CHARACTERIZATION OF MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MAGNESIUM ALLOYS WITH NEODYMIUM AND YTTRIUM PROCESSED BY EXTRUSION

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Abstract

Intensive efforts have been recently made for a production and a processing of Mg-based light structural alloys, which would provide a utilization in the automotive and aerospace engineering, medical implants and equipment, power generation system, etc. In this study, three magnesium alloys containing neodymium and/or yttrium were investigated in an extruded condition. Microstructure of the extruded samples was investigated by scanning electron microscope including EBSD and X-ray diffraction. Mechanical behaviour of the alloys was studied by compression deformation tests in the extrusion direction (ED) and transverse direction (TD). Acoustic emission technique was concurrently applied to investigate activity of particular deformation mechanisms. The highest mechanical strength was found in the WN43 alloy, which contained the highest amount of rare earths. On the other hand, no difference in a mechanical strength between N2 and W4 alloy was observed. This resulted from lower grain size and higher fraction of precipitates found in the N2 samples. Different shape of the deformation curves between ED and TD was observed in two alloys containing neodymium. This behaviour corresponded to different texture formation and was attributed to an increased activation of twinning in one deformation direction, which was proved by acoustic emission measurement.

Keywords: Magnesium alloys, rare earths, mechanical properties, texture, twinning

1. INTRODUCTION

Magnesium and its alloys became very attractive material because of its low density and superior strength to weight ratio. A major interest comes from an automotive industry, in which weight reduction have strong impact on economic and environmental factors [1]. Magnesium is also biocompatibile and biodegradabile metal, what makes it very promising material for use in medicine. Today, the research in this regard is primarily focused on temporary implants [2].

Applicability of magnesium alloys continuously grows and there is a constant need for further improvement of the material properties. It can be done by variation of the alloys' composition and processing method. Extrusion and rolling are typical processing techniques for magnesium alloys. Materials processed by both techniques have usually much finer grains compared to as-cast condition, and therefore is stronger. However, strong texture is formed at the same time [3]. The latest reports showed, that this undesired texture could be effectively suppressed by addition of rare earths [3-5]. Grain orientation randomization is substantially affected by the amount of rare earths in the alloy. In [5] it is shown that with increasing amount of yttrium in ZM31-based alloy, the texture became significantly weaker. On the other hand, rare earths as the alloying elements could also effectively strengthen the final material [6-9]. Precipitation hardening is a typical strengthening mechanism of these alloys. Therefore, the mechanical properties could be substantially improved by a proper combination of magnesium alloy and processing technique. In this study, three magnesium alloys containing neodymium and/or yttrium were investigated. All three alloys were processed by extrusion. The effect of particular alloying elements on the microstructure and mechanical properties is addressed in the paper.

2. EXPERIMENTAL METHODS AND MATERIALS

Three magnesium alloys N2 (Mg - 2 wt.% Nd), W4 (Mg - 4 wt.% Y) and WN43 (Mg - 4 wt.% Y - 3 wt.% Nd) were conventionally casted and subsequently processed by hot extrusion. The extrusion parameters were: *T* = 350 °C, *ER* = 30 and a constant ram speed = 1 mm/s. Homogenization annealing was conducted prior to the extrusion for 16 h at 400 °C for - W4, WN43 and 550 °C for - N2 alloys, respectively. Composition of the alloys was identified by a spark emission spectroscopy and the results are shown in **Table 1**.

Table 1 Composition of the investigated magnesium alloys (wt.%)

	Y	Nd	Fe	Cu	Ni	Mg
N2	-	2.43	0.0297	0.0016	0.0014	balance
W4	3.33	-	0.0316	0.0037	0.0002	balance
WN43	3.46	3.53	0.0344	0.0039	0.0011	balance

The observation of microstructure was performed using a scanning electron microscope (SEM) ZEISS Auriga Compact equipped with EDAX 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. Texture of the extruded material was measured by an X-ray PANalytical XPert MRD diffractometer with CuKα radiation. Full pole figures were calculated using MTEX software [10].

Mechanical properties were investigated by compression deformation tests performed by INSTRON 5882 deformation machine. The compression tests were carried out in two directions - in extrusion direction (ED) and transverse direction (TD). All tests were performed at the room temperature using a constant deformation rate $1x10^{-3}\ s^{-1}$. For deformation tests, samples of $5x5x8\ mm^3$ were cut out from the billets. At least three samples were tested for each geometry test.

During deformation tests an acoustic emission (AE) was detected. The AE signal was acquired using a miniaturized MST8S (Dakel-ZD Rpety, Czech Republic) piezoelectric transducer (Ø 6 mm). A preamplifier with a gain of 40 dB was used. The set of AE parameters were obtained by threshold level detection, threshold level was set to 27 dB. The AE activity were represented by the AE count rate, which is the count number per time unit at a given threshold voltage level.

3. RESULTS AND DISCUSSION

3.1. Microstructure

Microstructure of the extruded samples was studied by SEM and EBSD. Fully recrystallized microstructure with uniform character of a grain size distribution and high angle grain boundaries was observed in all three alloys, see **Figure 1**. The average grain size of all three alloys was in range of mm after the homogenization annealing (not shown here). Extrusion led to substantial grain refinement with highest degree in case of WN43 (~10 μm) and lowest in case of W4 (~20 μm). Average grain size of the extruded N2 alloy was ~15 μm. Contrary to the substantially higher amount of alloying element in W4 compared to N2 alloy, grain refinement was more intensive in the latter one. This difference stems from different distribution of the precipitates in the matrix. **Figure 2** shows secondary phase distribution in all three alloys after the extrusion. Precipitates in the N2 alloy are located primarily at the grain boundaries, and therefore they positively affect stability of the grain structure. On the other hand, such precipitates were not found in the W4 alloy, only secondary phase particles which are aligned in stripes along the extrusion direction. It could be suspected that they were present in the matrix prior to the extrusion. Therefore, most of the yttrium atoms was dissolved in the matrix. Microstructure of the WN43 alloy presents combination of microstructure of both binary alloys. Stripes of the undissolved particles were observed along with fine precipitates in the grain boundaries. Presence of the particles in W4 and WN43 alloy, which were not dissolved during homogenization annealing, indicates that the annealing temperature should be increased.

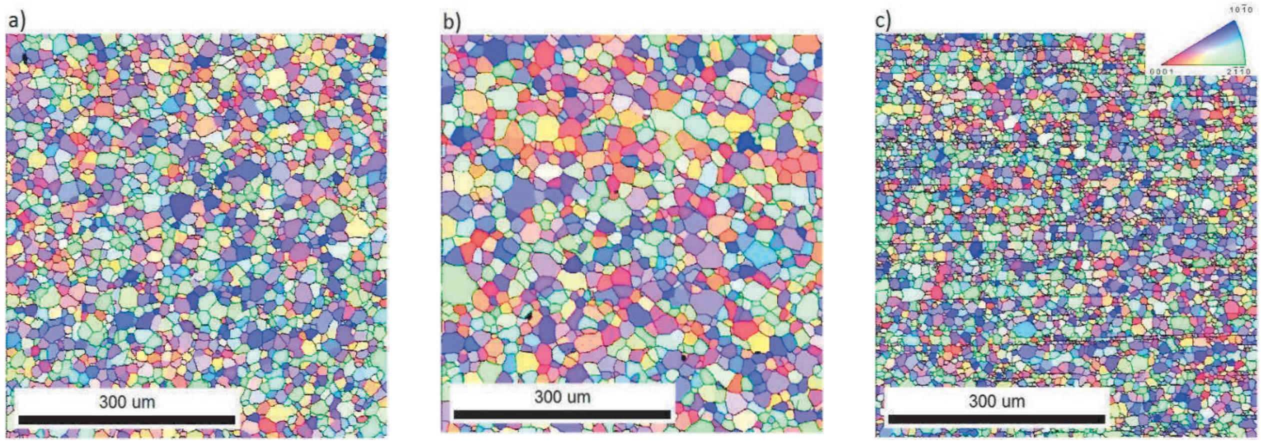

Figure 1 EBSD micrographs of **a)** N2, **b)** W4 and **c)** WN43

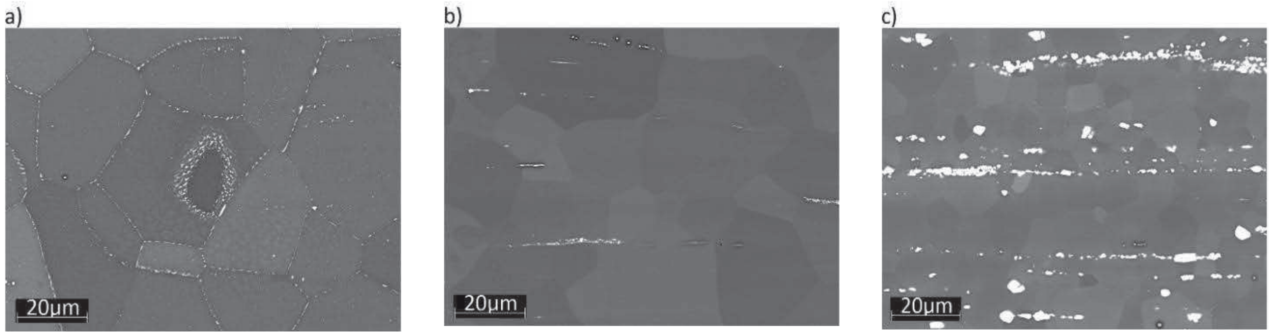

Figure 2 Secondary phase particles distribution in **a)** N2, **b)** W4 and **c)** WN43

3.2. Mechanical Properties

The mechanical properties of all three alloys were investigated in ED and TD direction during compression deformation tests. The resulting deformation curves are shown in **Figure 3** and the calculated values of yield compression strength (YCS) are shown in **Table 2**. Both ED and TD directions show similar values of YCS for N2 and W4. Overall similarity in the values of YCS for N2 and W4 was observed regardless of the different grain size and amount of precipitates. Therefore, solute solution hardening in cased of W4, as a strengthening factor, balanced negative effect of higher average grain size and lower amount of precipitates, when compared to the N2 alloy. In case of WN43, amount of particles in the material together with lower grain size resulted in substantial increase of YCS compared to both binary alloys. Beside overall improvement of the mechanical strength, higher YCS was measured in TD compared to ED. This difference could be explained by a higher number of particles aligned in the stripes along the extrusion direction.

Table 2 Yield compression strength measured in the ED and TD direction

	$\sigma_{0.2}$ (MPa)	
	ED	TD
N2	109 ± 1	110 ± 1
W4	105 ± 1	110 ± 5
WN43	146 ± 1	166 ± 2

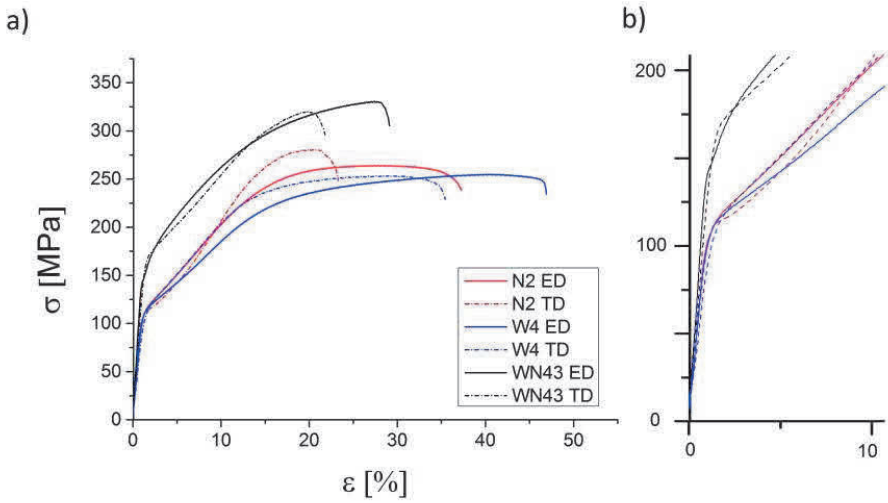

Figure 3 a) The true compression stress versus true strain curves of all Mg alloys in ED and TD direction, **b)** early stage of deformation presented in **a)**

Detail of the deformation curves for N2 and WN43 alloys, reveals difference in their shape for ED and TD in an early stage of the plastic deformation. However, no difference in the shape of the deformation curves for W4 was observed. Samples deformed along TD exhibited S-shape character of the deformation curve. It can be the result of twinning activity. A combination of the texture measurements and acoustic emission response, detected concurrently with the deformation tests, can help to analyse twinning activity. The measured (0001) and (10-10) pole figures for all three investigated alloys are shown in **Figure 4**.

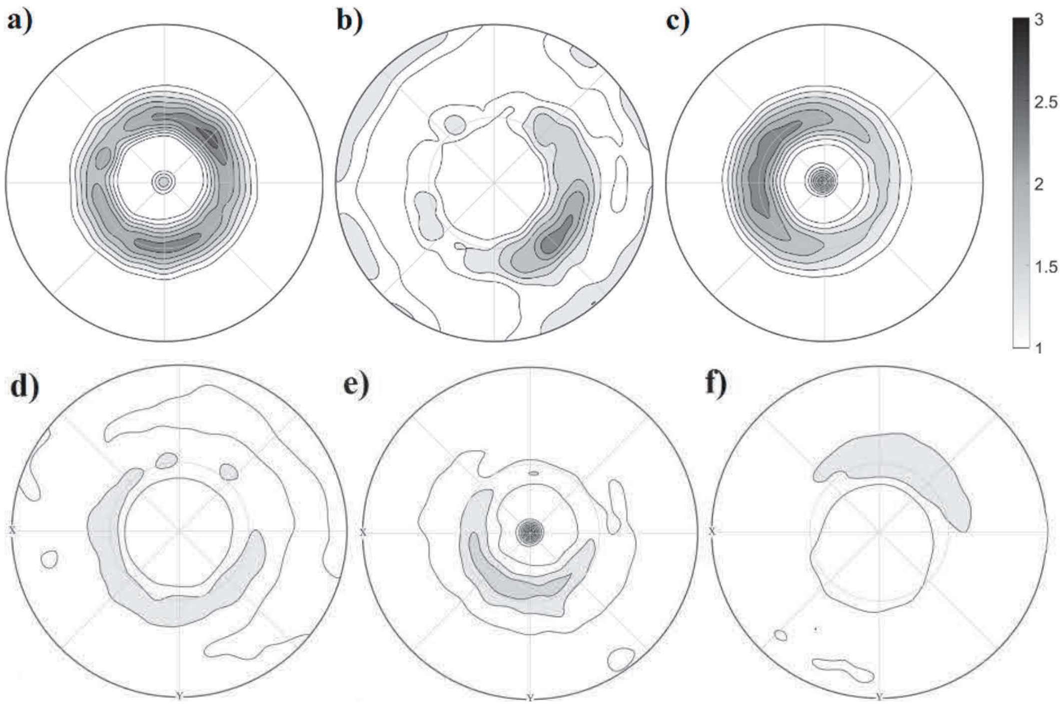

Figure 4 (0001) X-ray pole figure of a) N2, b) W4, and c) WN43 (plane perpendicular to ED) and (10-10) pole figure of d) N2, e) W4, f) WN43

It is evident that the typical "rare earth texture" was formed in all three alloys during extrusion. This texture type is formed by basal planes rotated ~45° from the extrusion direction symmetrically around ED [7]. Grains characterized by this texture element are well oriented for basal slip activation in both ED and TD directions. Therefore, difference of the deformation curve character doesn't stem from these grains. Nevertheless, in N2 and WN43 alloy, additional texture element in the centre of the (0001) PF formed during the extrusion process. It is well-known that the most popular tensile twins are activated in Mg when load is applied perpendicular to the c-axis [11]. Therefore, grains characterized by basal planes perpendicular to the ED, i.e. c-axis parallel to ED, are well oriented for activation of tensile twinning, preferentially during compression along TD. The overall texture is very weak in all three alloys, and volume fraction of grains represented by this texture element is low. Apparently, existence of this preferred orientation doesn't affect the yield point, but causes difference in the deformation curve character between ED and TD of the N2 and WN43 alloy. Preferential activation of the twinning during deformation along TD comparing to ED is well represented by the acoustic emission response, see **Figure 5**. In case of the deformation along ED, there is a substantial increase and sharp decrease of the AE signal around the yield point. In case of the deformation along TD, the AE signal is more pronounced and continues also during the first stage of the plastic deformation. AE technique is very sensitive to twinning and AE signal around YCS could be correlated to the twin nucleation.

Origin of the texture element in the centre of the (0001) PF is still under investigation. Nevertheless, these observations revealed that it is closely related to presence of neodymium in the alloy. This element was observed in both alloys containing neodymium, but not in the one containing yttrium. On the other hand, in a previous report regarding ZN11 alloy [8], this texture element was not observed. Therefore further investigation is still needed.

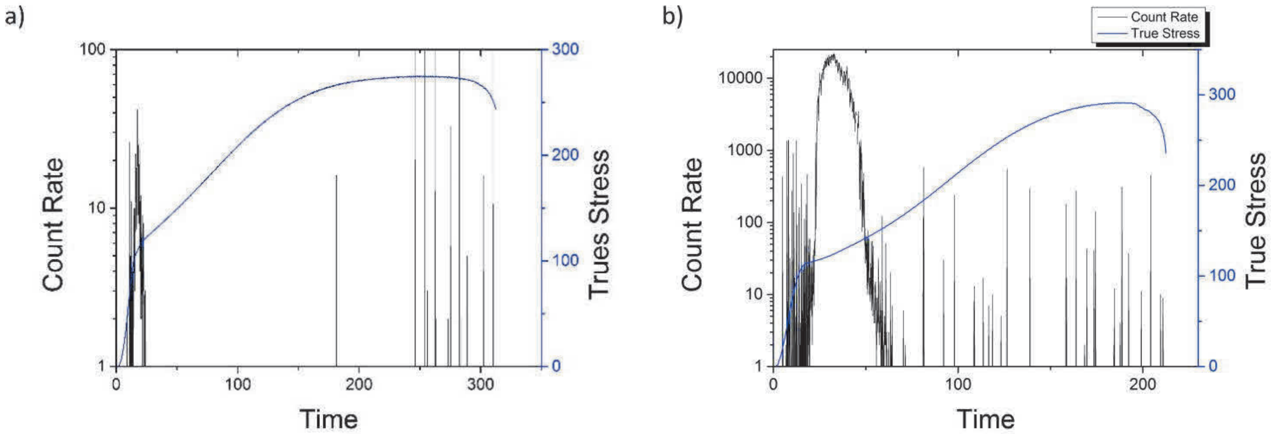

Figure 5 Compression test of N2 sample along the **a)** ED and **b)** TD with AE response

4. CONCLUSION

Microstructure and deformation behaviour was investigated in magnesium alloys: N2, W4 and WN43. Compression deformation tests were conducted in the extrusion and transverse direction. The Following conclusions can be drawn:

- Significant grain refinement was observed in all three alloys as a result of extrusion. Highest average grain size was measured for W4 and lowest for WN43 alloy, respectively.
- Weak texture with a rare earth character was measured in all three alloys. Additionally, texture element in the centre of (0001) pole figure was observed in the N2 and WN43 alloy, but not in the W4 alloy.
- Mechanical properties of the alloys corresponded to the microstructure. Highest strength was measured in WN43 alloy, which had lowest grain size and highest volume fraction of secondary phase particles.
- Specific texture of N2 and WN43 alloys resulted in activation of twinning during compression along TD. It is supported by S-shape of the deformation curve and pronounced acoustic emission activity around CYS.

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