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Effect of Zn/Er weight ratio on phase formation and mechanical properties of as-cast Mg–Zn–Er alloys

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ABSTRACT

The results of X-ray diffraction (XRD), differential scanning calorimetry (DSC) and microstructure observations have shown that various Zn/Er weight ratio leads to different phase compositions in the investigated alloys. The composition range for the formation of icosahedral quasicrystalline (I-phase) and face-centered cubic phase (W-phase) in Mg-rich corner is obtained. The Zn/Er weight ratio adjusting from10 to 6 results in the formation of I-phase exclusively as main secondary phase in the as-cast Mg–Zn–Er alloys. However, when the ratio is less than 0.8, it is mainly contributed to the precipitation of W-phase. When the ratio is in the range from 1 to 4, I-phase coexisting with W-phase is inclined to be formed. The results of tensile tests suggest that the as-cast Mg–5Zn–5.0Er alloy with I-phase and W-phase possesses the highest ultimate strength (UTS). It is concluded that UTS and elongation rate (ER) of as-cast Mg–Zn–Er alloys with I-phase as the main secondary phase are excellent. The UTS of the alloys with coexistence of I-phase and W-phase show the tendency of increasing firstly then decreasing with the increasing volume fraction of W-phase. The UTS declines when W-phase completely replaces I-phase in the alloy.

1. Introduction

Magnesium alloys have attracted a great deal of attention as structural materials due to their advanced properties such as low density, high specific strength and rigidity. Luo et al. [1] reported that the icosahedral I-phase was found in Mg–Zn–RE alloys. Mg–Zn–Y alloys containing I-phase as a secondary solidification phase exhibited good mechanical properties at room temperature as well as at elevated temperature [2]. Generally, there are three kinds of ternary equilibrium phases in Mg–Zn–Y alloy system. They are W-phase ($Mg_3Zn_3Y_2$, cubic structure), I-phase (Mg_3Zn_6Y, icosahedral quasicrystal structure, quasi-periodically ordered), and Z-phase ($Mg_{12}ZnY$) [3–5].

It has been reported [6–10] that the content variation of rare earth elements (RE) and element Y in the alloys can influence the phase formation and mechanical properties greatly. Kondo et al. [11] prepared the stable I-phase over a wide composition range in the Zn–Mg–Y system. The formation range of I-phase was investigated in several compositions in which a part of the liquidus surface had been determined [12]. Some of the Mg–Zn–RE alloys in the Mg corner are of technological interest since they exhibit good creep resistance at elevated temperatures. The creep strength of Mg–Y binary alloy is significantly improved by the small alloying with zinc by only 0.02–0.04 mol.% [13]. The steady creep rate of Mg–5.0Zn–2.5Er exhibits more superior creep resistance in the value of $2.73 \times 10^{-9}\ S^{-1}$ at 175 °C/70 MPa in [14] compared with AE42 of $56 \times 10^{-9}\ S^{-1}$ in [15]. Padezhnova et al. [16] investigated the microstructures of slowly cooled alloys and constructed the Mg comer of the Zn–Mg–Y diagram. Lee et al. [17] investigated that the effect of Zn/Y weight ratio (from 1.8 to 10) on the formation of I-phase and W-phase in the Mg-rich composition range of as-cast Mg–Zn–Y alloys with total solute content (Zn and Y) was less than 10 wt.% (wt., weight). In the previous research of Mg–Zn–Y–Zr alloys with different Y content [6], it was suggested that the formation of I-phase and W-phase was closely related with Zn/Y ratio. Partial phase diagrams of Mg–Zn–Sm [18] and Mg–Zn–Dy [19] have also been studied. Li et al. [20] discovered that I-phase with average composition $Mg_{30.02}Zn_{58.94}Er_{11.04}$ (in at.%, atom percent) and W-phase with average composition $Mg_{37.40}Zn_{38.20}Er_{24.40}$ formed in the as-cast Mg–Zn–Er alloy. However, phase diagrams in the Mg-rich region of as-cast Mg–Zn–Er system involving I-phase and other stable phase have not yet been constructed.

Therefore, the present work is designed to investigate the effect of Zn/Er weight ratio on microstructure and mechanical properties of as-cast Mg–Zn–Er alloys and discuss the phase formation range for I-phase and W-phase in Mg-rich corner of the alloys.

2. Experimental procedures

The alloys investigated in the present investigation are classified into three groups according to the addition of 3 wt.%, 5 wt.%

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and 7 wt.% Zn. Meanwhile, fixing the addition of Zn, the Zn/Er ratio value was changed to 0.8, 1, 2, 4, 6, 8 and 10 by adjusting the addition of Er. The nominal compositions and actual chemical compositions of the alloys were shown in Table 1. In this study, the different composition as-cast alloys were prepared by melting 99.9 wt.% magnesium, 99.9 wt.% zinc and binary Mg–20 wt.% Er master alloy in electric resistance furnace under anti-oxidizing flux. Holding for 30 min at 740 °C, the melt was poured into graphite mold which was preheated at 400 °C, and then cooled in the atmosphere, finally molten alloys were cast into rectangle ingots with 100 × 30 × 200 mm.

The microstructures of as-cast specimens were observed by scanning electron microscopy (SEM, HATACHI S3400N) and transmission electron microscopy (TEM, JEOL JEM 2100). The specimens for SEM observation were mechanically ground, polished, then etched with a solution of 4 vol.% (vol., volume fraction) nitric acid and 96 vol.% ethanol for several seconds. The specimens for TEM observation were firstly produced into a disc with a diameter of 3 mm by wet grinding, and further thinned by a twin-jet polishing machine in a solution of 80 vol.% methanol and 20 vol.% nitric acid at ∼20 V and 243 K. At last, the thinned disc specimens were performed on ion beam etching machine with an incident angle less than 10°. The chemical composition analysis of the ingots was conducted by X-ray fluorescence spectrometer (XRF, Magix PW2403). The phase identification was performed by X-ray diffraction (XRD, D/MAX-3C). The differential scanning calorimetry (DSC, NETZSCH-449C) measurement was confirmed under flowing argon atmosphere with a heating rate of 10 K min^{-1}, and characteristic temperature points of I-phase and W-phase existing in 20–30 mg specimens placed in an alumina crucible were determined. The tensile bars with a gauge length of 25 mm and 5 mm in diameter were machined from the alloys. Tensile experiments were conducted on the tensile testing machine (CSS-3902) with the constant strain rate of 1 mm min^{-1} at room temperature.

3. Results and discussion

3.1. Phase analysis

Table 1 shows phase constitutes of the as-cast alloys. It is suggested that the group of alloys containing 7 wt.% Zn include I-phase as a main secondary phase when the Er addition changing from 0.7 to 1.17 wt.%, correspond to Zn/Er at a weight ratio of 10, 8 and 6, respectively. However, when the addition of Er changes from 1.75 to 7.0 wt.% (the corresponding Zn/Er weight ratio is 4, 2 and 1), the main secondary phases of the alloys include I-phase and W-phase. Furthermore, the addition of element Er reaching 8.75 wt.% (Zn/Er weight ratio is 0.8) leads to a formation of W-phase as an exclusively secondary phase in the as-cast alloy. Additionally, when Zn/Er weight ratio is lower than 0.8 the element Er mostly forms W-phase, and the increasing volume fraction of W-phase can be attributed to that more Zn and Er are added in Mg–Zn–Er alloys. It has also been reported [2] that with increasing Y content, the grain boundaries were coarsened by lots of Mg–Zn–Y phases. The similar tendency reflecting the effect of Zn/Er weight ratio on phase formation in as-cast Mg–Zn–Er alloys is also found in the group alloys containing 3 wt.% and 5 wt.% Zn.

Fig. 1 shows three typical XRD patterns of the as-cast Mg–7Zn–1.17Er, Mg–7Zn–3.5Er and Mg–7Zn–8.75Er alloys, respectively. It is suggested that the phase compositions of as-cast alloys are different basically depending on the Zn/Er weight ratio. When the Zn/Er weight ratio is 0.8 in Mg–7Zn–8.75Er alloy, it is mainly contributed to the precipitation of W-phase; when the ratio is 2 in Mg–7Zn–3.5Er alloy, I-phase coexisted with W-phase is inclined to be formed; however, the ratio adjusting to 6 results in a formation of I-phase exclusively as a main secondary phase in the as-cast Mg–7Zn–1.17Er alloys. Consequently, the composition range for the formation of I-phase and W-phase in the Mg-rich corner has been obtained. The similar find has been reported in Mg–Zn–Y alloys. Xu et al. [6] have been reported that the addition of Zn/Y ratio (in wt.%) higher than 4.38 led to the precipitation of I-phase mostly in Mg–Zn–Y alloys. Further investigation suggested that W-phase formed as the ratio of Zn/Y lower than 1.10; both of I-phase and W-phase came into being at the ratio of Zn/Y between 1.1 and 4.38 in Mg–Zn–Y alloys. In conclusion, it is indicated that the formation of I-phase and W-phase is closely related with a ratio of Zn/RE (standing for Y or Er) addition in the Mg–Zn–Er alloys as well as in the Mg–Zn–Y alloys.

Fig. 2 shows the DSC results of the as-cast alloys Mg–7Zn–1.17Er, Mg–7Zn–3.5Er and Mg–7Zn–8.75Er, respectively. It is found that three endothermic peaks occur at 540 °C, 460 °C and 340 °C, which are standing for the presence of W-phase, I-phase

Table 1
Nominal composition, actual composition and main secondary phase of as-cast Mg–Zn–Er alloys in the investigation.

Zn (wt.%)	Nominal composition (wt.%)	Actual composition (wt.%)			Zn/Er Ratio	Main secondary phase
		Mg	Zn	Er		
3Zn	1-Mg–3Zn–0.3Er(10)	Bal.	3.1	0.3	10.3	I-phase
	2-Mg–3Zn–0.38Er(8)	Bal.	3.2	0.4	8.0	
	3-Mg–3Zn–0.5Er(6)	Bal.	3.0	0.49	6.1	
	4-Mg–3Zn–0.75Er(4)	Bal.	3.2	0.78	4.1	I-phase and W-phase
	5-Mg–3Zn–2.5Er(2)	Bal.	3.0	1.3	2.3	
	6-Mg–3Zn–3.0Er(1)	Bal.	3.2	2.8	1.1	
	7-Mg–3Zn–3.8Er(0.8)	Bal.	3.0	3.7	0.81	W-phase
5Zn	8-Mg–5Zn–0.5Er(10)	Bal.	5.2	0.5	10.4	I-phase
	9-Mg–5Zn–0.63Er(8)	Bal.	5.0	0.60	8.3	
	10-Mg–5Zn–0.83Er(6)	Bal.	4.8	0.77	6.2	
	11-Mg–5Zn–1.25Er(4)	Bal.	5.1	1.3	3.9	I-phase and W-phase
	12-Mg–5Zn–2.5Er(2)	Bal.	4.9	2.1	2.3	
	13-Mg–5Zn–5.0Er(1)	Bal.	4.9	4.9	1.0	
	14-Mg–5Zn–6.25Er(0.8)	Bal.	4.9	6.0	0.82	W-phase
7Zn	15-Mg–7Zn–0.7Er(10)	Bal.	6.4	0.62	10.3	I-phase
	16-Mg–7Zn–0.88Er(8)	Bal.	6.9	0.85	8.1	
	17-Mg–7Zn–1.17Er(6)	Bal.	6.3	1.0	6.3	
	18-Mg–7Zn–1.75Er(4)	Bal.	7.0	1.7	4.1	I-phase and W-phase
	19-Mg–7Zn–3.5Er(2)	Bal.	6.8	3.3	2.1	
	20-Mg–7Zn–7.0Er(1)	Bal.	6.7	6.6	1.0	
	21-Mg–7Zn–8.75Er(0.8)	Bal.	6.9	8.6	0.80	W-phase