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GRAIN REFINEMENT OF EXTRUDED AZ31 MAGNESIUM ALLOY BY ECAP PROCESS

ZJEMŇOVÁNÍ ZRNA PROTLAČOVANÉ HOŘČÍKOVÉ SLITINY AZ31 PROCESEM ECAP

Abstract

Extruded Mg – 3%Al – 1% Zn (AZ31) magnesium alloy was subjected to ECAP (Equal Channel Angular Pressing) processing at 523 K (250 °C). At the processing temperature of 523 K, fine grains with the average grain size of 2 – 3 μm are formed as a result of dynamic recrystallization originated by fine $Mg_{17}Al_{12}$ (γ) phase particles having 200 nm diameter dynamically – precipitated during ECAP processing. Microstructural evolution during ECAP was studied systematically using optical microscope and transmission electron microscope.

Keywords

AZ31, EX – ECAP, Optical microscopy, Transmission electron microscopy.

Abstrakt

Protlačená hořčíková slitina Mg – 3%Al – 1%Zn (AZ31) byla zpracována procesem ECAP (úhlové protlačování rovnostranným kanálem) realizovaného při teplotě 523 K (250 °C). Při teplotě 523 K bylo dosaženo zjemnění průměrné velikosti zrna 2 - 3 μm, které bylo dosaženo dynamickou rekrystalizací jemné γ faze $Mg_{17}Al_{12}$ s průměrnou velikostí 200 nm. Vývoj mikrostruktury během ECAP byl systematicky studován na optickém mikroskopu a využitím transmisní elektronové mikroskopie.

Klíčová slova

AZ31, EX – ECAP, Optická mikroskopie, Transmisní elektronová mikroskopie.

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1 INTRODUCTION

Demand of magnesium alloys has been increasing remarkably. However, the application of wrought alloys is still limited because of inferior workability of the magnesium alloys having limited slip systems at room temperature. Furthermore, strength of the conventional wrought magnesium alloys is lower than that of aluminium alloys, which prevents application to structural component that requires high strength. It has been reported that the strength of magnesium alloys is improved by means of grain refinement according to the Hall - Petch relation (1). Also, recent research reveals that high ductility can be obtained by structure control such as grain refinement and texture control even in magnesium alloys. Recently, Toth et. al. reported that the ductility is improved in fine – grained magnesium AZ31 (Mg – 3%Al – 1%Zn) alloys due to the activity of non – basal slip, grain boundary sliding (GBS) and recovery at high strained region. That is, grain refinement improves both strength and ductility in magnesium alloys [1].

$$\sigma_y = \sigma_o + \mathrm{k} \cdot d^{-\frac{1}{2}} \qquad (1)$$

where are:

σ_y – yield stress [MPa],

σ_o – stress for overcoming of Peierls-Nabarr friction stress of lattice[MPa],

k – constant, the measure of which is the value of shearing stress necessary for release of accumulated dislocations [–],

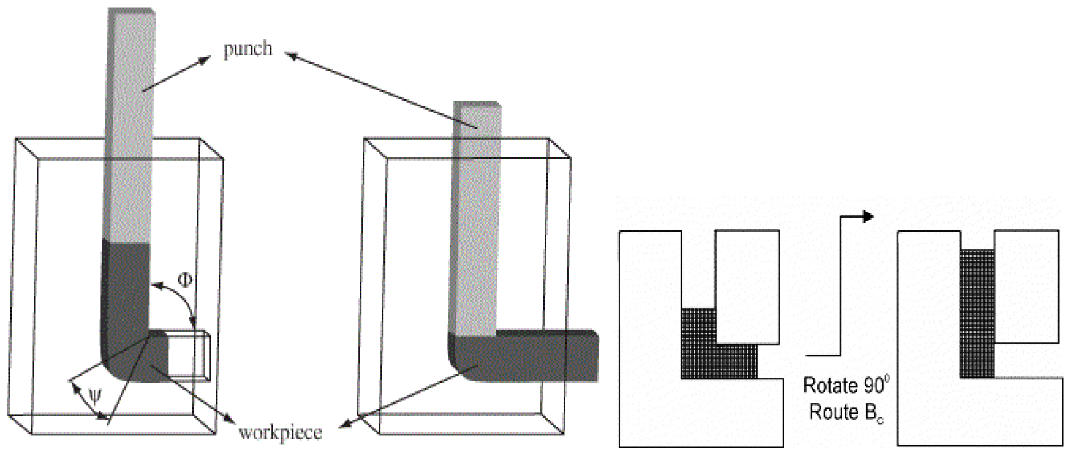
d – average grain size [nm].

Equal-channel angular extrusion (ECAE) also known as Equal Channel Angular Pressing (ECAP) is an emerging mechanical or thermomechanical method for the synthesis of bulk ultrafine-grained or nanomaterials. The uniqueness of ECAP is that fine grains are obtained without changing any of the dimensions of the sample. In the ECAP process, a well - lubricated billet is pressed into a die that containing two channels with the same cross section, intersecting at an angle called die channel angle. The material is subjected to shear deformation at the intersection plane. As the process is capable of maintaining the net dimensions of the workpiece, repetitive extrusion is possible, provided that the material exhibits sufficient ductility to withstand heavy deformations.

The Equal Channel Angular Pressing (ECAP) method is based on the dislocation activities and is configuration during increased deformation degree [1 – 3]. In **Fig. 1a** is present the principle of ECAP forming method. Sample is pressed through an L – shaped channel, it is constrained to deform primarily by simple shear along the plane of intersection between vertical and horizontal part of channel. For increasing the ECAP method efficiency, eg number of passes, is used deformation route Bc and ECAP channel geometry with helical horizontal part [4].

For creation of a very fine-grained structure formed by equiaxed grains separated by high-angle boundaries the angle of ϕ = 90° and the deformation rout Bc [1] when the extruded sample after separate pass is always rotated about longitudinal axis by 90° in the direction of clock hands, is the best. Using of deformation route Bc (**Fig. 1b**) leads to the achievement of the ultra-fine grained equiaxed structure.

For increasing of efficiency of the ECAP process the formed sample can be repeatedly extruded trough channel with the use of rotation of the sample between separate passes (used deformation routs are shown on **Fig. 1b**). When using samples rotation the activation of secondary slip systems and increasing of the amount of accumulated dislocations arise. According to the experimental publications [1 – 4] the highest efficiency has the combination of ECAP with using of deformation route Bc.



a) **b)**

Fig. 1 a) The principle of Equal Channel Angular Pressing (ECAP) method [5]; **b)** principle of the ECAP processing route Bc [6]

2 EXPERIMENTAL PROCEDURE

Magnesium alloy AZ31 was casted and hot extruded at temperature 430 °C to the blocks 40 x 40 – 1000 mm. The hot extrusion has a significant positive impact on the homogenously distribution of the typical intermetallic phases $Mg_{17}Al_{12}$ and average grain size reduction. The chemical composition of the experimental alloy AZ31 is presented in **Tab.1**.

Tab 1 Chemical composition of AZ31 magnesium alloy

Element	**Al**	**Zn**	**Mn**	**Si**	**Fe**	**Ni**	**Cu**	**Mg**	**Rest.**
Wt. %	3,100	1,200	0,500	0,100	0,005	0,005	0,050	94,740	0,300

An ECAP die used for the present study has two equal channels of dimensions 15 x 15 mm. The intersecting angle between the two channels is 90° and the angle of the outer arc at the intersection is 9,5°. Four – pass ECAP processing was carried out in the temperature 250 °C. The ECAP specimens (15 x 15 – 60 mm) were rotated by 90° around the longitudinal axis of the specimen after each pass (route B_c) to obtain a homogeneous microstructure.

The structural analysis of the initial state was carried out on the optical microscope NEOPHOT 2 and samples (from 1^{st} to 4^{th} pass) were before evaluation mechanically grounded (sandpaper on the SiC basis with roughness 800 and 1500) and then etched (3 % of Nital solution, time of immersion from 5 to 10 seconds, followed by washing and hot air drying).

3 EXPERIMENTAL RESULTS

To investigate the effectiveness of multiple pass ECAP on the grain refinement of AZ31, the specimens were subjected up to four ECAP passes at 250 °C and the resultant microstructures are shown in **Fig. 2**. The optical micrographs show increasingly homogenous equiaxed and refined microstructures after every additional pass. The evolution of the microstructure from highly distorted grains to equiaxed grains at the end of every additional ECAP pass indicates that continuous recovery, recrystallization and growth are active during multiple ECAP passes.

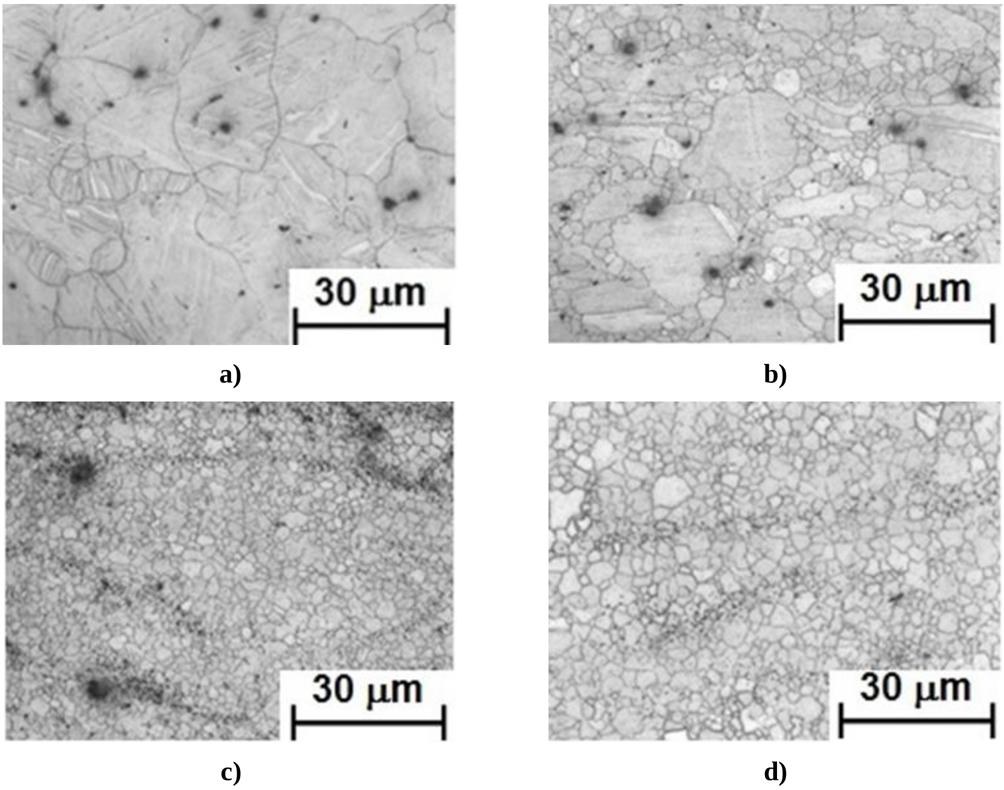


Fig. 2 Optical micrographs showing microstructure evolution of AZ31: **a)** before ECAP; **b)** after one pass; **c)** after three passes; **d)** after four passes of ECAP

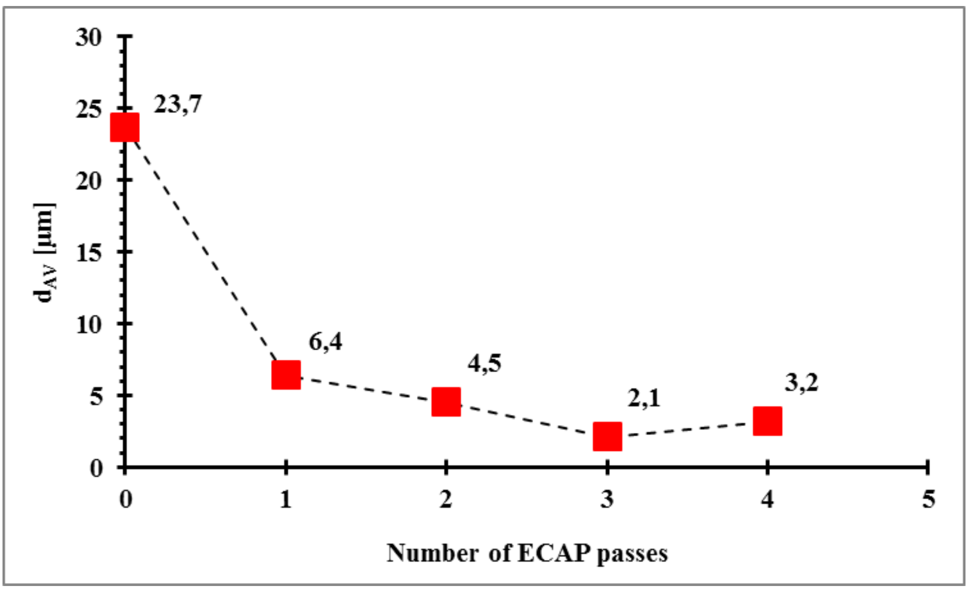


Fig. 3 The average grain size of AZ31 after different ECAP passes

In the **Fig. 3**, the grain size evolution after each ECAP pass is presented.

The optical micrographs and the surface relief both indicate significant grain boundary bulging after deformation. The grain boundary regions from the gridded surface observations show large undulations and the grids are distorted in these regions more than they are in the grain interiors These observations indicate that the specimen is undergoing dynamic recrystallization. So, although in this instance it may be true that the deformation is largely accommodated at the grain boundary regions, there were no sudden offsets that are indicative of the classical GBS.

Figure 4 presents BF images taken at different magnifications and corresponding SADP of samples after 1 (upper) and 3 (lower) passes, respectively. One can see that in case of sample after 1 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 AZ31 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.

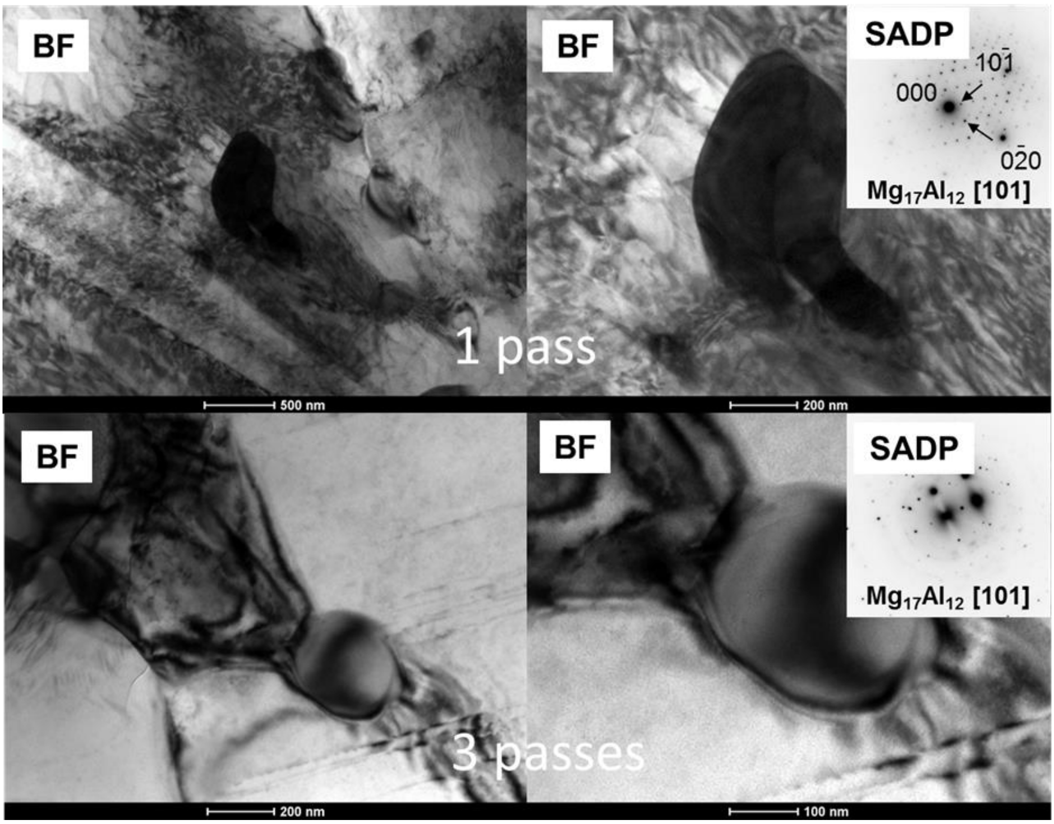


Fig. 4 BF images taken at different magnifications and corresponding SADPs of samples after 1 (upper) and 3 (lower) passes, respectively

4 DISCUSSION

It is well established that multiple pass ECAP is able to achieve good grain refinement in many initially coarse – grained materials. While models have hitherto been proposed based on studies on ductile materials like Al and Cu at room temperature [8], the limitations of such models on Mg processed above room temperatures have been highlighted in the earlier sections. **Fig. 5** is a schematic illustration of the model proposed for the grain refinement of Mg during ECAP [8]. The illustration applies to the grains in the centre portion of the billet [7 – 9].

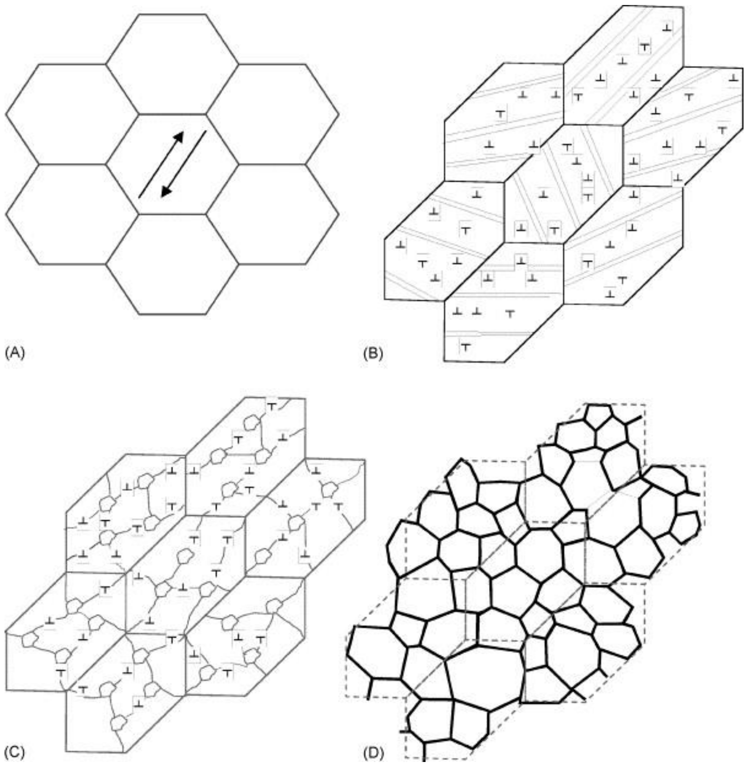


Fig. 5 Proposed grain refinement mechanism during ECAP of Mg at above room temperatures showing: **a)** equiaxed and coarse-grained microstructure before ECAP; **b)** immediately after shear deformation, original grains deformed along the shear plane, shear bands and dislocation pile-ups formed within grains; **c)** shortly after deformation, continuous recovery due to heating, leading to rearrangement of dislocations and formation of subgrain structures, nucleation of new strain free grains within shear bands also occurs; **d)** static growth from heating leads to formation of high angle boundaries from subgrains and recrystallized grains; equiaxed grains formed within boundaries of sheared microstructure [9]

AZ31 Mg alloy samples were processed by ECAP at 250 °C for up to four passes. The results showed that the grain refinement mechanism for Mg during ECAP is by a combination of mechanical shearing and subsequent continuous recovery, recrystallization and growth of grains and subgrain cells to produce refined and equiaxed grains within one ECAP pass [7 – 9]. Multiple ECAP passes showed that further grain refinement can be achieved with every additional pass. It was observed that temperature plays a key role in the effectiveness in the grain refinement of Mg by ECAP in addition to the processing route and amount of strain accumulation [9].

5 CONCLUSIONS

The hot extruded AZ31 magnesium alloy specimens were processed by ECAP at 250 °C for up to four passes. The metallographic analysis was made on the optical microscope. The results showed that ECAP has a significant influence on the grain refinement and with increases number of ECAP passes are applied influences of the dynamically recovery and recrystallization to produce refined, equiaxed grains and subgrains with high angle boundaries. With increased number of passes was proved that during ECAP have significant influences a recovery and recrystallization.

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