Research on the Corrosion Behaviors of Austenitic Steel in Molten Aluminum Alloy

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Abstract: Die corrosion has been a concern during aluminum alloy die casting. The casting parameters play a significant role in causing corrosion, such as the temperature of the aluminum alloy melt and working time. In order to study the effect of temperature and working time on dynamic corrosion behaviors, SDHA steel was stirred in molten ADC12 aluminum alloy at 650–800 °C. The corrosion morphology and corrosion product were investigated through X-ray diffraction (XRD) and scanning electron microscope (SEM) observations. The results show that the matrix reacts with aluminum alloy to form an Al₈Fe₂Si phase at experimental temperatures. The growth activation energy of the Al₈Fe₂Si phase is 89 kJ/mol. The dynamic corrosion rate rises with increasing temperature and holding time. The most serious corrosion was found when the experimental temperature reached 800 °C, which is closely related to the peeling of matrix and the formation of Al₈Fe₂Si at the grain boundary. Besides, the vanadium carbides in the matrix act as barriers to hinder the diffusion of Al and Si atoms effectively.

Keywords: aluminum alloy; corrosion; intermetallic compound; interface; steel

1. Introduction

Based on the strict CO₂ emission policy, automakers choose aluminum alloy as a lightweight material for engine blocks, cylinder heads, and wheels [1]. These large aluminum alloy parts could potentially reduce the weight of a vehicle by 30–40% [2]. Die casting is economical and is a common production method for aluminum alloy parts in which molten aluminum alloy is quickly injected into the die cavity at a high velocity [3]. This direct contact has long been regarded to cause die corrosion. An increasingly accepted view was that die corrosion is divided into soldering (static corrosion) and erosion (dynamic corrosion) [4,5].

Due to the reaction between iron and molten aluminum alloy, the soldering feature is the adhesion of intermetallic compounds (IMC) to the die surface during filling and solidification [6]. The microstructure of the IMC layer at the interface is similar to the intermetallic layer after hot-dip aluminizing (coating technology). Therefore, the soldering mechanism has been studied by a large number of immersion tests. The formation of IMC includes solid dissolution, element diffusion, and chemical reaction. In single (martensite) and dual (martensite and ferrite) phases of 12% Cr steel, aluminum diffused more easily into martensite due to the structural differences and large number of defects in martensite [7]. Additionally, Zhang [8] noticed that the precipitation phase along the austenite/ferrite and ferrite/ferrite interfaces could hinder aluminum diffusion and improve molten aluminum corrosion resistance. In addition, the formation of a structural compound in a solid is a workable method to mitigate aluminum diffusion. It has been reported that the netlike M₂B boride phase in Fe-B steel could effectively hinder aluminum diffusion, and a quantitative number model between the thickness loss and the diffusion inhibition factor was suggested [9]. By comparing the corrosion mechanism of unalloyed steel...
and ferritic and modified pearlitic cast irons in molten aluminum, graphite lamellas in ferritic and modified pearlitic cast irons were observed to act as an efficient barrier to aluminum diffusion [10]. Therefore, much attention has been paid to the effect of special microstructures in steel on corrosion resistance.

The erosion is the result of soldering and the impact of high-velocity molten aluminum alloy at the same location [11]. The erosion is marked by a loss of material from the die surface and the decline of surface hardness [12,13]. Additionally, the erosion is controlled by temperature, velocity, die surface modeling, and impact angle. A greater angle, temperature, and speed increase the erosion rate [14]. Additionally, some research showed that erosion might be caused by the impact of solidified particles (silicon particles and IMCs) during the filling [15]. A lower superheat results in more solidified particles, which promote erosion. According to the failure analysis of die casting shot sleeves, the recurrence and growth of IMCs on the surface damage erosion resistance [16]. Additionally, they proposed that the DIN 1.2367 steel with high Mo and low Si resists erosion better than AISI H13 steel. The literature proved that the flake graphite in the HT300 steel prevented the diffusion reaction at stirring compared to A3 steel and QT5381 steel [17].

Based on the complex erosion mechanism and the lack of standardized test equipment, the studies focused on evaluating the corrosion resistance of different materials through failure analysis and specific tests [4,9,11,12,16,17]. The effect of temperature and time on erosion was not systematically studied. In this work, the dynamic corrosion behaviors and IMCs evolution of SDHA steel in ADC12 aluminum alloy at different holding times and temperatures were investigated. Moreover, the effect of carbides and grain boundaries on dynamic corrosion resistance was critically discussed. This effect has an important guiding significance for selecting the appropriate aluminum alloy temperature and holding time during die casting.

2. Materials and Methods

2.1. Materials

SDHA is austenitic steel and its chemical composition is shown in Table 1. The steel was held for 45 min at 1170 °C for the solution treatment, then quenched in oil, and finally held for 3 h at 720 °C. The hardness of specimens after heat treatment was 44–46 HRC using a Rockwell hardness tester (69-1 type, Shangguang, Shanghai, China). The chemical composition of commercial ADC12 cast aluminum alloy is shown in Table 2. ADC12 cast aluminum alloy was melted by heating and used as an etchant.

Table 1. Chemical compositions of SDHA steel (wt.%).

| C  | Si  | Mn   | Cr  | Mo  | V   | P    | S    | Fe |
|----|-----|------|-----|-----|-----|------|------|----|
| 0.62 | 0.81 | 10.45 | 3.10 | 1.69 | 1.83 | 0.012 | 0.0140 | Bal. |

Table 2. Chemical compositions of ADC12 aluminum alloy (wt.%).

| Pb  | Ti  | Zn  | Si  | Mn  | Fe  | Mg  | Cu  | Al  |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.035 | 0.064 | 0.87 | 10.60 | 0.16 | 0.76 | 0.22 | 1.74 | Bal. |

2.2. Methods

The shape of specimens is shown in Figure 1a. The specimens were ground and polished mechanically. The equipment for corrosion tests was illustrated schematically in Figure 1b. In addition, the molten aluminum alloy is injected into the cavity at 650–800 °C during die casting [18,19]. Thence, 650, 700, 750, and 800 °C were selected as the temperatures of molten aluminum alloy; the holding times were 10 min, 20 min, and 30 min. Specific test parameters are shown in Tables 3 and 4. Regarding dynamic corrosion, the rotational and linear speeds of specimens were 120 r/min and 0.05 m/s, respectively. In order to evaluate weight loss and observe corroded surface morphologies, the tested
samples were immersed in 10% NaOH solution to remove residual aluminum alloy and IMCs. The corroded surface morphologies and IMC layer were analyzed by scanning electron microscope (SEM, Supra-40 type, Zeiss, Oberkohen, Germany), energy-dispersive spectrometer (EDS, X-MAX type, Oxford Instruments, Oxford, UK) and optical microscopy (OM, LV 150 type, Nikon, Tokyo, Japan). The electronic balance calculated the weight of the original samples and tested samples. The types of IMCs were analyzed with X-ray diffraction (XRD, D/MAX-2500 type, Rigaku, Tokyo, Japan).

![Figure 1. Shape of the samples and schematic illustration of the test equipment (a) shape of the sample; (b) test equipment.](image)

Table 3. Corrosion test parameters during rotation.

| Number | Temperature/℃ | Time/Min | Number | Temperature/℃ | Time/Min |
|--------|----------------|----------|--------|----------------|----------|
| 1      | 650            | 10       | 4      | 800            | 10       |
| 2      | 700            | 10       | 5      | 700            | 20       |
| 3      | 750            | 10       | 6      | 700            | 30       |

Table 4. Corrosion test parameters during static immersion.

| Number | Temperature/℃ | Time/Min | Number | Temperature/℃ | Time/Min |
|--------|----------------|----------|--------|----------------|----------|
| 1      | 650            | 10       | 7      | 750            | 10       |
| 2      | 650            | 20       | 8      | 750            | 20       |
| 3      | 650            | 30       | 9      | 750            | 30       |
| 4      | 700            | 10       | 10     | 800            | 10       |
| 5      | 700            | 20       | 11     | 800            | 20       |
| 6      | 700            | 30       | 12     | 800            | 30       |

3. Results

3.1. Microstructure of SDHA Steel

Figure 2 shows the microstructure of SDHA steel after heat treatment. Coarse grain boundaries and large vanadium carbide particles were found in the steel.

3.2. Phase Calibration of IMCs

To confirm the phase compositions of the IMCs, the cross-sections of the corroded samples were analyzed by XRD. The results clearly depicted that the IMCs of the specimens were identified as the Al$_8$Fe$_2$Si phase at 650–800 ℃, as shown in Figure 3a. Some researchers reported that the IMCs were composed of FeAl [20], FeAl$_2$ [21], Fe$_2$Al$_5$ [22], and Fe$_4$Al$_{13}$ [23]. In this study, the IMCs were identified as the Al$_8$Fe$_2$Si phase. Three distinct regions could be found according to the morphology of the IMCs and EDS line scan analysis: the solidified ADC12 aluminum alloy on the left, the IMC layer in the middle, and steel on the right.
3.3. Dynamic Corrosion Morphology of IMCs

Figure 4 shows the dynamic corrosion morphologies of the IMCs after 10 min at different temperatures. At 650 °C, the particles and long strips of IMCs peeled off, as shown in Figure 4a. When the temperature of molten aluminum alloy rose to 700 °C, there seemed to be fewer exfoliated compound particles. However, irregular voids were found due to the different diffusion rates of Fe, Si, and Al atoms in the IMC layer. Some researchers believed that the voids in the IMC layer were caused by unbalanced diffusion as Kirkendall holes [10]. Additionally, the large internal stress between the matrix and the IMC layer caused cracks in the IMC layer. These cracks accelerated the fracturing of the IMC layer at 750 °C. In addition, molten aluminum alloy infiltrated along the cracks and increased the diffusion rates of Al and Si atoms. As the molten aluminum alloy temperature raised to 800 °C, the morphology of the IMC layer could be significantly changed. Molten aluminum alloy penetrated into the matrix, resulting in the exfoliation of the matrix. The exfoliated matrix destroyed the integrity of the IMC layer.

To study the effect of time on the dynamic corrosion of molten aluminum alloy, the samples were rotated at 700 °C in molten aluminum alloy for different amounts of time. The morphologies of the IMC layer are shown in Figure 5. When the samples were rotated for 20 min, the interface between the matrix and IMC layer became more uneven, as shown in Figure 5a. Additionally, a small amount of matrix peeled off according to EDS analysis. At the same time, there were gaps between the IMC layer and exfoliated matrix due to the different expansion coefficients. The matrix peeled off severely when time increased to 30 min, as shown in Figure 5b.
3.4. Surface Morphologies of the Matrix after Dynamic Corrosion

According to the corrosion under washout of molten aluminum alloy, scholars have proposed three basic corrosion surface mechanisms: (1) The pits appear on the sample surface under the impact of molten aluminum alloy. (2) With local pressure fluctuations of molten aluminum alloy, the formation and collapse of bubbles causes cavitations. (3) Solid erosion is caused by the impact of solidified particles (Si particles, oxidation particles, and intermetallic particles) [24–26]. Figure 6 is the surface morphologies of the matrix at different temperatures after dynamic corrosion. Scratches appeared on the surfaces of the tested samples; Zhu [24] also observed a similar phenomenon. From Figure 6a, the surface was not corroded badly by molten aluminum alloy at 650 °C and only small pits appeared under the washout of molten aluminum alloy. When the temperature reached 700 °C,
grooves and holes were found on the surface, as shown in Figure 6b. As the temperature increased to 750 °C, surface morphology was seriously corroded by molten aluminum alloy, as shown in Figure 6c. A large broken area on the surface causes severe weight loss. At 800 °C, grain boundaries played a vital role in the weight loss of matrix in molten aluminum alloy. According to Figure 6d, the molten aluminum alloy preferentially invaded the matrix along the grain boundaries, resulting in intergranular corrosion. When the corrosion time increased to 20 min at 700 °C, some small holes were seen on the matrix surface, as shown in Figure 7a. In addition, the molten aluminum alloy also corroded the matrix along the grain boundary, which was similar to surface morphology at 800 °C. As shown in Figure 7b, with time raised to 30 min, the intergranular corrosion became more serious, resulting in a large amount of matrix spalling.

Figure 6. Surface morphologies of the matrix after dynamic corrosion for 10 min. (a) 650 °C; (b) 700 °C; (c) 750 °C; (d) 800 °C.

Figure 7. Surface morphologies of the matrix at 700 °C after dynamic corrosion. (a) 20 min; (b) 30 min.

3.5. Dynamic Corrosion Rates

The weight loss rate of corrosion in the molten aluminum alloy was affected by time and contact surface. Thus, in order to better evaluate corrosion resistance in molten
aluminum alloy, the corrosion rate \( r \) is introduced in this paper; \( r \) was calculated based on Equation (1) [27]:

\[
    r = \frac{\Delta m}{s \times t}
\]

where \( \Delta m \) is the difference between the weight of the original samples and tested samples in 10% NaOH solution (g); \( s \) is the area of the original samples (cm\(^2\)); \( t \) is the corrosion time (min). The result is shown in Figure 8. The corrosion rate increased with the increase of temperature and time. At 650 and 700 °C, the outermost layer peeled off after dynamic corrosion, but the bonding force between the IMC layer and matrix was excellent. Therefore, the IMC layer could resist the washout of molten aluminum alloy. When the temperature of molten aluminum alloy increased to 750 °C, the bonding force between the IMC layer and matrix became poor under thermal stress. At 800 °C, the invasion of molten aluminum alloy into the matrix caused the intergranular corrosion to increase the corrosion rate. Corrosion time had a significant influence on the corrosion rates. On the basis of corrosion rates at 700 °C for different amounts of time, the corrosion rate increased significantly after 20 and 30 min as a result of the exfoliation of the matrix.

![Figure 8. The dynamic corrosion rates for (a) different temperatures; (b) different times at 700 °C.](image)

3.6. Growth Activation Energy of the IMCs

Due to the spalling of the IMC layer under the washout, it is difficult to quantitatively explain the relationship between thickness, temperature, and time. Thus, the samples were immersed statically in molten aluminum alloy to determine the growth kinetics of the IMCs. During static immersion, the diffusion of Fe, Si, and Al atoms is controlled by different parameters, such as temperatures, time, elements of molten aluminum alloy, and steel types. These parameters will also affect the morphologies, thickness, and types of IMCs [28–30]. In particular, temperature and time play an important role in the formation of IMCs. Figure 9 shows the morphologies of the IMC layer after static immersion. Independent of morphology, thickness increased with immersion time for all specimens. It can be seen from Figure 10 that the thickness of the IMC layer has a linear relationship with the holding time. When SDHA steel was in contact with molten aluminum alloy, the atoms diffused into each other at the interface. When the content of Fe atoms exceeded the saturation value, the supersaturated Fe atoms reacted with Al and Si atoms at the interface to form IMCs. However, the contact between the matrix and the molten aluminum alloy was hindered by continuous IMC layer formation. Therefore, the growth rates of the IMCs depended on the diffusion of atoms in the layer. Due to a lower diffusion rate of atoms in the IMC layer, the holding time cannot significantly promote the growth of IMCs.
During static immersion, the thickness and the square root of the holding time were related to the growth of the IMC layer [31,32]. The kinetic constant of $\text{Al}_8\text{Fe}_2\text{Si}$ was calculated based on Equation (2):

$$l^2 = K \times t$$

Here, $l$ is the thickness of the layer (µm); $K$ is the kinetic constant of $\text{Al}_8\text{Fe}_2\text{Si}$; $t$ is the holding time (min).

Figure 11a presents the results of the thickness of the $\text{Al}_8\text{Fe}_2\text{Si}$ phase at different temperatures. The kinetic constant $K$ is the slope in the relationship between the thickness and the square root of time. The result revealed that the values of $K$ increased with temperature. Additionally, the relationship between layer thickness and temperature
conformed to the Arrhenius relationship; the effect of temperature on diffusion rates could be described by the Equation (3):

\[
K = K_0 \times e^{-Q/RT}
\]  

(3)

where \(K\) is the kinetic constant of \(\text{Al}_8\text{Fe}_2\text{Si}\); \(K_0\) is the coefficient of the growth constant; \(Q\) is the activation energy (J mol\(^{-1}\)); \(R\) is the gas constant (8.314 J mol\(^{-1}\) K\(^{-1}\)); \(T\) is the temperature (K). The kinetic constant \((K)\) is obtained by Equation (2).

**Figure 11.** The growth kinetic curve of the IMC layer. (a) The thickness as a function of the square root of time; (b) the curve of the natural logarithm of the parabolic coefficient and the reciprocal of temperature.

In order to evaluate the activation energy of \(\text{Al}_8\text{Fe}_2\text{Si}\) phase, the values of \(\ln K\) were drawn as a function of the \(1/T\), as shown in Figure 11b. According to the straight-line slope, the growth activation energy of \(\text{Al}_8\text{Fe}_2\text{Si}\) phase was 89 kJ/mol. In addition, some scholars calculated the activation energy of other Fe–Al compound phases. Kim [7] indicated that the activation energy of the \(\text{Fe}_4\text{Al}_{13}\) phase was 138 kJ/mol. The activation energy of the \(\text{Fe}_2\text{Al}_5\) phase was evaluated by Heumann [33] and Bouche [34] and was found to be 76 and 74 kJ/mol, respectively. Therefore, the activation energy of the \(\text{Al}_8\text{Fe}_2\text{Si}\) phase was lower than that of the \(\text{Fe}_4\text{Al}_{13}\) phase and higher than that of the \(\text{Fe}_2\text{Al}_5\) phase.

### 4. Discussion

It can be found from the above results that although the IMCs at differing times and temperatures are all \(\text{Al}_8\text{Fe}_2\text{Si}\), the interfaces between IMCs and matrix show different dynamic corrosion morphologies. Corrosion resistance is affected by the growth behavior of the IMCs and the evolution of the matrix microstructure. IMC formation is a complex chemical process including penetration, dissolution, and adhesion [35]. Due to a more significant diffusion coefficient from Fe to Al, the Fe atoms in the matrix diffuse more easily into the molten aluminum alloy, resulting in matrix loss [28]. At the same time, an IMC layer is gradually generated at the interface due to the reaction and diffusion of Al, Si, and Fe atoms. In addition, due to a relative motion between matrix and molten aluminum alloy, the IMC layer and matrix were subjected to impact and shear force under the washout of molten aluminum alloy. The formation, thickening, and spalling of the IMC layer are recycled under combined force. Fe atoms are accelerated to dissolve during the cycle, resulting in increased matrix loss. From the kinetic constants of \(\text{Al}_8\text{Fe}_2\text{Si}\) at different temperatures, the reaction rate increases rapidly with rising temperature. The diffusion reaction preferentially reacts at a high energy position, such as the cavity and dendrite gap [17]. This phenomenon was found in this work; the high-energy grain boundaries
with vacancies, dislocations, and segregation of elements promote the diffusion reaction, as shown in Figure 12. The IMCs formed along grain boundaries have a weak adhesion with the matrix. These IMCs are separated from the matrix under scouring force accompanied by exfoliation of the matrix. In addition, according to the corrosion rate results, holding time seems to play a more important role in corrosion rate. At the early corrosion stage, depression and deformation are found on the surface. This would cause small pits at the location of surface defects and tissue defects due to the cyclic impact of molten aluminum alloy. As holding time increases, the softening of the matrix causes the hardness to decrease. Additionally, protracted impact causes fatigue of the matrix, which reduces its strength (especially yield strength) and plasticity [11]. In both cases, a number of small pits become large and join together to finally form dents on the surface.

Figure 12. Morphologies of IMC layer at the grain boundary.

4.1. The Effect of Carbides on Corrosion

Due to large vanadium carbides in SDHA steel, it is necessary to study their effect on dynamic corrosion. In this study, it was found that the vanadium carbides had an influence on the diffusion reaction. From Figure 13, although the matrix chemically reacted with Al and Si atoms, vanadium carbides were still present in the intermetallic layer. It was shown that vanadium carbides with poor dissolution act as barrels to prevent the diffusion of Si and Al. However, when the matrix around the carbides was completely corroded, the carbides exfoliated and embedded in the IMC layer. Additionally, the vanadium carbides destroyed the integrity of the IMC layer, causing the formation of holes and the inflow of molten aluminum alloy along the cracks.

4.2. Dynamic Corrosion Mechanism

The formation of the IMC layer is controlled by atomic diffusion. The diffusion of the atoms through the IMC layer is the vital step during the formation process. The diffusion rates are determined by the diffusion coefficients, the thickness of the diffusion boundary layer, the saturation concentration contents, and the concentration contents [36]. Meanwhile, the dissolutions and agitation speeds also control the dynamic corrosion rates. Therefore, the dissolution of steels rotating in molten aluminum alloy can be described by the Equation (4) [37]:

\[ k = 0.62K^{2/3}v^{-1/6}w^{1/2} \]

Here, \( k \) is the dissolution rate constant; \( K \) is the diffusion rate constant of solute in the IMC layer; \( v \) is the angular speed of samples; \( v \) is the kinematic viscosity of the melt. Equation (4) shows that the diffusion rates of atoms in the IMC layer and the agitation speeds strongly affect dissolution rates. Additionally, the matrix is not mechanically removed in rotating conditions; the damage zone is confined within the matrix surface and IMC layer. The weight loss of the steel increases under the impact of molten aluminum alloy because the impact induces the peeling of the IMC layer and weakens the diffusion
barrier. In addition, as temperature and holding time increase, the IMC layer cannot adhere to the matrix well under thermal stress and peels off more easily when rotating.

**Figure 13.** The carbides at the interface between matrix and IMC layer after dynamic corrosion. (a) interface morphology; (b) map scanning analysis of Al; (c) map scanning analysis of Fe; (d) map scanning analysis of V.

Based on the influence of temperature and time on the dynamic corrosion behaviors of SDHA steel, it can be found that dynamic corrosion consists of the evolution of the IMC layer and microstructure changes; these two behaviors interact with each other. Therefore, dynamic corrosion can be divided into three steps, as illustrated in Figure 14. Firstly, due to the interdiffusion between Fe, Si, and Al atoms at the interface, the SDHA steel is corroded by molten aluminum alloy. At the same time, the voids resulting from the Kirkendall effect appear in the IMC layer. These voids reduce the binding force of IMCs and make them peel off easily. As corrosion proceeds, the vanadium carbides contact molten aluminum alloy, marking the second step of dynamic corrosion. Due to superior corrosion resistance in molten aluminum alloy, the vanadium carbides can effectively resist the invasion of molten aluminum alloy and protect the matrix. However, when the vanadium carbides are captured in the IMC layer, the embedded vanadium carbides lead to crack initiation and propagation, promoting the fragmentation and exfoliation of IMCs. In addition, the expansion difference between the matrix and the IMCs causes cracks at the interface. These cracks accelerate the spalling of the IMC layer and reduce the atomic diffusion time and further aggravate the dynamic corrosion. Al and Si atoms preferentially react with the Fe atoms at grain boundaries to cause intergranular corrosion, which is defined as the third step of dynamic corrosion. The diffusion of atoms in a specific direction causes severe exfoliation of the matrix and increases dynamic corrosion rates.
5. Conclusions

In this study, the dynamic corrosion behaviors of SDHA steel in molten aluminum alloy and the formation of IMCs were investigated. The results obtained are drawn as follows:

(1) During static immersion, the thickness of the IMC layer increases with increasing temperatures and holding times. The IMCs are identified as the \( \text{Al}_8\text{Fe}_2\text{Si} \) phase during corrosion. At temperatures from 650 °C to 800 °C, the kinetic constants of \( \text{Al}_8\text{Fe}_2\text{Si} \) increase from 3.73 \( \mu \)m\(^2\)/min to 19.89 \( \mu \)m\(^2\)/min. The growth activation energy of the \( \text{Al}_8\text{Fe}_2\text{Si} \) phase is calculated to be 89 kJ/mol.
(2) The dynamic corrosion rate increases with an increase in corrosion temperatures; the dynamic corrosion rate is highest at 800 °C with a value of 12.67 g/(cm²·min). At 800 °C, the diffusion reaction is preferentially carried out at the grain boundary. The formed IMCs with a poor bonding force lead to the spalling of the matrix under scouring force.

(3) Vanadium carbides in matrix with a lower dissolution rate can effectively inhibit the diffusion of Al and Si atoms. Additionally, a schematic diagram of the dynamic corrosion mechanism in molten aluminum alloy was proposed.

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