MICROSTRUCTURE EVOLUTION AND CORROSION RESISTANCE OF MAGNESIUM ALLOYS WN43 PROCESSED BY ECAP

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Abstract

This report shows first investigation of microstructure development and its effect on mechanical strength and corrosion resistance of non-commercial magnesium alloy WN43 processed extrusion (Ex) and equal-channel angular pressing (ECAP). Bimodal grain size distribution with high volume fraction of ultra-fine grains about 650 nm in diameter was achieved after 4 passes through ECAP. The microhardness measurement showed substantial increase of mechanical strength; however, initial corrosion attack investigated by linear polarization method was found to be more severe in the ECAPed condition. Deterioration of the initial corrosion resistance was attributed to high volume fraction of the grain boundaries and other lattice defects.

Keywords: WN43 alloy, ECAP, ultra-fine-grain microstructure, corrosion

1. INTRODUCTION

Magnesium alloys owing their unique properties as elastic modulus similar to the bones, non-toxicity, biocompatibility etc. are considered as an appropriate material for manufacturing biodegradable implants. Crucial advantage of magnesium is that magnesium is a natural element in human body and with carefully chosen alloying elements, the final implant could be harmless to the body. The most beneficial attribute of magnesium biodegradable implants would be shortened recovery time and rapid decrease of the re-operation costs. However, application of the magnesium alloys in this regard is still substantially limited due to its relatively low strength, low ductility and rapid corrosion leading to the hydrogen accumulation in the surrounding area and too rapid deterioration of mechanical properties.

In recent years, number of articles indicates a strong connection between microstructure and corrosion resistance. Corrosion is very complex process that depends on a large number of different parameters. Most of these parameters are connected with the microstructure which is affected by casting conditions and processing [1]. In this work, samples of the investigated material were processed by extrusion and ECAP. ECAP is today most utilized severe plastic deformation method for preparation of the ultra-fine-grain microstructure [2]. Repetitive processing may be conducted to achieve significant grain refinement of the microstructure [3]. The refinement of the microstructure in connection with the corrosion resistance is intensively studied, but non-conclusive yet. Higher corrosion resistance was observed after substantial grain refinement [5, 9], however, some studies also reported that the corrosion resistance decreases with decreasing grain size [4, 6].

It was repeatedly reported that addition of rare earth metals improve mechanical properties and may also have positive effect on the corrosion resistance of magnesium alloys. Currently, one of the most promising material for medical application are alloys containing yttrium and other rare earth elements generally in a form of mischmetal (WE type alloys) [7]. Some of the reports suggest that using pure rare earth elements instead of mischmetal could provide even better corrosion properties. Pure yttrium and neodymium have positive effect on the grain refinement and also corrosion resistance [7]. Therefore, for this study was chosen non-commercial magnesium alloy WN43.

2. EXPERIMENTAL METHODS AND MATERIAL

The investigated alloy WN43 was conventionally casted and afterwards processed by hot extrusion at T = 350°C with an extrusion ratio ER = 30 and a constant ram speed of 1 mm/s. Homogenization annealing at 400°C for 16h was performed prior to the extrusion. Composition of the alloy was identified by spark emission spectroscopy and the results are shown in **Table 1**. Rectangular billets were machined from the extruded bars and processed by ECAP. The processing was performed at the temperature range 290-330°C and the ram speed of 5-10 mm/min. The angle Θ between two intersecting channels and the corner angle ψ were 90° and 0°, respectively. Molybdenum disulphide grease was used as a lubricant. Four conditions of processed material were chosen for investigation - extruded (E), one pass (1P), two passes (2P) and four passes (4P) through ECAP.

Table 1 Composition of the investigation alloy WN43 (wt.%)

Y	Nd	Fe	Cu	Ni	Mg
3.46	3.53	0.0344	0.0039	0.0011	balance

The microstructure observation was performed using a scanning electron microscope (SEM) ZEISS Auriga Compact equipped with EDAX EDX detector and EBSD camera. The samples were mechanically polished using emery papers and diamond suspensions of the grain size decreasing down to 0.25 μm and afterwards electrochemically polished using Struers AC2 solution. EDX (Energy-dispersive X-ray) spectroscopy in SEM was used in order to specify composition of the secondary phases in Mg matrix.

Evolution of the mechanical strength after different number of ECAP passes was investigated by microhardness tests performed by fully automatic microhardness testing device QNESS Q10a. A Vickers indenter was used with an applied load of 0.1 kg for 10s. At least 100 indents were measured for each investigated condition.

Corrosion resistance was investigated by the linear polarization method in 0.1 M NaCl solution at the room temperature. At least three tests were performed for each condition. The samples were mechanically polished by 1200 emery paper prior to each measurement. The tests were conducted using the potentiostat AUTOLAB128N and three-electrode setup. The characteristics were measured in the potential range from 150 mV to 200 mV with respect to the open circuit potential (OCP) and constant rate of 1 mV s^{-1} after 10 min of stabilization. Additional rotation of 300 rpm was introduced to the samples in order to provide better homogeneity of the measurement.

3. RESULTS AND DISCUSSION

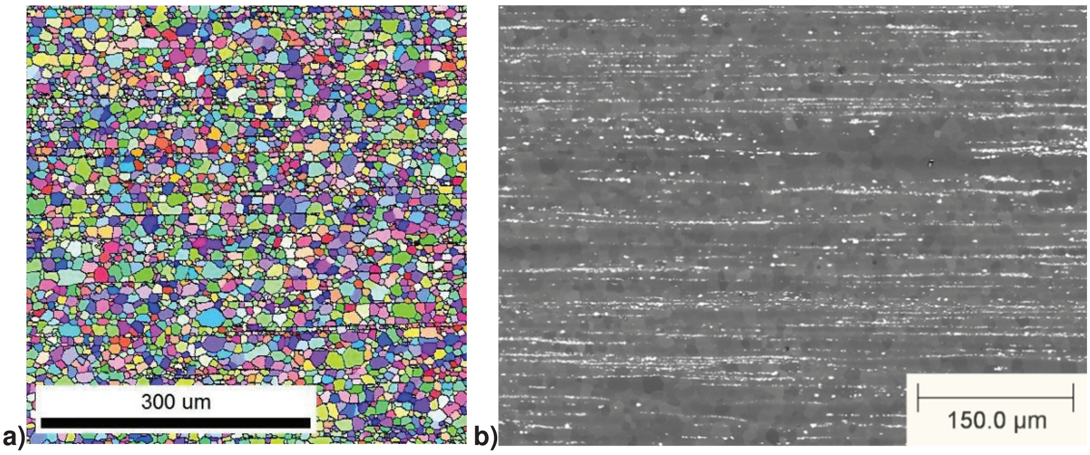

Figure 1 Microstructure of the WN43 alloy in extruded condition **a)** EBSD micrograph, **b)** secondary phase particles distribution

Microstructure of the investigated alloy in the extruded condition was studied by SEM and EBSD. Fully recrystallized microstructure with uniform character of the grain size distribution and average grain size of ~10 µm was observed, as shown in **Figure 1a).** In **Figure 1b)** is shown distribution of the secondary phases. Strong stripe-like structure along the processing direction is a result of the extrusion process. Composition of the secondary phase particles was identified by EDX in SEM. The quantitative analysis indicated that the particles are $Mg_{14}Nd_2Y$ intermetallic phase; nevertheless, precise identification in transmission electron microscope is still needed.

Effect of ECAP processing on the microstructure was investigated by EBSD and resulting micrographs are presented as **Figure 2.** After 1 ECAP pass the microstructure was highly inhomogeneous and bimodal character of the grain size distribution dominated, as shown in **Figure 2a)**. The grain size of the small grains was ~ 1 µm but size of the large grains was up to 23 µm in diameter. The area fraction of the small grains was low after the first pass, but rapidly increased after the next passes through ECAP. After 4 passes, submicron grains dominated the microstructure. Average grain size of the small grains was ~ 650 nm, but presence of much bigger grains with grain size up to 7 µm was still apparent, as shown **Figure 2c).**

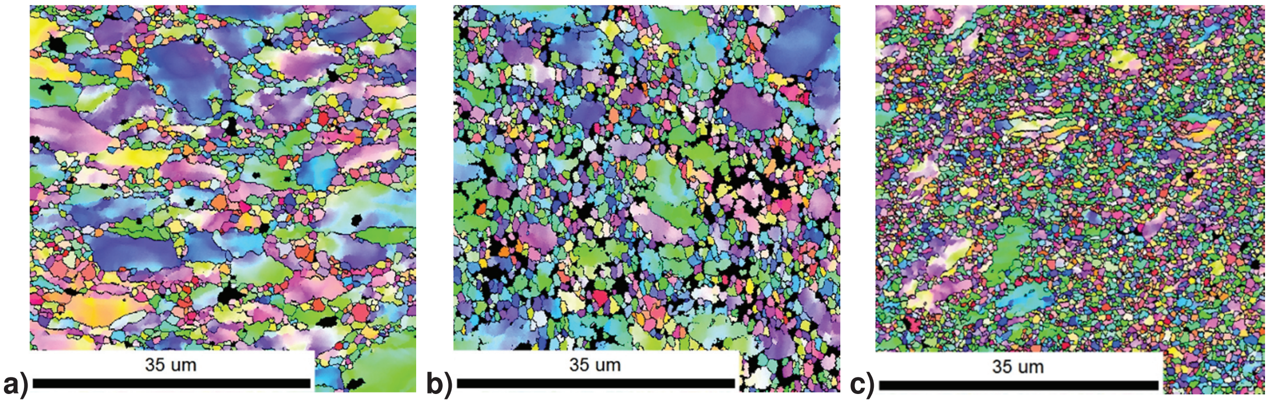

Figure 2 EBSD micrographs of WN43 processed by ECAP **a)** 1P, **b)** 2P, **c)** 4P

Effect of ECAP processing on the mechanical properties was measured by the microhardness test. The first results showed a homogeneous distribution of the microhardness in the cross-section of all investigated conditions. The average values of the microhardness as a function of the increasing number of ECAP passes are presented in **Figure 3.** Mechanical strength of the alloy increased rapidly with the increasing number of ECAP passes. This is result of the substantial grain refinement of the material, which is typical in magnesium alloys processed by ECAP [8].

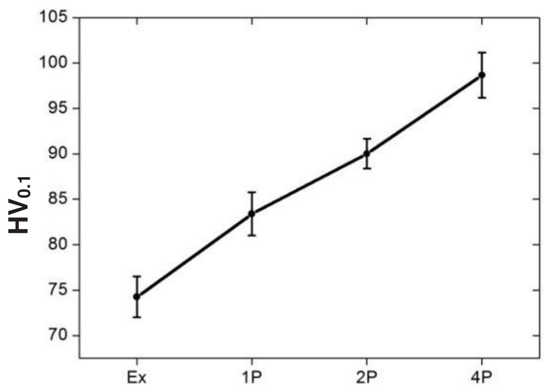

Figure 3 Microhardness as function of the increasing number of ECAP passes

Effect of ECAP on the initial corrosion attack was studied by linear polarization method after 10 minutes of immersion in 0.1M NaCl solution. The resulting values of the polarization resistance (R_p) are presented in **Figure 4.** It is shown that corrosion resistance of the WN43 alloy deteriorates with increasing number of ECAP passes. However, increase of the corrosion resistance was previously observed in different Mg alloys processed by ECAP [5, 9]. Moreover, it was reported increase of the corrosion resistance of the commercial WE43 after substantial grain refinement [10]. Increase of the corrosion resistance after ECAP was attributed to the grain refinement together with better distribution of the alloying elements present in secondary phases. Better distribution of alloying elements with positive effect on the corrosion layer stability could overwhelm negative effect of the high volume fraction of the lattice defects introduced during the processing, resulting in increase of the overall corrosion resistance [8, 9]. Zirconium present in the commercial WE43 alloy (less than 2%) was reported to have positive effect on the corrosion layer stability [6]. Therefore, its absence in the investigated alloy could have led to decrease of the corrosion resistance in the ultra-fine grained condition. Nevertheless, there is a report showing, that material processed by ECAP suffers from severe residual stress and corrosion resistance could be substantially increased after complete dynamic recrystallization when sufficient number of ECAP passes is applied [12]. Therefore, additional investigation is needed, in order to reveal effect of the grain refinement alloying elements and possible residual stress on the corrosion resistance of the investigated alloy processed by ECAP.

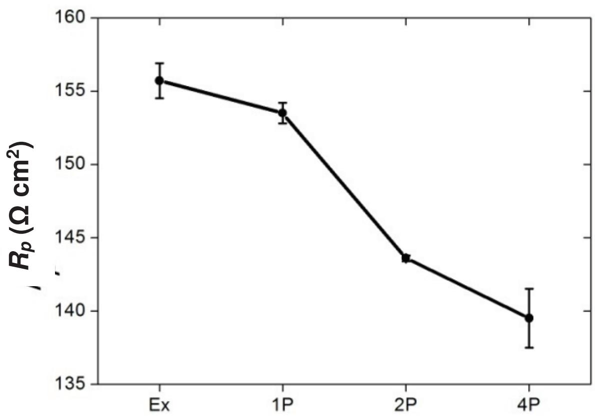

Figure 4 Polarization resistance R_p as a function of the increasing number of ECAP passes

4. CONCLUSION

This work is a report of the first examination of microstructure development and its effect on mechanical strength and corrosion resistance of the magnesium alloy WN43 processed by ECAP. The grain size distribution after four passes through ECAP was still bimodal but submicron grains dominated the microstructure. Evolution of the microhardness showed increasing tendency with increasing number of ECAP passes, which is effect of substantial grain refinement. Polarization resistance after 10 minutes of stabilization decreased with increasing number of ECAP passes. This is the most probably caused by the grain refinement, which resulted in substantial increase of lattice defects volume fraction.

ACKNOWLEDGEMENTS

The present work is a part of the Czech Science Foundation project 14-13415S. One of the authors (M.Z.) acknowledges financial support by the Charles University Grant Agency under the grant 250333/2016.

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