Investigation on texture evolution and recrystallization aspects of novel Mg-Zn-Gd-Y-Nd alloys

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Abstract

Magnesium exhibits substantial property enhancement for a vast range of applications via alloying by rare earth elements (REs). The effect of different amounts of alloying elements (Zn and REs such as Gd, Y and Nd) on the microstructure, texture and mechanical properties of Mg has been investigated here by means of optical microscopy, electron backscattered diffraction, shear punch test and hardness test. Results indicate that the recrystallization process along the grain boundaries consists of dynamic, metadynamic and static recrystallization, while dynamic recrystallization as the dominant mechanism gives rise to bimodal microstructures in as-extruded samples with superior properties. The recrystallized area is narrow at low amounts of alloying elements, whereas addition of higher amounts of alloying elements promotes recrystallization, expands recrystallized areas and weakens texture intensity. Moreover, it is observed that there are some optimal REs ratios promoting a partially recrystallized grain structure which in turn provides higher shear strength, hardness values, and more enhanced ductility as well as a weaker texture.

Keywords:

Mg-Zn-Gd-Y-Nd alloys; texture; recrystallization; extrusion.

1. Introduction

From giant parts in the transportation industry to tiny heart stents, magnesium (Mg) can be used in a large set of products [1, 2]. However, Mg, as the lightest of all commercial structural metals and the sixth most abundant element in the Earth's crust, depicts poor ductility due to its hexagonal close-packed (HCP) crystal structure, which, in turn, provokes limited slip systems. Besides, deformation induces crystallographic asymmetry (texture effect) restricting the activation of non-basal slip systems at room temperature, thereby limiting its ductility [3, 4]. Thus in order to fulfil the plastic compatibility criteria (von Mises condition), some extra slip systems in addition to the basal $\{0001\}\langle 10\text{--}12\rangle$ slip mode, such as prismatic and pyramidal slip modes, are required to be activated. Further, the critical resolved shear stress (CRSS) of these slip systems and twinning systems should be decreased.

To overcome these limitations and improve mechanical properties (including ductility), grain refinement and texture modification through alloying and deformation are the most effective approaches. In this regard, conventional thermomechanical processing such as extrusion and rolling are applied to Mg alloys in order to promote grain refinement and eliminate cast defects. Furthermore, the high temperature of these processes (higher than the recrystallization temperature) usually activates non-basal slip modes, and therefore promotes dynamic

recrystallization (DRX), resulting in further grain refinement, enhanced strength, and also providing a weaker texture intensity which is good for ductility [5].

Together with thermomechanical processing, the addition of alloying elements in general, and rare earth elements (REs) in particular, plays a significant role in the enhancement of Mg alloys' properties [6]. The function of REs in Mg differs from that of each element depending on their atomic radii, and therefore on their solid solubility [7]. The strengthening of Mg by solid solution and the reinforcement of intermetallic phases which remain stable at elevated temperatures are identified as the basic effects of REs additions [8]. Besides, satisfactory results have been achieved when adding REs in terms of texture modification associated with recrystallization and grain growth, and improved workability [9]. Amongst more common RE elements, Gd, Nd and Y have been found to bring about advantageous effects on casting characteristics, as well as electrochemical, mechanical and physical properties at ambient and high temperatures [3, 4, 10]. On top of that, it has also been reported the synergetic effects that Zn plays together with REs which led to unique characteristic microstructures [11, 12]. This has been promoted in the literature with regard to a large combination of Mg, Zn and RE although no systematic studies can be found [9, 13–16]. It is therefore clear that the correct design of Mg-Zn-RE alloys with the optimal amount of alloying elements and the most economical properties necessitates further studies.

Accordingly, the main purpose of the current work is to optimize the chemical composition and processing conditions to obtain Mg-based alloys with outstanding properties. Consequently, this will help to design micro-alloy systems at the minimum cost and to meet the requirements of loadbearing applications. For this purpose, four Mg-based alloys, Mg-Zn-Gd-Y-Nd, with different values of alloying elements were prepared. Hence the effect of alloying element variants was first investigated by microstructure observation using optical microscopy, shear punch test (SPT) and hardness measurements. This study was complemented by EBSD examinations which offered information on the microtextures.

2. Experimental Procedures

In the present study, four newly developed Mg-Zn-Gd-Y-Nd alloys were investigated. The chemical composition of the studied alloys, obtained by the Inductively Coupled Plasma (Arian ICP-OES spectrometer 730-ES) method, is summarized in Table 1. They were denoted alloy I, alloy II, alloy III and alloy IV, respectively. The alloys were designed with the nominal pattern where alloy I was almost only affected by Y, alloy II by Y and Gd, alloy III by Nd in addition to Y and Gd, and finally alloy IV enjoys the effects of all due to the presence of all three REs in almost 1 wt%. The nominal percent of Zn kept basically constant and close to the range 0.4–0.5 wt%, in all four alloys.

The alloys were produced by die casting. Accompanied by pure alloying elements, pure Mg (99.90%) was loaded into a steel mold and they were melted simultaneously. When the temperature of the sample reached 450 °C, a cover gas (Ar) was supplied in order to prevent the ignition and explosion of the melt. The melt was held at 780 °C for 30 min and then stirred continuously at 110 rpm for 5 min. After that, it was poured into a preheated (500 °C) permanent steel mold. During this process the cover gas was also used. The concentration of Gd, Y, Zn and Nd in these Mg-based alloys was analyzed by the Inductively Coupled Plasma method (Arian ICP-OES spectrometer 730-ES).

Round rods of 34 mm in diameter and 70 mm in height were machined from the as-cast ingots and were homogenized in Ar atmosphere at 520 °C for 12 h. Homogenized cylinders were extruded to round rods of 12 mm in diameter under an extrusion ratio of 8 at a temperature of 480 °C using a Zwick/Roell 250 testing machine. The extrusion speed was 1 mm/min. In order to reduce the deformation friction, graphite-based lubricant was used.

Metallographic specimens for optical microscopy, sectioned perpendicular to the extrusion direction (ED), were prepared by conventional grinding and diamond polishing (down to 0.25 µm) followed by etching in an acetic-picral solution. After microstructural observations, microhardness measurement was conducted on samples with the applied load of 50 g and dwell time of 15 s. Moreover, the shear stress-normalized displacement curves of the samples were obtained by the shear punch test (SPT). In this test, the load–displacement is recorded during testing and the average shear stress τ is calculated using the relation $\tau = P/\pi dh$ where P is the load on the punch, h is the specimen thickness, and d is the average of the die and punch diameter [17].

Crystallographic texture was studied by electron backscattered diffraction (EBSD) data provided by a Jeol scanning electron microscope (SEM). For EBSD analysis, specimens were extracted from the longitudinal section of the extruded sample and were prepared using colloidal silica as the final step polishing medium. The analyses of the acquired EBSD raw data were accomplished using Channel 5 and ATEX software [18], which gave rise to the microstructure map, pole figure, inverse pole figure and misorientation distribution chart for each alloy. (It should be mentioned that in all EBSD maps, the low angle grain boundaries (LAGBs), between 2° and 15°, are marked by red lines and the high angle grain boundaries (HAGBs), beyond 15°, by black lines.)

Table 1. Chemical composition (wt.%) of the four designed alloys I, II, III and IV.

Alloy	Nominal composition	Zn	Gd	Y	Nd	Mg	Total REs
I	Mg-0.5Zn-<0.1Gd-0.5Y-<0.1Nd	0.512	0.046	0.442	0.064	Bal.	0.552
II	Mg-0.5Zn-0.5Gd-0.7Y-<0.1Nd	0.493	0.462	0.692	0.058	Bal.	1.212
III	Mg-0.5Zn-0.6Gd-0.5Y-0.2Nd	0.409	0.588	0.484	0.143	Bal.	1.215
IV	Mg-0.5Zn-1Gd-1Y-1Nd	0.477	1.079	0.619	0.677	Bal.	2.375

3. Results

3.1 Microstructural Observations

The optical microstructures of the four current alloys in different states, namely as-cast and as-extruded are shown in Fig. 1. A different average grain size was observed in the as-cast microstructure of each alloy. The effect of increasing the amount of REs on grain size refinement is conspicuous. Examples of the as-extruded microstructure of the samples are illustrated in Fig. 1. Upon extrusion, necklaces of grains were noticed along the initial grain boundaries.

A bimodal microstructure of fine recrystallized grains and large unrecrystallized grains through the extrusion direction are noticed, especially in alloys I, II and III. However, alloy IV revealed an almost fully recrystallized grain morphology.

Fig. 1 Optical microstructure of the cross section of the alloys after casting and extrusion (all magnification bars correspond to 200 μm)

3.2 Mechanical Properties

Figure 2 illustrates the microhardness of the samples measured across the diameter of their cross sections. Generally, the hardness has many fluctuations in the as-cast condition. Extrusion at elevated temperature decreased the hardness amount slightly but a more uniform distribution was obtained.

Fig. 2 Vickers microhardness along the normalized distance of the **a** as-cast and **b** as-extruded samples, where d is the distance from the center and r is the sample's radius

Fig. 3 Shear stress vs. normalized displacement of the as-extruded samples

Figure 3 shows the shear stress–normalized displacement curves of the as-extruded samples. It is observed that higher amounts of alloying elements increased the shear strength of the samples. Although alloy IV achieved the highest shear stress of 136 MPa, it experienced lower ductility in comparison with other alloys. The second highest shear stress was attributed to alloy I, and alloy II and alloy III held the following positions, respectively, while the same trend was observed in their ductility.

3.3 Textural Observation

The extrusion texture of these four different alloys was studied along the extrusion direction by means of EBSD. Considering that the studied material is an extruded bar, the directionality of the extruded material is of importance, namely the extrusion direction (ED) and transverse direction (TD).

The microstructure of alloy I (Fig. 4a) is an excellent representation of the DRX phenomenon. Layers of nuclei formed across the grain boundaries and progress in expense of the deformed domain by formation of new layers of the DRXed grains. As can be seen, further deformation resulted in intertwining the layers from the two grain boundaries inside the domain grain in some parts, but the recrystallization rate was not high enough for the middle grain to be fully covered by the DRXed grains and the parent grain was still observable.

Alloy II (Fig. 4b) showed a bimodal microstructure with two components, i.e. a stretched coarse-grain region and a recrystallized fine grain region in the IPF map. In Alloy II, the middle grain was fully covered by the DRXed grains and some grain growth was also observable.

As depicted in Fig. 4c, a bimodal microstructure consisting of coarse unrecrystallized grains and fine DRXed grains was the most typical feature of alloy III. The fine grains accompanied by the coarse ones in the as-extruded microstructure were associated with partial dynamic recrystallization. However, alloy IV is an apparent example of a fully recrystallized material (Fig. 4d). The pole figures in Fig. 5 display the $\{0001\}$ $\langle 11\text{--}20 \rangle$ texture components in alloy I, II and III, while alloy IV represents a $\{0001\}$ basal texture. Moreover, it should be noted that the maximum intensity has decreased with the addition of higher amounts of REs and the progress of the recrystallization.

The inverse pole figures explain more precisely the presence of a fiber texture in alloy I with a maximum at the $\langle 11\text{--}20 \rangle$ position. This so-called “cylindrical texture”, normal of the basal planes $\langle 0001 \rangle$ are perpendicular to the extrusion axis, was regularly observed in the extruded rods of hexagonal materials [19]. Regarding the IPF for alloy II, the strongest texture peak is at a position between $\langle 11\text{--}20 \rangle$ and $\langle 0001 \rangle$. According to Stanford et. al. report [20], this peak was termed “RE texture component” and originated from the $\langle 11\text{--}21 \rangle$ direction being parallel to the extrusion direction. It was reported that the RE texture often existed in the extruded rods, and was hardly found in the extruded sheets [19]. Wang et. al. showed that the separated RE texture component exhibits a characteristic similar to the full texture component and they also contributed the ED-split of the full texture to the RE texture component, which occurred in alloy III as well [19].

As already stated, with the highest amount of alloying elements, alloy IV has the finest grain size and the most homogenous structure among all four extruded alloys. A strong basal texture can be noticed in this alloy. Beside the $\{0001\}$ basal texture, the c-axis of the recrystallized grains tilted very slightly from its normal direction to the extrusion direction and also spread in this direction. Therefore, a fiber texture appeared in the IPF of alloy IV in a way that plate normal was away from $\langle 0001 \rangle$ and approximately equivalent to the position in-between $\langle 11\text{--}22 \rangle$ and $\langle 10\text{--}11 \rangle$. The understanding of the operating slip systems on basal planes can provide suitable information about the origin of basal fiber texture.

The texture existing in the extruded Mg alloys can be also confirmed by the orientation distribution function, ODF, (calculated from EBSD data). The ODFs can provide treasured information regarding dominant components, their positions, volume fractions and overall texture sharpness.

The ODF distribution positions of the extruded samples for the selected sections of fixed ϕ_2 are shown in Fig. 6. According to Wang et. al.[19], the first row in Fig. 6 depicts a combination of $\{0001\}\{11-20\}$ and $\{10-10\}\{0001\}$ textures for alloy I, while the peak centered in $\phi_1 = 80^\circ$, $\phi = 80^\circ$ in $\phi_2 = 30^\circ$ section was a representation of $\{11-20\}\{0001\}$ texture. Occurrence of this peak was due to the presence of some elongated unDRXed grains in the DRXed region (Fig. 4(a)). Moreover, in the second row, the ODF section at $\phi_2 = 0^\circ$ displays that the main peak for alloy II was centered at a position with $\phi_1 = 60^\circ$ and $\phi = 0^\circ$ and a weak secondary peak around $\phi_1 = 0^\circ$ and $\phi = 0^\circ$. This two ODF sections were consistent with the $\{0001\}\{11-20\}$ fiber texture [19]. In addition to the $\{0001\}\{11-20\}$ fiber texture, alloy III revealed a peak centered in $\phi_1 = 90^\circ$, $\phi = 35^\circ$ in $\phi_2 = 30^\circ$ section. Finally, as shown in the last row, the ODF of alloy IV shows a combined $\{0001\}$ basal texture and a weak fiber texture of $\{11-20\}$ and $\{10-10\}$.

Fig. 4 EBSD microstructure of as-extruded **a** alloy I, **b** alloy II, **c** alloy III and **d** alloy IV

Fig. 5 Pole figures and inverse pole figures (IPF) of the as-extruded alloys

4 Discussion

The properties of Mg alloys are largely influenced by the microstructure and the texture. The development of RE-containing Mg alloys should consider how to achieve developed properties on the basis of low RE content regarding the high value and waste of RE resources. Hence the alloying design and the REs ratio are important factors in the case of properties. At room temperature, slip in Mg occurs predominantly by basal slip, while prismatic and pyramidal slip become more noticeable at increasing temperatures. It is worth remembering that as the extrusion temperature is 480 °C, slip becomes the main plastic deformation mechanism, and the migration of the grain boundaries is fully promoted. With the development of the deformation, the dislocation density increases and the grain boundaries confront dislocation pile-ups resulting in the formation of sub-boundaries and dislocation cells. Subsequently, high angle grain boundaries are formed during dislocation absorption by sub-boundaries, which in turn increases the misorientation among them. More importantly, and as already mentioned, RE elements promote the process of DRX by decreasing the SFE.

Regarding Fig. 1, the microstructure of the extruded alloys is an evident necklace structure along the grain boundaries. It is well accepted in the literature that these typical necklaces are indicative of the occurrence of dynamic recrystallization [21]. In the particular case of Mg-Zn-RE alloys, the recrystallization process occurred at the boundaries and not only did it consist of dynamically recrystallized grains (as expected during the extrusion process at elevated temperatures), but also of metadynamically recrystallized (MDRX) grains (the large grains within the necklace area). This is why the grains on the necklace were not so thin as expected when DRX is the only recrystallization phenomenon. This metadynamic phenomenon (i.e., static recrystallization where

the nucleation stage is skipped as nuclei are already present) occurred during the air cooling interval after the extrusion. Moreover, because DRX was only partial, a bimodal microstructure of fine recrystallized grains (either dynamically or metadynamically) and large unrecrystallized grains through the extrusion direction was noticed, especially in alloys I, II and III. Recrystallization in alloy I, with the lowest amount of alloying elements, started by the nucleation of the first layers of DRX nuclei along the grain boundaries. Formation of new layers of the recrystallized grains continued in expense of the deformed grains with basal texture but the recrystallization rate was not high enough for the layers to intertwine inside the domain grain. By increasing the REs amount, recrystallization proceeded in a higher rate in alloy II and the deformed grains covered by some newly developed fine DRX nuclei, some DRXed grains and few larger MDRXed grains. These MDRXed grains contained some twins with an $84\text{--}87^\circ$ misorientation angle, which was indicative of $\langle 10\text{--}12 \rangle$ extension twins [22]. Although these recrystallized grains had random texture, the wide extended grains gave rise to a sharper basal texture. Moreover, the formation of twins in alloys I and II can be attributed to Y as the dominant rare earth element in these alloys, while in alloys III and IV, no twins were detected, obviously. According to Fig. 4(c), the microstructure of alloy III corresponded to a no fully recrystallized material. This can be explained by the fact that the DRXed grains in the as-extruded conditions were very few, so the nucleation sites for SRX during the subsequent annealing were very low, and therefore it progressed very slowly. Having not enough time to complete recrystallization, then a bimodal grain size distribution should be apparent. Moreover, it is worth mentioning that, as reported in the literature [23], Gd and Nd have close grain refining effects. This has been explained in terms of the inhibition of the grain boundary migration, resulting therefore in grain refinement [23]. Moreover, it has long been established that the addition of alloying elements, more precisely the REs used in the present study, significantly lowers the stacking fault energy (SFE) and thereby facilitates the progress of dynamic recrystallization, which resulted in a wider area covered by the DRXed grains in alloy III in comparison to the other two alloys [24]. This wide randomly textured area weakened the overall texture of the material and preserved its high ductility as well.

On the other hand, alloy IV revealed an almost fully recrystallized grain morphology. This can be explained by two facts. First, and as mentioned above, the addition of alloying elements, here REs, reduced the SFE offering a higher driving force for recrystallization which facilitated the DRX phenomenon. And second, because the initial grain size in the as-cast condition was fine, the critical strain for the onset and finalization of DRX was lower, enhancing the chance to have a fully DRX microstructure with higher shear strength in comparison to alloys I, II and III, under the same processing conditions.

As a consequence of the observations pointed out in the above paragraphs, the texture of the present alloys during extrusion is formed through the process of deformation, plus recrystallization (which in turn is comprised of DRX, MDRX, SRX or a combination of all), and even some possible grain growth [25]. In hexagonal metals and alloys processed by rolling or extrusion, the texture evolution also depends mainly on the c/a lattice constant ratio [19]. The Peierls stress for non-basal slips decreases by decreasing the c/a ratio. For metals and alloys with $c/a = 1.623$, such as Mg, which almost has the ideal value of 1.633, the basal fiber texture often tends to be formed in the plastic deformation process. Such textures stem from the slip systems operating on basal planes [19]. In the case of metals and alloys with $c/a > 1.633$, such as Zn (1.856) or Cd (1.885), the basal poles tend to tilt $15\text{--}25^\circ$ away from the normal direction towards RD; in contrast, a material with $c/a < 1.633$, such as Ti (1.588), often shows a splitting of the

basal poles towards TD [16]. According to some authors, a basal texture with a slight tilt of poles from ND toward the TD and RD may occur in some Mg alloys; the RD spread of the basal texture is attributed to the activation of the pyramidal slip systems [26]. As a result, the presence of Zn in alloys affected the c/a ratio and caused the RD split of the texture, which is evidence for the activation of the pyramidal slip specially in alloy III.

Referring to Table 1, even slight changes in the amount of alloying elements, resulted in significant changes in the stored energy, microstructure, texture and strength of the alloys. The large difference existing in the atomic radii of Mg and Y allows strengthening of Mg by both solid solution and precipitation hardening [27, 28]. Likewise, Nd has the highest solid solubility in Mg and it has also been reported to increase the strength by both solid solution and precipitation hardening [29]. Additionally, Gd gives rise to solid solution strengthening when alloyed with Mg and weakens the texture by producing the rare earth texture component [30, 31]. Comparing the shear stress-normalized displacement curves of different alloys, it is evident that fine DRXed grains improved the strength, while their weak texture enhanced the ductility. Therefore, it is considered that the difference in the elongation between different alloys was not only due to the texture but also stemmed from their different bimodal structures which resulted from different amounts of alloying elements. As can be seen in Fig. 3, the presence of Nd along with Gd in alloy III decreased the strength but induced a lamellar bimodal microstructure of fine DRXed grains across the elongated deformed grains which enhanced ductility. On the other hand, alloy IV enjoying the presence of all REs at almost 1 wt% changed the decreasing trend of the shear stress in alloys and exhibited the highest shear strength among them. As a result, partial DRX is the key factor in order to give rise to the combination of the noble properties of high strength and large elongation. Adjusting the content of each rare earth element in order to save the costs due to the presence of RE, it should be noted that there are some preferable ratios among the alloying elements. Alloys demonstrated superior properties when Nd/Gd and Nd/Y ratios were about 1 and there was an obvious drop in the properties. This was showcased by alloys II and III when Y/Gd was about 1. Therefore, it can be concluded that although alloy IV, having the highest amount of alloying element, possessed the highest shear strength, in order to save ductility a bimodal structure of alloy III is favorable. As a result, a design with the amounts of $Zn \approx 0.5$, $0.6 < Gd < 1$, $Y \approx 0.5$ and $0.1 < Nd < 0.6$ in wt% is suggested, while with the lowest amount of alloying elements a combination of noble properties such as weak texture, bimodal microstructure, enhanced ductility and strength can be provided.

Fig. 6. The ODF sections of $\phi_2=0^\circ$ and $\phi_2=30^\circ$ for the investigated as extruded alloys.

5. Conclusion:

Four Mg–Zn–Gd–Y–Nd were designed in order to optimize the chemical composition and processing conditions to obtain micro-alloy system with outstanding properties at the minimum cost. The main conclusions are summarized as follows:

1. The maximum peak intensity decreased and the hardness increased through the addition of REs to Mg. The resulting RE texture was observable in the pole figures of alloys II and III, which means that there is a critical minimum amount for REs to be effective.
2. Dynamic, metadynamic and static recrystallization are the combined recrystallization processes developed at the boundaries and led to a bimodal microstructure of fine recrystallized grains (either dynamically or metadynamically) and large unrecrystallized grains along the extrusion direction. Gd as the most abundant alloying element in alloy III gave rise to a bimodal structure with a higher amount of stored energy and finer DRXed grains in the presence of Nd.
3. Alloy III had a partial DRXed microstructure which provided suitable ductility, while alloy IV had an equiaxed structure with a strong basal texture with the highest shear strength but lower ductility. So it can be concluded that higher amounts of alloying elements do not necessarily result in better properties and an alloying design concerning alloys III and IV may provide the context for the development of superior properties. Hence a design with the amounts of $Zn \approx 0.5$, $0.6 < Gd < 1$, $Y \approx 0.5$ and $0.1 < Nd < 0.6$ possess the best alloying design with a combination of favorable properties such as weak texture, bimodal microstructure, enhanced ductility and strength accompanied by having the lowest amount of alloying elements.

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