

(Y, Gd)H₂ phase formation in as-cast Mg-6Gd-3Y-0.5Zr alloy

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Abstract: In this work, a new (Y, Gd)H₂ precipitate was identified and systematically investigated in the as-cast Mg-6Gd-3Y-0.5Zr alloy by XRD, SEM with EDS, TEM with EDS techniques and thermodynamics analysis. Results show that the as-cast alloy contains α -Mg, Mg₂₄(Gd, Y)₅, and (Y, Gd)H₂ phase. The (Y, Gd)H₂ phase usually forms near the eutectic phase Mg₂₄(Gd, Y)₅ or in the α -Mg grains, displaying a rectangle-shape. The Mg₂₄(Gd, Y)₅ and (Y, Gd)H₂ phases crystallize in bcc and fcc structure, respectively, and the (Y, Gd)H₂ phase has a semi-coherent relationship with α -Mg matrix. The thermodynamics calculation results reveal that the hydrogen dissolved in the melt leads to the formation of hydrides. It is also found that the (Y, Gd)H₂ hydride can form directly from the liquid phase during solidification. Additionally, it can precipitate by the decomposition of Mg₂₄(Gd, Y)₅ phase due to absorbing hydrogen from the remaining melt.

Key words: Mg-6Gd-3Y-0.5Zr alloy; as-cast; microstructure; (Y, Gd)H₂ phase; formation mechanism

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1 Introduction

Mg-Gd-Y-Zr alloys have attracted increasing attention in the aerospace field because of their excellent mechanical properties in both room and elevated temperatures and excellent castability^[1,2]. The quality of Mg-Gd-Y-Zr alloy castings is closely related to melting condition, solidification process and humidity in the surrounding environment^[3,4]. It is well known that magnesium alloy melt has a strong hydrogen absorption capacity, and it is easy to form shrinkage porosity defects during the casting process^[5-7]. Therefore, degassing treatment during the melting process is essential. However, the formation of hydrides in rare earth magnesium alloys is still inevitable due to the high activity of rare earths^[8,9], and a cuboid REH₂ phase with a face-centered cubic structure is usually encountered in Mg-RE alloys except the RE-containing eutectic phases^[10]. Hydrides such as GdH₂^[11,12], NdH₂^[13,14], DyH₂^[10,15], etc. were reported in the literature.

However, some rare earth hydrides have not been found or accurately identified in Mg-Gd-Y-Zr alloys. Therefore, it is necessary to find and characterize the hydrides in the as-cast Mg-Gd-Y-Zr alloy.

Previous studies reported that the precipitation of hydrides had an adverse effect on the mechanical properties of Mg alloys^[14,16]. Kannan et al.^[17] pointed out that the hydrogen embrittlement effect was the main factor contributing to the reduced mechanical properties of the alloy due to the formation of hydrides. Yang et al.^[13] suggested that NdH₂ easily caused cracks and worsened the mechanical properties of the Mg-Nd alloys. Therefore, it is necessary to take measures to prevent the formation of hydrides for magnesium alloys. Recently, some researchers have investigated the formation mechanism of the hydrides. Huang et al.^[10] studied the precipitation behavior of RE hydride particles in Mg-RE (Dy, Gd, Y) alloys with different RE contents, and they considered that the high solid solution of RE in Mg matrix resulted in a higher REH₂ amount. Peng et al.^[11] found that the hydrogen-containing medium, compressive deformation stress, and Gd segregation were the prerequisites for GdH₂ formation. Zheng et al.^[12] suggested that the GdH₂ precipitates were produced during the alloy melting and casting process, and the growth of GdH₂ particles during the homogenization treatment process could be

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attributed to the inward diffusion of hydrogen generated by the decomposition of moisture on the surface of the sample. Yang et al.^[13] proposed that NdH₂ probably nucleated during the solidification process as a result of the reaction of Nd with hydrogen previously dissolved in Mg solution. Zhu et al.^[14] proposed that the formation of rare earth hydrides may be related to the hydrogen decomposition of the Mg-RE intermetallic phases during the solidification or high-temperature heat treatment process. Gan et al.^[15] pointed out that DyH₂ hydride could form during sample preparation at room temperature, such as mechanical polishing with water. However, as described above, the formation mechanisms of hydrides are still controversial, and further in-depth research is necessary. So, it would be interesting to get a deeper understanding of the origin of RE hydrides in Mg alloys, especially in Mg-Gd-Y-Zr alloys.

In the present work, the Mg-6Gd-3Y-0.5Zr alloy was prepared, and a new (Y, Gd)H₂ phase in the as-cast Mg-6Gd-3Y-0.5Zr alloy was identified and characterized by using SEM, TEM and HRTEM observations. Meanwhile, the corresponding formation mechanism was discussed.

2 Experimental procedure

The Mg-6Gd-3Y-0.5Zr (in wt.%) alloy was prepared using an electrical resistance furnace with the protection of a mixed gas atmosphere of CO₂ (99 vol.%) and SF₆ (1 vol.%) by using commercial pure Mg, Mg-30Gd (wt.%), Mg-30Y (wt.%) and Mg-30Zr (wt.%) master alloys as raw materials. After the raw materials were completely melted, the melt was degassed with high purity and dry argon gas (99.999%) for 30 min using a rotary degasser at 740 °C, and then the melt was held for 10 min to remove the residue. Finally, the surface of the melt was skimmed, and the alloy melt was gravity cast into a sand mould of $\Phi 100$ mm $\times$ 100 mm. The actual chemical composition of the alloy was measured by fluorescent spectrometer technique and detailed in Table 1.

Table 1: Chemical composition of as-cast Mg-6Gd-3Y-0.5Zr alloy used in this study (wt.%)

Gd	Y	Zr	Cu	Ni	Fe+Si	Mg
5.65	3.23	0.50	0.0266	0.0013	0.0126	Bal.

Phase analysis of the as-cast alloy was performed using the Smartlab (9 kW) X-ray diffractometer (XRD, Rigaku, Japan), and the scanning angle was 20°–90° with the scanning speed of 2°·min⁻¹. The microstructure and composition were observed and determined by a JSM-6510 scanning electron microscope (SEM, JEOL, Japan), a Tecnai G2 F30 transmission electron microscope (TEM, FEI, Netherland) and an energy dispersive X-ray spectrometer (EDS). Specimens for SEM observations were polished and etched with a mixed solution of 19 mL deionized water, 60 mL ethylene glycol, 20 mL glacial acetic acid and 1 mL nitric acid for 50 s. Specimens for TEM

observations were thinned to 50 μ m by grinding with SiC sandpapers and were punched to $\Phi 3$ mm discs. The final stage of thinning was performed by argon ion milling with an incident angle of 10° until perforation occurred.

3 Results and discussion

3.1 Microstructure

Figure 1 shows the XRD spectrum of the as-cast Mg-6Gd-3Y-0.5Zr alloy. It can be seen that the as-cast alloy contains two phases, α -Mg and Mg₂₄(Gd, Y)₅. The SEM microstructure of the as-cast Mg-6Gd-3Y-0.5Zr alloy is shown in Figs. 2(a) and 2(b). It can be seen that the network eutectic phases distributed in the α -Mg grain boundaries. According to the EDS results in Table 2 and XRD analyses, the eutectic phase is identified as Mg₂₄(Gd, Y)₅. Figures 2(c) and 2(d) are the TEM images of the eutectic phase. The selected area electron diffraction (SAED) pattern indicates that the Mg₂₄(Gd, Y)₅ phase has a bcc structure with a lattice parameter of $a=11.2$ nm, which is the same as that reported in previous works^[18, 19]. From Fig. 2(b), it also can be observed that there are some small particles with different shapes distributed around the eutectic phase and in the α -Mg grains. The EDS results of the particle phase are shown in Table 2. It can be clearly found that these particles are rich in Gd and Y elements, especially Y elements. However, the relevant phases are not detected in Fig. 1 due to the low sensitivity of XRD.

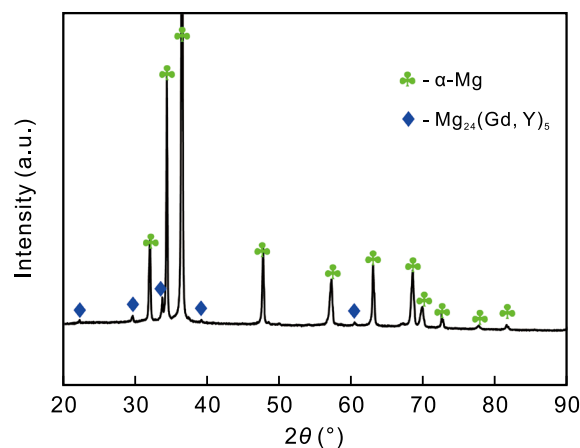


Fig. 1: XRD pattern of as-cast Mg-6Gd-3Y-0.5Zr alloy

A bright field TEM image of the particle phases is shown in Fig. 3(a). It can be seen that the particles are rectangular and the length is approximately 800 nm. The EDS analysis indicates that the particle phase is rich in Gd and Y elements, especially the Y element. Figures 3(b)–(d) show the SAED patterns of the particle phase in Fig. 3(a) along three different beam directions. The diffraction patterns provide an evidence for the hydride phase and display fcc symmetry along the $[\bar{1}12]$, $[01\bar{1}]$, $[10\bar{3}]$ axes, respectively. This particle can be indexed as (Y, Gd)H₂ phase according to the EDS and SAED analyses. It is known that Gd and Y elements belong to hcp crystal structure and have good alloying capability^[20]. The solid solubility of Gd in magnesium is greater than that of Y^[21]. Thus, the Y(Gd)

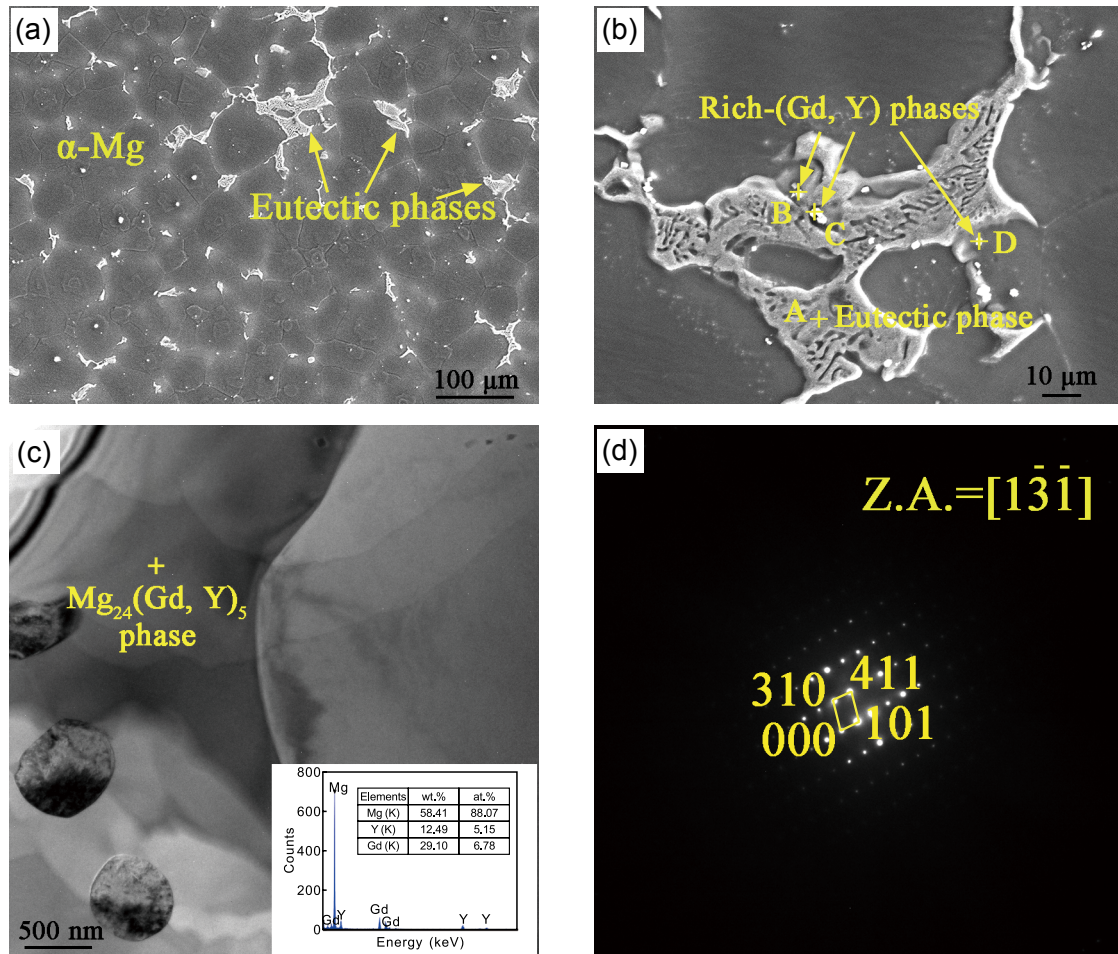


Fig. 2: SEM microstructure of as-cast Mg-6Gd-3Y-0.5Zr alloy (a and b); bright field TEM image of eutectic phase (c); and corresponding SAED pattern of eutectic phase in (c) and (d)

Table 2: EDS analysis results of spots marked in Fig. 2(b) (at.%)

Points	Gd	Y	Mg
A	5.28	5.98	88.74
B	23.72	54.65	21.63
C	24.12	56.84	19.04
D	11.71	57.38	30.91

substitutional solid solution is easily obtained. The precipitation of $(Y, Gd)H_2$ is more affected by Y element, and Y content is the majority in $(Y, Gd)H_2$. Figure 3(e) shows the interfacial structure between the $(Y, Gd)H_2$ phase and α -Mg matrix. It can be seen that the (111) plane of $(Y, Gd)H_2$ phase and (0001) plane of α -Mg phase are nearly parallel. The lattice misfit between the (111) $(Y, Gd)H_2$ and (0001) α -Mg is 13.24% according to Eq. (1), which satisfied the structure of the semi-coherent interface^[22, 23]:

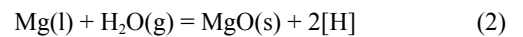
$$\delta = 2 (\alpha_{(Y, Gd)H_2} - \alpha_{Mg}) / (\alpha_{(Y, Gd)H_2} + \alpha_{Mg}) \quad (1)$$

where $\alpha_{(Y, Gd)H_2}$ and α_{Mg} represent the lattice constants of $(Y, Gd)H_2$ and Mg matrix.

3.2 Formation mechanism of $(Y, Gd)H_2$

Gd and Y usually exist as solid solution atoms in the α -Mg matrix or as intermetallic compounds to form eutectic phases,

which play a role of solid solution strengthening and second phase strengthening, respectively^[24]. As mentioned above, $(Y, Gd)H_2$ phases distributed around the eutectic phase and in the α -Mg grains are found in the as-cast Mg-6Gd-3Y-0.5Zr alloy. Most of the RE elements have a great tendency to absorb hydrogen at room temperature and quickly form hydride (REH_2) at elevated temperature, and those hydrides will not decompose until the temperature is higher than 1,000 K^[8, 25]. The H in the Mg melt mainly comes from the Mg liquid or the water vapor in the atmosphere. The main reaction is shown in Eq. (2):



According to the thermodynamic analysis of Luo et al.^[26] and Xu et al.^[27], the solid solubility of hydrogen in the Mg alloy melt can be expressed as:

$$N/P^{1/2} = 0.0882 \exp(-4987/T) \quad (3)$$

where N is the molar concentration of hydrogen, and P is the partial pressure of hydrogen. Taking $N=1$ and $P=1.01 \times 10^5$ Pa as the standard state, the standard dissolution free energy of hydrogen in Mg melt can be expressed as:

$$\Delta G_1^0 = -RT \ln K \quad (4)$$

where R is the gas constant ($R=8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$), and K is the equilibrium constant. The solute is dilute and according to the

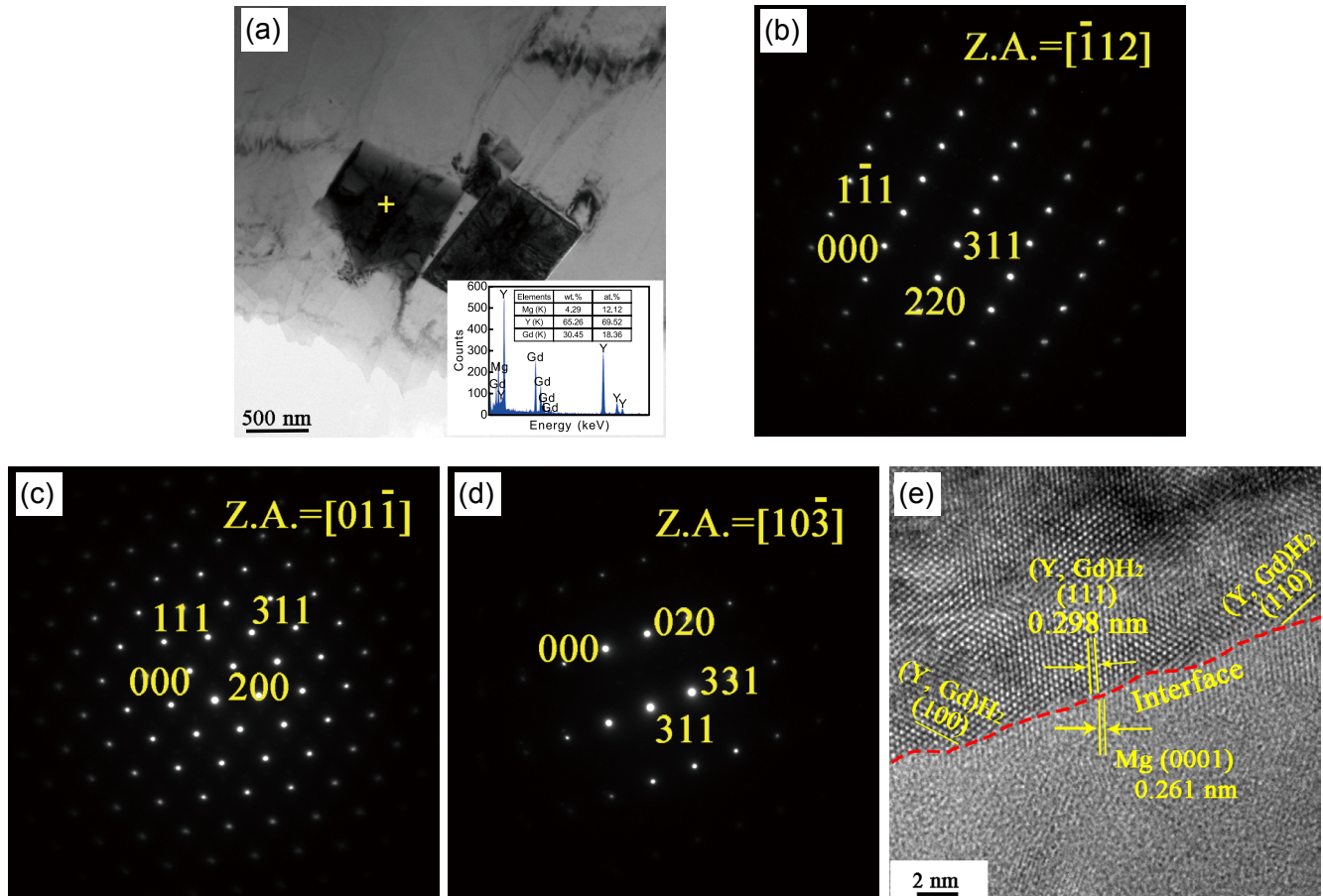


Fig. 3: Bright field TEM image of particle phase presented in as-cast Mg-6Gd-3Y-0.5Zr alloy (a); corresponding SAED patterns along three beam directions (b, c, and d); HRTEM image of (Y, Gd)H₂/α-Mg interface (e)

Sievert's law ^[27], $K=\alpha_{[H]}=0.0882\exp(-4987/T)$ can be obtained. Thus, Eq. (4) can be expressed as follows:

$$\Delta G_1^0 = -RT\ln K = -RT\ln[0.0882\exp(-4987/T)] \quad (5)$$

$$= 41462 + 20.19T$$

According to the Gibbs-Helmholtz equation $\Delta G_m = \Delta H_{298} - T\Delta S_{298}^0 + \int_{298}^T \Delta C_p dT - T \int_{298}^T \Delta C_p \cdot dT/T$ ^[28] and the thermodynamic data of the reaction substance presented in Ref. [29], the standard free energy change for the reaction between Mg melt and H₂O(g) [Eq. (2)] can be easily calculated and described as:

$$\Delta G_2^0 = -278782 - T\lg T + 98.34T \quad (6)$$

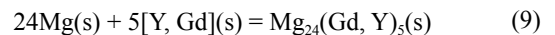
When $T=1,033$ K, $\Delta G_2^0 = -180,310$ J·mol⁻¹, which suggested that the reaction of Eq. (2) can proceed spontaneously at the standard state condition and has a strong reaction driving force. When $P_{H_2}=1.01 \times 10^5$ Pa and $T=1,033$ K, $N=7.06 \times 10^{-4}$ mol can be obtained from Eq. (3). In the actual condition, when $P_{H_2O}=10^3$ Pa, $\alpha_{[H]}=N=7.06 \times 10^{-4}$ mol, $\alpha_{Mg}=0.997$ mol, $\alpha_{H_2O}=P_{H_2O}/1.01 \times 10^5$ Pa = 0.0297 mol. The actual free energy change for the reaction between Mg melt and H₂O(g) [Eq. (2)] can be expressed as:

$$\Delta G_2 = \Delta G_2^0 + RT\ln J = \Delta G_2^0 + RT\ln[\alpha_{[H]}^2/(\alpha_{Mg} \times \alpha_{H_2O})] \quad (7)$$

$$= -180,310 + 8.314 \times 1,033 \ln[(7.06 \times 10^{-4})^2/(0.997 \times 0.0297)]$$

$$= -274,715 \text{ J} \cdot \text{mol}^{-1}$$

Therefore, the reaction of Eq. (2) can still proceed spontaneously, and the hydrogen dissolved in the melt is the cause of hydrides formation. According to Ref. [30], the reaction between the Y, Gd, H and Mg can be described as Eqs. (8) and (9). The corresponding ΔG -temperature graph of (Y, Gd)H₂ and Mg₂₄(Gd, Y)₅ can be obtained in Fig. 4 according to the previous works ^[31, 32] and the thermodynamic calculation method presented in Ref. [33].



It can be seen that the changes of Gibbs energy (ΔG) of (Y, Gd)H₂ and Mg₂₄(Gd, Y)₅ are negative below 900 K, which confirms that the (Y, Gd)H₂ and Mg₂₄(Gd, Y)₅ phase can coexist. When the solidification temperature decreases, the (Y, Gd)H₂ phase can nucleate directly from the liquid phase due to its higher melting point ^[34]. Thus, the (Y, Gd)H₂ phase forms in the α-Mg grains [Fig. 2(b)]. When the temperature drops to the eutectic temperature (about 800 K), the eutectic phase Mg₂₄(Gd, Y)₅ forms, and the ΔG of (Y, Gd)H₂ is lower than that of Mg₂₄(Gd, Y)₅. Therefore, the (Y, Gd)H₂ phase can also occur due to the decomposition of Mg₂₄(Gd, Y)₅ phase by absorbing hydrogen from the remaining melt, and some (Y, Gd)H₂ phases are distributed around the eutectic phase

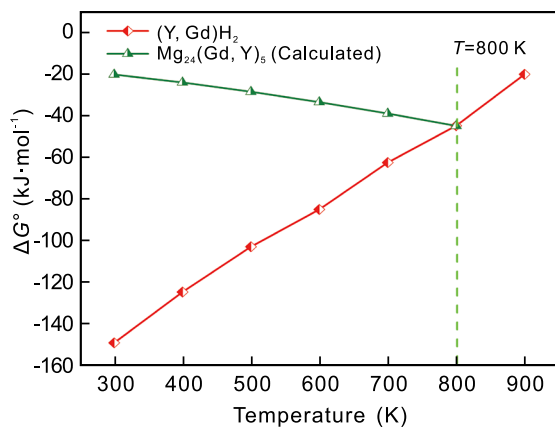


Fig. 4: ΔG° - T graph of $(Y, Gd)H_2$ and $Mg_{24}(Gd, Y)_5$ [31, 33]

$Mg_{24}(Gd, Y)_5$, as shown in Fig. 2(b). Similar results have been reported by Zhu et al. [14] in their investigation on the RE hydrides using binary Mg-La, Mg-Ce and Mg-Nd alloys.

4 Conclusions

(1) The microstructure of as-cast Mg-6Gd-3Y-0.5Zr alloy mainly consists of α -Mg, $Mg_{24}(Gd, Y)_5$ eutectic phase and $(Y, Gd)H_2$ phase. The $(Y, Gd)H_2$ phases are distributed around the eutectic phase $Mg_{24}(Gd, Y)_5$ and in the α -Mg grains.

(2) The $Mg_{24}(Gd, Y)_5$ phase has a bcc structure with a lattice parameter of $a=11.2$ nm. The $(Y, Gd)H_2$ phase with a rectangle-shape has a fcc structure, and the interfacial structure between the $(Y, Gd)H_2$ phase and α -Mg matrix shows semi-coherent relationship.

(3) The hydrogen dissolved in the melt causes the formation of hydrides. The formation mechanism of $(Y, Gd)H_2$ phase can be attributed to the direct nucleation in liquid phase and the decomposition of $Mg_{24}(Gd, Y)_5$ phase by absorbing hydrogen from the remaining melt.

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