S, Kim DI, Tóth LS, Suwas S. Analysis of microstructure and texture evolution in pure magnesium during symmetric and asymmetric rolling. Acta Materialia. 2009;57(17):5061-77.
13. Du Y, Zheng M, Ge Y, Jiang B, Shen M. Microstructure and texture evolution of deformed Mg-Zn alloy during recrystallization. Materials Characterization. 2018;145:501-6.
14. Barrett CD, Imandoust A, Oppedal AL, Inal K, Tschopp MA, El Kadiri H. Effect of grain boundaries on texture formation during dynamic recrystallization of magnesium alloys. Acta Materialia. 2017;128:270-83.
15. Galiyev A, Kaibyshev R, Gottstein G. Correlation of plastic deformation and dynamic recrystallization in magnesium alloy ZK60. Acta materialia. 2001;49(7):1199-207.
16. Biswas S, Dhinwal SS, Suwas S. Room-temperature equal channel angular extrusion of pure magnesium. Acta materialia. 2010;58(9):3247-61.
17. Humphreys FJ, Hatherly M. Recrystallization and related annealing phenomena: Elsevier; 2012.
18. Biswas S, Kim D-I, Suwas S. Asymmetric and symmetric rolling of magnesium: Evolution of microstructure, texture and mechanical properties. Materials Science and Engineering: A. 2012;550:19-30.
19. Fatemi-Varzaneh S, Zarei-Hanzaki A, Cabrera J, Calvillo P. EBSD characterization of repetitive grain refinement in AZ31 magnesium alloy. Materials Chemistry and Physics. 2015;149:339-43.
20. Estrin Y, Yi S, Brokmeier H-G, Zuberova Z, Yoon S, Kim H, et al. Microstructure, texture and mechanical properties of the magnesium alloy AZ31 processed by ECAP. International Journal of Materials Research. 2008;99(1):50-5.
21. Wang L, Mostaed E, Cao X, Huang G, Fabrizi A, Bonollo F, et al. Effects of texture and grain size on mechanical properties of AZ80 magnesium alloys at lower temperatures. Materials & Design. 2016;89:1-8.
22. Sadeghi A, Pekguleryuz M. Recrystallization and texture evolution of Mg–3% Al–1% Zn–(0.4–0.8)% Sr alloys during extrusion. Materials Science and Engineering: A. 2011;528(3):1678-

85.
23. Cottam R, Robson J, Lorimer G, Davis B. Dynamic recrystallization of Mg and Mg–Y alloys: crystallographic texture development. Materials Science and Engineering: A. 2008;485(1-2):375-82.
24. Zeng Z, Zhu Y, Xu S, Bian M, Davies C, Birbilis N, et al. Texture evolution during static recrystallization of cold-rolled magnesium alloys. Acta Materialia. 2016;105:479-94.
25. Koike J. Enhanced deformation mechanisms by anisotropic plasticity in polycrystalline Mg alloys at room temperature. Metallurgical and Materials Transactions A. 2005;36(7):1689-96.
26. Humphreys J, Rohrer G, Rollett A. Control of recrystallization. Recrystallization and Related Annealing Phenomena. 2017:527-67.