Research Article

Effect of Heat Treatment on Corrosion Behaviors of Mg-5Y-1.5Nd Alloys

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Corrosion behavior of Mg-5Y-1.5Nd alloy was investigated after heat treatment. The microstructure and precipitation were studied by scanning electron microscope (SEM) and energy dispersive spectrometer (EDS). The weight loss rates of different samples were arranged as T6-24 h>T6-6 h>T6-14 h>as-cast>T4. The open circuit potential (OCP) showed that T4 sample had a more positive potential than that of other samples. The potentiodynamic polarization curves showed that the T6-24 h sample had the highest corrosion current density of $245.362 \mu\text{A}\cdot\text{cm}^{-2}$, whereas the T4 sample had the lowest at $52.164 \mu\text{A}\cdot\text{cm}^{-2}$. The EIS results confirmed that the heat treatment reduced the corrosion resistance for Mg-5Y-1.5Nd alloy, because the precipitations acted as the cathode of electrochemical reactions to accelerate the corrosion process. The corrosion rates of different samples were mainly determined by the amount and distribution of the precipitations. The precipitations played dual roles that depended on the amount and distribution. The presence of the phase in the alloys could deteriorate the corrosion performance as it could act as an effective galvanic cathode. Otherwise, a fine and homogeneous phase appeared to be a better anticorrosion barrier.

1. Introduction

Mg-Y-Nd alloys have gained successful application because of their high strength and creep resistance [1]. Yttrium (Y) serves as an effective solid solution hardener; therefore, Y-containing Mg alloys have good mechanical properties [2, 3]. Neodymium (Nd) can refine the grain size and improve creep resistance of Mg alloys [4, 5]. Based on the Mg-Y-Nd system, many commercial alloys have been successfully developed, including WE43, WE54, and EW75 alloys [6–9]. Considerable effort has been dedicated to the corrosion properties of Mg-Y-Nd based alloys [10–12]. There are many different precipitated phases that may form in the Mg-Y-Nd system, such as the Mg_3Nd , $\text{Mg}_{12}(\text{YNd})$, and $\text{Mg}_3(\text{YNd})$ phases [13]. Davenport et al. [14] demonstrated that the Y-rich regions slowed the propagation of corrosion in WE43 alloy by a more homogenous distribution of Y. Corrosion resistance of Mg-Nd-Zn-Zr alloy was mainly ascribed to the grain refinement and the fine dispersed Mg_{12}Nd [15]. Williams et al. [4] studied the influence of Nd additions on the corrosion behaviors of Mg and found that cathodic activation

of corroded regions was proposed to derive from Nd-rich intermetallic grains.

Heat treatment can cause precipitated phases changes and redistribution of alloying elements. It has been reported that the precipitated phases have an effect on the corrosion resistance of aluminum-containing Mg alloys. Song et al. [16–19] found that a solution treatment decreases the amount of second-phase particles inside the grains and weakens strong pinning on dislocations. Wang et al. [20] studied the change in the microstructure of AZ91D alloy during T4 or T6 treatments. Lu et al. [21] analyzed the corrosion behaviors of Mg-3Zn alloy (T4 and T6) via electrochemical measurements, which found that large MgZn particles formed mainly on grain boundary and fine MgZn particles precipitated on matrix for T6 treatment. Ben-Hamu et al. [22] studied the relationship between microstructure and corrosion behavior of peak-aged Mg-Y-Gd-Zr alloy. From the above, the effect of heat treatment on the corrosion resistance of Mg-Al alloys has been widely reported, especially AZ91 Mg alloy. However, the effect of heat treatment on the corrosion properties of Mg-Y-Nd alloy has still not been well understood.

TABLE 1: Thermal process of the Mg-5Y-1.5Nd alloys in this study.

Heat treatment	Homogenizing	Aging
As-cast	—	—
T4	535°C, 20 h + water quenching	—
T6 (early-aged)	535°C, 20 h + water quenching	225°C, 6 h + water quenching
T6 (peak-aged)	535°C, 20 h + water quenching	225°C, 14 h + water quenching
T6 (overaged)	535°C, 20 h + water quenching	225°C, 24 h + water quenching

In this work, Mg-5Y-1.5Nd alloys after heat treatment were investigated to reveal the influence of precipitations on the corrosion resistance during heat treatment. The relationship between microstructure and corrosion behavior of different samples was analyzed. The results showed that heat treatment significantly affected microgalvanic corrosion between cathodic phases and anodic α -Mg matrix. The precipitations played dual roles that depended on the amount and distribution. The conclusion indicates that the corrosion properties of Mg-Y-Nd alloys can be modified by controlling the heat treatment process.

2. Experiments

2.1. Materials. The Mg-5Y-1.5Nd alloys were prepared by melting in an electrical resistance furnace in a steel crucible under protected gas consisting of Ar and $C_2H_2F_4$ in order to prevent burning of the melts. The materials were prepared from blending the appropriate proportions from ingots of commercial purity Mg (>99.95%), Y (99.9%), and Nd (99.9%). The highest smelting temperature was 850°C and the cast temperature was between 720°C and 750°C.

The samples were cut from the ingot by electric spark linear cutting machine. Actual compositions of the alloy were detected by inductively coupled plasma-atomic emission spectrometry (ICP-AES). The chemical compositions were as follows: Y, 5.02; Nd, 1.50; Al, 0.019; Fe, 0.013; balance Mg. The solution treatment (T4) was performed at 535°C for 12 h in argon atmosphere followed by water quench at 25°C. The aging alloy was T6 heat treatment at 225°C for 6 h, 14 h, and 24 h. The detailed thermal process of the samples in this study was shown in Table 1.

2.2. Experimental Procedures. For gas collection, cubes of $1 \times 1 \times 1$ cm were prepared and weighed and then sealed by epoxy with 1 cm^2 exposed surface. A funnel was inverted on the sample to transfer the generated hydrogen bubbles into a buret during the course of immersion. The samples were polished with up to 5000-grit SiC paper and then measured using three replicate samples for each alloy in 3.5% NaCl solution for 24 h.

The samples for electrochemical tests were cubes of $1 \times 1 \times 1$ cm. Before the tests, the electrodes were encapsulated in epoxy resin with a surface of 1 cm^2 exposed to 450 mL 3.5% sodium chloride solution (made with AR sodium chloride and deionized water) in a beaker at $25 \pm 1^\circ\text{C}$. The OCP, potentiodynamic polarization curve, and EIS were obtained from a Solatron 2273 system. The potentiodynamic scanning was performed at 0.5 mV/s after the cell was held at the OCP

for 400 s. The EIS was measured with the frequency ranging from 10,000 Hz to 0.1 Hz with 5 mV of amplitude of sinusoidal potential signals with respect to the OCP.

2.3. Analysis and Characterization Methods. The microstructures of Mg-5Y-1.5Nd alloys were characterized by SEM. EDS analysis was conducted on the alloys to confirm the phase identification. When the immersion test was completed, the corrosion products on the surface of different samples were removed in boiling chromic acid (20% CrO_3 + 1% AgNO_3) for 5 min [23]. The samples were then quickly washed with deionized water, dried, and weighed to obtain their final weight (W_1). The difference between W_0 and W_1 is the corrosion weight loss (Δw). The surface morphologies of samples without corrosion products were observed by SEM.

3. Results and Discussion

3.1. The Microstructure of Samples. The SEM photographs of Mg-5Y-1.5Nd alloy in different conditions were illustrated in Figure 1. As shown in Figure 1(a), the microstructure of as-cast alloy consisted of α -Mg matrix, particle phases, and skeleton eutectic phases. The close-up view clearly showed that a small quantity of skeleton eutectic phases was distributed on the surface of as-cast alloy, whereas amounts of particle phases were mainly distributed discretely (Figure 1(b)). The microstructure of Mg-5Y-1.5Nd alloy had changed during homogenization annealing (T4). The volume fraction of the precipitations decreased, and the skeleton eutectic phases were almost invisible. The precipitations were almost dissolved completely except for less particle phases, and the supersaturated α -Mg matrix was produced in Figure 1(c).

After T6-6 h heat treatment, amounts of particle phases precipitated along the grain boundary and within grains of the supersaturated α -Mg phase. Meanwhile, a small quantity of skeleton eutectic phases began to form. T6 treatment made Y and Nd atoms diffuse towards grain boundaries to form precipitations, and this process reduced Y and Nd atoms concentration in the α -Mg matrix. With a prolonged aging time, Y and Nd-content in the α -Mg matrix precipitated gradually and distributed uniformly (Figures 1(d)–1(f)). The precipitations would act as the cathode of electrochemical reaction to accelerate the corrosion process [24–26]. With a prolonged aging time, the amounts of skeleton eutectic precipitates increased and formed a continuous uniform distribution.

The EDS analyses were performed to characterize the components of as-cast Mg-5Y-1.5Nd alloy by more than three

FIGURE 1: The SEM photographs of Mg-5Y-1.5Nd alloys: (a) as-cast, (b) close-up view of (a), (c) T4, (d) T6-6 h, (e) T6-14 h, and (f) T6-24 h.

replicate spots. The result was basically identical in Figure 2. The white contrast particle phases and the eutectic phases were arranged to be $\text{Mg}_3(\text{Y,Nd})$ and $\text{Mg}_{12}(\text{Y,Nd})$, respectively. The great difference was that the content of Y in $\text{Mg}_3(\text{Y,Nd})$ phase was higher than that in $\text{Mg}_{12}(\text{Y,Nd})$ phase, but the Nd content was just the reverse. The contents of Y and Nd in the locations were rich due to the solute atoms enrichment in the solid/liquid interface during nonequilibrium solidification, whereas the solute atoms must be pushed into the grain boundary [27, 28].

3.2. Gas Collection and Weight Loss Rates. Gas collection was equivalent to employing a weight loss method for corrosion rate testing and was more convenient for calculating the average corrosion rate over a given period. In the current work, the corrosion rate was investigated by both hydrogen

evolution and weight loss tests measured simultaneously for each sample. Figure 3 showed the average hydrogen evolution rates of different samples in 3.5% NaCl for 24 h. The corrosion rates of different samples increased with the prolonging of immersion time. The T6-24 h sample showed the largest corrosion rate due to the amount and distribution of the precipitated phases, which acted as microcathodes for the microcells. During the period of immersion, the relative corrosion rates of the different samples can be ranked as T6-24 h > T6-6 h > T6-14 h > as-cast > T4.

The corrosion rate was calculated as follows: $C = (W_0 - W_1)/S$, where C is the weight loss rates ($\text{mg}\cdot\text{cm}^{-2}$), W_0 is the original weight (mg), W_1 is the final weight without corrosion products (mg), and S is the surface area (cm^2). Each type had three samples, so the corrosion rates were the average. Figure 4 shows that the T6-24 h sample had

FIGURE 2: The EDS results of different precipitations in as-cast Mg-5Y-1.5Nd alloy: (a) $Mg_{12}(Y,Nd)$ and (b) $Mg_3(Y,Nd)$.

FIGURE 3: The average hydrogen evolution rates of samples measured in 3.5% NaCl.

the highest corrosion rate of 1.3331 mg·cm⁻²·h⁻¹, whereas the T4 sample had the lowest at 0.3259 mg·cm⁻²·h⁻¹. The weight loss rates could be arranged as T6-24 h > T6-6 h > T6-14 h > as-cast > T4. When T6 sample was aged at 225°C for 6 h, bulky precipitated phases formed in the α -Mg matrix and along the grain boundary, resulting in the fact that the corrosion rate had a large increase. When the aging times increased from 6 h to 14 h, the precipitated phases were distributed uniformly to form a corrosion barrier, so the weight loss rate decreased to some extent. The amount of precipitated phases became the largest after the T6-24 h heat treatment, which acted as the cathode of electrochemical reactions. As a result, acceleration of the corrosion was more effective than the corrosion barrier, so the T6-24 h samples corroded most seriously.

3.3. Corrosion Products and Surface Morphologies. The corrosion products formed on the surface of samples were characterized by XRD in Figure 5. It could be demonstrated that Mg, Mg(OH)₂, Y(OH)₃, and Nd(OH)₃ were the main

FIGURE 4: The weight loss rates of samples after immersion in 3.5% NaCl for 24 hours.

FIGURE 5: The XRD of corrosion products formed on different samples.

components analyzed by Jade 5.0 software. However, the characteristic peaks of Mg in the corrosion products on

FIGURE 6: The surface morphologies without corrosion products of different samples: (a) as-cast, (b) T4, (c) T6-6 h, (d) T6-14 h, (e) T6-24 h, and (f) EDS results.

the T4 sample were higher than these of other samples, whereas the characteristic peaks of $\text{Mg}(\text{OH})_2$, $\text{Y}(\text{OH})_3$, and $\text{Nd}(\text{OH})_3$ were just the reverse. More interestingly, the characteristic peaks of $\text{Y}(\text{OH})_3$ and $\text{Nd}(\text{OH})_3$ in the corrosion products were found in the as-cast and peak-aged samples, which is not consistent with the literature [29, 30]. Normally, the amounts of $\text{Y}(\text{OH})_3$ and $\text{Nd}(\text{OH})_3$ in the corrosion products were less to detect. The phenomenon indicated that the as-cast and peak-aged samples had corroded seriously. Thus, characteristic peaks of $\text{Y}(\text{OH})_3$ and $\text{Nd}(\text{OH})_3$ appeared in the corrosion products.

After immersion test, the surface morphologies without corrosion products were shown in Figure 6. The corrosion attack of as-cast sample occurred horizontally in Figure 6(a), while the partial α -Mg matrix and precipitated phases

remained intact. The large uncorroded locations were irregularly distributed over the T4 samples, which indicated that the slightest damage occurred on the surface of alloys. Serious corrosion had occurred between the matrix and precipitates for T6-6 h sample, resulting in a large number of corrosion pits. However, corrosion on the surface morphologies of the peak-aged alloy (T6-14 h) showed a decreasing tendency. With the extension of aging time, the corrosion of overaged sample was aggravated gradually; even some corrosion areas were almost completely corroded. The most serious corrosion occurred in Figure 6(e); only a small quantity of precipitated phases existed.

3.4. Electrochemical Characterizations. Figure 7 shows the electrochemical characterizations, corrosion potential (E_{corr}),

FIGURE 7: The electrochemical characterizations, corrosion potential (E_{corr}), and corrosion current (I_{corr}) of Mg-5Y-1.5Nd alloy measured in 3.5% NaCl solution.

and corrosion current (I_{corr}) of different samples measured in 3.5% NaCl solution. The open circuit potential was comparatively stable before measurement. All the samples achieved a relatively stable value soon and equilibrium had been established. A corrosion product film formed on the surface of the samples, and no clear localized corrosion occurred. It was interesting to find that the T4 sample had a more positive OCP (-1.6148 V) than other samples, which meant lower driving on self-corrosion than as-cast and T6 samples. The OCP results implied that the electrochemical activity order could be arranged as $\text{T6-24 h} > \text{T6-6 h} > \text{T6-14 h} > \text{as-cast} > \text{T4}$.

The potentiodynamic polarization curves of Mg-5Y-1.5Nd alloy of different samples had been investigated. The cathodic Tafel slopes were similar for samples in different conditions, which indicated that hydrogen evolution reaction occurred [31, 32]. In contrast to the anodic branch, the cathodic branch exhibited linear Tafel behavior for potentials more negative than a critical potential. It is well known that the current associated with reduction of oxygen can be neglected and the cathodic process within the Tafel region was mainly hydrogen evolution in a static solution without stirring; the anodic polarization curves of magnesium alloys

have a complicated nature for the reason of simultaneous combination of both anodic dissolution and hydrogen evolution at anodic polarized area; thus, the Tafel extrapolation for calculating corrosion current density could be carried out via tangent line of cathodic branch. The values of corrosion current density of Mg-5Y-1.5Nd alloys were shown in Figure 7. The value of I_{corr} for T6-24 h sample was the highest of $245.362 \mu\text{A}\cdot\text{cm}^{-2}$, because $\text{Mg}_3(\text{Y,Nd})$ and $\text{Mg}_{12}(\text{Y,Nd})$ phases along the grain boundaries acted as cathode of the electrochemical reaction to accelerate the corrosion process. I_{corr} of T6-14 h sample became lower than T6-6 h, because the continuous distribution of $\text{Mg}_{12}(\text{Y,Nd})$ phases formed a corrosion barrier to protect the α -matrix. These results were consistent with the weight loss rate and surface corrosion morphologies mentioned above. The electrochemical impedance spectroscopy exhibited different characterizations of electrochemical corrosion reaction. The semicircle of EIS of T4 sample was the largest, which illustrated that hydrogen evolution reaction was most difficult on the α -Mg matrix. This phenomenon demonstrated that T4 sample with a single phase had the lowest microgalvanic reaction.

FIGURE 8: The schematic diagram of microstructure related to the corrosion mechanism: (a) as-cast, (b) T4, (c) T6-6 h, (d) T6-14 h, and (e) T6-24 h.

The corrosion of aged samples is consistent with the increased microgalvanic corrosion acceleration of α -phase that had an intrinsic corrosion rate. Increased aging time increased the amount of the precipitated phases. The increasing current corrosion density in the sequence aged samples can be attributed to a combination of these two effects: (i) Y and Nd elements precipitated from the grain, resulting in the fact that the potential of the matrix became lower, and (ii) increasing microgalvanic corrosion between the precipitated phases and matrix directly increased the corrosion rate. However, the peak-aged sample showed a decreasing trend compared with the early-aged sample, and then the overaged sample continued to increase compared with the peak-aged sample. This phenomenon indicated that the low corrosion rate of peak-aged sample was attributed to a continuous

network of the precipitated phases, which would be expected to act as a barrier to corrosion propagation.

3.5. Corrosion Mechanism. The schematic diagram of microstructure related to the corrosion mechanism was shown in Figure 8. Actually, there was a difference at the volume fraction and distribution of Mg₃(Y,Nd) and Mg₁₂(Y,Nd) phases in the Mg-5Y-1.5Nd alloy after heat treatment. In this study, the corrosion rates of different samples were mainly determined by the amount and distribution of the precipitations according to the classical theory of Song et al. [33, 34]. The phases played dual roles that depended on the amount and distribution. The presence of the phase in the alloys could deteriorate the corrosion performance as it could act as an effective galvanic cathode. Otherwise, a fine and

homogeneous phase appeared to be a better anticorrosion barrier.

4. Conclusion

(1) Mg-5Y-1.5Nd alloys under different conditions were investigated to reveal the effect of heat treatment on the electrochemical corrosion behaviors. The microstructure of different samples mainly consisted of α -Mg matrix and $\text{Mg}_{12}(\text{Y,Nd})$ and $\text{Mg}_3(\text{Y,Nd})$ phases. The precipitated phases almost dissolved into the α -Mg matrix. The amount and volume fraction of $\text{Mg}_{12}(\text{Y,Nd})$ and $\text{Mg}_3(\text{Y,Nd})$ phases increased with the prolongation of aging times.

(2) The T6-24 h sample showed the weight loss rate of $1.3331 \text{ mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$, whereas the T4 sample had the lowest rate at $0.3259 \text{ mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$. The electrochemical corrosion activity of different samples could be arranged as T6-24 h > T6-6 h > T6-14 h > as-cast > T4. Precipitated phases formed along the grain boundary after the aging treatment, resulting in the fact that the corrosion reaction increased rapidly. However, precipitated phases of peak-aged sample were distributed uniformly to form a corrosion barrier.

(3) The microstructural influence of corrosion can be understood from the interaction of the following three factors: (i) the surface film formed on the surface of the α -Mg can be more or less effective in hindering corrosion and more or less effective in controlling the form of corrosion as uniform corrosion or localized corrosion, (ii) the precipitated phases $\text{Mg}_{12}(\text{Y,Nd})$ and $\text{Mg}_3(\text{Y,Nd})$ can cause microgalvanic acceleration of corrosion of the matrix α -Mg, and (iii) the precipitated phases can act as a corrosion barrier and hinder the corrosion propagation in matrix.

(4) The electrochemical results confirmed that the microstructure and precipitated phases of Mg-5Y-1.5Nd alloys played an important role, which indicated that the corrosion resistance of Mg-Y-Nd alloy can be modified via heat treatment. This research is expected to provide a guiding insight into developing the Mg-Y-Nd based alloys.

Competing Interests

The authors declare that they have no competing interests.

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