MICROSTRUCTURE EVOLUTION OF AZ61 MAGNESIUM ALLOY PROCESSED BY ECAP METHOD

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Abstract

The article deals with the grain refinement and microstructure evolution of AZ61 magnesium alloy subjected to sever plastic deformation (SPD) by ECAP (Equal Channel Angular Pressing) process. The commercial hot extruded AZ61 alloy subjected to severe plastic deformation possesses a two-phase microstructure consisting of solid solution matrix and massive γ - phase $Mg_{17}Al_{12}$ distributed mostly at grain boundaries. Based on selected area diffraction and electron back scattered diffraction applied to a sample after the third pass, it can be concluded that plastic deformation induced by ECAP occurs mainly by slip mode forming a high density network of dislocation inside the grains. The grains size was significantly refined to 1.4 μm after the third pass of ECAP. The refinement of grain size is probably due to polygonization process associated with formation of high angle grain boundaries due to dislocations rearrangement. $Mg_{17}Al_{12}$ precipitates of size scattered from 100 to 200 nm and also the primary precipitates of Al_6Mn phase were observed in this alloy.

Keywords: Magnesium alloy, ECAP, optical microscopy, TEM

1. INTRODUCTION

In recent years, a lot of efforts have been developed on magnesium alloys because of their outstanding specific strength. Consequently, they are used today in several fields such as electronics, biomedical, aerospace, defense, and particularly in the automobile sector [1]. The attractive benefit of a drop in vehicle weight can be observed in terms of enhanced fuel efficiency and less exhaust CO_2 gas emissions. However, their widespread applications are limited compared to commercial structural materials such as steel and aluminium alloys [2,3]. A hexagonal close-packed structure of Mg alloys offers a limited number of independent slip systems that prevent excessive plastic deformation at ambient temperature [4].

Among the currently developed technologies of plastic deformation of metallic materials, severe plastic deformation (SPD) methods are worth to mention [5-7]. This plastic deformation technologies consists of transforming the micrometric structure of coarse-grained materials into ultra-fine (UFG with grain size in range of 100–1000 nm) and/or nanometric materials (grain size < 100 nm) by re-organizing the dislocation structure created during plastic deformation [8-9]. One of most popular SPD method is ECAP, i.e., Equal Channel Angular Pressing. During ECAP, a workpiece with a square or circular cross-section is pressed through an angle channel. Both channels (vertical and horizontal channel) are intersected by a shear plane, where a shear deformation in introduced into the material. Schematic illustration of ECAP principle is presented in **Figure 1**.

Severe plastic deformation methods in contrast to conventional technologies of plastic deformation are not used to shape the initial material, but primarily to transform the coarse-grained microstructure into ultra-fine or nanometric microstructure [10-11]. The obtained grain size and nature of the produced nanostructures depend mainly on: SPD process conditions, phase composition, and initial grain size of the starting material. Compared to the conventional (coarse-grained) materials that were subjected to SPD processing they are characterized by greater mechanical and physical properties [10-14].

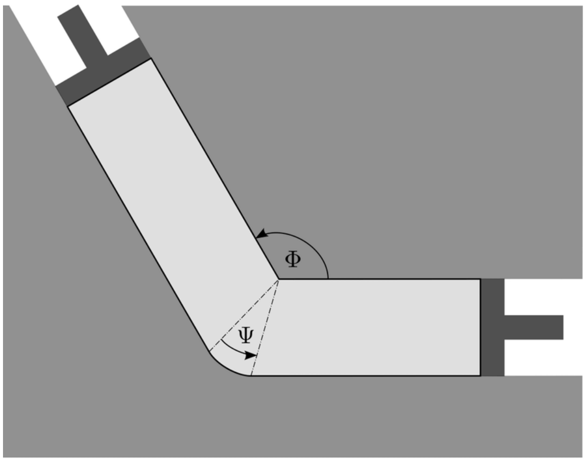


Figure 1 Schematic illustration of ECAP die geometry [14]

2. EXPERIMENTAL PROCEDURE

The experiments were concentrated on the influence of severe plastic deformation method ECAP on the grain microstructural evolution of AZ61 magnesium alloy.

2.1. Experimental material

Experimental magnesium alloy, containing by 6% Al and 1% of Zn (Chemical composition is given in **Table 1**). The samples were casted under argon protecting atmosphere and hot extruded at 430°C. Hot extruded rods were cut on square cross-sectional shape with dimensions 15x15 – 60 mm.

Table 1 Chemical composition of the AZ61 magnesium alloy

Element (wt. %)	Al	Zn	Mn	Si	Fe	Cu	Ni	Mg
AZ61	6.430	1.092	0.230	0.030	0.003	0.003	0.001	rest.

2.2. ECAP process parameters

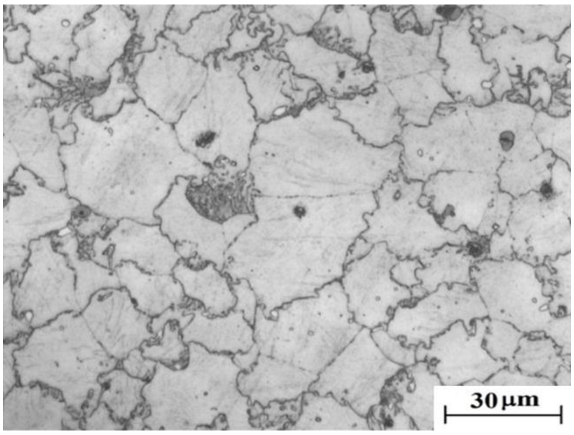
Due to the limited formability of investigated alloy at low temperature (below 200 °C), the extruded alloy AZ61 was deformed by the ECAP method at a temperature of 200 °C (the temperature is the same for the sample and the channel) with the constant deformation rate of 40 mm/min. Samples were rotated by 90° around the longitudinal axis (Bc processing route). The angle between two channels Φ = 90° and the angle of the outer curvature Ψ = 9.5°.

2.3. Microstructural investigation

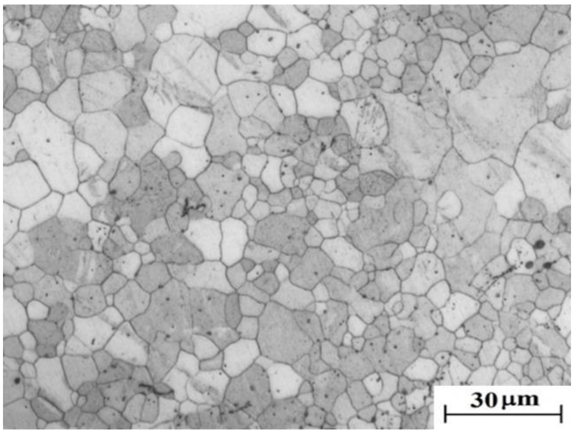
Microstructural analysis of investigated magnesium alloy was realized on the NEOPHOT 2, optical microscope. Detailed studies of plastically deformed grains, distribution and types of phases have been done by transmission electron microscopy (TEM) using Tecnai G2 F20 (200 kV) microscope fitted with high-angle annular dark field scanning transmission electron microscopy detector (HAADF-STEM). TEM specimens have been prepared by twinjet electro-polishing (Tenupol-5) using a solution of 10.6 g lithium chloride LiCl, 22.32 g magnesium perchlorate $Mg(ClO_4)_2$, 1000 ml methanol, and 200 ml 2-butoxy-ethanol at - 40 °C and 80 V.

3. INVESTIGATION RESULTS

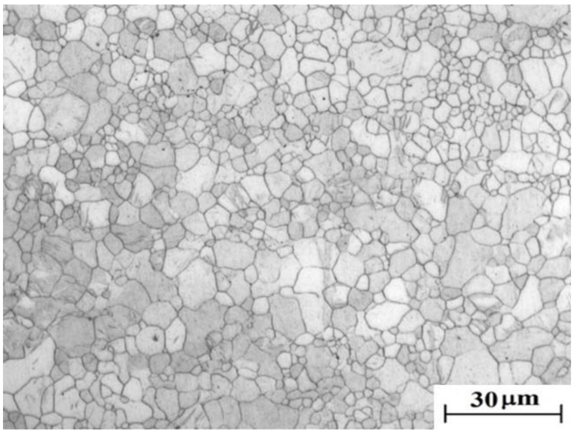
Figure 2 shows the optical microscopy (OM) images of hot extruded samples and of those processed by ECAP method. The grain size of the hot extruded state was typically non-uniform on the cross-section and the average grain size was ~ 24 μm (see **Figure 2**). The microstructures of samples after individual ECAP passes are shown in **Figure 2**. While many grains were already significantly refined already, just after the first pass, the microstructure was relatively heterogenous with grain size ~ 9 μm (see **Figure 2a**). The decomposition of the structure attributed to ECAP began in the grain boundary area, in order words, the dynamic recrystallization began, as can be seen in OM image of the sample after the first pass. After the first pass was grain distribution relatively heterogeneous (microstructure has a bimodal character). Further ECAP passes at the same temperature, a relatively homogeneous microstructure with an average grain size below 2 μm was obtained. This homogeneity after only three passes suggests that original grains were replaced by recrystallized grains (see **Figures 2b-d**).



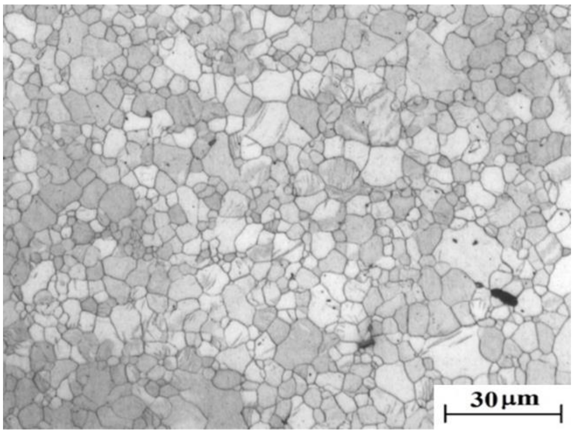
a) initial (hot extruded) state



b) after the first ECAP pass



c) after the second ECAP pass



d) after the third ECAP pass

Figure 2 The OM images of the microstructure of AZ61

TEM investigations of samples after 1 and 3 ECAP passes were performed in order to examine microstructural features, especially the change of grain size and distribution but also size and chemical composition of precipitates. **Figure 3** presents a set of bright field (BF) images taken at different magnifications and corresponding selected area diffraction patterns (SADP) of sample after 1 pass of ECAP. High density of dislocations, twins and formation of shear bands can be observed in BF images. The grains possess slightly elongated shape and mean size of about 5.4 μm. SADP was indexed to Mg with a [123] zone axis exhibiting a diffused character of reflects associated with a strongly deformed structure. Also an additional reflects can be recognized on SADP image, most probably originated from precipitates. BF and dark field (DF) microstructures and corresponding SADP of sample after 3 passes of ECAP are presented in **Figure 4**. In this case more refinement microstructure can be seen as is demonstrated on DF image performed from reflect of

(110) plane of Mg structure. The mean grain size is about 1.9 µm, what indicates that two additional passes of ECAP caused almost 2.5 times reduction of grain size. Higher magnification image shows individual grain of magnesium matrix with [001] zone axis having equiaxed shape indicating that the ECAP process causes loss of texture of grains before present in the samples after 1 pass of ECAP. Although, reduction of grain size was observed after 3 passes of ECAP, shape of the grains and lower dislocation density may indicate that the dynamic recrystallization occurred.

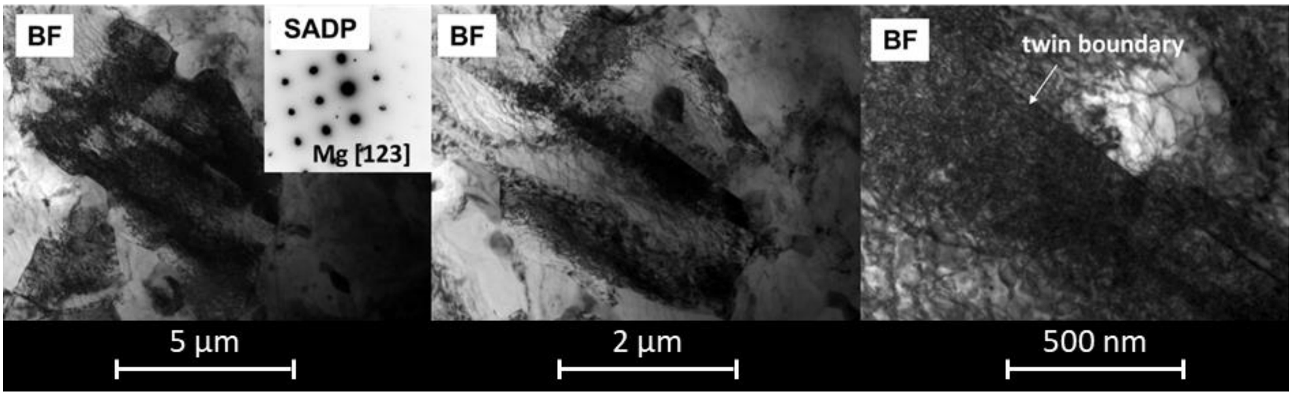


Figure 3 Set of bright field (BF) images and corresponding selected area diffraction pattern (SADP) of sample after 1 pass of ECAP

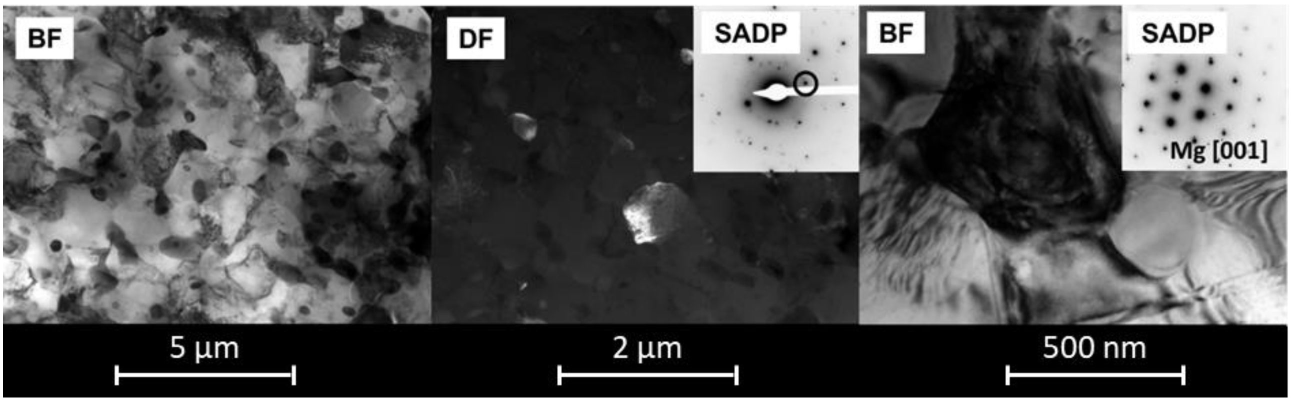


Figure 4 Set of bright field (BF) images and corresponding selected area diffraction pattern (SADP) of sample after 3 pass of ECAP

Figure 5 presents BF images taken at different magnifications and corresponding SADP of samples after one (upper) and 3 (lower) passes, respectively. One can see that in case of sample after one pass of ECAP, irregular precipitate of $Mg_{17}Al_{12}$ type with the [101] zone axis is located inside grain with high density of dislocations and twins. This type of precipitates is mainly present in AZ61 magnesium alloys. Also it can be assumed that these precipitates contain some amount of Zn. In the case of sample after 3 passes through ECAP performed at higher temperature the $Mg_{17}Al_{12}$ precipitates of size of about 200 nm possess rounded shape and mainly are located at the grain boundaries strongly affecting on adjacent area.

The Mg alloy AZ61 subjected to SPD by ECAP possesses the two-phase microstructure consisting of Mg grains of the size close to 1.5 µm after three passes and $Mg_{17}Al_{12}$ precipitates of the size scattered below 200 nm. Grain refinement of the investigated alloy was achieved by a combination of severe plastic deformation with subsequent initiation of dynamic recrystallisation. Grain refinement of the grain size from 24 µm to 1.5 µm was achieved after the third ECAP pass. Thus, it is apparent that Mg-Al-Zn grain refinement is made possible by the interaction of dislocations with recrystallised grains nucleated in the dislocation cells.

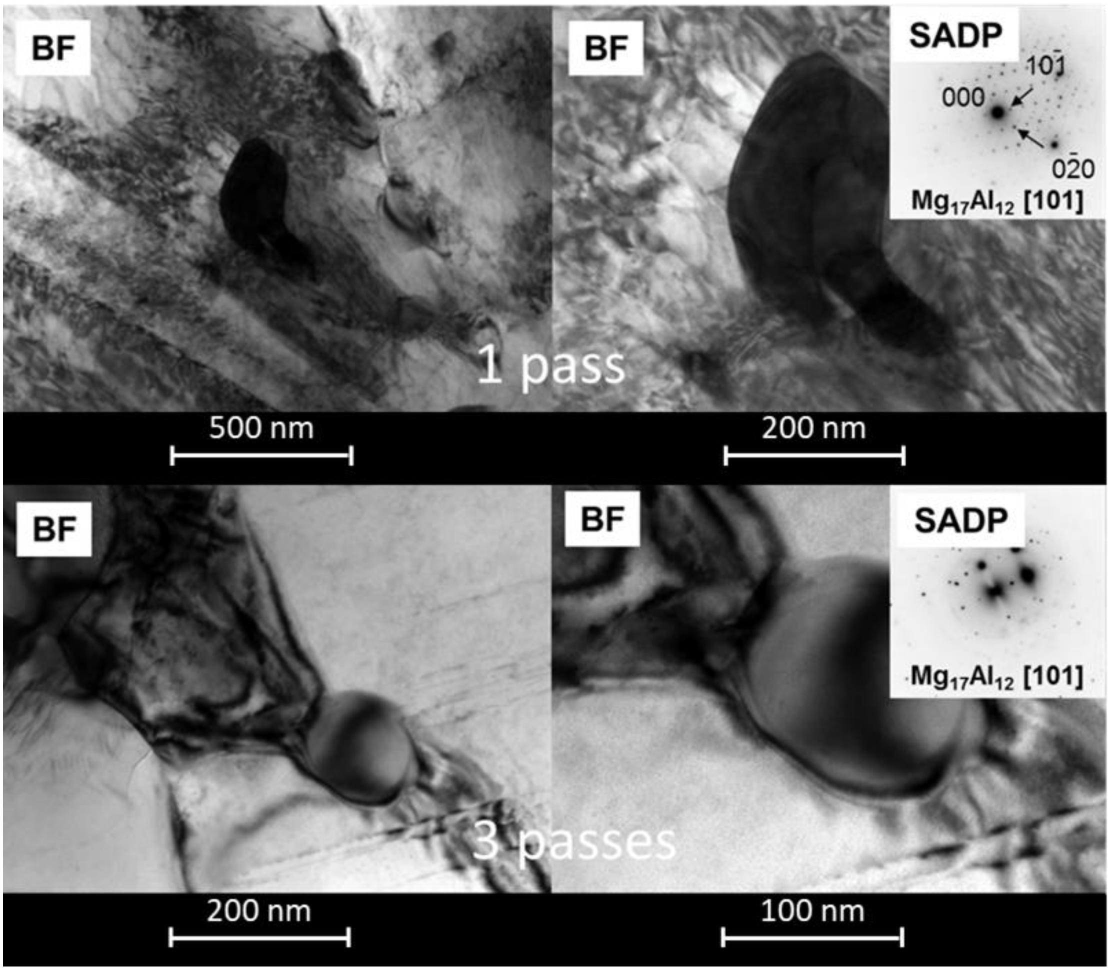


Figure 5 BF images taken at different magnifications and corresponding SADPs of samples after 1 (upper) and 3 (lower) passes, respectively

4. CONCLUSION

The hot extruded AZ61 magnesium alloy was processed by severe plastic deformation method ECAP method using up to three passes at 200 °C. The microstructure and tensile properties were studied using OM and, TEM. The following conclusions have been drawn:

- The TEM images of AZ61 magnesium alloy examined after the first pass show an increase of the dislocations density and deformation inside the grains. The results show the significant influence of the forming under SPD conditions on the formation of dislocation substructure.
- TEM microstructure of AZ61 magnesium alloy after three passes show Mg matrix microstructure and $Mg_{17}Al_{12}$ precipitate. TEM microstructure of AZ61 alloy after three passes show dislocation structure inside the grain.
- The AZ61 magnesium alloy subjected to SPD by ECAP passes possesses the two-phase microstructure consisting of Mg grains of the size close to 1.5 μm after 3 passes and $Mg_{17}Al_{12}$ precipitates of the size scattered from 100 to 200 nm. The primary precipitates of Al_6Mn phase were also observed.
- Refinement of the grain size is probably due to polygonization process associated with the formation of high angle grain boundaries due to rearrangement of the dislocations and to formation of subgrain structure.

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