STRUCTURE AND PROPERTIES OF AZ31 MAGNESIUM ALLOY AFTER COMBINATION OF HOT EXTRUSION AND ECAP

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Abstract
Equal channel angular pressing (ECAP) method was used for achieving very fine-grained structure and increased mechanical properties of AZ31 magnesium alloy. The experiments were focused on the, in the initial state, hot extruded alloy. ECAP process was realized at the temperature 250°C and following route Bc. It was found that combination of hot extrusion and ECAP leads to producing of material with significantly fine-grained structure and improves mechanical properties. Alloy structure after the fourth pass of ECAP tool with helix matrix 30° shows a fine-grained structure with average grain size of 2 μm to 3 μm and high disorientation between the grains. More experimental results are discussed in this article.

Keywords: ECAP, AZ31, Light optical microscopy, TEM, EBSD, Tensile test

1 Introduction

Equal channel angular pressing (ECAP) forming method leads to producing new materials with very-fine grained structure. It is many years well know that grain size has a significant impact, by the Hall-Petch eq. (1), on the mechanical and physical properties of material. Equation 1 indicates the impact of grain size (diameter) on the strength properties (yield strength) [1].

Very-fine grained materials can be produced by severe plastic deformation (SPD) methods using. SPD methods are based on the effect of dislocation activities and interactions. Adjacent grains normally have different crystallographic orientations and a common grain boundary. During plastic deformation, slip or dislocation motion must take place across this common boundary. Grains boundary acts as a barrier to dislocation motion [1-2].

It should be mentioned that, for high-angle grain boundaries (HAGBs), it may not be the case that dislocations traverse grain boundaries during deformation, rather, dislocations tend to “pile-up” at grain boundaries. These pile-ups introduce stress concentrations ahead of their slip planes, which generate new dislocations in adjacent grains [3-4].

Low-angle grain boundaries (LAGBs) are not effective in interfering with the slip process because of the slight crystallographic misalignment across the boundary. On the other hand, twin boundaries will effectively bloc slip and increase the strength of the materials. Boundaries between two different phases are also impediments to movements of dislocations [4].

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1.1 The principle of Equal Channel Angular Pressing

The Equal Channel Angular Pressing (ECAP) method is based on the dislocation activities and is configuration during increased deformation degree [3-5, 15-19]. In **Fig. 1** 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 [5, 20-23].

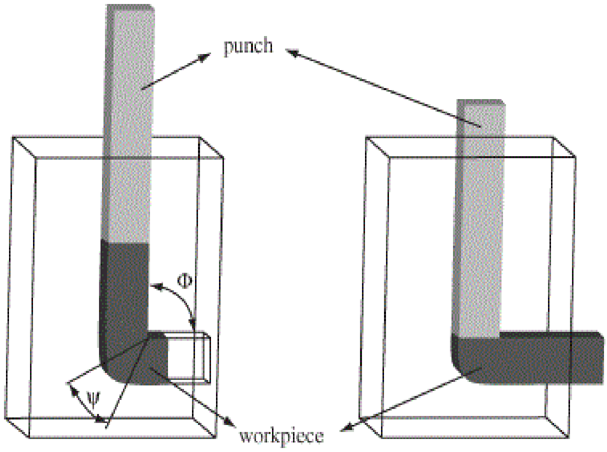

Fig. 1 The principle of Equal Channel Angular Pressing (ECAP) method [3]

2 Experiments

2.1 Material

Experiments were concentrated on the magnesium alloy with 3 wt.% of aluminium and 1 wt.% of zinc, AZ31.

Experimental material was casted and hot extruded at temperature 400°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, effect of hot extrusion on the structure of the AZ31 magnesium alloy shown **Fig. 2b**. The chemical composition of the experimental alloy AZ31 is presented in **Table 1**.

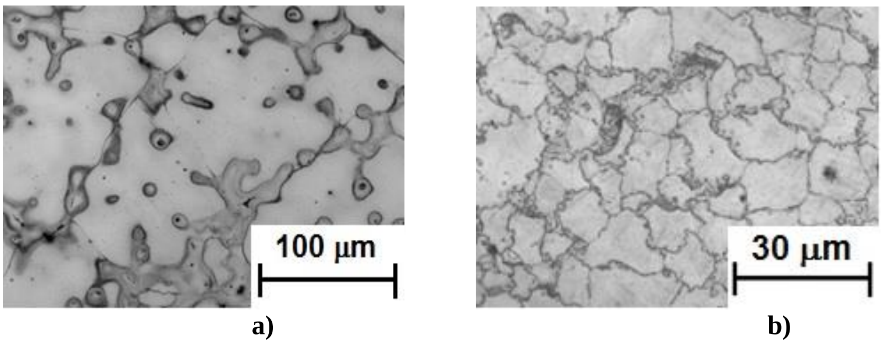

Fig. 2 Structure of AZ31: a) as – casted state, b) hot extruded (EX) [6]

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Table 1 Chemical composition of AZ31 magnesium alloy

Element	**Al**	**Zn**	**Mn**	**Si**	**Fe**	**Ni**	**Cu**	**Rest.**
Wt. %	3.100	1.200	0.500	0.100	0.005	0.005	0.050	0.300

The samples for metallographic evaluation on the light optical microscope NEOPHOT 2 were prepared by usual manner. Polishing of samples was made in two stages. In the first stage the samples were polished on cloth with use of the Al_2O_3 based polishing suspension. In the second stage the polishing was made on very fine velvet cloth with short fibres. Diamond powder with grain size of 1μm was used as polishing material. Diamond was applied by spraying and cloth was regularly wetted by alcohol – based liquid. The samples were then etched by Nital. Duration of etching varied from 5 to 10 seconds.
Microstructure of AZ31 in as – casted state is presented in **Fig. 2a**. The microstructure consists of solid solution matrix on the Mg base and intermetallic γ – phase $Mg_{17}Al_{12}$, or $Mg_{17}(Al,Zn)_{12}$near the grain boundary and nextprecipitates detected as Al_6Mn.

2.2 The ECAP process realization

Forming of aluminium alloys with the use of ECAP method was realized in the laboratory of the Department of Mechanical Technology, Faculty of Mechanical Engineering, VŠB – Technical university of Ostrava at the newly renovated DP2000 hydraulic press.
Samples, dimensions 15 x 15 – 60 mm, were extruded through tool with new geometry (with helix) of the channel (ϕ = 90°, ψ = 9,5° and α = 30°) and by selected deformation route Bc(maximal 4 passes to complete 360° rotation were realized). Extrusion was carried out at temperature of T = 250°C and chosen extrusion speed$v$ = 40 mm. min^{-1} (deformation speed$\dot{\varepsilon}$= 0.01 s^{-1}).
The hot extruded AZ31 sample with an average grain size of about 24 μm was subjected to first ECAP pass at 250°C. The longitudinal microstructure after the ECAP pass is shown in **Fig. 3b**, it is evident that microstructure exists from equiaxed grains. After first ECAP pass at 250°C were obtained average grain size of about 9.8 μm. **Fig. 3c** shown the microstructure of EX AZ31 after third ECAP pass, microstructure looks very-fine. Microstructure after four passes has a coarser grain compared with sample after third ECAP pass (**Fig. 3d**). This can be explained by metadynamic recrystallization (MDX) effect during individual ECAP passes. But microstructure looks still fine grained with relatively homogenized grain size distribution. The average grain size of EX – ECAPed AZ31 magnesium alloy after the third and fourth pass was 2.1 μm, 3.5 μm, respectively.
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.
Fig. 4 shows TEM micrographs of the microstructure of AZ31 after hot extrusion (**Fig. 4a**) and after four ECAP passes (**Fig. 4b**). Originated here many intermetallic inclusions in the vicinity of small grains. Due to the partial recrystallization of small defects formed within the grains and showed the heterogeneity of the structure here. Alloy structure after the fourth pass of ECAP tool with helix matrix 30° shows a fine-grained structure with average grain size of 2 μm to 3 μm and high disorientation between the grains (see **Fig. 4 a, b**). The experimental verification was achieved even with the largest increase deformable resistance during testing, refinement of large structures AZ31 alloys.

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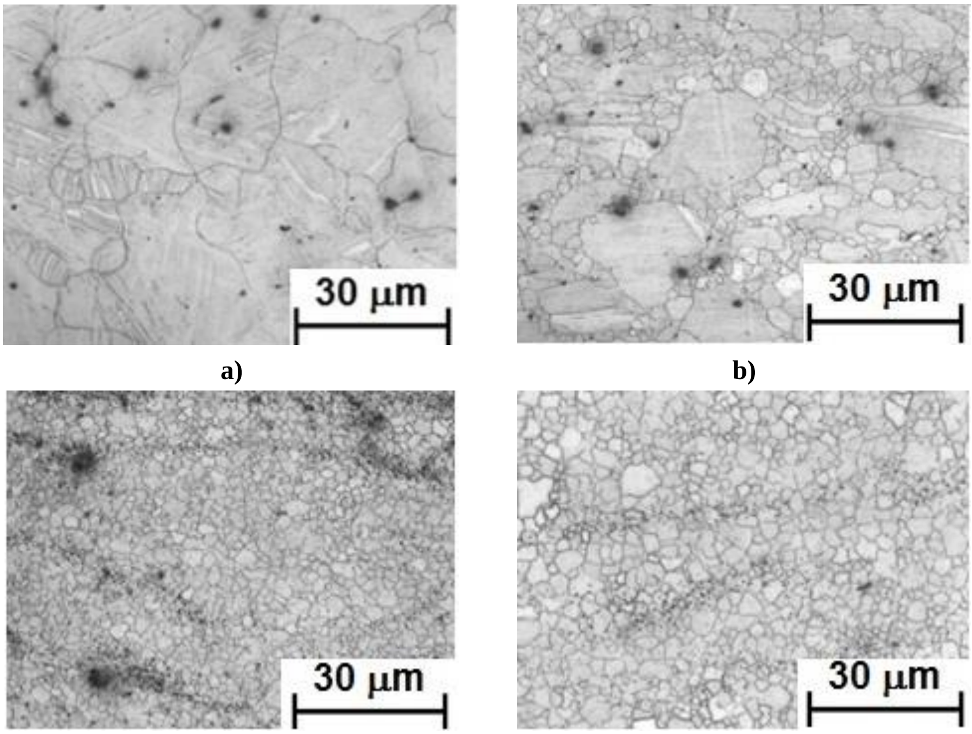
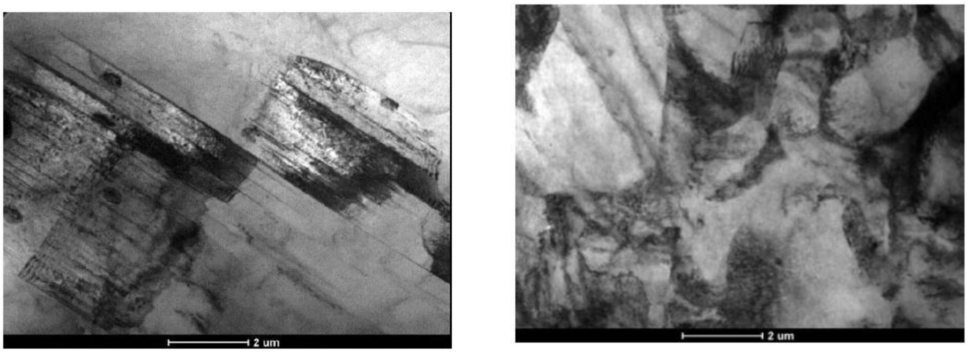


a) b) c) d)

Fig. 3 LOM - microstructure of AZ31: a) initial state -hot extruded (EX), b) one ECAP pass, c) third ECAP pass, d) fourth ECAP pass



a) b)

Fig. 4 TEM micrographs of AZ31: a) a) initial state -hot extruded (EX), b) fourth pass

2.3 Mechanical properties of EX – ECAP AZ31 magnesium alloy

Mechanical properties characterization of the material investigated was carried out with the use of Mini-Tensile Tests (M-TT) in servo-hydraulic system with 10kN capacity under strain rates corresponding with CSN EN 100002-1. M-TT specimens geometry and testing procedure was verified in [6]. Testing was performed under quasi-static loading conditions at room

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temperature. Strain was measured with the use of digital image correlation system ARAMIS by GOM [5, 7].

Table 2 Mechanical properties of AZ31

	YieldStrength[MPa]	**UltimateTensileStrength[MPa]**	**Ductility[%]**	**Hardness HV_{10}[-]**
Initial state - hot extruded(EX)	152	237	8	59
1st ECAP pass	193	329	9	68
2nd ECAP pass	202	341	11	71
3rd ECAP pass	205	343	11	73
4th ECAP pass	196	334	13	73

Results from M-TT measurements are presented in **Table 2**. It is evident that ECAP process has a positive impact on the mechanical properties increasing. M-TT results corresponding with metallographic analysis, the maximal value of YS and UTS were obtained on the sample after third ECAP pass. Evaluation of elongation shows an influence of recrystallization (DRX /MDX), as was confirmed by light optical microscopy and transmission electron microscopy. It can be said that recrystallization has an eminent role in grain refinement formation of Mg based alloys [6, 8-10].
It is well know that grain size has an impact on the strength properties increasing. The influence of grain size on the yield stress gives the Hall – Petch relationship (Eq.1).

$$\sigma_y = \sigma_0 + k \cdot d^{-\frac{1}{2}} \tag{1.}$$

where: σ_y[MPa] – yield stress
σ_0 [MPa] – stress for overcoming of Peierls-Nabarr friction stress of lattice
k [–] – constant, the measure of which is the value of shearing stress necessary for release of accumulated dislocations
d [µm] – average grain size [nm]

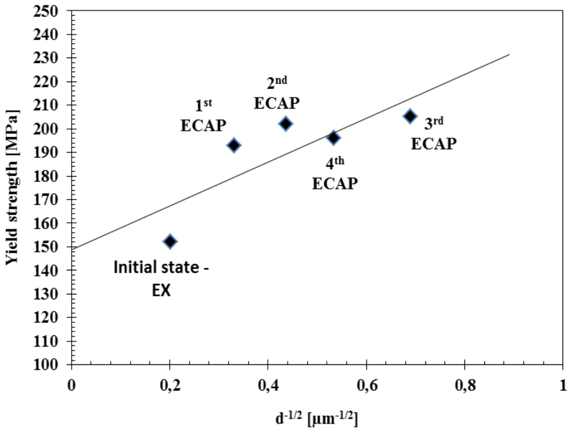

Fig. 5 Hall – Petch relationship for AZ31 processed by hot extrusion and ECAP

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The Hall – Petch relationship for EX – ECAP AZ31 is presented in **Fig. 5**. It can be seen that the strength of the EX – ECAPed AZ31increases with the decrease of grain size, but yield strength – grain size $d^{-1/2}$ is not linear. In [11] was presented strength dependence on the grain size for AZ31 magnesium alloys, with the similar texture, supports that the softening of ECAPed AZ31 alloys (a negative slope) which are typically observed despite the significant grain refinement, is due to the texture modification where the rotation of basal planes occurs towards the orientation for easier slip[12-14]. Thus, it can be concluded that the yield strength of the SPDed Mg alloy is determined by the competition of grain size strengthening effect and texture softening effect, and the texture softening depends on the texture type and texture intensity [15].

3 Conclusions

The hot extruded AZ31 magnesium alloy samples were processed by ECAP with helical horizontal part of the channel at 250°C for up to four passes. The metallographic analysis was made on the optical microscope and by using transmission electron microscopy (TEM). 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 and equiaxed grains and subgrains with high angle boundaries.

Micro – tensile tests evaluation of AZ31 after individual ECAP passes proved that ECAP process has a positive influence on the strength properties (YS, UTS and HV) increasing. With increased number of passes was proved that during ECAP have significant influences a recovery and recrystallization. Were achieved the samples with better plastically properties (Ductility), this point to effect of a softening.

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