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Research Paper

Effects of Equal Channel Angular Pressing (ECAP) Process with an Additional Expansion-Extrusion Stage on Microstructure and Mechanical Properties of Mg–9Al–1Zn

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ABSTRACT

In this study, equal channel angular pressing (ECAP) process with an additional expansion-extrusion stage named expansion-extrusion equal channel angular pressing (EECAP) process is utilized for producing bulk ultrafine grained (UFG) Mg–9Al–1Zn magnesium alloy. In this process, cyclic expansion-extrusion (CEE) and equal channel angular pressing (ECAP) processes are combined. AZ91 magnesium alloy was used for this experiment. An FEM simulation was performed to calculate the compressive hydrostatical stress. Furthermore, microstructural and mechanical properties were investigated. The experimental findings revealed that the ductility of the sample remains almost constant (0.03% drop) while ultimate tensile strength increased about 30%. The average value of microhardness improved from 48 Hv to 65 Hv (35%) and grain size refined from 144 µm to 3.4 µm. Despite the expansion part of the die, there is no need for back pressure. Another advantage of this new method is maintaining ductility while strengthening.

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1. Introduction

Severe plastic deformation (SPD) processes are well-known methods to improve the mechanical properties of metals through producing bulk UFG (100 nm < d < 1 μm) and nanocrystalline (NC) (d < 100 nm) structured materials [1, 2]. The material is subjected to shear deformation in SPD processes, resulting in an intense plastic strain. This leads to distorted grain boundaries with higher energy, promoting segregations and mechanical mixing that affect the mechanical properties [3]. Decades of research have been dedicated to developing and modifying the SPD techniques. Equal channel angular pressing (ECAP) [1, 2] and Cyclic extrusion compression (CEC) [3, 4] are two of the old and common SPD techniques for processing bulk metals. ECAP includes pressing of material into equal angular channels. In the CEC process, the sample is extruded back and forth. Also, due to the compression of the sample from both sides of the CEC die, large hydrostatic compressive stress is induced, and there is no need to remove the sample until the desired number of passes is conducted. Cyclic expansion-extrusion (CEE) [5] is a modified process introduced to eliminate the need for external backpressure. The CEE process is performed in two steps: expansion and extrusion. The expansion step provides the backpressure needed for extrusion, and the cycle can be repeated for any desirable passes without removing the sample from the die. Recent studies aim to fabricate finer grains through one pass of hybrid SPD techniques like Exp-ECAE [6] and CECAP [7]. In the Exp-ECAE process, an expansion cavity is located at the corner of channels, resulting in larger strains and consequently more grain refinement compared with conventional ECAP. CECAP is a result of the combination of CEC and ECAP, thus eliminating their drawbacks. In CECAP, two extrusion steps result in high hydrostatic pressure, which leads to ductility and strength improvement. Ensafi et al. [1] reported that CECAP includes significantly higher hydrostatic pressure than conventional ECAP. Also, they saw that after applying CECAP, the ultimate strength of AZ91 magnesium alloy was enhanced from 144 MPa to 234 MPa, and ductility increased from an initial value of 4% to 8% due to the effects of high hydrostatic compressive stress. In fact, hydrostatic pressure delays the crack initiation and also leads to closing the cracks and cavities or reducing the growth of cracks; these cause achieving higher ductility [2]. Besides SPD methods, other mechanical methods such as radial-shear rolling [1] by crushing the microstructure can produce ultrafine-grain materials with higher strength. Magnesium alloys are very popular due to their lightweight, high specific strength, damping capacity, and dimensional stability. Alloy AZ91, Mg (base)–Al 9%–Zn 1%–Mn 0.2%, is the cost-effective and most widely used magnesium because of its high strength, relatively excellent corrosion resistance, and good castability [2]. Since the HCP crystalline structure restricts slip to the basal planes, magnesium is difficult to deform at room temperature plastically [3]. SPD techniques at high temperatures have been developed to improve the mechanical behavior of Mg alloys.

In this study, the expansion-extrusion equal channel angular pressing (EECAP) process, which results from the combination of ECAP and CEE processes, is performed on AZ91 magnesium alloy. The extrusion section has been placed after the expansion part, and then the ECAP channel eliminates the need for backpressure. Low force requirement and simple design of the ECAP channel with a slight modification are the advantages of this method. The present investigation was initiated to examine the mechanics of this process, microstructure evolution, and mechanical properties of AZ91 alloy.

2. Principles of the EECAP process

Fig. 1 shows the schematic representation of the EECAP process. The cylindrical metal is pressed through the die using a punch (Fig. 1a). As the material reaches the end of the channel, the flow of the material fills the expanded part of the die (Fig. 1b,c) and expands in diameter. Then it is extruded to its initial dimension and finally is side extruded and flows through the 90-degree exit channel (Fig. 1d). Because of the angular part of the die, there will be no need for backpressure. For applying subsequent pass of EECAP, it is enough that another sample is inserted in the die and then pressed through the die using the punch until the first sample comes out from the die. Next, the first sample is inserted into the die again and then pressed until the second sample comes out from the die. Then, this cycle is repeated until the first sample comes out from the die for the second time. In this way, the two-pass EECAP processed sample is obtained. The effective plastic strain due to shear deformation during a one-pass ECAP is [1]:

$$\bar{\varepsilon}_1 = \frac{1}{\sqrt{3}}\left[2\cot\left(\frac{\phi}{2}+\frac{\psi}{2}\right)+\psi\cos ec\left(\frac{\phi}{2}+\frac{\psi}{2}\right)\right] \tag{1}$$

And the equivalent plastic strain in a one-pass CEE process is [2]:

$$\bar{\varepsilon}_2 = 4\ln\frac{D}{d} \tag{2}$$

The total plastic strain at one-pass EECAP is calculated from the Eq. (3)

$$\bar{\varepsilon}_{total} = \bar{\varepsilon}_1 + \bar{\varepsilon}_2 = \frac{1}{\sqrt{3}}\left[2\cot\left(\frac{\phi}{2}+\frac{\psi}{2}\right)+\psi\,\mathrm{cosec}\left(\frac{\phi}{2}+\frac{\psi}{2}\right)\right]+4\ln\frac{D}{d} \tag{3}$$

For this study, with considering the EECAP die parameters, the total plastic strain is obtained from Eq3: ε_{total}=2.02. As seen, the EECAP process induces significant plastic strain on the material. Thus, it is expected that more grain refinement is obtained compared with conventional ECAP. Also, as illustrated in the next sections, EECAP includes higher hydrostatic pressure leading to keeping ductility while strengthening of the Mg alloy.

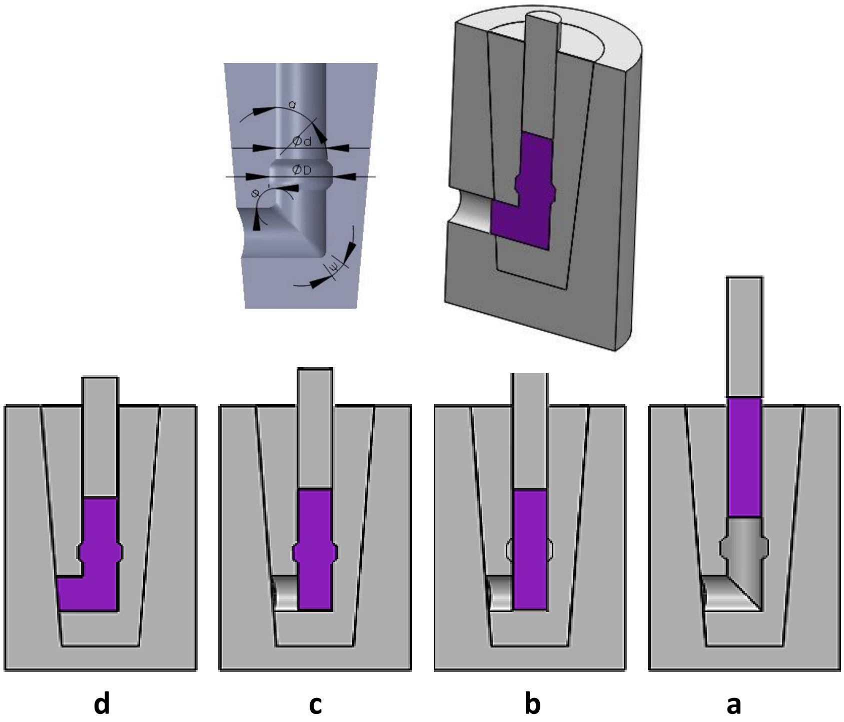

Fig. 1. Schematic of the EECAP process and die parameters and different stages of EECAP process.

3. Experimental and FEM procedures

An ingot of AZ91 alloy was machined to two cylindrical samples of 15 mm diameter and length of 50 mm. The EECAP die is made from H13 hot work tool steel. The die design is shown in Fig. 1. Two samples were pressed with HSS-Co5 punch through the EECAP channel at a constant temperature using a 100-ton hydraulic press (ram speed of 10mm/s). We used an electric heater to keep the temperature at the precise value of 330 °C. A thermocouple was attached to the heater to control the process temperature. In order to reduce friction, MoS_2 was used as a lubricant on the internal surface of the channel and around the sample. The first sample was pulled out of the die, and the second one remained in the channel as proof of the filled expansion region. Samples were cooled to room temperature by air. Fig. 2 shows the EECAP die equipment and the two consecutive EECAP processed samples; the first sample was processed entirely and came out from the die. The microstructure and microhardness of the second sample was investigated, and also a tensile test was conducted on the first sample using a SANTAM tensile machine at room temperature. The tensile test was carried out at a speed of 1 mm/min. The dimension of dog bone shape tensile test sample was selected based on ASTM- E8 Standards E8 (4 mm diameter and gauge length of 16.2 mm) from the initial and processed samples. The second sample was cut along the parallel to its axis to study the microstructure and homogeneity by optical microscopy (OM). The sample was polished and then etched (etchant Composition: 4.2 gr Picric Acid, 10 cc Acetic Acid, 10 cc water, 70 cc Ethanol). After preparing the microstructure images by OM, J-micro vision image analysis software was used to estimate the grain size. Vickers hardness test method using an optical measurement system (a Coopa microhardness testing machine) was performed at room temperature, and the sample was subjected to a load of 500 gr for 30 s. Commercial finite element method (FEM) software DEFORM^{TM} -3D V6.1 was used for the numerical study of the EECAP process. 3D model of the punch, die, and workpiece was designed by Solidworks 2016. The geometry of the specimen and die is similar to the experiment. To decrease the calculation times, half of the sample and dies were modeled. Punch and die were modeled to be rigid, and the workpiece with mechanical properties of AZ91 (as an elastic-plastic material model) was meshed by 50000 tetrahedral elements. Young modulus and Poisson ratio were considered as 41 GPa and 0.35, respectively. The friction coefficient was set to 0.1 between all contact surfaces. Displacement of the punch in every step was considered 0.1 mm.

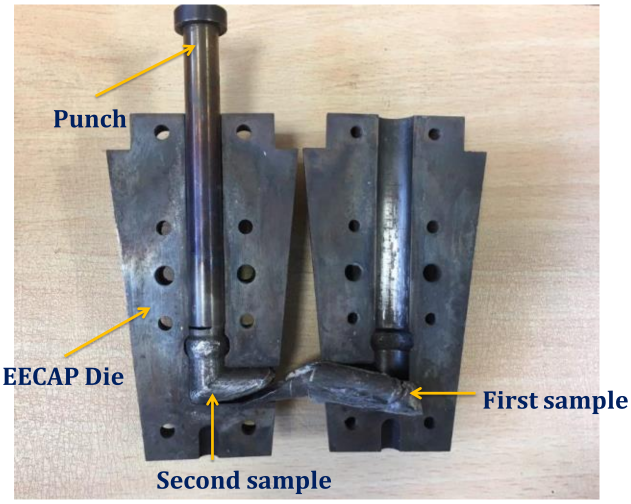

Fig. 2. Punch, die and sample after experiment

4. Results and discussion

Magnesium alloys include very weak workability and formability at room temperature; this is attributed to their HCP (hexagonal close-packed) crystal structure and an inadequate number of slip systems. The basal slip is the main slip system in the deformation of Magnesium at room temperature [3, 4]. By increasing temperature (at temperatures over 225°C), the further slip systems of magnesium alloys are activated, causing the enhancement of formability and ductility [5, 6]. In this study, the EECAP processing of AZ91 was done at 330 °C. Fig. 3 shows the microstructure obtained by OM from the deformation zone of the EECAP processed sample. Through the path shown in Fig. 3a the grain size is reduced because the material experiences more plastic strain. According to J-micro vision software, after one pass of EECAP, the grain size was refined from 144 µm to 3.4 µm. AZ91 alloy microstructure consists of a solid solution of α-Mg phase and an intermetallic compound β-Mg phase ($Mg_{17}Al_{12}$) at the grain boundaries and another intermetallic compound of $Mg_{17}Al_{11.5}Zn_{0.5}$ or $Mg_{17}(Al,Zn)_{12}$. Due to the HCP structure of α phase, poor ductility is expected but distribution of bcc structure of β phase can help the deformation process [3, 4]. Braszczynska-Malik claimed that during the ECAP process, plate-like precipitates morphology changes into a spherical shape in order to minimize the total energy (elastic strain energy and interfacial energy) [5]. One could spot these particles as dots inside the coarse grains. HCP structure has a limited number of slip systems, so plastic deformation happens via dislocation slips and deformation twinning [6]. Basal slip system (α-type) and non-basal slip system named (c+α) (pyramidal)-type is activated at elevated temperature; thus, dislocation reaction between two types arises. Fig. 3 shows the presence of twinning, which increases the strength and ductility. As observed in Fig. 3c,d, by applying a certain level of plastic strains at a temperature of 330 °C, dynamically recrystallized (DRXed) twins are formed. Indeed, along the deformation path (Fig. 3b-f) at 330 °C partial recrystallization occurs near twin or grain boundaries. Dynamically recrystallization (DRX) behavior in the boundaries and the original grains during hot deformation results in grain refinement. During DRX, a microstructure containing finer and more equiaxed grains is developed. This microstructure, with refined and equiaxed grains, can enhance the ductility and superplastic behavior of magnesium alloys [6]. On the other hand, by applying severe plastic strains, the hard β phase networks are broken and then distributed randomly through the grains. β phase has a pining effect on the recrystallized grains and prevents them from growth. Precipitated β phase and grain refinement cause mechanical property improvement [6-9]. The more homogeneous and equiaxed β phase, the more improvement of formability and mechanical properties of magnesium alloys [10]. Also, it was reported that the β-$Mg_{17}Al_{12}$ phase could be helpful in improving the corrosion resistance of AZ91 alloys [11, 12]. Finally, at the end of the EECAP process, fine grains can be seen in a heterogeneous pattern which is predictable after only one pass (Fig. 3f). In other words, point f, which experienced the highest plastic strain at relatively high temperature, includes more DRX and consequently more grain refinement. In Table 1, the obtained grain size from this study (3.4 μm) is compared with the results of some other SPD techniques. As is obvious, compared to other SPD techniques, the EECAP process led to a significant reduction of grain size.

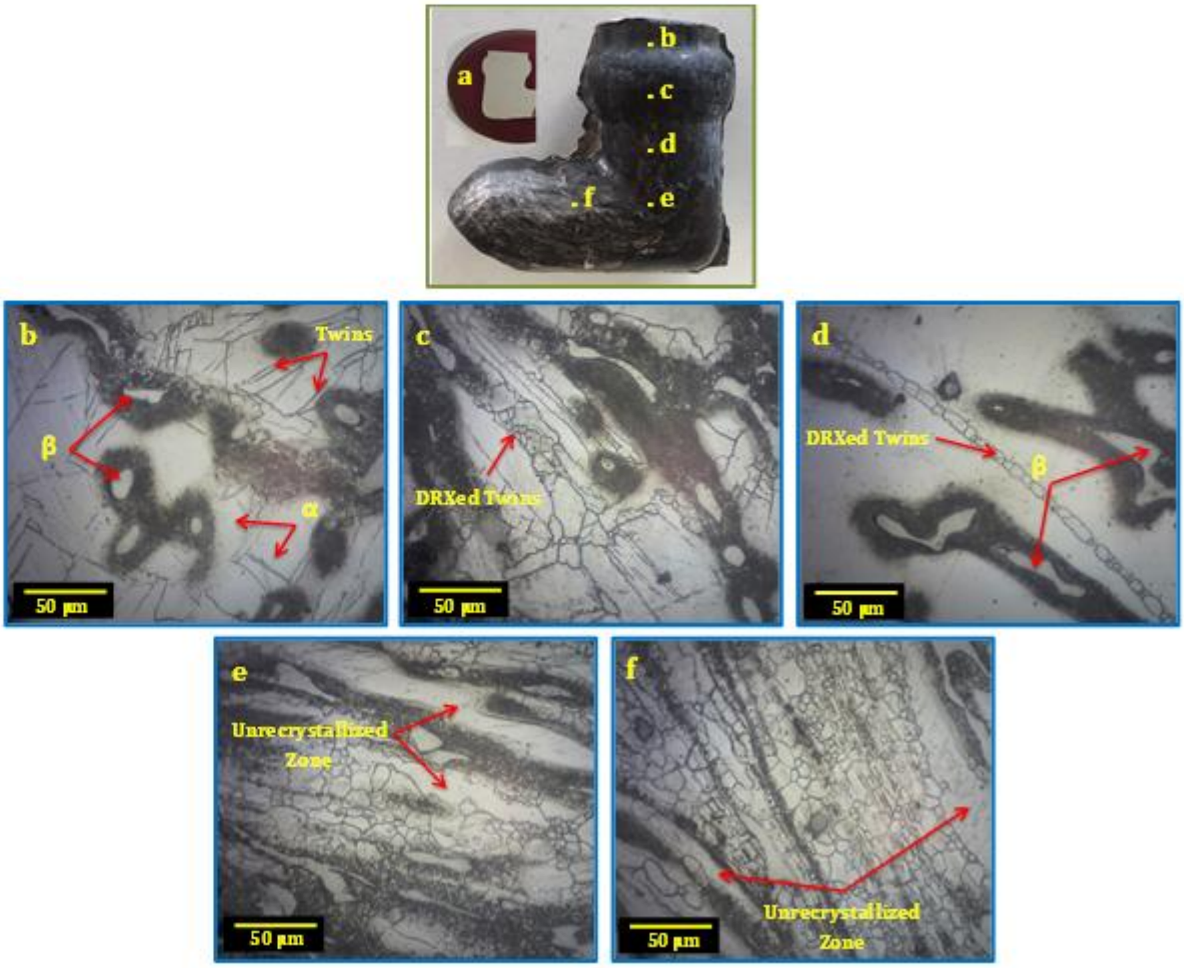

Fig 3. EECAP processed sample (a) and Optical images of EECAP processed AZ91 sample (b-f)

Table 1. The comparison of the mean grain size obtained from this study with that of other techniques performed on AZ91 magnesium alloy

Number	SPD process	Mean grain size (μm)	References
1	1 pass EECAP	3.4	This study
2	1 pass PTCAP	9	[10]
3	1 pass PTCAP + TBE	12.4	[13]
4	1 pass PTCAP	12.6	[13]
5	1 pass CECAP	3	[14]
6	1 pass ECAP	4.3	[9]
7	1 pass ECAP	> 8	[15]
8	1 pass RFE	3	[16]

The reduction of grains size of the magnesium alloys after performing severe plastic deformation (SPD) methods is commonly seen in previous studies [6, 10, 14, 17, 18]. Fig. 4 shows the as-cast sample microstructure and selected regions of the processed sample. The initial unprocessed state of the sample (Z0) contains a dendritic structure with large grains (about 144 μm) with random orientations. During the EECAP process, DRX and breaking β phase occurrence leads to grain refinement. An important factor in HCP structure is grain orientation. Slip activation starts in some grains with proper orientations leading to the dislocation density increment and continues with DRX behavior so that the grain boundaries rotation is forced and high angle boundaries are created [19] and finally results in fine grains surrounding coarse grains. Because of the larger shear strain in the outer regions of Fig. 4a, an inhomogeneous microstructure with finer grains appears from Z5 to Z1, Z2, Z3, and Z4. Region Z5, which is the central region of the EECAP processed sample (as obvious in Fig. 4a), is the least affected and includes coarser grains, β phase networks, and unrecrystallized this region experiences lower strain compared with the other regions. Heterogeneous structure and oriented fine grains can be seen in outer regions.

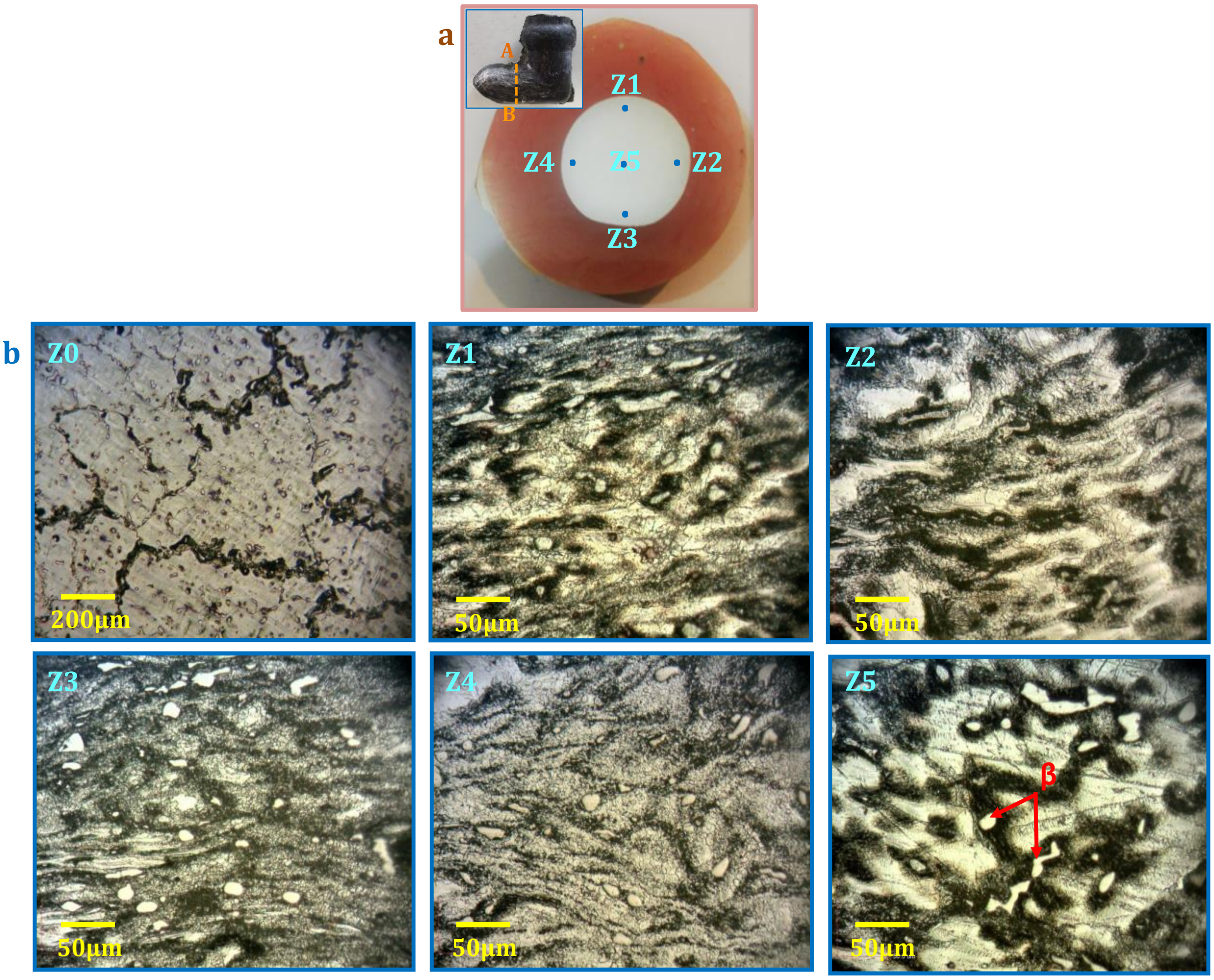

Fig. 4. EECAP processed sample in the A-B transverse cross section (a) and (b) optical images of unprocessed AZ91sample (Z0) and EECAP processed sample (Z1-Z5).

The microhardness test investigates the homogeneity along the deformation path (Fig. 5). The sample was tested along paths (A-B), (C-D), and (E-F). The distance of each point from the next is 10 mm. Fig. 5 demonstrates the comparison of these three paths and as-received hardness. Initial hardness was 48 Hv and was increased to a maximum of 102 Hv. As the paths include expansion, extrusion, and passing through the ECAP channel, inhomogeneity is to be expected. β phase in the grain boundaries is brittle and hard, which can increase the hardness as well as reduction of the grain size. Left (A-B) and right (E-F) paths show higher hardness; however, some irregularities is observed. The homogeneous microstructure can be achieved by increasing the number of passes, which leads to microhardness homogeneity [10, 17, 18]. It was proved that microhardness is a single-valued function of true effective strain [19]. Concerning Fig. 5, at the first distances of the three paths, the value of hardness is lower than that of the end distances because the material, by passage through the deformation regions, experiences more plastic strain and consequently more grain refinement leading to higher values of hardness at the end distances of the paths. Microhardness change during the EECAP process in the transverse cross-section is demonstrated in Fig. 6. The maximum hardness is about 80 Hv. The distribution of β phase, which has a higher hardness than the α-Mg phase [17-19], could cause fluctuations in the hardness profiles of Figs. 5 and 6. In fact, while applying the SPD methods, the hard phase of $Mg_{17}Al_{12}$ can be mostly found in two forms: first, the form of dissolution phase into the grains. Second, the form of rigid particles has a pinning effect. The distribution and dissolution of the $Mg_{17}Al_{12}$ phase are influenced by the deformation temperature and the amount of plastic strain [17]. Furthermore, As we know from the Hall-Petch equation, the smaller the grain size, the higher hardness gets [18]. So, as grain refinement happens, microhardness increases. Owing to the lack of slip systems in the magnesium alloys, the hardness and strength are more dependent on the grain size [19]. As is obvious in Fig. 6, the hardness value of the center of the EECAP processed sample (point 3) is lower than that of points 1 and 5 located on the two ends of both paths of A-B and C-D. This agrees with Fig. 4, which exhibits that the center of the EECAP processed sample (Z5) includes less grain refinement than outer regions. Moreover, studies on CEC and ECAP processes proved that homogenous strain distribution could be achieved by optimizing the die geometry and dimension [20, 21]. However; because of the strain hardening in the scale of SPD deformation, the non-uniformity of strain doesn’t affect the uniformity of microhardness or other mechanical properties [20].

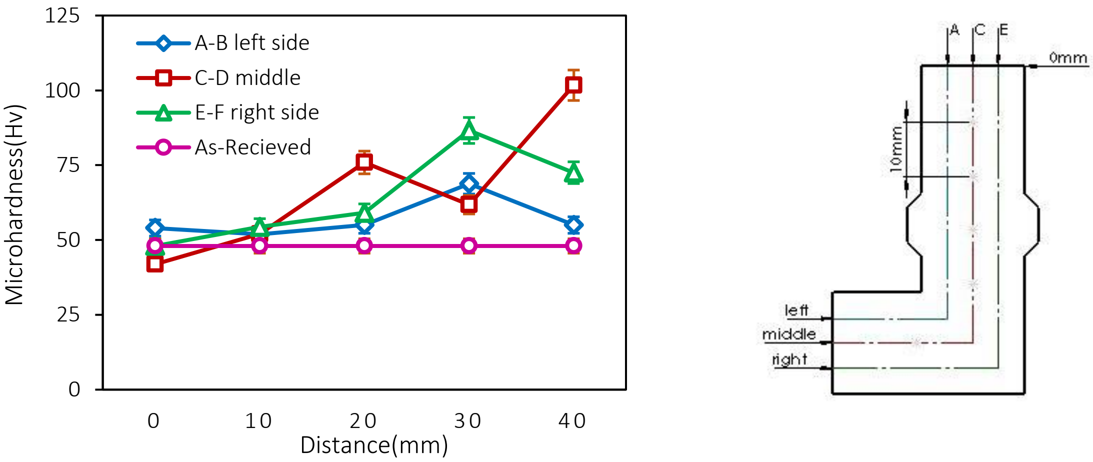

Fig. 5. Microhardness variations along three deformation paths and as received sample of AZ91.

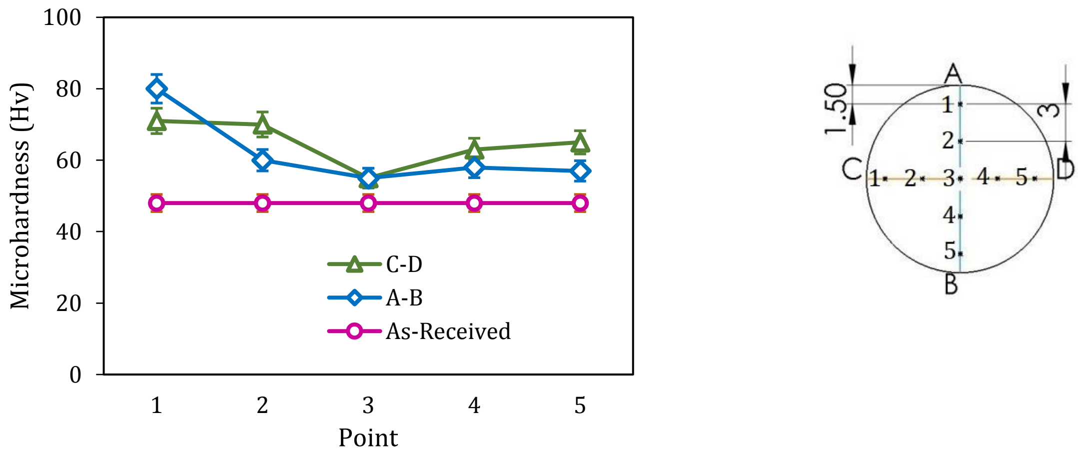

Fig. 6. Microhardness variations along the two perpendicular paths and as received sample of AZ91.

Fig. 7 shows the Engineering stress-strain diagram obtained from the tensile test. Due to limited slip systems at room temperature and the special distribution of β phase along the grain boundaries, the as-received sample has low ductility and strength. As seen in each plot of Fig. 7, the stress is enhanced by increasing the applied strain until reaching a peak value (UTS); This is attributed to the work hardening caused by the dislocations accumulation. After one pass of the EECAP process, the strength increased from 162 MPa to 211 MPa. An increase in dislocation density and grain refinement leads to higher strength. The increment of strength as a result of the reduction of grain size is also confirmed by Hall–Petch equation for strength [17]. In other words, during the EECAP process, the grain refinement, due to the DRX, and the distribution and dissolution of β phase cause the improvement of mechanical properties. Thus, during the EECAP process, dislocations strengthening, grain boundaries strengthening, and second phase particle strengthening are responsible for improving mechanical properties. Ductility is slightly decreased from 4.5% to 4.2%, which is predictable because, from Fig. 3f and Fig. 4, it is evident that the EECAP process, in addition to the significant grain refining, causes more homogeneous precipitation and distribution of β phase through the microstructure compared to the as-received state. Another reason for maintaining the ductility is that in the EECAP process there is significantly high hydrostatic compressive stress. Fig. 8 illustrates the mean stress (hydrostatic compressive stress) in the ECAP, and the EECAP processed samples. In SPD processes, besides the effective strain, the shear strain and hydrostatic compressive stress play important roles in the production of ultrafine-grained metals having simultaneously high strength and sufficient ductility. High levels of hydrostatic pressure prevent the initiation and movement of cracks and postpone the failure leading to high ductility. In addition, higher hydrostatic compressive stress causes a significant enhancement of deformability of the hard to form and brittle metals such as hcp metals like magnesium alloys [17]. Hydrostatic pressure might result in the activation of different slip systems in a magnesium alloy [18]. As it can be seen in Fig. 8, EECAP shows significantly higher hydrostatic pressure. So, it is claimed that EECAP is more effective than ECAP in the grain refinement and the improvement of mechanical properties of metals. Also, according to Bin Chen et al. [19], after one pass of ECAP, initial grains remain in the microstructure, and the saturation of grain size occurs in ECAP after 4-8 passes. Thus, further passes of EECAP are recommended to obtain better ductility [20]. Enhancement of strength while maintaining ductility is one of the advantages of this new hybrid method. This phenomenon is called the ‘‘paradox of strength and ductility in SPD-processed metals’’ [21]. Through the SPD process, high angle boundaries and strain increase simultaneously, which interrupts dislocation motion (strengthening). They also boost sliding grain boundaries and grain rotation as a deformation mechanism (ductility improvement).

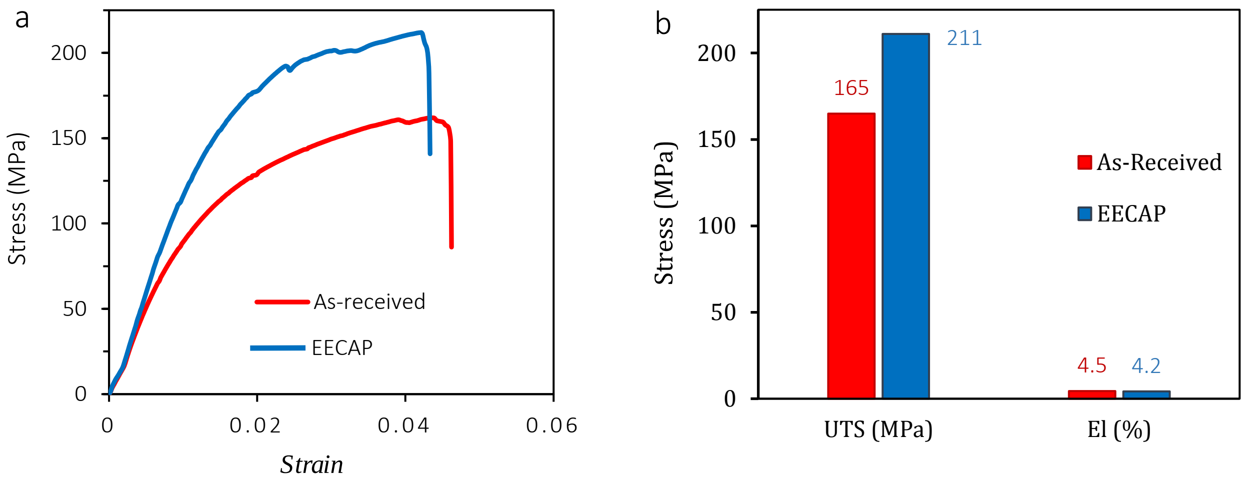

Fig. 7. Stress vs. strain curves for EECAP processed AZ91 (a) and (b) the ultimate tensile stress (UTS) and the elongation (El) obtained from (a).

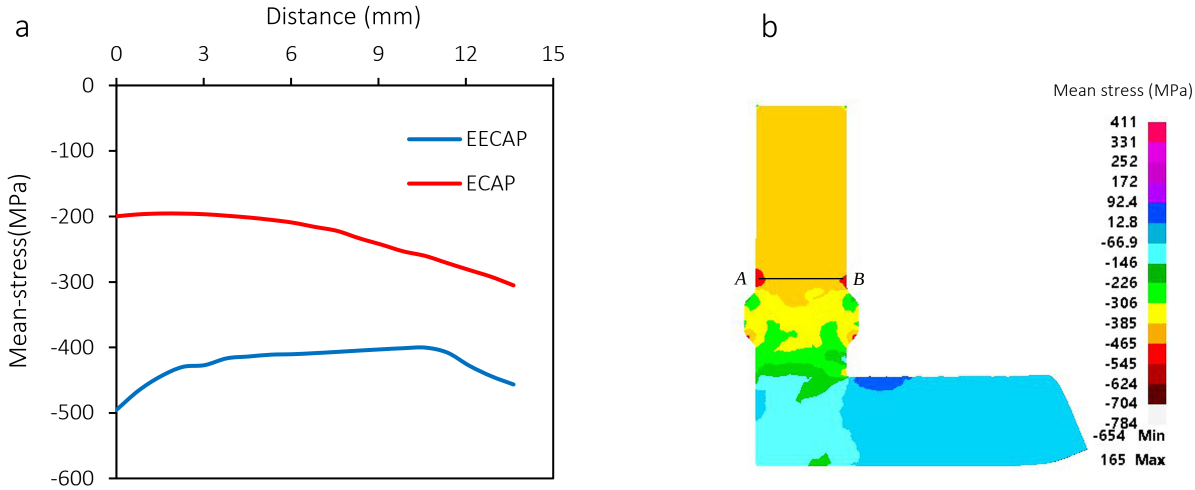

Fig. 8. Mean stress-distance curve along A-B path for ECAP and EECAP process (a) mean stress plot from FEM analysis (b)

5. Conclusion

1. In this research, the effects of equal channel angular pressing (ECAP) process with an additional expansion-extrusion stage, the expansion-extrusion equal channel angular pressing (EECAP) process on the microstructure, and the mechanical properties of Mg–9Al–1Zn magnesium alloy were studied. After only one pass of EECAP, results were as follows:
2. In comparison with ECAP, higher hydrostatic pressure was exerted.
3. Like the CEE process, backpressure is unnecessary, and a simple die setup would suffice.
4. The ultrafine-grained microstructure was achieved after inducing high strains in the process.
5. Due to the grain size reduction, 35% modification of the microhardness was observed.
6. The use of EECAP improved the ultimate tensile stress by about 30%; also, ductility remained almost unharmed due to the higher hydrostatic pressure as a dominant factor.

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