Effect of Sn content on the mechanical properties and corrosion behavior of Mg-3Al-xSn alloys

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Abstract

The effect of Sn content on the mechanical properties and corrosion behavior of Mg-3Al-xSn alloys was investigated by SEM-EDXS, XRD, electrochemical measurements, and scanning Kelvin probe force microscopy (SKPFM). The results showed that when the Sn content was 1.4 wt%, Sn dissolved in the α-Mg matrix and then precipitated as an intermetallic compound (Mg2Sn). The combined results of mass loss, hydrogen evolution, and electrochemical measurements indicated that Mg-3Al-1Sn had a low corrosion rate. The SKPFM results showed that the Volta potential of Mg2Sn particles, Al-Mn, and β-Mg17Al12 phases were 100, 80, and 50 mV higher than the matrix, respectively. Therefore, the Mg2Sn phase that formed in Mg-3Al-xSn served as a local cathode due to its high potential, which accelerated microgalvanic corrosion along with the secondary local cathode (Al-Mn). The Sn solution strengthening and secondary phase strengthening (fine Mg2Sn particles) improved the mechanical properties of the Mg-3Al-xSn alloys.

1. Introduction

Over the past decade, Mg-Al series alloys have been widely used due to their low density, high specific strength, good electrical conductivity, and easy mechanical processing [1–5]. However, their poor mechanical properties and corrosion resistance have restricted their applications in automobiles, 3C products, and aeronautical fields [4–6]. Therefore, minor alloying elements are often added to modify the alloy microstructure and form new intermetallic compounds, which improves their tensile strength and corrosion resistance.

Sn is relatively cheap and can improve the corrosion resistance, mechanical properties, and castability of Mg alloys [7–11]. Park et al [12] revealed that when Sn was added into Mg-5Al-1Zn alloys, it stabilized the Mg(OH)2 layers on the alloy surface, which dramatically improved the corrosion resistance. Zeng et al [13] reported that Sn decreased the cathodic reaction and the hydrogen evolution rate of alloys due to its high hydrogen overpotential. Moreover, Polina et al [14] indicated that Sn dissolved in the α-Mg matrix of AT53 and AT34 alloys and facilitated the formation of a protective layer that reduced the anodic dissolution rate of the matrix [15]. However, excessive Sn addition to Mg alloys can result in the formation of Mg2Sn intermetallic compounds, which increase the hydrogen evolution rate and deteriorate the corrosion resistance of as-cast AM70 magnesium alloys [16, 17]. Mg2Sn intermetallic compounds in magnesium alloys were assumed to be the cathode phase responsible for severe microgalvanic corrosion [18, 19]. However, there remains an incomplete understanding of how Mg2Sn phases influence the microgalvanic corrosion of Mg-Al alloys.

Recently, scanning Kelvin probe force microscopy (SKPFM) has been used to determine the Volta potential difference between the α-Mg matrix and secondary phases, such as Al2Y, Al2Nd, Al2Gd, and Al-Mn [20–23]. This has increased the understanding of the local microgalvanic corrosion behavior of such alloys. Therefore, the
effect of Sn content on the mechanical properties and corrosion behavior of Mg-3Al-xSn alloys were investigated by SEM-EDXS, electrochemical measurements, and SKPFM. The main focus was on the effect of Mg2Sn on the mechanical properties of Mg-3Al-xSn alloys and their corrosion mechanism.

2. Experimental procedure

Mg-3Al-xSn (x = 0, 1.0, 1.5, 2.0) alloys were prepared using pure Mg (>99.9 wt%), Al (>99.9 wt%), Sn (>99.9 wt%), and Al-10Mn master alloy ingots. First, pure Mg and Al were heated to 720 °C in a steel crucible inside a resistance furnace, and the melt was protected by a mixture of CO2 and SF6 gases in a volume ratio of 6:1. When the temperature of the melt decreased to 680 °C, Sn wrapped in aluminum foil was added. After holding for 15 min at 700 °C, the melt was poured into a metal mold (180 mm in length and 40 mm in diameter) preheated to 200 °C. Subsequently, these experimental alloys were processed into Φ38 × 40 mm by a lathe. Samples were first homogenized at 400 °C for 12 h in a box-type resistance furnace and then hot extruded at 300 °C into bars with 16 mm diameters at an extrusion speed of 2.5 mm s⁻¹. The chemical compositions of the specimens were analyzed by inductively coupled plasma atomic emission spectroscopy (ICP-AES), and the results are shown in Table 1. The extruded bar was processed into a standard tensile specimen, and the remainder was used for microscopic observations and electrochemical tests. Tensile tests were carried out at a strain rate of 1 mm min⁻¹ using an electronic tensile testing machine (CMT5105) at room temperature.

Table 1. Chemical composition of the Mg-3Al-xSn magnesium alloys (wt.%).

| Alloys   | Al  | Mn  | Sn  | Mg   |
|----------|-----|-----|-----|------|
| Mg-3Al   | 2.96| 0.34| N.D.*| Balance |
| Mg-3Al-1Sn| 2.98| 0.36| 0.94| Balance |
| Mg-3Al-1.5Sn| 2.93| 0.32| 1.42| Balance |
| Mg-3Al-2Sn| 2.94| 0.34| 1.81| Balance |

N.D.*: Not detectable.

Figure 1. Scanning electron micrographs in backscattered electron mode of as cast alloys: (a) Mg-3Al, (b) Mg-3Al-1Sn, (c) Mg-3Al-1.5Sn, and (d) Mg-3Al-2Sn.
To obtain microstructural characteristics, all samples were cut from as-cast and extruded bars, then polished by 600–2000 # silicon carbide (SiC) paper and 5–0.5 μm diamond polishing paste on an MP-2A metallographic polishing machine. Then, sample surfaces were corroded by a 4% nitric acid/ethanol solution. Microstructural characteristics were observed by optical microscopy (OM, Nican M200) and scanning electron microscopy (SEM, TESCAN VEGA 3) equipped with energy-dispersive x-ray spectroscopy (EDXS).

Surface potential maps of the phases in the Mg-3Al-xSn alloys were obtained by atomic force microscopy (AFM, Agilent Technologies 5500 Scanning Probe Microscope) working in the KFM-AM mode. A probe with a gold-plated tip was used to simultaneously acquire topographic and surface potential images. The images were scanned with a pixel resolution of 512 × 512 and a scan rate of 1 Hz. The tip force constant was 1 N m$^{-1}$, and all measurements were conducted at room temperature.

Specimens for corrosion tests, including potentiodynamic polarisation, H$_2$ gas collection, and mass loss evaluation, were ground with a 2000 grit SiC paper, cleaned in alcohol, and then dried in air. The weight of each sample was measured using an analytical balance with an accuracy of 0.0001 g. Corrosion experiments were performed by placing samples in a 3.5 wt% NaCl solution for 24 h at room temperature. H$_2$ was collected into an inverted acid dropper above the corroded samples. The volume of H$_2$ was obtained by measuring the change in the liquid level. Then, samples were soaked in a mixed solution of 20% CrO$_3$ and 1% AgNO$_3$ for 15 min to remove corrosion products. Finally, samples were dried in the air and weighed. All tests were repeated five times to assess their reproducibility. The hydrogen evolution and weight loss rates of samples were calculated by the following formulas $[24$–$27]$:

![Figure 2. The EDX spectrum of as-cast Mg-3Al-xSn alloys: (a) corresponding EDS of Mg-3Al, (b) corresponding EDS of Mg-3Al-1.5Sn and (c) corresponding EDS of Mg-3Al-2Sn.](image-url)
where \( M_1 \) is the original weight, and \( M_2 \) is the weight after corrosion for 24 h; \( H_1 \) is the original liquid level of the acid dropper, and \( H_2 \) is the liquid level after 24 h; \( A \) is the total surface area of the samples; \( t \) is the corrosion time.

All electrochemical measurements were carried out on an electrochemical workstation (Princeton P4000). The three-electrode system was composed of a working electrode with an exposed area of 1 cm\(^2\), a platinum counter electrode, and a saturated calomel electrode as the reference electrode. Polarization scans of the potentiodynamic polarization tests were in the range of \( \pm 250 \text{ mV}_{\text{SCE}} \) with a scan rate of \( 1 \text{ mV}_{\text{SCE}} \text{s}^{-1} \) after samples were immersed for 15 min to stabilize the open circuit potential. Electrochemical impedance spectra were obtained at frequencies from 100 kHz to 10 MHz. The disturbance amplitude of the open circuit potential was 5 mV. The EIS results were fitted using ZView-Impedance software, and the fitting errors of the parameters were less than 10%. All tests were repeated five times to reduce errors.

3. Results

3.1. Microstructure

Figure 1 presents the SEM images of the Mg-3Al alloys with different Sn contents. Two different phases were observed, and the corresponding EDS results are listed in figure 2. As shown in figure 1(a), the microstructure of the matrix and Mg-3Al-1Sn alloys consisted of an \( \alpha \)-Mg matrix and \( \beta \)-Mg\(_{17}\)Al\(_{12}\) and Al-Mn particles, according to the EDS results (figure 2(a)). When 1.5 wt.% Sn was added into the alloy, some bright particles with sizes of \( \sim 2 \mu\text{m} \) were formed (figure 1(c)). The corresponding EDS results (figure 2(b)) indicated that these particles were an Sn-rich intermetallic compound. As the Sn content increased to 2.0 wt.%, the Sn-rich intermetallic compound particles coarsened, as shown in figures 1(d) and 2(c).

To further analyze the Sn distribution in the alloy, EDXS elemental maps of Mg-3Al-2Sn are shown in figure 3. Significant amounts of Sn were evenly distributed throughout the matrix alloy, but it tended to accumulate on the white particles. It can be inferred that a small amount of Sn was present in the matrix alloy in the form of a solid solution. When the content of Sn exceeded the solid solubility of the Mg matrix, an Sn-rich intermetallic compound formed, as shown in figure 3(d). The electronegativities of Mg, Al, Mn, and Sn are 1.31, 1.61, 1.55, and 1.96, respectively. The electronegativity difference between Mg and Sn was higher than that of the other two elements, which suggests that the Mg-Sn phase would form more easily than others.
The Sn-rich intermetallic compounds in the as-cast Mg-3Al-2Sn alloy were analyzed by XRD, as shown in figure 4. Many peaks corresponding to $\alpha$-Mg and $\beta$-Mg$_{17}$Al$_{12}$ were observed in the patterns of the Mg-3Al-xSn alloys, while peaks associated with Mg$_2$Sn were found in the patterns of the Mg-3Al-1.5Sn and Mg-3Al-2Sn alloys. Therefore, it was concluded that the Sn-rich intermetallic compound was Mg$_2$Sn.

Optical microstructures of the hot-extruded Mg-3Al-xSn ($x = 0, 1.0, 1.5, 2.0$) alloys are shown in figure 5. Many coarse $\alpha$-Mg grains were observed in the Mg-3Al alloy after hot extrusion, and the average grain size of the alloy matrix was measured via the linear intercept method at 103 $\mu$m, as shown in figure 5(a). Upon increasing the Sn content, the grain size of the Mg-3Al alloys containing Sn decreased. At 1.5 wt.% Sn, the grain size decreased to 66 $\mu$m, as shown in figure 5(c), and the average grain size of the Mg-3Al-1Sn and Mg-3Al-2Sn alloys were 81 and 78 $\mu$m, respectively.

Recent studies have used the different Volta potentials between the secondary phases and the alloy matrix to understand local microgalvanic corrosion behavior [28]. In this study, the Volta potential of the intermetallic compounds in Mg-3Al and Mg-3Al-1.5Sn alloys were determined using SKPFM. Figures 6 and 7 show the topographic images, surface potential maps, and surface potential profiles of the $\alpha$-Mg matrix, $\beta$-Mg$_{17}$Al$_{12}$, Al-Mn, and Mg$_2$Sn phases in the Mg-3Al and Mg-3Al-2Sn alloys. As shown in figures 6 and 7, strip-shaped Al-Mn...
particles exhibited cathodic behavior with a potential 80 mV higher than the surrounding matrix alloy. In contrast, the potentials of the submicron Mg$_2$Sn particles and β-Mg$_{17}$Al$_{12}$ were 100 mV and 50 mV higher than the matrix, respectively. These values are similar to those observed for the Mg-Al alloys \cite{29}, which suggests that a more intense galvanic couple formed between Mg$_2$Sn and the α-Mg phase in the Mg-3Al-2Sn alloy.

3.2. Corrosion and electrochemical analysis

Figure 8 shows the hydrogen evolution and weight loss rates of Mg-3Al-xSn alloys after immersion in a 3.5\% NaCl solution for 24 h. The H$_2$ volume and weight loss rate first decreased with the Sn content and then increased significantly. Mg-3Al-1Sn exhibited low hydrogen evolution and weight loss rates of $0.18 \times 10^{-2}$ ml·cm$^{-2}$·h$^{-1}$ and $0.21 \times 10^{-2}$ mg·cm$^{-2}$·h$^{-1}$, respectively. It also had a maximum corrosion rate at 2.0 wt.\% Sn, i.e. an H$_2$ volume of $1.69 \times 10^{-2}$ ml·cm$^{-2}$·h$^{-1}$ and a weight loss rate of $2.07 \times 10^{-2}$ mg·cm$^{-2}$·h$^{-1}$. The immersion test results indicated that the lowest corrosion rate and best corrosion resistance were obtained at 1.0 wt.\% Sn.

The polarization curves of Mg-3Al-xSn in 3.5 wt.\% NaCl solution are displayed in figure 9. The corrosion potential ($E_{corr}$) of Mg-3Al-1Sn was higher than other alloys, and $E_{corr}$ of the matrix alloy was $-1.41$ V$_{SCE}$. It first increased to $-1.35$ V$_{SCE}$ for Mg-3Al-1Sn, then decreased to $-1.426$ V$_{SCE}$ for Mg-3Al-1.5Sn, and finally decreased to $-1.451$ V$_{SCE}$ for Mg-3Al-2Sn. The fitting data of the polarization curves in table 2 show that the alloy prepared with 1.0 wt.\% Sn exhibited the lowest corrosion current density ($I_{corr}$) of 34.56 μA·cm$^{-2}$.

Figure 6. (a) Topographic image; (b) surface potential map; (c) potential profile of Al-Mn intermetallics in the Mg-3Al alloy.
The Nyquist plots and Bode plots of Mg-3Al-xSn alloys immersed in 3.5 wt.% NaCl solution are shown in figure 10. The Nyquist plot of Mg-3Al consists of two capacitive loops and a short low-frequency inductive loop, while the other alloys all displayed a capacitive loop and an inductive loop. The capacitive loop diameters were ordered as follows: Mg-3Al-1Sn > Mg-3Al > Mg-3Al-1.5Sn > Mg-2Sn. It has been reported that a larger capacitive loop diameter corresponds to a higher corrosion resistance [30]; therefore, the addition of 1.0 wt.% Sn enhanced the anticorrosion performance of Mg-3Al. In figure 10 (b), the value of $|Z|$ for Mg-3Al-1Sn was also the highest, and most of the phase angles in figure 10 (c) exceeded 50°.

The corresponding equivalent circuit of each alloy is shown in figure 11. Figure 11 (a) represents Mg-3Al, and figure 11 (b) represents Mg-3Al-xSn (x = 1.0, 1.5, 2.0). In figures 11 (a) and (b), $R_s$ is the solution resistance, $R_f$ and CPE$_f$ are the resistance and capacitance of the corrosion product layer, $R_{ct}$ is the resistance to transfer charge during corrosion, $L$ and $R_L$ represent inductance resistance, and CPE$_{dl}$ is the capacitance of the electric double-layer at the interface of the metal surface and the corrosive medium [31–33].

Table 3 lists the fitting parameters obtained from the EIS spectra of the studied alloys. The polarization resistance ($R_p$) can be calculated as follows [34]:

$$R_p = R_s + R_f + R_{ct}$$  \(\text{(3)}\)

The $R_p$ value of Mg-3Al-1Sn was the highest (227.54 Ω·cm$^2$), while Mg-3Al-2Sn had the lowest value (133.37 Ω·cm$^2$). Metalknikov et al [14] reported that a high $R_p$ value indicates a better corrosion resistance, indicating that Mg-3Al-1Sn has the highest corrosion resistance.
3.3. Mechanical properties

The stress-strain curves and tensile properties of the extruded Mg-3Al-xSn alloys at room temperature are shown in figure 12. The ultimate tensile strength of the Mg-3Al-xSn alloys increased with the Sn content, reaching a maximum of 271 MPa at 1.5 wt.% Sn; however, as the Sn content further increased, the ultimate tensile strength decreased to 250 MPa. The elongation of the extruded Mg-3Al-xSn alloys decreased from 13.8% to 9.4% as the Sn content increased, as shown in table 4.
4. Discussions

4.1. Effect of Sn content on the corrosion resistance of alloys

Electrochemical potential differences between secondary-phase particles and the $\alpha$-Mg matrix influence the corrosion behavior, which is usually quantitatively examined to evaluate the microgalvanic corrosion behavior of alloys [20, 35]. In this study, the Volta potential of the Al-Mn phase showed a higher corrosion potential (30 and 80 mV) than that of the Mg$_{17}$Al$_{12}$ and $\alpha$-Mg phases, indicating that Al-Mn served as a local cathode in Mg-3Al. It was also revealed that Mg$_2$Sn served as a local cathode during the microgalvanic corrosion of Mg-3Al-Sn.

Figure 10. The Nyquist (a) and Bode (b), (c) plots of the Mg-3Al-xSn ($x = 0, 1.0, 1.5, 2.0$) alloys.
alloys. Therefore, the effect of microgalvanic couple corrosion between Mg2Sn/Al-Mn phases and the matrix increased the corrosion rate and decreased the polarization resistance of Mg-3Al-2Sn alloys.

To further understand the corrosion process of Sn-containing Mg-Al alloys, a schematic of the corrosion mechanism is shown in figure 13. According to the microstructural characteristics and SKPFM results, well-defined anodic α-Mg and cathodic Al-Mn phases within the Mg-3Al alloy were confirmed (figure 13(a)). When 1.0 wt.% Sn was added into the Mg-3Al alloy, most Sn dissolved in α-Mg (figures 3 and 13(b)), which decreased the weight loss rate of Mg-3Al-1Sn to $0.21 \times 10^{-2} \text{mg cm}^{-2} \text{h}^{-1}$. Similar results have been previously reported, in which Sn dissolved in the α-Mg matrix increased the corrosion resistance by stabilizing the oxide layer of corrosion products and reducing the anodic dissolution rate of the matrix [15–17, 19]. As the Sn content further increased, Mg2Sn formed in Mg-3Al-1.5Sn, which served as a local cathode due to its high potential and accelerated microgalvanic corrosion along with the secondary local cathode, Al-Mn (figure 13(c)). Significant amounts of cathodic Mg2Sn in Mg-3Al-2Sn further deteriorated its corrosion resistance, as shown in figure 13(d).

### 4.2. Effect of Sn content on the mechanical properties of alloys

The strengthening mechanism of Mg-3Al-xSn alloys may be attributed to grain refinement strengthening, solution strengthening, and secondary-phase strengthening. According to the Hall-Petch relationship, grain size has a significant effect on mechanical properties. In this work, the average grain size of the Mg-3Al-xSn alloys decreased from 103 to 66 μm during dynamic recrystallization due to hot extrusion. The lower average grain size increased the surface tension and the interaction of neighboring grains, which resulted in the formation of a hard deformation area near grain boundaries [36]. Therefore, the formation of fine grains increased the deformation resistance, which greatly improved the tensile properties of the extruded Mg-3Al-xSn alloys. Moreover, the atomic radius of Sn ($R_{\text{Sn}} = 0.14 \text{nm}$) is larger than Mg ($R_{\text{Mg}} = 0.136 \text{nm}$), which allowed Sn to dissolve and evenly distribute throughout the Mg matrix, as shown in figure 3. These solid solution Sn atoms caused lattice distortion, increased the dislocation motion resistance, remarkably impeded slip, and improved the strength of the magnesium alloy. For example, a Δσ increase of 29.8 MPa was caused by the dissolution of Sn in Mg [37]. As shown in figures 1(c) and 7, many fine Mg2Sn particles were uniformly distributed in the α-Mg grain in

### Table 2. Electrochemical parameters from the potentiodynamic polarization curves of the Mg-3Al-xSn (x = 0, 1.0, 1.5, 2.0) alloys.

| Specimen     | $E_{\text{corr}}$(V) | $i_{\text{corr}}$(μA·cm$^{-2}$) |
|--------------|----------------------|-----------------------------------|
| Mg-3Al       | -1.41                | 56.21                             |
| Mg-3Al-1Sn   | -1.35                | 34.56                             |
| Mg-3Al-1.5Sn | -1.426               | 83.70                             |
| Mg-3Al-2Sn   | -1.451               | 124.22                            |

Figure 11. Equivalent circuit of the EIS spectra for the Mg-3Al (a) and Mg-3Al-xSn (x = 1, 1.5, 2) alloys (b).
Table 3. Electrochemical parameters of studied alloys attained from the EIS data.

| Specimen       | R_s (Ω·cm²) | CPE_f (F·cm⁻²) | CPE_dl (F·cm⁻²) | C_film | n       | R_f (Ω·cm²) | C_dcl | n       | R_f (Ω·cm²) | L (H·cm⁻¹) | R_d (Ω·cm²) | R_p (Ω·cm²) |
|----------------|-------------|----------------|-----------------|--------|---------|-------------|--------|---------|-------------|-------------|-------------|-------------|
| Mg-3Al         | 3.3         | 9.6 × 10⁻⁶     | 0.99            | 71.4   | 1.0 × 10⁻⁴ | 0.66        | 63     | 87.9    | 101.1       | 167.3       |             |             |
| Mg-3Al-1Sn     | 3.1         | 1.8 × 10⁻⁵     | 0.96            | —      | —       | —           | 89.6   | 35.2    | 134.9       | 227.5       |             |             |
| Mg-3Al-1.5Sn   | 3.1         | 7.1 × 10⁻⁶     | 1.02            | —      | —       | —           | 60.6   | 18.1    | 80.1        | 143.8       |             |             |
| Mg-3Al-2Sn     | 3.2         | 7.3 × 10⁻⁶     | 0.98            | —      | —       | —           | 51.5   | 16.3    | 78.7        | 133.4       |             |             |
Mg-3Al-1.5Sn that facilitated the formation of pinning points and dislocations. During tensile deformation, an increasing number of pinning points and dislocations in the grain interior due to Mg$_2$Sn hindered deformation and increased the deformation resistance and ultimate tensile strength. However, the coarser Mg$_2$Sn particles in Mg-3Al-2.0Sn separated the matrix alloy under loading, which reduced the tensile strength. Meanwhile, the

Figure 12. Engineering stress-strain tensile curves for Mg-3Al-xSn (x = 0, 1.0, 1.5, 2.0) alloys.

Figure 13. Schematic image of corrosion change of the Mg-3Al-xSn alloys.
solution strengthening and secondary phase strengthening also weakened the plasticity of the alloys, as shown in figure 12 and table 2.

5. Conclusions

(1) Increasing the Sn content in Mg-3Al-xSn alloys first caused Sn to dissolve in the $\alpha$-Mg matrix, but Mg$_2$Sn particles precipitated when the Sn content exceeded 1.0 wt.%.

(2) As the Sn addition increased, the H$_2$ volume and weight loss rates first decreased, followed by a significant increase. Mg-3Al-1Sn had the best corrosion potential, and its larger polarization resistance (determined by EIS) indicated a better corrosion resistance.

(3) The Volta potentials of Mg$_2$Sn particles, Al-Mn, and $\beta$-Mg$_{17}$Al$_{12}$ phases were 100, 80, and 50 mV higher than the matrix, respectively. The results showed that the Mg$_2$Sn phase served as a local cathode and accelerated microgalvanic corrosion, along with the secondary local cathode, Al-Mn.

(4) The addition of Sn improved the mechanical properties of the Mg-3Al-xSn alloys by solution strengthening and secondary phase strengthening.

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