GRAIN REFINEMENT OF AZ61 MAGNESIUM ALLOY BY ECAP PROCESS

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Abstract

The article describes the influence of a new ECAP forming tool draft (helix) for achieve a very fine grained structure of magnesium alloy AZ61. It was realized detailed measurements of hardness (HV) distribution and unconventional micro tensile tests for small samples (M-TT) after the individual passes through deformation route Bc. Effect of ECAP technology on the structure of AZ61 alloy on the optical microscope and electron microscope was measured.

Keywords: ECAP, magnesium alloy AZ61, micro tensile test, TEM, EBSD

1. INTRODUCTION

The ECAP technology is a very progressive forming method for producing material with very fine grained (UFG - Ultra Fine Grained) structure. By the Hall - Petch eq. (1) it can be assumed that UFG structure has an impact on the final mechanical properties of material as in the initial state (material without very fine grained structure):

$$YS = \sigma_o + kd^{-\frac{1}{2}} \qquad (1)$$

where *YS* present Yield stress, σ_o is a material constant for the starting stress for dislocation movement (or the resistance of the lattice to dislocation motion), *k* is a strengthening coefficient and *d* is the average grain size.

The ECAP method is based on extrusion of the sample through the tool with an internal L-shaped channel, without any change of cross-section of the sample, as it is evident from **Figure 1**. The sample is inserted from above into the vertical channel and then extruded through the tool. This operation is then repeated in order to achieve the required degree of deformation of the material leading to a refinement of the structure [1-3].

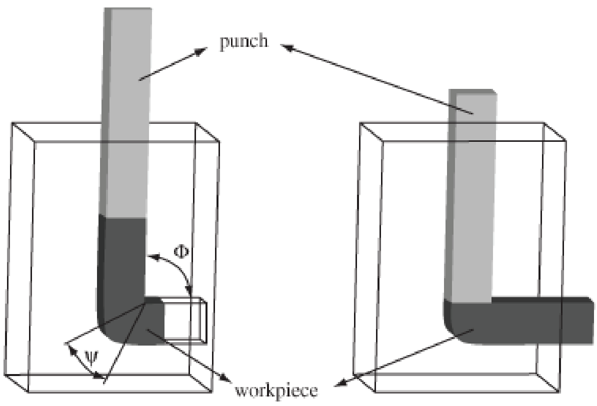


Figure 1 Principle of the ECAP process

We use tool with modified geometry for increasing the efficiency of the ECAP process (**Figure 2**). The principle is applying an additional torsion deformation of the sample passing through the horizontal part of the channel made in the shape of a helix with angle γ = 30°. The material will be strengthened very intensively, allowing us to achieve a high degree of deformation of material at a lower number of passes through the forming tool [1].

Figure 2 New concept of ECAP tool (helix in the horizontal part of channel)

2. EXPERIMENTAL PROCEDURE

2.1. Material

The investigation was carried out on magnesium alloy AZ61. Chemical composition of AZ61 alloy is presented in the **Table 1**. This material is in initial state after casting and extrusion at 430°C. For experimental purposes from the belts of alloy the test samples of the underlying dimensions 15 x15 - 60 mm in the direction of extruding were made [4 - 6].

Table 1 Chemical composition of AZ61

Weight [%]	Fe	Si	Mn	Ni	Cu	Mg	Zn	Al
AZ61	0.003	0.030	0.230	0.001	0.003	92.210	1.092	6.430

2.2. ECAP process

Extrusion was realised at 200°C (temperature of samples and in the deformation zone), the extruder feet rate was 40 mm/min ($\dot{\varepsilon} = 0.01\ s^{-1}$).

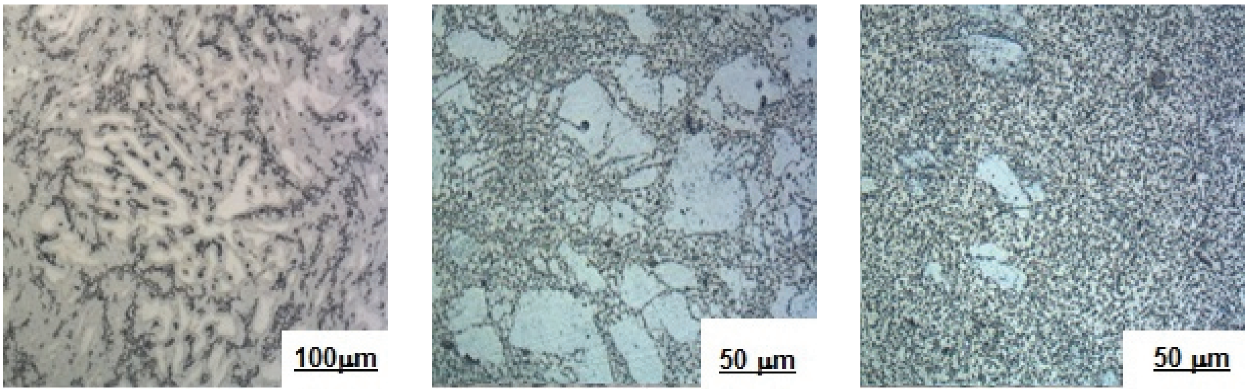
Three repetitive pressings were carried out on each sample with rotation about longitudinal axis by 90° in the same direction, this procedure has been designated as route Bc. For the next evaluation were taken samples after 1st and 3rd pass.

3. INVESTIGATION RESULTS

3.1. Microstructure and grain size

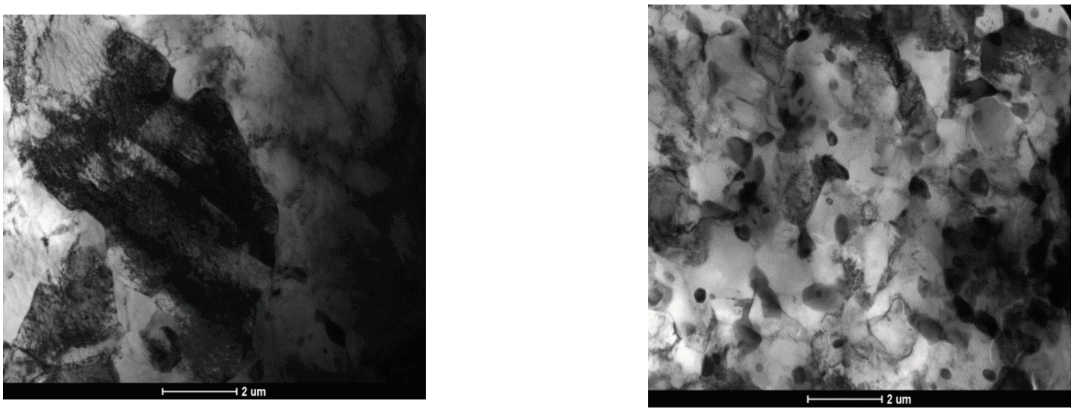
The extruded material was then divided into individual series for manufacture of testing specimens for metallographic evaluation.

The typical microstructure of the used alloy AZ61 is presented in **Figure 3a**. The basic microstructure of magnesium alloy AZ61 consists of solid solution matrix on the Mg base and massive γ- phase $Mg_{17}Al_{12}$, or $Mg_{17}(Al,Zn)_{12}$ with fine precipitates of these phases near the grain boundary. In **Figure 3b-c**, is presented microstructure of AZ61 after 1st, resp. after 3rd pass by ECAP tool with helix.



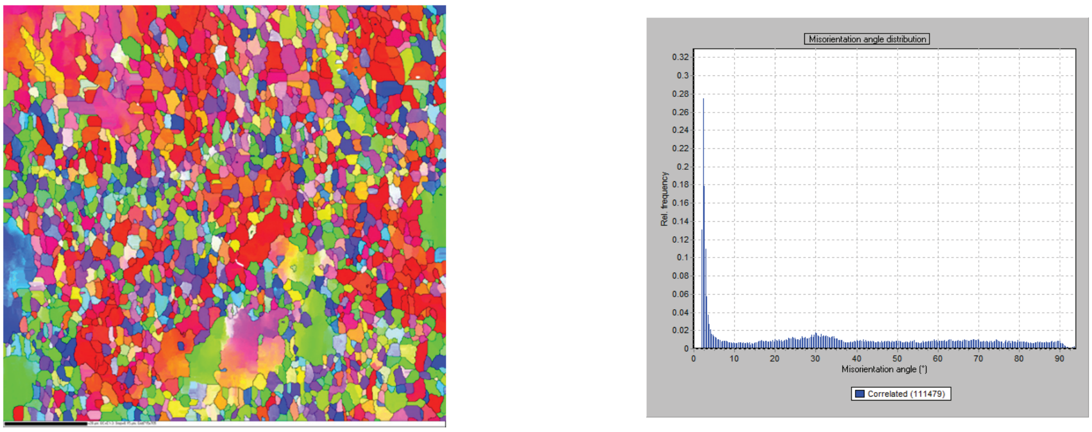
a) b) c)

Figure 3 Microstructure of magnesium alloy AZ61: **a)** initial state, **b)** after ECAP, the 1st pass, **c)** after ECAP, the 3rd pass



a) b)

Figure 4 TEM analysis of AZ61 microstructure: **a)** BF, ECAP 1st pass, **b)** BF, ECAP 3rd pass



a) b)

Figure 5 EBSD analysis of AZ61 microstructure after 3rd pass: **a)** EBSD, **b)** Misorientation profile correlated

The grains looks highly deformed which correspond to the route Bc that provides largest grain refinement and the most uniform structure.

The Mg commercial alloy AZ61 subjected to SPD by ECAP passes the two phase microstructure consisting of Mg grains of size close to 1.8 µm after 3 passes and $(Al, Zn)_{12}Mg_{17}$ precipitates of size scattered from 100 to 200 nm. Also the primary precipitates of Al_6Mn phase were observed in this alloy.

Based on selected area diffraction (SADP) and electron back scattered diffraction (EBSD - **Figure 5**) results obtained in TEM (**Figure 4**) and SEM respectively, it can concluded that plastic deformation of magnesium alloys induced by ECAP occurs mainly by slip mode forming the high density network of dislocation inside grains. The refinement of grain size is probably due to polygonization process associated with formation of high angle grain boundaries due to rearrangement of same signed dislocations.

As it could be seen (**Table 2**), during ECAP process occurred grain refinement. Final average grain size of AZ61 is 1.8 µm.

Table 2 Results of grain size measurement

Condition	Initial state	1st pass by ECAP	3rd pass by ECAP
Grain size [µm]	23.6	5.4	1.8

3.2. Mechanical properties

Materials with nanostructure are of the recent interest and demand for such a kind of materials is gradually increasing. With development of such a kind of materials is closely related demand on description of the material properties as development of these materials is usually carried out on small size samples. Thus for material mechanical properties assessment special testing procedures has to be employed. There has been performed extensive attention to development of a Micro Tensile Test (TM-TT) technique for testing of miniature samples. M-TT samples are made of same material volume as in the case of widely used small punch test which is requiring known correlation parameters for the material investigated, while M-TT should be able to assess also unknown materials, as there is carried out the same evaluation as in the case of standard tensile test. M-TT samples were made by [3].

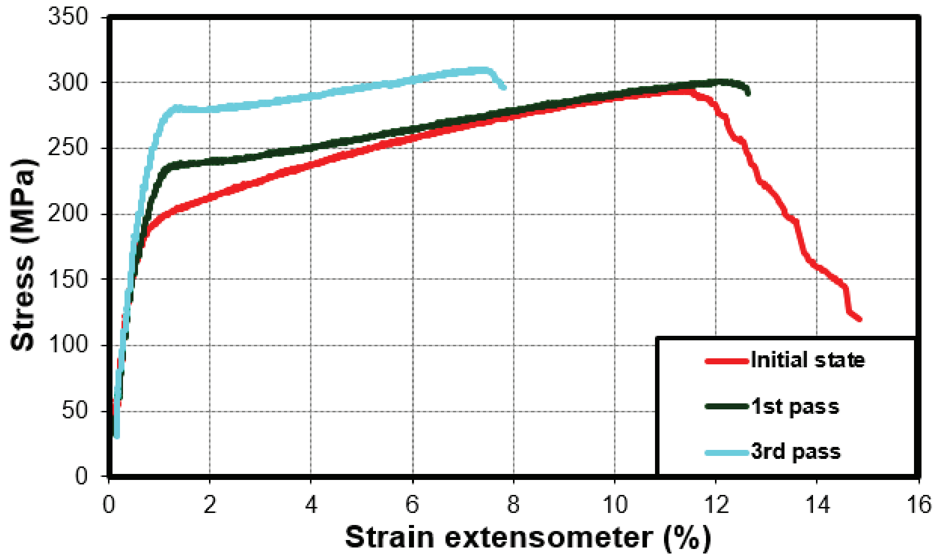


Figure 6 M-TT records for mechanical extensometer

Figure 6 presents actual results of mechanical properties measured by micro tensile tests. Results showed the positive impact of the ECAP technology on the strength parameters of AZ61 magnesium alloy.

As last was a measurement of hardness evaluation trough sample cross section on the Vickers hardness tester.

Mechanical properties of the samples in the initial state and after each passes through ECAP are presented in the **Table 3**.

Table 3 Mechanical properties of AZ61 magnesium alloy

	Yield stress [MPa]	Ultimate strength [MPa]	Ductility [%]	Contraction [%]	Hardness HV10 [-]
Initial state	180.9	295.3	14.3	17.2	79.2
1st pass	197.0	301.5	12.2	14.1	87.7
3rd pass	260.2	310.4	7.4	8.4	91.6

From the Micro tensile tests results is evident a positive effect of ECAP process on the final mechanical properties of AZ61 alloy. There is an increase of strength characteristics of the material (YS, US and HV). Simultaneously are deteriorating plastic properties of the material. It can be assumed that ECAP forming process leads to deformation strengthening from accumulated dislocations in the grain boundaries areas.

4. CONCLUSION

It may be stated on the basis of the obtained results that the new geometry of the ECAP tool has considerable influence of efficiency of the grain refinement process. It follows from metallographic analysis that substantial structure refinement takes place already after the 1st and 3rd passes. Due to the fact that the semi-products used for specimens showed already considerable heterogeneity. It will be necessary to achieve in future a very good homogeneity (Heat treatment application T4 - Solution treatment) for the subsequent ECAP process. Future work will focus on the influence of lower temperature on strengthening and final structure grain refinement.

- The TEM analysis after 1st pass show that there are presented in the structure of the severely deformed alloy. The BF (Bright Field) show the dislocation structure and stacking faults as result of interaction between matrix and precipitate during plastic deformation process.
- TEM microstructure of AZ61 alloy after 3 passes showing Mg matrix microstructure and $Mg_{17}Al_{12}$ precipitate. TEM microstructure of AZ61 alloy after 3 passes showing dislocation structure inside the grain.
- The refinement of grain size is probably due to polygonization process associated with formation of high angle grain boundaries due to rearrangement of same signed dislocations.
- Tensile tests results show a positive impact of the ECAP technology on the increase the mechanical properties. After the 3rd pass it will receive the highest values of Yield strength (~80 MPa) and Hardness (~12).

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