THE EFFECT OF SEVERE PLASTIC DEFORMATION ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AS-CAST AZ31

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Abstract: *The evolution of microstructure and mechanical properties of a magnesium cast alloy (AZ31) processed by equal channel angular pressing (ECAP) at two different temperatures were investigated. The as-cast alloy with an average grain size of 360 μm was significantly refined to about 5 μm after four ECAP passes at 543 K. Grain refinement was achieved through dynamic recrystallization (DRX) during the ECAP process in which the formation of necklace-type structure and bulging of original grain boundaries would be the main mechanisms. ECAP processing at lower temperature resulted in finer recrystallized grains and also a more homogenous microstructure. The mechanical behavior was investigated at room temperature by tensile tests. The obtained results showed that the ECAP processing can basically improve both strength and ductility of the cast alloy. However, the lower working temperature led to higher yield and ultimate strength of the alloy.*

Keywords: *Equal Channel Angular Pressing (ECAP), AZ31, Magnesium Alloy, Dynamic Recrystallization.*

1. INTRODUCTION

Magnesium and magnesium alloys have attracted much attention in automotive and aerospace industries due to their low density (1.74 g/m^3) and high specific stiffness [1]. However, the use of Mg and its alloys is limited because of their poor workability at room temperature. This disadvantage is attributed to the hexagonal close packed crystal structure of magnesium and its alloys and consequently insufficient number of independent slip systems at room temperature. In fact, plastic deformation in magnesium alloys occurs almost entirely by slip planes because the critical resolved shear stress for the basal planes at room temperature is much smaller than that for non-basal planes. But the basal planes provide only two independent slip systems and it is not enough to satisfy the von Mises criterion [2].

As the grain size plays an important role on mechanical properties improvement, using the methods of the magnesium alloys grain refinement to provide a fine equiaxed structure can be useful. Severe plastic deformation (SPD) techniques are widely used to produce such fine grain structures and to improve the mechanical properties of the material. ECAP as the most frequently used SPD technique is able to impose a high shear strain to the material without any change in the cross sectional area [3]. There are four processing routes in the ECAP process that introduce different slip systems and can influence the final microstructure; in route A the sample is pressed without rotation, in route B_C the sample is rotated by 90° after each pass, in route B_A the sample is rotated by 90° in alternate directions between passes and in route C the sample is rotated 180° between passes [3]. However, it was reported that route B_C can produce an ultrafine structure with equiaxed grains more rapidly in comparison with other routes [4].

Various studies showed that the ECAP processing can produce ultrafined grain structures and has a positive effect on the mechanical properties of materials such as aluminium, steel, and copper alloys [3]. It has been observed that grain refinement in FCC materials by ECAP occurs through formation of an elongated array of sub-grains or cells leading

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to a structure of equiaxed grains [5]. However, the grain refinement in HCP materials such as magnesium alloys occurs through the dynamic recrystallization and activation of both basal and non-basal slip systems [6].

As the SPD of Mg alloys at low temperatures usually leads to the formation of deep cracks and segmentation in the alloys, it is reasonable that the process is carried out at elevated temperatures [7]. Deformation at high temperature leads to the activation of non-basal slip systems resulting in a better workability of the magnesium alloys [2]. Numerous researches were carried out to improve the workability and microstructural characteristics of the magnesium alloys by varying the die geometry and experimental parameters. Despite the fact that the ECAP of an extruded AZ31 alloy at 473 K failed in the die angle of 90˚, it was successfully performed up to 4 passes in a die angle of 110˚ [8]. Figueiredo et al. also could eliminate the cracking of the ZK60 alloy by an increase in the die angle [9]. It was demonstrated that increasing the die angle and therewith reducing the shear localization and spreading the deformation zone in the shear region results in the deformation without any crack [9,10].

It was proposed that by reducing the pressing speed one can ECAP the alloy at lower temperature without cracking. Kang et al. ECAPed the AZ31 samples with no damage at 473 K while they reduced the pressing speed and subsequently flow localization in the alloy [11].

Another approach to overcome the difficulties in ECAP of magnesium alloys was the use of the back-pressure in the ECAP die [12–14]. A greater grain refinement and more homogenous microstructure was obtained by ECAP of an extruded AZ31 alloy at lower temperature as reported by Xu et al. [12]. They conducted the ECAP of AZ31 alloy at a temperature as low as 100 °C after applying the back-pressure so that a uniform structure and a sub-micrometer grain size was achieved after eight passes [12]. Applying a back-pressure three times of its yield stress facilitated the pressing of a twin-roll cast AZ31 alloy at room temperature [15].

Since the initial coarse grains make the ECAP processing of magnesium and its alloys much difficult [16,17], a pre-processing like extrusion or rolling is required to achieve the exceptional grain refinement in the ECAPed product [18–21]. The use of an intermediate step of rolling of pure magnesium facilitated its grain refinement through the ECAP and led to a more uniform structure [17]. Matsubara et al. [18] found that making the extrusion prior to the ECAP is effective in producing the ultrafine grains and superplastic ductility of Mg-9% Al.

Since the ECAP of magnesium alloys in cast or annealed condition is very difficult because of their coarse grains structure, most of the previous studies focused on the ECAP of these alloys in wrought condition or using a preliminary step prior to process. To the best of authors' knowledge, few research studies were done on ECAP of magnesium cast alloys without any intermediate step. Therefore, the present work was undertaken to evaluate the workability and grain refinement of as-cast AZ31 during the ECAP process. The effect of deformation temperature on the mechanical properties as well as microstructure of the ECAPed alloy is introduced in this work.

2. EXPERIMENTAL PROCEDURE

A cast AZ31 magnesium alloy with the chemical composition of (Mg-3.1 wt.% Al-0.8wt. % Zn- 0.2 wt.% Mn) was used for all experiments. The cylindrical ECAP samples were machined with a size of 65 x 9.9 mm (length x diameter). All samples were then homogenized at 693 K for 2h, followed by water quenching. An ECAP die was designed and manufactured from AISI D6 tool steel as shown in Fig. 1. In order to have a continuously homogenous temperature, four electrical heating elements were placed into the die. The temperature of the channel was measured using a K-type thermocouple, which placed near the channel. The angle between two intersecting channels (φ) and the outer arc of curvature (ψ) were 90° and 20°, respectively. Both channels have a circle cross section with a diameter of 10 mm. In order to have less friction in the exit channel and to reduce the pressing force, the exit channel was made shorter than the length of the sample. This design of the die

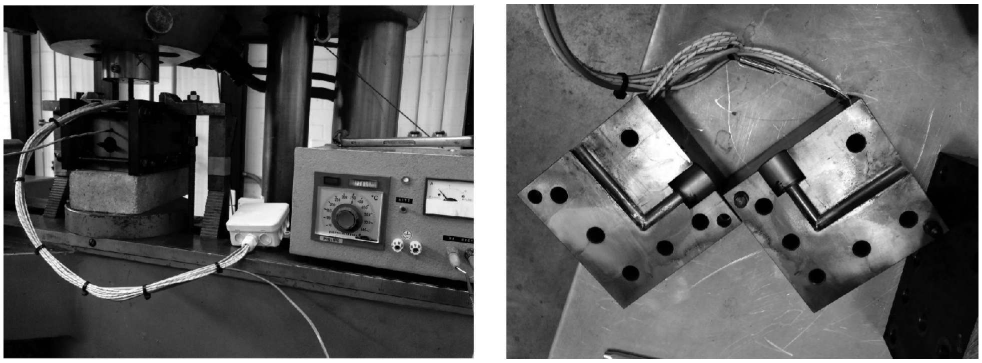

Fig. 1. Experimental setup (left) and ECAP die (right).

provides an equivalent imposed strain about 1 for each pass according to equation 1 [22]:

$$\varepsilon_{eq} = \frac{1}{\sqrt{3}}\left[2\cot\left(\left(\frac{1}{2}\varphi\right)+\left(\frac{1}{2}\psi\right)\right)+\psi\, cos\left(\left(\frac{1}{2}\phi\right)+\left(\frac{1}{2}\psi\right)\right)\right] \quad (1)$$

The processing speed and temperature were controlled and kept constant during the experiments. ECAP tests were performed at two different temperatures, 543 K and 523 K, using route BC, in which samples were rotated 90° clockwise after each ECAP pass. MoS_2 was used as lubricant in the process. For all ECAP tests, the constant ram speed was selected 3 mm/min.

After ECAP, the samples were cut in cross sectional plane perpendicular to the pressing direction, polished in three steps with 6, 3 and 1 μm diamond suspension and then etched by picric-acetic solution. All optical micrographs were prepared by polarized light microscope. Tensile tests were carried out at room temperature on the initial and processed samples by Zwick/Roell universal testing machine. Standard tensile specimens according to ASTM standard E8-03, with a gauge length of 20 mm and a diameter of 4 mm were machined in pressing direction. Average grain size measurements were carried out using the intercept method.

3. RESULTS AND DISCUSSION

3.1. Microstructure Evolution

Optical micrographs of the as-cast and the homogenized AZ31 alloy are shown in Fig. 2.

As can be seen, after homogenization of the samples, large equiaxed grains can be observed in the microstructure. The average grain size of the

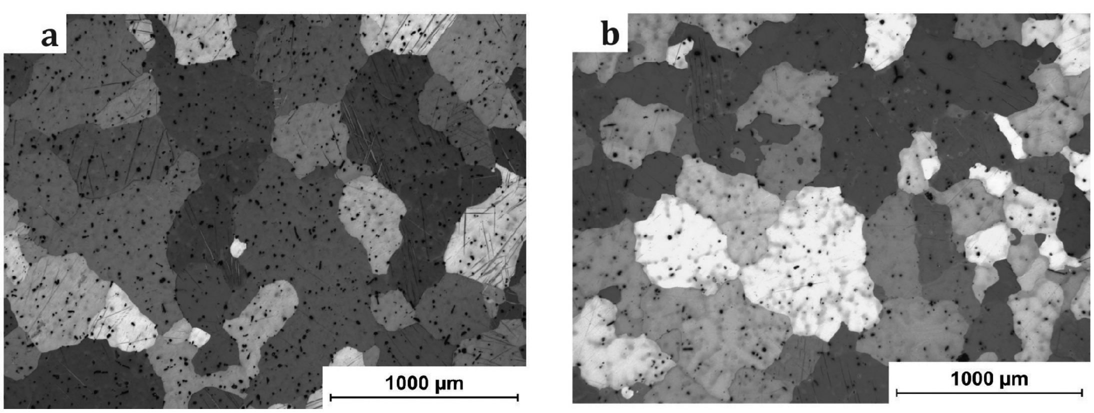

Fig. 2. Optical micrographs of (a) as-cast and (b) homogenized AZ31 alloy.

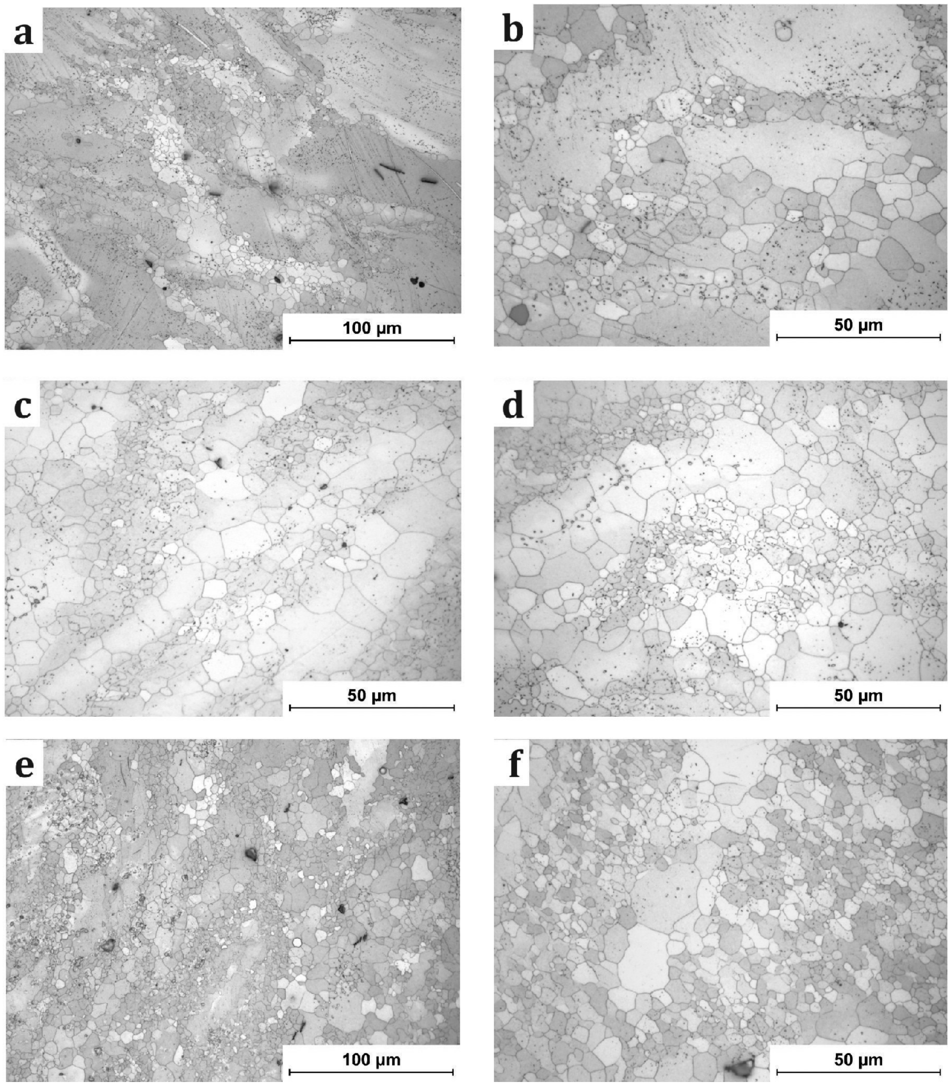

Fig. 3. Optical micrographs of the ECAPed samples at 543 K after (a) and (b) one pass, (c) two passes, (d) three passes, (e) and (f) four passes.

samples was 360 µm (before) and 390 µm (after homogenization), respectively.

The cross sectional microstructure after one ECAP pass at 543 K is shown in Fig. 3a and b. The microstructure consists of a bimodal structure of fine and initial coarse grains. In other words, it could be observed that some grains were refined during the first pass, however, a necklace-type structure of coarse grains is surrounded by the fine grains. For this reason, the microstructure is still non-uniform. These new fine grains form along the original grain boundaries, where the stress concentration was higher. The reason of this phenomenon is that the grain boundaries act as preferred nucleation sites for the recrystallization of new grains. The formation of new recrystallized grains and also the necklace structure imply the occurrence of dynamic recrystallization (DRX). DRX is a predominant softening mechanism in alloys with low stacking fault energy (SFE) [23]. In magnesium and magnesium alloys dynamic recrystallization occurs instead of dynamic recovery due to the limited number of independent slip systems despite their high SFE (γ=125 mJ/m^2) [23]. Different mechanisms for DRX of magnesium alloys have been suggested. Galiyev et al. [24] reported that new fine grains nucleate along the initial grain boundaries, attributing to the stress concentration at the grain boundaries and activation of both basal and non-basal slip systems. Figueiredo and Langdon [16] developed a model for the grain refinement in Mg alloys. They state that there is a critical grain size (d_c) which plays the main role in structure homogeneity and dominant refinement mechanism. Based on their model, when the initial grain size is bigger than the critical grain size, a bimodal microstructure in a necklace manner will be obtained after the first ECAP pass and it becomes homogenous in the next passes. While in material with initial fine grains, the microstructure will be homogeneous after only one pass. Since the as-cast AZ31 had coarse grains, it was expected to achieve a bimodal structure after the first pass; the microstructure shown in Fig. 3 is in agreement with the proposed model.

Fig. 4 indicates the formation of bulges in old grain boundaries and leaving a dislocation-free region behind the migrating boundary is considered as a nucleation mechanism which occurs at the start of DRX. It is usually assumed to be related to strain-induced grain boundary migration mechanism [23].

A large number of twins, which were formed after the first ECAP pass, can be seen in Fig. 5. Such as other materials, also in Magnesium alloys the slip is the first mechanism, which causes the deformation. Nevertheless, due to the lack of sufficient slip systems in magnesium, this mechanism cannot continue. For this reason, twining plays an important role in the deformation of magnesium and its alloys. However, the magnitude of twining shear is small and deformation continues and occurs by slip [23]. It is worth mentioning that twins also can be the nucleation sites for DRX.

As can be seen in Fig. 3c to f, further ECAP passes improve homogeneity of the microstructure. Increasing the strain level with further passes, the grains are refined and the recrystallized structure is distributed more uniformly. After four ECAP passes, the microstructure is still not completely homogenous; it consists of fine equiaxed grains as well as some pre-existing grains with an average size of 5 μm.

Fig. 6 shows the microstructure of samples after the first ECAP pass at 523 K. As shown in Fig. 6a-d, recrystallization mechanism after the first pass was similar to that one at 543 K. The recrystallized microstructure developed by further ECAP passes. Decreasing the temperature resulted in increasing the volume fraction of new recrystallized grains and therefore a higher grain

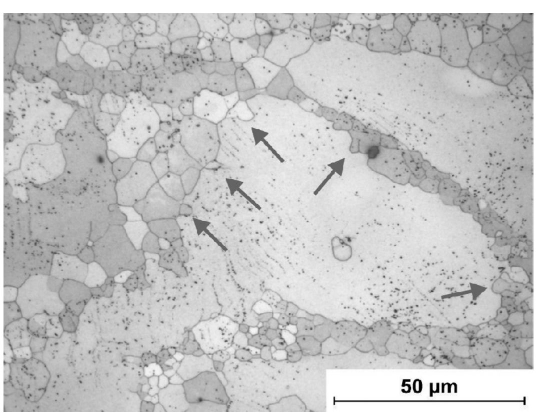

Fig. 4. Microstructure evolution of the ECAPed sample at 543 K representing the bulging of the initial grain boundaries and new grains formation.

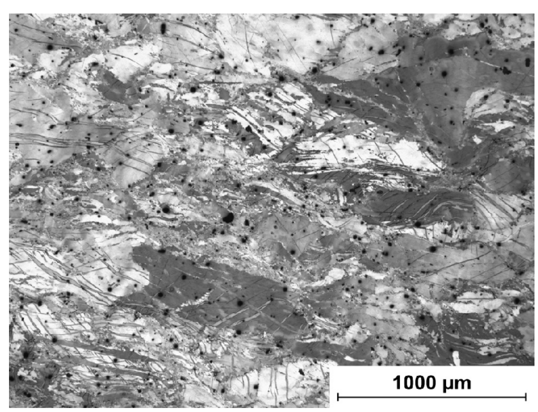

Fig. 5. Structure of specimen after one pass ECAP at 543 K representing a large number of twins.

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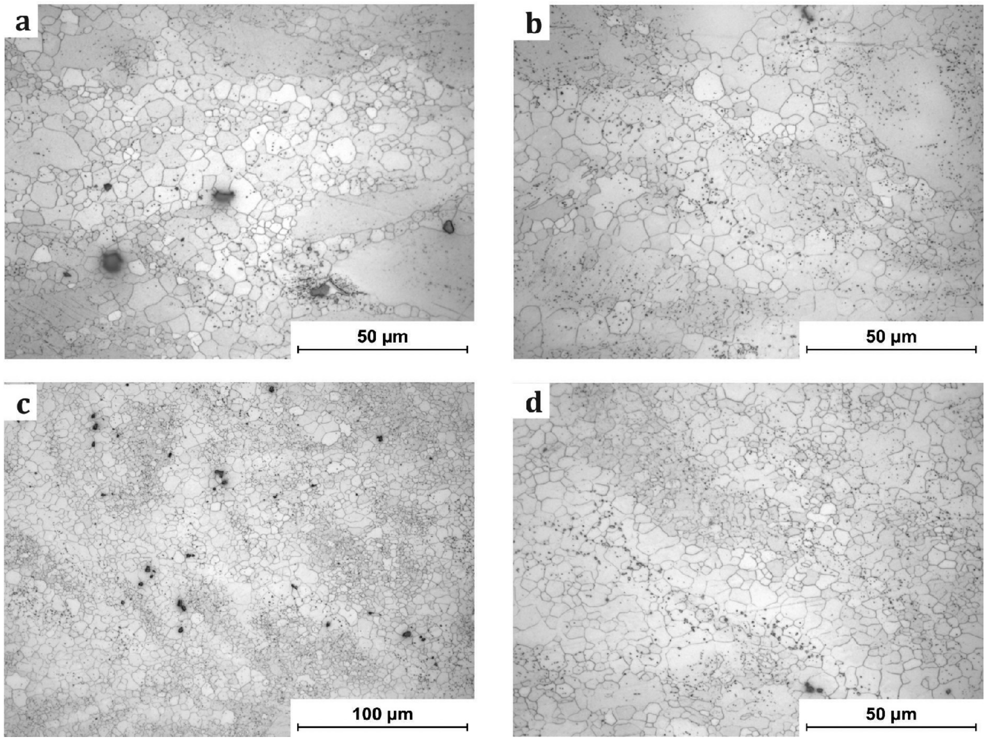

Fig. 6. Typical microstructure of the ECAPed samples at 523 K after (a) one pass, (b) two passes (c) and(d) four passes.

refinement. As can be observed in Fig. 6c and d, after four ECAP passes recrystallization has been developed over the entire structure. An almost homogenous microstructure of fine grains with an average grain size of 3.5 µm was formed as well. The finer grains and more homogenous structure can be a consequence of the low probability of grain growth at lower temperatures. Grain growth involves the migration of high angle grain boundaries and the higher temperature leads to higher mobility of grain boundaries and retard the DRX phenomenon [23]. In other words, ECAP at 543 K is not able to develop the uniform new recrystallized grains after 4 passes while at 523 K it shows a better response.

The reported results on the ECAP of cast magnesium alloys showed that this process was not effective in reducing the grain size. The ECAP of a magnesium alloy Mg- 0.6% Zr in the cast condition with a grain size of 70 µm resulted in a large final grain size of 30 µm and the formation of a non-uniform microstructure [20]. Moreover, a cast Mg- 0.9% Al alloy with initial grain size of 100 µm was refined only to 78 µm after two passes pressing at 673 K and to 17 µm after pressing at 473 K [7]. Unlike the reported results, it seems that in present investigation, ECAP processing had a significant effect on grain refinement of cast AZ31 alloy.

3.1.1. Mechanical Properties

The effect of grain refinement by the ECAP on the mechanical properties of AZ31 alloy was studied by tensile test at room temperature. Fig. 7a and b show the engineering stress-strain curves of the as-cast, homogenized, and ECAPed samples at 543 K and 523 K. It can be stated that the ECAPed samples exhibit a higher strength and ductility compared to the as-cast and only homogenized material. The relationship between mechanical properties and the number of ECAP passes is given in Fig. 8. ECAP at 543 K up to the second pass resulted in a significant enhancement of both yield and ultimate strength values but

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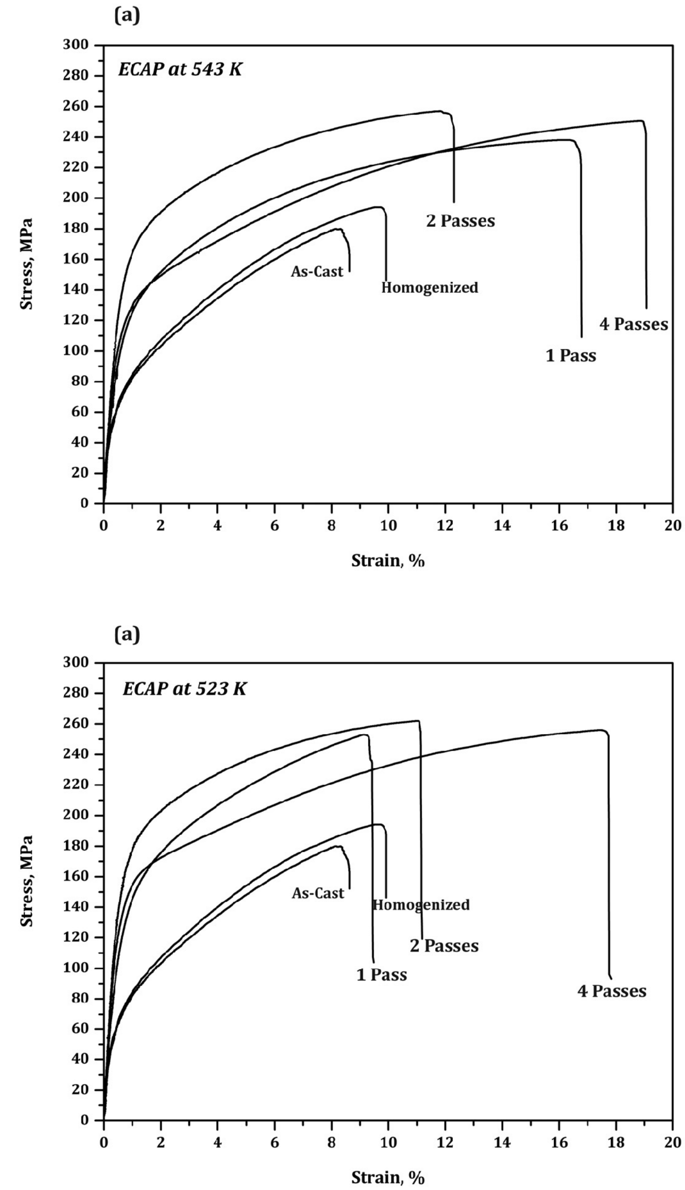


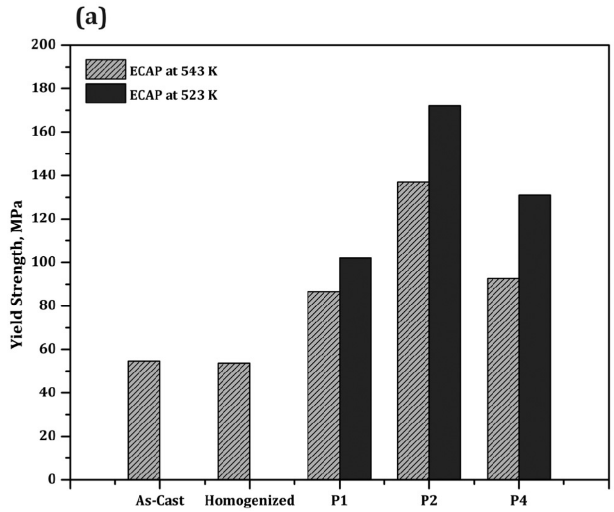
Fig. 7. Engineering stress-strain curves of AZ31 samples before and after ECAP processing at (a) 543 K and (b) 523 K.

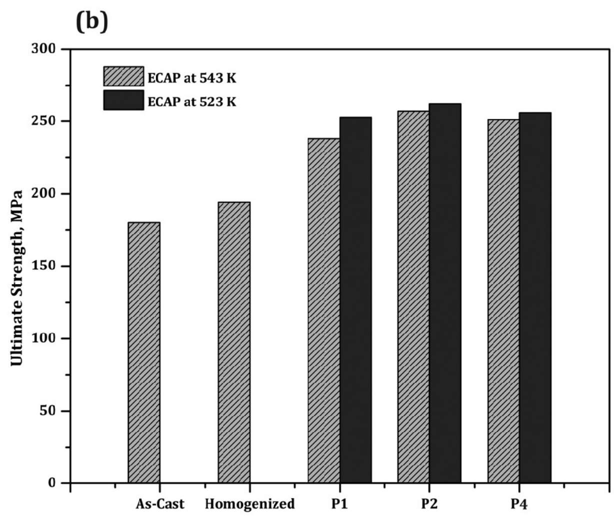
after four passes, the ultimate strength remained almost stable while the yield strength decreases (Fig. 8a and b). However, the yield strength was still higher than at the initial states of the alloy.

As can be seen in Fig. 7b, the same type of trend is observed after reducing the ECAP temperature to 523 K. The lower ECAP temperature leads to higher values of the yield and ultimate strength.

Concerning ductility, the elongation of ECAPed material at 543 K was increased significantly from 8.1% in as-cast condition to 18.8% after four ECAP passes (see Fig. 8b). However, a decrease after the second pass can be observed. The specimen processed at 523 K exhibited a continuous increase in elongation with successive passes while it slightly decreased in comparison with the specimens ECAPed at 543 K.

Both strength and ductility enhancement after





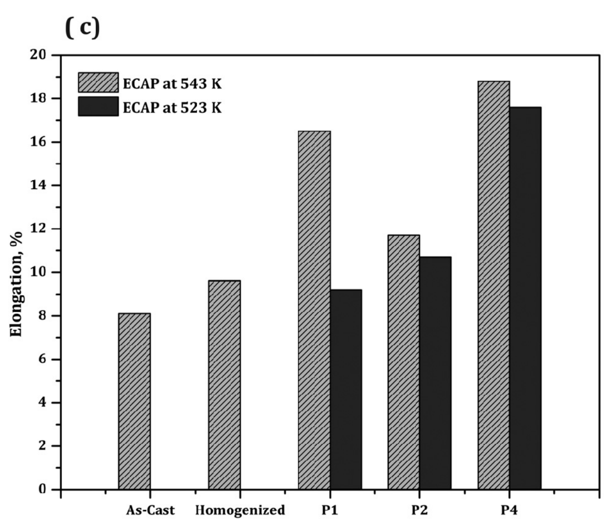


Fig. 8. Tensile properties of AZ31 samples before and after ECAP: (a) yield strength, (b) ultimate strength and (c) elongation (extracted from Fig. 7).

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ECAP processing are the consequence of grain refinement. According to the Hall-Petch relation, which states the relationship between yield strength and grain size, decreasing the grain size leads to higher strength. Therefore, dynamic recrystallization and grain refinement as a consequence of ECAP processing resulted in an enhancement of strength. Increasing the strength of material processed at lower temperature can also be related to the more homogenous microstructure and finer grain size achieved by ECAP at 523 K compared to 543 K.

A similar trend in elongation of the ECAPed magnesium alloys has been reported by other researchers. They showed that the elongation enhancement is also attributed to the texture modification occurring during the ECAP process. The ECAP process can modify the distribution of basal planes or non-basal planes to some degrees, so that it is favorable for dislocations to slip during the tensile test [13, 25, 26]. Therefore, grain refinement and texture modification through ECAP are the main reasons for the ductility enhancement. However, this softening through the texture modification can be also the reason of a drop in yield strength after the fourth pass.

4. CONCLUSIONS

In this study, an as-cast AZ31 alloy was ECAPed at two temperatures, namely 523 K and 543 K, up to four passes through the route B_C. The microstructure evolution and mechanical properties of the samples were studied. Based on the obtained results, the following conclusions can be drawn:

1. ECAP is an effective method for grain refinement of as-cast AZ31 alloys. A significant grain refinement was obtained after four passes through dynamic recrystallization and formation of new recrystallized grains. Nucleation of new fine grains occurs along the initial grain boundaries and a necklace-type structure is achieved during the ECAP process. Optical micrographs show that this recrystallization phenomenon starts via bulging the grain boundaries.
2. The presence of twins in the microstructure of the sample after one ECAP pass implies that the severe plastic deformation of AZ31 alloy begins with twinning then followed by slipping.
3. Reducing the temperature, a more homogenous and finer grains were obtained after four ECAP passes. It can be attributed to the lower probability of grain growth at lower temperatures.
4. The ECAP processing led to an improvement in both elongation and strength of the material. The grain refinement obtained by dynamic recrystallization during ECAP is the main reason for strength enhancement. Increasing the elongation is mainly caused by the formation of a homogenous fine structure and texture modification. Due to finer grains obtained at lower temperature, the yield and ultimate strength exhibited a slightly increase after reducing the temperature.

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