ULTRAFINE GRAINED MG-ZN-ZR MAGNESIUM ALLOY VIA MICROALLOYING WITH RARE EARTH ELEMENTS AND HOT EXTRUSION

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ABSTRACT

Grain refinement and enhancement of the tensile properties of Mg-Zn-Zr alloy via addition of rare earth (RE) elements at microalloying levels and utilization of hot extrusion process were investigated. The extrusion of the RE-containing Mg-Zn-Zr alloy replaced the coarse grain size of the as-cast ingot with an ultrafine grained (UFG) microstructure with the recrystallized grain size of ~ 1 µm. In this regard, the effect of the RE elements during solidification and the occurrence of the dynamic recrystallization (DRX) during hot extrusion process were found to be responsible. Accordingly, the ultimate tensile strength (UTS) and total elongation were significantly enhanced, which reveals the synergistic effect of RE addition and hot extrusion process on the enhancement of the mechanical properties of magnesium alloys.

KEYWORDS: *Magnesium alloys, Recrystallization, Grain refinement, Mechanical properties*

1 INTRODUCTION

Zinc has been frequently used as a major alloying element in magnesium alloys. Not only this element improves the strength of Mg alloys but it also increases their formability, where both grain refinement [1] and solid solution strengthening [2] have been noted. Another critical element is zirconium, which is a grain refiner for Mg alloys [3]. Accordingly, the ZK series of magnesium alloys have gained a significant attention. Among the Mg-Zn-Zr alloys, the composition of Mg-0.5Zn-0.5Zr with incorporation of rare earth (RE) elements has gained a recent considerable attention [9-11]. For instance, a combination of Gd, Y, Nd, and Er has been extensively added to the Mg-0.5Zn-0.5Zr alloy and the formation and effects of long period stacking order (LPSO) structures have been discussed in details [4-6]. The effect of Sm addition to the Mg-0.5Zn-0.5Zr alloy has been also investigated and it has been reported that the resulting intermetallic particles could act as heterogeneous nucleation sites for dynamic recrystallization during hot extrusion via particle stimulated nucleation mechanism (PSN) mechanism for enhancement of mechanical properties and weakening the basal texture in the as-extruded alloys [4,5]. For the Mg-2.5Nd-0.5Zn-0.5Zr alloy, it has been shown that the coarse microstructure with continuous network of intergranular eutectic $Mg_{12}Nd$ phase can be refined by the high strain rate rolling (HSRR) process [6]. Besides these works, the effect of RE in the form of mischmetal addition, due to its lower price [7], to the Mg-0.5Zn-0.5Zr alloy is important in the as-cast and wrought conditions. The present work provides a systematic investigation to unravel the effects of microalloying with rare earth elements and hot extrusion in a lean Mg-Zn-Zr alloy.

2 EXPERIMENTAL DETAILS

Pure Mg, as well as Mg-50Zn and Mg-33Zr master alloys and cerium-based mischmetal RE (48.7Ce-26.4La-19.6Nd-5.3Pr) were used as the charge materials in an induction furnace for melting under the protection of a gas mixture containing CO_2+SF_6 and pouring into a cylindrical metallic mold shown in **Figure 1a**. The Mg-0.5Zn-0.5Zr alloy with 0 and 0.5RE (wt%) was considered in this work. The homogenized ingot (400 °C - 12 h) was preheated at the extrusion temperature of 250 °C for 1 h, which was followed by extrusion with a ratio of 12:1 at this temperature as schematically depicted in **Figure 1b**. Microstructure observations were carried out using optical microscopy and scanning electron microscopy (SEM, TESCAN VEGA II XMU) equipped with an energy dispersive X-ray spectroscopy (EDS) detector for elemental analysis. A solution containing 4.2g picric acid, 70 ml ethanol, 10 ml acetic acid, and 10 ml distilled water was used for chemical etching. Average grain size was measured using the standard intercept method according to the ASTM E112-96. Room temperature tensile tests were performed using ASTM-E8 round tensile specimens (**Figure 1c**) at a cross head speed of 1mm/min. The tests were repeated once and it was revealed that the reproducibility of results is in the valid range.

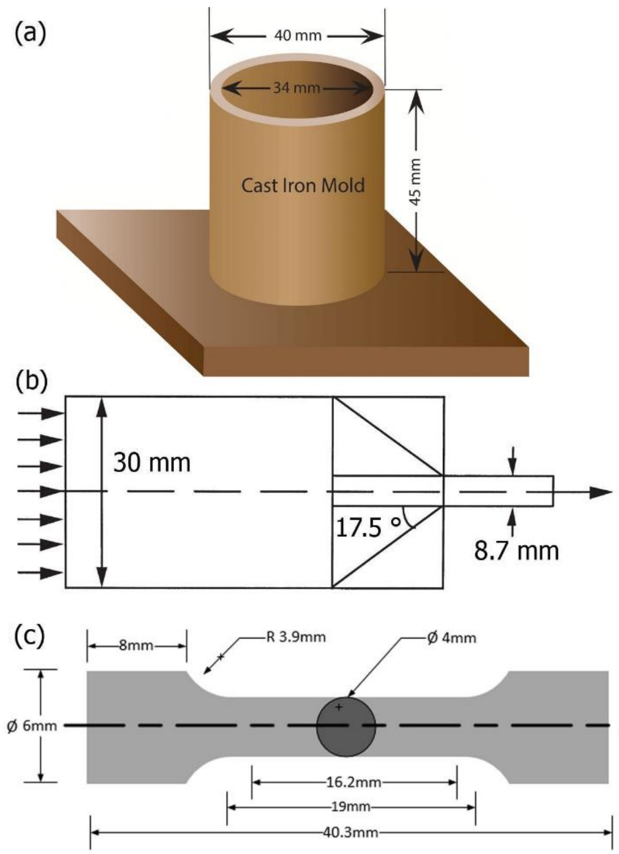
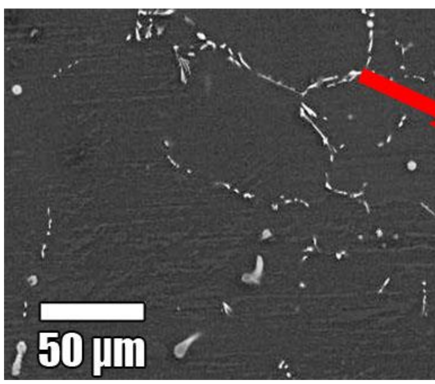


Figure 1: Details of (a) metallic mold (b) extrusion die, and (c) tensile specimen.

3 RESULTS AND DISCUSSION

The optical micrographs of the as-cast alloys are shown in **Figure 2**. The Mg-0.5Zn-0.5Zr alloy contains coarse α-Mg grains with the average size of 1320 μm and the introduction of 0.5 wt% RE resulted in a moderate grain refinement effect in the as-cast condition (1030 μm). This refinement can be related to the enrichment of RE solutes near the solid/liquid interface during solidification, which enhances the undercooling effect via constitutional undercooling. The backscattered electron (BSE) SEM micrograph of the Mg-0.5Zn-0.5Zr-0.5RE alloy is shown in **Figure 3**, where a bright intergranular constituent can be observed. The high brightness implies the presence of elements with high atomic numbers. The EDS spectrum of the grain boundary region reveals the presence of RE (Ce, La, Pr, and Nd) and Zr. This explains the observed brightness due to high atomic number of these elements. Based on EDS, the atomic ratio of RE/Mg was obtained as ~ 0.07 which is in a good agreement with the atomic ratio of 1/12=0.083 for the $Mg_{12}RE$ compound [8].

Figure 2: Optical micrographs of as-cast alloys.



Element	Wt.%	At.%
Mg	61.90	89.34
Zn	0.28	0.15
Zr	8.16	3.14
La	3.69	0.39
Ce	15.87	3.97
Pr	2.32	0.58
Nd	7.79	1.89

Figure 3: SEM image and EDS point analysis of the Mg-0.5Zn-0.5Zr-0.5RE alloy.

The microstructure of the extruded Mg-0.5Zn-0.5Zr-0.5RE alloy is shown in **Figure 4**. Compared to the as-cast alloys, a remarkable grain refinement can be observed due to the dynamic recrystallization (DRX) induced by the hot deformation process [9]. By applying the extrusion process, the average grain size has decreased from 1030 to the recrystallized grain size of 1.3 µm. Moreover, the second phase particles have been fragmented and dispersed along the extrusion direction. Therefore, the extruded Mg-0.5Zn-0.5Zr-0.5RE alloy with the ultrafine grained (UFG) microstructure is expected to show huge enhancements in mechanical properties.

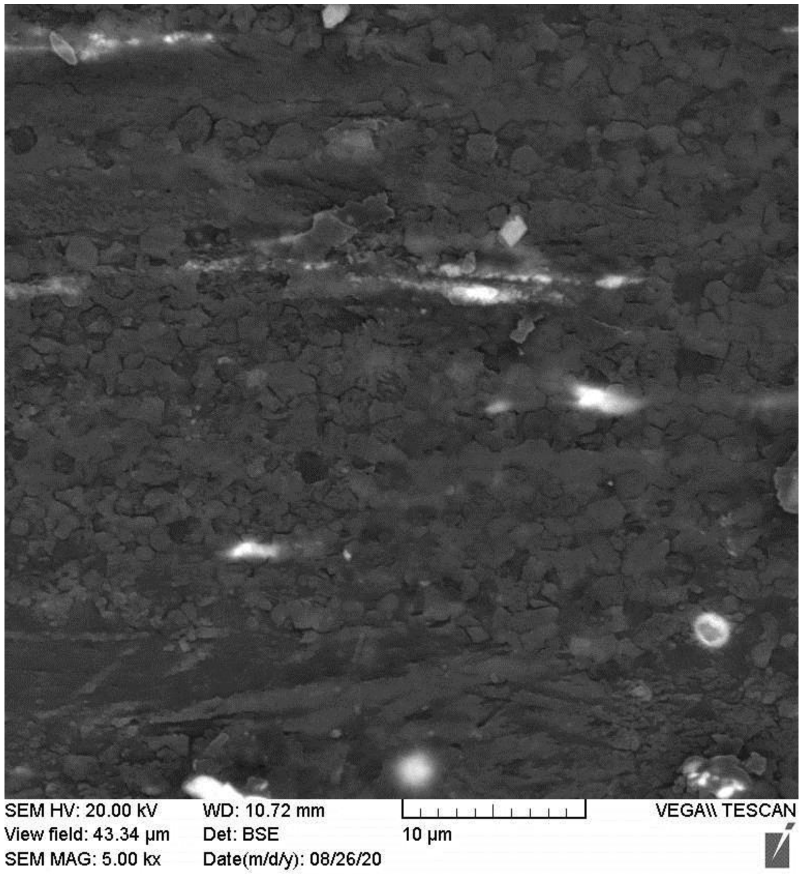


Figure 4: SEM image of the extruded Mg-0.5Zn-0.5Zr-0.5RE alloy.

Tensile engineering stress-strain curves of the as-cast alloys are illustrated in **Figure 5**, where the mechanical properties slightly enhanced by the addition of 0.5 wt% RE due to the moderate grain refinement. The tensile stress-strain curve of the extruded Mg-0.5Zn-0.5Zr-0.5RE alloy is also shown, where it can be observed that both ultimate tensile strength (UTS) and total elongation (%El) were significantly enhanced by the extrusion process. It is comprehensible that these remarkable improvements are related to the grain refinement through recrystallization mechanism and also dispersion of fine intermetallics during the extrusion process. This proves the favorable effects of the ultra grain refinement in the enhancement of the mechanical properties of magnesium alloys.

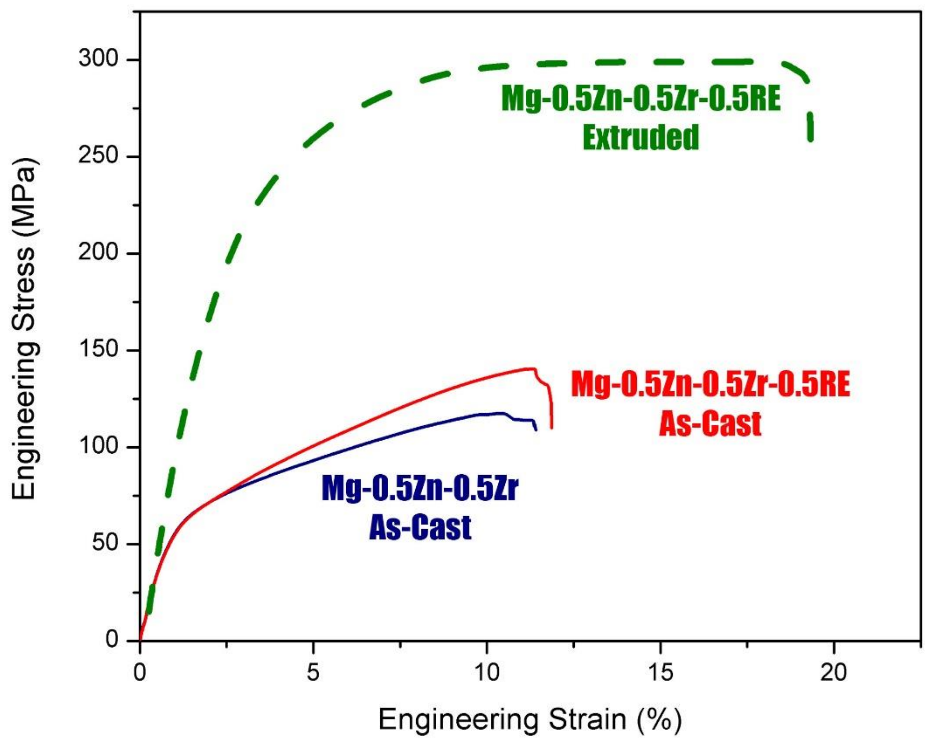


Figure 5: Tensile stress-strain curves.

4 CONCLUSIONS

In summary, grain refinement and enhancement of the tensile properties of Mg-Zn-Zr alloy via addition of rare earth (RE) elements at microalloying levels and utilization of hot extrusion process were investigated. The extrusion of the RE-containing Mg-Zn-Zr alloy replaced the coarse grain size of the as-cast ingot with an ultrafine grained (UFG) microstructure with the recrystallized grain size of ~ 1 µm. In this regard, the effect of the RE elements during solidification and the occurrence of the dynamic recrystallization (DRX) during hot extrusion process were found to be responsible. Accordingly, the ultimate tensile strength (UTS) and total elongation were significantly enhanced, which reveals the synergistic effect of RE addition and hot extrusion process on the enhancement of the mechanical properties of magnesium alloys.

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