MECHANICAL PROPERTIES AND MICROSTRUCTURE OF ULTRAFINE-GRAINED MAGNESIUM ALLOYS CONTAINING NEODYMIUM AND ZINC

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Abstract

Two experimental magnesium alloys containing neodymium and zinc (Mg-5Nd-1Zn and Mg-1Nd-1Zn) were processed by severe plastic deformation (SPD) method - equal channel angular pressing (ECAP). The effect of SPD processing on the microstructure was studied by light microscopy (LM) and scanning electron microscopy (SEM) including electron backscatter diffraction (EBSD). A significant grain refinement was observed after 8 passes of ECAP in both alloys resulting in homogenous ultra-fine grained condition. Microstructure evolution significant affected mechanical properties, which were studied by microhardness measurements and compression deformation tests. Both yield compression strength σ0.2 and microhardness significantly increased after ECAP when compared to the extruded or as-cast counterparts.

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

1. INTRODUCTION

Magnesium (Mg) is metal with the lowest density (ρ = 1740 kg.m^{-3}) among structural metals resulting in high specific strength, which can be utilized for weight saving in automotive industry and aerospace applications. [1]. Disadvantages include low Young's modulus, high chemical affinity and low corrosion resistance [2].

Materials used in aircraft manufacturing industry must meet strict technical standards. Recently, magnesium alloys were generally allowed for construction of passenger and crew seats in transport aircrafts by legally binding SAE Aerospace Standard (AS) 8049C [3]. However, a magnesium alloy must pass defined flammability test. Two magnesium alloys - Elektron21 and WE43 successfully passed this specifically designed test, while the most common AZ31 alloy failed [4]. Lifting the ban on Mg-based alloys in aircraft industry creates a new stimulus for developing non-flammable (ignition-proof) high strength magnesium alloys.

Generally, elements that have high solubility limit in magnesium matrix and form only thermally stable intermetallic phases are considered as good candidates for materials with high strength and increased ignition temperature [5]. It was shown that Nd and Gd cause an increase of the ignition temperature in binary Mg alloys due to the existence of Nd_2O_3 and Gd_2O_3 surface oxides [6,7,8].

Mechanical properties of polycrystalline materials depend on many parameters including the grain size. The relation between the strength and the grain size is given by Hall-Petch equation [9,10]:

$$\sigma_{0.2} = \sigma_0 + k_y d^{-\frac{1}{2}}, \quad (1)$$

where $\sigma_{0.2}$ is the yield strength (MPa), σ_0 is material constant for the starting strength for dislocation movement (MPa), k_y is the strengthening coefficient (MPa.m$^{1/2}$) and finally d is the average grain size (m). From equation

(1) it follows, that the ultrafine-grained (UFG) materials have increased strength when compared to the coarse-grained ones. UFG materials can be produced by severe plastic deformation (SPD) techniques. The most frequently used current techniques of SPD are equal channel angular pressing (ECAP) [11], high pressure torsion (HPT) [12-15], accumulative roll-bonding (ARB) [16], twist extrusion [17] or multi-directional forging [18]. ECAP is widely used for its simplicity and good results [19].

In the present study, commercial magnesium alloy ZN11 alloy (1 wt% of Zn and 1 wt% of Nd) and newly developed alloy NZ51 with higher content of Nd (1 wt% of Zn and 5 wt% of Nd) were studied. These alloys were processed by ECAP to achieve better mechanical properties especially increased strength.

2. EXPERIMENTAL MATERIALS AND PROCEDURES

ZN11 alloy (1 wt% of zinc and 1 wt% of neodymium) was extruded at 400 °C with the extrusion ratio of 30 (ZN11 E). NZ51 alloy (1 wt. % of zinc and 5 wt. % of neodymium) was in as-cast state (NZ51 C).

Both alloys were processed by ECAP with the die with channels intersecting with the angle of 90° using route B_C pressing speeds of 3-10 mm.min^{-1} (increasing with the number of passes) [20]. Two billets were produced for each alloy - after one pass (1P) and after 8 passes (8P). ZN11 alloy was processed by ECAP at temperatures 250 °C (last pass) to 350 °C (first pass) and NZ51 at 320 °C (last pass) to 355 °C (first pass). Microstructure was investigated by scanning electron microscope (SEM) including EBSD and light microscope (LM). Mechanical properties were studied using microhardness measurement and compression tests. Microhardness was measured using Vickers hardness method with the load of 0.5 kgf (HV 0.5) for loading time of 10 s. More than 100 experimental points (indents) were evaluated for each specimen. Compression tests were performed on cuboid specimens with aspect ratio 3:2 (4 mm x 4 mm x 6 mm).

3. RESULTS AND DISCUSSION

3.1. Microstructure

The microstructure of ZN11 alloy was observed by LM in a plane perpendicular to the extrusion direction (ED) and is shown in **Figure 1**. Microstructure of extruded state is homogenous with average grain size 5 - 10 μm in diameter. After ECAP, microstructure is more refined and therefore EBSD was utilized for observations. Inverse pole figure (IPF) maps of ZN11 alloy after 1 and 8 passes (1P and 8P) are shown in **Figure 2**. Microstructure of ZN11 1P is bimodal - large grains (~20 μm in diameter) are surrounded by small grains (~3 μm in diameter). Specimen after 8 passes is almost homogenous and contains grains about 1 μm in diameter.

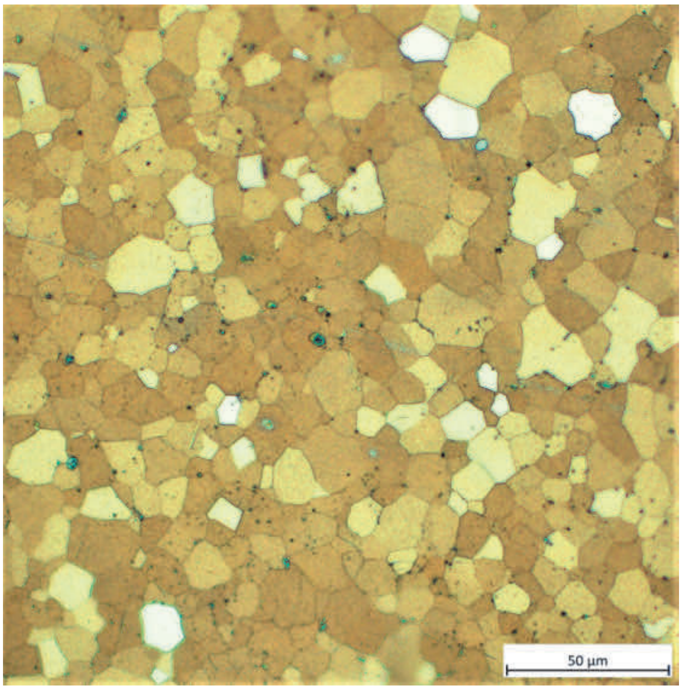

Figure 1 Microstructure of ZN11 E (extruded state)

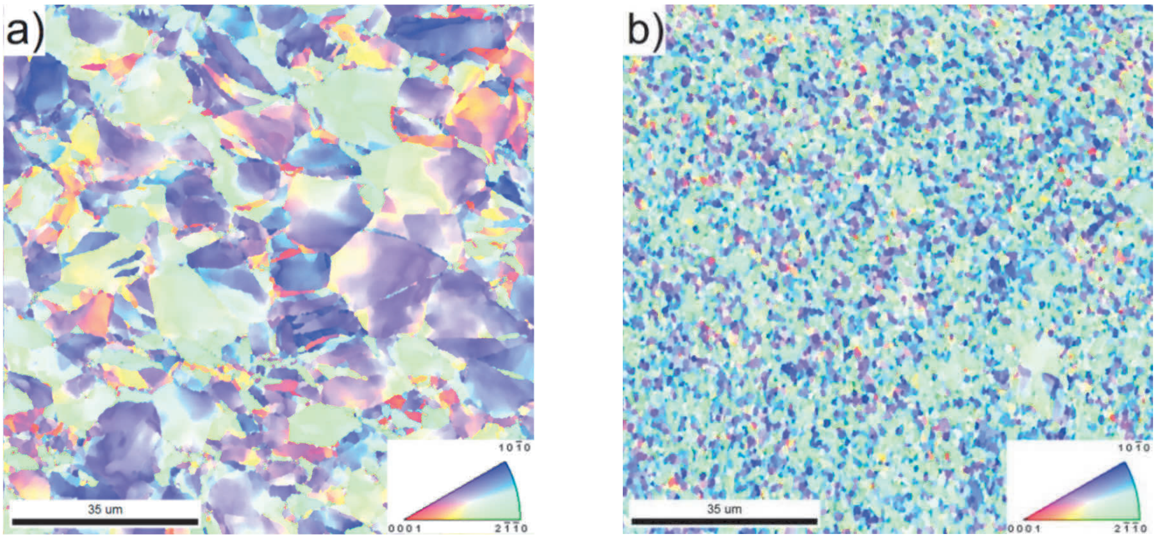

Figure 2 IPF maps of ZN11 a) 1P, 2) 8P

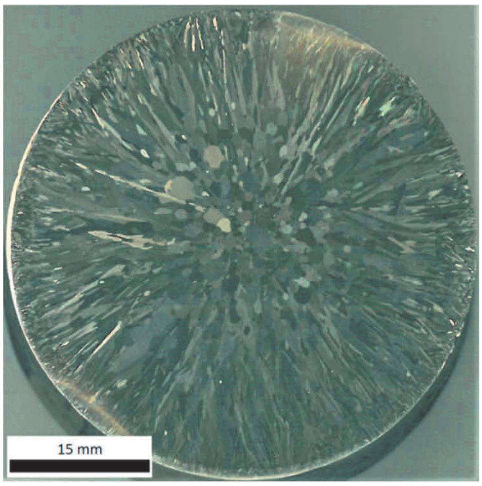

Figure 3 Microstructure of NZ51 (cast state)

The microstructure of NZ51 is shown in **Figure 3**. The cast material was produced in the shape of cylinder. It is clearly observed in **Figure 3** that the microstructure is not homogenous - grains in the centre of cylinder are equiaxed while grains near the edge of the sample are elongated in the radial direction. Some of the grains are larger than 1 mm in diameter. After one ECAP pass (1P), many large grains remained in the specimen, but they were significantly deformed and some small grains formed around them (**Figure 4a**). The microstructure of NZ51 8P (**Figure 4b**) is similar to the case of ZN11 8P. Microstructure is fine grained and almost homogenous with the grain size about 1 µm in diameter.

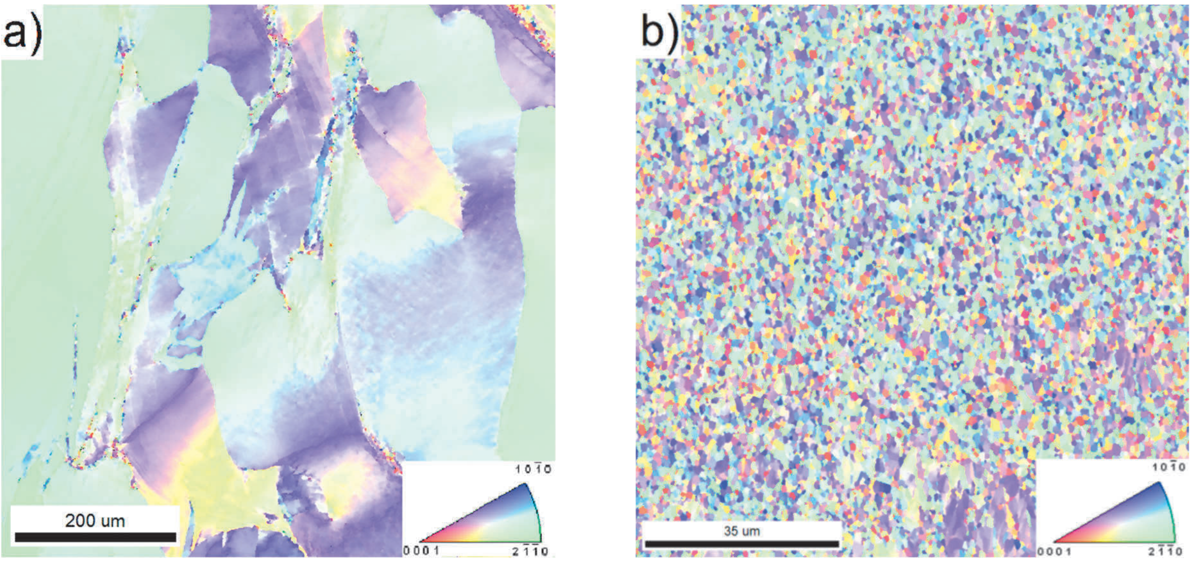

Figure 4 IPF maps of NZ51 a) 1P, b) 8P

EBSD observations of microstructure can be used for determination of average grain size and fraction of high-angle grain boundaries (HAGB). Achieved data are shown in **Table 1**. The fraction of HAGB significantly increased with the ECAP processing and reached for than 80 % for the 8P states. This suggests matured, homogeneous UFG structure, with equilibrium grain size for given processing temperature and speed.

Table 1 Average grain sizes and fraction of high-angle grain boundaries (HAGB, missorientation angle > 15°) measured from EBSD

Sample	**Average grain size** (µm)	**Fraction of HAGB** (%)
ZN11 1P	8.6 ± 6.2	51
ZN11 8P	1.1 ± 0.6	84
NZ51 1P	520 ± 260	38
NZ51 8P	1.2 ± 0.7	83

3.2. Mechanical properties

Compression samples of ZN11 alloy were oriented such that the loading direction was parallel to the extrusion direction. In the case of NZ51 alloy, the material was taken from the centre of the as-cast cylinder and the loading direction was parallel to the axis of the as-cast cylinder.

Yield strength $\sigma_{0.2}$ and compressive strength σ_P were evaluated from the deformation curves and achieved values are shown in **Table 2**. Yield strength of ZN11 increased dramatically after one pass of ECAP (ZN11 1P) and further after 8 passes. After 8 ECAP passes, yield strength was more than two times higher than for the extruded state (96 vs. 236 MPa). Compressive strength also increased, but not so significantly (from 268 to 351 MPa). Mechanical properties of NZ51 were also enhanced after one pass of ECAP, but the increase was not so large, which can be attributed to the limited refinement after 1P in NZ51 alloy. The differences between ZN11 1P and NZ51 1P can be explained by different initial states - the microstructural of NZ51 C was much coarser than the extruded one (ZN11 E). On the other hand, $\sigma_{0.2}$ and σ_P are similar for both alloys in the case of samples after 8 ECAP passes. This is fully attributed to the equivalent microstructure consisting of small equiaxed grains.

Table 2 Yield strength $\sigma_{0.2}$ and compressive strength σ_P of experimental materials

Sample	$\boldsymbol{\sigma_{0.2}}$ (MPa)	$\boldsymbol{\sigma_P}$ (MPa)
ZN11 E	96 ± 5	268 ± 9
ZN11 1P	173 ± 6	301 ± 9
ZN11 8P	236 ± 7	351 ± 16
NZ51 C	67 ± 7	174 ± 7
NZ51 1P	98 ± 6	268 ± 10
NZ51 8P	248 ± 7	354 ± 15

The results of microhardness measurement are summarized in **Table 3**. Microhardness was not measured on NZ51 C due to its large grains. As you can see in **Table 3**, the results of microhardness has very similar upward trend like the compressive strength for both alloys.

Table 3 Microhardness results

Sample	ZN11 E	ZN11 1P	ZN11 8P	NZ51 1P	NZ51 8P
Microhardness HV 0.5	48 ± 1	64 ± 2	75 ± 2	55 ± 3	79 ± 2

4. CONCLUSION

The effect of severe plastic deformation by ECAP on microstructure of magnesium alloys containing neodymium and zinc (ZN11 E and NZ51 C) was studied. Initial extruded state of ZN11 E has homogenous microstructure with average size of grains of approx. 20 μm. The microstructure after one pass (ZN11 1P) was bimodal with large initial grains from extrusion (~20 μm) and newly formed small (~3 μm) grains. The microstructure of NZ51 C was very coarse and non-homogenous. One pass of ECAP led to deformed structure of NZ51 alloy without significant fragmentation. After 8 ECAP passes, the microstructure of both alloys was significantly refined with average grain size 1 μm, homogeneous and containing more than 80 % of HAGB. Grain refinement led to improvement of mechanical properties - yield strength, compressive strength and microhardness. It is concluded that ECAP processing of ZN11 and NZ51 magnesium alloys was successful in terms of microstructural refinement and improvement of strength.

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