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Formation and characterization of microstructure of as-cast Mg–6Gd–4Y–xZn–0.5Zr (x = 0.3, 0.5 and 0.7 wt.%) alloys

Y.J. Wu[a,b], C. Xu[a], F.Y. Zheng[a,b], L.M. Peng[a,b,*], Y. Zhang[a,b], W.J. Ding[a,b]

[a]National Engineering Research Center of Light Alloy Net Forming, Shanghai Jiao Tong University, Shanghai 200240, China
[b]State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University, Shanghai 200240, China

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ABSTRACT

Mg–6Gd–4Y–xZn–0.5Zr (x = 0.3, 0.5 and 0.7 wt.%) alloys were prepared via conventional ingot metallurgy (I/M) in this study. The as-cast microstructures of these alloys were established by X-ray diffraction (XRD) analyses, optical microscope (OM), scanning electron microscope (SEM) and transmission electron microscope (TEM) observations. Lamellar stacking order (SF) and 14H-type long period stacking order (LPSO) structure within α-Mg matrix are formed in the three as-cast alloys. The eutectic secondary phase is $(Mg,Zn)_{24}(Gd,Y)_5$ for the alloy containing 0.3 wt.% Zn, while, it is $(Mg,Zn)_3(Gd,Y)$ for the alloys containing 0.5 wt.% Zn and 0.7 wt.% Zn. Moreover, X phase-$(Mg,Zn)_{12}(Gd,Y)$ is formed in the latter two as-cast alloys.

1. Introduction

Mg–RE–Zn alloys have attracted much attention, due to solution strengthening, aging strengthening [1–7] and long period stacking order (LPSO) structures strengthening [8–26]. At present, the LPSO structures have been observed in Mg–RE–Zn/Cu/Ni/Al alloys (RE = Y, Dy, Ho, Er, Gd, Tb, Tm alloys) [8–26]. There are various types of LPSO structures in these alloys such as 6H, 10H, 14H, 18R, and 24R [8–23]. Among them, the Mg–Y–Zn and Mg–Gd–Zn alloys were focused on.

Recently, the eutectic secondary phase with 18R-LPSO structure in the as-cast Mg–Y–Zn alloy, such as $Mg_{97}Y_2Zn_1$, has been determined as $Mg_{10}YZn$ by Zhu et al. [8], instead of X-$Mg_{12}YZn$ by Luo et al. [9] which was commonly accepted in the early literatures. During subsequent solution treatment, the $Mg_{10}YZn$ phase with 18R-LPSO structure transforms into the X-$Mg_{12}YZn$ with 14H-LPSO structure [8]. At present, the eutectic secondary phase with fcc structure in the as-cast Mg–Gd–Zn(–Zr) alloy, such as $Mg_{97}Gd_2Zn_1$ [24], $Mg_{96.5}Gd_{2.5}Zn_1$ [17], $Mg_{96.82}Gd_2Zn_1Zr_{0.18}$ [16] and $Mg_{96.32}Gd_{2.5}Zn_1Zr_{0.18}$ [18,19] alloys, has been determined as β-$(Mg,Zn)_3Gd$ by Yamasaki et al. and Y.J. Wu et al. [16–19]. Interestingly, during subsequent solution treatment, the β-phase with fcc transforms into a novel X-phase with 14H-LPSO structure [17–19]. For clarity, the constitute phases of Mg–Y–Zn and Mg–Gd–Zn(–Zr) alloys were summarized in Table 1.

At present, no LPSO structure has been observed in both the as-cast or solution-treated Mg–Gd–Y–Zr alloys [2–7]. Interestingly, the addition of (0.5–2.3) wt.% Zn to the as-cast Mg–Gd–Y–Zr alloys results in the formation of LPSO structure [7,20,21,23,24]. However, there are no reports on the influence of addition of trace amount of Zn (Zn = 0.3, 0.5 and 0.7 wt.%) to the microstructure of Mg–6Gd–4Y–0.5Zr alloy. Whether LPSO structure forms in these alloys is worthwhile to investigate. This paper mainly reports the formation and characterization of microstructure of the as-cast Mg–6Gd–4Y–

* *Corresponding author at:* National Engineering Research Center of Light Alloy Net Forming, Shanghai Jiao Tong University, Shanghai 200240, China. Tel.: +86 21 54742911; fax: +86 21 34202794.
E-mail address: plm616@sjtu.edu.cn (L.M. Peng).

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Table 1 – Phase constitutes of Mg–Y–Zn(–Zr) and Mg–Gd–Zn(–Zr) alloys.

System	Alloy (at.%)	Condition	Phase constitute
Mg–Y–Zn	$Mg_{97}Y_2Zn_1$[8]	F (I/M)	α-Mg + $Mg_{10}YZn$(18R-LPSO) + LPSO within matrix
	$Mg_{96}Y_3Zn_1$[8]	T4	α-Mg + X-$Mg_{12}YZn$(14H-LPSO) + LPSO within matrix 18R-LPSO → 14H-LPSO
Mg–Gd–Zn(–Zr)	$Mg_{97}Gd_2Zn_1$[24]	F (I/M)	α-Mg + β-$(Mg,Zn)_3Gd$(fcc) +14H-LPSO within matrix
	$Mg_{96.5}Gd_{2.5}Zn_1$[17]		
	$Mg_{96.32}Gd_{2.5}Zn_1Zr_{0.18}$[18,19]	T4	α-Mg + X-$g_{12}GdZn$(14H-LPSO) +14H-LPSO within matrix (fcc → 14H-LPSO)
	$Mg_{96.82}Gd_2Zn_1Zr_{0.18}$[16]		

xZn–0.5Zr (x = 0.3, 0.5 and 0.7 wt.%) alloys, especially the stacking faults (SF), 14H-type LPSO structure within Mg matrix and lamellar X-Mg_{12}(Gd,Y)Zn phase in grain boundaries.

2. Experimental Procedures

The studied alloys were Mg–6Gd–4Y–xZn–0.5Zr (x = 0.3, 0.5 and 0.7 wt.%). The ingots were prepared via conventional ingot metallurgy (I/M). Pure Mg and Zn, and Mg–25%Gd (wt.%) and Mg–25%Y (wt.%) and Mg–30%Zr (wt.%) master alloys were needed and preheated to about 523 K in a baking oven. They were melted in an electric resistance furnace with a mild steel crucible under a protective gas (0.3% SF_6 and 99.7% CO_2). Pure Mg was put and melt in the electric resistance furnace; the two master alloys of Mg–25%Gd (wt.%) and Mg–25%Y (wt.%) and pure Zn metal were put and melted at about 993 K, in sequential order; Mg–30%Zr (wt.%) was added into and melted at about 1053 K. After adding of flux named JDMJ including $MgCl_2$, KCl, $BaCl_2$, NaCl, CaF_2, MgO and foamer, which was invented by Zhai C.Q. [27] and refining for 10 min at 1023 K, the melt was held for 20 min. Finally, the melt was poured at about 1013 K into the mild steel mold preheated to 473 K. The alloy ingots were cooled in air, cut into small specimens. Actual chemical compositions of these alloys were determined by an inductively coupled plasma analyzer (PerkinElmer, Plasma 400).

Phase analysis was carried out by X-ray diffraction (XRD, Rigaku D/max 2550 V) with copper foil target, and scanning speed of 1°/min, and start angle of 10° and end angle of 80°. XRD analyses were made by MDI Jade 5.0 software. To characterize the microstructure, specimens, etched by 4% HNO_3 and 96% ethanol, were observed by the Zeiss reversal optical microscope (OM, Axio Observer A1) and a scanning electron microscope (SEM, FEI SIRION 200) at 5–20 kV equipped with an Oxford energy disperse X-ray spectrometer (EDS). For further study the microstructure and structure, transmission electron microscope (TEM) observations were carried out. The thin foils were thinned by a twin jet electropolishing in a solution of 5% $HClO_4$ and 95% ethanol under conditions of 20 mA, 75 V and 233 K and then by low energy beam ion milling with the incidence angle of 4° and the voltage of 3.5 V for 0.5 h. TEM observations were performed using a JEOL-2010 operating at 200 kV equipped with EDS.

3. Results and Discussion

Actual chemical compositions of GWZ64.3K, GWZ64.5K and GWZ64.7K alloys were listed in Table 2. It shows that the actual chemical compositions of GWZ64.3K, GWZ64.5K and GWZ64.7K alloys are Mg–6.09Gd–4.04Y–0.34Zn–0.51Zr, Mg–6.00Gd–3.98Y–0.48 Zn–0.49Zr and Mg–6.08Gd–4.12Y–0.75Zn–0.53Zr, respectively. Therefore, the differences between the actual and the designed are little, which satisfy the need.

Fig. 1 shows XRD patterns of the as-cast Mg–6Gd–4Y–xZn–0.5Zr (x = 0.3, 0.5 and 0.7 wt.%) alloys. It can be observed that typical α-Mg peaks are present in the three alloys. Besides, $(Mg,Zn)_{24}(Gd,Y)_5$ (composition will be shown in SEM–EDS results below) phase occurs in the Mg–6Gd–4Y–0.3Zn–0.5Zr alloy. It is a $Mg_{24}Y_5$-type phase, which has bcc structure with lattice parameter of 2.24 nm in the Mg–Gd–Y–Zr alloys [2]. While, interestingly, $(Mg,Zn)_3(Gd,Y)$ and Mg_{12}(Gd,Y)Zn phases (compositions will be shown in SEM–EDS results below) occur in both the Mg–6Gd–4Y–0.5Zn–0.5Zr and Mg–6Gd–4Y–0.7Zn–0.5Zr alloys, due to higher Zn content (≥0.5 wt.%). $(Mg,Zn)_3(Gd,Y)$ is a Mg_3Gd-type phase, which has DO_3 structure with lattice parameter of 0.74 nm in the Mg–Gd–Zr alloys [16–19].

Fig. 2 shows the OM images of the typical as-cast Mg–6Gd–4Y–xZn–0.5Zr (x = 0.3, 0.5 and 0.7 wt.%) alloys. One eutectic secondary phase, is distributed at grain boundaries in the Mg–6Gd–4Y–0.3Zn–0.5Zr alloy; while, two phases are distributed at grain boundaries of the Mg–6Gd–4Y–0.5Zn–0.5Zr and Mg–6Gd–4Y–0.7Zn–0.5Zr alloys. Moreover, lamellae are formed within α-matrix in the three alloys.

Fig. 3(a)–(f) shows SEM images and EDS spectra of the α-Mg, $(Mg,Zn)_{24}(Gd,Y)_5$, lamellae and the square-shaped phase in the as-cast Mg–6Gd–4Y–0.3Zn–0.5Zr alloy. The compositions of the α-Mg and eutectic secondary phase were established to be Mg–1.46 ± 0.1 at.% Y–1.06 ± 0.1 at.% Gd–0.3 ± 0.1 at.% Zn [Fig. 3(c)] and Mg–6.07 ± 0.1 at.% Y–8.34 ± 0.1 at.% Gd–1.92 ± 0.1 at.% Zn [Fig. 3(d)]. Therefore, the eutectic secondary phase is $(Mg,Zn)_{24}(Gd,Y)_5$, which is consistent with the result of XRD (Fig. 1). While, the composition of lamellae within α-matrix is Mg–4.64 ± 0.1 at.% Y–1.93 ± 0.1 at.% Gd–3.66 ± 0.1 at.% Zn [Fig. 3(e)], where the contents of Gd and Y are between those of the α-Mg and $(Mg,Zn)_{24}(Gd,Y)_5$ phase. It can be concluded that the elements of Gd and Y are enriched in

Table 2 – Actual chemical compositions of GWZ64.3K, GWZ64.5K and GWZ64.7K alloys.

No	Alloy	Composition/wt.% (at.%)
1	GWZ64.3K ($Mg_{97.44}Y_{1.20}Gd_{1.01}Zn_{0.12}Zr_{0.15}$)	Mg–6.09Gd–4.04Y–0.34Zn–0.51Zr ($Mg_{97.47}Y_{1.21}Gd_{1.03}Zn_{0.14}Zr_{0.15}$)
2	GWZ64.5K ($Mg_{97.47}Y_{1.20}Gd_{1.02}Zn_{0.20}Zr_{0.15}$)	Mg–6.00Gd–3.98Y–0.48Zn–0.49Zr ($Mg_{97.46}Y_{1.19}Gd_{1.01}Zn_{0.20}Zr_{0.14}$)
3	GWZ64.7K ($Mg_{97.35}Y_{1.20}Gd_{1.02}Zn_{0.29}r_{0.15}$)	Mg–6.08Gd–4.12Y–0.75Zn–0.53Zr ($Mg_{97.47}Y_{1.24}Gd_{1.03}Zn_{0.31}Zr_{0.16}$)