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Study of corrosion mechanism of cast iron in molten aluminum

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Abstract

The corrosion mechanism of three aluminum ingot mold materials used by Chinese aluminum electrolytic enterprises in molten aluminum was studied: The three materials are pearlite cast iron (PCI) contains vermicular graphite, lamellar graphite, and spherical graphite respectively. After 800°C molten aluminum corrosion for 4 h, the intermetallic compound (IMC) layers of Fe2Al5 and FeAl3 is formed at the interface. It was found that the volume of spherical graphite in IMC was reduced to 20%, and lamellar graphite aggregated in the IMC layer. The average thickness of the Fe2Al5 layer in PCI1, PCI2 and PCI3 is about 180 μm, 100 μm and 101 μm, respectively. The weight loss rate of PCI1 is 2.57 (mg cm−2)min−1, PCI2 is 2.20 (mg cm−2)min−1, and PCI3 is 2.24(mg cm−2)min−1. There are dispersed Cr and Mo carbides in the IMC layer. Si-rich ribbon-shaped region was found at the interface between cast iron and IMC. The needle-like precipitates (Al4C3) were observed only at the only at the boundary between cast iron and IMC in PCI1.

1. Introduction

Aluminum has high specific strength and good formability, and is widely used in aerospace, automotive, construction and other fields [1–4]. However, the aluminum liquid has high chemical activity [5–7] and is easy to corrode aluminum suction tubes, aluminum ingot molds, crust breaker, and forming molds, etc. The composition produced by Fe and Al usually has a rough interface, which causes the workpiece of the aluminum product to adhere to the surface of the mold [8, 9]. And the exfoliated compounds as impurities seriously affect the quality of aluminum or aluminum alloy products. The corrosion process of Fe-based materials by high-temperature aluminum liquids is usually having two steps. First, aluminum reacts with Fe-based materials to form Fe-Al intermetallic compound (IMC) layers such as Fe2Al5 and FeAl3. Second, Fe-Al intermetallic layer peeling off. Cast iron is widely used in aluminum electrolysis, casting, molding and other production processes due to its high cost-effectiveness, stable mechanical properties [10], and good aluminum liquid corrosion resistance [11].

Bouché K et al [12] studied the corrosion behavior of Armco iron (99.78%) in a 700–900°C aluminum bath. The corrosion products are Fe2Al5 and FeAl3, and the interface between the Fe2Al5 phase and iron is extremely irregular. Shahverdi H R et al [13] found that Fe2Al5 is always the main producing phase in Fe-Al compounds and takes precedence over FeAl3. Because Fe2Al5 is the orthorhombic cell structure, there are a large number of vacancies in the c-axis, and Al is more likely to diffuse along these vacancies. Hwang S H et al [14] researched the corrosion of carbon steel (C content 0.2–1.1 wt%) in liquid aluminum at 660°C. It is concluded that the thickness of the Fe-Al compound layer decreases as the carbon content increases. Awan G H et al [15] discussed the effect of different Si content in molten aluminum on the thickness and morphology of Fe-Al intermetallic compounds. In the pure aluminum solution, a thicker layer of Fe2Al5 compound is formed on the surface of the steel, and the compound form as ‘fingers’ to matrix growth. When the Si content in the aluminum liquid is large, the compound layer are thin and flat. Lin M B et al [30] found that in the hot-dip aluminized process of high-silicon ductile iron, the coating layers are Al, Fe-Al compound, and Si-rich in order from the outside to the inside. The Fe-Al compound layer contains a large amount of tongue-like Fe2Al5 and a few rod-like FeAl3, and no Fe-Al-Si phase exists, the existence of the Si-rich layer effectively prevents the growth of the compound layer.
to the matrix. Huang Y X, Wan L [16] have studied the microstructure and properties of friction stir lap welding Al/steel joint with ultrastrong interface. A localized Si enrichment region was found at the Al-Fe interface and the growth of the IMC layer was suppressed. Therefore, an ultrathin interfacial layer appeared. It comprises a Fe₄Al₁₃ layer, a single Fe₂Al₅ layer, a Fe-rich layer and a chaotic layer. In their other research, Wan L, Huang Y X [17] studied AA6061 aluminum alloy and AISI 316 L steel friction welding and the formation of FeAl and FeAl₃ intermetallic compound (IMC) layer. The thickness of IMC layers declined from 4 to 0.2 μm with friction time decreasing from 40 to 10 s, while a 15 welding groove machined on the end of steel helped realize thinning of IMC layer to a thickness of 0.3 μm. Balloy D et al [10] studied the corrosion mechanism of unalloyed steel (UAS), ferritic cast iron (FCI) and pearlite cast iron (PCI) in aluminum liquid and found that Al has the largest diffusion depth in UAS. The flake graphite in FCI and PCI can effectively prevent the diffusion of Al. Thin layers rich in Al and Si appear between the PCI matrix and Fe₂Al₃, effectively preventing element diffusion.

In this paper, the corrosion behavior of three aluminum ingot mold materials used by Chinese aluminum electrolytic enterprises in molten aluminum were studied, the weight loss rate of three kinds of materials were obtained, the vacancies, needle-like precipitates, Si-rich regions and Cr, Mo carbides in the compound layer during corrosion were discussed.

2. Materials and methods

2.1. Materials

Three kinds of cast iron were used as experimental materials. The three kinds of cast iron are all aluminum ingot mold materials used by Chinese aluminum electrolytic enterprises. Their chemical composition (wt%) are listed in table 1. The scanning electron microscope (SEM) images of the three types of cast iron microstructures are shown in figures 1(a)–(c). Figures 1(d)–(f) corresponds to the rectangular areas A, B, C in figures 1(a)–(c), respectively. All three kinds of cast iron are pearlitic cast iron. The pearlite structure is granular in PCI1 and layered in PCI2. At some grain boundaries of PCI3, eutectic Cr, Mo carbide precipitates were observed and showed a typical lattice structure [18].

The corrosion samples and machined into a cylinder of diameter 8 mm and height 8 mm. Samples were ground to the average roughness of the surface less than 3.2 μm and rinsed by ultrasonic bath with ethanol. Aluminum ingot was divided into small pieces with an average mass of 30 g and descaled by 5% NaOH solution respectively [19] and washed by ethanol before melting.

Table 1. Chemical composition of the materials (wt%).

| Material | C  | Cr | Ni | Mn | Si | Mo | Cu | Ti | Zn | Al | Fe |
|----------|----|----|----|----|----|----|----|----|----|----|----|
| PCI1     | 3.55 |    |    |    | 0.58 | 2.17 |    |    |    |    | Bal. |
| PCI2     | 3.20 | 0.36 |    |    | 0.16 | 2.50 |    |    |    |    | Bal. |
| PCI3     | 2.35 | 1.35 |    |    | 0.97 | 2.21 | 0.40 |    |    |    | Bal. |
| Al       |    |    | 0.03 |    |    | 0.25 | 0.05 | 0.03 | 0.05 |    | Bal 0.35 |
3. Methods

In this research, heating equipment is a 5 kW well-type resistance furnace. A schematic diagram of the corrosion experiment is shown in figure 2. The entire experiment was carried out in an air atmosphere at room temperature (25 °C). The aluminum pieces was heated to 800 °C in alumina crucible by well-type resistance furnace and held for 30 min. As the molten aluminum reaches the predetermined temperature, the cylinder corrosion sample was placed into molten aluminum immediately and kept for 4 h. The aluminum crucible was taken out of the furnace and cooled at room temperature (25 °C).

3.1. Characterization

The corrosion sample was cut perpendicular along the diameter of the bottom surface. The morphology of the cross section of corrosion samples was observed by Scanning electron microscope (SEM: FEI SIRION, Netherlands). Main element contents of key points and concentration profiles of selected path were measured by Energy Dispersive Spectrum (EDS: GENESIS 7000).

4. Results and discussion

4.1. Microstructure of intermetallic compound

The microstructure of the sample after corrosion of molten aluminum is shown in figure 3. The darkest area is solid Al, the middle is IMC, and the brighter is cast iron. There are many free rod-shaped Fe-Al IMCs in solid aluminum [10]. Also, graphite is present in IMC and solid aluminum in corrosion samples. The graphite in the solid Al area of the PCI3 corrosion sample remains spherical, which is same as the graphite morphology in cast iron. In the PCI1 corrosion samples, the morphology of graphite in Al and IMC is completely different.
graphite in solid aluminum is linear and tends to be perpendicular to the IMC interface. Spheroidal graphite exists only in cast iron and IMC layers, and its volume is significantly reduced. Ten spherical graphites were selected from each of the cast iron and the IMC layer. Use Photoshop software to count the pixels of graphite spheres. It is found that the ratio $r$ of spherical graphite to spherical graphite in cast iron in the IMC is about $0.34$, that is, the cross-sectional area ratio of the graphite sphere is $r \approx 0.34$. Because the graphite is spherical, its volume ratio $R = r^3$ is about $0.20$. In the PCI2 sample, graphite aggregates in the IMC layer and is dispersed in solid aluminum. It can be observed that the spheroidal graphite and lamellar graphite forms a graphite barrier that hindered the growth of the IMC.

As shown in figure 3, the IMC has two layers (IMC1, IMC2). This is similar to the morphology of the IMC layer after corrosion of pure iron in molten aluminum in other studies [4, 12, 13]. The IMC1 layer is thinner and its interface with Al (Interface 1) has a sawtooth shape. The IMC2 layer interface with the cast iron (Interface 2) is tongue-shaped. The interface between IMC1 and IMC2 (Interface 1–2) is flatter. Although the IMCs of three samples have similar morphology, the thicknesses are different.

The top-view SEM images of corrosion samples are shown in figure 4. The surface morphology of three cast iron samples corroded by molten aluminum were irregular, but generally corresponded to the morphology of the FeAl3 layer in figure 3. Compared to PCI2 and PCI3, the surface of PCI1 is relatively flat, and there are some granular IMCs on the surface. There is a large block IMC on the surface of PCI2. The surface of PCI3 is covered with granular compounds, and the particles are small. The surface morphology of the three corrosion samples is due to the long time of immersion in molten aluminum, and the surface of FeAl3 near the Al side has sufficient time to form and join together. The raised granular and bulk morphology are newly generated FeAl3 at the interface between the FeAl3 layer and Al. These raised FeAl3s are more susceptible to erosion by molten aluminum and peel off, which ultimately leads to a reduction in the weight of the corroded samples.

In figure 5, the IMC dimensions of the three samples were measured. The thickness of IMC1 layer of three samples was almost the same. The tongue-shaped region of the IMC2 layer of the PCI2 sample is thin and wide, while the tongue-shaped region of the PCI1 sample is thick and narrow. The average thickness of the Fe2Al5 compound layer of PCI1, PCI2 and PCI3 is about 180 $\mu$m, 100 $\mu$m and 101 $\mu$m, respectively.
seen that under the same corrosion conditions, PCI1 has the highest weight loss rate, which is at 2.57 (mg/cm²) min⁻¹. PCI2 is at 2.20 (mg/cm²) min⁻¹, PCI3 is at 2.24 (mg/cm²) min⁻¹. This is because the first process of corrosion of cast iron by molten aluminum is to generate IMC, and then the IMC peeled off. The lamellar graphite in the IMC of PCI2 is dense and has a large surface area, which acts as a barrier and prevents the growth of the IMC layer, thereby reducing the weight loss rate of PCI2 in the molten aluminum. In addition, the weight loss rate of the corrosion sample increases as the thickness of the IMC layer increases. Since the hardness of IMC generated by corrosion in an aluminum melt at 800 °C is about HV1000 [20], the fracture toughness is poor and cracks generated. The IMC layer is corroded by molten aluminum and is easily peeled off at the crack.

4.2. Weight loss rate

Figure 6 shows the average weight loss rate of cast iron samples after corroded in molten aluminum. It can be seen that under the same corrosion conditions, PCI1 has the highest weight loss rate, which is at 2.57 (mg/cm²) min⁻¹, PCI2 is at 2.20 (mg/cm²) min⁻¹, PCI3 is at 2.24 (mg/cm²) min⁻¹.

4.3. Phases composition

Corrosion samples were analyzed by EDS line scanning. The Al, Fe and Si elements are scanned in the direction of the arrows in figures 3(a), (e), (f). As shown in figure 7(a), there are few Fe and Si elements in solid Al. The Al element is in agreement with the fluctuation of the Fe element. Because the free rod-shaped Fe-Al IMC in the solidified Al layer is scanned. However, in figure 7(c), the fluctuation of the Al element does not correspond to the Fe element. This is due to the line scan path passing through the spheroidal graphite, as shown in figure 3(f). The IMC layer in figure 7 consists of two parts. At Interface1, the composition of Al and Fe elements changed drastically, Al content decreased, and Fe content increased significantly. However, in the IMC region, the Al and Fe concentrations did not change. This type of concentration change is consistent with the characteristics of reaction diffusion. Al element diffuses from the surface of the sample to its interior, and when the content of Al exceeds its maximum solubility in cast iron, Fe and Al form IMC. In figure 7(a), the concentrations of Al and Fe at Interface2 of the IMC layer are changed. The Al element content is gradually reduced, and the Fe element content is increased. There is a transitional area near Interface2 in cast iron. The Si element fluctuates significantly at Interface2 of the PCI3 sample, and there exist a Si-rich ribbon area, as shown in figure 7(c). It will be explained in detail later.

Region A and B in figure 3(a) correspond to figures 8(a), (b), 4(b) corresponds to figures 8(c), (d), and 4(c) corresponds to figures 8(e), (f). The EDS analysis was performed on the IMC layer and cast iron zone of three samples in figure 8. The composition results are shown in table 2.

Since the IMC layer is thin and varies in thickness and has significant compositional fluctuations, it does not have a definite stoichiometric constitution, making it difficult to analyze the IMC layer product by XRD [21]. Therefore, according to the EDS results in table 2, ratio of Fe-Al composition, and the research results of Cheng, W. J., Wang, C. J. [22, 23], it is considered that point 1 is FeAl₅ (θ phase), point 2 is Fe₆Al₇ (η phase), point 3 is the cast iron region where Al and Si are enriched, point 4 and 5 is Cr, Mo carbide. In the study of Gupta S P. [24], the solubility of Si in Fe-Al IMC was lower than 2 wt% between 873 K and 1073 K (600 °C to 800 °C). This result is consistent with our findings.

In figures 8(b), (d) and (f), a ribbon-shaped Al, Si element-rich cast iron area can be seen. The enrichment of Si is influenced by the growth of the Fe-Al IMC layer, which in turn affects the growth of the IMC layer. When the surface of the cast iron is in contact with the molten aluminum, Al atoms diffuses into cast iron to form an
Al-rich region. When the Al content reaches the reaction concentration, Fe-Al IMC is produced. Due to the similar atomic size of Si and Al, and the low solubility of Si in Fe-Al IMC, Si atoms are continuously extruded from the compound layer during the formation of Fe-Al IMC, and gradually enriched in the cast iron side of Interface 2 to form a rich Si ribbon area. On the other hand, the concentration gradient of Si element at Interface 2 increases, causing Si atoms to diffuse into the IMC. These Si atoms occupy the vacancies in Fe$_2$Al$_5$, increase the density of the IMC layer, hinder the diffusion of the Al atoms in Fe$_2$Al$_5$ in the c-axis direction [22], and slow down the growth rate of Fe$_2$Al$_5$ IMC. Therefore, it can be concluded from figures 8(b), (d), (f) and table 2 that as the thickness of the IMC layer increases, the Si content of the Si-rich region increases.

In figure 8(b), there are some black needle-like precipitates in the IMC and Al-Si-rich regions near the Interface 2. These precipitates were also found in the study of Balloy D. [10], which gave the assumption that these precipitates were Al$_4$C$_3$ or small flake graphite transformed by cementite in pearlite. According to the Balloy D. study, Al$_4$C$_3$ is usually present in the form of (Fe$_3$C)$_x$(Al$_4$C$_3$)$_y$ in pearlitic cast iron, and when the Al content exceeds 17%wt, Al$_4$C$_3$ decomposes into graphite. However, they are both pearlite cast iron, which were found only in PCI1. Therefore, it is considered that the graphite which produces Al$_4$C$_3$ is not produced by the decomposition of cementite in the pearlite. There is also no presence of vermicular graphite around the precipitate, as shown in figure 8(b). The reason for the formation of Al$_4$C$_3$ should be that Al atom is larger than Fe atom, the replacement of the Fe atom by the Al atom causes a lattice distortion in the region where the Al is concentrated. The change in lattice constants allows more C atoms to enter the lattice gap of α-Fe, causing supersaturation of C elements. When the C and Al contents are saturated, Al$_4$C$_3$ is produced, and because of the increasing Al content, Al$_4$C$_3$ decomposes to produce graphite [10].

In figure 8(c), voids can be observed in the IMC layer of PCI2. There are two reasons for the appearance of voids: (1) The IMC layer grows first along the direction perpendicular to the growth interface (the c-axis direction of Fe$_2$Al$_5$ [22]), and then laterally closes [25]. The molten aluminum corrodes for a long time, the interface 2 tends to be flat [26]. Due to the difference in diffusion rates of Fe and Al, the Kendall phenomenon occurs and voids are generated. [27]. (2) The reaction time is long enough. The growing IMC will bypass the flake graphite and wrap it inside. Because the lattice constants of Fe$_2$Al$_5$ and α-Fe are different, the volume of Fe$_2$Al$_5$ will become larger and stress will be generated during the growth process, so that the brittle graphite will gradually agglomerate and break, thereby generating voids.
It can be found from the proportion of the components in Table 2 that point 5 in Figure 8(f) is an enriched region of Mo and Cr carbides, in which the Mo content is particularly high, which is also present in the sample before corrosion, as shown in Figure 1(f). Mo and Cr carbide-rich regions in the cast iron which were not in

![Figure 8. SEM image of the microstructure of cast iron corrosion samples. (a) (b) PCI1; (c) (d) PCI2; (e) (f) PCI3.](image)

**Table 2. Average Chemical Composition of the Phases Present in Figure 8 (EDS Measurements).**

| Material | point | Al   | Fe   | Si   | Cr  | Mo  | C    | Phase                                      |
|----------|-------|------|------|------|-----|-----|------|--------------------------------------------|
| PCI1     | 1     | 77.02| 21.82| 1.17 |     |     |     | FeAl3                                      |
|          | 2     | 70.3 | 28.45| 1.25 |     |     |     | FeAl3                                      |
|          | 3     | 26.98| 69.63| 4.39 |     |     |     | Cast iron with Al-Si rich                 |
| PCI2     | 4     | 76.89| 22.86| 0.25 |     |     |     | FeAl3                                      |
|          | 5     | 73.3 | 26.25| 0.45 |     |     |     | FeAl3                                      |
|          | 6     | 20.99| 75.87| 3.14 |     |     |     | Cast iron with Al-Si rich                 |
| PCI3     | 7     | 78.85| 19.86| 0.92 | 0.22| 0.15|     | FeAl3                                      |
|          | 8     | 72.62| 25.21| 1.39 | 0.41| 0.37|     | FeAl3                                      |
|          | 9     | 59.19| 15.18| 0.89 | 2.37| 12.24| 10.13| Fe₃Al₅ with Cr,Mo carbide                  |
|          | 10    | 21.06| 74.84| 2.47 | 0.87| 0.76|     | Cast iron with Al-Si rich                 |
|          | 11    | 0.54 | 23.7 | 3.04 | 1.64| 16.05| 55.03| Cast iron with Cr,Mo carbide              |
direct contact with the molten aluminum and it’s did not change significantly after 4 h of corrosion. However, in point 4 of figure 8(e), it can be seen that a dotted strip-shaped region appear, and the EDS results indicate that the region is the Fe2Al5 compound enriched by Mo and Cr carbides. The Mo and Cr carbides are embedded in the compound, and the distribution is more dispersed than point 5. According to the research theory of Zhang X. and Chen W. [18, 28], it can be inferred that during the corrosion process, Al atoms continuously diffuse into the matrix and reacts with Fe in the grid-like Mo and Cr carbide regions to produce Fe2Al5. The carbides of Mo and Cr have good stability and are not easily reacted with Al, so they are surrounded by Fe2Al5. This kind of surround is not only in the physical sense, but also metallurgical bonding occurs, and the Al-Mo metal bond is generated and it’s bonding force is strong [29]. Moreover, due to the different lattice constants between Fe2Al5 and α-Fe, the volume of Fe2Al5 grows larger, resulting in more dispersion of Cr and Mo carbides relative to the original position. Further, as shown in figure 8(f), in the Al-Si-rich region in the cast iron, Mo and Cr carbides are also scattered. This may be due to the influence of high temperature and elemental diffusion during the corrosion process, which increases the vacancy density and exacerbates the segregation of Mo and Cr in the cast iron, promotes the combination of Mo, Cr and C, and precipitates Mo and Cr carbides. The Cr and Mo carbides dispersed in the IMC and near the interface prevents the diffusion of Al, reduce the thickness of Fe2Al5. These scattered Cr, Mo carbides pin the IMC layer, inhibiting the peeling of the IMC layer and reducing the corrosion rate. The IMC thickness of the PCI3 is significantly lower than that of the PCI1 figure 5, while the ribbon-shaped Si rich area in the PCI3 is also narrower than in the PCI1 figure 8. This indirectly indicates that the presence of Cr and Mo carbide in cast iron is helpful in resisting corrosion of molten aluminum.

5. Conclusion

(1) During the corrosion of cast iron in molten aluminum, the graphite in the cast iron acts as a barrier to prevent the growth of intermetallic compounds. The appearance of intermetallic compounds has no effect on the spheroidal graphite, but causes the lamellar graphite to aggregate.

(2) After 4 h of corrosion in molten aluminum at 800 °C, the average thickness of the Fe2Al5 compound layer of PCI1, PCI2 and PCI3 is about 180 μm, 100 μm and 101 μm, respectively. The weight loss rate of PCI1 is 2.57 (mg/cm2) min⁻¹, PCI2 is 2.20 (mg/cm2) min⁻¹, and PCI3 is 2.24 (mg/cm2) min⁻¹.

(3) A Si-rich ribbon-shaped region appears at the interface between the cast iron and the compound, and reduces the growth rate of the Fe-Al IMC layer.

(4) The needle-like precipitates appears at the boundary between the cast iron and the Fe-Al IMC in PCI1, which are graphite produced by decomposition of Al4C3. This precipitates are not found in the other two cast irons.

(5) Voids are found in the IMC layer of PCI2 sample, which are formed by the Kirkendall phenomenon or graphite collapse.

(6) Disperse Cr and Mo carbides appear in the compound layer. These Cr, Mo carbides provide protection for the Fe2Al5 IMC layer and reduce the peeling of the Fe2Al5 compound.

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