MAGNESIUM WITH Zr ADDITION PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING

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Abstract

Magnesium alloyed with 0.3 wt.% Zr was processed by extrusion followed by equal channel angular pressing (ECAP) for 1, 2, 4 and 8 passes. The material was characterized using microhardness measurements, compression tests and electron backscatter diffraction (EBSD). Coarse microstructure after extrusion was significantly refined. Microstructure after 1 and 2 ECAP passes was bi-modal and become homogeneous with average grain sizes ~2 μm after 4 ECAP passes. Zr addition was found to be critical for retaining refined microstructure. The highest microhardness was measured in the samples after 2 and 4 ECAP passes. Similar evolution was found for yield compression stress measured in normal direction. On the other hand, values of yield strength measured in extrusion direction were saturated in the samples after 2, 4 and 8 ECAP passes. Processing by ECAP led to significant microstructural refinement and substantial increase in strength.

Keywords: Magnesium alloys, ultrafine-grained materials, equal channel angular pressing, electron backscatter diffraction

1. INTRODUCTION

Severe plastic deformation (SPD) techniques are attractive and useful because they lead to very significant grain refinement to the submicrometer or nanometer level [1] and consequently the reduction of grain size results in changes in mechanical and other properties. Ultrafine-grained (UFG) materials processed by SPD techniques often show better mechanical properties in comparison with their coarse-grained counterparts. The most frequently used current techniques of SPD are equal channel angular pressing (ECAP) [1 - 4], high pressure torsion (HPT) [5 - 8], accumulative roll-bonding (ARB) [9], twist extrusion [10] or multi-directional forging [11]. ECAP proved to be capable of improving mechanical properties of advanced Mg-based alloys [12].

Magnesium has closed packed hexagonal crystal structure with parameters a = 0.320 nm, c = 0.520 nm and an axial ratio c/a = 1.624. Limited formability of hexagonal metals causes difficulties during SPD processes and requires precise optimization of processing conditions [13]. Temperature is one of the most important parameters [14]. At room temperature, the basal slip is mostly activated in magnesium [15]. 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 [16]. 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 [17, 18].

Alloying of Zr in magnesium results in stabilization of grain boundaries, which is critical for applications of fine-grained material at elevated temperatures. The maximum solubility of Zr in cast Mg is ~3.8 wt. % at the temperature 654 °C. From room temperature to approximately 573 K although higher solubility may be achieved using other processing techniques such as physical vapour deposition [19]. The alloy used in the present experiments is therefore on the limit of solubility and contains only very small amount α-Zr phases.

Magnesium with 0.6 % Zr was successfully processed by extrusion and 1 pass of ECAP to achieve very fine microstructure (~1 μm) and superplastic ductility [20].

The objective of this work is to investigate microstructure and mechanical properties of UFG magnesium alloyed with 0.3 wt.% Zr processed by extrusion (EX) and subsequently by ECAP.

2. EXPERIMENTAL MATERIAL AND PROCEDURES

Magnesium with Zr alloying element (0.3 wt.%) was used in this investigation. The extruded material was prepared from as-cast state by extrusion at 400 °C with the extrusion ratio of 30. The ECAP conditions needed to be optimized to obtain compact specimens without surface cracking. These optimal conditions were found: pressing temperature of 185-230 °C, pressing speed of 5-10 $mm \cdot min^{-1}$, MoSi lubricant. The billets with dimensions of 10 × 10 × 100 mm^3 were ECAPed through a 90° die via route B_C [21] for various numbers of passes.

Microstructure was investigated using electron backscatter diffraction (EBSD) in scanning electron microscope FEI Quanta. Mechanical properties were studied using Vickers microhardness (HV0.1) measurements and compressive tests.

3. RESULTS AND DISCUSSION

3.1. Microstructure

Microstructure of magnesium with Zr was investigated on the plane perpendicular to the direction of equal channel pressing (it is also perpendicular to the extrusion direction) using EBSD and the results are shown in **Figure 1**. In the case of material processed by extrusion and 1 pass of ECAP, the structure is not homogeneous and can be referred to as bimodal. It consists of large (~20 -70 μm) and small (~2 μm) grains. Microstructure of the sample after 2 ECAP shows further fragmentation of microstructure. The overall fraction of large grains significantly decreased and the microstructure consists of a lot of small grains (~2 μm) and same larger grains. After 4 ECAP passes, almost all large grains were refined and only few remnants of larger grains are observed on the large scan (**Figure 1c**). After 8 passes, sample has very homogeneous microstructure with the grain size of ~ 2 μm. Note that the size of small grains almost does not change with increasing number of ECAP passes.

Microstructure can be characterized by average grain sizes and fractions of high- and low-angle grain boundaries (HAGB, LAGB). The results are summarized in **Table 1**. Average grain sizes of the samples after 4 and 8 ECAP passes are ~2 μm. The fraction of HAGB is high (80 %) already after the first ECAP pass and is even higher after 8 passes (91 %). This very fine-grained microstructure is caused by zirconium which stabilizes grain boundaries so it is very good grain refiner [22]. ECAP processing of pure magnesium (with no Zr addition) leads to much coarser microstructure [23 - 26].

Processing by ECAP at elevated temperatures is a dynamic phenomenon. Severe deformation induces large deformation into the material and is responsible for substantial formation of dislocations. Thermally activated processes such as recovery and continuous and discontinuous recrystallization occur. It is believed that fine grained structure after one or two ECAP passes is formed mainly by discontinuous recrystallization. New fine grains are formed at stress concentrations (namely original grain boundaries) [27, 28].

After 4 or more ECAP passes microstructure is homogeneous and consists of small grains only. Grain size saturates as the result of balance between deformation induced by ECAP and recovery and recrystallization processes. Hindering of grain boundary migration is therefore critical for achieving very fine microstructures. In simple Mg-0.3Zr binary alloy, Zr atoms reduce grain boundary mobility due to grain boundary segregation [20].

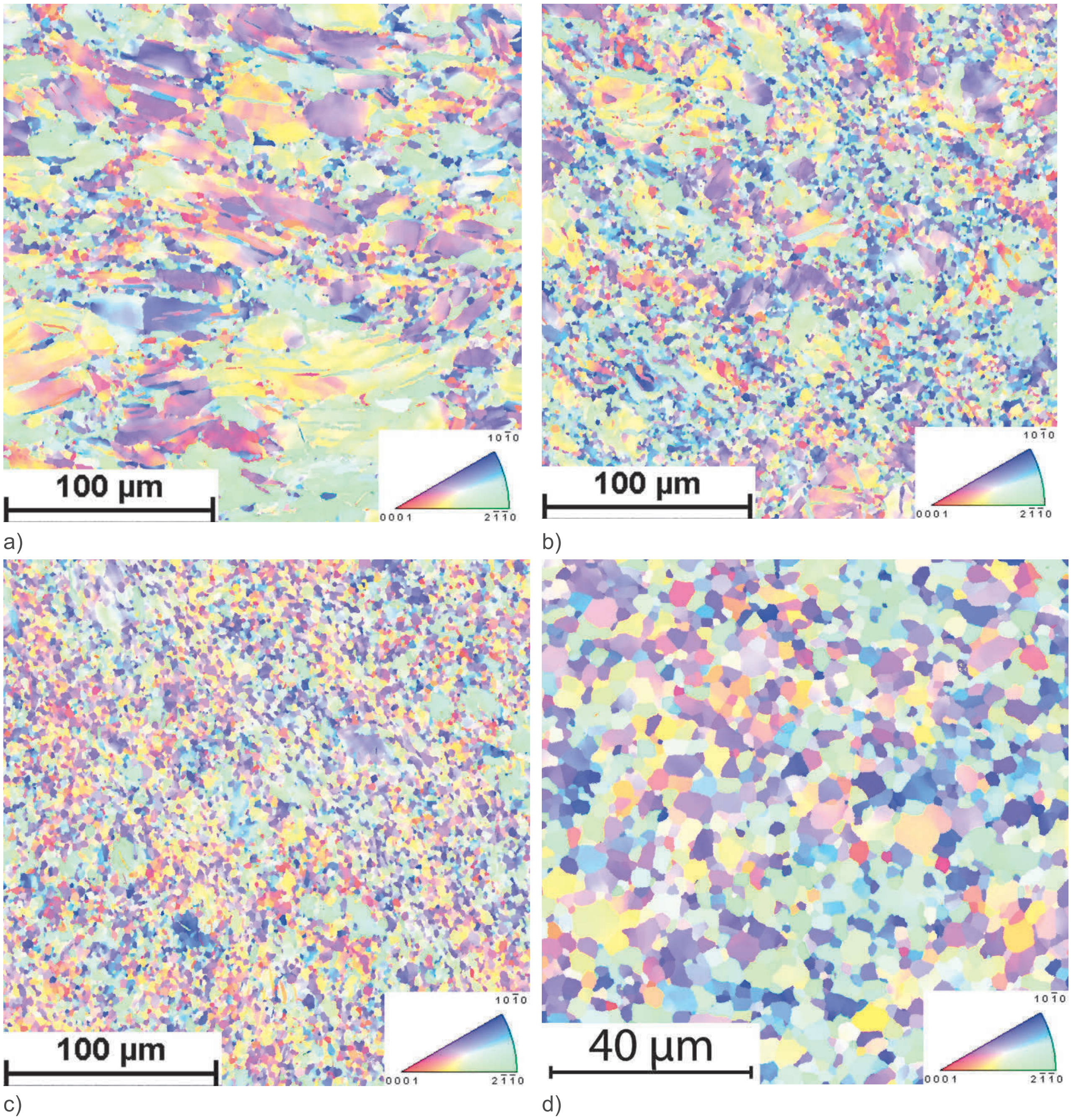

Figure 1 Inverse pole figure maps of MgZr alloy processed by a) extrusion and 1 pass of ECAP, b) extrusion and 2 passes of ECAP, c) extrusion and 4 pass of ECAP and d) extrusion and 8 pass of ECAP

Table 1 Average grain sizes and fraction of high-angle grain boundaries (HAGB, misorientation angle > 15°) and low-angle grain boundaries (LAGB, 5° < misorientation angle <15°) measured from EBSD

Sample	Average grain sizes	Fraction of HAGB	Fraction of LAGB
1P	Bimodal microstructure (~20 - 70 µm and ~2 µm)	0.80	0.20
2P	Bimodal microstructure (~20 µm and ~2 µm)	0.80	0.20
4P	(1.8 ± 1.1) µm	0.82	0.18
8P	(2.1 ± 1.3) µm	0.91	0.09

3.2. Mechanical properties

Mechanical properties were studied by microhardness measurements and compression tests. Microhardness was measured in the plane perpendicular to the extrusion direction using Vickers hardness method (100 g, 10 s) and the results are summarized in **Table 2**. At least 100 experimental points were measured in each specimen. Microhardness values increase from 37 HV (extruded) to 47 HV in the sample after 2 and 4 ECAP passes. Microhardness of the specimen after 8 passes is slightly lower, but the difference is not significant.

Results of the compression tests are summarized in **Table 3**. Yield strength $\sigma_{0.2}$ measured in normal direction increase with deformation up to 4 ECAP passes, while yield strength measured in extrusion direction saturates at 130 MPa after 2 ECAP passes. Mechanical properties of deformed materials depend mainly on grain size, dislocation density and texture. Typical grain sizes can be inferred from EBSD orientation maps. Despite dislocation density was not yet studied for this alloy, we can rely on previous results for low-alloyed magnesium alloys [23] - dislocation density tends to increases rapidly up to 2 ECAP passes, then decreases with increasing number of additional passes and eventually saturates. This is explained by the decreasing grain size and reduced length scale for dislocation annihilation [29, 30]. Evolution of grain size and assumed evolution of dislocation density accounts for increase in hardness and strength with induced deformation up to two ECAP passes.

Table 2 Results of the microhardness measurements (Vickers method, 100 g, 10 s)

Sample	Microhardness HV0.1
Extr.	37.4 ± 2.5
1P	43.8 ± 2.6
2P	47.2 ± 1.6
4P	47.2 ± 2.1
8P	44.5 ± 2.1

Table 3 Results of the compression tests: yield strength $\sigma_{0.2}$ measured in two directions - in extrusion direction (ED) and in direction perpendicular to the extruded direction - normal direction (ND)

Sample	ND	ED
Extr.	-	55 MPa
1P	114 MPa	116 MPa
2P	137 MPa	131 MPa
4P	140 MPa	128 MPa
8P	105 MPa	130 MPa

4. CONCLUSION

This research focused on microstructure and mechanical properties of magnesium with 0.3 wt.% Zr. Studied alloy was extruded and processed by ECAP. Microstructure was first bimodal (after 1 and 2 ECAP pass) and then homogeneous and fine-grained with average grain sizes ~2 µm. Fraction of HAGBs was very high (91 %) after 8 ECAP passes. The highest microhardness was measured in the samples after 2 and 4 ECAP passes. Similar evolution was found for yield compression stress measured in normal direction. On the other hand, values of yield strength measured in extrusion direction were saturated in the samples after 2, 4 and 8 ECAP passes.

ACKNOWLEDGEMENTS

Authors acknowledge financial support by GACR 16-08963S. M. J. acknowledges financial support by ERDF project CZ.02.1.01/0.0/0.0/15003/0000485. Authors gratefully acknowledge Dr. Jan Bohlen from Helmholtz Zentrum Geesthacht who provided experimental material.

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