Effect of Solution Treatment on the Microstructure and Mechanical Properties of Sand-Cast Mg–9Gd–4Y–0.5Zr Alloy

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Abstract Sand-cast Mg–9Gd–4Y–0.5Zr (wt%) alloy was solution-treated at 500–565 °C in the time range of 0.5–30 h in air or vacuum to investigate its microstructure evolution and mechanical properties. The results showed that solution treatment temperature had a significant influence on the dissolving rate of eutectic phase and grain growth. Taken both of them into consideration, 510–520 °C was considered to be the optimum solution treatment temperature range for this alloy. It should be noted that the trace (0.4–0.9 vol%) and insoluble cuboid-shaped phase precipitated during solution treatment was identified to be YH₂, of which the hydrogen was thought to come from both the melting and solution heating process. In addition, the 3D morphology and dissolving process of Mg₂₄(Gd,Y)₅ eutectic phases in the as-cast alloy were also discussed via in-situ observation under X-ray tomography.

KEY WORDS: Mg–Gd–Y–Zr alloy; Solution treatment; Microstructure; Rare-earth hydride

1 Introduction

The combination of low weight, good strength and castability makes Mg alloys to be the promising engineering materials for the automotive and aviation industries [1–4]. Among them, Mg–RE alloys with addition of rare-earth elements have received considerable interest due to their potential for achieving higher strength and better creep resistance at

elevated temperatures [5]. Especially, Mg–Gd–Y system alloys were thought as the most promising heat-treatment-strengthening magnesium alloys because of the high solubility (23.49 and 12.5 wt%) of gadolinium and yttrium in magnesium [6]. Currently, the aging precipitation behavior and its strengthening mechanism of the alloys have been widely investigated. It was reported that the dominant strengthening precipitates were metastable β' and β'', which played a key role in impeding basal slip of the dislocations [7, 8]. The precipitation sequence was also cleared and reported, i.e. S.S.S.S → β''(D0₁₉) → β'(cbco) → β1(fcc) → β(fcc) [9].

However, to our knowledge, only a few of researches have been technically concerned on the microstructural evolution and its effect on the mechanical properties during solution treatment of Mg–Gd–Y–Zr alloy. Gao et al. [10] investigated the microstructure evolution of Mg–15Gd–5Y–0.5Zr alloys both in the as-cast condition and after solution treatment from 500 to 540 °C in the time range of 0.5–72 h. It was observed that the skeleton-like Mg₅(Gd,Y) phase in the as-cast alloy experienced a spheroidized process as dissolving into the matrix during solution treatment.

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Moreover, it was thought that the cuboid-shaped phase with composition of $\text{Mg}_2\text{Y}_3\text{Gd}_2$ may occur during solution treatment. However, because of its trace amount and non-unique composition detected by EDS, the cuboid phase was identified as hydride [11], oxide [12] and Mg–RE compounds with different stoichiometric ratios [7, 10] in different investigations. Therefore, the real identity of this cuboid phase was still under dispute. The present study was conducted to investigate the solution treatment behavior of Mg–9Gd–4Y–0.5Zr (wt%) (GW94) alloy from 500 to 565 °C, focusing on the grain growth and eutectic dissolution. At the same time, the composition of the cuboid phase and its characterizations were also discussed.

2 Experimental Procedure

GW94 alloy was prepared from high-purity magnesium, gadolinium, yttrium and Mg–30Zr (wt%) master alloys by melting in an electric resistance furnace at about 780 °C under protection with an anti-oxidizing flux and pouring into a sand mold of tensile test bars [13]. The chemical composition of the alloy determined by an inductively coupled plasma atomic emission spectrum is Mg–9.4Gd–3.76Y–0.41Zr (wt%).

The as-cast specimens with dimension of $\Phi 12 \text{ mm} \times 7 \text{ mm}$ were cut from the cast bars and underwent solution treatment at different temperatures in the time range of 2–16 h. Solution treatment temperatures of 500, 510, 520 and 530 °C selected preliminarily determined on the basis of calculated phase diagram of Mg–xGd–4Y–0.5Zr (wt%) alloy system [6] and literature data [7, 10]. In addition, the temperature of 565 °C approaching to the solidus temperature was also employed for investigating whether the cuboid phase can be removed by solution treatment. For identifying the cuboid phase, solution treatment under vacuum atmosphere was conducted by sealing the sample into a vacuum quartz glass tube with vacuum degree of $\sim 10^{-6}$ torr. The detailed solution treatment conditions in this work are summarized in Table 1.

Optical microscopy (OM) and scanning electron microscope equipped with energy disperse spectroscopy

(SEM, Philips XL30ESEM-FEG/EDAX) were utilized to observe microstructures. The specimens for OM observation were etched in a 4 vol% nital after mechanical polishing to reveal the grain boundaries. The mean grain size, d , was measured by the linear intercept method using the equation $d = 1.74 L$, where L is the linear intercept [13]. The average value of the grain sizes with error bars was obtained by evaluating 100 grains.

The major phases of the alloy in different conditions were identified by X-ray diffraction (XRD) (RigakuD/max 2400 X-ray diffractometer) with CuK_α radiation. To describe the morphology evolution of eutectic phase during solution treatment, X-ray tomography (XRT) with a resolution of 1 μm (Versa XRM-500) was carried out on the samples with a size of 0.5 mm $\times$ 0.8 mm $\times$ 0.9 mm in both the as-cast and the as-solutionized states. The images were detected on a moving CCD camera as the sample was rotated. The output from the CCD was fed through a reconstruction algorithm [14] producing binary grayscale bitmap and RGB color of the cross-section, of which brighter place indicates having higher Z (relative atomic mass).

Time-of-flight secondary ion mass spectrometry (TOF–SIMS) (ION TOF–SIMS 5) with a 30 keV Bi⁺ primary ion source was utilized to characterize H, Mg, Gd, and Y elements for the solutionized GW94 alloy. Second ion mass spectra from multiple projectiles were generated simultaneously (instrument and target surface conditions remained constant through the measurement). The system pressure for the ToF–SSIMS measurements was $\sim 3 \times 10^{-7}$ torr or lower. The sample for SIMS has a dimension of 10 mm $\times$ 10 mm $\times$ 10 mm, and the observed fresh surface without contamination plane was prepared by iron milling. The area of analysis was 100 $\mu\text{m} \times 100 \mu\text{m}$. Data were acquired for both positive and negative secondary ions. The differences between the expected and observed masses for both positive and negative ions after calibration were less than 20 ppm.

The samples for tensile tests were standard casting test bars with gauge dimension of $\Phi 12 \text{ mm} \times 72 \text{ mm}$. Tensile tests were performed at initial strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ and room temperature.

Table 1 Designed solution treatment conditions for GW94 Mg alloy

Temperature	Time					Atmosphere
500 °C	2 h	4 h	6 h	8 h	16 h	Air
510 °C	2 h	4 h	6 h	8 h	16 h	Air
520 °C	2 h	4 h	6 h	8 h	16 h	Air
530 °C	2 h	10 h	6 h	8 h	16 h	Air
565 °C	2 h	6 h	30 h			Air
510 °C	0.5 h					Vacuum
520 °C	6 h					Vacuum

3 Results and Discussion

3.1 As-Cast Microstructure

Typical microstructures and corresponding EDX analysis results of the GW94 alloy in the as-cast condition are shown in Fig. 1. The as-cast alloy had fine grains with an average size of $\sim 47 \mu\text{m}$, and consisted of approximate equiaxed dendrites of α -magnesium matrix (arrow 1 in Fig. 1b) surrounded by white skeleton and some cuboid of

Fig. 1 OM **a** and SEM **b** images of the as-cast GW94 alloy and the EDX results of the skeleton phase **c**, the cuboid-shaped phase **d**, corresponding to A and B points from **b**, respectively

non-equilibrium eutectic phase (arrows 2 and 3 in Fig. 1b). The semi-quantitative EDX results in Fig. 1c and d indicate that the skeleton phase and the cuboid phase (positions A and B in Fig. 1b) have approximate stoichiometric composition of $\text{Mg}_{24}(\text{Gd},\text{Y})_5$ and $\text{Mg}_5(\text{Gd},\text{Y})$, respectively. According to the phase diagram of Mg–Gd–Y–Zr alloy calculated by Guo et al. [6] and the diffraction pattern reported by Gao et al. [5], the cuboid phase in the as-cast alloy was thought to be $\text{Mg}_5(\text{Gd},\text{Y})$ with fcc crystal constant: $a = 2.223 \text{ nm}$. The XRD shown in Fig. 2a further verified that the as-cast GW94 alloy were dominantly made up of α -Mg phase and Mg_{24}Y_5 eutectic compounds, where some positions of Y were substituted by Gd atoms. After the solution treatment at 510°C for 6 h, the microstructure of this alloy only consisted of α -Mg matrix with the eutectic phase $\text{Mg}_{24}(\text{Gd},\text{Y})_5$ dissolving into the matrix, as described in Fig. 2b. It should be noted that there is an unknown small peak at $2\theta \approx 29.5^\circ$, which will be discussed in the following sections.

3.2 Microstructural Evolution During Solution Treatment

The three-dimensional morphology and spatial distribution of $\text{Mg}_{24}(\text{Gd},\text{Y})_5$ phase in the as-cast GW94 alloy were characterized by thresholded XRT images shown in Fig. 3a. It reveals that the $\text{Mg}_{24}(\text{Gd},\text{Y})_5$ phases had 3D irregular morphology and were not connected. The total volume fraction of the eutectic phase can be exactly evaluated to be 6.4% with the size of individuals varying considerably. Figure 3b presents the four biggest ones in Fig. 3a, which had the volumes of 154,120.3, 53,560.75, 37,975.56, and 37,916.7 μm^3 , respectively. However, the minimum one with approximate granular shape was only about 1.110689 μm^3 . Figure 4 illustrates the microstructure evolution of GW94 alloy during solution treatment via in-situ XRT tomography. According to SEM image of the as-cast alloy (Fig. 1b), it is clear that the brightest places (white in grayscale and yellow in RGB bitmap) were

Fig. 2 XRD patterns of Mg–9Gd–4Y–0.5Zr alloy: **a** the as-cast, **b** the as-solutionized (510 °C for 6 h)

$\text{Mg}_{24}(\text{Gd},\text{Y})_5$ and the grain interior (matrix) was in dark or in blue (Fig. 4c, f). The RE element-enriched zones near the grain boundaries were represented by green in Fig. 4c, f. It is interesting to find that after solution treatment several particles (as indicated by red arrows in Fig. 4d) with higher content of RE elements than $\text{Mg}_{24}(\text{Gd},\text{Y})_5$ (as implied by the inset of Fig. 4d and the orange particle located in bottom of Fig. 4f) precipitated on the surface of the specimen. In-situ slices images of Fig. 4c and d demonstrate that the decomposition of eutectic phase was from its edge to its center in the light of the change of color. It indicates that the dissolving process of eutectic phase involved diffusion of solute atoms from the grain boundary to grain interior. Figure 4f also shows that the eutectic phase in the center of the sample was much easier to dissolve than that in the fringe field. However, due to the

small size of the cuboid phase and the similar RE content to eutectic phase, the cuboid phase was not identified separately.

The microstructural evolution with solution time and temperature is shown in Fig. 5. It implies that solution treatment condition had significant influence on both the dissolution rate of eutectic phase and the grain growth of the alloy. At 500 °C, although the amount of the eutectic phases dramatically decreased after being held for 2 h (Fig. 5a), it seems that eutectic phases could not be completely dissolved into matrix until the solution treatment time increased to 8 h, as indicated from the SEM images under BSE (backscattered electron) mode of Fig. 6a, b. However, Fig. 6c presents that no eutectic phase was found after solution treatment at 510 °C for 2 h. It can be concluded that the dissolution rate of the eutectic phase was considerably high at temperatures higher than 510 °C. Figure 7 summarizes the influence of solution treatment time on the average grain size of this alloy solutionized at various temperatures. Generally speaking, the rate of grain growth increased with increasing solution treatment time. Furthermore, it is evident that the rates of grain growth were slow at 500–520 °C in the range of 0–8 h. The grain sizes of the samples solutionized at 500–520 °C for 8 h were all lower than 100 μm. However, when the temperature increased to 530 °C, grains grew up quickly with the increase of time. After held at 530 °C for 8 h, the average grain size of this alloy was coarsened to 160 μm. Therefore, the solution treatment temperature of this alloy should be lower than 530 °C and the solute treatment time should be under 8 h to avoid abnormal grain growth. In consideration of economy and efficiency, solution treatment temperature of 510 and 520 °C with high dissolution rates of eutectic phase was preferred for GW94 alloy.

Fig. 3 **a** Threshold of XRT result showing the $\text{Mg}_{24}(\text{Gd},\text{Y})_5$ in the as-cast alloy and **b** the four biggest $\text{Mg}_{24}(\text{Gd},\text{Y})_5$ in **a**

Fig. 4 In-situ X-ray tomography (XRT) images of the as-cast alloy **a–c** and under solutionized conditions (510 °C for 0.5 h) **d–e**: **a, d** the 3D reconstruction of the sample, **b, e** interior showing segregated GBs with high content of Gd, Y elements (in grayscale), **c, f** the same cross sectional slice (in RGB bitmap)

Furthermore, it should be noted that the determination of solution treatment time for practical application should also take the sample size into consideration.

3.3 Characterization of the Cuboid-Shaped Phase in Solutionized Alloy

Except for the dissolution of eutectic phase and grain growth, another profound change of this alloy after solution treatment was that some cuboid phase was generated and unevenly distributed in the matrix as indicated in Fig. 6. This phase in solutionized Mg–RE alloy also has been widely observed but not well understood in previous studies [7, 10–12]. The compositions of a mass of (more than 50) cuboid phases in the as-solutionized alloys were examined by EDX and five typical of them are listed in Table 2. It

manifests that the cuboid phase has a wide composition range of 7.75–71.28 wt% Mg, 51.44–10.35 wt% Y and 40.81–18.37 wt% Gd. Obviously, the semi-quantitative EDX analysis cannot provide an unified stoichiometric composition for cuboid phase, but it was sure that the cuboid phase was rich in RE.

The area fractions of cuboid phase after different solution treatment conditions were quantified through image analysis as plotted in Fig. 8. It shows that the area fraction of cuboid phase was in a narrow range of 0.4%–0.9%, changing little with varying solution treatment condition. This is also the reason why this cuboid phase cannot be markedly characterized by XRD in Fig. 2b. However, despite of their trace amount and small size, it was reported that the cuboid phase was harmful to the mechanical property [11]. The special morphologies of these cuboid

Fig. 5 OM images of GW94 alloy solution heat treated at **a** 500 °C for 2 h, **b** 500 °C for 8 h, **c** 500 °C for 16 h, **d** 510 °C for 2 h, **e** 510 °C for 8 h, **f** 510 °C for 16 h, **g** 520 °C for 2 h, **h** 520 °C for 8 h, **i** 530 °C for 8 h

Fig. 6 SEM images of GW94 Mg alloy solid solution-treated at **a** 500 °C for 6 h, **b** 500 °C for 8 h, **c** 510 °C for 2 h

Fig. 7 Grain size variation with solution treatment time at different temperatures

phases were gathering into clusters as shown in Fig. 9a, b, which is responsible for their detrimental effect on ductility due to easily causing great stress concentration and even inducing micro-cracks. Figure 9c shows the microstructure of the alloy solutionized at appropriate solidus temperature of 565 °C for 30 h. It indicates that the cuboid phase was highly stable and could not be eliminated during solution treatment. Therefore, the cuboid phase would also be harmful to the hot workability. Nodooshan et al. [15] reported that the cuboid-shaped particle was separated from the matrix during the tensile test, causing microcracks and premature failure. Furthermore, due to its consumption of alloying elements of Gd and Y, the existing of cuboid phase would also weaken the aging precipitation.

Mg–Gd–Y alloy has been developed and investigated for decades. However, there were different opinions about the cuboid phase which was encountered in the as-cast specimens and observed more frequently in the solutionized alloy. These phases were identified to be $Mg_5(Gd, Y)$ [7, 16], $Mg_2Gd_3Y_3$ [10], GdH_2 [11, 17, 18] or Gd_2O_3 [12]. This kind of phase has an fcc crystal structure with $a \approx 0.53$ nm [7]. It is well known that Mg_5Gd has an fcc crystal structure with $a = 2.22$ nm. Moreover, the dissolution temperature of Mg_5Gd was supposed to be about 500 °C [6]. In view of its special lattice constant and high thermal stability (existence at 565 °C for 30 h), the cuboid

Fig. 8 Area fraction of the cuboid-shaped phase in Mg–9Gd–4Y–0.5Zr alloy under different solution temperatures and time

phase should not be $Mg_5(Gd, Y)$. In other words, the cuboid phases in the as-cast alloy and as-solutionized alloy may be two different phases. The former was $Mg_5(Gd, Y)$, which would be dissolved into α -Mg matrix during solution treatment at temperatures higher than 500 °C. Lately, the fcc phase in as-solutionized alloy was identified to be RE hydrides based on XRD. As shown in Fig. 2b, the unknown small peak in solutionized GW94 Mg alloy has 2θ of 29.54°. According to PDF card, it might be a peak of Gd_2O_3 (lattice plane (111), $2\theta = 29.04^\circ$, $a = 0.531$ nm), GdH_2 [(111), $2\theta = 29.14^\circ$, $a = 0.531$ nm] or YH_2 [(111), $2\theta = 29.65^\circ$, $a = 0.521$ nm]. The identification of Gd_2O_3 also has been discussed, relying on XRD and TEM analysis. In this paper, solution treatment of the samples was conducted at 510 and 520 °C for 6 h in vacuum atmosphere. As shown in Fig. 8, the microstructure result shows that the cuboid phase was generated during solution treatment without atmosphere of oxygen, excluding the possibility of being Gd_2O_3 . In order to determine whether this cuboid phase was hydride or not, SIMS was used to detect H. As shown in Fig. 10, the images SIMS of Mg^+ , Y^+ , Gd^+ and H^+ present a uniform element distribution except for some spots unevenly interspersing. Apparently, the spots correspond to the cuboid phases in the matrix. It can be seen that all the spots contained high contents of Y and H (Fig. 10b and d) and almost none of Mg (Fig. 10a). However, only several big spots contained high

Table 2 Compositions of five typical cuboid phases examined by EDX

	MgK wt% (at.%)	YL wt% (at.%)	GdL wt% (at.%)	Total wt% (at.%)
1	71.28 (92.63)	10.35 (03.68)	18.37 (03.69)	100 (100)
2	29.35 (66.76)	31.03 (19.03)	39.62 (19.94)	100 (100)
3	07.75 (27.55)	51.44 (50.02)	40.81 (22.43)	100 (100)
4	54.97 (85.41)	20.41 (08.67)	24.62 (05.91)	100 (100)
5	43.60 (78.39)	27.76 (13.65)	28.63 (07.96)	100 (100)

Fig. 9 SEM images in back scattered electron (BSE) mode of this alloy solution-treated at **a** 510 °C for 6 h, **b** 520 °C for 6 h, **c** 565 °C for 30 h

Fig. 10 SIMS results of **a** Mg⁺, **b** Y⁺, **c** Gd⁺, **d** H⁻

content of Gd (Fig. 10c). It seems that the cuboid phase was YH₂ with fcc crystal structure and $a \approx 0.52$ nm, where a few of Y atoms in some cuboid phases may be substituted by Gd due to their similar feature. RE hydride was reported

to be highly stable up to 1000 °C, so it is reasonable to observe the cuboid phase existing at 565 °C as shown in Fig. 7d. Furthermore, with high volume fraction, other kinds of cuboid-shaped RE (Dy, Nd, Ce, etc.) hydrides have been

Fig. 11 a SEM image of the GW94 sample after solutionized at 520 °C for 6 h and element distribution of **b** Mg, **c** Gd, **d** Y using EDX

Fig. 12 Mechanical properties of the as-cast and as-solutionized (at 520 °C for 6 h) GW94 Mg alloy

easily identified in the Mg–RE alloys with high content of RE elements by XRD diffraction [11]. However, the origin of H and the formation mechanism of the RE hydride are still not well understood [11]. According to Fig. 8, the area fractions of the samples solutionized in vacuum atmosphere were slightly lower than those exposed in air, indicating that hydrogen probably came from both the air during solution treatment and the sample interior involved during melting.

Therefore, the melting quality and the heat treatment atmosphere are very important for decreasing the cuboid phase in solutionized Mg–Gd–Y alloy.

3.4 Determination of Solution Treatment

It has been illustrated that the cuboid phase in solutionized GW94 alloy was detrimental to ductility. However, according to the analysis above, the cuboid phase cannot be eliminated during solution treatment and its volume fraction was small, keeping at a level of less than 1% under all solution treatment conditions. Therefore, the cuboid phase was not taken factor into consideration for determining solution treatment. To our knowledge, there has been still no quantitative method to determine optimum solution treatment, and the complete dissolution of coarse eutectic phases along with no apparent grain growth was always considered as the advisable solution treatment conditions. Among the advisable conditions, low temperature with short time was preferred in view of economy. In addition, homogenization of solute atoms should be checked using map scanning of EDX.

Given grain growth and eutectic phase dissolution, the optimum solution treatment temperature range for this

sand-cast alloy was 510–520 °C. And it has been discussed that for this GW94 samples with dimension of $\Phi 12 \text{ mm} \times 7 \text{ mm}$, 2–8 h was suitable as the solution treatment time. In this paper, 520 °C for 6 h was selected as the solution treatment condition to compare the mechanical properties between the as-solutionized and the as-cast alloys. Figure 11 shows SEM image (Fig. 11a) of the sample solutionized at 520 °C for 6 h and Mg, Gd, Y element distributions (Fig. 11b–d). It can be seen obviously that after solution treatment, the alloy has a homogenized microstructure. Figure 12 provides the mechanical properties of the as-cast and the as-solutionized GW94 Mg alloy. It can be seen that the elongation of this alloy after solution treatment was significantly improved from 2.2% to 11.7%. The microstructural evolution during solution treatment as shown in Figs. 3 and 4 indicated that the coarse and hard eutectic phases at the grain boundaries with high volume fraction of 6.4% should be dominantly responsible for the ductility improvement. The detrimental effect of this eutectic phase on ductility was reported by Mu *et al.* [19], who demonstrated that stress concentration caused by Mg_{24}Y_5 was prone to be released by generating cracks at the near solute-enriched region. With bypassing rather than breaking the eutectic phase, the cracks inclined to propagate rapidly into the matrix or along grain boundaries, causing little grain deformation and low ductility. Generally, the ductility improvement was always accompanied with compromising strength [20]. However, for this alloy, with ductility improved by 430%, the yield strength only decreased slightly from 150 to 133 MPa. Although both dissolution of the hard eutectic phase and grain growth may dramatically decrease the strength of the alloy after solution treatment, the conspicuous solution strengthening effect of Gd and Y element [21, 22] effectively offset the strength drop. Therefore, the similar strengths of the as-cast and as-solutionized GW94 alloys were a comprehensive effect among dissolution of the hard eutectic phase, grain growth, and solution strengthening.

4 Conclusions

In this study, solution treatments at different temperatures, for different time, and in different atmosphere were employed on sand-cast Mg-9Gd-4Y-0.5Zr (wt%) alloy, and the microstructural evolution and mechanical properties of the alloys were investigated. The main conclusions can be summarized as follows:

- (1) The eutectic phase of $\text{Mg}_{24}(\text{Gd,Y})_5$ in as-cast specimens was disconnected, having three-dimensional irregular shape and various sizes. In-situ XRT observation reveals that the dissolution of these eutectic

phases during solution treatment starts from the phases in the center of the sample and then spreads to those in external region.

- (2) The inevitable and invariable cuboid phase in as-solutionized Mg-Gd-Y alloy was identified as YH_2 , where a few of Gd atoms may substitute some positions of Y.
- (3) Taken grain growth and eutectic phase dissolution into consideration, the optimum solution treatment temperature range for this sand-cast alloy was 510–520 °C.
- (4) After solution treatment, the ductility of this sand-cast alloy can be improved by 430% without pronounced decrease of strength.

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