

Short Communication

Effect of Temperature Treatment on Microstructure and Electrochemical Properties of 6061 Aluminum Alloy

Zhisheng Wang, Runhua Fan, KunChen, QingHong Li, Yansheng Yin

College of Ocean Science and Engineering, ShanghaiMaritime University, 1550 Hai gang Avenue, Shang hai201306, China

*E-mail: zhishengwang1985@126.com ; rhfan@shmtu.edu.cn

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The corrosion behavior of 6061 aluminum alloy under heat treatment at different temperatures and oxidation states was studied. The anode film was prepared by acidizing 6061 aluminum alloy as matrix. The effect of oxidation treatment on the microstructure and corrosion resistance of 6061 aluminum alloy was studied by SEM, XRD, and EDS. The results showed that the corrosion of 6061 aluminum alloy first decreased and then showed a general trend with the increase of temperature. Comparative analysis of the samples treated under different temperatures, in solution with pH=2 revealed that the corrosion resistance of the samples weakened with the increase of temperature, which was the weakest at temperature around 550°C. With the increase of temperature, the corrosion rate of 6061 aluminum alloy showed an increasing trend. The results showed that appropriate temperature treatment is beneficial to improve the corrosion resistance of 6061 aluminum alloy, and the corrosion sensitivity of 6061 aluminum alloy is: oxidation +550°C heat treatment > oxidation +350°C heat treatment.

Keywords: Heat treatment condition, corrosion morphology, surface scanning, line-surface scanning, 6061 aluminum alloy

1. INTRODUCTION

Aluminum and aluminum alloys are widely used in transportation, aerospace, and other industries because of their excellent properties such as low density, good welding performance, high plasticity, and corrosion resistance [1–3]. Aluminum 6061 alloy has good process plasticity, corrosion resistance, and strong corrosion resistance; therefore, it is widely used in modern transportation industry and other fields [4,5]. Heat treatment technology is a successful process applied extensively in treatment of aluminum surface; and this technology has witnessed a lot of advances and attracted significant research attention [6–8]. However, the aluminum alloy under some specific heat treatment conditions can still corrode with appearance of cracks [10,11]. This study intended to explore the

corrosion sensitivity of 6061 aluminum alloy by comparing the corrosion behavior of samples under different heat treatment conditions. Aluminum alloy has several excellent properties, such as low density and high strength-to-weight ratio; thus, it has been applied to various economic fields and many spheres of human life. In order to meet the needs of social development, aluminum surface treatment technology and corrosion resistance need to be further improved [3–5]. Aluminum is a metal material with the largest usage and the most extensive application area among non-ferrous metals, and its application scope is still expanding rapidly [12,13]. Many researchers have extensively studied and improved the surface film properties of aluminum alloy, leading to its widespread application prospects in various fields [3–9]. When the fresh surface of metallic aluminum is exposed to the atmosphere, it is immediately covered with a very thin oxide film, which rapidly regenerates once damaged [14]. Appropriate heat treatment is beneficial to improving the physical and chemical properties of aluminum alloy and improving the service life of aluminum products, which are widely used in various fields of life and industry [15–17].

2. EXPERIMENTAL

2.1. Sample preparation

After homogenizing at 550°C for 4 h, and maintaining the temperature at 400° for 2 h, the sample was annealed for 2 h at 300 °C. The main material used in the experiment was 6061 aluminum alloy. The components in the supply state of 6061 aluminum alloy are listed in Table 1. The samples with dimension of 10 mm × 10 mm × 3 mm were cut. The specimens were mechanically ground using SiC papers with grit sizes of 240, 400, 800, and 1200 and then degreased in acetone and ethanol, and finally washed with pure water. After the cleaning and rusting process, the samples were dried for 24 h, and the solution was placed at room temperature under humid environment for some time. Solution of Na₂SO₄(0.5 molL⁻¹) was employed as the corrosive medium. The pH of the medium was adjusted to 2 by dropwise addition of H₂SO₄ to the Na₂SO₄ solution at 22°C (humidity was 50% RH) for 10 days.

Table 1. Chemical compositions of 6061 Al alloys (wt.%)

Material	Cu	Mn	Mg	Zn	Cr	Ti	Si	Fe	Zr	Al
wt.%	0.20	0.11	0.99	0.008	0.21	0.015	0.51	0.32	0.05	allowance

2.2 Oxidation treatment

The sample was placed in a muffle furnace (SX-2.5-10) for heat treatment, and the temperature was set at 350 and 550°C, respectively. Briefly, the anodizing treatment process with sulfuric acid was carried out under the following conditions: sulfuric acid (20%), aluminum ion (20 g L⁻¹), current

density= 2Adm^{-2} , concentrated sulfuric acid, oxidation voltage 18V and 22V, oxidation temperature 24°C , and oxidation time 60min. Further, the 6061 aluminum alloy samples were processed into plate samples. The bare 6061 aluminum alloy was labeled as sample 1, and the 6061 aluminum alloy anodized with sulfuric acid was labeled as sample 2. After cleaning, grinding, polishing, and drying the flat samples, five parallel samples in each group of samples were obtained.

2.2 Characterization test

Elemental composition of micro regions of samples was analyzed using an INCA energy spectrometer. The phase composition was analyzed by X-ray diffraction(XRD).The morphology of the surface and cross section was characterized by scanning electron microscopy(SEM, Jsm-7500F), and composition of the products was analyzed by energy-dispersive X-ray spectroscopy (EDS). The tube pressure and tube flow were 40kV and 100mA, respectively. The scanning range was 20° and the scanning rate was $10^{\circ}\text{min}^{-1}$.The electrochemical properties of the as-obtained samples were characterized on an electrochemical workstation (Gamry, 1010E), using a three-electrode system (the auxiliary electrode was a platinum wire, the reference electrode was calomel saturated electrode, and the working electrode was the 6061ALalloy sample). Before testing, the working electrode was cleaned and polished. The excitation signal used for electrochemical impedance spectroscopy (EIS) was 10 mV, while the frequency range was 10^4 – 10^{-2}Hz , which was measured at the self-etching potential by using Zview software to analyze the results.

3. RESULTS AND DISCUSSION

3.1 Corrosion morphology observation

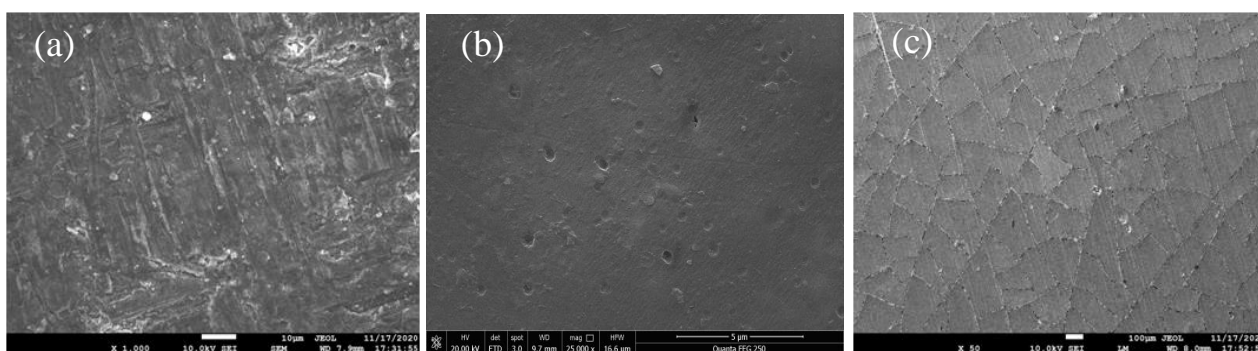


Figure 1. Microstructure of sample surface (a: non-oxidized and 550°C , 4h heat treatment, b: Oxidation treatment at 22 V and 350°C , c: Oxidation treatment at 22 V and 550°C , 4h heat treatment)

Figure 1 shows the appearance morphology of 6061 aluminum alloy under different heat treatment conditions. Figure 1(a) exhibits the surface morphology of sample subjected to heat treatment at 350°C , showing the existence of obvious dendrite segregation in the alloy, and the

precipitation phase on the grain boundary is continuously thick. Figure 1(b) shows the appearance morphology of sample treated at 550°C. Clearly, the precipitated phase at grain boundary gets back dissolved into the matrix, the dendrite segregation is basically eliminated, and the grain boundary becomes thinner. Figure 1(c) shows the appearance morphology of sample treated at 550°C, with grains extending into fibrous structure, phase transition at grain boundary becomes discontinuous and finer, and the precipitated phase at grain boundary becomes more discrete and finer.

3.2 Effect of oxidation treatment on phase composition of oxide film

Figure 2(a) shows XRD pattern of non-oxidized samples treated at 550°C, and Figure 2(b) exhibits XRD pattern of oxidized samples treated at 550°C. Notably, the oxide film before treatment is mainly composed of R-Al₂O₃ phase. Figure 3 displays energy spectrum images of the surface morphology and structure of the section of the treated 6061 aluminum alloy, exhibiting obvious irregularity in the distribution. Analysis of the precipitate in 6061 aluminum alloy indicated the excessive precipitation of Al-Fe-Si phase in the aluminum alloy, as shown in Figure 2. After oxidation treatment, the oxidation film was still dominated by R-Al₂O₃ phase [10–12].

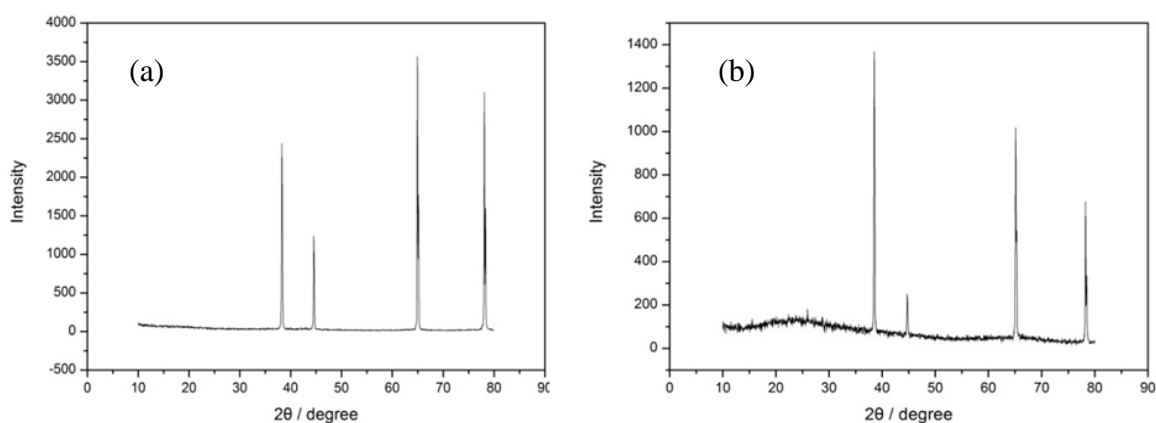


Figure 2. XRD patterns of ceramic coatings under oxidation treatment (a: Non-oxidized and 550°C, 4h heat treatment, b: Oxidation treatment at 22 V and 550°C, 4h heat treatment)

3.3 Effect of oxidation treatment on the cross section structure of 6061 aluminum alloy

Figure 3(a) demonstrates that the thickness of grain boundary phase is of sub-micron level after oxidation treatment at 18V and 24°C for 1h followed by heat treatment at 350°C for 4h. At the same time, precipitation of a large number of fine second phase compounds was observed in the grains. The precipitation of compounds is the most important structural change characteristic of 6061 aluminum alloy in the oxidation process, and has a significant influence on its performance. On the one hand, the precipitation of the second phase of these compounds resulted in the decrease of alloying element content; however, numerous nano particles were also formed in the grain after the oxidation treatment at 24°C for 1h, which is the main reason for the significant improvement of the strength and hardness

of 6061 aluminum alloy [3]. On the other hand, due to the inhibition effect of grain boundary and the second relative dislocation motion and plastic deformation within the grain, the tensile plasticity of 6061 aluminum alloy after oxidation at 24°C for 1 h decreased significantly.

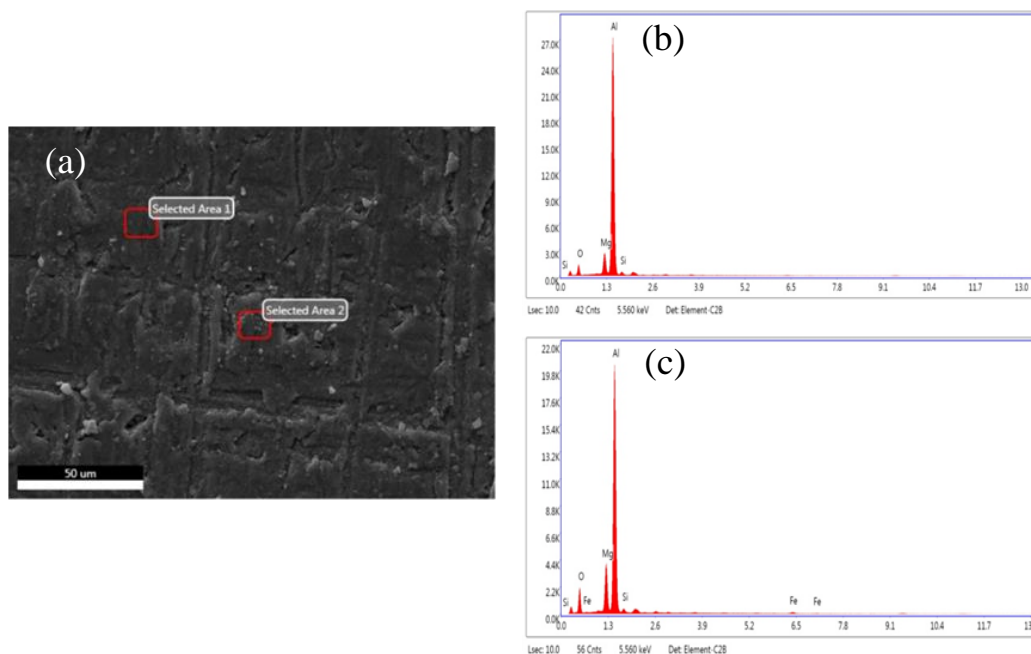


Figure 3. Energy spectrum of aluminum alloy after 18V, 24°C, 1 h oxidation treatment; followed by 350°C, 4 h heat treatment.

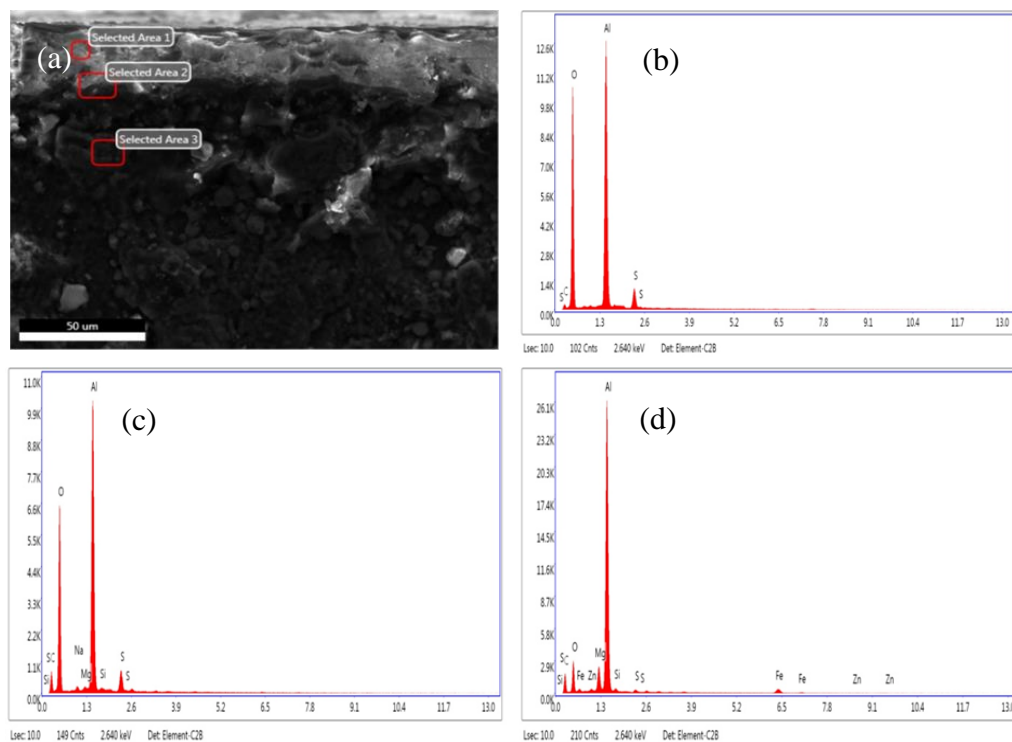


Figure 4. Energy spectrum of aluminum alloy after 18V, 24°C, 1 h oxidation treatment; followed by 550°C, 4 h heat treatment.

At the same time, the grains were obviously found to coarsely change into spherical particles, and the number of grains decreased obviously. Figures 4(b–d) show the SEM and EDS images of the original 6061 aluminum alloy samples after heat treatment at 350°C for 4 h. Clearly, the grain of the aluminum alloy is distributed with granular or long and short rod-like second phase, and the energy spectrum shows that the main components are Al, O, Mg, and Si, that is, the ternary compound of Al, Mg, and Si is the main strengthening phase of the aluminum alloy.

3.4 Electrochemical measurements

Figure 5 shows the variation in 6061 aluminum alloy in Na₂SO₄ solution under different heat treatment states. According to the provisions [7–9], a material may be considered to be corrosion sensitive in a corrosive medium. It was found that the 6061 aluminum alloy in three states has corrosion sensitivity. The corrosion sensitivity of 6061 aluminum alloy is: oxidation +550°C heat treatment > oxidation +350°C heat treatment.

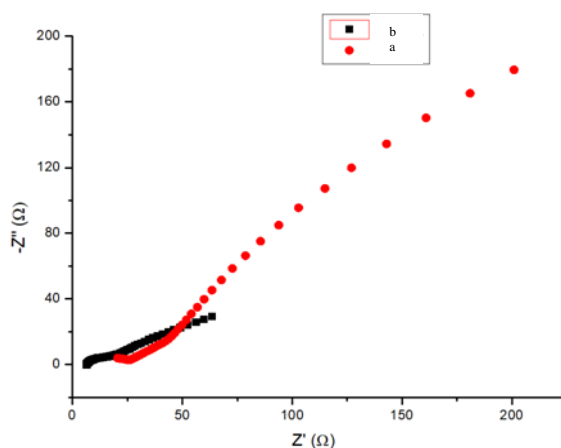


Figure 5. Nyquist plots of 6061 aluminum alloy at pH=2 (a: Oxidation treatment at 550°C + 4h heat treatment, b: non-oxidized at 550°C)

Table 2. Impedance parameters of 6061 aluminum alloy at pH=2

pH=2	Rs(Qcm ²)	Q(cm ² s ⁿ Q)	N	Rt(Qcm ²)
a	4.0	2.68×10 ⁻⁵	0.81	3.53×10 ⁴
b	4.8	2.76×10 ⁻⁵	0.79	4.22×10 ⁴
c	5.6	3.31×10 ⁻⁵	0.71	8.68×10 ⁴

The electrochemical impedance of 6061 aluminum alloy under oxidation treatment and 550°C+4h heat treatment indicates higher corrosion resistance than that under non-oxidized and

550°C+4h heat treatment condition. At oxidation treatment and 550°C+4h heat treatment, the diameter of capacitive semicircles on day 10 increased significantly compared to non-oxidized and 550°C+4h heat treatment. Furthermore, a semi-finite Warburg element (W) appeared on day 10 at pH=2, which illustrates that the electron transfer was mainly controlled by diffusion. The constants of 6061 aluminum alloy at different temperatures are shown in Figure 5, and the electrochemical parameters are listed in Table 2. The solution impedance (R_s) presented different values under the effect of heat treatment, exhibiting the highest value of R in comparison with sample with 550°C heat treatment. Figure 6 exhibits that the circuit of metal subjected to heat treatment under different temperature conditions in solutions with pH=2 was found to be $R(QR)(QR)$. With the increase in the heat treatment temperature, the corrosion reaction continued and the layer of rust on 6061 Aluminum alloy increased [18–20]. Accumulation of products layer on the surface of the sample directly affects the R_p value of the sample [19]. The R value following 10 days of exposure to temperature at 550°C was higher compared to that at temperature of 550°C for non-oxidized sample, indicating the formation of protective film on the surface of the sample.

- R_s — Solution resistance
- R_p — Resistance of electrochemical film
- R_{ct} — Charge transfer resistance
- Q_p — Resistance of passivated film
- Q_{dl} — Double layer capacitance

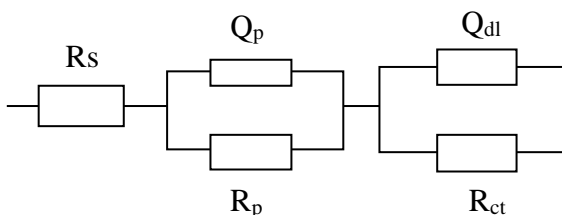


Figure 6. Schematic illustration and equivalent electrical circuits used for fitting EIS diagrams.

4. CONCLUSIONS

After oxidation and heat treatment at 550°C, the 6061 aluminum alloy was prone to element segregation near the grain boundary, resulting in the potential difference between the precipitation phase and the grain boundary and leading to the corrosion of the material and generation of the intergranular fracture. Owing to the uniform distribution of elements near the grain boundary, the potential difference between the precipitation phase at grain boundary and that around grain boundary was large, at grain boundary it was continuous, and inside the grain it was large and loose. However, a potential difference was observed between the precipitation zone and the precipitation phase at grain boundary, thus the corrosion of the material occurred and its corrosion sensitivity was the highest. The corrosion sensitivity of 6061 aluminum alloy is as follows: oxidation +550°C heat treatment > non-oxidized +550°C heat treatment. Under the heat treatment at 550°C, the 6061 aluminum alloy samples fractured along the crystal, but secondary cracks were present in the corrosion fracture. Dimples were observed in the corrosion fracture of the sample subjected to heat treatment at 550°C, which corresponded to ductile fracture. Thick second phase particles were distributed within the crystal boundary of the original 6061 aluminum alloy. After being oxidized at 24°C for 1 h, the ratio of 6061

aluminum alloy and the original 6061 aluminum alloy was obviously refined after being oxidized at 550°C for 4 h. After 24°C, 1 h oxidation treatment+550°C heat treatment, the grains also precipitated in the form of irregular particles. After being oxidized at 18V for 1 h, the fundamental reason for the plasticity of 6061 aluminum alloy was the obvious change of its structure during the oxidation treatment. The original impact fracture of 6061 aluminum alloy and the oxidation treatment of 6061 aluminum alloy show the characteristics of brittle fracture along the crystal.

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