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MICROSTRUCTURAL, THERMICAL AND MECHANICAL CHARACTERISATION OF ZK60 ALLOY PROCESSED THROUGH ECAP

Florina-Diana DUMITRU1, Brândusa GHIBAN1, José María CARRERA-MARRERO2,3, Oscar-Fabian HIGUERA-COBOS2,4, Gheorghe GURĂU5, Nicolae GHIBAN1

1Politehnica University of Bucharest, 2University Politecnica de Catalunya, 3Fundació CTM Centre Tecnològic, 4Universidad Tecnológica de Pereira, 5Dunarea de Jos University of Galati

Key words: magnesium alloys, ECAP, mechanical properties

Abstract: In recent years, a growing volume of research has been conducted on studying the effects of equal channel angular pressing processing. Equal channel angular pressing (ECAP) is a severe plastic deformation (SPD) technique, which improves the mechanical properties of the processed materials because of the ultrafine grained structures resulted. This paper investigates the microstructural evolution after ECAP, the recrystallization temperature for 3 heating rates (10, 20 and 40°C/min) and the room temperature tensile properties of the as-received and ECAP processed ZK60 alloy.

1. INTRODUCERE

Since 1989 when Herbert Gleiter presented the concept of nanocrystalline materials (ultrafine grained materials with a grain size under 100 nm), these materials have known a rapid development because of its technological and scientific importance [1]. Ultrafine grained materials can be obtained through a variety of methods, but in recent years, severe plastic deformation (SPD) techniques have been the focus of intense research [2], because they can produce metallic materials which have grain sizes ranging from 50 to 500 nm [3] and thus enhancing material properties such as improved tensile strength, hardness, toughness, fatigue life, and the optical and electrical related properties [4]. The interest in grain-size reduction is driven by the possibility to produce ultrahigh strength metals and high strain rate superplasticity.

Equal channel angular pressing (ECAP) is widely known as one of the techniques to impose severe plastic deformation on bulk materials to produce ultra-fine grained materials, without causing a significant change in the dimensions of the processed parts [5].

In the ECAP process, a metal billet is pressed through a die having two channels of equal cross-section intersecting at an internal angle, Φ, which is usually between 60° and 160° [6]. Another important feature of the die is the presence of an additional angle, Ψ, that defines the arc of curvature at the outer point of intersection of the two channels [7].

The billet undergoes simple shear deformation when it is pressed through the intersecting corner. With enough accumulation of plastic strain a new structure of submicrometer or even nanometer grain size replaces the former grain size [8].

Due to their high specific properties (superior specific stiffness and strength over many structural materials [9]), magnesium alloys are being viewed as the future materials for automobile, aerospace and electronics industries. However, their application potential is impeded by poor formability and limited ductility at room temperature, which is attributed to the hexagonal crystal structure and consequent non-availability of adequate numbers of independent slip systems [10].

The aim of this paper is to investigate the microstructural, thermal stability and mechanical behavior of a ZK60 magnesium alloy processed by ECAP.

2. EXPERIMENTAL PROCEDURE

The material used in this study is a ZK60 magnesium alloy, with the chemical composition (wt.%) listed in Table 1.

<table>
<thead>
<tr>
<th>Element</th>
<th>Zn</th>
<th>Zr</th>
<th>Al</th>
<th>Mn</th>
<th>Other elem.</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5.49</td>
<td>0.55</td>
<td>0.005</td>
<td>0.025</td>
<td>max 0.3</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

The as-received magnesium alloy was machined into square specimens with dimensions of 10 mm x 10 mm x 40 mm. The ECAP process was performed using a tool steel die with the internal angle between the two channels of 90° [11], having a strain of ε = 1.07 per pass. Back-pressure was used to ensure a uniform strain-stress distribution and to avoid failure during extrusion as reported in low ductile materials [12].

The samples were first lubricated with graphite before being inserted in the die. The ECAP was carried out following a different route from the major variants (Route A, Route B_a, Route B_b and Route C) which involved 180° back-and-forward rotation of the billet, along the transverse axis, by rotating the die with 90° between each pass. According to Valiev et al. [13] this type of processing is however equivalent to route A.

Prior to each pass, the billets were preheated to the respective temperature of ECAP in the die. The exact temperature of the ECAP was monitored through a thermocouple plugged into a multimeter placed exactly near the plane of intersection of the two channels. The extrusion speed of ECAP tests was 17.30 mm/s. The ECAP process was conducted at 250 ± 5 °C for 4 passes.

Samples with and without ECAP, cut in the extrusion direction, were prepared according to polishing and etching standards in order to reveal the microstructure of the material, which was observed with an Olympus BX51 optical microscope.

The ECAP specimens were cut into the middle and three samples were collected from the center of each billet. The transformation temperatures of ZK60 magnesium alloy after the ECAP process were determined by differential scanning calorimetry (DSC) using a 2920 MDSC calorimeter. Three
heating rates (10, 20 and 40°C/min) were applied in the DSC experiments under a nitrogen atmosphere and in a temperature range from 100 to 450°C.

Each specimen was placed in an aluminum crucible (6.5 mm inner diameter and 1 mm height) and introduced into the DSC furnace, while an empty Al crucible was used as a reference. The information obtained was processed using TA Instruments software.

The tensile properties were evaluated at room temperature in the longitudinal direction, but due to the fact that the samples in the sixth and seventh pass cracked, the tensile tests were performed only on samples taken during the first five passes and the sample without ECAP processing. The mini-tensile specimens with standard dimensions of the calibrated section of 4x1x4 mm were tested to failure using a DEBEN microtensile machine with an initial head speed of 3.3x10^-7 mm/s.

The microstructural observations were performed using a JEOL JSM 6400 scanning electron microscope.

3. RESULTS AND DISCUSSION

3.1 Microstructure

The results of the microstructural evolution through ECAP process are presented in Figure 1. It can be seen that the microstructure of ZK60 without ECAP is heterogeneous, with large grains surrounded by fine grains (Figure 1).

![Figure 1: Optical microstructure of the as-received ZK60](image1)

After the first pass a large grain refinement may be noticed, caused by dynamic recrystallization of the material during deformation and static recrystallization which may occur between passes (Figure 2a).

In the second and third pass (Figures 2b and 2c), along with a greater reduction in grain size, an elongation of large grains in the extrusion direction can be seen. It should be noted that after 4 passes of ECAP (Figure 2d), some coarse grains remain still visible, which indicates that after the ECAP process the microstructure is not fully refined. A similar structure ZK60 alloy was reported by He et al. [14] after 8 passes of ECAP at 240°C.

3.2 Calorimetric analysis

The DSC curves of ZK60 magnesium alloy with 1 to 4 passes of ECAP, obtained by continuous heating at three different heating rates (10, 20 and 40°C/min) are shown in Figure 3.

![Figure 3: DSC curves of the ZK60 alloy with 1 to 4 passes of ECAP at a heating rate of 40°C/min (a) and the curves for the three heating rates for ZK60 with 4 passes (b)](image2)

The recrystallization process can be observed as the first exothermic peak (peak 1) in Figure 3. It is also believed that the second endothermic peak (peak 2) corresponds to the eutectic temperature of the alloy, according to the Mg-Zn diagram [15] and to the investigations of Yu et al. [16].

It can be noticed a slight decrease of the peak temperatures as the number of ECAP passes increases, and that the value of the maximum peak temperature for the ECAP specimen increases at larger heating rates. Similar results have been obtained by Higa et al. [17] while investigating the thermal stability of ECAP processed copper.
3.3 Mechanical properties

Figure 4 shows the true stress - true strain curves for both the as-received and the ECAP processed alloy for the first 4 passes. For the ECAP processed samples, it can be noticed that the ductility varies in these samples, showing values ranging from 0.17 to 0.26. Also there is no simple relationship between the number of passes and ductility. The mechanical properties derived from the tensile tests (yield stress, ultimate tensile strength and elongation-to-failure) are presented in Table 2.

![Figure 4: True stress – true strain curves for the ZK60 magnesium alloy with and without ECAP](image)

Table 2. Mechanical properties of ZK60

<table>
<thead>
<tr>
<th>Number of ECAP passes</th>
<th>Yield stress, MPa</th>
<th>Ultimate tensile strength, MPa</th>
<th>Elongation -to-failure, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>216.24</td>
<td>285.11</td>
<td>14.86</td>
</tr>
<tr>
<td>1</td>
<td>207.03</td>
<td>310.51</td>
<td>23.21</td>
</tr>
<tr>
<td>2</td>
<td>158.26</td>
<td>258.99</td>
<td>17.31</td>
</tr>
<tr>
<td>3</td>
<td>144.18</td>
<td>235.64</td>
<td>25.91</td>
</tr>
<tr>
<td>4</td>
<td>131.66</td>
<td>244.60</td>
<td>21.60</td>
</tr>
</tbody>
</table>

It can be noticed that, both the yield stress as the ultimate tensile strength decrease with the number of ECAP passes. After the first pass of ECAP, the material presented an increase of ultimate tensile stress, which can be explained by the large number of dislocations introduced in the first stage of deformation. The reduction of the resistance with the number of passes may be caused by dynamic recrystallization mechanisms which occur during the process and leads to the elimination of dislocations, but also to the possible static recrystallization between passes. Similar behavior was reported by Lin et al. [18], which presented a slight reduction in mechanical properties with the number of passes of a magnesium alloy ZK60 after 4 passes under cyclic extrusion compression (CEC). Furthermore, the ductility of ZK60 processed by ECAP showed slightly increased values, despite of the appearance of the recrystallization phenomena. After the third pass of ECAP elongation remains almost constant.

The fracture surfaces of the ZK60 tensile samples in as-received state and after being deformed by ECAP can be seen in Figure 5. In the sample without ECAP (Figure 5a), one can see that the fracture surface typical contains cleavage facets, which is a common fact when deformation is governed by twinning, as it occurs in magnesium alloys at low temperatures [19].

After the first ECAP pass (Fig. 5b), the fracture surface consists of some cleavage planes and a large population of cavities of different sizes and shapes, which indicate the nucleation, growth and coalescence of cavities during fracture [20]. The fracture surface of the third pass sample does not show cleavage planes (Figure 5d), similarly to the specimens after 4 ECAP passes (Figure 5e). It can be observed that the strain at fracture was not much higher overall than that of the sample without deformation.

![Figure 5: Fracture surfaces of ZK60, in as-received state (a), and after 1 (b), 2 (c), 3 (d) and 4 (e) ECAP passes](image)

4. CONCLUSIONS

The microstructure of the magnesium alloy ZK60 processed by ECAP shows that grains are refined with the increasing number of ECAP passes at 250°C.

Using DSC analysis the recrystallization temperature of the ZK60 magnesium alloy has been studied. It was found that the peak temperature has a tendency to slightly decrease with the increasing number of ECAP passes.

The mechanical properties after the micro-tensile tests show that the values of elongation-to-failure improve in comparison with those of the raw material, because a finer grain microstructure should lead to a greater ductility. After the first ECAP pass, both the ultimate tensile strength and elongation present higher values than the as-received ZK60 magnesium alloy.

The fracture surfaces of the tensile samples showed cleavage facets, in the sample without ECAP, and cavities with different sizes and shapes, in the ECAP processed samples.
5. ACKNOWLEDGEMENTS

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