The study on corrosion behavior and corrosion resistance of ultralow carbon high silicon iron-based alloy

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Abstract

The ultralow carbon high silicon iron-based alloy (sample A) with a single ferrite matrix was prepared. The corrosion performance difference and mechanism between it and conventional high silicon cast iron (sample B) were systematically studied using optical microscope, scanning electron microscope, electric corrosion equipment, electrochemical corrosion workshop and atomic force microscope. The results show that the corrosion rate of ultralow carbon iron-based alloys was significantly lower than that of high-silicon cast iron. For example, the static corrosion rate after 310 h, the corrosion rate of sample A was 0.2399 g/(m²·h), and the corrosion rate of sample B was 1.5159 g/(m²·h). The potential difference of the substrate was significantly reduced. That is, the ultralow carbon iron-based alloy exhibits better corrosion resistance, which is mainly attributed to the denser passivation film formed on its surface. The results of microhardness show that the hardness of Sample A was higher than that of Sample B.

1. Introduction

High-silicon iron-based alloy is a low-cost alloy composed of Fe, Si, and C elements, and has excellent corrosion resistance. It is widely used to manufacture various corrosion-resistant equipment, such as pumps, anode castings, reaction apparatus and compressors etc [1–10]. Silicon is the main corrosion-resistant element. With higher content from Si, corrosion resistance is bigger. Generally, the silicon content of high silicon iron-based alloy is 14%–16%. On the one hand, silicon can reduce the potential difference between ferrite and graphite by increasing the potential of ferrite and protect the substrate [1]. On the other hand, Si has a good affinity for oxygen, so a dense protective film of SiO₂ will be formed on the surface of high silicon iron-based alloys under certain corrosion conditions [2–5]. Corrosion resistance improved due to the substrate and the corrosion environment is separated by passivation film. With the increase of silicon content, the passivation film is more compact and complete, and the corrosion resistance of the alloy is further improved.

However, the mechanical properties of high silicon iron-based alloy are proportionally decreased as silicon content increases. Besides, there are a large number of flake graphite structures in the ordinary high silicon cast iron due to the high silicon content. The morphology, quantity and distribution of graphite have an important influence on the corrosion behavior of high silicon iron-based alloys [11–15]. It has been reported that the center A-type and edge D-type graphite exhibit the highest corrosion resistance [12]. Titanium has the characteristics of promoting the formation of D-type graphite in high silicon cast iron alloy [13]. Magnesium has the property of rounding the graphite tips [14]. Rare earth elements can improve the shape and distribution of graphite and refine the microstructure, consequently improving the corrosion resistance [14]. Copper can also refine graphite and improve its corrosion resistance in sulfuric acid. However, there are relatively few studies on the corrosion resistance performance reduce carbon content to avoid graphite precipitation in high silicon iron-based alloys.
Therefore, the project prepared a high silicon iron-based alloy with a single ferrite matrix, and systematically studied the difference and mechanism of corrosion performance between it and conventional high silicon cast iron. The research of this project is expected to promote the industrial application of ultralow carbon high silicon iron-based alloy, especially in components with high requirements for corrosion resistance, such as anodes in industrial stainless steel pickling baths.

2. Experimental

2.1. Materials
In this work, the ultralow carbon high silicon iron-based alloy and conventional high silicon cast iron were used to be studied. The chemical compositions of the experimental alloys were presented in table 1, tagged as A and B. The chemical compositions design were based on the reason that the existence of graphite in the high silicon cast iron, which leads to the number of corrosion micro batteries in the matrix increases, the corrosion resistance deteriorates. Therefore, ultralow carbon high silicon iron-base alloy with the single ferrite structure was obtained by reducing carbon content.

2.2. Microstructure
The microstructure and morphology of samples were observed by ZEISS AX10 optical microscope and FEI QUANTA FEG 450 field emission environmental scanning electron microscope. The elemental distribution was investigated using the equipped energy dispersive spectroscopy (EDS). The metallurgical and SEM samples were cut from the casting using a wire cutting machine and were eroded by 10% FeCl₃ solution after being ground and polished. The surface roughness of each sample was measured with a laser scanning confocal microscope (LSCM). Phase identification was carried out by x-ray diffraction (XRD, MXP21VAHF instrument using Cu Kα radiation at a voltage of 40 kV and a current of 130 mA). The XRD samples were cut into plates with 10 mm × 10 mm in size, and the surfaces were milled on sandpaper from 200# to 2000#. X-ray diffractometer was used to scan the angular 2θ ranged of 20 to 100 deg at a scanning speed of 10 deg · min⁻¹. Microhardness tester (THV-1MD) was used to characterize their properties. The surface potential distribution of samples was detected by scanning Kelvin atomic probe force microscope (SKPFM).

2.3. Corrosion test
The static corrosion sample (Φ20 mm × 5 mm) was processed by a wire cutting machine. The sample was ground, then washed in distilled water and acetone, dried and weighed. The total surface area of the sample was calculated. Periodically, the samples were removed from the 8.5% nitric acid solution, brushed and cleaned completely to remove any loose corrosion products, and then weighed to obtain the corrosion rate. The average value of three experimental values was used to evaluate the uniform corrosion resistance.

The ultralow carbon high silicon iron-based alloy developed in this work may be applied to the anode plate of the pickling line in steel mill. In order to simulate the field condition, the loading current corrosion experiment was carried out. Figure 1 shows the schematic diagram of the device. The specimen (25 mm × 10 mm × 5 mm) used to simulate the field condition was obtained with the wire cutting machine. The sample was anode, which was linked to the positive pole of the power supply, and the 430 stainless steel was cathode, which was linked to the negative pole of the power supply. The dimensions of the cathode were 40 mm × 20 mm × 5 mm. The current density of the sample can be changed by adjusting the DC power supply, and the temperature of the electrolyte can be changed by adjusting the temperature of the water bath. The specimen was immersed in a dissolving tank with 8.5% nitric acid solution, and the dissolving tank was placed in a thermostat water bath of 55 °C, and the current density was set to 30 A dm⁻² to simulating the field application condition. The specimens were removed periodically from the 8.5% nitric acid solution, brushed in distilled water to remove any loose corrosion products, dried and then weighed on the precision balance. The corrosion rates of static corrosion and loading current corrosion determined in weight loss method [16, 17] can be expressed as follows:
D = \frac{t}{m \cdot \Delta m} \int m_{0} - m_{t} \int S \int t

(1)

Where \( \Delta m \) is corrosion rate (\( \Delta m \) in g/(m\(^2\)·h)), \( m_{0} \) and \( m_{t} \) indicated that weight before corrosion and after corrosion (\( m_{0} \) and \( m_{t} \) in g), respectively. And \( t \) is the exposure time (in h), \( S \) is surface area, (in m\(^2\)). Note that the surface area of the sample refers to the area in contact with the nitric acid solution.

2.4. Electrochemical tests

The polarization curve measurements were performed with an RST5000 electrochemical workstation in a three-electrode system. High silicon iron-based alloy, saturated calomel electrode and Platinum plate were used as working electrode, reference electrode and auxiliary electrode, respectively. In electrochemical tests, the scale of the sample was 10 mm \( \times \) 10 mm \( \times \) 3 mm. The sample was encapsulated with epoxy resin, and the working area was 10 \( \times \) 10 mm\(^2\). The electrochemical specimen was ground with sandpaper and then washed with distilled water and ethanol. The samples were immersed in an electrolytic cell containing 3.5% NaCl solution for electrochemical tests to obtain the polarization curve and the corresponding corrosion results [18, 19]. The initial potential, the termination potential and the potential scanning rate were \(-1.5 \text{ V}, 1.5 \text{ V} \) and 1.25 mv s\(^{-1}\), respectively. Electrochemical impedance spectroscopy (EIS) measurements were conducted over a frequency range from 100 kHz to 10 mHz with an amplitude of 10 mV. The EIS data were analyzed by the software ZView 2.

3. Results and discussion

3.1. Microstructure

The microstructure of test samples shown in figure 2 was observed with an optical microscope (OM) and scanning electron microscope (SEM). It can be seen that the matrixes of specimens were all ferrite, as shown in figures 2(a), (b). Since the carbon content of specimens is very low, it can be approximately considered as Fe–Si alloy. According to the phase diagram of Fe–Si, When Si reaches 5%, the austenite tends to disappear [20]. Silicon is an element which makes smaller \( \gamma \) area of ferrum [2]. However, there were obvious differences in the morphology of the two materials. The microstructure of ultralow carbon high silicon iron-base alloy (A) was a single ferrite matrix without graphite (figure 2(a)), while the structure of high silicon cast iron (B) was ferrite matrix and graphite (figure 2(b)). The graphite was distributed in the crystal and the grain boundary as spherical, spotty and small petals (figure 2(d)), which is due to the promotion of graphitization by Cu element [15]. In sample B, there were also phases containing copper around the grain boundaries (figure 2(f)). Compared with the grain size of conventional high silicon cast iron, ultralow carbon high silicon iron-base alloy was obviously larger. The grain sizes of samples A and B were about 62~340 \( \mu \)m and 40~160 \( \mu \)m, respectively. In order to observe the phase change in the microstructure of samples, x-ray diffraction (XRD) was conducted on specimens, as shown in figure 3. There is a set of characteristic diffraction peaks of Fe\(_3\)Si phase are present in both specimens. Moreover, the characteristic diffraction peaks of the Cu\(_{15}\)Si\(_4\) phase is observed in sample B [15, 21].
3.2. Corrosion resistance
The results of the static corrosion rate of samples were calculated by weight loss method in 8.5% nitric acid solution are shown in figure 4. With the same condition, the static corrosion rate of sample A soaking in 8.5%
nitric acid solution for different times were significantly lower than that of sample B, which indicates that its corrosion resistance to nitric acid is significantly better than that of high silicon cast iron.

There are two reasons why the static uniform corrosion rate of sample A was significantly lower than that of sample B. On the one hand, the microstructure of sample A was a single ferrite matrix without graphite and homogeneous microstructure. On the other hand, there was copper around the grain boundaries of sample B. Copper can react with the diluted HNO₃, which can be confirmed by the appearance of the blue corrosion product. The blue corrosion product was copper nitrate. The reactive mode is:

\[ 3Cu + 8\text{HNO}_3\text{(diluted)} = 3\text{Cu(NO}_3)_2 + 2\text{NO} + 4\text{H}_2\text{O} \]  

In the above reaction, the copper reacts with the diluted HNO₃ and forms copper nitrate, which dissolves in solution. Therefore, the corrosion channel was formed due to the leaching of copper at the grain boundaries, leading to continuous corrosion and increasing corrosion rate.

Figure 5 shows the relation curve between the corrosion rate and the corrosion time of the samples through the above-mentioned loading current corrosion device. The corrosion rate of specimen A decreased with the increase of time. When the corrosion time was 1 ~ 3 h, the corrosion rate of sample B decreases obviously. The corrosion rate of sample B tended to equilibrium after 5 h, while the corrosion rate of sample A basically tended to equilibrium after 3 h. It can be found that the corrosion rate of sample A was lower than that of sample B at different times, and the difference of the corrosion rate gradually decreases with the extension of time in the study period. The major factor of sample A has good corrosion resistance was ultralow carbon microstructure without graphite. Due to the elimination of graphite, the quantity of micro-battery in the matrix was reduced, the corrosion resistance increased.

![Figure 4. The results of the static corrosion test.](image)

![Figure 5. The change of corrosion rate of samples with time.](image)
Results of potentiodynamic polarization curve of ultralow carbon high silicon iron-base alloy (A) and high silicon cast iron (B) in 3.5% sodium chloride solution at 25 °C are shown in figure 6. In order to eliminate the influence of experimental parameters on the polarization curve, the same experimental parameters were used. It can be seen from the figure that although the corrosion modes of the two samples are different, the anodes of the samples show obvious passivation behavior. The current does not increase with the increase of the potential when the potential exceeds about 500 mV, indicating that an effective SiO$_2$ corrosion product layer is formed on the metal surface. Table 2 shows the fitting results of the samples. The corrosion potential of sample A and sample B were $-0.314$ V and $-0.440$ V, respectively. The corrosion potential of sample A larger than that of sample B, which indicates that sample A is not easy to corrode compared with sample B. The corrosion current densities of sample A and sample B were $0.513 \times 10^{-6}$ and $1.676 \times 10^{-6}$ A cm$^{-2}$, respectively.

According to Faraday law, the relationship between corrosion rate and corrosion current density is as follows [22]:

$$
\nu = \frac{MI_{corr}}{nF}
$$

Where $\nu$ is the corrosion rate, $M$ is the atomic weight of the corroded metal, $n$ is the chemical valence state and $F$ is the Faraday constant. The corrosion rate is proportional to the corrosion current density. The corrosion current density of sample A less than that of sample B, which indicates that sample A is not easy to corrode compared with sample B. The corrosion current densities of sample A and sample B were $0.513 \times 10^{-6}$ and $1.676 \times 10^{-6}$ A cm$^{-2}$, respectively.

The results of potentiodynamic polarization curve are concordance with the results of weight loss.

The EIS plots, as seen in figure 7, have been obtained to explore the mechanisms of corrosion on the electrode/electrolyte interface. Figure 7(a) shows the Nyquist diagram and the fitted equivalent electrical circuit of the two samples. There is only one capacitive reactance arc on EIS. The radius of the semicircular arc is related to the polarization resistance of the passive film [23]. The larger the capacitive arc radius, the greater the resistance to charge transfer and the stronger the corrosion resistance [24]. From figure 7(a) in comparison with sample B, the sample A provided the larger diameter which demonstrated the sample A has better corrosion resistance and low corrosion rate. In the equivalent circuit diagram, $R_s$ is the solution resistance, CPE is the constant phase element, and $R_t$ signifies the charge transfer resistance. The fitting result is shown in table 3. Compared with high silicon cast iron, $R_t$ of high silicon iron-based alloy has increased by 132.9%, indicating that the passivation film has higher density and stronger adhesion between the passivation film and the substrate.
Therefore, the passive film of sample A better prevents the penetration of \( \text{Cl}^- \) and reduces the contact between \( \text{Cl}^- \) and the substrate, thereby reducing the corrosion rate.

Figure 7 (b) shows the Bode magnitude plots and Bode phase angle plots for the two samples. It can be seen from figure 7 (b), the value of impedance modulus \(|Z|\) of the sample A is higher than that of sample B at low frequency. At 100 mHz, the impedance modulus of sample A is 17 276 \( \Omega \), and the impedance modulus of sample B is 12 626 \( \Omega \), which further proves that the electrode reaction resistance of the high silicon iron-based alloy passivation film is more larger, that is, the corrosion rate is lower.

3.3. Hardness

Figure 8 shows the microhardness of the two samples. The hardness of sample A and sample B are 556.8 HV0.5 and 500.7 HV0.5, respectively. The hardness of sample A is greater than that of sample B, and the mechanical properties of sample A are slightly lower. Although sample B has higher carbon content than sample A, sample B contains a higher copper content. Studies have shown that appropriate copper content reduces the hardness of high-silicon cast iron [25]. Therefore, the hardness of sample B is relatively low.

3.4. Corrosion products analysis

In order to study the reasons for the difference of corrosion resistance between ultralow carbon high silicon iron-base alloy (A) and high silicon cast iron (B), the corrosion morphology and EDS analysis of the samples corrosion for 11 h under the condition of 55 °C electrolyte temperature, 8.5% nitric acid solution and 30 A dm\(^{-2}\) current density was investigated, as shown in figure 9. The cross-section of the samples, as shown in figure 9. These SEM micrographs were obtained by scanning the surface of the oxidized layer and its cross-section using a field emission scanning electron microscope. It can be seen from figures 9(a) and (b) that the product film on the surface of sample A is relatively compact and homogeneous, and the protection of the material is better. It can be seen from figures 9(c) and (d) that the corrosion product film on the surface of the high silicon cast iron was clearly divided into small pieces and broken seriously, so the protection of the material is weak. The reason is due to the formation of a galvanic couple between graphite and matrix, electrochemical corrosion. The EDS analysis of the product film is SiO\(_2\) passivation film. (figure 9(c)). Ferrum atoms excretes from silicon-ferritic lattice and residual Si atoms are oxidized so the form protection film of silicon oxide which stops further corrosion [2]. High silicon cast iron can easily form a SiO\(_2\) passivation film to protect material because the free formation enthalpy of chemical Si-O binding is much more negative than for Fe–O [26]. At the same time, it can be seen from the figure that there are obvious cracks on the surface oxide film of two samples, which were caused by the function of the electric field [15]. As noted in figure 10, the oxide film of sample A was denser than that of the sample B. Compared with the sample B, the oxide film of sample A can better protect the substrate, indicating that the properties of sample A oxide film is better than that of sample B. Therefore, the
Figure 8. Hardness of the sample.

Figure 9. The surface morphology of the samples (a), (b) A and (c), (d) B and EDS analysis of (e) Point A.
elimination of graphite is beneficial in improving the quality of oxide film and enhancing the corrosion resistance. It is worth mentioning that the SiO₂ passivation film plays an important role in corrosion resistance.

The three-dimensional morphology of the oxide film on the sample surface is shown in figure 11. As shown in figure 11, the relative surface height of sample A was slightly lower and relatively uniform than that of sample B. The measurement indicates that the surface roughness of the two samples, after 11 h of galvanic corrosion, have obvious differences. The surface roughness of the sample B (8.453 ~ 12.078 μm) was higher than that of the sample A (4.909 ~ 7.337 μm). The reason is that the microstructure of sample A was a single ferrite matrix, and microstructure was homogeneous, so the surface oxide film was relatively compact and uniform. While the structure of sample B was ferrite matrix and graphite. The graphite on the surface of the sample may have spalled off during sample preparation, which leads to the relatively uneven surface of the sample. Therefore, the oxide film formed on the surface of sample B was relatively uneven and the surface roughness of the sample was relatively large. The microstructure of the sample had a great influence on the surface roughness of the oxide film. The surface roughness results are consistent with that measured the surface morphology of oxide film.

3.5. Potential distribution
Scanning Kelvin atomic probe force microscope (SKPFM) can detect the surface morphology and surface potential distribution. It has been reported that Kelvin probe force microscopy can be used to predict the corrosion behavior of alloy microstructure [27, 28]. In general, the potential difference rather than the absolute potential is exploited to characterize the metal activity between different regions. The larger the potential difference between regions, the easier to bring about galvanic corrosion. In order to further study the reasons for the difference of corrosion resistance between samples A and B, the surface potential distribution of the sample was detected by scanning Kelvin atomic probe force microscope (MFP-3D infinity) as shown in figure 12. The potential difference in different regions by distinguishing by using different colors. From this figure, compared with ultralow carbon high silicon iron-base alloy (sample A), the color of high silicon cast iron (sample B) in different areas of quite different. This indicates that the potential difference of sample B was greater than that of sample A. That means sample B is more prone to galvanic corrosion. As shown in figure 12(B), the larger potential difference of sample B was due to the structure of sample B was graphite and ferrite matrix. Compared with the matrix, graphite has a very low potential and is a strong cathode. Because of the existence of graphite, it
is easier to form a micro battery, which accelerates the corrosion rate of the material. This is one reason why the corrosion resistance of sample B is worse than that of sample A.

4. Conclusion

The ultralow carbon high silicon iron-based alloy with a single ferrite matrix was prepared. And the corrosion performance difference and mechanism between it and conventional high silicon cast iron have been systematically studied. Based on the results of previous experiments, we infer the following conclusions:

The results of OM, SEM and SKPFM show that sample A was a single ferrite structure with a small potential difference. The results of XRD, EDS, LSCM and corrosion tests show that the SiO₂ passivation film formed on the surface of sample A was denser than of sample B. Therefore, it can be seen that the corrosion resistance of ultralow carbon high silicon iron-base alloy was superior to high silicon cast iron. On the one hand, the uniform ferrite matrix without graphite in ultralow carbon high silicon iron-base alloy contributes to the decreased potential difference and elimination of corrosion micro-battery. On the other hand, the compact and uniform oxide film formed on the surface of ultra-low carbon high silicon iron-base alloy provides better protection for the material.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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