

Study of Sealing of Anodized Aluminium in Mixed Titanium-Cerium Salt Solutions

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5052 aluminium alloy as building structure material was used as the substrate to be anodized and then sealed respectively in boiling deionized water, titanium salt solution, cerium salt solution, and mixed titanium-cerium salt solutions to further improve the corrosion resistance of anodized aluminium and prolong the service life of 5052 aluminium alloy. The experimental results show that, the anodized aluminium sealed in boiling deionized water, titanium salt solution, cerium salt solution, and mixed titanium-cerium salt solutions shows different morphology features with different elemental composition and hydrophobic performance which can reduce the corrosion rate of 5052 aluminium alloy to significantly improve the corrosion resistance. $\text{Ti}(\text{OH})_2$, $\text{Ti}(\text{OH})_4$, $\text{Ce}(\text{OH})_3$ and $\text{Ce}(\text{OH})_4$ precipitates and gel film can be formed during synergetic effect between ammonium hexafluorotitanate and cerium(III) nitrate as a mixed sealing solution, which can play a combination of physical filling and covering effect. After sealing in mixed titanium-cerium salt solutions, the anodized aluminium is more flat and compact and exhibits hydrophobic performance, with the lowest weight loss of 0.62 mg/cm^2 , the highest charge transfer resistance of $4.06 \times 10^3 \Omega \cdot \text{cm}^2$ and the highest low-frequency impedance of $6.34 \times 10^3 \Omega \cdot \text{cm}^2$. The aluminium sealed in mixed titanium-cerium solutions shows excellent corrosion resistance and can provide better corrosion protection for 5052 aluminium alloy.

Keywords: Anodic oxidation; 5052 aluminium alloy; Building structure material; Sealing

1. INTRODUCTION

5052 aluminium alloy has medium strength, good molding performance, better weldability and corrosion resistance, which is widely used in the construction industry as window frames, light frames, handrails, decorative columns, and other building components [1-5]. The 5052 aluminium alloy as building components may be exposed to the natural environment for a long time, and affected by various factors such as rainwater and salt spray, resulting in severer corrosion. To improve the corrosion resistance of building components, the anodic oxidation process is usually used for surface

treatment. The anodized aluminium is generated on the surface of 5052 aluminium alloy which plays a good corrosion protection role [6-10]. Although the corrosion resistance of the anodized aluminium is better than that of the naturally occurring oxide film, the porous structure leads to the strong adsorption characteristics of the anodized aluminium, which is prone to severe corrosion when it is in a highly corrosive environment. Therefore, sealing treatment is needed to further improve the corrosion resistance of the anodized aluminium.

The traditional sealing processes include dilute chromate solution sealing, dichromate solution sealing, boiling deionized water sealing, nickel salt solution sealing and so on [11-14]. Although the use of dilute chromate solution, dichromate solution and nickel salt solution to seal the anodized aluminium can improve its corrosion resistance significantly, these solution contain heavy metal ions, which is harmful to the environment and human health. In recent years, in view of the disadvantages of traditional sealing processes, researchers have developed some typical environmental-friendly sealing processes, such as sol-gel sealing, titanium salt solution sealing, silicate solution sealing and cerium salt solution sealing [15-20]. Compared with the traditional sealing processes, the environmental-friendly sealing process has the advantages of low energy consumption, and the solution used is less harmful to the environment and human health.

However, the corrosion resistance of the anodized aluminium sealed in a single metal salt solution cannot meet the requirements of modern industry. Under the premise of not increasing the process and greatly decreasing the cost, it is of great significance to realize sealing of the anodized aluminium in a mixed solution. The research in this area is innovative and can provide new ideas for further improving the corrosion resistance of anodized aluminium and extending the service life of aluminium alloy building components.

2. EXPERIMENTAL

2.1 Preparation and pretreatment of 5052 aluminium alloy

The 5052 aluminium alloy sheet with a thickness of 3 mm was cut into long strips along the length direction, and then some samples with the size of 48 mm×20 mm were prepared. Before the experiment, the samples were pretreated according to the following process. Firstly, the 1200 mesh and 2000 mesh sandpapers were used to repeatedly grind the sample until the surface of the sample is smooth. Secondly, a cotton stick was dipped in acetone to wipe the sample after polishing. And then, ultrasonic cleaning was used to clean the sample in the absolute ethanol for 6 minutes. Thirdly, hot water was used to clean the sample after oil removal, and then the sample was immersed in the volume fraction of 15% hydrochloric acid solution for 1 minute; Finally, the sample was clean and dried.

2.2 Anodic oxidation of 5052 aluminium alloy

The pretreated 5052 aluminium alloy sample and the pure aluminium plate were installed on special fixtures, placed vertically facing each other and quickly immersed in the electrolyte (the main

component was 98% sulphuric acid 200 g/L). After the electrolyte was heated to about 24 °C and kept for 1 h, the power was turned on to conduct anodic oxidation experiment. The anode current density was set to 1.5 A/dm². The constant current mode lasted for 50 minutes, and the anodized aluminium was formed on the surface of the sample. After the experiment, the power was disconnected, and the samples were repeatedly washed with deionized water at room temperature.

2.3 Sealing of anodized aluminium

The anodized aluminium was immersed into a mixed titanium-cerium salt solution at 60°C (ammonium hexafluorotitanate (H₈F₆N₂Ti) 40 g/L, cerium(III) nitrate 4 g/L, hydrogen peroxide 10 mL/L) to be sealed. The sealing process lasted for 40 minutes. Moreover, the anodized aluminium alloy samples were also immersed in boiling deionized water, titanium salt solution at 60°C (ammonium hexafluorotitanate (H₈F₆N₂Ti) 40 g/L) and cerium salt solution at 60°C (cerium(III) nitrate 4 g/L and hydrogen peroxide 10 mL/L) respectively to seal the anodized aluminium. The sealing process also lasted for 40 minutes. For simplicity, the anodized aluminium sealed in boiling deionized water was called anodized aluminium sealed in BW. The anodized aluminium sealed in titanium salt solution was called anodized aluminium sealed in TS, and the anodized aluminium sealed in cerium salt solution was called anodized aluminium sealed in CS. The anodized aluminium sealed in mixed titanium-cerium salt solutions was called anodized aluminium sealed in TS and CS.

2.4 Characterization and performance test of anodized aluminium

Quanta 450 scanning electron microscope was used to select a representative area with a magnification of 30,000 times, and the surface morphology of different anodized aluminium was observed in the autofocus mode. The images captured by scanning electron microscope were processed by Image J software, and the morphology features of different anodized aluminium were extracted by converting the format, selecting the threshold value and adjusting the contrast in turn. The extracted pore areas are filled with red color, and the ratio of the red area to the total area of the image can be used as the basis for evaluating the compactness of different anodized aluminium. In addition, the elemental composition of different anodized aluminium was analyzed by X-max 80 spectrometer.

The contact angle of water droplets with a volume of 3 µL on the surface of different anodized aluminium was measured by Theta Lite contact angle meter, and the contact angle of water droplets on the surface of 5052 aluminium alloy were measured for comparison. According to the principle of taking the average value of multi-point measurement, a more real contact angle was obtained, and then the hydrophobicity of different anodized aluminium was evaluated.

The weight loss of different anodized aluminium after immersion in 3.5% sodium chloride solution at room temperature for 6 days was tested and its unit was mg/cm². In addition, a representative area was selected by scanning electron microscope and magnified 30,000 times, and the corrosion morphology of different anodized aluminium was observed in autofocus mode. Combined

with the weight loss and corrosion morphology, the effect of the anodized aluminium sealed in different solution can be evaluated.

Parstat2273 electrochemical workstation was used to test the electrochemical impedance spectroscopy (including Nyquist spectrum and Bode spectrum) of 5052 aluminium alloy and different anodized aluminium after immersion in 3.5% sodium chloride solution for 6 days. The reference electrode is a saturated calomel electrode, the auxiliary electrode is a platinum electrode and the working electrode is a 5052 aluminium alloy or different anodized aluminium sample with the exposed area of 1 cm². The electrochemical impedance spectroscopy test was started after the open circuit potential was stable, the disturbance potential was 10 mV, and the test frequency range was from 10⁵ Hz to 10⁻² Hz. Combined with the fitting results of electrochemical impedance spectroscopy, the effect of the anodized aluminium sealed in different solution was further evaluated.

3. RESULTS AND DISCUSSION

3.1 Analysis of surface morphology and elemental composition

Figure 1 shows the surface morphology of 5052 aluminium alloy and different anodized aluminium. The surface morphology of unsealed anodized aluminium is porous with cracks which is similar to the results reported by the researchers [21-23]. It can be seen from Figure 1(c), 1(d), 1(e) and 1(f) that, after sealed in boiling deionized water, titanium salt solution, cerium salt solution and mixed titanium-cerium salt solutions, four anodized aluminium with distinct morphological characteristics are obtained. Compared with the unsealed anodized aluminium, the pores on the surface of anodized aluminium sealed in BW, anodized aluminium sealed in TS, anodized aluminium sealed in CS and anodized aluminium sealed in TS and CS are significantly less, which indicates that the sealing treatment in boiling deionized water, titanium salt solution, cerium salt solution and mixed titanium-cerium salt solutions are effective. In comparison, the surface of the anodized aluminium sealed in TS and CS is smoother and denser, and its morphology quality is the best. Although the morphology quality of anodized aluminium sealed in TS and anodized aluminium sealed in CS are not as good as anodized aluminium sealed in TS and CS, they are both better than that of the anodized aluminium sealed in BW.

The alumina hydration reaction occurs during the sealing process in boiling deionized water (see the first reaction equation). During the reaction, boehmite alumina is formed, and the pores on the surface of anodized aluminium are filled and blocked by the volume expansion effect under high temperature conditions. Due to the uneven deposition of boehmite alumina, boiling deionized water has an unsatisfactory sealing effect on the anodized aluminium [24]. During the sealing process in titanium salt solution, the hydrolysis reaction of ammonium hexafluorotitanate occurs to form the precipitation of Ti(OH)₂ and Ti(OH)₄ (see the second and third reaction equations), and the anodized aluminium is sealed by the physical filling effect [25]. Moreover, Ce(OH)₃ and Ce(OH)₄ are precipitated by cerium ion precipitation during the sealing process in cerium salt solution (see the fourth, fifth and sixth reaction equations). In addition, the gradual deposition of Ce(OH)₃ and Ce(OH)₄

on the surface of the anodized aluminium may form a gel film, which covers the surface of the anodized aluminium and also plays a role in sealing pores. During the sealing process in mixed titanium-cerium salt solutions, the hydrolysis reaction of ammonium hexafluorotitanate to generate $Ti(OH)_2$ and $Ti(OH)_4$. Moreover, hydrogen peroxide can oxidize Ce(III) to Ce(IV) and form $Ce(OH)_4$ [26-27]. The anodized aluminium sealed in TS and CS has the best morphology quality and its surface is smoother and denser.

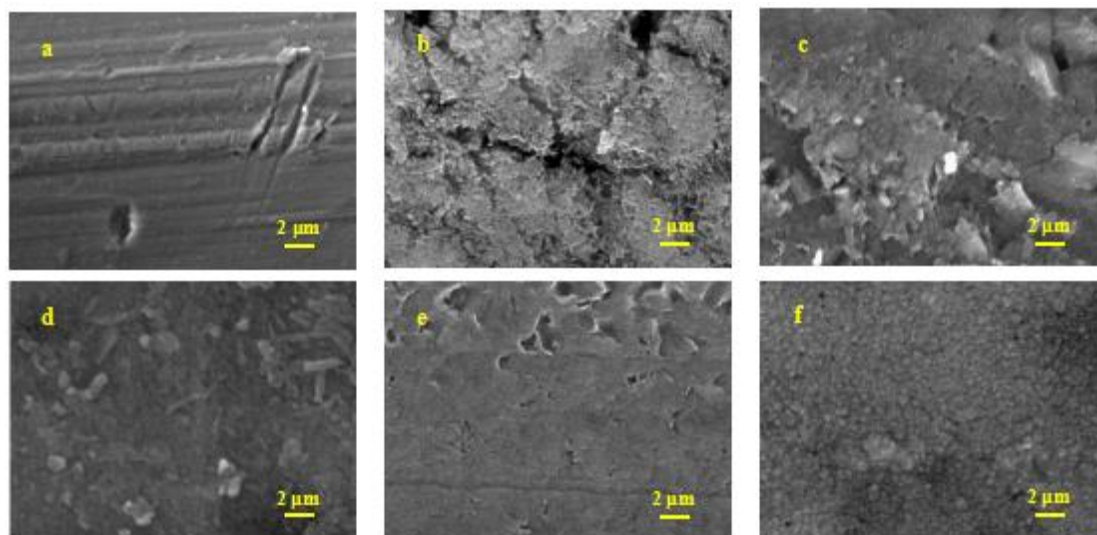


Figure 1. Surface morphology of 5052 aluminium alloy and different anodized aluminium: a-5052 aluminium alloy; b-unsealed anodized aluminium; c-anodized aluminium sealed in BW; d-anodized aluminium sealed in TS; e-anodized aluminium sealed in CS; f-anodized aluminium sealed in TS and CS

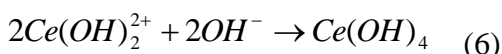
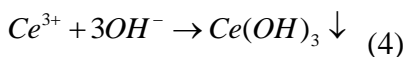
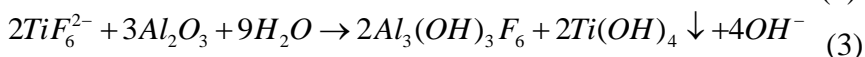
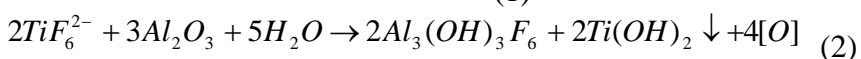
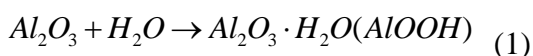


Figure 2 shows the morphological characteristics and pores distribution of different anodized aluminium. The pore area ratio of unsealed anodized aluminium, anodized aluminium sealed in BW, anodized aluminium sealed in TS, anodized aluminium sealed in CS and anodized aluminium sealed in TS and CS are obtained by calculation, which is 20.4%, 8.8%, 6.7%, 3.4%, and 1.5%, respectively. It is found out that the porosity of anodized aluminium after sealing is much smaller than that of unsealed anodized aluminium. Among them, the pore area ratio of the anodized aluminium sealed in TS and CS is the lowest, which is only 1.5%. It can be confirmed that mixed titanium-cerium salt solutions has the

best sealing effect on the anodized aluminium, so that more pores are filled and blocked to significantly improve the compactness.

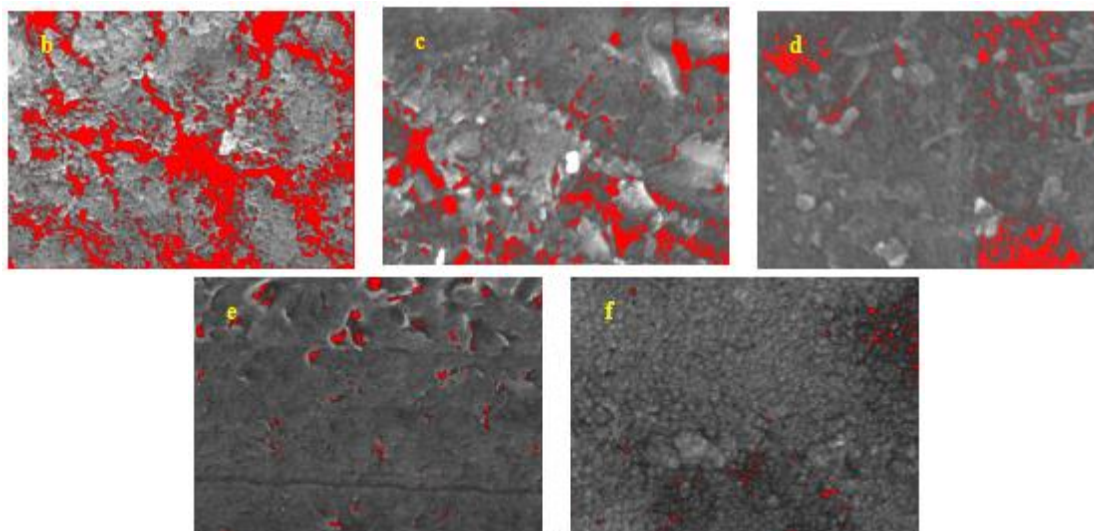
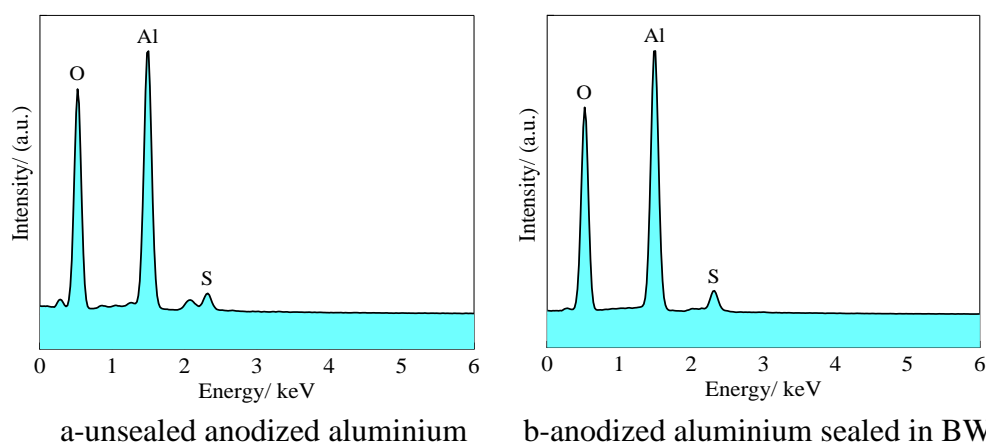


Figure 2. Morphological characteristics and pores distribution of different anodized aluminium: b-unsealed anodized aluminium; c-anodized aluminium sealed in BW; d-anodized aluminium sealed in TS; e-anodized aluminium sealed in CS; f-anodized aluminium sealed in TS and CS

It can be seen from Figure 3(a) and Figure 3(b), the elemental composition of unsealed anodized aluminium and anodized aluminium sealed in BW are similar. It can be seen from Figure 3(c) and Figure 3(d) that in addition to Al, O and S elements, Ti and Ce elements are respectively found. Ti element is introduced into the anodized aluminium in the form of Ti(OH)_2 and Ti(OH)_4 , while Ce element is introduced into the anodized aluminium in the form of Ce(OH)_3 and Ce(OH)_4 . It can be seen from Figure 3(f) that in addition to Al, O and S elements, Ti and Ce elements are also included in the anodized aluminium. Ti(OH)_2 , Ti(OH)_4 , Ce(OH)_3 and Ce(OH)_4 precipitates and gel films are formed simultaneously during the sealing process in mixed titanium-cerium salt solutions, so that Ti and Ce elements are introduced into the anodized aluminium at the same time, which is confirmed by the above analysis results.



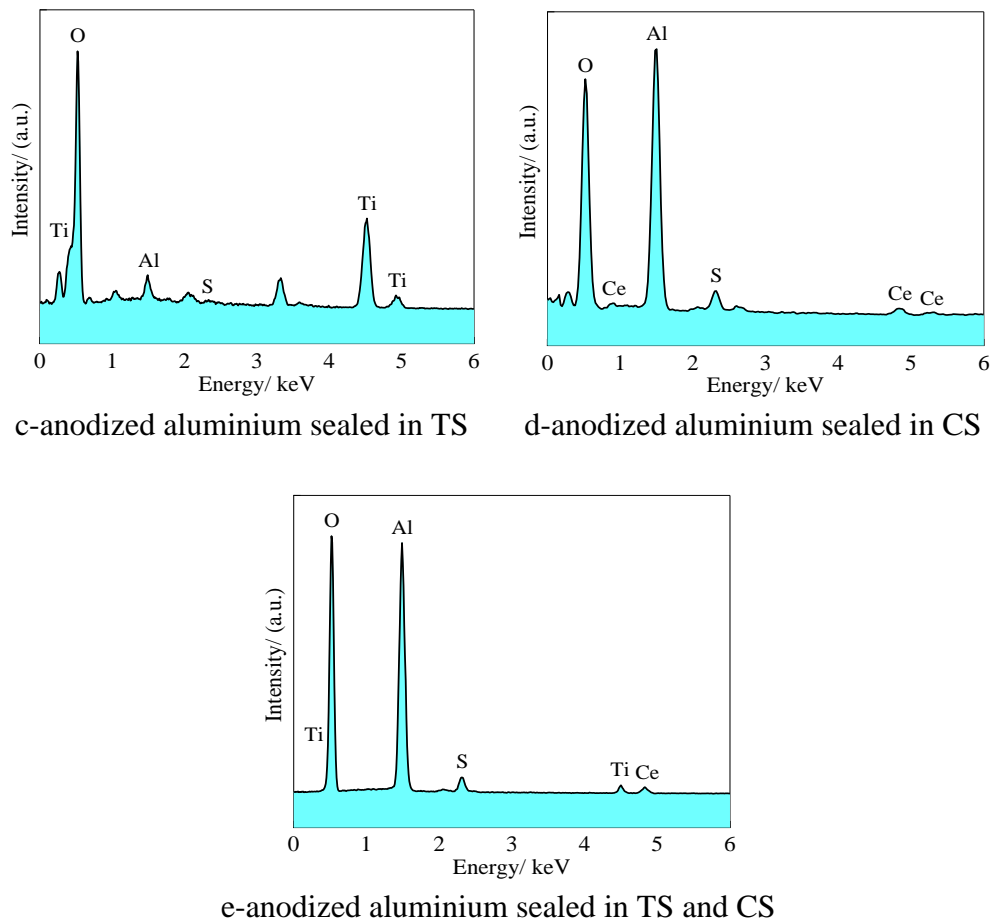


Figure 3. Energy spectrum of different anodized aluminium

3.2 Analysis of hydrophobicity, weight loss and corrosion morphology

Figure 4 shows the contact angle of water droplets on the surface of 5052 aluminium alloy and different anodized aluminium. According to the contact angle measurement results, it is known that 5052 aluminium alloy, unsealed anodized aluminium, anodized aluminium sealed in BW and anodized aluminium sealed in TS all exhibit hydrophilic performance, while the anodized aluminium sealed in CS and anodized aluminium sealed in TS and CS all exhibit hydrophobic performance, and the contact angle are 108.2° and 120.4° , respectively. Since a gel film may be formed during the sealing process in cerium salt solution and mixed titanium-cerium salt solutions, the covering effect reduces the surface energy of the anodized aluminium to increase hydrophobic performance [28]. In comparison, the surface hydrophobicity of the anodized aluminium sealed in TS and CS is better. The relationship between surface hydrophobicity and surface roughness is reported in some literatures [29-31]. The hydrophobic properties of anodized aluminium improve after sealing treatment, which may be due to the change of surface roughness.

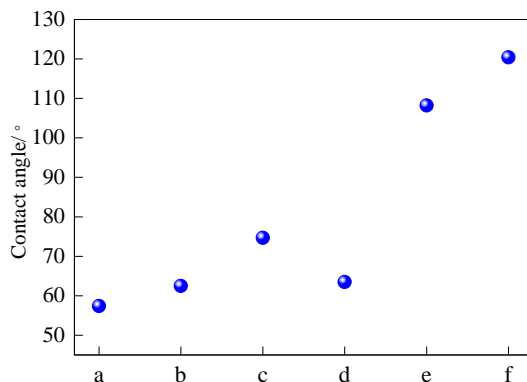


Figure 4. Contact angle of water drops on the surface of 5052 aluminium alloy and different anodized aluminium: a-5052 aluminium alloy; b-unsealed anodized aluminium; c-anodized aluminium sealed in BW; d-anodized aluminium sealed in TS; e-anodized aluminium sealed in CS; f-anodized aluminium sealed in TS and CS

Figure 5 shows the weight loss of different anodized aluminium. The order of weight loss from high to low is: unsealed anodized aluminium > anodized aluminium sealed in BW > anodized aluminium sealed in TS > anodized aluminium sealed in CS > anodized aluminium sealed in TS and CS. The anodized aluminium sealed in TS and CS has the lowest weight loss of 0.62 mg/cm^2 , this is due to the combination of physical filling effect and covering effect of the reaction products during the sealing process in mixed titanium-cerium salt solutions.

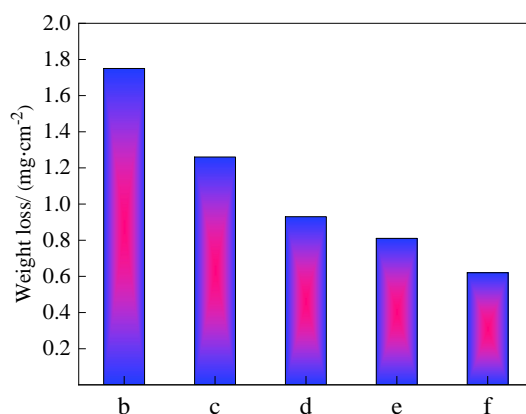


Figure 5. Weight loss of different anodized aluminium immersed in 3.5% sodium chloride solution for 6 days: b-unsealed anodized aluminium; c-anodized aluminium sealed in BW; d-anodized aluminium sealed in TS; e-anodized aluminium sealed in CS; f-anodized aluminium sealed in TS and CS

Figure 6 shows the corrosion morphology of different anodized aluminium after immersion in 3.5% sodium chloride solution for 6 days. By comparing Figure 6(a), Figure 6(b), Figure 6(c), Figure 6(d) and Figure 6(e), it can be seen that the corrosion degree of unsealed anodized aluminium, anodized aluminium sealed in BW, anodized aluminium sealed in TS, anodized aluminium sealed in

CS and anodized aluminium sealed in TS and CS are different. The corrosion degree of the anodized aluminium is significantly reduced after sealing. This is because different products generated during the sealing process play the role of filling and sealing the pores on the surface of anodized aluminium, which can prevent the corrosion medium from penetrating into the anodized aluminium and increase the corrosion resistance. After sealing in mixed titanium-cerium salt solutions, there are few pores on the surface of anodized aluminium, and its compactness is the best, showing a strong ability to prevent the erosion of corrosive media. In addition, the anodized aluminium sealed in TS and CS exhibits hydrophobic performance, which reduce the contact area with the corrosive medium, so its corrosion degree is the least.

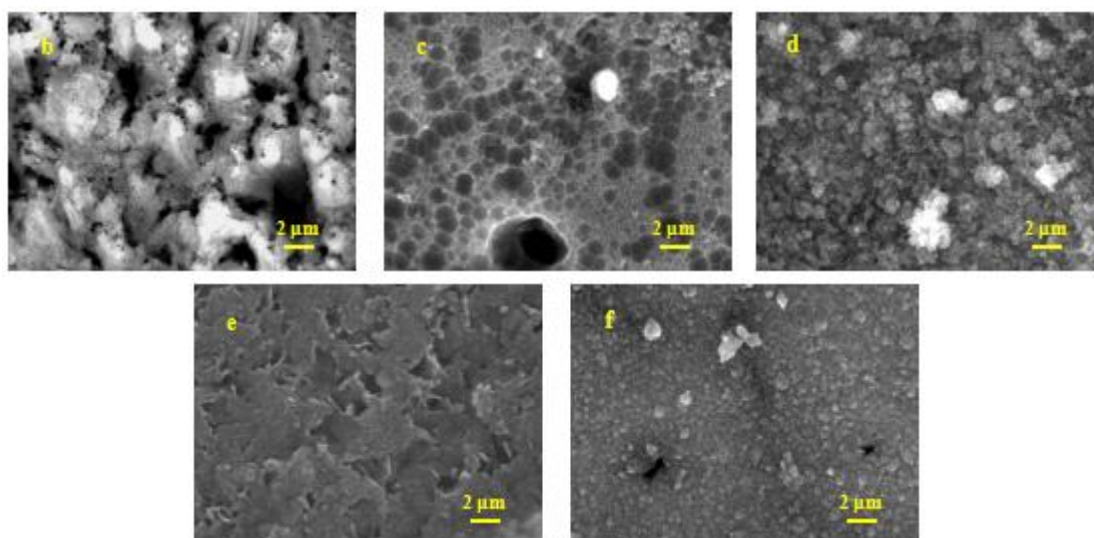


Figure 6. Corrosion morphology of different anodized aluminium after immersion in 3.5% sodium chloride solution for 6 days: b-unsealed anodized aluminium; c-anodized aluminium sealed in BW; d-anodized aluminium sealed in TS; e-anodized aluminium sealed in CS; f-anodized aluminium sealed in TS and CS

3.3 Electrochemical impedance spectroscopy analysis

Figure 7 shows the electrochemical impedance spectroscopy of 5052 aluminium alloy and different anodized aluminium after immersion in 3.5% sodium chloride solution for 6 days. Table 1 lists the charge transfer resistance and low-frequency impedance value of 5052 aluminium alloy and different anodized aluminium. It is reported that higher charge transfer resistance and lower low-frequency impedance can contribute to better corrosion resistance [32-34]. As can be seen from Table 1, the charge transfer resistance and low-frequency impedance value of 5052 aluminium alloy are the lowest. The charge transfer resistance of the anodized aluminium increases by $260 \Omega \cdot \text{cm}^2$, $620 \Omega \cdot \text{cm}^2$, $980 \Omega \cdot \text{cm}^2$ and $1540 \Omega \cdot \text{cm}^2$, respectively, after sealing in boiling deionized water, titanium salt solution, cerium salt solution and mixed titanium-cerium salt solutions. Moreover, the low-frequency impedance value are increased by $540 \Omega \cdot \text{cm}^2$, $1210 \Omega \cdot \text{cm}^2$, $1530 \Omega \cdot \text{cm}^2$ and $2460 \Omega \cdot \text{cm}^2$, respectively. It is further confirmed that sealing have certain pore sealing effect on the anodized aluminium, so that

the pores on the surface of anodized aluminium are filled and blocked, which effectively impedes the development of corrosion process.

Compared with the anodized aluminium sealed in BW, the charge transfer resistance and low-frequency impedance value of anodized aluminium sealed in TS, anodized aluminium sealed in CS and anodized aluminium sealed in TS and CS are increased sequentially. Among them, the anodized aluminium sealed in TS and CS has the highest charge transfer resistance of $4.06 \times 10^3 \Omega \cdot \text{cm}^2$ and the highest low-frequency impedance value of $6.34 \times 10^3 \Omega \cdot \text{cm}^2$.

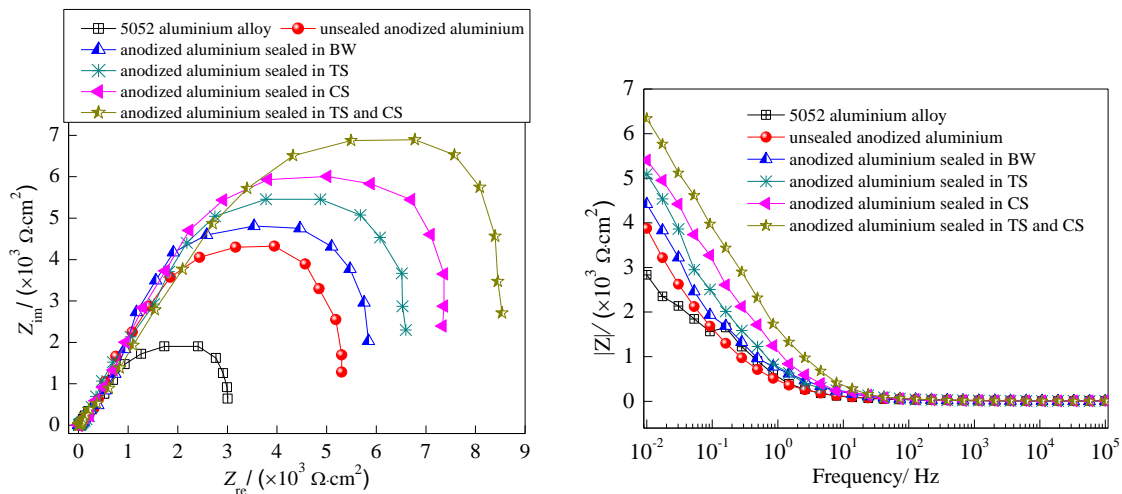


Figure 7. Electrochemical impedance spectroscopy of 5052 aluminium alloy and different anodized aluminium after immersion in 3.5% sodium chloride solution for 6 days

Table 1. Fitting results of electrochemical impedance spectroscopy

Different samples	Charge transfer resistance/ ($\Omega \cdot \text{cm}^2$)	Low-frequency impedance value/ ($\Omega \cdot \text{cm}^2$)
5052 aluminium alloy	1.43×10^3	2.83×10^3
unsealed anodized aluminium	2.52×10^3	3.88×10^3
anodized aluminium sealed in BW	2.78×10^3	4.42×10^3
anodized aluminium sealed in TS	3.14×10^3	5.09×10^3
anodized aluminium sealed in CS	3.50×10^3	5.41×10^3
anodized aluminium sealed in TS and CS	4.06×10^3	6.34×10^3

There are still some pores on the surface of anodized aluminium sealed in BW, so that the corrosion resistance is not ideal. During the sealing process in titanium salt solution and cerium salt solution, titanium and cerium hydroxide is respectively formed to achieve good sealing effect, which effectively inhibits the charge transfer and hinders the development of electrochemical corrosion process. Therefore, the corrosion protection effect of anodized aluminium sealed in TS and anodized

aluminium sealed in CS on 5052 aluminium alloy is better than that of unsealed anodized aluminium and anodized aluminium sealed in BW. During the sealing process in mixed titanium-cerium salt solutions, titanium and cerium hydroxide and gel film are generated simultaneously, so that the anodized aluminium shows excellent corrosion resistance and can provide better corrosion protection for 5052 aluminium alloy.

4. CONCLUSIONS

In this paper, 5052 aluminium alloy as building structure material is used as the substrate for anodic oxidation, and the anodized aluminium is sealed in different solutions to further improve the corrosion resistance and extend the service life of 5052 aluminium alloy. The following conclusions are obtained.

(1) After sealed in boiling deionized water, titanium salt solution, cerium salt solution and mixed titanium-cerium salt solutions, four anodized aluminium with distinct morphological characteristics are obtained. The products generated during the sealing process in different solutions all play the role of filling and sealing the pores on the surface of anodized aluminium, which can prevent the corrosion medium from eroding 5052 aluminium alloy and hinder the development of the electrochemical corrosion process.

(2) The anodized aluminium sealed in mixed titanium-cerium salt solutions has the best performance. $\text{Ti}(\text{OH})_2$, $\text{Ti}(\text{OH})_4$, $\text{Ce}(\text{OH})_3$ and $\text{Ce}(\text{OH})_4$ precipitation and gel film are generated to play physical filling effect and covering effect. The surface of the anodized aluminium sealed in mixed titanium-cerium salt solutions is more flat and compact, and has hydrophobic performance with excellent corrosion resistance.

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