EVOLUTION OF COMPRESSION STRAIN AND MICROSTRUCTURE OF Mg-Nd AND Mg-Y ALLOYS PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING

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Abstract

Two binary alloys Mg-3Nd (N3) and Mg-3Y (W3) processed by hot extrusion and equal channel angular pressing (ECAP) were studied. Microstructure after extrusion and a different number of ECAP passes was investigated by scanning and transmission electron microscope. The experimental results showed significant grain refinement in both alloys and massive precipitation in the N3 alloy. Compression deformation tests were conducted in two directions - along with processing direction and transverse direction. A mechanical strength of both alloys was considerably increased (~300%) by ECAP processing due to several factors. Positive effect on the strength had particularly grain boundary hardening, solid solution hardening in the case of W3 and precipitation hardening in the case of N3. Additionally, rather weak texture, which was formed during ECAP in both alloys, resulted in systematically higher strength along the processing direction compared to the transverse direction in almost all samples processed by ECAP.

Keywords: Magnesium alloys, rare earth elements, ECAP, microstructure, mechanical properties

1. INTRODUCTION

Magnesium alloys have a great potential to be used as structural materials in larger scale due to their low specific weight, high specific strength and good recycling potential. Since magnesium alloys exhibit low ductility and poor cold working abilities, magnesium-based parts are frequently produced by casting without further mechanical processing. However, as-cast magnesium alloys have relatively low strength, which often limits their further application in industry.

In order to optimise Mg alloys, extensive research has been done to understand the role of individual element additions. It was found that rare earth elements (RE) could be used to improve mechanical properties by solid solution hardening as well as by precipitation hardening [1-3]. Moreover, the precipitates are stable even at elevated temperature, what promotes high-temperature applications [4]. Additionally, solid solution hardening could be exploited particularly by yttrium addition [5]. Other important RE element, neodymium, have a significantly positive effect on creep resistance by the combination of precipitation [6]. Today, part of the most commercially successful Mg alloys belongs to WE series containing yttrium and neodymium. This type of alloys offers good corrosion resistance together with good mechanical properties at elevated temperature.

It is well known that grain refinement is another effective option to enhance the strength of Mg alloys. It has been demonstrated that severe plastic deformation techniques, such as equal channel angular pressing (ECAP), could effectively modify the microstructure of Mg-RE alloys [7,8]. By grain refinement, Mg alloys with excellent mechanical properties and good corrosion resistance could be prepared. Consequently, such materials have great potential not only in structural application but also in a field of biomedicine [9]. In addition, RE containing magnesium alloys exhibit weak texture even after ECAP, what is beneficial for mechanical properties of the processed materials [8].

However, the literature is usually concentrated on the mechanical properties improvement of the as-cast alloys by a combination of more RE elements or by ageing. On the other hand, the present paper is focused on the effect of individual REs (Nd and Y) on the microstructure and mechanical properties of binary magnesium alloys prepared by hot extrusion and ECAP.

2. MATERIAL AND EXPERIMENTS

Magnesium alloys with addition of neodymium (Mg-3Nd; N3) and yttrium (Mg-3Y; W3) were prepared. The cast billets were homogenized for 16 h at 400 °C for W3 and 550 °C for N3 alloy and then quenched in water. Both alloys were extruded with the ram speed of 1 mm/s and an extrusion temperature of 350 °C and subsequently processed by ECAP. The ECAP processing was performed up to eight passes (8P) for both alloys. Samples after one pass (1P), two passes (2P), four passes (4P) and eight passes (8P) were prepared following route B_C [10]. This route is considered to be the most effective one in order to achieve the uniform fine-grain microstructure [11]. The ECAP processing was performed at the temperature range of 285-335 °C and the ram speed of 5-10 mm/min with an internal angle of $\Theta = 90°$ and corner angle $\psi = 0°$.

Microstructure of all samples was characterized by scanning electron microscope (SEM) ZEISS Auriga Compact equipped with electron backscattered diffraction (EBSD) camera. Samples for microstructure observations were mechanically polished with decreasing grain size down to 50 nm. Additional ion-polishing was used on samples for EBSD by Leica EM RES102.

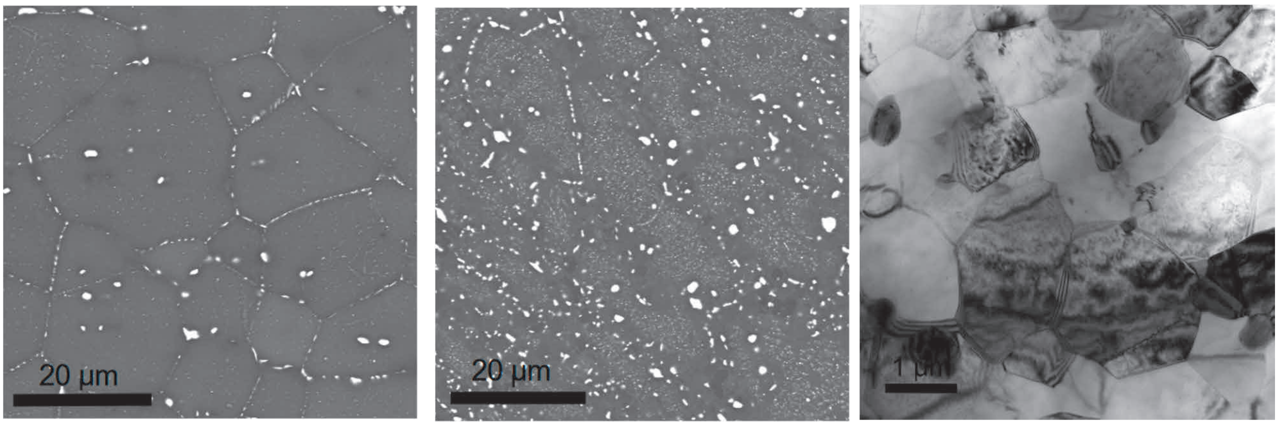
Compression deformation tests at ambient temperature were performed by Instron 5880 with an initial strain rate of 10^{-3} s^{-1}. Deformation tests were carried out in two directions - along with processing direction (X) and transverse direction (Y). Samples with a dimension of 4 x 4 x 5 mm^3 were cut from the billets. At least three measurements were performed for each investigated condition.

3. RESULTS AND DISCUSSION

3.1. Microstructure

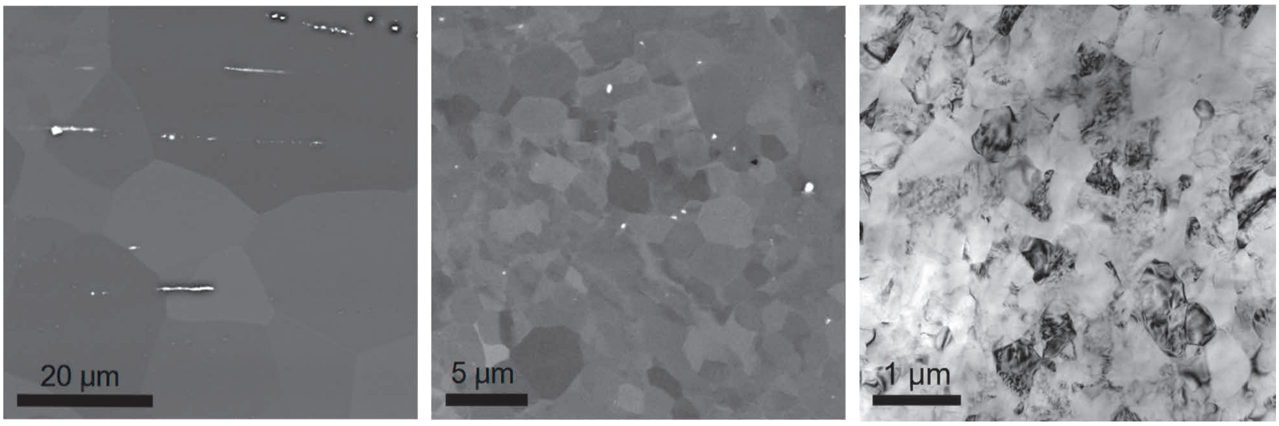
The microstructure of initial extruded (Ex) materials was formed by grains with a uniform character of a grain size distribution and a high fraction of high angle boundaries. The characteristic microstructure of both extruded alloys can be seen in **Figure 1a)** and **Figure 2a)**. The average grain size of the extruded N3 and W3 alloys was ~15 μm and ~20 μm, respectively. Contrary to the significantly higher amount of alloying element in the W3 alloy (atomic percent), grain refinement during extrusion was more intensive in the N3 alloy. As reported in our previous paper [12], this difference occurred due to the severe precipitation in the N3 alloy, which occurred during extrusion and intensified dynamic recrystallization. Only secondary phase particles observed in the W3 alloy were large ones aligned in the stripes parallel with the extrusion direction. Therefore, they could be considered as undissolved leftovers from homogenization annealing (mostly oxides) and unlike in the N3 alloy, no precipitation occurred during extrusion.

Figure 1b) and **Figure 2b)** illustrate microstructure of the alloys after one pass through ECAP. The microstructure exhibits a significant change of the grain size and distribution of secondary phase particles, particularly in the N3 alloy. The microstructure of both alloys was inhomogeneous with the areas of coarse grains surrounded by refined ones. After 4 passes through ECAP, microstructure was still inhomogeneous (not shown here). The mixture of areas with ultra-fine grains and grains with several microns in diameter were found in both alloys. Uniform grain size distribution with equiaxed grains was finally achieved in the 8P samples. The microstructure of both alloys after 8 passes through ECAP was investigated by TEM and the corresponding images are shown in **Figures 1c)** and **Figure 2c)**.



a) b) c)

Figure 1 The microstructure of the N3 alloy examined by SEM: a) Ex; b) 1P; and by TEM c) 8P (bright field)

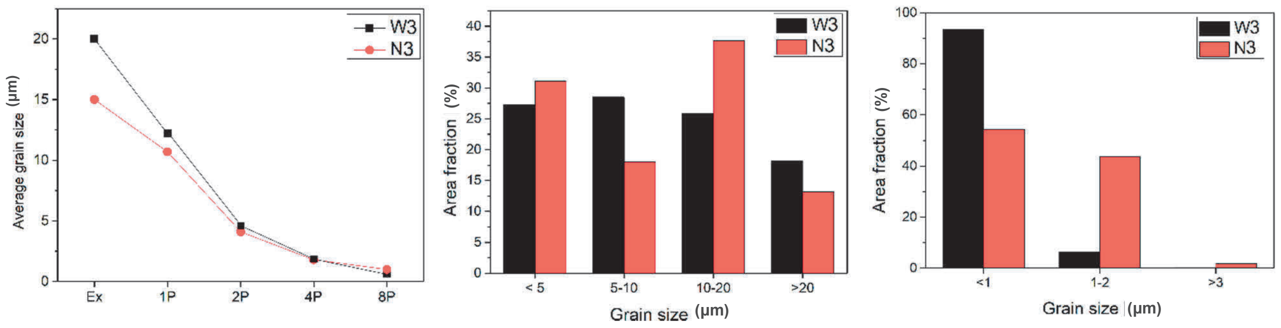


a) b) c)

Figure 2 The microstructure of the W3 alloy examined by SEM: a) Ex ; b) 1P; and by TEM c) 8P (bright field)

The grain size distribution of both alloys after one and eight ECAP passes recalculated from an EBSD data are summarized in **Figure 3b)** and **Figure 3c)**, respectively. As shown in **Figure 3b)**, the area fraction of grains with size <5 µm and >20 µm are similar for both alloys. However, most of the grains in the W3 alloy have a diameter in the range of 5-10 µm, while grains with a size of 10-20 µm have the largest area fraction in the N3 alloy. With the further processing up to eight passes, the grain size distribution of the smallest grains was significantly different between the two investigated alloys. The W3 alloy contained a much higher fraction of grains <1 µm than the N3 alloy and, consequently, average grain size measured in the W3 alloy was ~600 nm, while it was ~1 µm in the N3 alloy. The evolution of the average grain size as a function of increasing number of ECAP passes is shown in **Figure 3a)**.

As shown in **Figure 1** and **Figure 2**, type and distribution of the secondary phase particles changed after ECAP processing. Three types of particles were observed in the extruded condition of the N3 alloy by TEM (not shown here). The first type, plate-like precipitates, was identified as Mg_3Nd and these particles were found in the grain boundaries as well as in the grain interior. Particles of other two types were located primarily in the grain boundaries and were identified as $Mg_{12}Nd$ and $Mg_{41}Nd_5$. Nevertheless, processing by ECAP resulted in a more pronounced precipitation in the N3 alloy. **Figure 1b)** shows severe precipitation occurred in the large unrefined grains. On the other hand, particles in the grain boundaries were much larger and could be considered as a later stage of the precipitation sequence. After eight passes through ECAP, only homogenous distribution of small spherical particles was observed. The diffraction pattern of these particles was not clear but the results indicate that they are $Mg_{41}Nd_5$ equilibrium phases [13]. On the other hand, only randomly located small particles were found in the W3 alloy after the first pass through ECAP and no changes were observed even after eight passes. These particles were identified by TEM as $Mg_{24}Y_5$ [5].



a) b) c)

Figure 3 a) average grain size evolution as a function of the number of ECAP passes; b) grain size distribution of 1P condition; c) grain size distribution of 8P condition

3.2. Compression deformation tests

Figure 4 illustrates the evolution of an yield compression strength (YCS) in the X and Y direction as a function of increasing number of ECAP passes. The extruded samples exhibit the lowest strength, particularly due to the coarser grain structure. In addition, similar values of the YCS were observed for both alloys in both measured directions regardless the different grain size and amount of precipitates (N3 vs. W3). Negative effects of higher average grain size and smaller amount of precipitates observed in the W3 sample were balanced by solute solution hardening, what correlates with the previous results [15]. After ECAP processing, the YCS increased significantly in both alloys and also in both investigated directions. The strength increase of more than 300% was measured in both 8P samples compared to the corresponding extruded conditions. A sharp increase of mechanical strength is not usually observed in Mg alloys processed by ECAP because of strong specific texture development [10]. Nevertheless, in alloys containing REs, a weak texture and consequently sharp increase of mechanical strength were reported previously [7, 8, 11]. The major impact on the hardening of ECAP processed materials has grain refinement. A higher fraction of small grains was observed in the W3 alloy, compared to the N3 alloy, from the first pass through ECAP, as shown in **Figures 3b),c)**. Therefore, higher YCS measured in almost all ECAPed samples of the W3 alloy, compared to the N3 alloy, could be attributed particularly to the grain boundary hardening and solid solution hardening, similar as in extruded samples. The only sample where this conclusion was not true was a comparison of the 1P samples deformed along X-direction. Apparent contradiction with the results measured in Y direction could be explained by texture favourable for twinning in X-direction in both samples (not shown here). Activation of twinning in the W3 alloy decreased yield point of the W3-1P sample, but severe precipitation in the N3 alloy prohibited similar behaviour.

Other systematic difference in the YCS was observed between samples deformed in the X and the Y direction. The strength in all samples is lower when deformed in the transverse (Y) direction. As shown, ECAP led to the formation of microstructure with equiaxed grains and homogeneous distribution of particles; therefore, no differences should have been observed especially after the final eight pass through ECAP. However, specific texture development was observed along with grain refinement during ECAP. During deformation at the room temperature, basal slip should be the dominant deformation mechanism, especially when grains are small and twinning is hard to activate [14]. Hence, for a better understanding of the differences between values of YCS measured in X and Y direction, Schmid factor (SF) for basal slip was calculated from the EBSD data. Average values of the Schmid factor for both alloys and all samples processed through ECAP are displayed in **Table 1**. In almost all conditions, values of the SF are lower in the X direction for both alloys. Due to the fact that lower SF indicates harder activation of basal slip during deformation [14], SF differences explain well higher strength measured in the X direction.

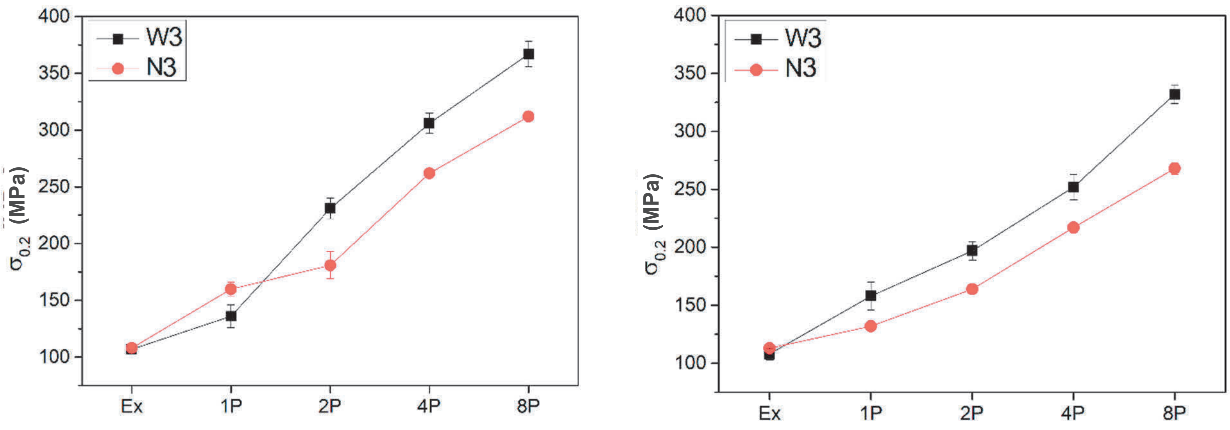


Figure 4 The compression deformation tests in a) X and b) Y direction

Table 1 Average values of the Schmid factor calculated for different samples

	N3 - X	N3 - Y	W3 - X	W3 - Y
1P	0.29	0.36	0.30	0.30
2P	0.26	0.35	0.26	0.36
4P	0.30	0.31	0.20	0.31
8P	0.26	0.32	0.26	0.31

CONCLUSION

Extrusion and ECAP were used to prepare two binary magnesium alloys with neodymium and yttrium in an ultra-fine grain condition. Microstructure and mechanical strength evolution were investigated in both alloys. The following results could be summarized from this study:

- Significant grain refinement in both alloys was achieved by ECAP with a final grain size of ~600 nm and ~1µm for W3 and N3 alloy, respectively. Additionally, severe precipitation was observed in case of the N3 alloy, which led to the formation of a dense distribution of small $Mg_{41}Nd_5$ particles. Such precipitation was not observed in the W3 alloy.
- ECAP processing resulted in a considerable increase of the strength of both alloys. The yield compression strength increased by 300% comparing extruded and ECAPed samples in case of both materials. Such increase was attributed to grain boundary hardening, solid solution hardening in the case of W3 and precipitation hardening in the case of N3. Additionally, rather weak texture, which formed during ECAP in both alloys, resulted in systematically higher strength along the processing direction compared to the transverse direction in almost all samples processed by ECAP.

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