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Grain refinement in AZ31 alloy processed by equal channel angular pressing

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ABSTRACT

Purpose: of this paper was to investigate the effect of grain refinement in AZ31 magnesium alloy processed by ECAP and to study microstructure evolution and hardness response of AZ31 alloy associated with ECAP processing.

Design/methodology/approach: The microstructure of AZ31 magnesium alloy after two passes of equal channel angular pressing at 150°C, 180°C and 250°C was studied by means of metallographic and transmission electron microscopy. The hardness changes after ECAP processing were determined by Vickers hardness.

Findings: The grain refinement in AZ31 magnesium alloy was successfully carried out using ECAP processing at 150, 180 and 250°C. The grain size decreases nearly 10 times after 2 passes of ECAP at 150 and 180°C, but microstructure is characterized by bimodal grains structure. The rather homogenous grains were achieved after ECAP processing at 250°C. Processes of dynamic recrystallization during ECAP were observed. The hardness increase related to grain refinement proceeded by ECAP is in accordance with Hall-Petch relationship.

Research limitations/implications: The ECAP processing were carried out only after 2 passes, therefore in order to describe in detail the microstructural changes connected with grain refinement, the analysis of more passes of ECAP processing should be done.

Practical implications: The development of highly ductile magnesium alloys allows to apply these materials as structural materials. The grain refinement of materials leads to the significant improvement of the mechanical properties and plasticity. The present results extend the knowledge about grain refinement in AZ31 alloy proceeded by ECAP.

Originality/value: The microstructural studies of AZ31 alloy after grain refining by ECAP processing performed by transmission electron microscopy were presented.

Keywords: AZ31 magnesium alloys; Equal channel angular processing; Grain refinement; Microstructure

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MATERIALS

1. Introduction

Magnesium alloys, as a one of the lightest structural materials known as an energy-saving “green materials”, are increasingly used in many industries, particularly in automotive, electronics and aerospace industry, because of their features like low density (1.74 g/cm^3), high specific strength, high damping capacity, excellent screening ability against electromagnetic radiation and recyclability [1,2]. Recently, most of components from Mg alloys are manufactured by many casting methods, mainly pressure die-casting. The mechanical properties of components prepared by casting processes often do not meet industrial requirements [3]. Therefore, it is essential to improve the mechanical characteristics of magnesium alloys by plastic deformation to extend their application. However, magnesium alloys exhibit poor formability at ambient temperature due to their hexagonal close packed structure with 2 slip systems: $(0001)\langle 11\bar{2}0\rangle$ and $(10\bar{1}0)\langle 11\bar{2}0\rangle$.

Grain refinement is an effective approach of improving the strength and ductility. In recent years, one of the successful method in refining the grain size of metals or alloys has become equal channel angular pressing (ECAP) [4, 5]. It was proved that ECAP process enables obtaining ultra-fine structure of aluminium, steels, copper and magnesium alloys [6-10]. The activation of non-basal slip systems in magnesium alloys with their hcp structure requires elevated pressing temperature. For that reason, to avoid the grain growth and dynamic recrystallization during equal channel angular pressing, the process temperature should be selected carefully.

The effect of grain refinement in magnesium alloys processed by ECAP was reported, however significantly different final grain sizes were obtained for the same Mg alloys at similar ECAP conditions [11, 12]. The aim of the present paper was to determine the effect of two passes of ECAP processed at different temperature on grain refinement of AZ31 magnesium alloy.

2. Experimental procedure

As cast ingots of magnesium alloy AZ31 (Mg-3%Al-1%Zn-0.3Mn, wt.%) was supplied by Jiangxi Royal, Ltd. Pieces with initial thickness of 20 mm were hot-rolled at 350°C to a thickness 10 mm and cut into billets of square section of 10 mm and length of 50 mm. The grain size after hot-rolling was measured by the method of secants on optical microscopy images from longitudinal section. The average of grain size was 9.7 μm.

As prepared, samples were pressed by ECAP at temperature 150, 180 and 250°C at the pressing speed 0.1 mm/s. The ECAP die is illustrated schematically in Fig. 1. The ECAP die was constructed of H13 tool steel with a square-shaped channel of 10.2 x 10.2 mm and designed as two parts. Die was surrounded by electric resistance heater ensuring accuracy to ±5°C. An internal angle ϕ between two parts of channel equals 90° and the corner angle (ψ) of 0° with a small radius of approximately 2 mm.

The hydraulic press was used for pressing with a maximum applicable load of 400 kN. For each pass, ECAP die was heated to proper temperature and a sample was held in die channel for 10 minutes to reach temperature stabilization. Before putting it in ECAP die channel, a sample was coated with graphite assembly paste (Molydal Multigraph) to assure lubrication during ECAP process. For each samples 2 passes were carried out and B_c route (the rotation of sample by 90° in the same direction between passes) was used. For the microstructural examinations the samples were cut longitudinally to the extrusion direction and from the middle of billets.

Light (LM) and transmission electron microscopy (TEM) studies were carried out after ECAP processing at 150, 180 and 250°C. Specimens for LM investigation were prepared by mechanical grinding and polishing and etched in solution containing 1ml nitric acid, 20 ml acetic acid, 24 ml distilled water and 75 ml glycol. For TEM study, thin foils were electropolished in solution of LiCl + perchlorate + methanol + buthyloxyethanol at -45°. The prepared TEM specimens were examined using Philips CM20 (200 kV).

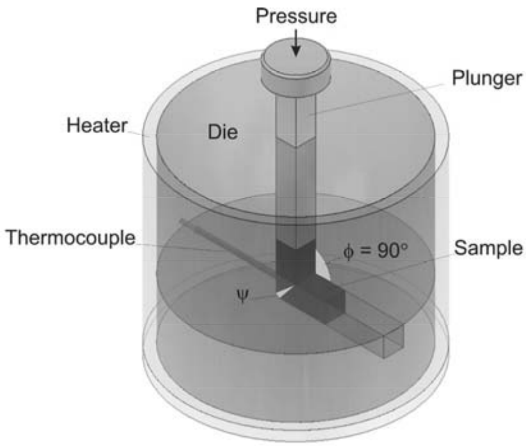


Fig. 1. Schematic illustration of the facility for ECAP

The hardness measurements of investigated samples were determined by Vickers hardness at the load of 49.03 N (HV5).

3. Results and discussion

3.1. Microscopic observation

The surfaces of ECAP samples after all passes do not exhibit cracks or fractures, what suggest a uniform flow in the process. However, fracture characterized by the formation of a series of segments along the direction of the extrusion at 150°C at various strain rates was reported [13].

The metallographic examination of AZ31 magnesium alloy samples after 2 passes of ECAP process at 150, 180 and 250°C has shown that microstructure is characterized by refined grains (Fig. 2). In each sample, very fine grains were observed and the average grain sizes were measured as 1.1, 1.4 and 4.2 μm, for pressing temperatures 150, 180 and 250°C, respectively (Table 1).

Table 1.
Average grain size of AZ31 alloy after 2 passes of ECAP

Condition	Grain size in μm
Initial (hot-rolled at 350°C)	9.7
ECAP, 250°C	4.2
ECAP, 180°C	1.4
ECAP, 150°C	1.1

a)

b)

c)

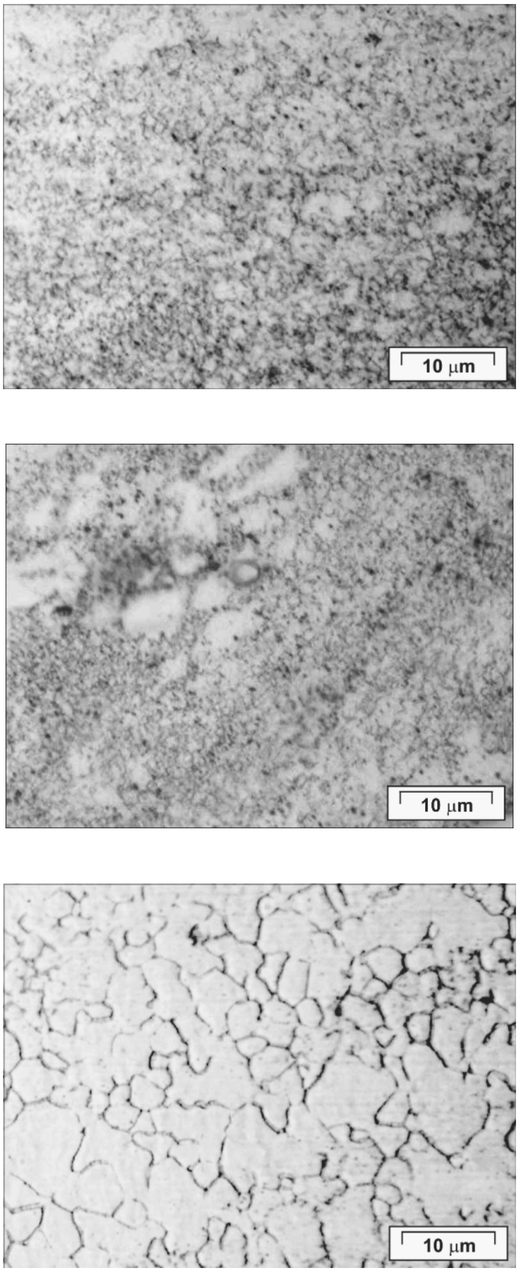


Fig. 2. Grain microstructure of the AZ31 alloy processed by ECAP after 2 passes at: (a) 150°C, (b) 180°C, (c) 250°C

The grains of as extruded samples at 150 and 180°C characterize so-called “bimodal” distribution (Fig. 2), which was observed in earlier studies [14]. For these samples, the microstructure consists of very fine grains (0.2-1 μm) as well as coarse grains (< 5 μm). Bimodal distribution of grains is probably due to limited slip systems in magnesium, therefore only favourable oriented grains were deformed and refined as first during ECAP process and areas of less deformed and larger grains were left in microstructure. It was found that fully homogenized grains structure can be achieved after 4 to 8 passes in ECAP experiments at 200°C [14,15].

On the other hand, it was reported that a homogenous ultrafine structure grain structure may be achieved after a single pass of ECAP when using an alloy with an initial fine microstructure [14].

The plastic shear deformation by the ECAP causes accumulation of large plastic strain and increase of structural defects. It is believed that continuous dynamic recrystallization occurs during ECAP, however nucleation and growth of new grains do not take place. After 2 passes of ECAP process at 250°C, there is an evidence of the rearrangement of dislocations within the grains and formation of subgrain boundaries (Fig. 3). The occurrence of dislocation in some recrystallized grains is observed.

a)

b)

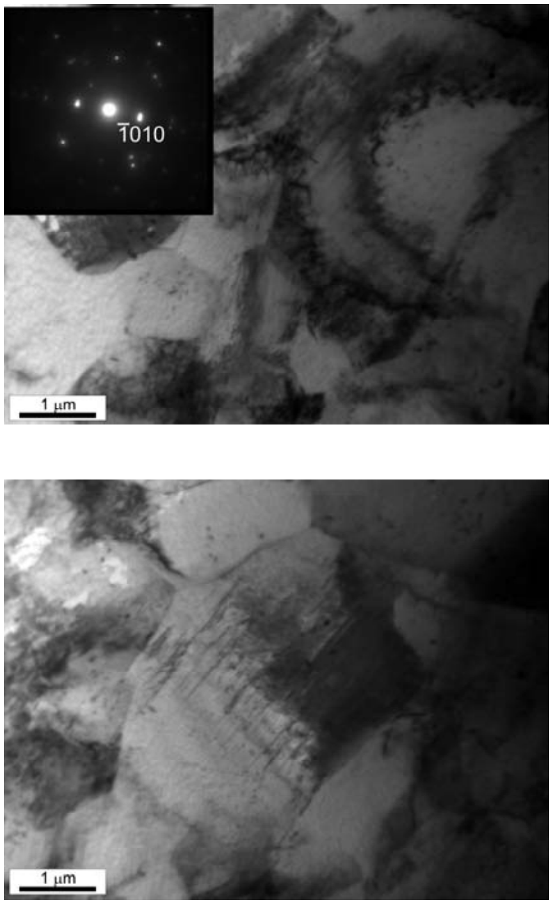
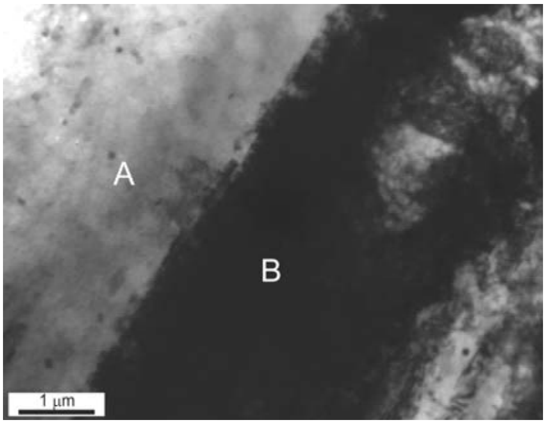


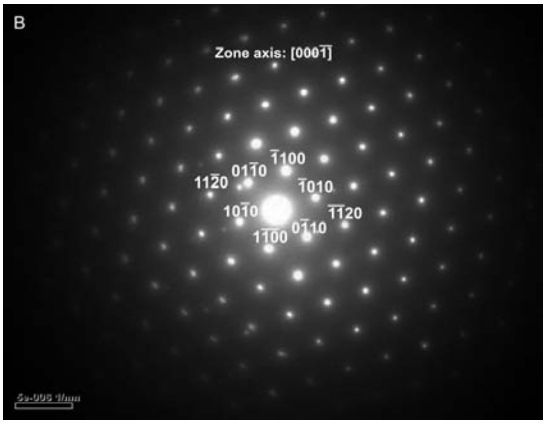
Fig. 3. TEM micrographs of microstructure of the AZ31 alloy after 2 passes of ECAP at 250°C: (a) rearrangement of dislocations and subgrain boundaries formation and electron diffraction pattern, (b) well defined grain boundaries

It was found that the microstructure evolution during ECAP process can be related to the gradual transformation of subgrain boundaries from low to high angle with pass number by absorbing dislocation [16]. In Fig. 4, the low angle boundaries between two grains (A and B) proved by selected area diffraction patterns has shown.

a)



b)



c)

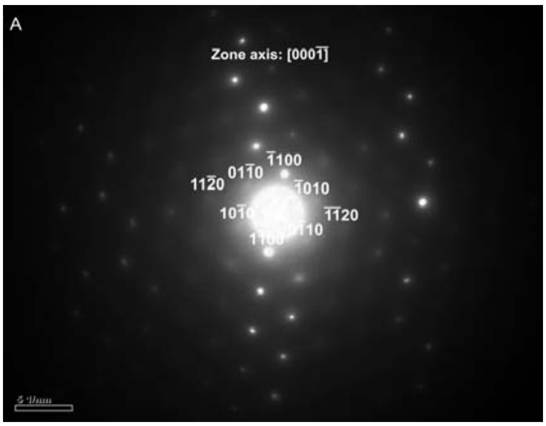


Fig. 4. TEM micrographs of microstructure of the AZ31 alloy after 2 passes of ECAP at 180°C: (a) grain boundary between grains A and B, (b) and (c) electron diffraction patterns of grains

The microstructure with sufficient amount of dislocation accommodated by subgrain boundaries during ECAP can be transformed into fine grained microstructure with high angle boundaries. Such regions with high dislocation density are preferable for dynamic recrystallization processes.

Decreasing the ECAP process temperature from 250 to 180 and 150°C leads to more effective grain refinement. It might be attributed to the repression of dynamic recrystallization and grain growth. Figs. 5 and 6 shows TEM micrographs of microstructure of the AZ31 alloy after 2 passes of ECAP at 150°C. The microstructure consists of very fine grains (less than 1 µm) and small amounts of larger grains (< 5µm). The high dislocation density and dislocation complexes inside most of the grains was observed (Fig. 6), however some regions contained very low density of dislocation. Dislocation density in this sample is higher than in sample deformed at 250°C.

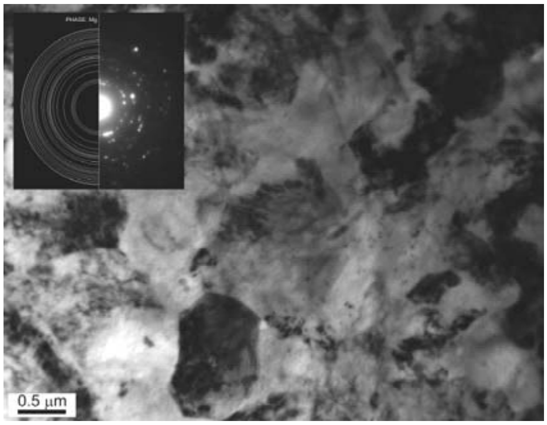


Fig. 5. TEM micrographs of microstructure of the AZ31 alloy after 2 passes of ECAP at 150°C: fine grains structure and partial ring like electron diffraction pattern

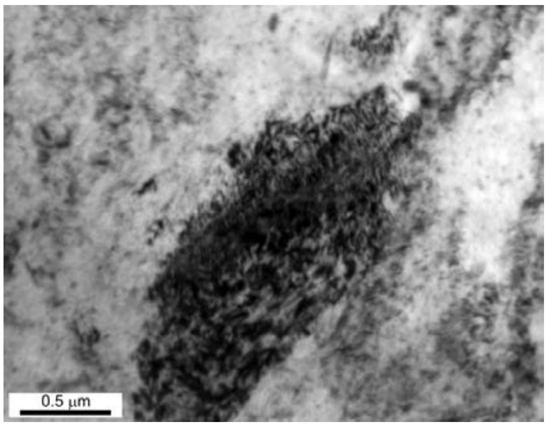


Fig. 6. TEM micrographs of microstructure of the AZ31 alloy after 2 passes of ECAP at 150°C: high dislocation density within grain

Comparing the microstructures deformed by two ECAP passes at 250°C and at lower temperature (150 and 180°C), it can be concluded that grain refinement proceed by ECAP is more effective at lower temperature as a result of structural defects accumulation and suppression of thermally activated processes. However, the microstructure achieved at 150 or 180°C is characterized by bimodal grains, while deformed microstructure by ECAP at 250°C consists of homogenous grains. In regions with higher dislocation density new grains can nucleate and grow. Therefore, the bimodal structure as a combination of new recrystallized grains and primary coarse grains are observed. During the ECAP pass, dislocation might accumulate at grain boundaries and stimulate dynamic recrystallization there. This process can be repeated in next passes of ECAP bringing about grain refinement. At this point, it is worth mentioning that the size of initial grains can play an important role in grain refinement proceeded by ECAP.

3.2. Hardness results determined by Vickers indentation

The hardness changes after ECAP processing were determined by Vickers hardness at the load of 49.03 N. The HV5 value for hot-rolled sample at 350°C as initial for ECAP deformation was 62. The HV5 values of AZ31 alloy after two passes of ECAP at 150, 180 and 250°C are plotted versus $1/\sqrt{d}$, where d is the average grain size. As shown in Fig. 7, the dependence of hardness on grain size demonstrates Hall-Petch relationship. It confirms the strengthening effect induced by the grain refinement proceeded by ECAP. The Hall-Petch relationship for Mg alloys deformed by ECAP and as well as Al alloys was reported [16, 17, and 18].

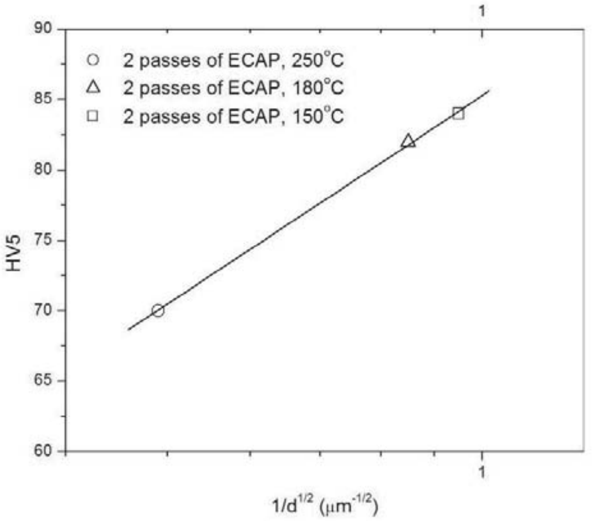


Fig. 7. The Vickers hardness changes during ECAP at 150, 180 and 250°C for AZ31 alloy versus $1/\sqrt{d}$ (Hall-Petch relationship), where d is an average grain size

4. Conclusions

The grain refinement in AZ31 magnesium alloy processed by equal channel angular pressing (ECAP) were examined using light and transmission electron microscopy and hardness measurements.

The following conclusions can be drawn from this investigation:

- The grain refinement in AZ31 magnesium alloy was successfully carried out using the ECAP processing at 150, 180 and 250°C.
- The grain size was reduced nearly 10 times after 2 passes of ECAP at 150 and 180°C (from 9.7 to 1.1 μm). However, the bimodal grain structures were achieved.
- The homogenous grains with an average size of 4.2 μm were attained after 2 passes of ECAP processing at 250°C.
- The accumulation of high dislocation density especially at 150 and 180°C due to intense shear deformation were observed.
- Dynamic recrystallization occurs during ECAP processing particularly at pressing temperature of 250°C. The sub-grain boundaries formation and the low-angle boundaries between grains were observed.
- The hardness increase (form 62 to 84 of HV5) is related to grain refinement proceeded by ECAP. The dependence of hardness on grain size demonstrates Hall-Petch relationship.

Acknowledgements

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