Corrosion behavior of Cu/Al casting-rolled clad plates in different alkaline solution

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Abstract: The effect of pH value and different kinds of anions on the corrosion behavior of Cu/Al casting-rolled clad plates in the alkaline solution was evaluated by means of scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD), weight loss analysis, 3D confocal laser scanning microscopy (CLSM) and electrochemical test. Results show the corrosion mainly occurs on the aluminum side. The corrosion resistance of the Cu/Al decreases as the pH value increases. When $\text{pH} \geq 12$, the dissolution of the film layer is faster than the passivation process. The addition of Cl^- ions reduces the corrosion resistance of the Cu/Al clad plates, which leads to pitting corrosion. The higher the concentration of Cl^- ions, the more prone the pitting to occur. The addition of SO_4^{2-} ions causes the denudation of the samples. The corrosion resistance of the Cu/Al is better in the alkaline solution containing NO_3^- ions than that in the solution containing Cl^- ions or SO_4^{2-} ions. When adding SO_4^{2-} , NO_3^- and Cl^- to the pure alkaline solution, the corrosion resistance of the Cu/Al clad plates decreases.

Key words: Cu/Al clad plates; alkaline solution; pH; anions; electrochemistry; casting-rolled

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1 Introduction

As a new type of composites, the Cu/Al casting-rolled clad plates, which possess the advantages of both copper and aluminum, are broadly used in production and life^[1-6]. They have good heat dissipation, good electromagnetic shielding, smooth surface and stable size, good rigidity, and good mechanical processing performance. At present, in large heating equipment, transformer, smelting equipment, rectifier cabinet and control cabinet and other power instruments, the Cu/Al clad plates have been used to replace pure copper bar to make bus bars, which can greatly save copper and reduce costs, and have a considerable market and economic prospects^[7]. The potential of the copper is higher than the aluminum, and they form galvanic corrosion when they contact with each other, so the copper accelerates the corrosion process of the aluminum. Therefore, the corrosion resistance is also an important index to evaluate materials

in application^[8,9].

In an alkaline environment, such as the petroleum, synthetic ammonia, chlor-alkali, concrete and nuclear industries, the unstable structure of the aluminum and its alloys contribute to the poor corrosion resistance^[10-12]. At the same time, the aluminum alloys also undergo pitting corrosion, crevice corrosion and stress corrosion in the alkaline atmosphere, which increases the potential danger^[13-15]. It has been studied that the addition of Cl^- ions to acidic and neutral solutions increases the dissolution rate of aluminum, either by the participation of Cl^- ions in the dissolution reaction, or by the incorporation of Cl^- ions into the oxide film^[16,17]. In acidic and neutral solutions, NO_3^- ions can be reduced to NH_3 by elemental aluminum or nascent hydrogen^[18], and SO_4^{2-} ions can react with elemental aluminum to form Al(OH)SO_4 and AlSO_4^+ ^[19]. However, since these research works were conducted in acidic and neutral solutions, they provide little information about the anodic dissolution of pure aluminum in alkaline solution in the presence of Cl^- , NO_3^- and SO_4^{2-} . These anions competed with the hydroxyl ions adsorbing on the surface in alkaline solutions which made the corrosion process complicated. Due to the incorporation of anions, the anodic dissolution of the aluminum is also considered to be impacted by the change in chemical composition of the surface film. Hence, it is of vital importance to elucidate the impact of these anions on the corrosion behavior of the Cu/Al

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in the alkaline solution.

In this study, the influence of pH and Cl^- , SO_4^{2-} and NO_3^- on the corrosion behavior of the Cu/Al clad plates was studied by using the scanning electron microscope (SEM), energy dispersive spectroscopy (EDS), electrochemical test and gravimetric measurements. The experimental results obtained were discussed according to the adsorption of the anions on the surface and subsequent incorporation into the surface in the alkaline solution.

2 Experimental apparatus and methods

2.1 Materials preparation

The 1050 industrial pure aluminum and the industrial pure copper T2 strip (0.4 mm thick) were used in the experiment, and a continuous casting-rolling mill with working size of Φ (960–980) $\times$ (1,600–2,100) (unit: mm) [20] was employed. The aluminum was melted in a medium-frequency induction furnace. After surface treatment and stress relief annealing of the copper plate, the molten aluminum was poured on it and then cast rolled into shape on the casting mill under the protection of atmosphere. The pouring temperature of the liquid aluminum was 690 °C, and the preheating temperature of the copper plate was 200 °C. Finally, the Cu/Al cast-rolling clad plate with the dimensions of 20 mm $\times$ 20 mm $\times$ 3 mm was obtained.

Prior to the corrosion experiments, the cross-sections of the samples were ground with abrasive paper progressively, to a surface finish of 2,000 grit, and then polished to give a mirror-like surface. The coupons were then cleaned with an alcoholic solution, dried with a hair dryer, and finally reserved in the drying vessel for at least 48 h.

2.2 Immersion test

The samples were immersed in the prepared solution (NaOH with a pH of 8, 9, 10, 12 and 13) for 48 h at room temperature in each group of experiments. In addition, to evaluate the effect of Cl^- , SO_4^{2-} and NO_3^- on the corrosion behavior of the Cu/Al clad plates, solutions containing 0.01 M NaOH, 0.01 M NaOH+0.1 M NaCl, 0.01 M NaOH+0.1 M Na_2SO_4 , and 0.01 M NaOH+0.1 M KNO_3 , respectively, were prepared. At least three replicated specimens were tested to ensure accuracy. The pH of the 0.01 M NaOH solution was equal to 10. The experiment was conducted at room temperature.

2.3 Specimen detection

A scanning electron microscope (SEM) (Hitachi s-3400N) equipped with an 132-10 energy disperse spectroscopy (EDS) was used to observe the corrosion morphology of the coupons. The 3D surface morphology was observed by confocal laser scanning microscopy (CLSM, OLYMPUS LEXT OLS4000). The constituents of the corrosion products were determined with an X-ray diffractometer (Shimadzu XRD-7000).

2.4 Electrochemical analysis

Prior to electrochemical tests, the uncorroded specimens were sealed with the hot melt adhesive, leaving an exposed area

(cross-section) of 0.6 cm². The tests were carried out using a VSP-300 electrochemical workstation. A traditional three-electrode system was employed using a platinum filament as the counter electrode, saturated calomel electrode (SCE) as the reference electrode, and the test samples as the working electrode. The potentiodynamic polarization curves were measured within -1 V–1 V [relative to the open-circuit potential (OCP)].

2.5 Gravimetric measurements

The expression $(g_0 - g_1)/s$ was used to characterize the weight change of the Cu/Al, where g_0 represents the initial weight, g_1 is the final weight, and s is the total surface area of the sample. The Cu/Al clad plate samples were weighed using a vacuum scale with a precision of 0.001 g, prior to and after immersion corrosion. The 50 mL H_3PO_4 and 1,000 mL distilled water were mixed to remove the corrosive medium residue and the corrosion products, and then the specimens were washed with ultrasonic cleaners for 30 min and were finally dried with hot air prior to conducting the weight loss measurements. A linear equation, $W = ax + b$, was used to fit the relationship between the weight loss and pH. Among which, W represents the weight loss of the Cu/Al clad plate, x is the pH value.

3 Results and discussion

3.1 Corrosion morphology and corrosion products

Figure 1 shows the corrosion morphology of cross-sections of Cu/Al clad plates without removing the corrosion products, in the NaOH solution with different pH values for 48 h. When $\text{pH} \leq 10$, in the initial age of the corrosion process, there is very little OH^- , so the formation rate of corrosion products is slow. There are only a few ring-shaped corrosion products at or near the interface of the Cu and the Al, mainly on the Al side. The obvious galvanic corrosion can be observed at the interface when $\text{pH} = 12$, and at the same time, the corrosion products peel off from the matrix. When $\text{pH} = 13$, the severe denudation appears on the aluminum, a large area of the matrix peels off while the copper matrix remains unchanged.

Figure 2 shows the surface morphology of Cu/Al clad plates after immersion for 48 h in four kinds of solution containing different ions, respectively. It can be seen from Fig. 2(a) that few corrosion products which are in ring shape appear at the interface of the sample after corrosion in the NaOH solution, mainly on the Al side. When adding Cl^- to the NaOH solution, as shown in Fig. 2(b), a lot of corrosion products are generated. The corrosion products are cracked and partly stacked together, appearing as a bulge on the Al. With the addition of SO_4^{2-} , there is no obvious pitting on the aluminum matrix, and punctuated corrosion products covers on the matrix, as shown in Fig. 2(c). When NO_3^- is present, the surface of the aluminum matrix is covered with a thin layer of snow-flake shaped corrosion products [Fig. 2(d)], no pitting occurs.

The 3D CLSM images can directly reflect the macroscopic morphology and the depth of the corrosion pits of Cu/

Fig. 1: Corrosion morphology of Cu/Al clad plates in NaOH solution with pH value of 8 (a), 9 (b), 10 (c), 12 (d) and 13 (e) for 48 h

Fig. 2: Corrosion morphology of Cu/Al clad plates in 0.01 M NaOH solution with addition of none (a), 0.1 M NaCl (b), 0.1 M Na_2SO_4 (c), and 0.1 M KNO_3 (d)

Al clad plates after 48 h immersion in different kinds of alkaline solution (as shown in Fig. 3). When $\text{pH} \leq 10$, local corrosion is the main corrosion form. Corrosion pits with the maximum depth of 2.519 μm are observed on the surface of the sample when $\text{pH}=8$. The maximum depth of the corrosion pits increases to 3.122 μm when $\text{pH}=9$, and 3.831 μm when $\text{pH}=10$. Obvious denudation is observed with a maximum depth of 8.543 μm when $\text{pH}=12$, and 53.582 μm when $\text{pH}=13$. According to the three-dimensional corrosion morphology of samples in the 0.01 M NaOH solution with the addition of 0.1 M NaCl [Fig. 3(f)], the corrosion pits have a maximum depth of

223.699 μm , while the denudation and corrosion pits are observed on the samples in the 0.01 M NaOH solution with the addition of 0.1 M Na_2SO_4 [Fig. 3(g)], with a maximum depth of 7.022 μm . For samples immersion corrosion in the 0.01 M NaOH solution with the addition of 0.1 M NaNO_3 [Fig. 3(h)], the number of corrosion pits is relatively small, with a maximum depth of 5.536 μm , and a small amount of denudation near the interface of the Cu and Al is observed.

Figure 4 shows the elemental distribution on the surface of Cu/Al clad plates in the 0.01 M NaOH solution with the addition of different ions for 48 h immersion corrosion. The

Fig. 3: 3D CLSM images of Cu/Al clad plates after immersion corrosion under different kinds of alkaline solution for 48 h: in NaOH solution of pH=8 (a), pH=9 (b), pH=10 (c), pH=12 (d), and pH=13 (e); and 0.01 M NaOH+0.1 M NaCl (f); 0.01 M NaOH+0.1 M Na_2SO_4 (g); 0.01 M NaOH+0.1 M KNO_3 (h)

Fig. 4: Elements distribution on surface of Cu/Al clad plates in 0.01 M NaOH solution with addition of none (a), 0.1 M NaCl (b), 0.1 M KNO_3 (c), and 0.1 M Na_2SO_4 (d)

newly generated corrosion products on the Al side mainly consist of oxygen elements. As shown in Fig. 4(a), oxygen elements are concentrated on the white bulk corrosion products. In NaOH solution, localized corrosion tends to appear at the interface of the Cu and Al. With addition of NaCl solution, the amount of oxygen elements increases and they almost cover the whole aluminum surface. Chlorine elements show little coverage because the final corrosion product AlCl_4^- is easily soluble in water and hard to stay on the surface. In the NO_3^- containing solution, the oxygen elements are uniformly distributed on the surface. Besides, a small amount of nitrogen

elements uniformly adsorbs on the surface. In the alkaline solution with the addition of SO_4^{2-} , oxygen elements are mainly distributed at the interface of the Cu and the Al. There are more sulfur elements adsorbed on the surface of the matrix than the chlorine elements and nitrogen elements.

3.2 Potentiodynamic polarization curves

Figure 5 shows the potentiodynamic polarization curves of the Cu/Al clad plate measured in different pH of the NaOH solution. Table 1 shows the corresponding fitting results. The corrosion current density reflects the corrosion resistance qualitatively [21].

Fig. 5: Potentiodynamic polarization curves of Cu/Al clad plate measured in different pH of NaOH solution

Table 1: Fitting results of polarization curves

pH values	8	9	10	12	13
J_{corr} ($\mu\text{A}\cdot\text{cm}^{-2}$)	3.320	4.214	4.325	30.917	403.52
E_{corr} (V vs. SCE)	-0.49	-0.53	-0.56	-1.27	-1.44

The corrosion current density (J) increases as the pH increases, as shown in Table 1. When $\text{pH} \leq 10$, the anodic current density progressively increases as the potential increases until reaching the pitting potential. However, when $\text{pH}=12$, the anodic current density remains constant with the increase of applied potential below the pitting potential, and there is no pitting occurs when the pH reaches 13.

Figure 6 shows the potentiodynamic polarization curves of the Cu/Al clad plate measured in NaOH solution with the addition of different concentrations of NaCl. Table 2 shows the corresponding fitting results. With the increase of the NaCl concentration added to the NaOH solution, the corrosion current density increases

Fig. 6: Potentiodynamic polarization curves of Cu/Al clad plate measured in NaOH solution with addition of different concentrations of NaCl

while the corrosion potential decreases, which indicates that Cl^- ions reduces the corrosion resistance of the Cu/Al. Without Cl^- ions, the anodic current density progressively increases with increasing applied potential. However, in the presence of Cl^- ions, the anodic current density remains constant below the pitting potential. The pitting potential moves to a negative value when the concentration of Cl^- ions increases, indicating that the higher the concentration of Cl^- ions, the more prone the pitting to appear. Pitting is observed at about $-0.45V_{\text{SCE}}$ in the 0.01 M NaCl containing alkaline solution, $-0.67V_{\text{SCE}}$ in the 0.1 M NaCl containing alkaline solution, and $-0.72V_{\text{SCE}}$ in the 0.3 M NaCl containing alkaline solution. The lowest pitting potential in the 0.3 M NaCl containing alkaline solution represents the quickest formation rate of AlCl_4^- , and the pitting appears due to the formation of AlCl_4^- [22]. The pitting easily occurs on the surface of the specimen in the Cl^- -containing solution. The pitting develops vertically and horizontally, and eventually causes serious damage to the material [23], so the corrosion resistance decreases.

Table 2: Fitting results of polarization curves

Solution	0.01 M NaOH +0 M NaCl	0.01 M NaOH +0.01 M NaCl	0.01 M NaOH +0.1 M NaCl	0.01 M NaOH +0.3 M NaCl
J_{corr} ($\mu\text{A}\cdot\text{cm}^{-2}$)	4.325	153.567	180.036	198.087
E_{corr} (V vs. SCE)	-0.65	-1.38	-1.36	-1.36

Figure 7 shows the potentiodynamic polarization curves of the Cu/Al clad plate measured in NaOH solution with the addition of solutions containing different types of corrosive ions, and the corresponding fitting results are listed in Table 3. When the anions are added, the corrosion current density increases, which implies that the added anions (Cl^- , SO_4^{2-} , NO_3^-) reduces the corrosion resistance of the Cu/Al. In the presence of Cl^- , pitting occurs at about -0.7 V. In the solution containing SO_4^{2-} , pitting appears after the first passivation area and then the second passivation area appears immediately. When the solution contains NO_3^- , the corrosion current density almost remains constant under the applied potential on the anodic polarization curve. Compared with the other two kinds of anions, with the presence of NO_3^- , the corrosion current density is the smallest which indicates the highest corrosion resistance of Cu/Al.

Fig. 7: Potentiodynamic polarization curves of Cu/Al clad plate measured in NaOH solution with addition of different kinds of corrosive ions

Table 3: Fitting results of polarization curves

Solution	0.01M NaOH	0.01 M NaOH +0.1 M NaCl	0.01 M NaOH +0.1 M Na ₂ SO ₄	0.01 M NaOH +0.1 M KNO ₃
J_{corr} ($\mu\text{A}\cdot\text{cm}^{-2}$)	4.325	180.036	126.266	88.434
E_{corr} (V vs. SCE)	-0.65	-1.36	-1.39	-1.15

3.3 Weight loss analysis

Figure 8 shows the weight loss of Cu/Al clad plates after immersion corrosion. As can be seen in Fig. 8(a), when the pH value of the solution increases, the weight loss of the Cu/Al clad plates gradually increases. When the pH reaches 13, the weight loss increases rapidly, which is 16 times greater than that of 12. When $\text{pH} \leq 12$, the relationship between the weight loss and pH can be expressed through a

linear function: $w=2.5x-19.4$ (x is pH value ranging from 8 to 12). Furthermore, the corrosion sensitivity of Cu/Al clad plates increases as pH increases in an alkaline solution. Figure 8(b) shows that weight loss increases in the order of $0.01 \text{ M NaOH} < 0.01 \text{ M NaOH}+0.1 \text{ M KNO}_3 < 0.01 \text{ M NaOH}+0.1 \text{ M Na}_2\text{SO}_4 < 0.01 \text{ M NaOH}+0.1 \text{ M NaCl}$. These results confirm that adding Cl^- , SO_4^{2-} , and NO_3^- to the NaOH solution decreases the corrosion resistance of Cu/Al clad plates.

Fig. 8: Weight loss of Cu/Al clad plates under different kinds of solution

3.4 Corrosion mechanism

Figure 9 shows the schematics diagram of the reaction and adsorption process between the matrix and ions during corrosion under different kinds of solution. OH^- participates in both the formation and the dissolution of corrosion products, the corresponding reaction equations are listed as Eqs. (1), (2) and (3) [24].

Although the formation rate of corrosion products was fast, the corrosion products were present in the form of $\text{Al}(\text{OH})_4^-$ at last, which was the most stable compound in the solution. The enhanced chemical film dissolution rate with increasing pH was attributed to both the rapid removal of $\text{Al}(\text{OH})_4^-$ ions from the film/solution interface, and the fast supply of OH^- ions towards the film/solution interface, which resulting in the decrease of the film growth rate and the thinning of the barrier layer film. The equation for the reaction is [25].

Cl^- ions contribute to the dissolution of both the inner film

and the outer film, because the size of Cl^- ions is small, which penetrates the film layer [Fig. 9(b)]. Reaction equations are as follows [26-28].

The formation of the AlCl_4^- is the main reason causing pitting [23]. With the increase of the concentration of Cl^- ions, the AlCl_4^- forms rapidly, thus the pitting occurs more easily.

When SO_4^{2-} ions are present in the alkaline solution, the competitive adsorption between SO_4^{2-} ions and OH^- ions occurs [23].

However, due to the large size of SO_4^{2-} ions, they are only adsorbed on the surface of the materials and are unable to penetrate to the inside of the film layer, as shown in Fig. 9(c).

Fig. 9: Schematic diagram of the reaction and adsorption process between the matrix and ions during corrosion under different kinds of solution: (a) 0.01 M NaOH; (b) 0.01 M NaOH+0.1 M NaCl; (c) 0.01 M NaOH+0.1 M Na₂SO₄; (d) 0.01 M NaOH+0.1 M KNO₃

In the NO_3^- ions containing alkaline solution, $\text{Al(NO}_3)_3$ forms through the incorporation of NO_3^- ions into the surface hydroxide film [Fig. 9(d)], which is then dissolved chemically into the solution as a complex anion of $\text{Al(NO}_3)_4^-$ [23].

The slight denudation is also observed on the surface of the matrix.

4 Conclusions

(1) Because the potential of the Al is lower than that of the Cu, they form a galvanic couple in the corrosion process, in which, Al acts as the anode and Cu acts as the cathode. The corrosion mainly occurs on the Al side, especially the area near the interface of the Cu and the Al.

(2) When $\text{pH} \leq 10$, there are no obvious passivation areas observed on the anodic polarization curves due to the

insufficient supply of OH^- , the local corrosion appears on the matrix. When $\text{pH} \geq 12$, the dissolution of the film layer is faster than the passivation process, and large area of denudation occurs on the surface of the matrix. When $\text{pH} \leq 12$, the relationship between the weight loss and pH is expressed through a linear function: $w = 2.5x - 19.4$ (x ranges from 8 to 12). However, when pH reaches 13, the weight loss increases sharply, and the weight loss of the matrix is 16 times greater than that as pH is 12.

(3) The higher the concentration of Cl^- ions, the more prone the pitting to appear. When only SO_4^{2-} ions exist in the alkaline solution, pitting is observed on the surface of the aluminum, but repassivation appears immediately. Sulfur elements are more easily adsorbed on the surface of the matrix than the chlorine elements and nitrogen elements to form corrosion products. The corrosion products peel off as the corrosion goes on, which accelerates the corrosion rate of the Cu/Al clad plates. With the presence of Cl^- ions, the depth of the corrosion pits of the Cu/Al clad plates are the deepest and the weight loss is the greatest in the alkaline solution.

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