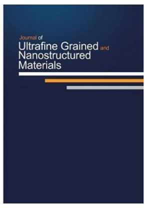

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Grain Refinement and Enhancement of Mechanical Properties of Hot Extruded Rare-Earth Containing Magnesium Alloy

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ABSTRACT

The effects of rare earth addition and hot extrusion process on the grain refinement of magnesium alloy were studied. The as-cast Mg-6Al-1Zn (AZ61) alloy had the average grain size of ~ 64 µm and its micro-structure consisted of α-Mg and $Mg_{17}Al_{12}$ phase. By partial substitution of Al with Gd to reach Mg-4.8Gd-1.2Al-1Zn alloy, it was observed that the $Mg_{17}Al_{12}$ phase disappeared and two new intermetallic phases, i.e. $(Mg,Al)_3Gd$ and Al_2Gd, were identified. The extrusion process showed significant effects on the shape and size of intermetallics and grain size of the matrix. The grain size of the extruded Mg-6Al-1Zn alloy was refined from 64 µm to 13.4 µm as a result of recrystallization. Regarding the Mg-4.8Gd-1.2Al-1Zn alloy, the grain refinement was much more pronounced, where the extruded grain size has been refined from 698 µm to 2.4 µm (extruded at 385 °C) and 1.3 µm (extruded at 320 °C). This was related to the presence of fine and widely dispersed intermetallic phases. Tensile strength and total elongation of extruded alloys were much higher than their as-cast counterparts and the extruded Mg-6Zn-1Al alloy showed magnificent mechanical properties. The latter was related to the absence of intermetallic particles, which act as stress risers.

Keywords: *Magnesium alloys; Rare-earth addition; Microstructure; Tensile properties.*

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1. Introduction

As the lightest structural materials, magnesium alloys have attracted considerable attention for many industries [1,2]. However, they suffer from poor room-temperature ductility. It has been demonstrated that the addition of rare earth (RE) elements, especially Gd to Mg is an effective way to modify the microstructure and attaining superb mechanical properties [3-6]. It was shown in the published works that the addition of Gd to Mg-2Al-1Zn alloy can lead to the formation of (Mg,Al)3Gd and Al_2Gd phases followed by the enhanced tensile properties at both room and elevated temperatures [5]. Moreover, the addition of Gd to AZ31 alloy is an effective way to inhibit the formation of $Mg_{17}Al_{12}$ phase along grain boundaries [7] and to improve the rolling capability [8]. It has been shown that like Al_2Y [9] and Al_2Ca [10] compounds, the Al_2Gd phase formed during solidification is an effective inoculant for heterogeneous nucleation of Mg [11]. Conclusively, it seems that the Mg-Gd-Al-Zn system is a good candidate for developing high performance magnesium alloys.

Moreover, the grain refinement by hot working under dynamic recrystallization (DRX) conditions has been used for the enhancement of mechanical properties of Mg alloys [12-17]. The hot deformation behavior of magnesium alloys during hot compression [14-16,18], hot torsion [19], and shear punch tests [20] has been demonstrated so far. The hot extrusion process has become a promising method, which can be optimized for grain refinement by adjusting the extrusion ratio, pressing temperature, and ram speed. The extrusion process can significantly affect the microstructure of the material and can alter the shape, size, and distribution of intermetallic compounds. This needs to be investigated for new classes of Mg alloys.

The present work aims to evaluate the effect of substitution of Al with Gd and hot extrusion process on the microstructure and mechanical properties of AZ61 magnesium alloy to pave the way for further development of magnesium alloys containing rare earth elements.

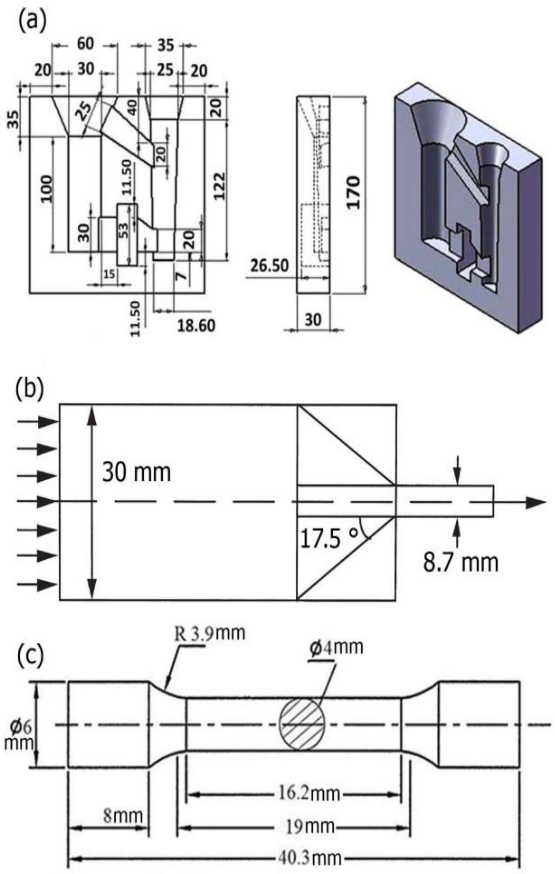

Fig 1- Schematic representation of (a) casting mold, (b) extrusion mold, and (c) tensile test specimen.

2. Experimental materials and procedure

The Mg-6Al-1Zn and Mg-4.8wt.%Gd-1.2wt.%Al-1wt.%Zn ingots were prepared from pure Mg and several masteralloys by heating to the temperature of 770 °C under protective gas atmosphere of 5%SF_6+CO_2 in an induction furnace, holding the melt at that temperature for 5 min for homogenization purposes, and then casting in a cast iron mold. Schematic representation of the mold is shown in Figure 1a. After homogenization at 400 °C for 14 h, the hot extrusion process with a ratio of 12:1 at 320 and 385 °C was performed (See Figure 1b). Optical microscopy (OM) and scanning electron microscopy (SEM) were used for microstructural investigations after etching in a solution containing 17 ml ethanol, 1 g picric acid, 2.5 ml acetic acid and 2.5 ml water. Tensile test specimen was prepared according to ASTM E8-04 standard (See Figure 1c) and this test was carried out at a crosshead speed of 1 mm/min.

3. Results and discussion

Figure 2 shows the XRD patterns of as-cast alloys. It can be seen that the Mg-6Al-1Zn (AZ61) alloy consists of α-Mg and $Mg_{17}Al_{12}$ phase. By partial substitution of Al with Gd to reach Mg-4.8Gd-1.2Al-1Zn alloy, it can be seen that the $Mg_{17}Al_{12}$ phase disappeared and the diffraction peaks of two new intermetallic phases, i.e. $(Mg, Al)_3Gd$ and Al_2Gd, can be identified. The morphology of these phases can be seen in Figure 3b, where the $(Mg, Al)_3Gd$ compound shows branched morphology but the Al_2Gd intermetallic has nearly spherical shape.

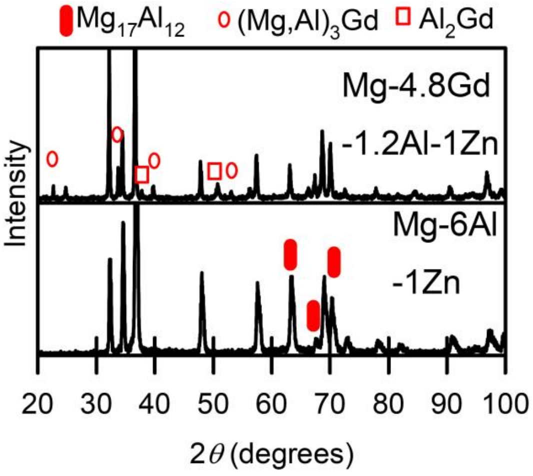

Fig 2- XRD patterns of the as-cast alloys. The rest of peaks belong to α–Mg. Note that the majority of high-intensity diffraction peaks of Mg17Al12 phase coincide with peaks of α–Mg.

The as-cast structures are shown in Figure 3. In Figure 3a, the presence of the non-equilibrium eutectic $Mg_{17}Al_{12}$ phase at grain boundaries of α-Mg matrix in the Mg-6Al-1Zn alloy is evident. In this way, it is possible to estimate the grain size of this alloy by consideration of eutectic $Mg_{17}Al_{12}$ phase at grain boundaries, which was determined as ~ 64 µm. However, it was possible to determine the average grain size of Mg-4.8Gd-1.2Al-1Zn alloy by macroetching (5% Nital solution) as shown in Figure 3b, where the grain size was determined as ~ 698 µm. It can be seen that the as-cast grain size of the AZ61 alloy is much finer. It is known that aluminum is a potent grain refiner for α-Mg based on the growth restriction mechanism [21], and hence, its partial substitution by Gd might be unfavorable. It should also be noted that Al_2Gd phase forms during solidification and acts as a nucleation agent [11]. However, it seems that this effect is completely masked by the decrease in aluminum content of the melt. Based on the tensile curves shown in Figure 4, the as-cast tensile strength of AZ61 is higher, which might be related to its finer grain size.

Figure 5 shows the extruded microstructures. It can be seen that the grain size of Mg-6Al-1Zn alloy extruded at 385 °C has been reduced from 64 µm to 13.4 µm as a result of recrystallization. Moreover, the grain-boundary eutectic $Mg_{17}Al_{12}$ phase has been disappeared. Regarding the Mg-4.8Gd-1.2Al-1Zn alloy, the grain refinement is much more pronounced, where the extruded grain size has been refined from 698 µm (as-cast condition) to 2.4 µm (extruded at 385 °C) and 1.3 µm (extruded at 320 °C). This enhanced refinement can be related to the presence of intermetallic compounds at extrusion temperature in this alloy, where the fractured and widely dispersed particles are available as shown in Figure 5b and Figure 5c. These particles produce more intricate deformation

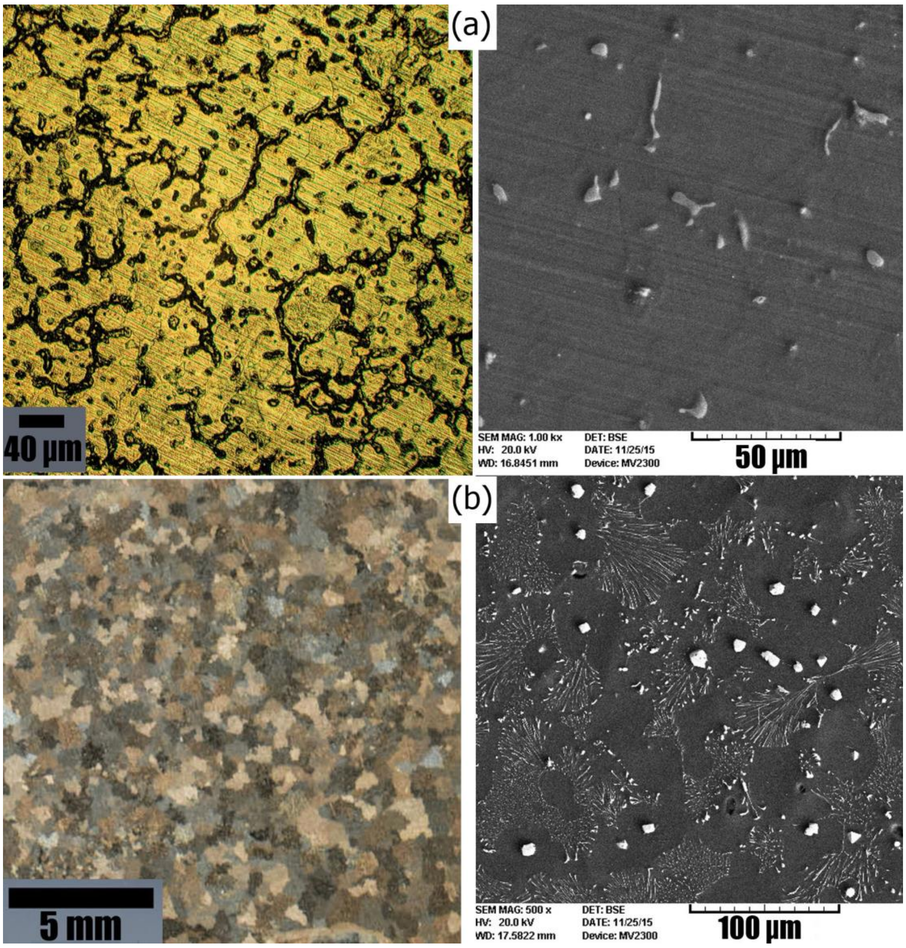

Fig 3- Macrostructure and microstructure of the studied alloys (a) Mg-6Al-1Zn alloy and (b) Mg-4.8Gd-1.2Al-1Zn alloy.

patterns, promote recrystallization, and inhibit the growth of fine recrystallized grains. Note that based on the Mg-Al phase diagram, the $Mg_{17}Al_{12}$ phase dissolved in the matrix at temperatures higher than 300 °C.

The obtained tensile curves for the extruded alloys are also shown in Figure 4. It can be seen that the tensile strength and total elongation of extruded alloys are much higher than their as-cast counterparts. It can be seen that by grain refinement from 2.4 µm to 1.3 µm, the tensile strength of Mg-4.8Gd-1.2Al-1Zn alloy enhanced as expected. However, the extruded Mg-6Zn-1Al alloy with average grain size of 13.4 µm shows a magnificent mechanical properties. In fact, its tensile strength is near that of Mg-4.8Gd-1.2Al-1Zn alloy extruded at 320 °C but its total elongation is evidently higher. This might be related to the effect of intermetallic phases present in the Mg-4.8Gd-1.2Al-1Zn alloy, which act as stress risers. These results demonstrate the importance of hot extrusion on the enhancement of mechanical properties of magnesium alloys and magnificent grain refinement by addition of rare earth element Gd and hot extrusion process.

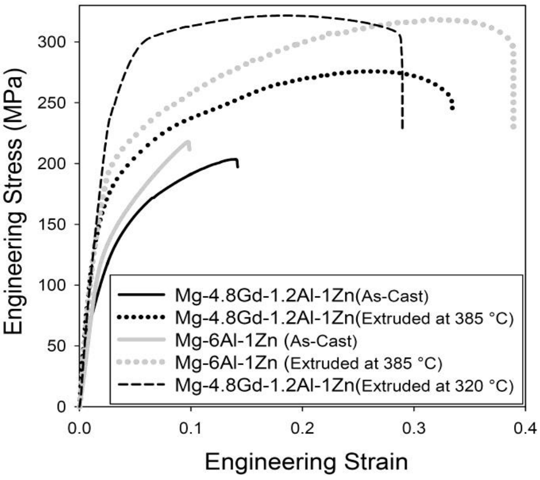

Fig 4- Tensile stress-strain curves.

5. Conclusions

The effects of partial substitution of Al by rare earth Gd and also the hot extrusion conditions on the grain refinement of magnesium alloy were studied. The following conclusions can be drawn from this work:

(1) The α-Mg and $Mg_{17}Al_{12}$ phases were identified for the as-cast Mg-6Al-1Zn (AZ61) alloy. However, the (Mg, $Al)_3Gd$ and Al_2Gd phases appeared and the $Mg_{17}Al_{12}$ phase disappeared by partial substitution of Al with Gd.

(2) The shape and size of intermetallics and the grain size of the matrix was significantly altered

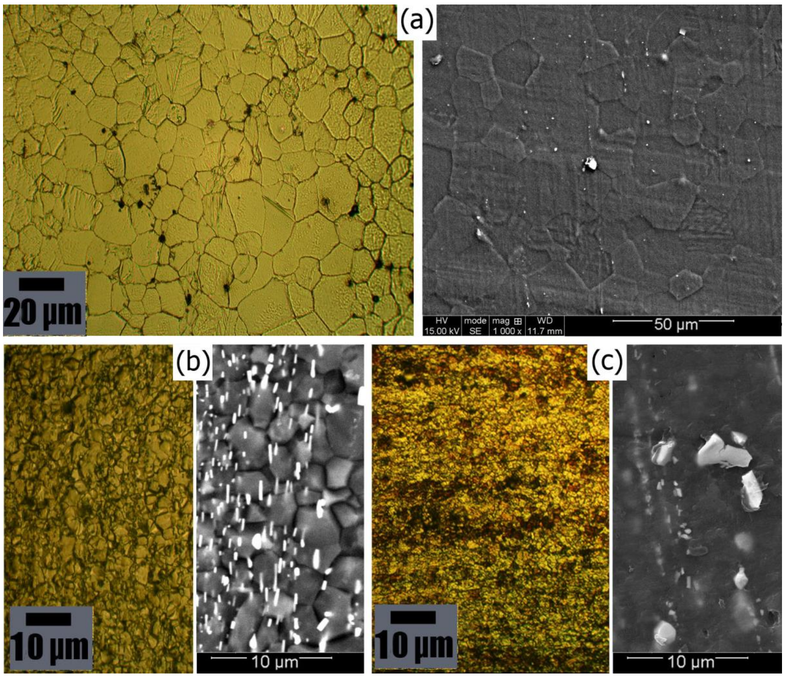

Fig 5- Extruded microstructures obtained in the present study: (a) Mg-6Al-1Zn alloy extruded at 385 °C, (b) Mg-4.8Gd-1.2Al-1Zn alloy extruded at 385 °C, and (c) Mg-4.8Gd-1.2Al-1Zn alloy extruded at 320 °C.

after the extrusion process. The grain size of the extruded Mg-6Al-1Zn alloy was refined from 64 µm to 13.4 µm as a result of recrystallization. Regarding the Mg-4.8Gd-1.2Al-1Zn alloy, the grain refinement was much more pronounced, where the extruded grain size has been refined from 698 µm to 2.4 µm (extruded at 385 °C) and 1.3 µm (extruded at 320 °C).

(3) The extruded alloys showed much better tensile strengths and total elongations compared with their as-cast counterparts. By grain refinement from 2.4 µm to ~ 1 µm after extrusion, the tensile strength was increased in the Mg-4.8Gd-1.2Al-1Zn alloy from 275 to 322 MPa. However, the extruded Mg-6Zn-1Al alloy showed magnificent mechanical properties. In fact, its tensile strength was near the Mg-4.8Gd-1.2Al-1Zn alloy extruded at 320 °C but its total elongation was ~ 10% higher. This was related to the absence of intermetallic particles, which act as stress risers.

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