Mechanical properties and structure of AZ61 magnesium alloy processed by equal channel angular pressing

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Abstract. An equal channel angular pressing (ECAP) procedure has been developed to produce a fine-grained AZ61 magnesium alloy. The results show that the microstructure can be effectively refined with increasing equivalent strain during ECAP. For increasing ECAP process efficiency was conventional tool as a helix in the horizontal part of channel built. This fine-grained alloy has an excellent strength accompanied by reasonable good tensile ductility. The success of the development of this ECAP procedure can offer a good opportunity for the development of magnesium alloys with good mechanical properties.

1 Introduction

It is well known that there is relationship between grain size and properties of the materials. The grain Refinement causes a significant increase in strength in bulk metallic materials due to the Hall–Petch relationship which connects the mechanical properties and microstructural features such as the grain size. This relationship illustrates the potential for the mechanical strength increase by decreasing the size of grains and subgrains and this has been main driving force in the expansion of ultrafine grained (UFG) and nanostructured (NS) materials. From the practical point of view, materials with refined structure are most readily manufactured in bulk form by samples subjected to severe plastic deformation (SPD) processes. Over the last few decades, severe plastic deformation techniques have been extensively utilized to fabricate a bulk (NS) materials that exhibit an unusual properties which is very attractive for diverse structural and functional applications [1]. Ultrafine grained metals and its alloys made by severe plastic deformation techniques exhibits in fact excellent mechanical properties which are a result of significant grain refinement connected with dislocation strengthening.

The equal-channel angular pressing method is at present one of the most promising and commonly used of all Severe Plastic Deformation processes. In this technique, a sample with circular, square or rectangular cross section is pressed through a die consisting of two channels. These channels have identical cross-section and intersects at a specific angle of (Φ). The work sample to be processed is prepared to fit tightly the die channel and has to be well lubricated in order to minimalize friction coefficient. The specimen is inserted to the first channel of a die and then pressed through the second channel (with a constant speed) using a plunger. Ideally, severe deformation process occurs by simple shear in the area of intersection of two channels. As the work sample is pressed through this area, new regions of sample are exposed to shearing strain providing
a homogeneous shear deformation of the specimen apart from its end regions, which result in strain accumulation and grain/subgrain refinement [1-3, 9].

2 Experimental procedure

2.1 Experimental material
Experiments were conducted on a commercial AZ61 alloy to evaluate the potential for achieving an ultrafine grain size through the use of EX-ECAP two-step processing procedure of hot extrusion plus equal-channel angular pressing.

Tested material was in initial state casted and extruded at 430°C (703 K) to the dimensions 40x40 – 1000 mm (OML Skawina). Extrusion of as-casted Mg alloys has a positive impact on the homogenous grain size distribution [13] and homogenous distribution of the phases Mg17 (Al, Zn)12 [5]. The chemical composition of the material investigated is shown in Table 1.

| Table 1. Chemical composition of AZ61 alloy |
|-----------------|-----|-----|-----|-----|-----|-----|-----|
| 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
Samples were ECAPed at 200°C (473 K) on the hydraulic press DP 2000. Deformation route Bc (rotation about longitudinal axis by 90° in the same direction) was applied.

The ECAP process 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 [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. It is possible to use various types of deformation route changes for the ECAP process [1-3].

Equal channel angular pressing that was carried out using die with modifying tool geometry. ECAP + TE, i.e. Equal-Channel Angular Pressing combined with Twist Extrusion) combination was used for process efficiency increase. The tool contains an in-built helix with spiral angle of 30°. Advantage of the in-built helix consists in creation of counter-pressure, which enables an increase of the deformation degree and thus achievement of substantial grain refinement at smaller number of passes through the ECAP tool in comparison with the previously used tool geometries [3, 6]. Material of the ECAP tool is HOTVAR, that exhibits better strength stability during forming processes at higher temperatures [4].

2.3 Investigation procedure
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) and energy dispersive X-ray (EDX) EDAX microanalysis. 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(ClO4)2, 1000 ml methanol, and 200 ml 2-butoxy-ethanol at - 40°C and 80 V. EBSD data was collected using a high resolution FEI FEGSEM Quanta 3D equipped with TSL software.

From the reason of small sample dimension the Micro-Tensile Test Technique was developed in [10-12], that was applied for current material characterization.
3 Investigation results

3.1 Microstructure
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 1 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 2. 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, since the ECAP process was conducted at 200°C [7-8].

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

Figure 2. BF and dark field (DF) microstructures and corresponding SADPs of sample after 3 passes of ECAP

Figure 3 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 \( \text{Mg}_{17}\text{Al}_{12} \) 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 \( \text{Mg}_{17}\text{Al}_{12} \) precipitates of size of about 200 nm possess rounded shape and mainly are located at the grain boundaries strongly affecting on adjacent area.

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

Figure 4 present BF images at two different magnifications performed in two beam condition for Mg [761] zone axis in sample after 3 passes of ECAP. There are several precipitates of size of about 200 nm at the grain boundaries but no precipitates inside the grain. In the higher magnification image the stacking faults contrast (or dislocation pileups) is visible between two neighboring \( \text{Mg}_{17}\text{Al}_{12} \) precipitates. Therefore, it can conclude that location of small precipitates on grain boundaries strongly effecting these areas due to blocking movement of dislocations during plastic deformation. Extremely fine grains with average grain size of 0.5 \( \mu \text{m} \) are formed by dynamic recrystallization being attributable to dislocation pileups that occur in the vicinity of dynamically precipitated \( \gamma \)-\( \text{Mg}_{17}\text{Al}_{12} \) phase particles of less than 100 nm diameter. Such mechanism can be successfully adapted to our case even the grain and particle sizes are small different.
3.2 Mechanical properties

Mechanical properties characterization of the material investigated was carried out with the use of Mini-Tensile Tests (M-TT). M-TT specimens geometry and testing procedure was verified in [10-12]. Testing was performed under quasi-static loading conditions at room temperature. Strain was measured with the use of digital image correlation system ARAMIS by GOM. Four specimens' batches were tested: initial state – MPO, state after one pass – MP1, state after two passes – MP2 and the state after three passes MP3. Specimens were extracted in the longitudinal direction from ECAPed bars. Three specimens per condition were tested. Youngs modulus $E$, offset yield stress $R_{p0.2}$, ultimate tensile strength $R_m$, uniform elongation at maximum force $A_g$ and cross section reduction $C_R$ were evaluated. Examples of obtained tensile curves together with the specimens geometry can be found in figure 5. Summarized averaged resulting values of tensile test can be seen in table 2. Results clearly show strength parameters increase after the first pass by about 20% together with uniform elongation increase by almost 40%. However subsequent passes do not have any significant influence on the tensile properties that remain almost the same, except the yield stress that is gradually decreasing.

![BF images at two different magnifications performed in two beam condition for Mg [761] zone axis in sample after 3 passes of ECAP](image)

*Figure 4. BF images at two different magnifications performed in two beam condition for Mg [761] zone axis in sample after 3 passes of ECAP*

![Obtained tensile curves of AZ61 processed by ECAP method](image)

*Figure 5. Obtained tensile curves of AZ61 processed by ECAP method*
Table 2. Mechanical properties of AZ61 measured by M-TT

| Specimen | No. of passes | E [GPa] | Rp0.2 [MPa] | Rm [MPa] | Ag [%] | CR [%] |
|----------|---------------|---------|-------------|----------|--------|--------|
| MP0      | Initial state | 35,8    | 205,3       | 285,8    | 11,3   | 29,6   |
| MP1      | 1st pass      | 35,8    | 230,0       | 309,4    | 15,3   | 25,3   |
| MP2      | 2nd pass      | 38,0    | 217,5       | 308,7    | 15,4   | 24,7   |
| MP3      | 3rd pass      | 37,6    | 213,7       | 301,2    | 16,4   | 24,7   |

4 Conclusion
The main aim of the experiments is a refinement of the structure of magnesium alloy AZ61 using the minimum number of ECAP passes with special tools. To increase the degree of distortion, thereby achieving the desired structure is an important factor in appropriate modification tool geometry. Geometric adjustment of instruments is particularly evident in the new instrument ECAP helix is located 30° in the horizontal part of the channel, which shows an overall increase in efficiency of the SPD process.

Structure investigations demonstrate that ECAP process allows obtaining highly deformed grains that contains high density of shearing bands. Further ECAP process also causes subsequent grain refinement. The TEM analysis shows that there are presented in the structure of the severely deformed alloy two different areas, those defects free and with high dislocation density. It was also confirmed the presence of main hardening phase that occurs after artificial ageing. It was also found that the grain refinement occurs on the subgrain level.

Results from M-TT shows an highest increase of strength parameters (Rp0.2 and Rm) after first pass, but after further passes decrease Rp0.2 and Rm. It is evident that during ECAP process softening process occurs. This hypothesis was confirmed by Ag and CR parameters measurements and microstructure investigations. The Young's modulus of AZ61 studied is introduced in Table 2. E value seems by very low for typical magnesium alloy (prox. 42GPa). In the initial state (casted and hot extruded) is E = 36GPa, lower value of Young's modulus may be caused by hot extrusion after casting.

It was confirmed a positive impact of ECAP tool modification on the grain refinement process. During ECAP occurred due to high degree of deformation softening (polygonization and dynamic recrystallization).

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