Effect of heat flux on metal corrosion in non-boiling media: testing system, heat transfer simulations, and corrosion study on Al alloy

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ABSTRACT
A dual-circulation testing system was constructed to study the effect of heat flux density ($q$, $-22.28 \sim +22.54$ kW m$^{-2}$) on the corrosion of an Al–Cu–Mg–Mn alloy in 0.5 M H$_2$SO$_4$ at a constant metal surface temperature (50°C) under different controlling heat transfer states. Their heat transfer parameters were determined quantitatively by COMSOL simulation to keep them except $q$ in constant, highlighting the influence of $q$ on metal corrosion. Compared with the $q = 0$ condition, the positive heat flux ($q > 0$) improved the surface layer performance of the alloy to inhibit its anodic corrosion process, decreasing its corrosion current density ($i_{corr}$) by 56% with increasing $q$ to $22.54$ kW m$^{-2}$. While $q < 0$, the opposite effect occurred, increasing its $i_{corr}$ by 52% at $q = -22.28$ kW m$^{-2}$. Heat flux also changed the corrosion reaction’s apparent effective activation energy ($E_a$) and pre-factor, and $E_a$ played a dominating role in changing $i_{corr}$.

Introduction
Heat exchangers play an essential role in various industrial processes, which transfer heat energy from one fluid to another through heat transfer surfaces [1]. These fluids may be corrosive to the metallic heat exchangers, producing corrosion products to cause additional thermal resistance [2–5] and damage their thermal performance [6,7]. Moreover, the corrosion failure of heat exchangers also leads to significant economic losses and potential security threats [8]. Therefore, it is necessary to pay attention to the corrosion behaviour of metals under heat transfer conditions.

In a usual corrosion system, metal’s corrosion behaviour is generally studied under isothermal conditions, i.e. the metal and test solution is in thermal equilibrium, and there is no temperature difference in the test solution. Under this condition, the effect of temperature on corrosion has been extensively studied in different media [9–11] to verify its critical influence on corrosion. However, in a heat transfer system, a thin thermal boundary layer (or temperature boundary layer) will occur in the fluid medium adjacent to the metal surface because of the heat flux flowing across the metal/fluid medium interface, among which there is a significant temperature difference in the fluid medium to conduct heat across the interface [12,13]. Under this condition, the heat flux may change the metal’s surface temperature ($T_M$), so we must keep a constant $T_M$ to investigate its effect on metal corrosion. Unfortunately, it is challenging to alter heat flux but maintain a constant $T_M$ in the test system. In this case, the heat flux’s influence on metal’s corrosion behaviour is unclear until now.

Ross [14] pointed out that the essential factors influencing the metal corrosion of heat exchangers included the direction of the temperature gradient at the metal/liquid interface, the nature of the fluid (gas or liquid), and the velocity profiles in corrosive fluids. These factors determined $T_M$, the solubility of corrosion products, and the concentration distribution of corrosive species adjacent to the metal surface, e.g. dissolved oxygen (DO), which affects the corrosion product film and passivation film on metals. Willem Faes et al. [15] also reviewed corrosion and corrosion prevention in heat exchangers and gave similar perspectives. Nevertheless, they did not mention the effect of heat flux on metal corrosion and related mechanisms.

Most of the research on the corrosion of heat exchangers concerned the corrosion behaviour of metals [16–19] and the corrosion prevention effect of inhibitors [20,21] or other protection methods [22–24] under specific conditions encountered in the practical fields. In these studies, the effects of heat flux and $T_M$ were usually combined, ignoring the effect of heat flux. Some works tried to separate their effects and clarify the influence of heat flux on corrosion. T. Prosek et al. [25] studied the initiation and expansion of pitting corrosion of austenitic stainless steel at a constant $T_M$ of 60°C with heat flux density ($q$) from $-15$ to $+74$ kW m$^{-2}$. The negative $q$ means the heat flux flowing from solution to metal, i.e. $T_M < T_{solution}$. In contrast, the positive $q$ denotes the opposite heat flux direction and $T_M > T_{solution}$. They found that the positive heat flux led to increased resistance to pitting corrosion, ascribed to the improvement of the passive film’s protective ability with more oxygen available on the metal surface. They also studied the influence of $q$ (0–42 kW m$^{-2}$) and $T_M$ (30–90°C) on the intergranular corrosion (IGC) of stainless steel under non-boiling conditions. They found that the effect of $q$ on IGC may be smaller than $T_M$ [26]. Andon et al. [27] used novel test rigs to study stainless steel corrosion in nitric acid under controlled $q$ and $T_M$. Their results showed that the more positive $q$ conditions (i.e.
q > 0) resulted in a slightly lower corrosion rate than those under isothermal conditions. These works focused on the local corrosion of stainless steel and generally separated the effects of q and T_M. However, the other heat transfer parameters in the metal/solution interface were not controlled, e.g., surface temperature distribution, the thickness of the thermal boundary layer (δ), and the metal surface’s solution flow rate, which also affect metal corrosion. Thus, it is necessary to control these parameters quantitatively to determine the heat transfer state of the metal/solution interface under heat exchanger conditions.

It is not easy to test these parameters directly in a natural heat transfer system, so we try to solve them using COMSOL Multiphysics, a widely used numerical simulation software based on the finite element method in scientific research [28]. The heat flux, temperature field and distribution, fluid flow, and thermal behaviour in the heat transfer systems can be simulated and modelled using this software [29–33]. Therefore, we can control some heat transfer conditions only with the change in q values to investigate its influence on metal corrosion.

This study constructed a dual-circulation testing system to investigate the effect of heat flux on the corrosion of an Al–Cu–Mg–Mn alloy in a 0.5 M H_2SO_4 solution under controlling heat transfer states. The test system’s heat transfer state was determined using COMSOL simulation to obtain q, T_M, δ, the temperature distribution in the thermal boundary layer, and the solution flow rate distribution near the metal surface. Under the different controlled heat transfer states, all the above heat transfer parameters except q are constant. Thus, the effect of q (−22.28–22.54 kW m⁻²) on the corrosion of the Al alloy in a 0.5 M H_2SO_4 solution at a constant T_M of 50°C was investigated using potentiodynamic polarisation, electrochemical impedance spectroscopy (EIS), and weight loss tests in the constructed test system. The corroded test samples were characterised using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and 3D optical microscope analysis. The involved mechanisms were discussed to improve understanding of the heat flux’s effect on metal corrosion in non-boiling media. Moreover, this study will provide a credible method to support further study in heat transfer systems.

**Experimental**

**Testing system**

Figure 1 presents the schematics of the constructed testing system, including two circulatory systems for deionised water and the test solution, respectively. The cylindrical water chamber (80.4 cm³) and the spherical solution chamber (69.6 cm³) were made of polytetrafluoroethylene (PTFE) and glass, respectively. The metal test sample (Φ20 × 4 mm) was placed in a reserved slot on the centre of the end surface of the water chamber and sealed with two O-ring gaskets (Φ14 mm) on its two surfaces to form a working area of 1.54 cm² by pressing the metal fixture (Figure 1).

The two glass containers with deionised water and test solution were placed in two thermostat water baths to control their temperature separately. Deionised water and test solution at controlling temperature were pumped into the water and solution chambers and circulated in the two circulatory systems. Two flow metres were used to control their inlet flow rates (Q, precision ± 1.0 L·h⁻¹). The thermocouples (precision ± 0.1°C) were used to measure the temperature of deionised water, test solution, and metal sample side surface (T_M, measure) from a hole in the water chamber end side (Figure 1). A reference electrode with a glass salt bridge and a counter electrode was placed in the solution chamber for electrochemical tests. By changing the temperature of deionised water in the water chamber (T_water), T_solution in the solution chamber, and the inlet Q values, we can obtain different heat transfer conditions in the test system. Generally, the test system can reach a relatively stable controlling condition within 10 min.

**Materials and test solution**

A commercial Al–Cu–Mg–Mn alloy was used in this study, whose chemical composition is (wt-%): Cu 4.26, Mg 1.48, Mn 0.69, Fe 0.13, Si 0.041, Ti 0.025, Ga 0.015, and Al balance. Before the test, the test samples were grounded with SiC paper from 400° to 1000°, then ultrasonically cleaned in distilled water, rinsed with acetone, and dried in atmospheric conditions. Sulphuric acid (98 wt-%) in analytical grade and deionised water was used to prepare a 0.5 M H_2SO_4 solution as the test media.

**Electrochemical tests**

All the electrochemical tests were conducted with a CS 350 Corrtest electrochemical workstation. The treated test sample (Φ20 × 4 mm) sealed between the two chambers was used as the working electrode (working area 1.54 cm²). A platinum (Pt) plate electrode (2.0 cm²) and a saturated calomel electrode (SCE) were used as the counter electrode and reference electrode. Before each measurement, the test system ran for 2 h to ensure the working electrode’s steady heat transfer state and free corrosion potential (E_corr). The polarisation curves were measured in the potential range of ±150 mV vs. E_corr at a scan rate of 0.5 mV s⁻¹. The EIS measurements were performed at E_corr with the AC voltage amplitude ±10 mV in 10 kHz–0.01 Hz. All the EIS data were fitted using Zview2.0 software. All the electrochemical tests under different heat transfer conditions were repeated more than three times.

**Weight loss test**

The treated test sample (Φ20 × 4 mm) was accurately weighed (precision ±0.1 mg) and then placed in the test system under different heat transfer conditions for 5 h. After that, the specimen was taken out, brushed with running water to remove corrosion products, rinsed with distilled water and acetone, and finally dried and weighed again to obtain its weight loss data. The test sample’s weight loss mainly comes from its corrosion in the test solution (i.e. 0.5 M H_2SO_4) [34,35]; thus, the corrosion rate (P_w, mm·y⁻¹) of the test sample can be calculated via the following equation [36]:

\[
P_w = 87.6 \times \frac{\Delta W}{S \times t \times \rho}
\]

where ΔW is the weight loss (mg), S is the exposed area of the test sample (1.54 cm²), ρ is the sample density (2.68 g cm⁻³),
and \( t \) is the immersion time (5.0 h). All the weight loss tests under different heat transfer conditions were repeated more than three times.

**Surface characterisation**

The surface morphologies of the Al alloy specimens after corroding under different heat transfer conditions were observed by a 3D optical microscope (KEYENCE VHE-1000) and SEM (Nova NanoSEM 450) with EDX (EDAX-Genesis). Corrosion products on the test samples were analyzed by XRD (SmartLab-SE) using Cu Kα radiation and XPS (ESCALAB 250Xi). The XRD pattern was analyzed with Xadata 4.1 software. The XPS spectrum was analyzed with XPS peak 4.1 software.

**Results**

**Determining the heat transfer states by COMSOL simulation**

A comprehensive numerical model for continuous liquid flow heat transfer was established in COMSOL by iteratively coupling the heat transfer of Newtonian fluids and fluid flow. In this model, the fluid dynamics module (for solving momentum and continuity equations) and the heat transfer module (for solving energy balance equations) were iteratively coupled to study Newtonian liquid and heat transfer metal’s temperature distribution. The heat transfer module obtained the flowing liquid’s velocity distribution by solving the Navier–Stokes equation \([37]\). Therefore, this model can simulate the test system’s temperature field to determine the interface temperature, heat flux, and other concerned parameters.

A simplified geometric structure of the test system was generated in COMSOL to facilitate simulation calculations, as shown in Figure S1a (Supporting information, SI). Two physical fields of solid heat transfer and laminar flow were added in sequence, coupled using the flow coupling in the multiphysics field. Since laminar flow unilaterally affected solid heat transfer, the laminar flow was used as the source in the coupling interface and solid heat transfer as the target. The computational domain was meshed using free triangle meshes, as shown in Figure S1b (SI). The transient solver was used to solve the constructed model.

Different heat transfer conditions can be formed by controlling different \( T_{\text{water}} \) and \( T_{\text{solution}} \) denoted as their temperature difference in this paper. For example, \( \Delta T_{70-30} \) denotes the positive heat flux condition with \( T_{\text{water}} = 70°C \) and \( T_{\text{solution}} = 30°C \); while \( \Delta T_{30-70} \) denotes the negative heat flux condition with \( T_{\text{water}} = 30°C \) and \( T_{\text{solution}} = 70°C \). Different heat transfer conditions were designed and simulated using the above model in COMSOL to determine their heat transfer states.

Figure 2 presents the test system’s critical simulation results to determine the controlling heat transfer states used for the subsequent corrosion study. Because of the influence of the fluid’s inlet \( Q \) on the heat transfer process \([38]\), the temperature distribution in the test solution with different \( Q \) at a stable heat transfer state was investigated, as typically shown in Figure 2(a). In this study, the inlet \( Q \) of the test solution and deionised water were equal. When inlet \( Q = 5 \text{ L h}^{-1} \), there was \( T = T_{\text{solution}} \) at \( d \geq 1.2 \text{ mm} \) (Figure 2(a)), so the thickness of the thermal boundary layer in the test solution \( \delta_{\text{solution}} \) could be determined as 1.2 mm. Similarly, when inlet \( Q = 20, 40, \) and \( 60 \text{ L h}^{-1} \), the \( \delta_{\text{solution}} \) values were 0.9, 0.7, and 0.6 mm, respectively, indicating that \( \delta_{\text{solution}} \) decreases with increasing inlet \( Q \) to accelerate the heat conduction \([39]\). Because of the slight decrease in \( \delta_{\text{solution}} \) when inlet \( Q > 20 \text{ L h}^{-1} \), we selected the inlet \( Q = 20 \text{ L h}^{-1} \) in this study.

Figure 2(b) presents the test solution’s temperature distribution with inlet \( Q = 20 \text{ L h}^{-1} \) to verify the possible change of \( \delta_{\text{solution}} \) under different heat transfer conditions. The
δ_solution value in Figure 2(b) was nearly the same (0.9 mm), suggesting a stable δ_solution under different heat transfer conditions. In addition, Figure 2(b) also shows that the centre temperature of the sample surface (T_{M, centre}) in the test solution is around 50°C under different heat transfer conditions.

Figure 2(c–d) presents the typical T_{M, centre} – t curve in the test solution, and the streamlined diagram of fluid velocity (u, m·s^{-1}) distribution at ΔT_{70-30} and inlet Q = 20 L·h^{-1}. The T_{M, centre} of the test sample reached 50.09°C at 10 min and then maintained at a relatively stable value (50 ± 0.2°C) (Figure 2(c)), implying a relatively stable heat transfer state. In this case, the test system needs about 10 min to reach a stable heat transfer state. Figure 2(d) indicates that the stable test solution’s flow velocity (u_solution) near the test sample surface was very slow and stable in the test system, e.g. at d = 0.01 and 0.90 mm, there was u = 0.0019 and 0.017 m·s^{-1}, respectively.

Figure 3 shows the stable temperature distribution on the test sample surface in the test solution under different heat conditions of (a) ΔT_{70-30}, (b) ΔT_{60-40}, (c) ΔT_{50-50}, (d) ΔT_{40-60}, (e) ΔT_{30-70} with inlet Q = 20 L·h^{-1}.
transfer conditions with inlet \( Q = 20 \text{ L h}^{-1} \). According to Figure 3, the \( T_M \) values were around 50°C, and its deviation was within 0.9°C and decreased with the temperature difference across the metal/solution interface. In this case, we can generally consider that the \( T_M \) is uniformly distributed at 50°C.

Figure S2 (SI) gives a general view of the stable temperature distribution in the test system at \( \Delta T_{70-30} \) and inlet \( Q = 20 \text{ L h}^{-1} \). The \( T_M \), centre in the water and solution chambers was 50.71 and 50.17°C, respectively, displaying a slight temperature difference (0.54°C) across the test sample due to the excellent heat transfer performance of the Al alloy. The heat flux density (\( q \)) was calculated as 22.28 kW m\(^{-2}\). Similarly, the \( q \) values can also be calculated under other heat transfer conditions.

Based on the above simulation, Table 1 presents five different heat transfer conditions in this study, denoted as \( \Delta T_{70-30} \), \( \Delta T_{60-40} \), \( \Delta T_{50-50} \), \( \Delta T_{40-60} \), and \( \Delta T_{30-70} \), and the corresponding heat transfer parameters with inlet \( Q = 20 \text{ L h}^{-1} \). In Table 1, the \( T_M \) values were 50 ± 0.5°C under all conditions, while their simulation results were 50 ± 0.4°C (see Figure 3), indicating a good consistency between them. In summary, under different heat transfer conditions in Table 1, because \( \delta_{\text{solution}} \) (Figure 2(b)), \( u_{\text{solution}} \) (Figure 2(d)), and \( T_{\text{sol}} \) (Figure 3) are controlled as constant with a stable inlet \( Q \), the influence of \( q \) on the test sample's corrosion can be highlighted in the test system. Thus, the effect of \( q \) on the corrosion of the Al–Cu–Mg–Mn alloy in 0.5 M H\(_2\)SO\(_4\) was investigated under these controlled heat transfer states (Table 1).

**Effect of heat flux on the corrosion of the Al alloy**

**Polarisation behaviour**

Figure 4 presents the typical polarisation curves of the Al–Cu–Mg–Mn alloy in 0.5 M H\(_2\)SO\(_4\) under different heat transfer conditions (\( t = 2 \text{ h} \)). All the polarisation curves in Figure 4 display typical features of activation-controlled corrosion processes similar to those in the acid media (HCl and HCl + H\(_2\)SO\(_4\)) of Al alloys [40–42], suggesting that heat flux does not change the corrosion mechanism in the test system. Compared with the thermal equilibrium condition (\( \Delta T_{50-50} = 0 \)), at \( \Delta T_{70-30} \) and \( \Delta T_{60-40} \) \( q \) > 0), \( E_{\text{corr}} \) moved positively, and anodic polarisation current density \( (I_a) \) decreased obviously; while at \( \Delta T_{30-70} \) and \( \Delta T_{40-60} \) \( q < 0 \), \( E_{\text{corr}} \) moved negatively, and \( I_a \) increased obviously. The cathodic polarisation curves did not change significantly under different conditions (Figure 4). These results imply that heat flux primarily influences the anodic process on the Al alloy specimen. The positive heat flux inhibits it while the negative heat flux accelerates it, resulting in the above changes in polarisation curves.

The polarisation curves in Figure 4 were fitted using the data in the weak polarisation region to obtain \( E_{\text{corr}} \), corrosion current density \( (i_{\text{corr}}, \text{mA cm}^{-2}) \), anodic Tafel slope \( (b_a) \), and cathodic Tafel slope \( (b_c) \), as shown in Table 2. The general corrosion rate \( R_i \left( \text{mm y}^{-1} \right) \) was calculated using Equation (2):

\[
P_i = 1.09 \times \frac{M}{\rho} i_{\text{corr}} \tag{2}
\]

where \( M \) is the atomic weight of Al (26.98 g mol\(^{-1}\)), and \( \rho \) is the density of the Al alloy (2.68 g cm\(^{-3}\)). Furthermore, \( \eta_i(\%) \) is defined as Equation (3) to show the change percentage of corrosion rate under different heat transfer conditions:

\[
\eta_i(\%) = \left( 1 - \frac{i_{\text{corr}}}{i_{\text{corr}}^0} \right) \times 100 \tag{3}
\]

where \( i_{\text{corr}}^0 \) is the \( i_{\text{corr}} \) value when \( q = 0 \).

According to the results in Table 2, the order of \( i_{\text{corr}} \) under different heat transfer conditions is \( \Delta T_{70-30} < \Delta T_{60-40} < \Delta T_{50-50} < \Delta T_{40-60} < \Delta T_{30-70} \). At a constant \( T_M \) (50°C), the positive heat flux \( (q > 0) \) reduced the corrosion rate, and the negative heat flux \( (q < 0) \) accelerated it. Moreover, the \( \eta_i \) increased with increasing \( |q| \), indicating that larger \( |q| \) will significantly affect the corrosion rate.

**EIS results**

Figure 5 shows the typical EIS results of the Al alloy at \( E_{\text{corr}} \) in 0.5 M H\(_2\)SO\(_4\) under different heat transfer conditions (\( t = 2 \text{ h} \)). The total system impedance under different heat transfer conditions was relatively small (around 30–70 Ω cm\(^2\)) and changed with \( q \), indicating a quick corrosion rate in the test system and the influence of \( q \) on corrosion. These impedance results can be compared to the Al in 0.1 M HCl (around 150 Ω cm\(^2\)) [41] and in 1.0 M HCl (around 10 Ω cm\(^2\)) [42], but their EIS features were different due to the difference in test media.

All the Nyquist and Bode diagrams in Figure 5 have similar features and consist of two capacitive loops without an inductive loop in the low-frequency range [42], further proving that heat flux does not change the corrosion mechanism in the test system. The high-frequency capacitance loop (1–10\(^4\) Hz) was determined by the electric double-layer capacitance \( (C_{dl}) \) parallel to the charge transfer resistance \( (R_{ct}) \), where \( C_{dl} \) is generally simulated with a constant phase element (CPE\(_{dl}\)) [43]. The low-frequency capacitance loop (10\(^{-2}\)–1 Hz) was related to the film on the metal surface [44]. The change of the two capacitance loops under different heat transfer conditions (Figure 5(a)) suggests that \( q \) influences the Faraday process and the surface film on the Al alloy to change its corrosion rate.

Figure 5(a) also presents an equivalent circuit (EC) to fit these EIS results, where \( R_i \) is the solution resistance, \( R_p \) is the film resistance, and \( C_f \) is the film capacitance. The fitting parameters are listed in Table 3, and the fitting curves are displayed in Figure 5, indicating good fitness with the raw data. In Table 3, the polarisation resistance \( (R_p) \) equals \( R_{ct} + R_f \) inversely proportional to \( i_{\text{corr}} \).

The results in Table 3 indicate that the order of 1\(/R_p\) is \( \Delta T_{70-30} < \Delta T_{60-40} < \Delta T_{50-50} < \Delta T_{40-60} < \Delta T_{30-70} \) which is generally consistent with the results obtained from the polarisation curves (Table 2). The \( R_{ct} \) and \( R_f \) values increased while \( C_f \) and CPE\(_{dl}\)-T (indicating \( C_{dl} \)) decreased with the more positive \( q \), suggesting that the corrosion process of the Al alloy is inhibited and its surface layer may be improved with the positive heat flux. In contrast, \( R_p \) and \( R_f \) generally decreased with more negative \( q \), but \( C_f \) and CPE\(_{dl}\)-T changed without evident tendency, implying that the negative heat flux can accelerate the corrosion of the Al alloy and damage its surface layer performance. These results clearly show that the heat flux not only influences the corrosion process of the Al alloy but also changes its surface film, which will be discussed later.
Table 1. Heat transfer conditions and corresponding parameters in this study (inlet Q = 20 L h⁻¹).

| Conditions       | $T_{\text{water}}$ °C | $T_{\text{solution}}$ °C | $t_{\text{M, measure}}$ °C | $t_{\text{M, center}}$ °C | $\Delta t_{\text{solution}}$ mm | Heat flux $W$ | $q$ kW m⁻² |
|------------------|-----------------------|--------------------------|----------------------------|----------------------------|---------------------------------|---------------|-------------|
| $\Delta T_{70-30}$ | 70.0                  | 30.0                     | 49.5                       | 50.2                       | 0.9                             | +3.47         | -22.54     |
| $\Delta T_{60-40}$ | 60.0                  | 40.0                     | 49.7                       | 50.1                       | 0.9                             | +1.73         | +11.24     |
| $\Delta T_{50-50}$ | 50.0                  | 50.0                     | 50.0                       | 50.0                       | 0                               | 0             | 0          |
| $\Delta T_{40-60}$ | 40.0                  | 60.0                     | 50.2                       | 50.3                       | 0.9                             | -1.72         | -11.17     |
| $\Delta T_{30-70}$ | 30.0                  | 70.0                     | 50.4                       | 50.1                       | 0.9                             | -3.43         | -22.28     |

Weight loss results

The weight loss tests were performed in the test system to verify the influence of heat transfer on the corrosion of the Al alloy. Figure 6 presents the obtained $P_w$ values under various heat transfer conditions (Table 1), in which the error bars are used to show the data fluctuation under the same condition. The change percentage of $P_w$ ($\eta_w$) is also displayed in Figure 6, which is calculated similarly to $\eta_i$ as:

$$\eta_w(\%) = (1 - \frac{P_w}{P_0}) \times 100$$

where $P_0$ is the $P_w$ value when $q = 0$.

The results in Figure 6 show that the $P_w$ order is $\Delta T_{70-30} < \Delta T_{60-40} < \Delta T_{50-50} < \Delta T_{40-60} < \Delta T_{30-70}$, and $|\eta_w|$ increases with increasing $|q|$, which is generally consistent with the results obtained from the polarisation curves and EIS results (Table 2 and Table 3). It should be noted that there is $P_w > P_i$ (Table 2) under each heat transfer condition, which may be because $P_w$ is the average corrosion rate of the whole test period (5 h), but $P_i$ is the simultaneous corrosion rate at the test time (2 h). However, the influence of heat flux on $P_w$ and $P_i$ (or $1/P_i$) displays the same change tendency as changing $q$, further verifying that $q$ has an apparent influence on the corrosion rate.

Characterisation of corrosion surface

Figure 7 presents the optical surface morphologies of the Al alloy corroded in 0.5 M H₂SO₄ for 2 h under different heat transfer conditions. A corrosion product layer can be seen changing on the Al alloy. Figure 6 presents the obtained weight loss results, and the corresponding EDX analysis for the Point A~C on their surfaces is shown in Figure S3 (SI). In Figure 8, small local corrosion pits occur on the test sample, similar to the reports of D. Mercier et al. [45], which should be related to the oxide layer's ununiform dissolution on the test sample. Their EDX analysis results (Figure S3, SI) are similar and show Al, O, and C elements, suggesting that Al oxides occur on their surfaces.

These corroded samples’ 3D morphologies and pit depth profiles were measured to obtain their deepest depth using a 3D optical microscope, as shown in Figure S4 (SI). The local corrosion pit’s depth at $\Delta T_{70-30}, \Delta T_{60-40}, \Delta T_{50-50}, \Delta T_{40-60}$ and $\Delta T_{30-70}$ is about 19.0, 21.6, 23.5, 26.2, and 34.4 μm, respectively. These results imply that the negative heat flux also improves the local corrosion of the Al alloy, and the positive heat flux inhibits it.

In order to verify the composition of the corrosion products on the Al alloy’s surface, XRD and XPS analyses were performed for the Al alloy corroded in 0.5 M H₂SO₄ for 2 h under different heat transfer conditions, as shown in Figure S4 (SI) and Figure 9. The XRD patterns (Figure S4, SI) indicate that the corrosion product layer under different heat transfer conditions should be Al₂O₃ and Al₂(SO₄)₃·18H₂O. The XPS survey spectrum in Figure 9(a) also shows O 1s, Al 2p, and S 2p spectra. Thus, the high-resolution O 1s, Al 2p, and S 2p spectra were further analyzed (Figure 9(b–d)) to verify their detailed compositions.

Two peaks with binding energy (BE) of 530.2 and 531.9 eV in the O 1s spectrum (Figure 9(b)) correspond to Al₂O₃ and Al₂(SO₄)₃ [46,47]. The main Al 2p peak (BE = 74.56 eV) in Figure 9(c) can be associated with Al–O bonds of Al₂O₃ [48], and another peak (BE = 77.23 eV) is assigned to the combination of the Cu 3p spectrum [49]. The S 2p spectrum in Figure 9(d) (BE = 169.13 eV) can be associated with Al₂(SO₄)₃ [50,51]. These XPS analysis results further prove that Al₂O₃ and Al₂(SO₄)₃ occur on the Al alloy’s surface after immersion in 0.5 M H₂SO₄.

It should be noted that because the Al surface can be oxidised easily in the air, the detected Al₂O₃ might result from the oxidation of its surface during the immersion test in 0.5 M H₂SO₄ [45] or grow in the air before the analysis. In addition, Al₂(SO₄)₃·18H₂O can be easily dissolved in 0.5 M H₂SO₄, so it should precipitate on the test sample after the immersion test. Even so, the above results of Figure 7(a–c), S5 (SI), and 9 still imply that Al₂O₃ and Al₂(SO₄)₃ should be the corrosion product on the Al alloy, discussed later.

Discussion

Corrosion process in the test system

There is an Al₂O₃ oxide layer on the Al alloy’s surface [52–54]. Therefore, when the Al alloy specimen is immersed in the H₂SO₄ solution, the oxide film dissolution occurs.

$$\text{Al}_2\text{O}_3 + 6\text{H}^+ \leftrightarrow 2\text{Al}^{3+} + 3\text{H}_2\text{O}$$  (5)
In the meantime, Al alloy’s acidic corrosion starts, whose cathodic reactions include:

\[
2H_{\text{ads}}^+ + 2e^- \rightarrow H_2 \quad (6)
\]

\[
O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \quad (7)
\]

For the high H\(^+\) concentration in 0.5 M H\(_2\)SO\(_4\), the primary cathodic reaction should be Equation (6). The anodic reactions include \[55,56\]:

\[
\text{Al} + H_2O \leftrightarrow \text{AlOH}_{\text{ads}} + H^+ + e^- \quad (8)
\]

\[
\text{AlOH}_{\text{ads}} + 5H_2O + H^+ \leftrightarrow \text{Al}^{3+} \cdot 6H_2O + 2e^- \quad (9)
\]

\[
2\text{AlOH}_{\text{ads}} + H_2O \leftrightarrow \text{Al}_2O_3 + 4H^+ + 4e^- \quad (10)
\]

Equations (9) and (10) occur in parallel after Equation (8), resulting in the dissolution and oxide formation processes on the Al surface \[56\].

By XPS analysis, D. Mercier et al. \[45\] detected the oxide layer on 1050 Al alloy in pH = 3 H\(_2\)SO\(_4\) solution. They found that a native oxide film was about 4.3 ± 0.3 nm, whose composition is a mixture of aluminium oxide, pseudo-boehmite, and bayerite covered with some carbonaceous contamination. Its thickness increased to 5.1 ± 0.3 nm after 30 000 s immersion in the H\(_2\)SO\(_4\) solution, proving the oxide film growth and the formation and dissolution of the film in dynamic equilibrium. These results also indicate that the oxide layer on Al metal in H\(_2\)SO\(_4\) solution is complex and challenging to clarify. However, the aging of the oxide film on Al metals can lead to the formation of Al\(_2O_3\) by a deprotonation process \[52\]. XRD (Figure S5, SI) and XPS analysis results (Figure 9) also display the existence of Al\(_2O_3\) on the test samples. Thus, we can consider the Al\(_2O_3\) oxide layer on the Al alloy test samples.

For the dissolution process, Al\(^{3+}\) can further form various complexes as follows \[55,57,58\]:

\[
\text{Al}^{3+} + H_2O \leftrightarrow [\text{AlOH}]^{2+} + H^+ \quad (11)
\]

\[
[\text{AlOH}]^{2+} + X^- \leftrightarrow [\text{AlOHX}]^+ \quad (12)
\]

In 0.5 M H\(_2\)SO\(_4\), HSO\(_4^−\) is the dominant anion \(pK_a = 1.9\) \[59\], so Equation (12) can be rewritten as:

\[
[\text{AlOH}]^{2+} + \text{HSO}_4^- \leftrightarrow [\text{AlO(HSO}_4^-)]^+ \quad (13)
\]

HSO\(_4^-\) can further replace OH\(^-\) through the ligand exchange in 0.5 M H\(_2\)SO\(_4\) \[59,60\] and then be hydrolyzed as follows:

\[
[\text{AlO(HSO}_4^-)]^+ + \text{HSO}_4^- \leftrightarrow [\text{Al(HSO}_4^-)_2]^+ + H_2O \quad (14)
\]

\[
[\text{Al(HSO}_4^-)_2]^+ + H_2O \leftrightarrow \text{Al}_2(SO}_4)^3^- + [\text{AlO(HSO}_4^-)]^+ + 2\text{HSO}_4^- + 4H^+ \quad (15)
\]

In this case, the formation of Al\(_2(SO}_4)^3^-\) is feasible \[61\], which is also supported by the XRD (Figure S5, SI) and XPS analysis results (Figure 9).

The controlling step in the Al alloy dissolution is the complexation reaction between the hydrated cation and anion, i.e. Equation (13) \[55\]. However, Ford et al. proposed that Equation (8) would be the rate-determining step for the bare Al alloy surface \[56\]. In this case, we used the total anodic reaction to describe the anodic behaviour of the Al alloy in acid media. Based on Equations (8)–(10), the total anodic reactions can be generally written as:

Dissolution

\[
\text{Al} + 6H_2O \leftrightarrow \text{Al}^{3+} \cdot 6H_2O + 3e^- \quad (16)
\]

Oxide formation \[62\]

\[
2\text{Al} + 3H_2O \leftrightarrow \text{Al}_2O_3 + 6H^+ + 6e^- \quad (17)
\]

| Conditions | \(q\) kW·m\(^{-2}\) | \(b_0\) mA·dec\(^{-1}\) | \(b_1\) mA·dec\(^{-1}\) | \(E_{\text{corr}}\) V vs. SCE | \(i_{\text{corr}}\) mA·cm\(^{-2}\) | \(\rho\) mm·y\(^{-1}\) | \(\eta\) % |
|------------|-------------------|----------------|----------------|----------------|----------------|----------------|----------------|
| \(\Delta T_{70-10}\) | +22.54 | 179 | 73 | -0.513 | 0.33 | 3.65 | 56.2 |
| \(\Delta T_{60-40}\) | +11.24 | 268 | 88 | -0.513 | 0.55 | 6.03 | 27.6 |
| \(\Delta T_{50-50}\) | 0 | 185 | 87 | -0.523 | 0.76 | 8.33 | 0 |
| \(\Delta T_{40-60}\) | -11.17 | 254 | 92 | -0.540 | 0.89 | 9.81 | -17.8 |
| \(\Delta T_{30-70}\) | -22.28 | 223 | 102 | -0.539 | 1.15 | 12.66 | -52.0 |

**Table 2.** Fitting results obtained from the polarisation curves in Figure 4.

Figure 5. (a) Nyquist and (b) Bode plots of the Al alloy at \(E_{\text{corr}}\) in 0.5 M H\(_2\)SO\(_4\) under different heat transfer conditions \((t = 2\) h\), and the insertion in (a) is the equivalent circuit to simulate the Al/solution interface.
According to the above discussion, Figure S6 (SI) presents a schematic diagram for the corrosion processes on the Al alloy in the H2SO4 solution. The formation of various complexes can impede the dissolution of Al [45], and their hydrolysis leads to the formation of stable hydrous oxides on the Al surface [55], resulting in the dynamic dissolution and formation of the oxide layer. Therefore, the states of the oxide film and the adsorption layer of various complexes on the Al alloy surface can influence its corrosion behaviour.

**Kinetic analysis for the effect of heat flux on corrosion**

The electrochemical and weight-loss data (Figures 4–6) and corrosion surface morphologies (Figures 7 and 8, and S4, SI) indicate that the heat flux has an evident influence on the corrosion rate of the Al alloy in 0.5 M H2SO4. According to the conventional chemical reaction kinetics, the corrosion rate \( r \) of the dissolution of Al alloys in H2SO4 solution can be described as [63]:

\[
r = k \cdot C_{H_2SO_4}^n \quad (18)
\]

where \( k \) is the reaction rate constant, \( C_{H_2SO_4} \) is the H2SO4 concentration (0.5 M), and \( n \) is the reaction order of H2SO4. Under different heat transfer conditions, the \( C_{H_2SO_4} \) in the test system can be seen as a constant (0.5 M), and the corrosion mechanism of the Al alloy is not changed, resulting in a constant reaction order \( n \), so the heat flux may only influence \( k \) to change \( i_{corr} \).

Generally, we can use the Arrhenius equation to describe the \( k \) of the complex corrosion reaction [64,65] and \( i_{corr} \) to describe the corrosion rate. Thus, there is [66]

\[
i_{corr} = Aexp\left(-\frac{E_a}{RT}\right) \quad (19)
\]

| Conditions | \( q \) (kW·m²⁻¹) | \( R_i \) (Ω·cm²) | CPEa-Ω·cm²·Hz⁻¹ | CPEa-Ω·cm² | \( R_{dl} \) (Ω·cm²) | \( R_p \) (Ω·cm²) | \( G_i \) (Ω·cm²) |
|------------|----------------------|--------------------|-----------------|----------------|---------------------|---------------------|----------------|
| ΔT70-30    | 22.54                | 3.6                | 0.36            | 0.98           | 43.8                | 0.14                | 19.9           | 63.7          |
| ΔT50-50    | 11.24                | 3.0                | 0.65            | 0.90           | 34.7                | 0.19                | 14.4           | 49.1          |
| ΔT30-70    | 0                    | 3.2                | 0.73            | 0.90           | 23.7                | 0.25                | 9.1            | 32.8          |
| ΔT20-60    | 11.17                | 4.3                | 0.55            | 0.90           | 22.6                | 0.25                | 8.7            | 31.2          |

The fitting parameters of the EIS results in Figure 5.

**Figure 6.** \( P_w \) and \( \eta_w \) values of the Al alloy immersed in 0.5 M H2SO4 for 5 h under different heat transfer conditions.

**Figure 7.** Surface morphologies of the Al alloy corroded in 0.5 M H2SO4 for 2 h under different heat transfer conditions: (a, d) ΔT70-30, (b, e) ΔT50-50, (c, f) ΔT30-70 with (a–c) and without (d–f) corrosion products.
where \( A = A_0 \times (3F_{\text{SO}_4}^n) \), \( E_a \) is the apparent effective activation energy, \( A_0 \) is the apparent Arrhenius exponential pre-factor, \( R \) is the universal gas constant, and \( T \) is the absolute temperature.

To verify the detailed effect of \( q \) on \( k \), we tested polarisation curves at different \( T_M \) (30, 40, 50, 55, 60, 65°C) with \( q = 0, +11.43, \) and \(-11.33 \text{ kW·m}^{-2}\) and obtained \( \ln i_{\text{corr}} - 1/T \) curves under different \( q \), as shown in Figure 10. These \( \ln i_{\text{corr}} - 1/T \) curves were linearly fitted to obtain \( A \) and \( E_a \) values under different \( q \), as shown in Table 4.

Compared with the \( q = 0 \) condition, when \( q = +11.43 \text{ kW·m}^{-2} \), \( A \) and \( E_a \) increased, but both \( A \) and \( E_a \) decreased when \( q = -11.33 \text{ kW·m}^{-2} \) (Table 4). Apparently, the change of \( E_a \) dominated the \( k \) to make \( i_{\text{corr}} \) decrease at \( q = +11.43 \text{ kW·m}^{-2} \) and increase at \( q = -11.33 \text{ kW·m}^{-2} \), as shown in Figure 10, which is consistent with the above results in Tables 2 and 3 and Figure 6. These results indicate that \( q \) can affect \( A_0 \) and \( E_a \) of the corrosion reaction, as shown in Equation (19), and \( E_a \) plays the dominating role in changing the corrosion rate.

**Effect of heat flux on the surface film of the Al alloy**

According to the above discussion about the corrosion process on Al alloys and the characterisation of corrosion surface (Figure 7(a–c), Figure 9, and S5, SI), there is a surface film on the Al alloy in 0.5 M H\(_2\)SO\(_4\), including the oxide film (e.g. Al\(_2\)O\(_3\), Figure 9 and S5, SI) and other adsorption surface layers of various complexes, see Equations (8)–(14) and Figure S6 (SI). Moreover, the EIS results (Figure 5)

\[ \text{Figure 8. SEM images of the Al alloy corroded in 0.5 M H}_2\text{SO}_4 \text{ for 2 h under different heat transfer conditions: (a) } \Delta T_{70-30}, \text{ (b) } \Delta T_{50-50}, \text{ (c) } \Delta T_{30-70} \text{ (with corrosion product).} \]

\[ \text{Figure 9. (a) XPS survey spectrum and high-resolution spectra (b) O 1s, (c) Al 2p, and (d) S 2p for the Al alloy corroded in 0.5 M H}_2\text{SO}_4 \text{ for 2 h at } \Delta T_{70-30}. \]
indicate that the surface film on the Al alloy changed with different $q$, but combining the change of the oxide film and the adsorption surface layer on the Al alloy.

To verify the effect of $q$ on the oxide layer of the Al alloy in 0.5 M $\text{H}_2\text{SO}_4$ [67], the Mott–Schottky (MS) measurements were performed under $\Delta T_{70-30}$, $\Delta T_{50-50}$, and $\Delta T_{30-70}$ conditions (see Table 1). The MS relationship for an $n$-type semiconductor passive layer can be written as [68–70]:

$$\frac{1}{C^2} = \frac{2}{\varepsilon e_0 N_D} \left( E - E_{FB} - \frac{KT}{e} \right)$$

(20)

where $C$ is the electrode capacitance ($\text{F/cm}^2$), $E$ is the electrode potential (V vs. SCE), $E_{FB}$ is the flat band potential, $\varepsilon$ is the dielectric constant of the passive film (in $\varepsilon_{\text{Al}_2\text{O}_3} = 8$ [71], $e_0$ is the permittivity of vacuum ($8.854 \times 10^{-12}$ F/cm), $e$ is the electron charge ($1.602 \times 10^{-19}$ C), $N_D$ is the donor density for n-type semiconductor ($\text{cm}^{-3}$), $K$ is the Boltzmann constant, and $T$ is the absolute temperature. According to Equation (20), $N_D$ can be calculated from the $C^{-2}$-$E$ curve slopes in the linear region, generally indicating the defect density in an oxide film [69].

As shown in Figure 11, the $C^{-2}$-$E$ relationship of the specimens corroded in 0.5 M $\text{H}_2\text{SO}_4$ for 2 h under different heat transfer conditions was recorded at a frequency of 1 kHz with the AC voltage amplitude $\pm 10$ mV in a potential range of $-100$ mV to $300$ mV (vs. $E_{\text{corr}}$) in successive steps of 20 mV [68]. Under $\Delta T_{70-30}$ and $\Delta T_{50-50}$ conditions, the MS plots displayed two linear regions above or below around $-0.36$ V (vs. SCE), as shown by the straight lines drawn in Figure 11, and the positive slopes indicated that the oxide film was n-type. The obtained $N_D$ values based on Equation (20) are also listed in Figure 11, indicating the difference in the oxide film under different potential ranges and heat transfer conditions.

According to the $N_D$ values in Figure 11, it can be concluded that the positive heat flux ($q > 0$) improves the oxide film performance ($N_D (\Delta T_{70-30}) < N_D (\Delta T_{50-50})$). In contrast, no MS characteristic was evident in all potential ranges at $\Delta T_{30-70}$ (Figure 11(a)), revealing the destruction of the oxide film with negative heat flux ($q < 0$). These results further prove that heat flux across the metal/solution interface has an evident influence on the oxide film formation of the Al alloy in $\text{H}_2\text{SO}_4$.

Based on the COMSOL analysis of the heat transfer state in the test system, different $q$ produces different temperature distributions in the test solution’s heat boundary layer (Figure 2(b)), leading to the local temperature difference near the metal surface. Such local temperature differences can change the solubility of the metal surface layer and the concentration distribution of corrosive species adjacent to the metal surface [14], e.g. DO, $\text{H}^+$, and $\text{HSO}_4^-$. Therefore, the metal corrosion process will be affected. In this test system, the influence of DO, $\text{H}^+$, and $\text{HSO}_4^-$ (pK<sub>1</sub> = 1.9) can be ignored in the 0.5 M $\text{H}_2\text{SO}_4$ solution, so only the solubility of the metal surface layer is the main issue to be considered. Based on the above discussion, Figure 12 presents the schematics of the heat flux influence on the surface layer of the Al alloy in the $\text{H}_2\text{SO}_4$ solution.

When positive heat flux flows across the test sample, e.g. at $\Delta T_{70-30}$ and $\Delta T_{50-40}$, the fluid temperature near the metal surface decreases quickly in the heat boundary layer to reach $T_{\text{solution}}$, as shown in Figure 12(a). Owing to the low-temperature solution near the sample surface, the solubility of the formed oxide layer (Equations (5), (10), and (17)) and the complexes adsorption layer (Equations (11)–(14)) will be lowered to stabilise the surface film [45]. In this case, the positive heat flux facilitates the oxide layer and
adsorption layer formation, resulting in lower $N_D$ values of the oxide layer (Figure 11) and the stable adsorption layer to reduced $C_f$ and CPE$_{dl}$ (Table 3). Meanwhile, the stabilised surface film can increase $R_p$, $R_b$, and $R_p$ (Table 3) to decrease $\iota_{corr}$. When $q < 0$, e.g. at $\Delta T_{30.70}$ and $\Delta T_{40.60}$, the high-temperature solution near the sample surface will weaken the surface film to increase its solubility (see Figure 12(c)). Thus, the opposite effect occurs, decreasing $R_p$, $R_b$, and $R_p$ (Table 3) to increase $\iota_{corr}$. Meanwhile, the quick dissolution of the surface film may be unstable to produce fluctuations in the $C_f$ and CPE$_{dl}$, not showing an evident change tendency (Table 3).

According to the above discussion, on the one hand, heat flux can affect the formation of the oxide film and the adsorption layer on the Al alloy to change its surface film performance and therefore change the corrosion rate. On the other hand, heat flux can also change the $E_a$ and $A_0$ of the corrosion reaction of the Al alloy to change its corrosion rate. In summary, the effect of heat flux on the corrosion behaviour of the Al alloy in the H$_2$SO$_4$ solution has been verified under controlling heat transfer states. Meanwhile, the constructed testing system combined with the COMSOL simulation has been proved valid in investigating metal corrosion under heat transfer states in non-boiling media. This method will support further study on the effect of heat flux on the performance of corrosion inhibitors, scale inhibition, and other protection methods in heat transfer systems.

**Conclusions**

A dual-circulation testing system, including deionised water and the test solution, was constructed to study heat flux’s effect on metal corrosion in non-boiling media. By controlling the inlet flow rate ($Q$), the deionised water temperature ($T_{water}$), and the test solution temperature ($T_{solution}$), different heat transfer conditions can be achieved in the testing system. Their heat transfer parameters were determined quantitatively by COMSOL simulation, including heat flux density ($q$), metal surface temperature ($T_M$), the thermal boundary layer thickness ($\delta$), and the distributions of $T_{solution}$ and the solution flow rate ($\nu_{solution}$). Under different controlling heat transfer states, all the above heat transfer parameters except $q$ are constant in the test system to highlight the influence of $q$ on metal corrosion.

In the testing system, the effect of $q$ ($\sim 22.28$–$22.54$ kW m$^{-2}$) on the corrosion of an Al–Cu–Mg–Mn alloy in 0.5 M H$_2$SO$_4$ was investigated at a constant $T_M$ (50°C) under different controlled heat transfer conditions. Compared with the thermal equilibrium condition ($q = 0$), the positive heat flux ($q > 0$) from the metal to the test solution can improve the surface layer performance on the Al alloy to mainly inhibit its anodic process, decreasing its corrosion rate with increasing $q$. Its corrosion current density ($i_{corr}$) decreased by 56% at $q = 22.54$ kW m$^{-2}$. While $q < 0$, the opposite effect occurred, increasing the corrosion rate with increasing $|q|$. Its $i_{corr}$ increased by 52% at $q = -22.28$ kW m$^{-2}$. Apparently, heat flux can influence metal corrosion behaviour, even at a constant metal surface temperature in the heat transfer system.

Heat flux can also influence the apparent effective activation energy ($E_a$) and the apparent Arrhenius exponential pre-factor ($A_0$) for the dissolution of the Al alloy in 0.5 M H$_2$SO$_4$. $E_a$ and $A_0$ increased when $q > 0$, while they decreased when $q < 0$. However, the change of $E_a$ played a dominating role in changing the corrosion rate of the Al alloy.

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