EXPERIMENTAL STUDY OF ULTRAFINE-GRAINED COMMERCIAL MAGNESIUM ALLOY AZ31 PREPARED BY SEVERE PLASTIC DEFORMATION METHOD

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Abstract

Ultrafine-grained (UFG) magnesium materials offer unique properties, for example high strength-to-weight ratio or improved corrosion resistance. Severe plastic deformation methods are very efficient for manufacturing UFG compact bulk material. Namely equal-channel angular pressing (ECAP) and high pressure torsion (HPT) currently attract significant attention of material science community. This contribution summarizes experimental study of UFG magnesium alloys. Investigation utilized light microscopy, scanning electron microscopy, microhardness measurements and other techniques. Results are discussed in light of other experimental studies.

Keywords: Ultrafine-grained (UFG) materials, Severe plastic deformation (SPD) techniques, Magnesium alloys

1. INTRODUCTION

Severe plastic deformation (SPD) techniques are attractive because they lead to very significant grain refinement to the submicrometer or even the nanometer level [1]. Ultrafine-grained (UFG) materials processed by SPD offer unique properties, for example better mechanical properties or corrosion resistance. A variety of special techniques are used for the production of bulk (UFG) materials, e.g. equal channel angular pressing (ECAP) [1], high pressure torsion (HPT) [2, 3], accumulative roll-bonding (ARB) [4], twist extrusion [5] or multi-directional forging [6]. Among these techniques, which introduce SPD in the material, the HPT is a very efficient method of grain refinement [7]. HPT was also successfully applied for producing consolidated metal powders [8]. On the other hand, ECAP is quite an easy and widely-used SPD technique.

Magnesium and magnesium alloys have hexagonal lattice which results in difficulties with SPD processes and requires precise optimization of several process parameters [9]. Temperature is one of the most important conditions [10]. At room temperature, the basal slip is mostly activated in magnesium [11]. Because of fewer number of slip systems in hexagonal closed packed lattice, basal slip does not offer five independent slip systems which are required for uniform deformation according to von-Mises criterion [12]. The deformation twinning provides additional independent deformation mode at room temperature. At elevated temperatures, the critical shear stresses for prismatic and pyramidal slip systems reduce significantly and the twinning contribution becomes less crucial [13, 14]. The objective of this work is to sum up, correlate and discuss various properties of UFG magnesium alloy AZ31 processed by extrusion (EX) and subsequently by ECAP (EX-ECAP).

2. EXPERIMENTAL MATERIAL AND PROCEDURES

AZ31 magnesium alloy (composition in weight%: 3.623% Al, 1.361% Zn, 0.291% Mn, 0.180% Ca, 0.004% Cu, 0.003% Fe, 0.002% Ni, 0.0014% Si, and the rest is Mg) was used in this investigation. The extruded material was prepared from as-cast state by extrusion at 350 °C with the extrusion ratio of 22. The ECAP conditions needed to be optimized to obtain compact specimens without surface cracking. These optimal conditions were found: pressing temperature of 180 °C, pressing speed of 50 mm.min⁻¹, MoSi lubricant. The
billets with dimensions of $10 \times 10 \times 100$ mm were ECAPed through a $90^\circ$ die via route $B_C$ for various numbers of passes.

Microstructure was investigated using light microscopy and scanning electron microscopy. Mechanical properties were studied using Vickers microhardness (HV0.1) measurements. Dislocation density was measured by positron annihilation spectroscopy. A $^{22}\text{Na}_2\text{CO}_3$ positron source with the activity of 1.5 MBq was used in positron lifetime measurements. The source spot with diameter of 1 mm was deposited on a 2 $\mu$m thick Mylar foil and sealed between two identical specimens of the studied material.

3. RESULTS AND DISCUSSION

Microstructure of the extruded (EX) and EX-ECAP material is shown in Fig. 1 a and b, respectively. Microstructure and texture of this material was studied in detail in Janecek et al. [15] using electron backscatter diffraction (EBSD) and transmission electron microscopy. The extruded material contains large grains of 50–100 $\mu$m mixed with relatively fine grains of 2–10 $\mu$m. Structure of the alloy after 1 and 2 EX-ECAP passes is also bimodal. On the contrary, microstructure of the EX-ECAP alloy after 4 passes is nearly homogeneous. Average grain size is approximately equal to 1 $\mu$m, which is smaller than the grain size of the same material processed by ECAP only [16]. Finally, microstructure of the EX-ECAP material after 8 and more passes is homogeneous but the probability of crack formation may increase.

![Fig. 1](image-url) Microstructure of the AZ31 alloy processed by a) extrusion and b) extrusion and 4 passes of ECAP (EX-ECAP); plane perpendicular to the pressing direction, scanning electron microscopy (SEM), magnification 4000$\times$ and 15000$\times$, respectively

Results from the microhardness measurements depending on the number of EX-ECAP passes are published in Vratna et al. [17]. The value of microhardness increases up to the fourth pass and then declines continuously with increasing strain. We can correlate these results with dislocation densities shown in [18].

During ECAP processing, dislocation density increases and reaches its maximum in the sample subjected to 2 EX-ECAP passes. However, further ECAP processing leads to a gradual decrease of dislocation density. This fact indicates probably recovery of dislocation structure connected with development of UFG structure. Grain refinement seems to have more major impact on microhardness than dislocation density in this stage of processing. Corrosion resistance is other interesting point of view in this investigation which plays an essential role in applications. Corrosion properties, studied in detail in diploma thesis [19] and published in [20], depend significantly on the number of EX-ECAP passes. Corrosion resistance of the alloy after 4 EX-ECAP passes at room temperature in standard 0.1 M NaCl solution is very good but only at shorter...
immersion times (< 1 day). Material after 8 EX-ECAP passes shows good corrosion resistance not only at shorter immersion times but also at longer immersion times (≈ 3 days and more). This investigation indicates that the homogeneity of microstructure is a determinative factor for good corrosion resistance. Structure stability at elevated temperatures is another factor influencing applicability of this UFG material. Some of the results are shown in [21]. Dependence of microhardness on annealing temperature is shown in Fig. 2. The samples were annealed for 1 hour at various temperatures.

![Fig. 2 Dependence of microhardness on temperature during isochronal annealing (for 1 hour)](image)

One can see that microhardness value does not change until the temperature reaches about 170 - 190 °C. Significant decrease of microhardness was observed between 170 and 230 °C and then between 400 and 450 °C. The first decline of microhardness could be caused primarily by recovery process – migration of dislocations into grain boundaries, their rearrangements or mutual annihilation of dislocations with opposite sign. These processes occur commonly at homologous temperatures \((T/T_M) \approx 0.3 - 0.5\). Melting temperature \((T_M)\) of pure magnesium is equal to 650 °C \((T_M(AZ31) \approx 636 °C [22])\). At higher temperatures, the process of grain growth and recrystallization occurs. Microstructure of annealed specimens is shown in Fig. 3.
Fig. 1 Microstructure of the AZ31 alloy processed by extrusion and 4 passes of ECAP (EX-ECAP) annealed for 1 hour at a) 210 °C, b) 300 °C, 400 °C (all SEM, magnification 8000×) and d) 500 °C (light microscopy, polarized light, magnification 500×)

4. CONCLUSIONS

This paper summarized, correlated and discussed various properties of UFG magnesium alloy AZ31 processed by extrusion (EX) and subsequently by ECAP (EX-ECAP). Microstructure was studied using scanning electron microscopy (see Fig. 1) and the results were correlated with other important properties (mechanical properties, dislocation density and corrosion resistance). Structure stability at elevated temperatures was studied using microhardness measurements (see Fig. 2) and microstructure observations (see Fig. 3). We demonstrated that the microhardness value does not change until the temperature reaches about 170 - 190 °C. Two significant decreases of microhardness were observed in the temperatures between 170 and 230 °C and then between 400 and 450 °C. These changes were caused mainly by recovery processes and grain growth, respectively.
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LITERATURE
