Effect of Precipitated Phases on Corrosion of $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ Alloy with Long-Period Stacking Ordered Structure

Xi-Mei Zong^{1,2,3} · Dan Wang^{1,2} · Wei Liu^{1,2} · Kai-Bo Nie^{1,2} · Chun-Xiang Xu^{1,2} · Jin-Shan Zhang^{1,2}

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Abstract The microstructure of the precipitated phases of $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloys with long-period stacking ordered structure before and after heat treatment is discussed. The corrosion properties of the as-cast (F), solid-solution (T4) and aging-treated (T6) alloys in 1% NaCl solution are studied. The hydrogen evolution and electrochemical measurements display that the as-cast $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloy with the continuous network eutectic phase exhibits the greatest corrosion resistance, while T6 sample with some needle-like phases and the particle phases is the worst among the three alloys. It is proposed to be mainly related to the amount, composition, microstructure and distribution of the precipitated phases.

KEY WORDS: Mg–Gd–Zn; Corrosion properties; Long-period stacking ordered (LPSO); Microstructure

1 Introduction

Nowadays, magnesium alloys have been widely researched and applied in automotive and aerospace industries due to the excellent properties of high strength and low density [1, 2]. Specifically, among the numerous Mg alloys, the discovery and development of Mg alloys with long-period stacking ordered (LPSO) structure have attracted great interests recently. Because they have distinctive microstructure and outstanding mechanical properties at both room temperature and elevated temperatures [3–5]. Though various magnesium alloys have the relatively good mechanical properties,

the poor corrosion resistance mainly limits their widespread applications.

Amounts of studies demonstrated that the corrosion of Mg alloys greatly depended on the amounts, distribution and microstructure of the second phase which could play different roles in different Mg alloys [6–12]. On the one hand, the isolated coarse precipitates, as a galvanic cathode, could cause micro-galvanic corrosion and therefore accelerate corrosion rate. For instance, the ternary $\text{Mg}_{12}\text{DyZn}$ phase with 18R LPSO structure in Mg–2Dy–0.5Zn alloy could act as micro-cathodes during corrosion process and accelerate the corrosion of α -Mg matrix [7]. Coy et al. [9] reported that the potential difference between the phase enriched in Zr and α -Mg matrix was higher than that between the other secondary phase and α -Mg matrix. Consequently, as an active cathode phase, the phase enriched in Zr increased the corrosion rate of the magnesium alloys. These studies indicate that the second phases in Mg alloys are unfavorable to the corrosion resistance. On the other hand, fine and continuously distributed precipitates in α -Mg matrix can act as a corrosion barrier to retard the development of corrosion. Zhang et al. [11] reported that $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy showed the lowest corrosion rate in their studied alloys due to the continuous distribution and moderate volume fraction of LPSO

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✉ Jin-Shan Zhang
jinshansx@tom.com

¹ College of Materials Science and Engineering, Taiyuan University of Technology, Taiyuan 030024, China

² Shanxi Key Laboratory of Advanced Magnesium-Based Materials, Taiyuan 030024, China

³ Department of Chemistry, Taiyuan Normal University, Taiyuan 030031, China

phase. The addition of Ce could improve the corrosion resistance of AZ91D alloy because of high volume fraction of β phase appearing continuous network structure [12]. These researches show that the continuous structure of second phases is beneficial to the corrosion resistance. In general, the corrosion rate of Mg alloys is affected by the precipitated phases significantly.

Heat treatment is one of the effective methods to adjust the second phases and therefore to affect the mechanical properties, as well as corrosion properties of Mg alloys [13–15]. According to our previous work, it was confirmed that the mechanical properties were closely linked to the different precipitated phases in $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloy with LPSO structure before and after heat treatment [3]. However, the influence of the different precipitated phases on the corrosion resistance of $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloy with LPSO structure has been barely studied. The aim of our work is to study the corrosion properties of the alloys in the as-cast (F), solid-solution (T4) and aging-treated (T6) conditions and then investigate the effect of precipitated phases on the corrosion process.

2 Experimental

2.1 Material Preparation and Microstructure Analyses

The $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloy with LPSO structure was prepared by well-type crucible resistance furnace from high-purity Mg (99.9%), Gd (99.9%), Zn (99.9%) and Mg–30Zr (wt%) master alloy in a shielding gas $\text{CH}_2\text{FCF}_3 + \text{N}_2$ atmosphere at 760 °C. Then it was cast into a preheated mold to keep homogeneous. The prepared $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloy ingot was cut into cylinder-shaped specimens with diameter of 30 mm and thickness of 3 mm. And part of these specimens was performed by solid-solution treatment at 500 °C for 30 h (T4), the aging-treated at 200 °C for 50 h (T6) in a SX2-8-10 box-type high-temperature electric resistance furnace and then quenching in water at 70 °C. The specimens for immersion and polarization tests were ground to a 2000 grit SiC paper, subsequently rinsed with acetone in an ultrasonic bath and dried in warm air. The specimens for metallographic observation were further ground to a 3000 grit SiC paper and then etched in 3% nital. The detailed steps about the process of disposing samples can refer to the literature [16, 17].

The surface microstructure of the specimens was observed by scanning electron microscopy (SEM, JSU-6700F). Phase structure was detected by X-ray diffraction (XRD, Model Y-2000, Aolong, China), using monochromatic $\text{Cu-K}\alpha$ radiation and 0.05° step length. The scanning range was between 20° and 80°.

2.2 Corrosion Testing

The corrosion resistance of the alloys was studied by immersion and polarization tests. The corrosion medium is 1% NaCl solution at room temperature with pH of 7.0. To record hydrogen evolution, the specimens were placed in a beaker and a calibrated hydrogen collection tube was set up over the corroding specimen. The corrosion depth, D_{corr} (mm), and corrosion rate, R_{corr} (mm/year), were calculated using the following equations [18, 19]:

$$D_{\text{corr}} = \frac{M \times V_{\text{h}}}{24.79 \times \rho \times A}, \quad (1)$$

$$R_{\text{corr}} = \frac{353 \times M \times \Delta V_{\text{h}}}{\rho \times A \times t}, \quad (2)$$

where V_{h} is the volume of hydrogen evolved in mL, M is the apparent atomic weight of the sample in g/mol, ρ is the density of the sample in g/cm³, A is the surface area of the sample in mm² and t is the test duration in hour (h). The corrosion rate was averaged from five tests.

In the case of the polarization curves, the potentiodynamic polarizations were performed in 1% NaCl solution at room temperature using Land CS350 (CorrTest, China) electrochemical system. The scan scope was set from –2000 to –1200 mV versus open-circuit potential (OCP) with a constant scan rate of 2 mV/s. A classical three-electrode cell was used with a platinum as counter electrode and a saturated calomel electrode as reference electrode, and the samples were sealed by resin with an exposed area of 10 mm × 10 mm as working electrode.

3 Results

3.1 Microstructures

Figure 1 displays the SEM images of $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloys with LPSO structure in F, T4 and T6 conditions. Figure 1a shows that F alloy mainly consists of α -Mg matrix and eutectic phase, $(\text{Mg,Zn})_3\text{Gd}$, which locate at the grain boundaries with network continuous structure. Besides, the content of LPSO phase in F sample is too low to be observed in Fig. 1a, and therefore, it cannot be detected by XRD shown in Fig. 2. After T4 and T6 treatment (Fig. 1b, c), a number of block and lamellar phases can be found at the grain boundaries, which were determined as 14H-LPSO structure [3, 20]. According to the previous studies, Mg–Gd–Zn alloys were the type II alloys, in which the LPSO phase was nonexistent or with small volume fraction in the as-cast ingots but precipitated after heat treatments [4, 5, 21]. This was why there was little LPSO in F alloy but numerous in T4 and T6 alloys. Besides, in T4 alloy there are a lot of face-centered cubic

Fig. 1 SEM microstructure of $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloys: **a** F alloy, **b** T4 alloy, **c** T6 alloy

Fig. 2 XRD patterns of F, T4 and T6 $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloys

(fcc) of needle-like phase precipitating in the grains from matrix. And the composition was $\text{Mg}_{25.82}\text{Zn}_{25.97}\text{Gd}_{1.10}\text{Zr}_{47.11}$ enriched in Zr which is confirmed by EDS (Fig. 3a).

The microstructure of T6 alloy is displayed in Fig. 1c. Numerous fine-stripe lamellar phase with 14H-LPSO structure appears not to be at the grain boundaries but within grains. In addition, there are also some needle-like

Fig. 3 EDS spectra for **a** needle-like phases, **b** particle phases

phases locating dispersedly on the grain surface. It is more important that a kind of tiny particle phase with the composition of $\text{Mg}_{58.90}\text{Zn}_{13.76}\text{Gd}_{27.34}$ (Fig. 3b) precipitates at the grain boundaries [3]. The needle-like phase and particle phases can be detected in XRD patterns in Fig. 2. It can be concluded that after different heat treatments, $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloy could precipitate different phases with different composition and microstructures.

3.2 Corrosion Properties

The corrosion depths for 74-h immersion of F, T4 and T6 alloys as a function of corrosion time are shown in Fig. 4. Evidently, with increasing immersion time each corrosion depth curve appears to be approximately linear with corrosion time but at different slopes. The two lines of T4 and T6 are close to each other, especially at the beginning of the corrosion time within 20 h. Furthermore, the slopes of T4 and T6 alloys are steeper than that of F alloy, which means that T4 and T6 alloys are poorer resistant to corrosion than F alloy in 1% NaCl solution.

It is known that the hydrogen evolution trend directly reflects the corrosion rate of magnesium alloys. So the corrosion rates were calculated according to the volume of hydrogen with Eq. (2). Figure 5 shows the corrosion rates of F, T4 and T6 alloys as function of corrosion time. The

Fig. 4 Corrosion depths of F, T4 and T6 $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloys as a function of immersion time

Fig. 5 Corrosion rates of F, T4 and T6 Mg_{95.8}Gd₃Zn₁Zr_{0.2} alloys as a function of immersion time

Fig. 6 Corrosion rates of F, T4 and T6 Mg_{95.8}Gd₃Zn₁Zr_{0.2} alloys in 1% NaCl solution

curves of T4 and T6 take the similar tendency and rise slightly with increasing immersion time. However, the corrosion rate of F sample is almost stable within 24 h, indicating a passivation trend. After immersion for 72 h, the corrosion rates of F, T4 and T6 alloys are 0.896, 3.318 and 3.689 mm/year, respectively, as shown in Fig. 6. Thus, the results obtained from the immersion tests indicate that F alloy is more resistant to corrosion than T4 and T6 alloys in 1% NaCl solution.

The potentiodynamic polarization curves of F, T4 and T6 Mg_{95.8}Gd₃Zn₁Zr_{0.2} alloys are shown in Fig. 7. It can be seen from cathodic branch that the current density is nobler for T4 and T6 samples than that of F sample. It is revealed that the hydrogen evolution rate of T4 and T6 alloys is faster than that of F alloy. For anodic branch, T6 alloy exhibits the highest anodic current density, while that of F alloy exhibits the lowest. The values of major electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), cathodic Tafel slopes (β_c) and anodic Tafel slopes (β_a) of the composite materials were evaluated from such polarization plots using Tafel

Fig. 7 Polarization curves of F, T4 and T6 Mg_{95.8}Gd₃Zn₁Zr_{0.2} alloys in 1% NaCl solution

analysis and presented in Table 1. From the electrochemical parameters (i_{corr} , β_a and β_c), the polarization resistance (R_p) was calculated according to the following equation [22]:

$$R_p = \frac{\beta_a \beta_c}{2.3(\beta_a + \beta_c) i_{\text{corr}}} \quad (3)$$

It shows that the current density (i_{corr}) values of the three samples rise in the order of $F < T4 < T6$, while the polarization resistance (R_p) values decrease in the same order. Evidently, F alloy exhibits the best corrosion resistance, while T6 sample is the worst one. The polarization curve results are in good consistent with the immersion tests.

Figure 8 shows the SEM images of F, T4 and T6 alloys after immersion in 1% NaCl solution for 24 h. Some microcracks can be seen at the grain boundaries (Fig. 8d–f), which results from the corrosion product drying after removal from the test solution. In Fig. 8a, d, the eutectic phase is observed clearly and corroded barely. The matrix adjacent to the eutectic phase is covered by the compact and uniform corrosion products, whereas the matrix farther from the grain boundaries appears a few local corrosion and some areas have not been corroded, where the areas are not covered by the corrosion products. Based on these observations, it reveals that the eutectic phase acts as galvanic cathodes accelerating corrosion. So, the eutectic phase supports the cathodic reaction locally and increases the local pH (due to hydroxyl ion generation), and hence, corrosion products are formed nearby the eutectic phase.

However, the corrosion product in T4 alloy takes different morphologies. The main parts adjacent to the eutectic and LPSO phases are covered by a compact oxide film (Fig. 8b, e), which could effectively block the solution

Table 1 Fitting results of the polarization curves of F, T4 and T6 Mg_{95.8}Gd₃Zn₁Zr_{0.2} alloys

Alloy	E_{corr} (V)	i_{corr} (A/m ²)	β_a (V/dec)	β_c (V/dec)	R_p (Ω m ²)
F alloy	−1.6656	0.427	0.19173	0.22081	0.1045
T4 alloy	−1.6802	0.569	0.13882	0.20799	0.0636
T6 alloy	−1.6853	0.763	0.14017	0.20492	0.0474

Fig. 8 SEM images of Mg_{95.8}Gd₃Zn₁Zr_{0.2} alloys immersed in 1% NaCl solution for 24 h: **a** F alloy, **b** T4 alloy, **c** T6 alloy, **d–f** corresponding high magnification images of **a–c**, respectively

penetration, while the other little parts are mainly made up of coarse particles, hardly postponing the penetration of solution. These nonuniform morphologies of the corrosion product in T4 alloy may be caused by the needle-like precipitates. The discontinuously distributed precipitates accelerated the corrosion of the substrate through galvanic corrosion and resulted in the discontinuous oxide film on the surface. The similar results were reported by Zhang *et al.* [23] in which the Mg–5Gd–1Zn–0.6Zr alloy with needle-like phase had poor ability to resist corrosion. Besides, the needle-like precipitate enriched in Zr is identified by EDS in Fig. 3a. According to the study by Coy *et al.* [9], Zr-rich precipitates exhibited relative Volta potential differences with respect to the magnesium matrix. This difference is sufficient to develop significant galvanic

coupling, hence favoring the dissolution of the matrix. Thus, it is deduced that the needle-like phase enriched in Zr may play a similar role in T4 alloy. And that is why the corrosion resistance of T4 alloy is worse than that of F alloy though the main part is covered by compact film in T4 alloy. And more researches on the mechanism will be performed in our future work.

Compared with F and T4 alloys, T6 alloy suffers from most severe corrosion in fairly large area (Fig. 8c, f). The corrosion products on the surface are mainly formed by numerous loose and irregular particles, which cannot effectively restrain the penetration of solution. This may be due to the precipitates of LPSO, needle-like, and the particles phases (Fig. 1c), which act as cathode and facilitate the corrosion.

4 Discussion

It should be noted that there was numerous LPSO phase in both T4 and T6 alloys. Up to now, the effect of LPSO phase on the corrosion properties is unclear. Some researches showed that the LPSO structure played a positive role in improving corrosion resistance of Mg alloys [23, 24]. However, Izumi *et al.* [19] found that the presence of the LPSO phase involved in the filiform corrosion process, whereas single-phase microstructure delayed the occurrence the filiform corrosion. Srinivasan *et al.* [25] reported that LPSO phase facilitated the corrosion in Mg–10Gd–2Zn alloy also. Zhang *et al.* [11] observed that the increase in the volume of bulk X phase containing the LPSO structure in Mg–Zn–Y alloys increased the corrosion rate due to the galvanic coupling effect. But $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy showed the lowest corrosion rate among the studied alloys due to the continuous distribution and moderate volume fraction of LPSO phase. Anyway, whether LPSO phase facilitates the corrosion of Mg alloys or not depends on the morphology, amount and distribution in the grains. Unfortunately, although there was 14H-LPSO phase in both T4 and T6 alloys in our work, they took discontinuous distribution at the grain boundaries or within the grains. It is believed that the critical factors determining the rate of galvanic corrosion include the cathode-to-anode area ratios and the difference in potentials of the two phases [15]. In T4 and T6 alloys, 14H-LPSO phase developed with α -Mg matrix alternately, which increased the cathode-to-anode area ratio and resulted in facilitating the corrosion rather than retarding. So the galvanic corrosion of T4 and T6 alloys is more severe than that of F alloys.

On the basis of the above results, no matter eutectic, LPSO, needle-like and the particle phases, these precipitated phases that acted as the cathodes in the micro-galvanic corrosion result in the preferential corrosion of the matrix. Thus, the corrosion initially happened in the vicinity of the second phases where the surfaces were covered by the products with prolonging immersion time. Though the different precipitated phases played the similar role in the corrosion process, the corrosion rates of F, T4 and T6 alloys were different in immersion tests. Combined with the compactness of the surface film, it can be concluded that the corrosion products covering on the surface of the alloys could play a key role as a protective film. A higher corrosion resistance of the as-cast alloy is due to the more compact protective film. On the other hand, although precipitated phase serves mainly as a galvanic cathode and accelerates anodic dissolution, it could retard the corrosion of the alloy due to its continuous structure to some extent [26, 27]. The precipitated phase could have a barrier influence because it is inert, which effectively reduces the

surface reactive area, so less area of α -Mg matrix is available to be corroded. In our study, SEM images showed that more continuous networks of the eutectic phase were observed in F alloy (Fig. 1a), while LPSO, needle-like and the particle phases in T4 and T6 alloys did not appear to be a continuous distribution compared with the eutectic phase in matrix, and thus, the barrier effect was poor. This is another reason why T4 and T6 alloys have higher corrosion rates than F alloy.

5 Conclusion

The microstructures of F, T4 and T6 $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloys with LPSO structure were discussed, and the effect of the precipitated phases on the corrosion properties of the alloys immersed in 1% NaCl solution was studied. It can be concluded that before and after different heat treatments $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloy appeared a different corrosion rates. The corrosion resistance of F alloy is the best. This may be attributed to the two key factors: relatively compact film and continuous eutectic phase. There are various LPSO and needle-like phase precipitated from the matrix in both T4 and T6 alloys. The discontinuous structure of LPSO and needle-like phases and loose film result in deteriorated corrosion resistance of the two alloys. Besides, in T6 sample some particle phases precipitate at the grain boundaries, which brings about severe corrosion of the matrix. Therefore, precipitated phases with different composition and microstructure significantly dominate corrosion rates of the $\text{Mg}_{95.8}\text{Gd}_3\text{Zn}_1\text{Zr}_{0.2}$ alloys.

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