Galvanic corrosion of copper/titanium in aircraft structures using a cyclic wet/dry corrosion test in marine environment by EIS and SECM techniques

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Abstract

Galvanic corrosion behavior between copper and titanium in natural seawater has been studied using wet/dry cycle corrosion test by electrochemical impedance spectroscopy (EIS) and scanning electrochemical microscopy (SECM). The EIS was performed on Cu/Ti galvanic couple in natural seawater after wet/dry cyclic corrosion test. The charge transfer resistance (R_{ct}) and the film resistance (R_f) of Cu/Ti galvanic couple had a higher value due to the formation of corrosion products containing hydroxides and chlorides of copper. The release of Cu^+ and Ti^{2+} into the solution from local anodic surface as well as the consumption of dissolved oxygen at the corresponding cathodic surface was successfully monitored by SECM. SEM analysis confirmed the presence of corrosion products. SEM/EDX analysis showed that Cu and Ti were enriched in corrosion products at the surface of Cu/Ti galvanic couple after corrosion testing. FIB-TEM analysis confirmed that the nanoscale oxide layers containing Cu and Ti were identified in the rust of the Cu/Ti galvanic couple which had a beneficial effect on corrosion resistance of Cu/Ti galvanic couple by making the protective corrosion product in wet/dry cyclic test.

Keywords SECM · EIS · Galvanic corrosion · Copper · Titanium

1 Introduction

While corrosion comes in many forms, the type that aircraft technicians are most interested in is galvanic corrosion. The driving force for galvanic corrosion is the potential difference between two or more metals or alloys in a conductive medium that generates current flow between the anodic and cathodic members [1]. Galvanic corrosion is corrosion between two or more electrically connected different metals originally, where the more active one acts as anode and corrodes, while the less active one is cathode [2]. This will increase the corrosion rate of the anodic metal and reduces that of the cathodic alloy [3]. Galvanic corrosion is very common in municipal infrastructure and industrial and has been studied by many researchers [4].

Copper and its alloy possess high electrical, thermal and mechanical properties. Hence, they are used in desalination plants, heat exchangers, plumbing and transformers. Various studies were reported to control the corrosion in different environments [5–9].

TiO_2 film with few nanometers thickness will form on titanium surface which shows remarkable corrosion resistance. Therefore, copper acts as anode while titanium behaves as cathode in Cu/Ti.

The corrosion process mainly reflects the corrosion of copper in the Cu/Ti couple because of higher resistance of titanium. Cu–Ti possesses higher anticorrosion properties, good electrical and thermal conductance for specific applications [10, 11]. Factors affecting the galvanic corrosion are surface area, conductance, temperature, amount

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of oxygen, extent of potential difference between the dissimilar alloys [12, 13]. It was already reported the corrosion of steel and other alloys in different media [14–19]. SECM is one of the important techniques for analyzing the corrosion because of higher spatial resolution. The corrosion properties of copper/titanium sample have been extensively studied in natural seawater [20–25]. SECM and EIS studies were carried out in natural seawater to monitor the corrosion process. The Cu/Ti galvanic couple was characterized by FE-SEM/EDX, XRD, and TEM/EDX analysis.

It is reported in this paper that the results of experiments designed to follow the corrosion processes occurring at copper and titanium samples directly exposed to natural seawater, the metals being electrically connected as a galvanic couple. No polarization of the samples was performed, thus allowing the systems to evolve spontaneously as it occurs in naturally driven corrosion processes. Experiments were conducted by SECM and EIS in natural seawater. The aim of the present study is to verify that the corrosion of copper electrode can occur in Cu/Ti couple. It is also aimed to monitor the release of Ti^{2+} and Cu^+ ionic species of Cu/Ti. The sample was characterized by FE-SEM and TEM analysis.

2 Experimental procedure

2.1 Preparation of the sample

The experiments were performed on 99.5% pure copper and 99.5% pure titanium, both supplied as sheet of thickness of 1 mm. The dimension of the sample tested is 2 cm × 1 cm. The titanium sample composition in mass% is 0.01Al, 0.05V, 0.01Cr, 0.02Cu, 0.002Fe, 0.01Mn, 0.03Mo, 0.01Nb, 0.01Zr, 0.01Si, 0.05Sn, Ti forms the rest. The chemical composition of copper sample in mass% is 0.01Ag, 0.01Zn, 0.03Co, 0.002Al, 0.02Fe, 0.001Mn, 0.02Ni, 0.01Pb, 0.01Zr, 0.002P, 0.01Sn, Cu forms the rest. The sheets were cut and mounted into an epoxy resin sleeve, so that only a square area of 1 cm² formed the testing metal substrate. Moreover, the dimension of the separation between Cu and Ti placed in the epoxy resin is found to be around 5 µm. For the galvanic couple experiments, two electrodes were embedded in the resin and connected electrically at the back. The mount with the samples was then polished with silicon carbide paper down to 1200 grit, washed thoroughly with distilled water, dried with acetone and finally placed in the electrochemical cell. Testing was carried out in natural seawater collected from the Ennore beach of Indian Ocean, Chennai in Tamil Nadu, India. The solution was naturally aerated and experiments were conducted at room temperature.

2.2 EIS and SECM measurements

A wet/dry cyclic test was conducted under the condition (12 h immersion in natural seawater and 12 h in the dry) for 15 days. The EIS measurements were taken periodically in natural seawater for electrochemical characterization. The EIS was performed in a conventional three-electrode cell, using Cu/Ti galvanic couple as the working electrode and saturated calomel electrode (SCE) as the reference electrode. A frequency response analyzer was used for EIS measurements with amplitude of 10 mV over a frequency range of 40 kHz to 1 mHz. All measurements were carried out at the open circuit potential at room temperature (25 °C). The EIS experimental data were analyzed using curve fittings.

The Scanning Electrochemical Microscope consisted of model CHI900 (CH-Instruments, Texas, USA) and operated with a diameter of 10 µm platinum microelectrodes as SECM tips. The movement of the tip was controlled in the x, y and z directions using optically encoded inch-worm piezo motors. A bipotentiostat designed for SECM was used to control the potentials of the sample and tip separately. A video microscope mounted above the cell was used to aid in positioning the microelectrode over the sample. All measurements were performed using an Ag/AgCl reference electrode and a platinum wire as a counter electrode. The copper–titanium galvanic couple sample was mounted horizontally at the bottom of flat cell. The sample of galvanic couple was examined at the OCP for all SECM experiments. A line scan was observed across the galvanic couple area in the sample at a constant height of 50 µm from the surface at a scan rate of 20 µm s^{−1} in the x direction. The platinum tip was scanned at a constant height above the sample while all experiments were performed.

2.3 Surface analysis

The surface state of the corrosion product on the Cu/Ti galvanic couple was observed by FE-SEM and FIB-TEM (Focused Ion Beam-Transmission Electron Microscope) analysis. After the cyclic corrosion test, the Cu/Ti galvanic couple was cast in resin and polished using emery paper, followed by diamond paste. Carbon was then evaporated on the sample in order to compensate for charging effects. A cross section of the rusted steel was examined using FE-SEM at an acceleration voltage of 20 kV and irradiation current of 10 µA. The quantity of Cu, Ti and O in the rust was measured by EDX (energy dispersive X-ray) analysis. TEM observation was performed with EDX analysis. The rust was cut from the inner rust by FIB.

Then, the line profile analysis was carried out in order to identify the quantity of oxides of Cu and Ti in the rust.

The crystalline nature of corrosion products formed at the Cu/Ti couple in natural seawater for 15 day was analyzed with X-ray diffractometer (XRD; Bruker model D8, Germany) using Cu K α radiation λ -1.5406 Å. The 2 θ angles were swept from 10° to 100° in steps of one degree.

3 Results and discussion

3.1 Electrochemical impedance spectroscopy (EIS)

Figure 1 shows the typical EIS plots of Cu/Ti galvanic couple in natural seawater. The Cu/Ti galvanic couple corrosion behavior is investigated by EIS in order to elucidate the influence of titanium on the corrosion of copper. A stable thin oxide film of TiO₂ with few nanometers thickness

on titanium surface shows remarkable corrosion resistance. Therefore, Cu acts as anode while Ti acts as cathode in Cu/Ti galvanic couple. The characteristics of galvanic couple corrosion mainly reflect the corrosion of copper because the Titanium has a higher corrosion resistance compared with Cu.

It can be seen from the EIS plots that two time constants are needed to fit the EIS plots with the increase of wet/dry cyclic corrosion time. This is due to the fact that more and more corrosion products accumulated on the matrix surface, which leads to a new double electrode layer and displays as another time constant. The equivalent electrical circuit shown in Fig. 2 was used to fit the EIS plots by using Z-view software and the fitted results are listed in Table 1. The impedance of CPE is defined as the following equation [26]:

$$Z_{CPE} = 1/Y(j\omega)^n \quad (1)$$

where Y is the modulus, j is an imaginary unit, ω is the angular frequency, and n is the CPE exponent ($-1 \leq n \leq 1$). CPE describes an ideal inductor for $n = -1$, an ideal resistor for $n = 0$, and an ideal capacitor for $n = 1$ [27]. The “ n ” value in CPE is an indication of surface roughness; the lower value of it indicates the rougher or more heterogeneous surface. Therefore, the oxidation of the Cu side, as well as the dissolution of Cu/Ti intermetallic phases in the intersection of Cu/Ti couple, leads to roughening of the surface on the Cu side and at the joint interface. R_s is the solution resistance, CPE_{dl} is the double layer capacitance, R_{ct} is charge transfer resistance, CPE_f is the capacitance of outer passivating film and corrosion product layer, R_f is the resistance of outer passivating film and corrosion products.

As the wet/dry cyclic corrosion test time increases, more corrosion products will accumulate on the metal surface, which will block the pathways of O₂ or Cl⁻ from corrosive

Fig. 1 EIS behavior of Cu/Ti galvanic couple after wet/dry cycles test in marine environment for various days

Fig. 2 Equivalent circuit diagram for Cu/Ti galvanic couple after wet/dry corrosion test in marine environment

Table 1 The fitted results of EIS using equivalent circuit

Time/d	R_s (Ω)	R_f (Ω)	CPE_f (μF)	n_f	R_{ct} (Ω)	CPE_{dl} (μF)	n_{dl}
1	65.25	13,985	39.45	0.7162	30,562	142.50	0.8912
3	67.90	11,945	48.27	0.6924	24,865	320.45	0.8825
6	69.46	9994	96.36	0.6655	21,998	449.91	0.8714
9	72.38	8518	111.69	0.6211	18,867	612.65	0.8555
12	75.85	7046	115.22	0.6105	16,545	761.34	0.8467
15	79.15	6035	124.48	0.6065	15,652	980.85	0.8358

medium to metal surfaces. The accumulated corrosion products on the metal surface will crack due to the limited space of the metal surface. This results in the increase of the corrosion area (S) and decrease of R_{ct} and R_f and subsequently enhances the corrosion rate. Therefore, the formation of corrosion products will simultaneously change the area of the corroding surface, the resistances of passivating film and charge transfer. This subsequently causes the fluctuations on both CPE_f , CPE_{dl} and the corrosion rate. The film resistance, R_f of galvanic couple is decreased from $1.37 \times 10^4 \Omega \text{ cm}^2$ at 1 day to $7.51 \times 10^3 \Omega \text{ cm}^2$ at 15 day. The charge transfer resistance, R_{ct} is also decreased from $2.95 \times 10^4 \Omega \text{ cm}^2$ at 1 day to $1.57 \times 10^4 \Omega \text{ cm}^2$ at 15 day with increase in wet/dry cyclic corrosion tests. It can be shown that the potential difference between the Cu/Ti couple metals accelerates the corrosion process of Cu, especially the corrosion rate of the area adjacent to Ti, which consequently causes the fast increase of the Cu area exposed to the solutions and the larger value of CPE_f .

The potential difference between their components can initially partly hinder the surface thermodynamic refining actions, accelerate the diffusion of aggressive Cl^- from the natural seawater to the corrosion active points on Cu and initiate pitting corrosion. This subsequently results in the decrease of R_f .

3.2 Scanning electrochemical microscopy (SECM)

Figure 3 shows the SECM images of the Cu/Ti couple immersed in natural seawater for different wet/dry cyclic corrosion test at -0.70 V . The variation in color in the SECM images explains the corrosion behavior that represents the local anodic and cathodic area in the galvanic couple. The tip current measured at the couple and along the titanium surface is much smaller (-1.0 nA) than over the copper (-5.1 nA). This is due to the consumption of dissolved oxygen at the titanium surface. The quantity of oxygen is decreased by the consumption of dissolved oxygen as

Fig. 3 SECM topographic images obtained near the Cu/Ti couple at the tip potential of -0.70 V in natural seawater by wet/dry cycles test at 1 day, 3 day, 6 day, 9 day, 12 day and 15 day

the cathodic reaction occurs at the titanium surface due to the occurrence of anodic dissolution of Cu^+ at the copper surface. It can be shown from the figure that the corrosion activity in the copper increases rapidly than that of titanium. The tip current decreased at the couple with increasing wet/dry cyclic corrosion test, which is shown by increase of red color. Figure 4 represents the currents of O_2 reduction measured with different wet/dry cyclic corrosion test above the Cu/Ti electrode at -0.70 V. The

Fig. 4 Line profile diagram of Cu/Ti couple in natural seawater at different days at the potential of -0.70 V

amount of oxygen dissolved in solution decreases when exposure time increases, as a result of its consumption in the cathodic reaction. The cathodic current decreases from -4.2 nA at 1 day to -1.0 nA at 15 day. The gradient of oxygen quantity reaches the entire electrolyte depth, in spite of some further dissolution of oxygen from the air, and consequently the average quantity decreases with time [26].

Figure 5 presents the SECM images of Cu–Ti couple at $+0.34$ V at different cyclic wet/dry corrosion tests. The current measured at copper surface is significantly higher than that of titanium surface. This is due to the anodic dissolution of copper surface. The tip current measured at copper surface decreases from 8.1 nA at 1 day (light blue) to 3.7 nA at 15 day (greenish yellow). This shows that the dissolution of copper decreases with increase in test time due to decrease in the oxidation of copper. This is due to the formation of corrosion products containing hydroxides and chlorides of copper at the metal surface, which hinders the further oxidation of the metal. This results in the decrease in volume of the electrolyte. As the number of cyclic corrosion tests increases, the decrease in the tip current at the couple and along the copper surface is significantly seen. Figure 6 shows the steady-state currents monitored at the UME polarized at $+0.34$ V at different cyclic wet/dry corrosion tests. The anodic current at copper surface decreases slowly from 7.9 nA at 1 day to 3.6 nA at 15 day. The decrease in the anodic current with increase in cyclic

Fig. 5 SECM topographic images obtained near the Cu/Ti couple at $+0.34$ V under wet/dry cycles in natural seawater at 1 day, 3 day, 6 day, 9 day, 12 day and 15 day

Fig. 6 Line profile diagram for Cu^{2+} obtained above the Cu/Ti couple at the tip potential of +0.34 V in natural seawater at different days (Wet and Dry cycles)

corrosion test is due to the formation of corrosion products which prevent further oxidation of the electrodes. The quantity of Cu^+ ions slowly decreased with increase in cyclic wet/dry corrosion tests. This behaviour is due to the formation of corrosion products which hinder the dissolution of copper as Cu^+ ions from metal into

solution. It is also due to the sacrificial action of copper electrode, which prevents the release of Ti^{2+} ions from the metal into the solution.

Figures 7 and 8 present the SECM images and line scan analysis of Cu-Ti couple at +0.51 V at different cyclic wet/dry corrosion tests, respectively. It can be seen from the Figs. 7 and 8 that the dissolution of titanium decreases as the test time increases. This is due to the formation of corrosion products at the metal surface, which hinders the further oxidation of the metal. This results in the decrease in volume of the electrolyte [27]. The decrease in the anodic current with increase in cyclic corrosion test is due to the formation of corrosion products which prevent further oxidation of the electrodes. The galvanic coupling of Cu with Ti produces decrease in the dissolution rate of copper and titanium since the precipitation of corrosion products occurs on this metal. Therefore, the surface is blocked by the corrosion products and the corrosion reaction slows down. The titanium surface is protected due to the sacrificial action of copper whose corrosion products prevent the dissolution of titanium. It is evident that the anodic dissolution of copper takes place at the copper surface [28]. As the W/D cyclic corrosion test increases, the anodic dissolution of copper decreases due to the formation of the corrosion products. The formation of corrosion products on the surface of copper prevents the further

Fig. 7 SECM topographic images obtained near the Cu/Ti couple at +0.51 V under wet/dry cycles in natural seawater of at 1 day, 3 day, 6 day, 9 day, 12 day and 15 day

Fig. 8 Line profile diagram for titanium obtained above the Cu/Ti couple at the tip potential of +0.51 V in natural seawater at different days (wet and dry cycles)

oxidation of copper. The corrosion products also hinder the oxidation of titanium surface due to the sacrificial action of copper.

3.3 Field emission-scanning electron microscopy (FE-SEM)

Figure 9 shows the SEM image of the corrosion products formed on the tested samples in the natural seawater

Fig. 9 SEM image of the corrosion products formed on the Cu/Ti couple after wet and dry cyclic corrosion test

after wet/dry cyclic corrosion tests. It can be observed that a dense and continuous corrosion product layer precipitate at the couple and along the copper surface. The characterization of galvanic couple mainly reflects the copper anode side because the titanium cathode side has a higher corrosion resistance compared to copper. It can be seen from the figure that the potential difference between the Cu/Ti couple metals accelerates the corrosion process of Cu, especially the corrosion rate of the area adjacent to Ti, which consequently causes the fast increase of the Cu area exposed to the solutions. In comparison, it can be seen that the corrosion of Ti is extremely slight, and its main corrosion type is pitting. It can be seen that the corrosion rate of copper is much higher than the titanium. The rate of corrosion is slowly decreased as the test time increased due to the formation of corrosion products on the metal surface. SEM was used in order to identify the corrosion products of Cu/Ti galvanic couple after 15 days of wet/dry cyclic corrosion test. Figure 10 shows the EDX results for the Cu/Ti galvanic couple. EDX analysis confirms the presence of Cu and Ti in the inner rust of Cu/Ti galvanic couple. This is due to the formation of complex oxides containing Cu and Ti during corrosion test. This result implies that the complex oxides of copper containing hydroxides and chlorides are formed during the cyclic corrosion test, which can enhance the barrier properties of the Cu/Ti galvanic couple.

Fig. 10 EDX mapping of the corrosion products formed the Cu/Ti couple after wet and dry cyclic corrosion test

3.4 X-ray diffraction studies (XRD)

The XRD analysis of the Cu/Ti galvanic couple exhibits only Cu and Ti peaks, as shown in Fig. 11. However, the intensity of copper peaks is much higher than that of the titanium peaks. Therefore, it is confirmed that the corrosion rate of copper is much higher than that of titanium. This result is in consistent with EIS and SECM studies.

3.5 TEM analysis

TEM observation was conducted in order to investigate the nanostructure of the rust formed on the Cu/Ti galvanic couple as shown in Fig. 12. The inner rust was cut by FIB (focused ion beam), then employed to TEM. It was

Fig. 11 XRD analysis of corrosion products formed the Cu/Ti couple after 15 days of wet and dry cyclic corrosion test

(b)

Fig. 12 a Bright field image and **b** line profile of EDX according to the spots in **a** for the Cu/Ti galvanic couple in natural seawater (after wet/dry cycles test)

possible to select the rust containing oxides of Cu and Ti by EDX analysis. The micrograph of Fig. 12a depicts the spot position of analysis. Figure 12b shows the elemental composition of Cu, Ti and Oxygen in mass% corresponding to each spot in Fig. 12a. This figure clearly indicates an enrichment of oxides containing Cu and Ti. Therefore, it is concluded that nanoscale layers containing oxides of

Cu and Ti were formed in the rust and these nanoscale layers could increase the corrosion resistance of the Cu/Ti galvanic couple.

3.6 Corrosion mechanism

The main reactions responsible for corrosion of copper and titanium in natural seawater at neutral pH are reduction of dissolved oxygen and of water at the cathode,

whereas, at the anode the oxidation of metal takes place, either

or

By setting the tip potential to +0.34 V versus Ag/AgCl/KCl(sat.), the oxidation of the Cu⁺ can be detected through their oxidation to Cu²⁺ at the UME.

On the other hand, by setting the tip potential to +0.51 V versus Ag/AgCl/KCl(sat.), the oxidation of the Ti²⁺ can be detected through their oxidation to Ti⁴⁺ at the UME.

The resulting Cu⁺ ions further react with corrosive ions to form CuCl₂[−] and Cu₂O, which is oxidized to the atacamite or the isomorphous phase paratacamite [Cu₂(OH)₃Cl], which has minor protective characteristics as shown in the following equations [29].

Since no ions were originally present in the electrolyte, they can only originate from corrosion processes at the metal directly exposed to the aqueous environment. In this way, soluble Cu⁺ ions would diffuse away in the electrolyte and will be eventually detected at the tip through their reduction. It must be noticed that the electrons released by copper will be consumed by soluble oxygen in the cathodic half-reaction. As a result of these

reactions, depletion of oxygen will occur near the cathode and accumulation of metal cations will occur near the anode. The cathodic and anodic activities were well separated and located on titanium and copper respectively, as anticipated in a galvanic couple with copper oxidizing sacrificially and preventing the corrosion of titanium. The quantity of dissolved molecular oxygen was sensed by applying a constant potential of −0.70 V to the SECM tip and by scanning it over the surface. The quantity of cuprous ions was detected by applying a constant potential of +0.34 V to the SECM tip whereas the quantity of Ti²⁺ ions was detected by applying a constant potential of +0.51 V to the SECM-tip. SECM-image operating at the oxygen-reduction potential showed a difference in the current values across the transition between copper and titanium surfaces.

4 Conclusion

It has been shown that the corrosion processes related to copper dissolution that take place in an Cu/Ti galvanic couple exposed to natural seawater were investigated by scanning electrochemical microscopy (SECM) using a platinum ultramicroelectrode as SECM tip and electrochemical impedance spectroscopy (EIS). The tip current measured at the potential of −0.70 V decreased with increase in wet/dry cyclic corrosion test. This was due to the consumption of dissolved oxygen at the titanium surface. The quantity of Cu⁺ emanating from the dissolving copper electrode was determined by wet/dry cyclic corrosion test at the tip potential of +0.34 V. The anodic current decreased as the W/D cyclic test increased. This was due to the formation of corrosion products which prevent further oxidation of the electrodes. On the other hand, the quantity of Ti²⁺ ions was found to be insignificant by applying the potential of +0.51 V at the SECM tip. The tip current was much smaller than that of copper. This behaviour was due to the sacrificial action of copper electrode which prevents the release of Ti²⁺ ions from the metal into the solution. The charge transfer resistance (R_{ct}) and the film resistance (R_f) of Cu/Ti couple had a higher value after 1 day wet/dry cyclic corrosion test time and the value decreased slowly with increase in wet/dry cyclic corrosion test time. SEM/EDX and TEM/EDX analysis showed that Cu and Ti were enriched in corrosion products after wet/dry cyclic corrosion testing.

Compliance with ethical standards

Conflict of interest There is no conflict of interest.

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