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Effect of Arsenic Alloying on the Microstructure and Corrosion Properties of Mg-Al Alloy

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We report that arsenic alloying significantly accelerates the corrosion process of Mg-Al alloy. The microstructure of Mg-Al-As alloy consists of α -Mg grains, β -Mg₁₇Al₁₂ particles, and Mg₃As₂ particles. Gravimetric tests revealed that the average corrosion rates of Mg-Al-As and AZ80 magnesium alloys are 75.48 mm/y and 7.06 mm/y. The acceleration of the corrosion process in Mg-Al-As alloy is mainly attributed to the Mg₃As₂ phase, which quickly hydrolyzes to form a Mg(OH)₂ film.

Keywords: Magnesium corrosion, Electrochemistry, Arsenic

1. INTRODUCTION

Magnesium alloys, the lightest metallic engineering materials, exhibit a great number of applications in the medical, automobile and aerospace industries, with the characters of low density, superior castability, high specific strength, and good biocompatibility[1,2]. However, their poor corrosion resistance significantly restricts the application of magnesium alloys. Mg-Al alloys are extensively used in commercial applications due to the appropriate aluminum contents, which are typically 2-10%. The corrosion rate of Mg-Al alloys decreases with increasing amounts of aluminum[3–5]. The Mg₁₇Al₁₂ phase(β -phase) is the main compound formed during solidification of Mg-Al alloys along the grain boundaries and interdendritic interfaces. Compared with the Mg matrix, the Mg₁₇Al₁₂ phase is more inert to reaction with chloride solutions. Consequently, a continuous Mg₁₇Al₁₂ phase can protect the matrix, and a discontinuous Mg₁₇Al₁₂ phase can couple with the matrix to accelerate corrosion. At the same time, Al-rich substrate can also act as a barrier against

corrosion[6]. On the other hand, Al₂O₃ existing in the corrosion film of Mg-Al alloys can significantly improve the protectiveness of the film[7].

Many researchers have aimed to modify Mg-Al alloys by the addition of Li, Ca, Mn, rare elements, etc.[8–10]. Choi studied the effect of minor Ti addition to AZ31, AZ61 and AZ91 alloys on the corrosion behavior in 3.5 wt.% NaCl solution and reported that minor Ti addition can significantly improve the corrosion resistance of AZ31 and AZ91, but it makes AZ61 more vulnerable to corrosion[11–13]. Lia found that indium addition can enhance the anodic activity of AZ63 magnesium alloy in 3.5 wt.% NaCl solution. Kohama et al. found that Ca addition can improve the discharge properties of Mg-Al-Mn alloy by forming a less-adhesive film[14].

Arsenic lies in the fourth period and the fifth main group of the periodic table of elements, which is rarely used for magnesium alloys. Eaves proposed that arsenic can significantly inhibit magnesium corrosion under conditions of acidic bulk solution by inhibiting hydrogen atom recombination[15]. G. Williams et al. researched the localized corrosion of Mg-As alloy by SVET and found that arsenic can suppress cathode kinetics. The impact of arsenic on the corrosion behavior of Mg-Al alloys has not been studied[16]. The aim of the present study was to investigate the influence of arsenic addition on the corrosion behaviors of Mg-Al alloys in naturally aerated 3.5 wt.% NaCl solution by gravimetric, hydrogen evolution and electrochemical measurements.

2. EXPERIMENTAL

2.1. Test materials

AZ80 and Mg-Al-As magnesium alloys (hereafter referred to as alloy 1 and alloy 2) were prepared to commercial pure Mg, pure Al, pure Zn, MnCl₂ (99.9%) and pure As (99.5%). First, pure magnesium was melted in a resistance furnace with a graphite crucible under the protective atmosphere of argon gas. Next, pure Al and Zn were put into the crucible at 720 °C. After 20 min of standing, MnCl₂ was put into the melted alloy with stirring. Then, pure As was added to the crucible at 720 °C. After 15 min of standing, the melt was poured into a copper mold with water cooling. Chemical compositions of the alloys were measured by inductively coupled plasma atomic emission spectroscopy (ICPAES) and are listed in Table 1.

Table 1. Nominal con	positions of	the material	tested.
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Material	Elements(wt.%)						
_	Al	Zn	Mn	Fe	As	Mg	
Alloy 1	7.53	0.729	0.123	0.007	0	Bal.	
Alloy 2	7.89	0.0239	0.059	0.006	0.154	Bal.	

2.2. Microstructure observation

Each specimen for microstructure observation was ground with 5000# SiC abrasive papers,

polished to 0.5 μ m, and subsequently etched in an acetic-nitric reagent (1 g oxalic acid + 1 mL acetic acid + 1 mL nitric acid+ 150 mL H₂O). The samples were examined by OM and SEM measurement. The constituent phases were identified by XRD and EDX.

2.3. Corrosion tests

2.3.1. Gravimetric tests and hydrogen evolution

Specimens for gravimetric and hydrogen evolution tests were cut into cubes (15 mm *15 mm *5 mm). All surfaces of the samples were ground with 2000# SiC abrasive papers, and the flanks were bound with fishline. Then, the samples were immersed in 3.5 wt.% NaCl aqueous solution at 25 \pm 0.5 °C and placed in a container with a thermostat.

The volume of hydrogen was measured by a cylinder that was above a burette, and the burette covered the sample. The cylinder was initially full of solution, which was displaced by the evolution of hydrogen.

The samples for gravimetric tests were weighed before and after the immersion test with an accuracy of 0.01 mg. After the tests, the samples were immersed in 200 gL⁻¹ chromium trioxide (CrO₃), 10 g L⁻¹ silver nitrate (AgNO₃) and 20 g L⁻¹ barium nitrate (Ba(NO₃)₂) to remove the corrosion products.

The average corrosion rates were evaluated by the gravimetric tests and calculated by the following equation:

 $P_{AW} = (W_0 - W_1) * \rho^{-1} * S^{-1} * t^{-1}$ (1)

where P_{AW} is the average corrosion rate (mm/y), W_0 is the weight of the specimen before immersion, W_1 is the weight of the specimen after immersion, ρ is the density of the specimen, S is the exposed area of the specimen, and t is the immersion time.

2.3.2. Electrochemical tests

Electrochemical tests were carried out in 3.5 wt.% NaCl solution at 25 °C using a standard three-electrode system with an electrochemical workstation (CHI 660e). A platinum plate (20 mm * 20 mm * 10 mm), a saturated calomel electrode (SCE) and a Luggin capillary were used as the counter electrode, reference electrode and electrolytic bridge, respectively. The exposed area of the work electrodes was 1 cm². The potentiodynamic polarization curves were scanned from -100 mV to +250 mV relative to the open circuit potential (OCP) with a scan rate of 1 mV/s. Electrochemical impedance spectroscopy (EIS) measurements were conducted for immersion times ranging from 1 h to 24 h. The frequency ranged from 100 kHz to 10 mHz, and the amplitude of the sinusoidal potential signal was 5 mV around the OCP. The measurements were repeated at least three times to ensure reproducibility of the polarization measurements.

3. RESULTS AND DISCUSSION

3.1. Microstructures



Figure 1. Optical micrographs of alloy 1 (a) (b) and alloy 2 (c) (d).

Fig. 1 shows the OM images of alloy 1 and alloy 2. It can be seen in Fig. 1 a and b that the microstructure can be divided into the matrix, the netlike gray zone, and island-shaped precipitates partly surrounded by the gray zone. Some lamellar precipitates were black and lamellar, which were different from other precipitates. In contrast to alloy 1, alloy 2 had less gray zone, more black lamellar precipitates and more tiny particles that were dispersed more uniformly. According to the XRD patterns shown in Fig. 2, alloy 1 consisted of the α -Mg and β -Mg₁₇Al₁₂ phases and alloy 2 was composed of the α -Mg, β -Mg₁₇Al₁₂ and Mg₃As₂ phases.



Figure 2. XRD patterns of the two investigated alloys.



Figure 3. SEM micrographs of alloy 1 (a) (b) and alloy 2 (c) (d). The BSE image (e) of alloy 2 in the unetched condition.

Fig. 3 shows the SEM micrographs of the investigated alloys. According to the SEM and EDS results, many divorced eutectic α -phase enriched Al atoms in alloy 1 were network-like and enclosed the Mg₁₇Al₁₂ phase. Meanwhile, part of the α -phase decomposed into the Mg₁₇Al₁₂ phase[17,18]. On the other hand, the Al-Mn-Fe phase was found in alloy 1. Because of the similarity between the arsenic L_{a2} (1.282 keV) and the magnesium K_{a1} (1.254 keV), the EDS mapping of alloy 2 was hard to analyze. The solubility of arsenic in magnesium is very low, and the Mg₃As₂ phase can form by the eutectic reaction between magnesium and arsenic only when the levels of arsenic are up to 65%[19]. Hence, it is difficult to discuss the diversity of arsenic in alloy 2 due to its low content. Alloy 2 had a significant lamellar eutectic structure that was different from alloy 1. The Mg₃As₂ phase, which was adjacent to the Mg₁₇Al₁₂ phase, was detected in the unetched sample of alloy 2.

At the beginning of cooling, α -Mg was generated first, and As atoms were exhausted to the solid-liquid interface from primary α -Mg due to the low solid solubility. Then, enriched As atoms generally inhibited the diffusion of Al atoms with increasing arsenic solubility. Along with constant cooling, a eutectic reaction happened to yield products that were eutectic β -Mg₁₇Al₁₂ and α -Mg, which enriched the high solubility of Al and decomposed into a lamellar eutectic structure through solid-state transformation. Because of the impact of As atoms, the Al content for the eutectic reaction in alloy 2 was less than that in alloy 1. Hence, the eutectic structure of alloy 2 was not netlike.

Material	Elements						
	position	Mg	Al	Mn	Fe	Zn	As
Alloy 1	1	88.41	11.26	-	-	0.33	-
	2	89.88	9.82	-	-	0.3	-
	3	68.87	29.08	-	-	2.05	-
	4	55.61	32.25	11.37	0.76	-	-
Alloy 2	5	66.71	33.2	0.04	0.04	-	-
	6	21.17	60.22	16.04	2.5	0.07	-
	7	77.65	-	0.08	-	-	22.27
	8	73.33	26.54	-	0.13	-	-

Table 2. EDS analysis results for the marked positions in Figure 3.

3.2. Electrochemical tests

Fig. 4 shows the potentiodynamic polarization curves of the investigated alloys over 72 h in 3.5 wt.% NaCl solution. The values of corrosion potential (E_{corr}) and corrosion current density (i_{corr}) and the slopes of cathodic polarization (β_c) and anodic polarization (β_a) calculated from the potentiodynamic polarization are listed in Table 3. Upon the addition of As, E_{corr} shifted to significantly negative values. At the same time, the higher cathodic and anodic currents in alloy 2 meant that the corrosion reaction was more active. The smaller β_c and β_a values, which were related to the degree of the corrosion reaction, also supported this conclusion.



Figure 4. Potentiodynamic polarization curves of the investigated alloys in 3.5 wt.% NaCl solution over three days at 25±1°C.

	Time	Ecorr	$\beta_c(mV/$	$\beta_a(mV/$	$I_{corr}(A \cdot cm^{-2})$
			decade)	decade)	
Alloy 1	72 h	-1.525	409.17	270.48	1.376e-3
Alloy 2	72 h	-1.552	145.37	156.79	2.35e-03

Table 3. The values extracted from the polarization curves of the investigated alloys.

Fig. 5 shows the EIS spectra of the investigated alloys and the equivalent circuit that can be applied to all EIS spectra. The EIS spectra are characterized by a capacitive loop at high frequency (HF) and medium frequency (MF) and an inductive loop at low frequency. It is generally believed that the first capacitive loops at high and medium frequencies are related to the charge transfer resistance of the working electrode and the capacitance of the electric double layer at the interface of the electrode/electrolyte [20–24].



Figure 5. Electrochemical impedance spectrum of (a) alloy 1 and (b) alloy 2 in 3.5 wt.% NaCl solution for different immersion times at 25±1°C; Equivalent circuit for EIS of the investigated alloys.

Table 4. Values calculated from EIS of the investigated alloys.

	Time	R _s (Ω·cm₋2)	$R_f(\Omega \cdot cm_{-2})$	CPE ₁ , ⊤(10 ⁻⁵ F ·cm-2)	CPE ₁ , p	$R_{ct}(\Omega \cdot cm_{-2})$	CPE _{2,T} (10 ⁻⁵ F⋅ cm ₋₂)	CPE _{2,P}	$L_1(H \cdot cm_{-2})$
Alloy 1	1 h	1.45E+00	3.22E+02	7.53E-05	9.46E-01	2.11E+02	1.21E-05	9.50E-01	2.84E+01
	5 h	8.28E-01	3.23E+02	1.20E-04	8.84E-01	2.19E+02	1.36E-05	9.41E-01	2.35E+01
	10 h	8.146e-1	3.101e+2	6.722e-5	9.422e-1	1.709e+2	9.722e-6	9.899e-1	2.106e1
	72 h	1.89E+00	8.24E+01	6.61E-05	9.57E-01	1.05E+02	9.57E-03	4.53E-01	2.97E+00
Alloy 2	1 h	5.09E+00	5.57E+02	1.46E-04	7.66E-01	3.78E+02	9.27E-06	1.00E+00	6.44E+01
	5 h	5.38E+00	1.41E+02	3.08E-05	9.09E-01	4.51E+01	2.01E-03	7.70E-01	7.69E+00
	10 h	2.74E+00	4.83E+01	2.16E-03	5.27E-01	2.49E+01	2.70E-05	1.00E+00	3.84E+00
	72 h	1.64E+00	1.14E+01	2.85E-04	8.44E-01	3.14E+00	3.08E-04	8.08E-01	8.68E-01

The inductive loop in the low-frequency region may be attributed to the adsorption and desorption of corrosion products on the electrode surface[23,25,26]. The equivalent circuit that considers the film of corrosion products and the charge transfer process on the surface of the samples is composed of three resistances: two constant phase elements (CPE) and an inductor. R_s is the solution resistance, R_f is the film resistance, R_{ct} is the charge transfer resistance, Q_1 and Q_2 are used to fit the time constant of the EIS spectrum, and L_1 is related to the adsorption of the intermediate product of the corrosion process. A constant-phase element has a special capacitance and associated phase angle.

In Fig. 5, all parameters of the capacitance arcs decreased during the immersion period, but alloy 1 and alloy 2 demonstrated distinct development trends. At the beginning of the 10 h, the capacitance arcs of alloy 2 shrank quickly; however, alloy 1 was more stable. The fitted data in Table 4 show the same trend as seen in Fig. 5. The decreasing rates of R_f and R_{ct} in alloy 2 were quicker than in alloy 1 during the immersion time. On the other hand, the R_f and R_{ct} of alloy 1 were always higher than those of alloy 2 and even reached six times higher than those of alloy 2 for 10 h.

3.3. Weight loss measurements

Fig. 6 shows the weight loss of the investigated alloys in 3.5 wt.% NaCl solution over 5 days. For alloy 1, the average corrosion rate over 5 days was 7.06 mm/y, and the rate was 75.48 mm/y for alloy 2. Fig. 7 shows the volumes and rates (V_h) of evolution hydrogen for the investigated alloys. At the beginning of the 10 h, alloy 2 reached a rapid average rate of hydrogen evolution that was 0.35061 ml·h⁻¹·cm⁻². The hydrogen volume of alloy 2 during the 10-h immersion was 2.12978 ml·cm⁻², which was even more than the hydrogen volume of the 50-h immersion of alloy 1. According to Fig. 9, the degressive V_h of alloy 2 after 48 h can be attributed to the decreasing surface area.



Figure 6. Average weight loss of the investigated alloys during immersion testing in 3.5 wt.% NaCl solution over five days at 25±1°C.



Figure 7. Hydrogen evolution (a) volume and (b) rate of the investigated alloys in 3.5 wt.% NaCl solution over three days at 25±1°C.

Fig. 8 shows the corroded surfaces of the samples after immersion times of 24 h. Alloy 1 clearly exhibited a pattern of typical pitting corrosion. At the same time, the black dendritic region, which corroded more seriously than did the white region, was α -phase. Furthermore, it can be observed that alloy 2 had undergone severe corrosion. Fig. 8 shows the surface morphologies of the investigated alloys. Combined with the results of Fig. 9, the corrosion types of alloy 1 and alloy 2 were confirmed as pitting corrosion and general corrosion, respectively. After immersion for 5 days, a size reduction was obviously observed in alloy 2, and the weight of the alloy 2 sample was reduced by 72.88%. Compared to alloy 1, alloy 2 had a significantly higher corrosion rate.



Figure 8. The corroded surfaces of the samples after an immersion time of 24 h in 3.5 wt.% NaCl solution at 25±1°C, examined by SEM: (a), (b) alloy 1; (c), (d) alloy 2.



Figure 9. The morphologies of the investigated alloys after gravimetric testing.

For analyzing the corrosion products of the Mg-Al-As alloy, $AgNo_3$ solution was used according to the Gutzeit test[27]. As AsH₃ gas passes through AgNo₃ solution, the gas deoxidizes unipositive Ag⁺ ions to form a black precipitate that can be clearly observed. The chemical equation for the Gutzeit test is:

 $AsH_3 + 6Ag^+ + 3H_2O = H_3AsO_3 + 6Ag + 6H^+$ (1)

3.4. Discussion

The present study indicates that the corrosion rate of Mg-Al alloy is significantly accelerated by the addition of arsenic. As mentioned above, alloying with arsenic reduced the content of the Mg₁₇Al₁₂ phase that induced the isolated island structure of the Mg₁₇Al₁₂ phase. Because the potential of the Mg₁₇Al₁₂ phase is higher than that of α -Mg solid solution, it acts twofold according to its structure[3,28]. The netlike Mg₁₇Al₁₂ phase encloses and protects the matrix due to its inertia. However, isolated Mg₁₇Al₁₂ phase attaches to the matrix to form galvanic coupling[13,29]. From the microstructures of the Mg₁₇Al₁₂ phase, the Mg-Al-As alloy is vulnerable to corrosion. However, it is difficult to merely attribute the initial high rate of corrosion to the microstructures of the Mg₁₇Al₁₂ phase. On the other side, Mg₃As₂ phase was synthesized in the Mg-Al-As alloy by the eutectic reaction between Mg and As.

The Tafel curve, EIS, gravimetric testing and hydrogen evolution results reveal that alloying Mg-Al alloy with arsenic yields lower corrosion resistance than that of the AZ80 alloy. According to the EIS results, the corrosion process of the Mg-Al-As alloy was constantly accelerating during the entire immersion time, but the AZ80 alloy reacted slowly at the beginning of the 10 h. It is certain that there were some matters or structures that existed in the Mg-Al-As alloy that could rapidly start the

process of corrosion. After an immersion of 24 h, most of the surface area of the Mg-Al-As alloy had undergone severe corrosion. However, the AZ80 alloy exhibited pitting corrosion, which is a typical mechanism of Mg-Al alloys due to its heterogeneity [13,17]. After 5 days of immersion, the sample (15 mm *15 mm * 5 mm) of the Mg-Al-As alloy had lost 72.9 wt.%, which was significantly different from the results observed for AZ80. Meanwhile, AsH₃ was detected in the corrosion products of alloy 2, which means that the hydrolytic reaction of Mg₃As₂ should be considered in the corrosion mechanism. Although merely a small quantity of Mg₃As₂ existed in the Mg-Al-As alloy, Mg₃As₂ was the only obvious difference, and the Mg(OH)₂ of the hydrolytic reaction is significant. Hence, the mechanism of quick corrosion in the initial stage can be reasonably correlated with the hydrolytic reaction. The chemical equation of the hydrolytic reaction is:

 $Mg_3As_2+6H_2O=3Mg(OH)_2+2AsH_3$ (2)

 $Mg(OH)_2$ is one of the hydrolytic products. The hydrogen evolution reaction that occurred at the $Mg(OH)_2$ film of the Mg surface was enhanced in contrast to that on a pristine Mg surface[30]. In other words, the $Mg(OH)_2$ film on the surface will accelerate corrosion. Combined with the hydrolytic reaction, the Mg_3As_2 phase absolutely acts to quickly start corrosion. Due to the lack of a protective structure, the corrosion is accelerated with the expanding area of $Mg(OH)_2$ film. Hence, the corroded area was similar to an expanding ripple on water.

The overall corrosion processes of the Mg-Al-As alloy are divided into two steps: (1) The Mg₃As₂ phase, which rapidly starts corrosion, quickly hydrolyzes to form a local film of Mg(OH)₂ on the sample surface, at the margin of which the matrix gradually corrodes. Hence, the corrosion area radiates to the uncorroded surface. (2) Because of the island-like β -Mg₁₇Al₁₂ phases, many microelectric couples are formed to accelerate the corrosion rate of the α -Mg matrix. Meanwhile, there is lack of a protective structure, and the corrosion type is general corrosion, which is different from the pitting corrosion of the dAZ80 alloy.

Although the poor corrosion resistance limits the application of Mg-Al-As alloy in structural material, the excellent discharge properties provide a bright future as a battery material. Both the uniform surface and low resistance of the reaction are excellent characteristics for Mg batteries.

4. CONCLUSION

The effect of arsenic alloying was investigated in 3.5 wt.% NaCl solution. According to the results, the average corrosion rate of Mg-Al-As alloy was ten times higher than that of the magnesium alloy AZ80, which means that the Mg-Al-As alloy is more vulnerable to corrosion. In the investigated alloys, the Mg₃As₂ phase was the important difference, which only existed in the Mg-Al-As alloy. The hydrolysis reaction of the Mg₃As₂ phase rapidly formed a film of Mg(OH)₂, under which the magnesium alloy corroded quickly.

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