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Effects of 0.5 wt% Ce addition on microstructures and mechanical properties of a wrought Mg−8Gd−1.2Zn−0.5Zr alloy

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ABSTRACT

Effects of 0.5 wt% cerium (Ce) addition on microstructures and mechanical properties of a wrought Mg−8Gd−1.2Zn−0.5Zr alloy were thoroughly investigated in this work. The results indicate that 0.5 wt% Ce addition has slight refinement on the as-cast grains and results in a lattice expansion of the dominant intermetallic phase Mg_3RE. After extrusion, 0.5 wt% Ce addition leads to higher level of recrystallization and finer dynamic recrystallization (DRX) grains and non-recrystallization stripes. In addition, there are much more dynamic precipitates on the DRX grain boundaries in the alloy with Ce addition, and the crystal structure ($Mg_{12}RE$) is different from those (Mg_5Gd) in the alloy with free Ce addition. Under peak-aging condition, 0.5 wt% Ce addition significantly changes the precipitates in the DRX grains from basal γ' phase to prismatic β' phase. As a result, the as-extruded Mg−8Gd−1.2Zn−0.5Zr alloy with 0.5 wt% Ce addition owns higher strength and more obvious precipitation hardening response than the alloy with free Ce addition.

1. Introduction

Mg alloys acted as the lightest metallic structural materials are increasingly interesting for lightweight applications in transportation to improve fuel efficiency, thus reducing climate-warming carbon dioxide emissions [1−3]. It is well known that RE-containing (RE denotes rare earth) magnesium alloys such as Mg−Gd−Zn−Zr systems have being receiving more and more attentions due to their remarkable age hardening response and high mechanical properties [4−9]. Therefore, many investigators have been paying attention to investigate the crystal structures of various precipitates precipitated during different aging stages, or to further improve the mechanical properties of the Mg−Gd based alloys by adding some other alloying elements [10,11]. To date, numerous high-strength Mg−Gd based alloys have been developed. For example, the Mg−11.7Gd−4.5Y−1Nd−1.5Zn−0.5Zr (wt%) alloy fabricated by hot-extrusion plus cold rolling and the hot-extruded Mg−10Gd−5.7Y−1.5Zn−0.65Zr (wt%) alloy both have over 500 MPa yield strength [11,12]. However, it has to be noted that these high strength alloys ordinarily contain very high RE c0ntent over 15 wt%, which directly results in expensive cost, thus limited their widely commercial applications.

Recently, Zheng et al. [13] reported that the Mg−8Gd−1Er−0.5Zr (wt%) alloy fabricated by rolling and ageing exhibits ultra-high strength, with the ultimate tensile strength (UTS) and yield strength (YS) of approximately 560 MPa and 518 MPa, respectively, at room temperature (RT). This strength is even higher than the conventional Mg−Gd based alloys with much higher RE content [11,12]. Although they adopted the expensive Erbium (Er), their work still indicates that little other RE addition into the Mg−8Gd based alloys can simultaneously accomplish high strength and relatively low cost. Nonetheless, the similar investigations are hitherto scarce. What is worse, the authors in Ref. [13] did not investigate the microstructures and mechanical properties of the Mg−8Gd−0.5Zr (wt%) alloy under same conditions, which resulted in poor understanding of the effects of the

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little Er addition. Therefore, to further develop low-cost and high-strength Mg–Gd based alloys, it is of overwhelming importance to investigate the effects of some other particularly cheap RE elements such as Cerium (Ce) on the microstructures and mechanical properties of the Mg–Gd based alloys.

Ce is one of the cheapest RE elements and was widely used in various Mg systems. It is reported that Ce addition can improve mechanical properties and increase alloy's extrusion property, resulting in good extruded sample without surface defects [14]. For example, Du et al. [15] reported that 0.5 wt% Ce addition clearly improved the strength of Mg–5.3Zn–0.2Ca (wt%) alloy because of the finer dynamic recrystallization (DRX) grains, the denser dislocations in non-recrystallized regions, and the much more fine precipitates. However, investigations of Ce addition into Mg–Gd based alloys were seldom reported. Additionally, as is well known, Zn addition into Mg–Gd alloys can enhance precipitation hardening response, finally resulted in good strength [16]. Thus, the Mg–8Gd–1.2Zn–0.5Zr (wt%) alloy was selected and then 0.5 wt% Ce was added into this alloy. Then, the microstructures of the studied two alloys in both as-cast and extrusion states were investigated. Afterwards, the tensile properties of the extruded samples were tested at both RT and high temperature (200 and 250 °C). Finally, the effects of 0.5 wt% Ce addition on the microstructures and mechanical properties of the Mg–8Gd–1.2Zn–0.5Zr alloy were analyzed and discussed.

2. Experimental procedures

Alloys with nominal compositions of Mg–8Gd–1.2Zn–0.5Zr (designated as A) and Mg–8Gd–1.2Zn–0.5Zr–0.5Ce (designated as B) were prepared by electric melting of pure Mg, Zn, Mg–20 wt% Ce and Mg–30 wt% Zr master alloys in a steel crucible under the protection of $CO_2 + 1.5$ vol% SF_6 mixture gas. The melt was fully stirred for approximately 15 min after its temperature reached to approximately 755 °C, and then kept static for approximately 20 min at this temperature. Afterwards, the melt was gradually cooled down to about 710 °C in half an hour. Finally, the melt was poured into a preheated iron mold with a diameter of 90 mm. The chemical compositions of the obtained alloys were examined by an inductivity coupled plasma atomic emission spectroscopy (ICP-AES), and the results are listed in Table 1. Before extrusion, the ingots were machined into billets with diameter of 82 mm. After preheated at 360 °C for approximately 2 h, the billets were directly extruded at the same temperature with an extrusion ratio of 7. Finally, aging treatments at 200 °C for 64 h were performed on the as-extruded samples.

Microstructures were characterized using optical microscopy (OM, Olympus–GX71), X–ray diffractometer (XRD, Bruker D8 FOCUS) at 40 kV and 40 mA with Cu K_α radiation ($\lambda = 0.15406$ nm), scanning electron microscopy (SEM, Hitachi S–4800) under an accelerating voltage of 10 kV, transmission electron microscopy (TEM, FEI Tecnai G^2 F20) equipped with energy-dispersive X–ray spectroscopy (EDS) operating at 200 kV and electron backscatter diffraction (EBSD) with a step size of 0.12 μm on FEI Nova 400 with EDAX-TSL system and an orientation imaging microscope (OIM) Analysis software. Specimens for OM and SEM observations were firstly grinded with different grades of SiC papers and then polished with Al_2O_3 suspension, finally slightly etched by a mixture of 5 ml acetic acid, 5 g picric acid, 10 ml H_2O, and 100 ml ethanol. The grain sizes of the as-cast samples presented in this work were tested using the Nano Measurer software. Thin foils with 3 mm in diameter for TEM observations were mechanically polished to approximately 20 μm and then ion–beam milled using a precision ion polishing system (Gatan 691) with cooling system by liquid nitrogen. The samples for EBSD observations were firstly prepared by standard metallographic technique, then polished using 0.08 μm colloidal silica for 25 min, and finally electro-polished at 20 V for approximately 80 s in an AC2 solution at −5 °C. To remove artifacts on the polished surface, samples were sonicated for 20 min after preparation in absolute ethanol.

Table 1
The chemical compositions of the studied two alloys.

Alloy	Composition (wt%)				
	Gd	Zn	Zr	Ce	Mg
A	8.12	1.19	0.47	/	Bal.
B	8.07	1.25	0.52	0.51	Bal.

Cylindrical tensile samples with gauge length and gauge diameter of 36 mm and 6 mm, respectively, were machined from extruded bars with the tensile direction parallel with extrusion direction (ED). Then, tensile tests were carried out on Instron 5869 tension tester at both RT and high temperatures (200 and 250 °C). The strain rate was set to be $1.0 \times 10^{-3}\ s^{-1}$ and the strain was measured by an extensometer attached to the sample. For each testing condition, at least three effective tests were carried out for every condition to confirm reproducibility and the values presented in this work are the averages. In addition, a 30 min holding was applied to balance the testing temperature before each high temperature test.

3. Results and discussion

3.1. Microstructures of the as–cast A and B samples

Fig. 1a and b shows OM images of the as-cast A and B alloys, respectively. Both microstructures are composed of α-Mg grains and semi-continuous reticular eutectic compounds. The average grain sizes of the as-cast A and B alloys are measured as 50 ± 6 μm and 41 ± 8 μm, respectively. This illustrates that Ce addition has slight grain refinement effect. Ce has very small solubility in Mg matrix [17]. During solidification, most Ce atoms will accumulate on the solid-liquid interface, thus resulted in greater constitutional supercooling. However, due to the limited Ce addition, the supercooling is also limited, subsequently slight grain refinement. In addition, comparison of Fig. 1a and b indicates that there are much more relatively small intermetallic particles on grain boundaries, which can be seen more clearly from the corresponding backscatter SEM images (Fig. 1c and d). Both alloys contain two kinds of intermetallic phases: one with blocky morphology and the other one with fishbone shaped morphology. But their corresponding XRD patterns (Fig. 2) suggest that both A and B alloys contains same dominant intermetallic phase, which can be reasonably indexed by Mg_3RE phase (a face-centered cubic structure) [18,19]. However, the diffraction peaks from intermetallic phase in B alloy locate at relatively lower degrees after 0.5 wt% Ce addition. Thus, Ce probably segregates in the Mg_3RE phase and results in a greater lattice parameter. To identify the crystal structure of the dominant intermetallic phase in both A and B alloy and to examine the Ce distribution in B alloy, TEM characterizations were conducted in this work. Fig. 3a and b shows the bright-field TEM (BF-TEM) micrographs of the fishbone shaped phase and the blocky phase, respectively, in the as-cast A alloy. The corresponding selected area electronic diffraction (SAED) patterns (Fig. 3c and d) illustrate that the fishbone shaped phase and the blocky phase both are Mg_3Gd, with the experimental lattice parameter of 0.7356 nm. In addition, the point EDS analysis results (Fig. 3e and f) demonstrate that these two phases contain some Zn although the Zn content in the blocky phase is relatively lower than that in the fishbone shaped phase.