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Effect on microstructure and corrosion resistance of semi-solid slurry of 7A04 aluminum alloy by electromagnetic stirring

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Abstract

The microstructures and corrosion resistance of electromagnetic stirring (EMS) semi-solid slurry and as-cast 7A04 aluminum alloy were compared in this work. The results show that the primary microstructure of the as-cast 7A04 aluminum alloy is mainly dendritic and columnar dendrite with obvious elements segregation. In contrast, semi-solid processing can effectively form homogeneous and round primary $\alpha$-Al grains, and significantly reducing element segregation. In addition, the main phases of the two forming methods (as-cast and semi-solid) both involved $\alpha$-Al, $\eta$-MgZn$_2$, $\theta$-Al$_5$Cu. With the decrease of solidification temperature, the semi-solid alloy formed by electromagnetic stirring increases the precipitation of brittle phase $\beta$-Al$_5$CuMg. The self-corrosion potential of the semi-solid alloy is greater than that of the as-cast alloy, and the polarization curve has obvious passivation characteristics. Semi-solid processing could improve corrosion resistance of 7A04 aluminum alloy obviously.

1. Introduction

High strength Al–Zn–Mg–Cu system aluminum alloys (type 7A04) are widely used in aerospace and rail transit due to the advantages of light quality, big ratio intension, good plasticity and recyclability [1–6]. In traditional casting, the microstructure of aluminum alloys show obviously dendrite with irregular coarse grain because of the wide solidification temperature range [7, 8]. As a result, segregation, shrinkage and hot crack defects are difficult to avoid in aluminum alloy [9–11], which may induce the weak of corrosion resistance [12–15]. Therefore, it is easy to form a weak area of corrosion and fracture. With the increasing demand of aerospace industry for aluminum alloy, the development of forming process to optimize the microstructure and corrosion properties of 7xxx series alloys has been widely concerned.

Semi-solid forming technology [16–18] is appropriate for low-cost near-net forming. The alloy was strongly stirred during solidification in the semisolid temperature range (between solidus and liquidus). The primary dendrites were fully crushed and obtained certain spherical primary solid phase. In addition, semi-solid forming technology can also effectively reduce segregation. Ordinary electromagnetic stirring is regarded as a good method for the rheological forming of semi-solid slurry due to non-contact, easy control, no pollution and simple operation [19–21]. It can inhibit the growth of dendrite arm and homogenize the composition and temperature distribution so as to obtain uniform small spherical primary particles. In our previous work, a large-volume semi-solid slurry of 7A04 aluminum alloy with primary $\alpha$-Al particles of average equivalent diameter of 73.5 $\mu$m and shape factor of 0.57 was prepared with a pouring temperature of 650 $^\circ$C combined with nominal stirring power of 0.6 kW kg$^{-1}$ for 40 s [22]. The microstructure and phase about as-cast 7xxx series aluminum alloy have been widely studied [23–25]. Some study has done on microstructure of semi-solid 7xxx series aluminum alloy [26, 27], but rarely on comparison between semi-solid and as-cast 7xxx series aluminum alloy slurry on major phases and corrosion behavior.
In this work, the microstructure, element segregation and corrosion resistance of as-cast and semi-solid 7A04 aluminum alloy slurry were comparatively studied by optical microscope (OM), X-ray diffraction (XRD), energy dispersive x-ray (EDX), polarization curves and electrochemical impedance spectroscopie (EIS). The ultimate purpose is to obtain 7A04 aluminum alloy with semi-solid microstructure and higher corrosion resistance with temperature-controllable electromagnetic stirrer.

2. Material and methods

7A04 aluminum alloy was used in this study and the chemical composition is shown in table 1. Semi-solid slurry of 7A04 aluminum alloy was prepared by electromagnetic stirring (EMS) with temperature-controllable electromagnetic stirrer. Primarily, the alloy was heated to 700 °C until fully melted, then stop heating but continue stirring. Furthermore, the samples were gathered at selected temperatures under the optimal process parameter combination (EMS voltage of 230 V, EMS frequency of 5 Hz) and then were quickly quenched in cold water. According to the differential scanning calorimetry (DSC) curve (figure 1), the solidus and liquidus temperatures were 478 °C and 652 °C, respectively. Because the solid phase ratio of the alloy is sensitive to temperature change, and the characteristics of electromagnetic stirring are considered, the temperature of slurry preparation is set in the range of 635 °C–645 °C. In contrast, samples of as-cast 7A04 aluminum alloy were prepared by water quenching without stirring under the same conditions.

The microstructures were characterized using optical microscope (OM), X-ray diffractometer (XRD) and energy dispersive x-ray (EDX). Samples for metallographic OM characterization were prepared according to the standard procedure and etched in mixed acid solution (1 vol% HF, 1.5 vol% HCl, 2.5 vol% HNO₃ and 95 vol% H₂O). Metallographs were carried out using a laser scanning confocal microscope (VHX-1000). Image Pro Plus software was used to estimate the average equivalent diameter \( D \) and average shape factor \( F \) of primary \( \alpha \)-Al grains to characterize the slurry quality under different experimental conditions. \( D \) and \( F \) were determined as follow [28]:

\[
D = \left(\frac{4A}{\pi}\right)^{1/2}
\]

\[
F = \frac{4\pi A}{P^2}
\]

Where \( A \) represents the average area of a primary \( \alpha \)-Al grain in \( \mu m^2 \), \( P \) represents the average circumference of a primary \( \alpha \)-Al grain in \( \mu m \), respectively.

The phases in the alloy were characterized by XRD using Cr- \( K\alpha \) target with a scan rate of 2° min⁻¹ on a XXQ2505D-XK3.2 x-ray diffractometer. The element segregation were characterized on HITACHI JSM-
6490LV scanning electron microscope (SEM) equipped with model GENESS 2000XMS energy dispersive x-ray (EDX) spectroscope.

In order to comparatively study the corrosion resistance, dynamic polarization and electrochemical impedance spectroscope tests in 3.5 wt% NaCl solution were performed on a CS310 electrochemical workstation. The scanning rate during dynamic polarization was set as 1 mV s\(^{-1}\) with the potential range of \(-0.5\) to 0.5 V relative to open circuit potential. EIS tests were carried out with frequencies from 100 kHz to 0.01 Hz. Ten points per tenfold frequency was chosen, and 5 mV perturbation amplitude was set.

3. Results and discussion

3.1. Influence of different molding methods on microstructures

Figure 2 shows the metallographic images of the as-cast and semi-solid samples. Primary solid phase and liquid phase can be observed in both samples. The liquid phases can be observed at the grain boundaries which mainly refers to a small amount of eutectic phase dissolved and distributed near the grain boundaries [10]. As shown in

![Figure 2. Metallographic images of (a) as-cast and (b) semi-solid 7A04 aluminum alloy.](image)

![Figure 3. XRD patterns for 7A04 aluminum alloy of as-cast and semi-solid sample.](image)
The dendrite size is significantly larger than that of semi-solid round grain [29]. In comparison, as shown in figure 2(b), the semi-solid sample is mainly composed of spherical or nearly spherical primary $\alpha$-Al grains, $\alpha_2$-Al and liquid phases. The primary $\alpha$-Al grains are round and evenly distributed.

Figure 3 shows the XRD pattern of as-cast and semi-solid 7A04 aluminum alloy. They both consist of three main phases, i.e. $\alpha$-Al, $\eta$-MgZn$_2$ and $\theta$-Al$_2$Cu. Figures 4(a) and 5(c) show the element distribution of as-cast and semi-solid primary grain inner. The main elements of as-cast grain inner are Al, Zn, Mg and Cu, with content of 96.78 at%, 0.91 at%, 1.27 at% and 0.13 at%, respectively. Combined with XRD pattern, the main phase are
matrix phase $\alpha$-Al, the strengthened phase $\eta$-MgZn$_2$ and the intermediate phase $\theta$-Al$_2$Cu, in an irregular continuous network distribution. In contrast, the main elements of semi-solid grain inner are Al, Zn, Mg, with content of 95.26 at%, 2.14 at% and 2.60 at%, respectively, without Cu element. Combined with XRD pattern, the main phase are $\alpha$-Al and $\eta$-MgZn$_2$. Figures 5(b) and (d) show the element distribution of as-cast and semi-solid primary grain boundary. The main elements of as-cast grain boundary are Al, Zn, Mg and Cu, with content of 88.10 at%, 1.84 at%, 1.96 at% and 6.69 at%, respectively. Likewise, the main elements of semi-solid grain inner are also Al, Zn, Mg and Cu, with content of 79.76 at%, 2.04 at%, 2.73 at% and 15.47 at%, respectively. Combined with XRD pattern, the main phases at primary boundary of as-cast and semi-solid are both $\alpha$-Al, $\eta$-MgZn$_2$ and $\theta$-Al$_2$Cu. The results show that there was no significant difference in main phase species and distribution of as-cast and semi-solid alloy. Therefore, the main reasons for the optimization performance of semi-solid technology may be morphology change and element homogenization rather than phase species change.

3.2. Effect of different solidification temperature on microstructures

Semi-solid rheological slurry samples are prepared by EMS at voltage of 230 V and frequency of 5 Hz at a near liquidus temperature of 645 °C, at 640 °C and 637 °C in semi-solid temperature range. The microstructure is shown in figure 5, and average equivalent diameter ($D$) and average shape factor ($F$) of the alloy is quantitatively analyzed at a given temperature and shown in figure 6. The sample electromagnetic stirred at 645 °C, which was slightly lower than the liquidus temperature, exhibits dendritic or columnar dendritic primary $\alpha$-Al without semi-solid round grain appeared. The average equivalent diameter of primary $\alpha$-Al is 50 $\mu$m but the average shape factor was only 0.14. The sample electromagnetic stirred at 640 °C, which was in the range of semi-solid forming temperature, exhibits microstructure of primary $\alpha$-Al appears obviously round grains with average equivalent diameter of 63 $\mu$m and average shape factor of 0.27. When the EMS temperature decreased to 637 °C, the melt solid ratio increased. Microstructure of primary $\alpha$-Al still presents round grains, but the average equivalent diameter increased to 75 $\mu$m. Average shape factor decreased slightly to 0.23, and the primary $\alpha$-Al grain roundness decreased.

In conclusion, the overall quality of the water-quenched tissue is the best at 637 °C. This is probably because when the temperature of the alloy drops from above the liquidus to 641 °C, the dendrite arm is broken under the effect of electromagnetic stirring, and only a small part of the grains grow into semi-solid grains with relatively large size. When the melt temperature continues to drop to 638 °C, most of the branching crystals are broken up and grow into larger semi-solid grains with a large increase. When the temperature further decreases, the primary $\alpha$-Al grain in the appropriate orientation will 'weld' at the contact point in the collision and gradually gather into a group, forming a 'large structure' with obvious agglomeration and resulting in an increase in grain size, which is consistent with the analysis in literature [30].
Figure 7 shows the main phases of semi-solid 7A04 aluminum alloy by electromagnetic stirring quenched at 645 °C, 640 °C and 637 °C. The main phases at 645 °C and 640 °C are α-Al, η-MgZn2 and θ-Al2Cu. When the temperature decreases to 640 °C, besides α-Al, η-MgZn2 and θ-Al2Cu, brittle phase S-Al2CuMg precipitates in grain boundary in a continuous network structure [25]. Figures 8(a), (c) and (e) show the element distribution of semi-solid primary grain inner at different temperature. The main elements at 645 °C and 637 °C are Al, Zn, Mg and Cu. The contents at 645 °C are 79.76 at%, 2.04 at%, 2.73 at% and 15.47 at%, respectively. Meanwhile, the contents at 645 °C are 79.76 at%, 2.04 at%, 2.73 at% and 15.47 at%, respectively. Combined with XRD pattern, the main phases are α-Al, η-MgZn2 and θ-Al2Cu at 645 °C and 637 °C. In contrast, the main elements at 640 °C are Al, Zn, Mg, with content of 96.80 at%, 1.73 at% and 1.47 at% respectively. Combined with XRD pattern, the main phases are α-Al and η-MgZn2. Figures 8(b), (d) and (f) show the element distribution of semi-solid primary grain boundary at different temperature. The main elements at 645 °C, 640 °C and 637 °C are Al, Zn, Mg and Cu. The main content at 645 °C are 88.10 at%, 1.84 at%, 1.96 at% and 6.69 at% respectively. The main content at 640 °C are 80.95 at%, 6.34 at%, 2.38 at% and 3.90 at%, respectively. Combined with XRD pattern, the main phases are α-Al, η-MgZn2, θ-Al2Cu and brittle phase S-Al2CuMg. This is probably because that the solid phase ratio is too high at 637 °C to homogenize alloy by EMS.

Figure 8. Energy-dispersive x-ray (EDX) analysis of primary grains of 7A04 alloy by Electromagnetic Stirring at 645 °C of (a) grain inner and (b) grain boundary, 640 °C of (c) grain inner and (d) grain boundary, and 645 °C of (e) grain inner and (f) grain boundary.
The results show that the samples at 645 °C and 640 °C are mainly composed of matrix phase $\alpha$-Al, strengthening phase $\eta$-MgZn$_2$ and aging strengthening phase $\theta$-Al$_2$Cu. However, 645 °C is near the liquidus, so the microstructure is dendritic. With further decreasing of temperature, besides $\eta$-MgZn$_2$ and $\theta$-Al$_2$Cu, brittle phase S-Al$_2$CuMg precipitates at 637 °C. Taken together, 640 °C in semisolid temperature range is better than...
645 °C and 637 °C to form better microstructure and phase. Therefore, proper temperature control is beneficial to round primary grain formation, alloy homogenization and brittle phase inhibition.

### 3.3. Influence of different molding methods on corrosion behavior

Figure 9 shows the polarization curves of the as-cast and semi-solid 7A04 aluminum alloy in 3.5 wt% NaCl solution measured by the three-electrode system. The electrochemical parameters of corrosion are listed in Table 2. The self-corrosion potential of the semi-solid sample is higher than that of the as-cast sample, and its polarization curve has obvious passivation characteristics. The corrosion current density, corrosion rate and $E_p$ (pitting potential) of the semi-solid sample are both lower than that of the as-cast sample, meanwhile $R_p$ (polarization resistance) and $|E_p - E_{corr}|$ are higher. All of this indicates that the semi-solid sample has higher corrosion resistance than that of the as-cast sample.

Figure 10 shows the electrochemical impedance spectrum (EIS) of the as-cast and semi-solid 7A04 aluminum alloy. The Bode curve is presented in Figure 10(a). The electrochemical impedance spectrum (EIS) of the as-cast 7A04 aluminum alloy consists of a high and medium frequency capacitive reactance arc and a medium and low frequency inductive reactance arc. The impedance spectrum of the semi-solid 7A04 aluminum alloy shows two capacitive reactance arcs, located in the high and low frequency regions respectively, and an inductive reactance arc. The high frequency region reflects the information between the original oxide film on the alloy surface and the corrosion solution which the alloy contacts with. While the low frequency region reflects the electrochemical action between the alloy and Cl$^-$ infiltrating into the fresh bare matrix metal surface after the passivation film is destroyed. The inductive reactance arcs of as-cast and semi-solid alloy are often considered to be caused by the weakened protection of the passivation film on the metal matrix during the pitting induction period [31, 32].

### Table 2. Parameters of polarization curves of 7A04 aluminum alloys in 3.5 wt% NaCl solution.

| State     | $E_{corr}$ (V) | $I_{corr}$ (A.cm$^{-2}$) | Corrosion rate (mm/a) | $E_p$ (V)   | $R_p$ (Ω.cm$^{-2}$) | $E_p - E_{corr}$ (V) |
|-----------|----------------|--------------------------|-----------------------|-------------|---------------------|----------------------|
| As-cast   | $-0.83$        | $8.592 \times 10^{-3}$   | 0.938                 | $-0.81$     | 303.61              | 0.02                 |
| Semi-solid| $-0.75$        | $3.094 \times 10^{-3}$   | 0.338                 | $-0.69$     | 842.96              | 0.06                 |

Figure 11. Energy-dispersive x-ray (EDX) line scanning of (a) as-cast sample and (b) semi-solid sample.
Table 3. Fitting results of EIS equivalent circuits of as-cast and semi-solid 7A04 aluminum alloy.

| State            | $R_{\text{en}}/(\Omega \cdot \text{cm}^2)$ | $R_{\text{ao}}/(\Omega \cdot \text{cm}^2)$ | $Y/(\Omega \cdot \text{cm}^{-2} \cdot \text{S}^{-1})$ | $n$ | $R_{\text{p}}/(\Omega \cdot \text{cm}^2)$ | $L/(\text{H} \cdot \text{cm}^2)$ | $R_{\text{ct}}/(\Omega \cdot \text{cm}^2)$ |
|------------------|------------------------------------------|------------------------------------------|------------------------------------------|-----|------------------------------------------|--------------------------------|------------------------------------------|
| As-cast          | 104.6                                    | 1920                                     | $1.266 \times 10^{-4}$                    | 0.8684 | —                                       | 2.518 $\times 10^{-4}$ | 4740                                      |
| Semi-solid       | $1 \times 10^{-7}$                       | 109.2                                    | —                                        | — | 3140                                    | $1.55 \times 10^{-3}$ | 5401                                      |

Comparing the impedance spectra of semi-solid and as-cast 7A04 aluminum alloy, it can be found that the semi-solid alloy has a large capacitive reactance arc. On the one hand, the semi-solid process changed the microstructure of 7A04 aluminum alloy to form compact, round and semi-solid alloy has extra capacitive reactance arc. On the other hand, the scanning results show that the as-cast $\alpha$-Al grain is enriched with higher Cu at the grain boundary. While the distribution of elements in the semi-solid $\alpha$-Al grain is relatively uniform, which also indicates that the semi-solid process can significantly reduce element segregation and make the distribution of elements more uniform due to electromagnetic stirring. The second phases of 7A04 aluminum alloy mainly contain Al$_7$Cu$_2$Fe, (AlCu)$_6$(FeCu) and MgZn$_2$. Among them, the potential of the Cu-rich second phases are more positive compared with $\alpha$ aluminum matrix, so the Cu-rich second phases is as cathode in the local corrosion. Semi-solid process significantly promotes the Cu element evenly distributed. Also it decreases the ratio of Cu of grain boundary and inner smaller than as-cast alloy, which reduces the cathodic and anodic area ratio of the galvanic corrosion. So it is easier to form a dense barrier layer on the surface of semi-solid alloy, effectively preventing Cl$^-$ from penetrating into the interior of the matrix metal. The manifestation on the curve is an extra capacitive resistance arc of semi-solid alloy.

Figure 10(b) is the Nyquist curve of the as-cast and semi-solid 7A04 aluminum alloy. The impedance value and phase angle of the as-cast and semi-solid aluminum alloys in the high-frequency region are not much different. It indicates that the corrosion performances at the beginning has little difference. In the low frequency range, the impedance modulus of the as-cast alloy is significantly lower than that of the semi-solid alloy. It probably indicates that the corrosion depth of the as-cast alloy is likely to be higher than that of the semi-solid alloy. This may be due to the large and irregular dendrite of the as-cast sample. The grain boundaries between the coarse dendrites are twisted and crossed, meanwhile the stress concentration is higher. So it becomes the weak areas of corrosion, and the corrosion pits are easily extended in the depth direction. The semi-solid alloy has obvious phase angle peaks in the low frequency range. It indicates that the matrix of the semi-solid alloy is corroded obviously. This may be because the semi-solid alloy has a large number of rounded grains whose overall size is smaller than the as-cast dendrites. Under the same corrosion time, the number of small semi-solid round grains falling off is more than as-cast alloy. In summary, the surface of the as-cast alloy has less grain separation, but the corrosion depth is higher. Meanwhile small grains on the surface of the semi-solid alloy matrix are more than as-cast alloy, but the corrosion depth is relatively shallow. The electrochemical analysis results are consistent with the three-dimensional morphology of literature.

ZSimpwin software was used to fit the equivalent circuit model of electrochemical impedance spectrum, as shown in figure 12. As can be seen from the impedance diagram, there are two time constants in the as-cast aluminum alloy. The equivalent circuit model $R(Q(\text{RL}))$ is shown in figure 12(a), where the solution resistance is $R_s$, the constant phase angle of the outer layer is $Q$, the oxidation film (outer layer) resistance is $R_{\text{ao}}$. 

![Figure 12. EIS equivalent circuits of (a) as-cast and (b) semi-solid 7A04 aluminum alloy.](image-url)
the capacitance of $Q$ is $Y$, the dispersion effect index is $n$, the inductance is $L$, and $R_t$ is the charge transfer resistance. According to the impedance diagram, it can be seen that there are three time constants of the semi-solid aluminum alloy. The equivalent circuit model $R(C(R(CR)))CLR$ is shown in figure 12(b). In addition to the above introduced parameters, there are also external layer capacitor $C_1$, double layer capacitor $C_3$, barrier layer capacitor $C_2$ and barrier layer resistor $R_b$. Table 3 shows the parameter comparison of fitting results between as-cast and semi-solid 7A04 aluminum alloy. It can be seen from table 3 that the oxidation film resistance $R_o$ of as-cast aluminum alloy is higher than that of semi-solid aluminum alloy. This may be caused by the formation of a compact barrier layer on the stable and flat matrix surface of semi-solid aluminum alloy which thins the oxide film due to the absorption of most components. The time constant of semi-solid aluminum alloy is more than that of as-cast aluminum alloy, which may be due to the barrier layer formed between the oxide film and the matrix of semi-solid aluminum alloy. The double layer resistance $R_t$ of semi-solid aluminum alloy is higher than that of as-cast aluminum alloy. This may be due to the relatively compact structure formed by the fine round semi-solid primary grain. Meanwhile, the homogeneity of the composition reduces the anode and cathode potential difference of the matrix. These can prevent the corrosion from deepening.

The above analysis indicates that compared with the dendrite microstructure in as-cast alloy, the distribution of elements in semi-solid near-spherical microstructure tends to be obviously homogeneous. The semi-solid forming technology can greatly reduce the element segregation, which can significantly improve the corrosion resistance of the alloy.

4. Conclusions

(1) The primary microstructure of the as-cast 7A04 aluminum alloy is mainly dendritic and columnar dendrite. The main phases are $\alpha$-Al, $\eta$-MgZn$_2$, $\theta$-Al$_2$Cu. The semi-solid 7A04 aluminum alloy shows homogeneous and round primary $\alpha$-Al grains. The main phases are $\alpha$-Al, $\eta$-MgZn$_2$, $\theta$-Al$_2$Cu, which are the same as the as-cast sample.

(2) With the decrease of water quenching temperature, the primary grain of 7A04 aluminum alloy changes from dendrite to round grain, and then the size of round grain increased again. The main precipitated phases start with $\alpha$-Al, $\eta$-MgZn$_2$, $\theta$-Al$_2$Cu, and then increased the brittle phase Al$_2$CuMg at 637 °C.

(3) The polarization curve of semi-solid 7A04 aluminum alloy has obvious passivation characteristics. And its impedance spectrum has a extra capacitive reactance arc compared with as-cast alloy. The results of polarization curve and electrochemical impedance spectroscopy show that the corrosion resistance of the semi-solid sample is better than that of the as-cast sample.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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