Microstructure evolution and electrochemical corrosion behavior of Al–Zn–Mg aluminum alloy

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Abstract
Electrochemical corrosion behavior of Sn-containing Al–Zn–Mg aluminum alloy has been studied in detail. The localized corrosion behaviors were studied by electrochemical impedance spectroscopy (EIS) analysis, and the potentiodynamic polarization measurements. The grain structure, grain-boundary microstructure, grain-boundary microchemistry, pitting and intergranular corrosion morphology were characterized and observed using SEM, EDS, TEM, SAED and HRTEM analyses. Based on these tests, the effects of grain-boundary on the corrosion resistance in our Sn-containing Al–Zn–Mg alloys before/after bake hardening were analyzed systematically. Finally, the relationship between chemical composition, microstructure evolution and corrosion behaviour was revealed. The results indicate that the bake hardening process improves the corrosion resistance compared to the pre-aging state. The grain size has little effect on the electrochemical corrosion behavior.

1. Introduction

Excellent mechanical properties are obtained of 7000 series aluminum alloy by regulating of aging treatment, which was suitable for the lightweight of aircraft, automobile and rail transit [1]. The using of 7000 series aluminum in structural and safety parts in the automotive industry has higher specific strength, specific stiffness and anti-collision absorption performance compared to conventional high strength steels [2–6].

However, the strengthening phase precipitated during aging treatment is an key factor affecting the corrosion behavior (pitting corrosion, intergranular corrosion, et al.), including the volume fraction, type, size and habit surface of precipitates. Abreu [7] studied the effect of T6 and T73 aging treatment on pitting corrosion and intergranular corrosion of AA7075 aluminum alloy using cyclic potentiodynamic polarization and electrochemical impedance spectroscopy, and found that local deep intergranular corrosion was easily formed of T6 state, while pitting corrosion of T73 state. The corrosion current density icorr of 7055 aluminum alloy under retrogression and reaging (RRA) state was less than that of T6 state [8, 9]. The change of corrosion behavior was due to the evolution of precipitates during aging treatment. In general, the precipitation phase of 7000 series aluminum is GP zone+η', GP zone+η' + η and η' + η after the T6 aging, the RRA treatment and the T7X treatment respectively. The precipitates enriched in the intra-granular and grain boundaries lead to pitting and intergranular corrosion. Metastable η' phase mainly causes intergranular corrosion, while equilibrium phase tends to cause intragranular pitting corrosion. With the increase of (Zn+Cu)/Mg ratio, the precipitate size was decreased, and the potential between grain boundary precipitates and the Al matrix was decreased which enhanced the intergranular corrosion resistance of Al–Mg-Zn-(Cu) alloys [10]. Xu [11, 12] investigated the relationship between surface integrity and corrosion fatigue behavior of 7B50-T7751, and found that the
surface layer microstructure refinement and compressive residual stress inhibited the occurrence and development of pitting and intergranular corrosion, prevented the initiation of corrosion fatigue at the surface. Al$_2$CuMg and Al$_7$Cu$_2$Fe intermetallic particles were the main precipitates in the 7A85 Al alloy, and the particles not only induced a defective film but also acted as cathode phases in the precipitate-matrix galvanic corrosion couple, which led to pitting corrosion initiation [13]. Finer grains and lower volume fraction and discontinuous distribution of the second phases were possessed of the spray formed 7050 aluminum which significantly enhanced the corrosion performance [14]. The correlations among the corrosion behaviour, grain-boundary microchemistry, and Zn content in Al–Zn–Mg-Cu alloys were studied and the results showed that low Zn content inhibited SCC propagation and other types of local corrosion due to the positive potential of grain boundary precipitates and less SCC cracking initiation sites [15].

In summary, the research on the corrosion resistance of 7000 series aluminum alloy mainly focuses on the aviation field, in which the fabrication process of aluminum alloy plate mainly includes hot rolled thick plate, solution treatment, pre-stretching and aging treatment. However, as a structural part of automobile body, the plate needs to experience hot rolled thick plate, cold rolled thin plate, heat treatment, stamping forming and paint baking treatment. The performance requirements, processing flow and service state of 7000 series aluminum alloy in the above two application fields are very different. Therefore, it is necessary to study the corrosion behavior of Al–Zn–Mg under different pre-aging and bake hardening treatments, and to characterize the microstructure evolution which could provide technical support for its application in the field of automobile lightweight.

2. Materials and experimental methods

2.1. Materials preparation and corresponding treatment

The high-purity aluminum, pure Zn, Al–20 Mg alloy, Al–5 Mn alloy, Al–10 Cr, Al–10 Zr alloy, Al–50 Sn alloy, Al–10Ti alloy and Al–20Si alloy were used for the casting of experimental Al–Zn–Mg alloy. The compositions were listed in table 1.

The ingot was hot rolled and cold rolled into 1.5 mm thickness plate. Based on the production rhythm and fabrication process of aluminum material plant and automobile plant, the aging treatment route was formulated as shown in figure 1. The solution heat treatments were studied using salt bath furnace at 470 °C for 5 min. Different pre-aging treatments (PA) were carried out immediately at 80 °C for 60 min (namely P-1), 120 °C for 20 min (namely P-2) and 160 °C for 10 min (namely P-3), respectively, then natural aged (NA) for 14 days, and finally bake hardened (BH) at 180 °C for 20 min (namely B-1, B-2 and B-3, respectively).

The electrochemical measurements were performed on a RST5100 electrochemical workstation and the specimens were immersed in 3.5 wt% NaCl aqueous solution. The Pt foil and saturated calomel electrode were utilized as the counter electrode and reference electrode respectively.
The potentiodynamic polarization curves and EIS were conducted to investigate the corrosion behavior. The open circuit potential reached stable after immersion for 1200 s, and the EIS measurement was performed under the frequency range of 100 kHz to 0.01 Hz and sinusoidal voltage amplitude of 5 mV. Then the potentiodynamic polarization measurements were proceeded in the voltage range of $-0.9 \sim 0.6$ V.

2.2. Microstructure characterization
The samples were corroded by Keller reagent ($1.0 \text{ ml HF, 1.5 ml HCL, 95 ml H}_2\text{O, 2.5 ml HNO}_3$). The corrosion surfaces of different samples were analyzed by Quanta 450FEG field emission scanning electron microscope (SEM). The specimens were mechanically grounded to 40 $\mu\text{m}$ thickness and punched out into a diameter of 3 mm, and then etched by double-jet polishing using a solution of 1000 ml methanol and 500 ml HNO$_3$ at 12 V voltage and $-30 ^\circ \text{C}$ temperature. High Resolution Transmission Electron Microscopy (HREM) and Selected Area Electron Diffraction (SAED) were performed on a JEM-2010F microscope. The corrosion surface was observed by 3D-MLM optical microscope (Olympus OLS4100).

3. Electrochemical corrosion behaviors

3.1. Open circuit potential (OCP) test
Figure 2 represents an open circuit potential (OCP) curve of the experimental specimens. It is obvious that the OCP is basically stable during the full soaking time. Figure 2(a) shows the OCP curve after pre-aging treatment. The OCP values of the P-1, P-2 and P-3 treatment conditions relative to the saturated calomel reference electrode (SCE) are $-974.04$, $-929.81$ and $-938.66$ mV versus SCE, respectively. The OCP curve of the bake-hardening state (figure 2(b)) reflects a higher OCP value than the OCP curve of the pre-aging state. The OCP values of the B-1, B-2 and B-3 treatment conditions are $-935.105$, $-891.425$ and $-907.08$ mV versus SCE, respectively. The change pattern is similar to the pre-aging state, decreasing from B-2 to B-3 and then to B-1.

3.2. Dynamic potential polarization curve test
Figure 3 shows the influence of heat treatment parameters on the evolution of the dynamic potential polarization curves. The polarization curves were tested after the OCP was stabilized ($2100 \text{ s}$), and the anode

\[ E \text{ (mV vs SCE)} \]

\[ t \text{ (s)} \]

\[ P-1 \]

\[ P-2 \]

\[ P-3 \]

\[ B-1 \]

\[ B-2 \]

\[ B-3 \]

\[ \text{Pitting potential} \]

\[ \text{Unstable passivation point} \]

Figure 2. OCP curve of pre-aging treatments and bake hardening. (a) pre-aging state; (b) bake hardening state.

Figure 3. Dynamic potential polarization curves at different heat treatment processes. (a) pre-aging state; (b) bake hardening state.
branch exhibits a passivation tendency, but there is no significant initial passivation current density \( \text{ipp} \), and there is an unstable passivation point. Figure 3(a) shows the polarization curve of the pre-aging state. The corresponding fitting corrosion parameters are shown in Table 2. Both P-2 and P-3 have higher pitting potentials \( E_{\text{pit}} \), and their values are \(-884.62 \) and \(-889.18 \) mV versus SCE, significantly greater than the \(-1022.87 \) mV versus SCE of P-1, and P-2 and P-3 enter the passivation state in advance; self-corrosion current density \( i_{\text{corr}} \) gradually decreases in the order of P-1 > P-3 > P-2, and the self-corrosion potential increases in the order of P-1 < P-3 < P-2. The results show that the corrosion rate of P-2 alloy in the corrosive medium is the lowest and the corrosion tendency is the smallest. Therefore, it has good corrosion resistance. Figure 3(b) shows the curve of bake hardening state, and the corresponding fitting corrosion parameters are shown in Table 2. Similar to the pre-aging state, \( E_{\text{pit}} \) and self-corrosion potentials increase sequentially according to B-1 < B-3 < B-2 while \( i_{\text{corr}} \) gradually decreases according to B-1 > B-3 > B-2, and the unstable passivation point still exists. The difference from the pre-aging state is that the \( E_{\text{pit}} \) values of the three test panels differ greatly, and the B-2 panel enters the passivation state at a significantly faster rate than the B-1 and B-3 panels. The formation of unstable passivation

| Specimens | \( E_{\text{corr}} \) (mV versus SCE) | \( i_{\text{corr}} \) (\( \mu \)A cm\(^{-2} \)) | \( E_{\text{pit}} \) (mV versus SCE) |
|-----------|---------------------------------|-----------------|---------------------------------|
| P-1       | \(-1215.2\)                     | 62.1143         | \(-1022.87\)                    |
| P-2       | \(-1105.14\)                   | 36.952          | \(-884.62\)                    |
| P-3       | \(-1118.69\)                   | 41.372          | \(-889.18\)                    |
| B-1       | \(-1191.41\)                   | 48.591          | \(-995.75\)                    |
| B-2       | \(-1055.16\)                   | 20.932          | \(-822.66\)                    |
| B-3       | \(-1092.14\)                   | 28.253          | \(-898.1\)                     |

Figure 4. EIS spectra at different heat treatment processes. Nyquist (a) and Bode (b) of pre-aging style; Nyquist (c) and Bode (d) after bake hardening; (e) equivalent circuit.
points on the polarization curve is due to the lower potential of the precipitates and the large potential difference between the matrix and the substrate, which will easily destroy the passivation film on the surface of the sample and hinder the formation of a stable passivation region.

3.3. Electrochemical impedance spectroscopy (EIS) analysis

Figures 4(a) and (b) represent the Nyquist diagram and the Bode diagram of a pre-aging state, respectively, while figures 4(c) and (d) show the Nyquist diagram and the Bode diagram of a bake-hardening state, respectively. All the Nyquist plots exhibit similar shapes, consisting mainly of two capacitive reactance arcs of the first quadrant and a single sense arc of the fourth quadrant, wherein the capacitive reactance arc in the high frequency region represents the presence of an oxide film (formed in the air), and the capacitive reactance arc in the intermediate frequency region is high which is likely to be caused by metastable or stable pitting behavior. In addition, the inductive arc is related to the surface activity of the electrode, the relaxation reaction, and the surface activation adsorption of the corrosion product [16]. The arc radius of the pre-aging and bake-hardened aluminum alloy sheets are reduced in the order of P-2 > P-3 > P-1 and B-2 > B-3 > B-1, respectively. In the pre-aging state, the impedance arc radius of P-2 and P-3 differs little, and similar laws exist in B-2 and B-3 in the bake-hardening state. The average impedance arc radius of the BH state is larger than the PA state. In general, larger impedance arc, better corrosion resistance.

In the Bode diagrams of figures 4(b) and (d), the apparent capacitive reactance behavior occurs in the high frequency region, the maximum value of the phase angle (θ) is less than 35°, and the impedance mode (|Z|) value is below 10Ω cm². Generally, the corrosion resistance of the surface oxide film is qualitatively judged by the impedance modulus |Z| at a low frequency and the θ value at a high frequency. Therefore, the results show that the oxide film formed in the air on the aluminum alloy panels is thin and incomplete. In the intermediate frequency region (1000 Hz ~ 1 Hz), the maximum θ value is close to −60°, which is much smaller than −90°. The narrower peak width in the intermediate frequency region means that the integrity and compactness of the oxide film formed during the 2100 s immersion time are poor. For the surface of the sample with poor oxide film quality, the corrosion resistance mainly depends on the charge transfer process. At about 0.1 Hz, |Z| reaches the maximum value, and then, when f < f_max |Z| is gradually reduced and the value of θ develops in a positive direction. This phenomenon well explains the inductive behavior of the low frequency region.

The above impedance spectrum has two capacitive reactance arcs and one inductive impedance arc, and contains three time constants, so the equivalent circuit R(Q(R(Q(R(QL)))))) of figure 4(e) is used to fit and analyze all the electrochemical behavior of experimental aluminum alloy sheet specimens in 3.5% NaCl corrosion medium. Where Rf represents the solution resistance, Rm is the membrane resistance, Rct represents the charge transfer resistance, L and Rf represent the inductive reactance and corrosion product adsorption resistance, respectively; Q represents a constant phase angle element, which is used to replace the pure capacitance in the equivalent circuit Non-ideal capacitive reactance behavior, Qdl and Qadh are equivalent membrane capacitance and equivalent electric double layer capacitance, respectively.

Table 3 shows the corresponding impedance spectrum fitting parameters of the pre-aged P-1, P-2, and P-3 aluminum alloy panels. The Rs value does not change much, indicating that the soaking conditions are basically the same. The Rs values of P-2 and P-3 aluminum alloy panels are basically the same but significantly lower than that of the P-1 state. The change trend of Y_f value is contrary to the change trend of R_f value, which may be caused by the decrease of surface energy. The n_f and n_d values are between 0.5 and 1, so on the alloy/electrolyte interface it forms a non-ideal capacitor. The Rct values of the P-1, P-2 and P-3 aluminum alloy panels are 189.3, 404.5 and 388.7, respectively, and the Y_d values are 3.742, 0.2462 and 0.2675, respectively. The low Rct value and the high

| State | P-1 | P-2 | P-3 |
|-------|-----|-----|-----|
| R_f (Ω·cm²) | 2.546 | 2.013 | 3.451 |
| Y_f×10⁻⁷(Ω⁻¹·cm²·s⁻¹) | 2.945 | 5.874 | 5.567 |
| n_f | 0.6536 | 0.6177 | 0.6832 |
| R_m(Ω·cm²) | 7.615 | 5.664 | 5.622 |
| Y_d×10⁻⁴(Ω⁻¹·cm²·s⁻¹) | 3.742 | 0.2462 | 0.2675 |
| Q_dl | 0.6968 | 0.7413 | 0.7274 |
| R_c(Ω·cm²) | 189.3 | 404.5 | 388.7 |
| L_H(Ω·cm²) | 708.4 | 937.3 | 815.5 |
| R_L(Ω·cm²) | 184.4 | 513.7 | 449.6 |
Ydl value mean that the sample surface is more susceptible to be attacked by anions such as OH\(^-\) and Cl\(^-\). The increasing L and Rs values from P-1, P-3 to P-2 indicate a decrease in the corrosion product of the activated adsorption after the relaxation reaction.

Table 4 details the impedance spectrum fitting parameters of the B-1, B-2, and B-3 aluminum alloy panels. Comparing with table 2, it can be seen that the evolution of corrosion behavior of bake-hardened state is similar to that of pre-aging state, decreasing in the order of B-2 > B-3 > B-1. The corrosion performance of the bake-hardened state is obviously stronger than that of the pre-aging ones with corresponding parameters, namely B-1 > P-1, B-2 > P-2 and B-3 > P-3.

In summary, the change trend of OCP, polarization curve and EIS demonstrate that the corrosion resistance of bake-hardening aluminum alloy panel is better than that of pre-aging condition under corresponding test parameters.

The corrosion resistance of P-1 is the worst; in the pre-aged and bake-hardened panels, the panel samples with different grain sizes exhibited comparable corrosion resistance, namely P-2 ≈ P-3 and B-2 ≈ B-3.

### 3.4. Precipitation of the second phase and its effect on corrosion resistance

Figures 5(a), (b) shows the bright-field TEM images of microstructure of the alloy before and after bake hardening treatment, respectively. The grain boundary of the P-1 aluminum alloy panel is composed of continuous precipitates, while the precipitates in the B-1 alloy panel grow discontinuously. In fact, the coarse grain boundary precipitates (GBP) is produced after bake hardening, and a grain boundary precipitate-free zone (PFZ) with a width of 73 nm is appeared in the B-1 aluminum alloy panel. Such a B-1 alloy panel can get improved corrosion resistance because of the more discreet and coarser GBP and wider PFZ. However, this phenomenon is not occurred in the P-1 aluminum alloy panel.

Here, in combination with the reduction of the precipitates number density and the formation of discontinuous precipitates in the grain boundary after bake hardening, it can be considered that the improved corrosion resistance after bake hardening (B-1 > P-1, B-2 > P-2 and B-3 > P-3) have two main reasons: 1) the decrease of the number density reduces the occurrence site of pitting; 2) the discontinuous precipitation of the grain boundary reduces the potential difference with the matrix, thereby improving the corrosion resistance.

On the other hand, from the polarization curve and impedance analysis, the corrosion resistance of P-2 and P-3, and B-2 and B-3 aluminum alloy panels with different grain sizes changed little respectively because the anions such as Cl\(^-\) in the electrolyte do not preferentially attack the grain boundaries [17].

The corrosion behavior of 7000 series aluminum alloy relies on the formation of precipitates [18]. Figures 5(a), (b) shows the TEM graphs of precipitates at grain boundaries and inner grain. The shape of GP region, \(\eta\) phase and their orientation relationship with matrix determines the corrosion behavior [19–21]. HREM graphs and Fourier transform diagram in <110> \(_\text{Al}\) direction are showed in figures 5 (c)–(h). GP zones after PA treatment is the main precipitated phase type in the matrix, and the Fourier transform diagram shows its coherent with the Al matrix in figure 5(f). The \(\eta\) phase is precipitated in the Al substrate after BH treatments, while the Fourier transform diagram shows its semi-coherent with the Al matrix in figures 5(g)–(h) [22, 23]. In particular, the appearance of these metastable hardening \(\eta\) phase will accordingly reduce the density of matrix precipitation [24, 25], thus reducing the pitting side and pitting surface, and finally promoting the inhibition of subsequent intergranular corrosion, so as to improve the corrosion resistance.

In order to much accurately and comprehensively detect the corrosion morphology and corrosion product type after potentiodynamic polarization, the bake hardening aluminum alloy panel is used to clarify the specific corrosion process. The typical morphology and corrosion products are characterized using SEM, and the 3D-
MLM optical microscope. The corrosion morphology is shown in figure 6. Usually, pitting corrosion is the main feature of electrochemical corrosion of aluminum alloy. It can be observed that the pitting pit is distributed over the entire corrosion interface. The corrosion products near the grain boundary on the substrate and the edge of the pit are similar to those in the matrix. It can be seen that small corrosion products are distributed inside the corrosion pit, indicating that chemical corrosion begins with a site that is easily corroded and then expands until it forms a large etch pit that eventually extends to the entire interface. It can be seen from the 3D-MLM surface images that the B-1 state presents the most corrosion pits, and the maximum corrosion depth is about 38.917 μm, while the B-2 state and B-3 state is about 27.806 μm and 33.916 μm, respectively.

Figure 5. TEM and HRTEM micrographs obtained along the [110]Al zone axis. (a) Pre-aging state (P-1); (b) bake hardening state (B-1); (c) and (d) GP zone and IFFT of P-1; (e) ~ (g) η' phase and IFFT of B-1.
Figure 6. Corrosion morphology of SEM (EDS) and 3D-MLM images for the alloy pane at different bake hardening state (a) B-1; (b) B-2; (c) B-3.
The corrosion products on the exposed surface are mainly $\text{Al}_2\text{O}_3$. The main corrosion reactions are as follows [26–29]:

\[
\begin{align*}
\text{Anodereaction} & : \quad \text{Al} \rightarrow \text{Al}^{3+} + 3e^- \\
\text{Cathodicreaction} & : \quad 2\text{H}_2\text{O} + \text{O}_2 + 4e^- \rightarrow 4\text{OH}^- \\
& \quad \text{Al}^{3+} + 3\text{OH}^- \rightarrow \text{Al(OH)}_3 \rightarrow \text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}
\end{align*}
\]

Among them, the cathodic chemical reaction of the formula (2) is generally present in a NaCl soaking solution which has not been subjected to oxygen scavenging treatment.

In 3.5% NaCl solution, due to the existence of incomplete oxide film on the sample surface, the free $\text{Cl}^-$ in the solution will preferentially attack the aluminum matrix lacking the protection of the oxide film to form aluminum chloride and oxychloride, etc, and the chemical equation for the aluminum matrix to participate in the reaction is as follows:

\[
\begin{align*}
\text{Al}^{3+} + 4\text{Cl}^- & \rightarrow \text{AlCl}_4^- \\
\text{Al}^{3+} + 2\text{Cl}^- + 2\text{OH}^- & \rightarrow \text{Al(OH)}_2\text{Cl}_2
\end{align*}
\]

The corrosion ion passes through the oxide film, first of all, it forms local soluble compounds or intermediates with some components in the oxide film at active point with lower equal potential (such as precipitation phase). When the oxide film on the aluminum surface is thinned to a certain value, the alloy matrix will be destroyed rapidly, resulting in the rapid development of pitting corrosion.

The main causes of corrosion are as follows:

1) the quantity change of precipitates which causes the corrosion potential difference between the anode material (precipitation phase) and the substrate, and the propagation direction of intergranular corrosion is determined by the orientation of habit plane of precipitates.

2) Intergranular corrosion occurs due to the different breakdown potential between PFZ zone and matrix;

3) The dissolution of continuously distributed precipitates at grain boundaries can form a strongly erosive occluded zone ring which could form continuous grain boundary corrosion.

4. Conclusions

In summary, the corrosion resistance of experimental aluminum alloy after bake hardening is appeared to be higher than that of pre-aging treatment, since the discontinuous precipitation at the grain boundaries led to decrease the potential difference between the matrix and the grain boundary precipitates, which strongly enhanced the corrosion resistance; meanwhile, the appearance of metastable hardening $\eta'$ phases reduces the density of matrix precipitation, thus reducing the pitting side and pitting surface, and finally promoting the inhibition of subsequent intergranular corrosion, so as to improve the corrosion resistance.

Data availability statement

No new data were created or analysed in this study.

Conflict of interest

The authors declare that they have no conflict of interest.

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