AZ31 Magnesium Alloy Subjected to Severe Plastic Deformation at Different Temperatures: Microstructure and Hardness

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Abstract. This paper focuses on a hot extruded AZ31 magnesium alloy processed by severe plastic deformation method TCAP (Twist Channel Angular Extrusion) at different temperatures. In order to characterize processing temperatures and the resulting structure and properties, the specimens were processed by 4 passes at 250°C, 230°C and 200°C. Microstructure and mechanical characterization was performed by Vickers hardness testing and metallographic analysis on an optical microscope.

1 Introduction

It is well established that grain size has a significant influence on the strength (hardness) of materials based on the Hall-Petch relationship (1) [1,2].

\[
YS(HV) = \sigma_o + k \cdot \frac{1}{\sqrt{d_{AVE}}}
\]  

where \( YS(HV) \) is yield stress and hardness, respectively. \( \sigma_o \) is the stress necessary for overcoming of Peierls-Nabarro friction stress, resistance of dissolved foreign atoms, resistance of precipitates from solid solution and lattice defects, \( k \) is the constant, the measure of which is the value of shearing stress necessary for release of accumulated dislocations and \( d_{AVE} \) is the average grain size.

It follows from the equation (1) that material yield value increases with decreasing grain size. This phenomenon is a driving force for research and development of high-strength structural materials, particularly of steels. It turns out that refining of grain can lead to increased drawability of metallic materials. On condition of identical strengthening mechanism refining of grains down to the level of nanometers can bring enormous increase in material strength.

Severe plastic deformation (SPD) is well known as an effective process in refining the grain size of metals and alloys to the nanometer range [3,4]. Several techniques have been presented as SPD methods, such as accumulative roll bonding (ARB) [5], twist extrusion (TE) [6] and friction stir processing (FSP) [7], but the most common methods are equal channel angular pressing (ECAP) [8] and high-pressure torsion (HPT) [9].

Interest in magnesium alloy has increased in recent years due to its high specific strength and low density compared to other structural metals and alloys. Weight reduction is an important factor in fuel economy, so magnesium alloys are attractive for use in the automotive [10]. As a consequence of its hexagonal close-packed (HCP) crystal lattice and limited slip systems, magnesium alloy lack ductility
at ambient temperatures [11]. At room temperature, deformation can be achieved by two dominant mechanisms: slip in the basal plane and mechanical twinning [12,13]. Magnesium alloy has three slip systems in the basal plane, of which two are independent, but in order to achieve homogenous deformation without cracks developing, five independent slip systems are required [14]. For this reason, the homogenous deformation of magnesium at room temperature without cracking is difficult and this limits its use in many applications.

1.1 Equal Channel Angular Pressing
A typical flowchart of this technology is shown in Figure 1. ECAP allows deformation of a metal sample by shear without changing its dimensions. A die comprises two channels with the same cross section, which intersect each other and create a bend. When extruding a sample through the die, the shear strain occurs in this place. The process can be repeated to achieve intensive plastic strain and a finer grain. The real deformation in one pass depends on the angle between axes of both the channels and to a less degree also on the bending radius. A sample can be rotated along its longitudinal axis between the particular passes, thus creating various technological routes of ECAP [15]. The technological route influences the process of refinement of grains and their shapes significantly [16].

1.2 Twist Channel Angular Pressing
The TCAP (twist channel angular pressing) is a method for increasing the efficiency of SPD. The horizontal part of the TCAP channel is manufactured into a helix shape with an angle of lead $\gamma = 30^\circ$, the TCAP die geometry thus combines the conventional ECAP method and the TE [17] method. During extrusion of an experimental sample with the use of a tool with modified geometry, the refining starts with shearing strain originated during the passes through the deformation zone, which is at the place of transition from the vertical position to the horizontal position of the TCAP extrusion channel. This part of the process is identical with the conventional ECAP process. After the sample passes through the deformation zone, the sample is then extruded into the helix. The effect of the helix in the horizontal part of the channel lies in the fact that the sample in the first stage is decelerated, which means it encounters a back pressure (BP). For continuation of the forming process, it is necessary to increase the extrusion force and this leads to an increase of strain intensity [18].
2 Experimental material and procedures

The experiments were concentrated on the influence of severe plastic deformation method TCAP on the grain refinement and tensile properties of hot extruded (at 400°C with ratio of 22) magnesium alloy AZ31.

2.1 Experimental material

The investigated material was supplied by the Institute of Non-ferrous Metals in Gliwice, Department of Light Metals (OML Skawina), Poland. The chemical composition of the experimental alloy is presented in Table 1.

| Weight [%] | Fe   | Si   | Mn   | Ni   | Cu   | Mg   | Zn   | Al   |
|-----------|------|------|------|------|------|------|------|------|
| AZ31      | 0.005| 0.100| 0.500| 0.005| 0.050| 94.740| 1.200| 3.100|

2.2 Experimental procedures

Due to the limited formability of investigated alloy at low temperature (below 200°C), the extruded alloy AZ31 was deformed by the TCAP method at a different temperature (200°C, 230°C and 250°C) for obtaining maximal grain refinement of deformed structure. TCAP processing was realized with the constant deformation rate of 40 mm/min and the samples were rotated by 90° around the longitudinal axis (Bc processing route).

The samples for metallographic analysis were cold mounted in resin, ground with silicon carbide papers and polished in cloth with diamond paste to a mirror-like finishing. The polished surface was etched with a solution of 5% vol. nitric acid and 95% vol. ethanol and observed on an optical microscope.

The hardness tests (HPO 350 testing machine) according to Vickers HV5 (load 50 N for 20 s) were carried out on etched metallographic samples.

3 Experimental results

3.1 Microstructure evolution

Microstructure of the investigated alloy in the as-cast condition is shown in Figure 3a-b. The microstructure consists of the solid solution of aluminium in magnesium (α-phase), surrounded by formations smaller amount of Mg17Al12 intermetallic (β-phase), nearly continuously distributed in the interdendritic areas along grain boundaries.
Figure 3. OM microstructure of the investigated alloy in two different conditions: as-casted (a-b) and hot extruded (c-d)

The microstructures of the casted and extruded AZ31 alloy are shown in Figure 3c-d. The initial microstructure is bimodal containing large grains elongated in the extrusion direction and smaller grains. An advantage of using the samples in the extruded initial state is the refinement of the cast grain having an average size of ~ 120 μm to a size of ~ 23 μm. The initial hot extrusion has a positive influence on the β-phase precipitates dissolution. It can be assumed that hot extruded alloy will have a higher plasticity and during TCAP processing will be no cracking of the specimens. We can assume that by using the initially extruded state of the samples, the number of TCAP passes necessary for the achievement of the desired refinement and homogeneity of the structure, can be reduced.

Taking into the melting temperature of the investigated alloy is ~ 625°C [19], recrystallization during deformation at 200°C is referred to as low temperature dynamic recrystallization. Obviously, deformation at those conditions, i. e. a relatively high temperature that is below the typical ductile transition temperature (225-250°C) necessary for activating pyramidal slip, caused mechanical failure.
Figure 4. OM microstructure of the investigated alloy processed by TCAP at 250°C: 1 pass (a); 2 passes (b); 3 passes (c) and after 4 passes (d)

Figure 4a-d shows the OM images of AZ31 magnesium alloy subjected to TCAP method. After 1 pass of TCAP at 250°C, the microstructure is more heterogeneous with very fine grains below 9 µm. With further TCAP processing at the same temperature, the volume fraction of fine grains increases while that of large grains decreases. The average grain size is further homogenous after 3 passes of TCAP (4.8 µm). Some grains are refined to a few micrometers with little residual strain whereas other large grains undergo distortion with trace of plastic deformation. This heterogeneity is believed to result from the different orientations of the grains in relation to the shear direction imposed by TCAP.

The results show a positive influence of TCAP processing realised at lower temperature on the Vickers hardness values and distribution (standard deviation in Table 2). After the 3 passes, the hardness increases (compared to the initial state) by approximately 22% (samples processed by TCAP at 250°C), by approximately 29% (230°C) and 38% (200°C), respectively.

| Processing method | 250°C  | 230°C  | 200°C  |
|-------------------|--------|--------|--------|
| Initial state (hot extruded) | 55±2.6 | 55±2.6 | 55±2.6 |
| 1 pass TCAP        | 59±1.8 | 62±2.1 | 68±1.9 |
| 2 passes TCAP      | 63±1.7 | 67±1.9 | 72±1.3 |
| 3 passes TCAP      | 66±0.9 | 71±1.0 | 76±0.6 |
| 4 passes TCAP      | 62±1.3 | 65±1.7 | 73±0.9 |

4 Conclusions
The hot extruded (EX) AZ31 magnesium alloy was subjected to severe plastic deformation method twist channel angular pressing (TCAP) using up to 4 passes at different temperatures (250°C, 230°C or 200°C). The following conclusions have been drawn:

- The effect of TCAP processing on the microstructure and the hardness of hot extruded AZ31 magnesium alloy was studied by means of optical microscopy (OM) and hardness testing.
TCAP is successfully used to achieve a reasonably homogenous ultra-fine grained (UFG) structure for the AZ31 alloy. With increasing number of passes, the initial coarse-grained structure has been transformed gradually into a very fine-grained microstructure with an average grain size of 4.8 µm (sample deformed at 250°C), 2.9 µm (230°C) and 1.7 µm (200°C), respectively.

- The TCAP processing leads to relatively slightly increasing of Vickers hardness values. With increasing number of passes, the standard deviation (inhomogeneity factor) decreases to minimum.

- It was observed that temperature plays a key role in the effectiveness in the grain refinement of magnesium alloy.

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