



Review on ultrafined/nanostructured magnesium alloys produced through severe plastic deformation: microstructures

Mahmoud Fatemi^{1*} and Abbas Zarei-Hanzki²

1. Shahid Rajaee Teacher Training University, Tehran, Iran

2. School of Materials & Metallurgical Engineering, University of Tehran, Tehran, Iran

Received: 25 May 2015; Accepted: 12 October 2015

* Corresponding Author: E-mail: mfatemi@ut.ac.ir, Tel: +98-21-61114116, Fax: +98-21-88006046

Abstract

A review on the microstructural evolution in magnesium alloys during severe plastic deformation was presented. The challenges deserved to achieve ultrafine/ nanostructured magnesium were discussed. The characteristics of the processed materials are influenced by three main factors, including i) difficult processing at low temperatures, ii) high temperature processing and the occurrence of dynamic recrystallization and grain growth processes, and iii) a combined effect of grain refinement and crystallographic texture changes. Reviewing the published results indicate that there are two potential difficulties with severe deformation of magnesium alloys. First, it is very hard to achieve homogeneous ultrafined microstructure with initial coarse grains. The second is the dependency of microstructure development on the initial grain size and on the imposed strain level. It was clarified that different grain refining mechanisms may be contributed along the course of multi-pass severe deformation. It was clarified that discontinuous recrystallization takes places during the first stages of deformation, whereas continuous refinement of the recrystallized grain may be realized at consecutive passes. Shear band formation as well as twinning were demonstrated to play a significant role in grain refinement of magnesium alloy. Also, the higher the processing temperature employed the more homogeneous microstructure may be achieved with higher share of low angle grain boundaries.

Keywords: *magnesium, nano/ ultrafine grain, recrystallization, SPD.*

1. Introduction

Severe Plastic Deformation (SPD) techniques enjoy great popularity thanks to their ability to produce considerable grain refinement in fully dense, bulk-scale work-pieces, thus promising structural applications. The achievable grain sizes lie within the sub-micrometer (100-1000 nm) and nanometer (<100 nm) ranges. SPD-

processed materials with such grain sizes are generally referred to as nanomaterials, although only the latter ones can be regarded as being nanostructured according to the conventional definition. Though most SPD processing was limited to soft metals and alloys, more recent attention has focused on

the processing of difficult-to-work materials such as magnesium-based alloys.

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). Summarizing previous studies showed that the processing of magnesium alloys by SPD processes deserves significant challenges.

Accordingly, the characteristics of the processed materials are influenced by three main factors: 1. the processing of many magnesium alloys are relatively difficult at low temperatures thanks to the low symmetry hexagonal crystal structure, resulted in the development of premature cracking or segmentation, and 2. SPD processing should be carried out at high temperatures to prevent cracking. As a result, the dynamic recrystallization and grain growth processes occur inevitably and thereby may diminish the grain refinement effect, 3. the post-SPD mechanical properties result from a 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, 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 [1]. 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 demonstrated that the degree of grain refinement by SPD, itself, is strongly coupled to the development of texture and sub-structural evolutions [4].

Different SPD techniques have been used for processing the magnesium alloys, such as equal channel angular extrusion (ECAP) [2, 5-

7], Multi-axial forging (MDF) [8], accumulative roll bonding (ARB) [9], and accumulative back extrusion (ABE) [10]. The earliest attempts at severe straining of magnesium alloys go back to ECAP processing of AZ91 alloy by Mabuchi et al. [11], where fine grains of 1 μm size were achieved. Subsequently, the development of ultrafine microstructure with mean grain size of 0.7 μm was reported after eight passes ECAP at 200°C by Mabuchi et al. [11] and Lin et al. [7]. Nevertheless, severe cracking was observed after multi-pass ECAP processing at temperature below 200°C [13].

To overcome premature cracking in magnesium alloys, special approaches were employed during ECAP processing through applying back pressure at the die exit [10, 13] as well as increasing the die angle [5, 14]. The experiments have shown that the development of segmentation or cracking in the alloys during processing may be reduced or even eliminated through the application of a back-pressure during the pressing operation. This may be attained by preventing the flow to be localized in narrow regions. Accordingly, advantages of using a back-pressure in the processing of the AZ31 alloy by ECAP include the introduction of greater grain refinement, a potential for pressing at lower temperatures, and a more rapid transition to a homogeneous microstructure [10]. Moreover, the processing of magnesium alloy is viable at lower temperatures in the presence of back pressure. For example, it has been claimed that AZ31 alloy could be successfully deformed through eight pass ECAP at 150°C [15].

The second technique, increasing the channel angle in the die, reduces the magnitude of the equivalent strain. It was shown by FEM that cracking and segmentation may be reduced or even eliminated by increasing the angle Φ , and this effect was confirmed experimentally by pressing two billets of a ZK60 magnesium alloy through dies with different channel angles [13]. Sound samples were obtained by increasing the angle to 110° [15] and 120° [16] during ECAP processing of AZ31 alloy at 180 and 175°C, respectively. However, the results indicated that the latter was associated with an increase in the final mean grain size [17].

Rotating die ECAP (RD-ECAP) was also suggested as a continuous method for

processing straining ZE41A alloy [18], to iron out the discontinuity of the process. The microstructure observation after 16 passes indicated that RD-ECAP was effective in producing a more homogenous microstructure. The obtained microstructure was characterized by a more homogenous grain size distribution possessing a mean size of 1.5 μm . Moreover, ECAP processing was utilized in conjunction with differential speed rolling to achieve improved mechanical properties in AZ31 alloy through controlling grain size distribution as well as texture [19].

2. The Obtained Microstructures

Ultrafine/ nanostructured materials processed by SPD methods are characterized by a very high density of grain boundaries and are interface-controlled materials. Accordingly, their grain boundaries and grain sizes are in the center of all structural investigations. Various degrees of grain refinement and microstructure homogeneity were achieved in magnesium alloys after applying different processing techniques and routes. For example, it was reported that high pressure torsion (HPT) may work more effectively to produce nanostructured magnesium compared to ECAP [20], thanks to the presence of compressive stress component. The disadvantage of fairly small dimensions limits the potential applicability of the HPT

samples to electronic and medical pieces. Accumulative roll bonding (ARB) or processing resulted in a significant grain refining in AZ61 alloy at 300 and 400°C [9]. Moreover, the results reported for AZ grade alloys indicate that the higher AL content of the alloy the development of ultrafine grained structure is promoted [21].

Summarizing the published results denote that ultrafine grained sheets may be produced using ARB process, though elongated structure formed along the transverse direction. Recently, multi-directional forging (MAF) was also utilized to produce fine grained magnesium alloys. Xing [22, 23] and Miura [23] achieved an ultrahigh strength and a mean grain size of less than 1 micron by applying a cumulative strain of 4 using decreasing temperature technique. The next work published by Miura et. al. included the imposition of cumulative strain of 2 at room temperature, where a mean grain size of 0.6 μm and yield stress of 480 MPa were attained [24]. Very recently, a novel continuous SPD method called Accumulative Back Extrusion (ABE) was employed by present author and his supervisor to process AZ31 alloy. Accordingly, the experimental alloy was successfully deformed at low temperature of 80°C, where an inhomogeneous microstructure including nano-grain was developed [25]. Figure 1 displays the typical microstructure achieved after single pass ABE.

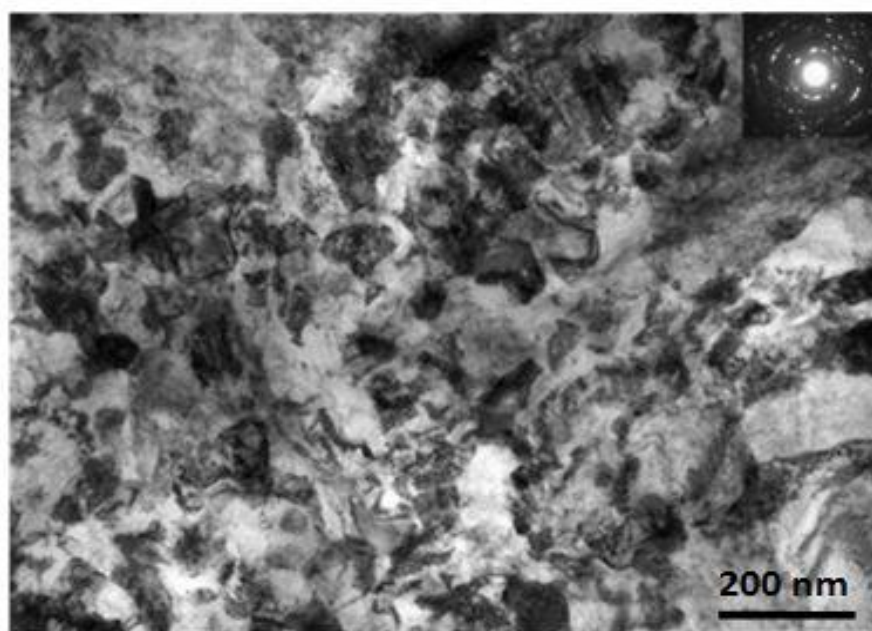


Fig. 1. Typical nano grains developed after single pass ABE

Though it is difficult to produce ultrafine/nano-grained microstructure in pure magnesium, it is viable to attain such a microstructure in magnesium alloys. Nevertheless, reviewing the published results indicates that there are two additional potential difficulties with magnesium alloys. First, it is very hard to achieve homogenous ultrafined microstructure with initial coarse grains. This was encountered, for example, during ECAP processing of cast Mg-Al alloy [26]. This limitation for coarse grain magnesium alloy is persistent even by employing a two-step EX-ECAP processing [12, 27]. The second is the dependency of microstructure development on the initial grain size and on the imposed strain level. The application of two pass ECAP of an extruded ZK60 with initial grain size of 2 μm resulted in a homogenous microstructure with a mean grain size of 0.2 μm [28]. However, a bimodal structure was formed after similar processing of an AZ31 alloy with initial mean grain size of 10 μm . The development of homogenous microstructure is more difficult in experimental alloy containing higher alloying elements [29]. A bimodal microstructure was also observed in AZ91 alloy even after 4 ECAP passes [30], where increasing the number of passes to eight yielded a homogenous fine microstructure.

The microstructural investigations of the SPD processed magnesium alloys showed that the mean grain size decreases as the imposed strain is increased. Kim et al. [31] found that grain boundary disorientation is increased with increasing cumulative strain, owing to the increasing the specific grain boundary area. This provides a proper driving force for the occurrence of dynamic recrystallization. However, the grain refinement during ECAP processing of magnesium alloy continues up to the fourth pass after that no further refining was reported [21, 32-34]. Ding et al. [17] believed that as the DRX completes, the microstructure is stabilized and no significant changes in grain size distribution may be recorded.

The published results implied that increasing the deformation velocity leads to a microstructures with smaller grain sizes [17, 35]. This is related to the effect of strain rate on the grain size developed during dynamic recrystallization [36]. Serre et al. [35] claimed

that increasing the deformation rate slightly reduces the final grain size of a magnesium alloy. However, no similar trend was reported in case of FCC metals. The observations made by Berbon et al. [37] demonstrates that deformation velocity has no effect on the final grain size of aluminum.

The homogeneity of obtained microstructure plays a key role in achieving the superior mechanical properties in materials processed by SPD [10, 22]. Some experiments have been conducted to evaluate the homogeneity of microstructure and mechanical properties of materials processed by SPD methods [38, 39]. In discontinuous SPD methods such as equal channel angular pressing (ECAP) and twist extrusion (TE), different processing routes have been developed where the billet is removed between each separate passage through the die. It has been shown that the efficiency of process in grain refinement and structural homogeneity is significantly affected by changing the rotation routes and the total amount of strain imparted [6, 7]. A high microstructural homogeneity by ECAP has been achieved when the processing route B_C was used [40]. There has been no similar route development for continuous methods such as high pressure torsion (HPT), where the work-piece is not removed between passes throughout the processing operation. This issue is of great importance as there are significant difficulties in the SPD processing of magnesium alloys [41].

A systematic work on evolution of homogeneity in severely deformed magnesium alloy was carried out very recently by Figueiredo and Langdon [6]. The results showed that there are variations in the grain size distributions and in the micro-hardness values of the disk-shape sample processed by HPT. Also, their observations demonstrated that the deformation is not homogeneous in the through-thickness direction during HPT. Moreover, the experiments on AZ31 specimens subjected to ABE processing demonstrated an evolution toward structural homogeneity with increasing temperature [42]. Micro-hardness measurements showed significant inhomogeneity after single pass ABE, which was attributed to the bimodal recrystallized grains nucleated either at prior

grain boundaries or within the shear bands. The grain refinement during ABE may assist in achieving reasonable homogeneity even after a few passes. Moreover, the strain reversal imposed during ABE may result in enhanced nucleation of new grains thereby leading to a rapid rate homogeneity achievement.

3. Microstructure Development Mechanism

There is no longer any doubt that extreme grain refinement is achievable with most malleable and even with many hard-to-deform materials through SPD processing. Despite this body of innumerable experimental evidence documented, the mechanisms of grain refinement, which are pivotal in designing the routes to property improvement, are far from being understood. In particular, there is no generally accepted scenario of grain fragmentation by subdivision of grains, and the underlying processes have remained a riddle for researchers to the present day. However, it has been well-recognized that the mechanism of grain refinement in magnesium alloys processed by SPD significantly differs from the mechanism contributed in FCC metals such as aluminum and copper.

In the FCC metals, the initial stages of straining produces an array of elongated sub-grains oriented with their longer axes aligned parallel to the primary slip system and with the low angle boundaries [22, 43]. Further straining introduces additional dislocations and these dislocations re-arrange and annihilate consistent with the low-energy dislocation structures theory [44]. The latter eventually gives an array of reasonably equiaxed grains separated by high angle boundaries. This microstructure is typically achieved after 4 passes ECAP in FCC structures. The related evolutions have been documented in detail in several materials including high-purity aluminum [45].

Microstructure study during SPD processing of magnesium alloys was first addressed by Kim and Kim [46] in an ECAPed AZ31 alloy. It was discussed that the sub-grain boundaries gradually transformed from low to high angle ones with pass number by absorbing dislocations generated during ECAP [46]. Xia et al. [47] obtained an inhomogeneous microstructure in an ECAPed

magnesium alloy and suggested that the recrystallization was preferably commenced in the grains with desired orientation for activation of slip systems. With increasing deformation passes the deformation is extended to the other grains, and consequently the progress of microstructure recrystallization is completed.

3.1. Discontinuous recrystallization

In contrast to FCC metals, many researchers believe that in magnesium and its alloys, the new ultrafine grains developed during severe deformation mainly form through the occurrence of discontinuous dynamic recrystallization (DDRX) along the original boundaries. The DDRX is found primarily in many materials with low to medium Stacking Fault Energy (SFE) including magnesium alloys. Although, the occurrence of DRX in magnesium has been attributed to the constraints imposed by the lack of easily activated slip systems, rather than to its SFE [48]. In fact the primary grain refinement in magnesium alloys during SPD methods is characterized by the nucleation of fine grains along the prior GBs. This is attributed to the development of stress concentrations at the boundaries and the subsequent activation of both basal and non-basal slip processes [49]. The initial large grains are ultimately consumed by these new smaller grains as the magnitude of strain (the number of deformation passes) increases. This is in agreement with the microstructural observations made during fundamental studies [36], where DDRX was recognized as the dominant mechanism during hot deformation of magnesium alloys.

Nucleation of fine grains occurs along pre-existing grain boundaries [49] and ends up to a necklace-like array of new grains, so that a critical grain size, d_c , is needed in order to achieve a homogenous equiaxed ultrafine grains. Figueiredo and Langdon [6] suggested the principle of grain refinement in magnesium alloys, as is schematically illustrated in Figure 2. The left column denotes the initial condition for grains either larger than d_c or smaller than d_c and the right column shows the microstructures after processing through one pass of ECAP. Thus, for $d > d_c$, new grains form along the

boundaries in a necklace-like manner but the initial grain structure is coarse so that processing by ECAP produces a bimodal structure, as shown in Figure 2b.

In this condition, the centers of these larger grains are not yet consumed by the formation of the smaller grains. Conversely, if $d < d_c$, the initial grain size is sufficiently small that few passes of ECAP produces a homogeneous array of ultrafine grains, as shown in Figure 2d. During two-step processes, such as EX-ECAP process, the preliminary extrusion is then used to produce a grain size that is smaller than the critical size d_c . The concept of bimodality may be more pronounced in the magnesium with high alloying additions. This is related to the role of alloying element in hindrance of grain boundary motion, which leads to smaller recrystallized grains [50].

Similar effect for AL addition was showed by Furukawa et al. [51] in Mg-AL-Zn alloys.

Most of the published results are in correspondence with the model proposed by Figueiredo and Langdon [6]. Different initial microstructures resulted in different homogeneities in the final ECAPed microstructure holding similar processing parameters. It has been observed that a homogenous ultrafine grained microstructure and multimodal microstructure was attained after similar ECAP processing of ZK60 alloy possessing initial grain size of 3 and 100 μm , respectively [28].

Present authors showed that the majority of DRXed grains formed during ABE process are originated from grain boundary bulging micro-mechanism, often called strain-induced boundary migration (Fig. 3a). The formation of new grains by the latter mechanism was

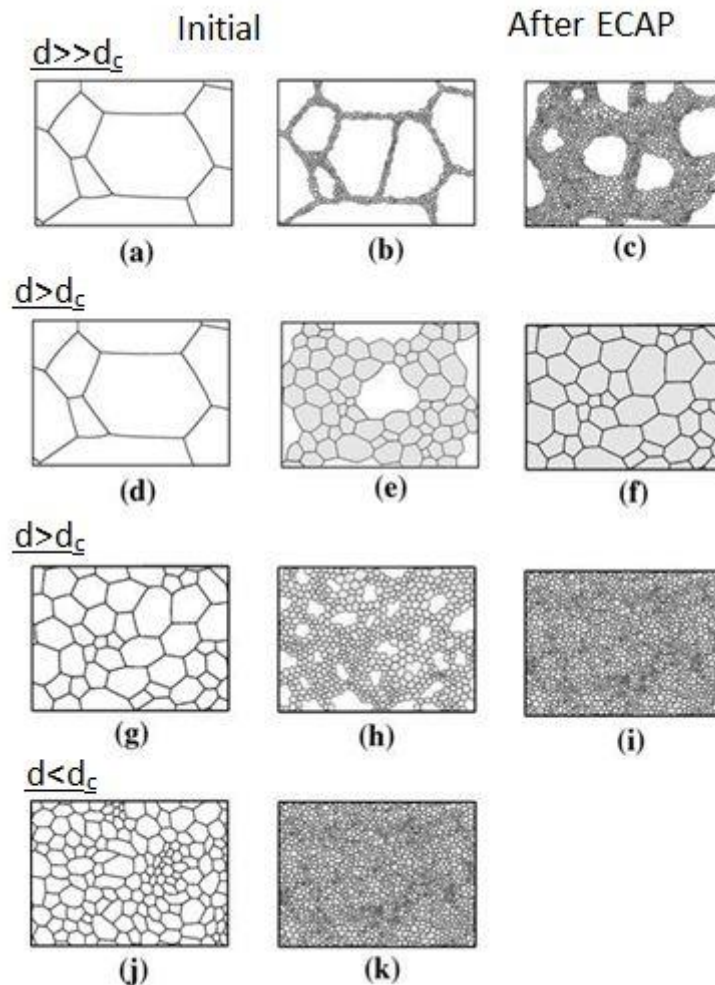


Fig. 2. A model for the grain refinement of magnesium alloys processed by ECAP [6]

also confirmed through local misorientation measurements by EBSD (Fig. 3b). It is believed that the local boundary migration occurs where there is a stored energy difference across a high-angle boundary. Accordingly, the migrating boundary is anchored by the pre-existing cell/sub-GBs and a grain boundary bulge develops [49]. However, in the case of AZ31 magnesium

alloy, cells/sub-grains have been hardly found. Instead, it has been demonstrated that after bulging out a segment of grain boundary, a bridging dislocation wall forms between the bulged region and the parent grain. During further deformation, the disorientation of this bridging wall gradually increases, and the bulged region becomes a DRXed grain [52].

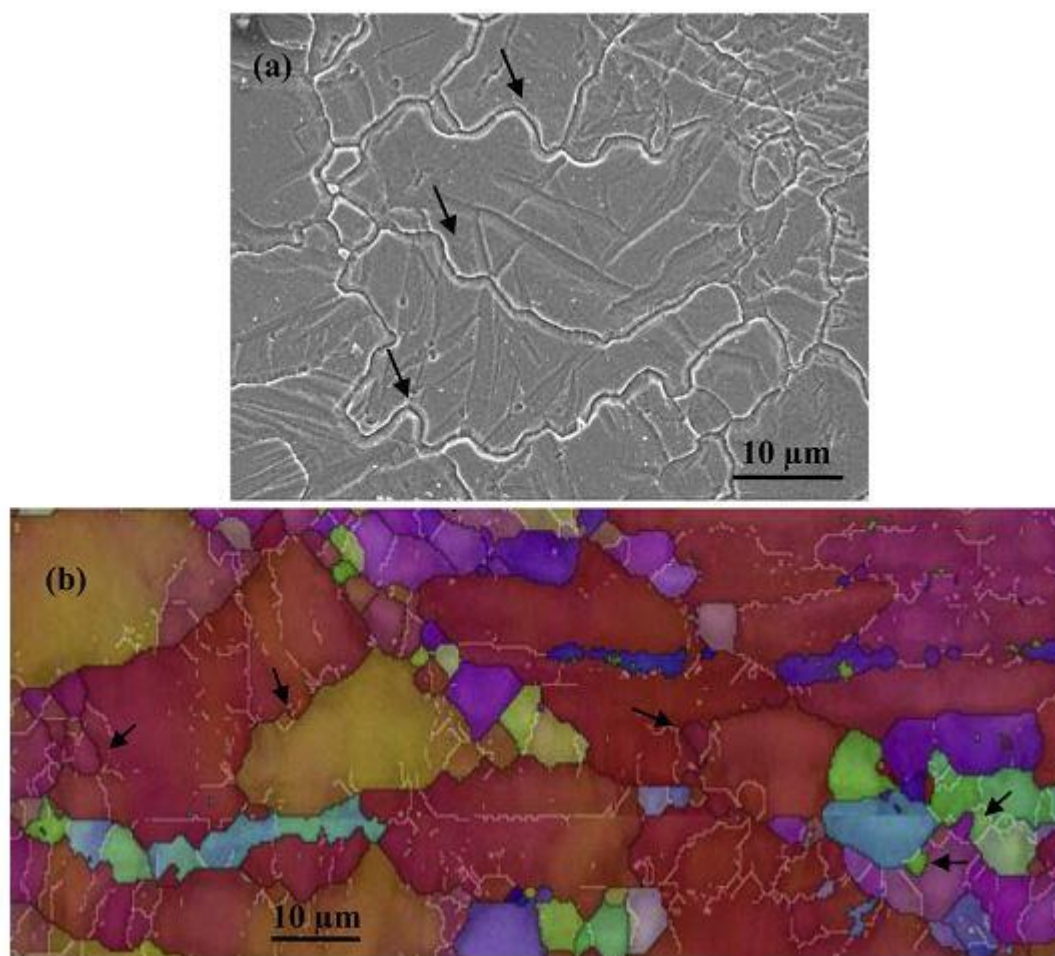


Fig. 3. a) and b) The microstructure of AZ31 alloy deformed to the first step of ABE process showing the bulging of GBs [53]

3.2. Shear banding

Generally speaking, in all SPD processes the deformation localization is observed through shear band formation. As is well established if the material work-hardening ability diminishes and the continuous flow becomes unstable, the localized flow commences along the shear bands [54]. Microstructural studies of the magnesium alloys processed by SPD methods denoted that the bands are considered to be the regions of microstructural refinement [54, 55].

The introduction of bands inclined to rolling direction during DSR of AZ31 alloy was reported to be the main microstructural features [56]. It has been reported that no apparent increase in the amount of the shear bands is observed with increasing deformation passes and the strain tends to concentrate around the shear bands formed by the previous pass. It has been reported that the fracture of the specimens can be accompanied by shear banding at high strain rates [57]. Also the

appearance of shear bands was observed by Lapovok et al. [58] during ECAP of AZ31 magnesium alloy. Moreover, the non-detrimental pronounced role of shear banding in developing a random recrystallization texture was also discussed during accumulative roll bonding (ARB) [59].

The results obtained by present authors noticed that shear banding phenomenon may be considered as a grain refining mechanism during SPD synthesis of AZ31 alloy, without harmful effect leading to premature fracture. This may be achieved where the deformation pattern associate with diffused shear bands well distributed all over the material. The latter was realized to be attainable in ABE process where adequate shear zones were anticipated during deformation [60]. The shearing deformation actually achieved inside a shear band is extremely large [61]. This may promote a high density of dislocations followed by continuous dynamic recrystallization. This may end up subdividing the bands by high angle grain boundaries and in turn grain refinement.

Su's results [62] showed that continuous recovery, recrystallization and grain growth in shear bands play a key role in effective grain refinement during ECAP of AZ31 alloy. Moreover, Su et al. [62] reported that formation of needle like shear bands is the dominant feature in AZ31 alloy during ECAP at 200°C. Within these bands, there were newly formed grain boundaries segmenting the bands into sections. The latter recrystallization mechanism was discussed to contribute during the microstructural evolution. TEM analysis showed that dislocations can experience continuous static recovery after deformation to be rearranged to form sub-grain boundaries. This intricate network of low angle boundaries can continue to experience static growth to form non-equilibrium grain boundaries like those shown in Figure 4. Eventually, continued growth of such grains can produce equilibrium grain boundaries which are much smaller than the prior grain size.

3.3. Twinning

The contribution of twinning was found to play an effective role during microstructural evolution in magnesium alloy. The degree to

which twinning contribute during deformation is influenced by temperature, c/a ratio, alloying elements, strain rate, and initial texture. It has been reported that during the primary stage of SPD processes, the main deformation mode is tensile twinning, while during the following passes the main deformation mode is dislocation slip [34]. The critical resolved shear stresses (CRSSs) for extension and contraction twinning was estimated to be 2 MPa and in the range from 76 to 153 MPa (114 MPa on average), respectively [24]. The high flow stresses that developed during severe deformation enabled the activation of contraction twins in addition to extension twins. These stresses permitted contraction twinning to take place even though the Schmid factor was low [34]. This can be explained in terms of the low accommodation strains likely to have been associated with these twins, while higher Schmid factor twins could not form due to their much higher accommodation strains [63, 64].

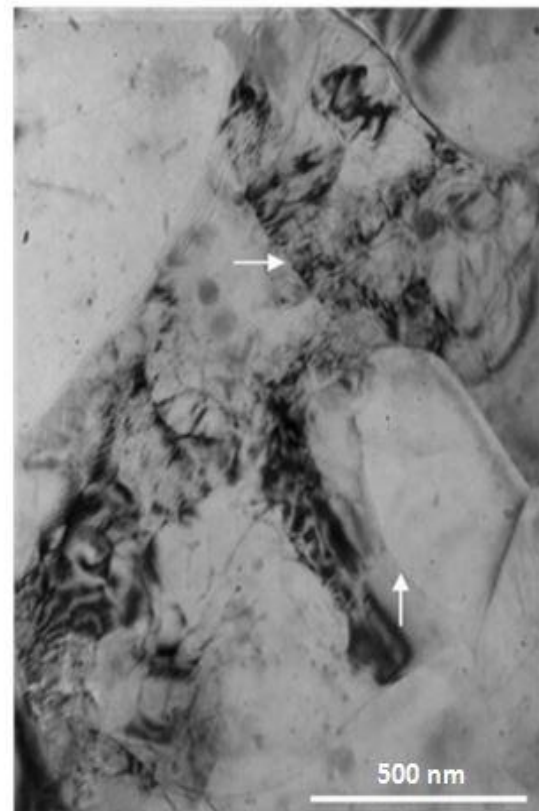


Fig. 4. Representative TEM micrographs of AZ31 after ECAP, showing the presence of subgrain structures [62]

The presence of both types of twins lead to significant grain fragmentation. Miura et al. [24] demonstrated that the twinning plays the main role in grain refinement of MDFed magnesium alloy, where original grains were subdivided into ultrafine ones by mechanical twinning at room temperature (Fig. 5). Similar observation made by present author in ABEed AZ31 alloy (Fig. 6). Lapovok [13] showed

that twinning occurs during the first two ECAE passes; the twin density stays constant at 200°C but decreases with subsequent passes for the other two temperatures. Together with the fact that the recrystallized grain area increases with the ECAE temperature, the observation suggested that twins are the preferred recrystallization sites.

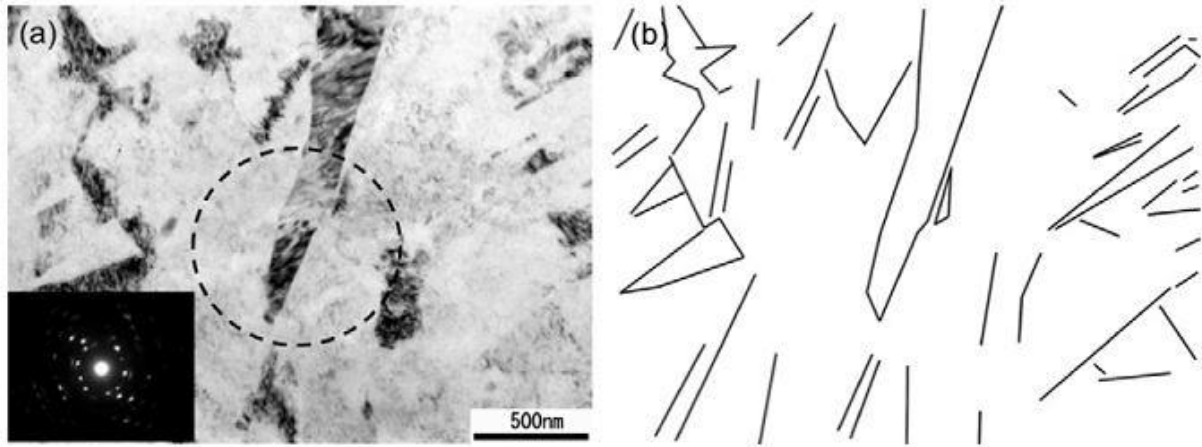


Fig.5. Microstructure of a multipass MDFed sample. The twin boundaries visible in (a) are identified more precisely in (b) [24]

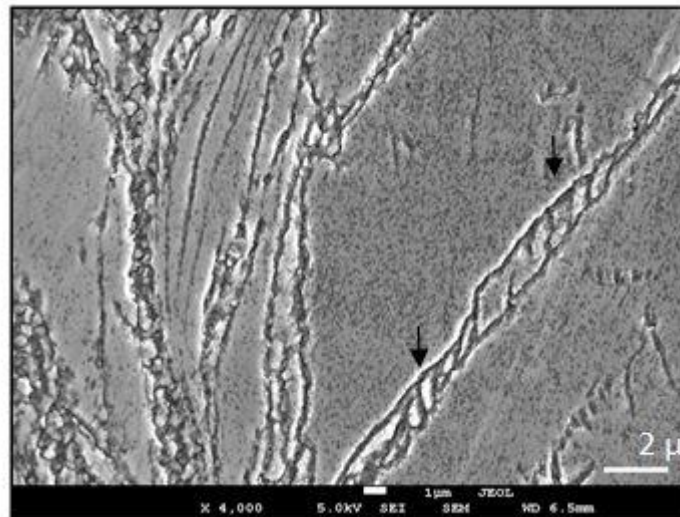


Fig. 6. Recrystallized grains within twin during in an ABE AZ31 alloy

3.4. Continuous Recrystallization

Kim and Kim [46] found a bimodal distribution of grain size after 1 extrusion pass; because the existence of equiaxed grains was discovered after the passes that followed, they suggested that dynamic recrystallization occurred during ECAE. However, they believed that the generation of new grains was

not due to a process of nucleation and growth, but to the gradual transformation of low-angle sub-grain boundaries into high-angle grain boundaries through the absorption of dislocations generated during ECAE, as was proposed for aluminum. Also, Jin et al. [34] suggested that the process of grain refinement consisted of the gradual transformation of

low-angle sub-grain boundaries into high-angle grain boundaries due to continuous dynamic recovery and recrystallization. Similarly, Miure and Sakai [65] reported that dynamic grain evolution in Mg alloy during multiaxial forging can be resulted from grain fragmentation by kink band taking place in original grain interiors, and so controlled by deformation-induced continuous reactions assisted by dynamic recovery, i.e. continuous DRX.

The earlier studies on DRX in AZ31 alloy have denoted that the recrystallized grains remain virtually unchanged as deformation proceeds [17]. It has also been observed during ECAP of AZ31 alloy that once dynamic recrystallization takes place and new grains are formed, dislocation density will decrease sharply at these regions, although the fraction of fine grains increases with ECAP pass number [65]. This ends to the substitution of initial grains by DRX fine grains during subsequent deformation pass, but no evolution was considered for the pre-formed DRX grains. In addition, Ding et al. [17] believed that the size of the fine grains obtained during

ECAP was not gradually refined by successive extrusion passes. However, the EBSD observation made by present author points out that additional refinement may be achieved through applying the consecutive pass [53]. New boundaries with low to medium angle disorientation are developed within the grains. It appears that, upon further straining, the LABs transform into high angle boundaries (HABs) and subdivide the DRXed grains to finer ones.

This is typically considered as continuous grain refinement. In order to precisely characterize this repetitive grain refinement, the boundary spacing histogram was measured for the experimental material deformed to different passes, through counting the HABs and LABs characterized by $>5^\circ$ disorientation. Thus, the histogram includes the distances between boundaries and sub-boundaries. As illustrated in Figure 7, the boundary spacing curve shifts to smaller one with increasing strain, which denotes the development of new LAB and HABs within prior grains. This evolution brings the smallest average spacing down to about 0.5 μm after four passes.

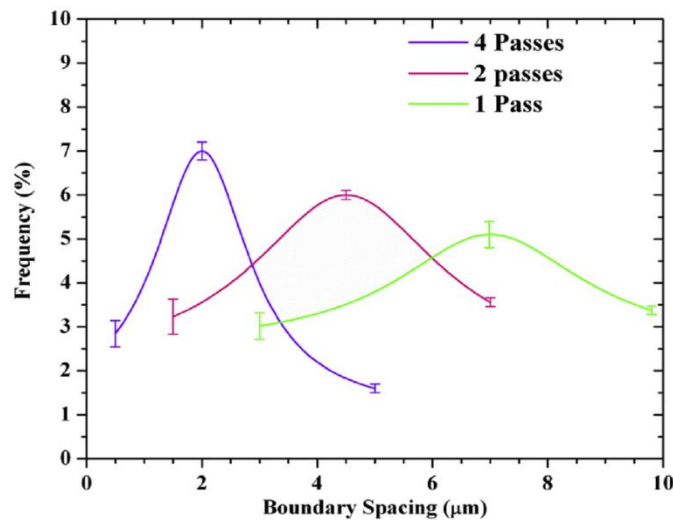


Fig. 7. Boundary spacing histogram in AZ31 alloy deformed to various number of ABE passes [53]

4. Effect of Temperature

When the temperature alters the deformation mechanisms (such as from twinning to slip), the relative activities of these mechanisms, or the fraction of recrystallized grains, noticeable transitions in the microstructure features with temperature will occur. As the new grains formed during SPD are developed through dynamic recrystallization process, it is

therefore expected that a lower temperature gives a smaller grain size. Despite the fact that a low extrusion temperature is desirable for producing an ultrafine-grained Mg alloy, there is a minimum workable extrusion temperature. Accordingly, the lower the extrusion temperature used, the finer the grain size obtained.

Considering the temperature effect, a procedure of “successive decreasing temperatures for each deformation pass” has been proposed. Grain sizes smaller than 500 nm were obtained in an AZ31 following ECAP at 150 and 100°C with a moderate back pressure of 50 MPa [12], while a more complicated multiple-temperature scheme from 200 to 115°C using an ECAP die of 120° resulted in grains of 370nm with yield strength of 372MPa [17]. Compared to Mg alloys, it is more critical to process pure magnesium at lower temperatures to prevent excessive grain growth. Ultrafine-grained pure magnesium with an average grain size of 0.8µm was produced by multi-pass ECAP at room temperature with the application of a back pressure [66].

It should be noticed that larger DRX grain size at high temperature would result a more homogenous final microstructure. For example, a bimodal inhomogeneous microstructure was obtained after ECAP processing of an AZ31 alloy with initial grain size of 22 µm at 150°C, while increasing the processing temperature to 200°C yielded a homogenous microstructure [6]. Ding et al. [17] showed that when the Zener–Hollomon parameters are plotted against the grain sizes (d), the relationship $d = AZ^n$ can be established for the new grains formed during ECAP, where A and n are the fitting parameters. Similar results were reported by Janeček et al. [67] for an AZ31 alloy processed by EX-ECAP. However, their results indicate that the

dislocations generated by ECAP after two passes did not reach the level necessary for the formation of dislocation cell-structure and further development of HAB. Because of high processing temperature, the dislocations annihilation process by the dynamic recovery occurred fast enough such that the grains could not be further refined.

Yoshida et al. [68] followed the texture evolution in AZ31 alloy during one pass at two different temperatures, 250 and 300 °C. At both temperatures, the dominant deformation mechanism changed during the course of one pass. Although basal slip is by far the easiest slip mode, the initial texture induced non-basal slip activity in the initial stages of deformation. At the lower temperature, tensile twinning and prismatic were active, but at the higher temperature pyramidal $\langle c + a \rangle$ was active.

At elevated temperatures, annihilation of dislocations by cross slip becomes easier. Dislocations, therefore, have a greater opportunity to be annihilated within grain interiors at a higher temperature, and consequently the absorption of dislocations into a boundary becomes less frequent, so that the increase of boundary disorientation becomes less efficient at elevated temperatures. This is in agreement with the electron back scattered diffraction (EBSD) results obtained after single pass ABE process, where the share of low angle boundaries was increased with processing temperature (Fig. 8).

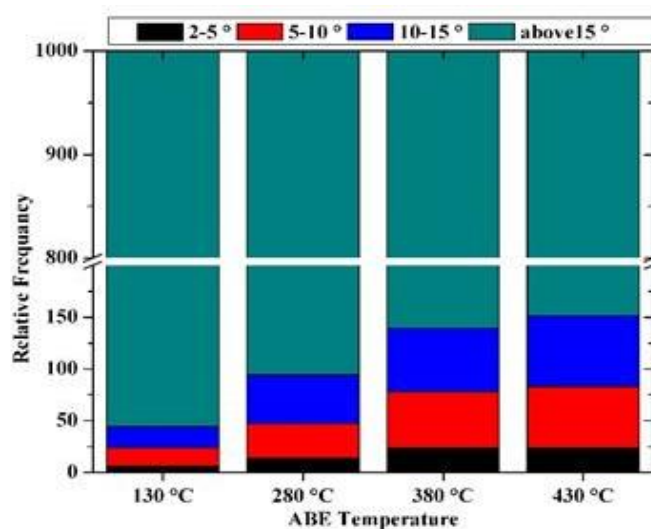


Fig. 8. The distribution of boundary misorientation in AZ31 alloy after single pass ABE at different temperature

Conclusions

- A complex contribution of different recrystallization mechanism may be realized during severe deformation of magnesium alloy.
- Twinning may play significant role during SPD through twin segmentation into ultrafine grains.
- DDRX may be operative at initial stages of SPD route, whereas CDRX may further refine the recrystallized grains during consecutive passes.
- Formation of well-distributed shear band during SPD of magnesium alloy may play an effective role in grain refinement with no detrimental effect.
- Higher temperature results in a more homogenous final microstructure after SPD processing as well as an increased share of low angle grain boundary.

References

- [1]. Agnew, S., Plastic anisotropy of magnesium alloy AZ31B sheet, in: *Magnesium Technology*, TMS 2002, pp. 169-174.
- [2]. Del Valle, J., Ruano, O., Influence of texture on dynamic recrystallization and deformation mechanisms in rolled or ECAPed AZ31 magnesium alloy, *Materials Science and Engineering: A*, Vol. 487 (2008) pp. 473-480.
- [3]. Kim, H. K., The grain size dependence of flow stress in an ECAPed AZ31 Mg alloy with a constant texture, *Materials Science and Engineering: A*, Vol. 515 (2009) pp. 66-70.
- [4]. Zhu, Y. T., Lowe, T. C., Observations and issues on mechanisms of grain refinement during ECAP process, *Materials Science and Engineering: A*, Vol. 291 (2000) pp. 46-53.
- [5]. Figueiredo, R., Cetlin, P., Langdon, T., The processing of difficult-to-work alloys by ECAP with an emphasis on magnesium alloys, *Acta Materialia*, Vol. 55 (2007) pp. 4769-4779.
- [6]. Figueiredo, R. B., Langdon, T. G., Grain refinement and mechanical behavior of a magnesium alloy processed by ECAP, *Journal of materials science*, Vol. 45 (2010) pp. 4827-4836.
- [7]. Lin, H., Huang, J., Langdon, T., Relationship between texture and low temperature superplasticity in an extruded AZ31 Mg alloy processed by ECAP, *Materials Science and Engineering: A*, Vol. 402 (2005) pp. 250-257.
- [8]. Biswas, S., Suwas, S., Evolution of sub-micron grain size and weak texture in magnesium alloy Mg-3Al-0.4 Mn by a modified multi-axial forging process, *Scripta Materialia*, Vol. 66 (2012) pp. 89-92.
- [9]. Del Valle, J., Pérez-Prado, M., Ruano, O., Accumulative roll bonding of a Mg-based AZ61 alloy, *Materials Science and Engineering: A*, Vol. 410 (2005) pp. 353-357.
- [10]. Xu, C., Xia, K., Langdon, T. G., Processing of a magnesium alloy by equal-channel angular pressing using a back-pressure, *Materials Science and Engineering: A*, Vol. 527 (2009) pp. 205-211.
- [11]. Mabuchi, M., Iwasaki, H., Yanase, K., Higashi, K., Low temperature superplasticity in an AZ91 magnesium alloy processed by ECAP, *Scripta materialia*, Vol. 36 (1997) pp. 681-686.
- [12]. Matsubara, K., Miyahara, Y., Horita, Z., Langdon, T., Developing superplasticity in a magnesium alloy through a combination of extrusion and ECAP, *Acta materialia*, Vol. 51 (2003) pp. 3073-3084.
- [13]. Lapovok, R., Thomson, P., Cottam, R., Estrin, Y., The effect of grain refinement by warm equal channel angular extrusion on room temperature twinning in magnesium alloy ZK60, *Journal of Materials Science*, Vol. 40 (2005) pp. 1699-1708.
- [14]. Furui, M., Kitamura, H., Anada, H., Langdon, T. G., Influence of preliminary extrusion conditions on the superplastic properties of a magnesium alloy processed by ECAP, *Acta Materialia*, Vol. 55 (2007) pp. 1083-1091.
- [15]. Figueiredo, R. B., Langdon, T. G., Principles of grain refinement in magnesium alloys processed by equal-channel angular pressing, *Journal of materials science*, Vol. 44 (2009) pp. 4758-4762.
- [16]. Lapovok, R., Estrin, Y., Popov, M. V., Langdon, T. G., Enhanced Superplasticity in a Magnesium Alloy Processed by Equal-Channel Angular Pressing with a Back-Pressure, *Advanced Engineering Materials*, Vol. 10 (2008) pp. 429-433.
- [17]. Ding, S., Chang, C., Kao, P., Effects of processing parameters on the grain refinement of magnesium alloy by equal-channel angular extrusion, *Metallurgical and Materials Transactions A*, Vol. 40 (2009) pp. 415-425.
- [18]. Ding, R., Chung, C., Chiu, Y., Lyon, P., Effect of ECAP on microstructure and mechanical properties of ZE41 magnesium

- alloy, *Materials Science and Engineering: A*, Vol. 527 (2010) pp. 3777-3784.
- [19]. Kim, W., Yoo, S., Chen, Z., Jeong, H., Grain size and texture control of Mg-3Al-1Zn alloy sheet using a combination of equal-channel angular rolling and high-speed-ratio differential speed-rolling processes, *Scripta materialia*, Vol. 60 (2009) pp. 897-900.
- [20]. Zhilyaev, A. P., Langdon, T. G., Using high-pressure torsion for metal processing: Fundamentals and applications, *Progress in Materials Science*, Vol. 53 (2008) pp. 893-979.
- [21]. Pérez-Prado, M., Ruano, O., Grain refinement of Mg–Al–Zn alloys via accumulative roll bonding, *Scripta Materialia*, Vol. 51 (2004) pp. 1093-1097.
- [22]. Xing, J., Soda, H., Yang, X., Miura, H., Sakai, T., Ultra-fine grain development in an AZ31 magnesium alloy during multi-directional forging under decreasing temperature conditions, *Materials transactions*, Vol. 46 (2005) pp. 1646-1650.
- [23]. Miura, H., Yu, G., Yang, X., Multi-directional forging of AZ61Mg alloy under decreasing temperature conditions and improvement of its mechanical properties, *Materials Science and Engineering: A*, Vol. 528 (2011) pp. 6981-6992.
- [24]. Miura, H., Maruoka, T., Yang, X., Jonas, J., Microstructure and mechanical properties of multi-directionally forged Mg–Al–Zn alloy, *Scripta Materialia*, Vol. 66 (2012) pp. 49-51.
- [25]. Fatemi-Varzaneh, S., Zarei-Hanzaki, A., Paul, H., Characterization of ultrafine and nano grained magnesium alloy processed by severe plastic deformation, *Materials Characterization*, Vol. 87 (2014) pp. 27-35.
- [26]. Yamashita, A., Horita, Z., Langdon, T., Improving the mechanical properties of magnesium and a magnesium alloy through severe plastic deformation, *Materials Science and Engineering A*, Vol. 300 (2001) pp. 142-147.
- [27]. Horita, Z., Matsubara, K., Makii, K., Langdon, T. G., A two-step processing route for achieving a superplastic forming capability in dilute magnesium alloys, *Scripta Materialia*, Vol. 47 (2002) pp. 255-260.
- [28]. Figueiredo, R. B., Langdon, T. G., Principles of grain refinement and superplastic flow in magnesium alloys processed by ECAP, *Materials Science and Engineering: A*, Vol. 501 (2009) pp. 105-114.
- [29]. Kim, W., Park, J., Wang, J., Yoon, W., Realization of low-temperature superplasticity in Mg–Al–Zn alloy sheets processed by differential speed rolling, *Scripta materialia*, Vol. 57 (2007) pp. 755-758.
- [30]. Máthis, K., Gubicza, J., Nam, N., Microstructure and mechanical behavior of AZ91 Mg alloy processed by equal channel angular pressing, *Journal of Alloys and Compounds*, Vol. 394 (2005) pp. 194-199.
- [31]. Kim, W., Hong, S., Kim, Y., Min, S., Jeong, H., Lee, J., Texture development and its effect on mechanical properties of an AZ61 Mg alloy fabricated by equal channel angular pressing, *Acta Materialia*, Vol. 51 (2003) pp. 3293-3307.
- [32]. Guo, Q., Yan, H., Chen, Z., Zhang, H., Grain refinement in as-cast AZ80 Mg alloy under large strain deformation, *Materials Characterization*, Vol. 58 (2007) pp. 162-167.
- [33]. 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*, Vol. 30 (2009) pp. 4557-4561.
- [34]. Jin, L., Lin, D., Mao, D., Zeng, X., Chen, B., Ding, W., Microstructure evolution of AZ31 Mg alloy during equal channel angular extrusion, *Materials Science and Engineering: A*, Vol. 423 (2006) pp. 247-252.
- [35]. Serre, P., Figueiredo, R. B., Gao, N., Langdon, T. G., Influence of strain rate on the characteristics of a magnesium alloy processed by high-pressure torsion, *Materials Science and Engineering: A*, Vol. 528 (2011) pp. 3601-3608.
- [36]. Fatemi-Varzaneh, S., Zarei-Hanzaki, A., Beladi, H., Dynamic recrystallization in AZ31 magnesium alloy, *Materials Science and Engineering: A*, Vol. 456 (2007) pp. 52-57.
- [37]. Berbon, P. B., Langdon, T. G., Tsenev, N. K., Valiev, R. Z., Furukawa, M., Horita, Z., Nemoto, M., Fabrication of bulk ultrafine-grained materials through intense plastic straining, *Metallurgical and Materials Transactions A*, Vol. 29 (1998) pp. 2237-2243.
- [38]. Xu, C., Horita, Z., Langdon, T. G., The evolution of homogeneity in an aluminum alloy processed using high-pressure torsion, *Acta materialia*, Vol. 56 (2008) pp. 5168-5176.
- [39]. Xu, C., Horita, Z., Langdon, T. G., The evolution of homogeneity in processing by

- high-pressure torsion, *Acta materialia*, Vol. 55 (2007) pp. 203-212.
- [40]. Fukuda, Y., Oh-Ishi, K., Horita, Z., Langdon, T., Processing of a low-carbon steel by equal-channel angular pressing, *Acta Materialia*, Vol. 50 (2002) pp. 1359-1368.
- [41]. Kang, F., Wang, J. T., Peng, Y., Deformation and fracture during equal channel angular pressing of AZ31 magnesium alloy, *Materials Science and Engineering: A*, Vol. 487 (2008) pp. 68-73.
- [42]. Fatemi-Varzaneh, S., Zarei-Hanzaki, A., Processing of AZ31 magnesium alloy by a new noble severe plastic deformation method, *Materials Science and Engineering: A*, Vol. 528 (2011) pp. 1334-1339.
- [43]. Langdon, T. G., The principles of grain refinement in equal-channel angular pressing, *Materials Science and Engineering: A*, Vol. 462 (2007) pp. 3-11.
- [44]. Kuhlmann-Wilsdorf, D., High-strain dislocation patterning, texture formation and shear banding of wavy glide materials in the LECD theory, *Scripta materialia*, Vol. 36 (1997) pp.173-181.
- [45]. Kawasaki, M., Horita, Z., Langdon, T. G., Microstructural evolution in high purity aluminum processed by ECAP, *Materials Science and Engineering: A*, Vol. 524 (2009) pp. 143-150.
- [46]. Kim, H., Kim, W., Microstructural instability and strength of an AZ31 Mg alloy after severe plastic deformation, *Materials Science and Engineering A*, Vol. 385 (2004) pp. 300-308.
- [47]. Xia, K., Wang, J., Wu, X., Chen, G., Gurvan, M., Equal channel angular pressing of magnesium alloy AZ31, *Materials Science and Engineering: A*, Vol. 410 (2005) pp. 324-327.
- [48]. kaibyshev, R., Sitdikov, O., On bulging mechanism of dynamic recrystallisation in: Third International Conference on Recrystallization and Related Phenomena, 1996.
- [49]. Galiyev, A., Kaibyshev, R., Gottstein, G., Correlation of plastic deformation and dynamic recrystallization in magnesium alloy ZK60, *Acta materialia*, Vol. 49 (2001) pp. 1199-1207.
- [50]. Kaibyshev, R., Galiev, A., Sitdikov, O., On the possibility of producing a nanocrystalline structure in magnesium and magnesium alloys, *Nanostructured Materials*, Vol. 6 (1995) pp. 621-624.
- [51]. Furukawa, M., Utsunomiya, A., Matsubara, K., Horita, Z., Langdon, T., Influence of magnesium on grain refinement and ductility in a dilute Al-Sc alloy, *Acta materialia*, Vol. 49 (2001) pp. 3829-3838.
- [52]. Sun, D., Chang, C., Kao, P., Microstructural Aspects of Grain Boundary Bulge in a Dynamically Recrystallized Mg-Al-Zn Alloy, *Metallurgical and Materials Transactions A*, Vol. 41 (2010) pp. 1864-1870.
- [53]. Fatemi-Varzaneh, S., Zarei-Hanzaki, A., Cabrera, J., Calvillo, P., EBSD characterization of repetitive grain refinement in AZ31 magnesium alloy, *Materials Chemistry and Physics*, Vol. 149 (2015) pp. 339-343.
- [54]. Wu, P., Chang, C., Kao, P., The distribution of dislocation walls in the early processing stage of equal channel angular extrusion, *Materials Science and Engineering: A*, Vol. 374 (2004) pp. 196-203.
- [55]. Segal, V., Deformation mode and plastic flow in ultra fine grained metals, *Materials Science and Engineering: A*, Vol. 406 (2005) pp. 205-216.
- [56]. Huang, X., Suzuki, K., Watazu, A., Shigematsu, I., Saito, N., Microstructural and textural evolution of AZ31 magnesium alloy during differential speed rolling, *Journal of Alloys and Compounds*, Vol. 479 (2009) pp. 726-731.
- [57]. Klimanek, P., Pöttsch, A., Microstructure evolution under compressive plastic deformation of magnesium at different temperatures and strain rates, *Materials Science and Engineering A*, Vol. 324 (2002) pp. 145-150.
- [58]. Lapovok, R., Tóth, L., Molinari, A., Estrin, Y., Strain localisation patterns under equal-channel angular pressing, *Journal of the Mechanics and Physics of Solids*, Vol. 57 (2009) pp. 122-136.
- [59]. Quadir, M., Ferry, M., Al-Buhamad, O., Munroe, P., Shear banding and recrystallization texture development in a multilayered Al alloy sheet produced by accumulative roll bonding, *Acta Materialia*, Vol. 57 (2009) pp. 29-40.
- [60]. Fatemi-Varzaneh, S., Zarei-Hanzaki, A., Naderi, M., Roostaei, A. A., Deformation homogeneity in accumulative back extrusion processing of AZ31 magnesium alloy, *Journal of Alloys and Compounds*, Vol. 507 (2010) pp. 207-214.

- [61]. Murr, L., Trillo, E., Pappu, S., Kennedy, C., Adiabatic shear bands and examples of their role in severe plastic deformation, *Journal of materials science*, Vol. 37 (2002) pp. 3337-3360.
- [62]. Su, C., Lu, L., Lai, M., A model for the grain refinement mechanism in equal channel angular pressing of Mg alloy from microstructural studies, *Materials Science and Engineering: A*, Vol. 434 (2006) pp. 227-236.
- [63]. Barnett, M., Keshavarz, Z., Beer, A., Ma, X., Non-Schmid behaviour during secondary twinning in a polycrystalline magnesium alloy, *Acta materialia*, Vol. 56 (2008) pp. 5-15.
- [64]. Sakai, T., Miura, H., Mechanical Properties of Fine-Grained Magnesium Alloys Processed by Severe Plastic Forging, *Intechopen*.
- [65]. Eskandari, M., Ductility Improvement in AZ31 Magnesium Alloy Using Constrained Compression Testing Technique”, *Materials Science and Engineering A*, Vol. 576 (2013) pp. 74-81.
- [66]. Kim, W., An, C., Kim, Y., Hong, S., Mechanical properties and microstructures of an AZ61 Mg alloy produced by equal channel angular pressing, *Scripta materialia*, Vol. 47 (2002) pp. 39-44.
- [67]. Li, J., Xu, W., Wu, X., Ding, H., Xia, K., Effects of grain size on compressive behaviour in ultrafine grained pure Mg processed by equal channel angular pressing at room temperature, *Materials Science and Engineering: A*, Vol. 528 (2011) pp. 5993-5998.
- [68]. Janeček, M., Yi, S., Král, R., Vrátná, J., Kainer, K., Texture and microstructure evolution in ultrafine-grained AZ31 processed by EX-ECAP, *Journal of materials science*, Vol. 45 (2010) pp. 4665-4671.
- [69]. Yoshida, Y., Cisar, L., Kamado, S., Kojima, Y., Effect of microstructural factors on tensile properties of an ECAE-processed AZ31 magnesium alloy, *Materials transactions*, Vol. 44 (2003) pp. 468-475.