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Effects of Rare Earth (Ce and La) on Steel Corrosion Behaviors under Wet-Dry Cycle Immersion Conditions

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Abstract: Rare earth (RE) elements are beneficial to improving corrosion properties in low-carbon and low-alloy steels. In this paper, corrosion performance of Q235B steel and Q355B steel samples after RE alloying under wet-dry cycle immersion conditions were analyzed. Experimental results show that corrosion rate was significantly decreased. It was probably due to the grain refinement by RE alloying, which increased the density of protective rust layers and improved corrosion resistance. The formation of small-sized spherical RE inclusions also inhibited the precipitation of MnS and weakened micro galvanic corrosion. Additionally, RE atoms tended to segregate towards grain boundaries and a RE concentration region is formed between rust layers and matrix to impede the access from contacting corrosive ions. A corrosion resistance schematic of RE atom segregation was proposed based on microstructure morphology and element distribution results.

Keywords: rare earth; immersion tests; inclusion; segregation; microstructure

1. Introduction

Low-carbon and low-alloy steels have been widely used as structural materials due to their excellent engineering properties. However, when these two kinds of construction steels exposed to atmospheric corrosion environment, they are prone to be attacked and corrosion will result in potential safety problems and economic losses [1,2]. Many methods including multi-alloying, coatings and inhibitors have been investigated to decelerate the steel corrosion [3–6]. Among them, multi-alloying is an effective method to decrease destructive corrosion. Valid chemical elements such as Cu, P, Cr and Ni are proved effective in preventing atmospheric corrosion with a rising cost [3,7,8]. Considering the economic and environmental reasons, attention has been paid to the development of cheaper alloying technologies applied to materials.

In recent years, many studies have shown that rare earth (RE) elements can not only purify molten steels but also modify inclusions due to their strong affinity for oxygen and sulfur [9,10]. Inclusions are considered to have negative effects on the corrosion properties of steels [11]. The atmospheric corrosion originates from pitting corrosion and spreads to the entire surface to form uniform corrosion. It is well-known that manganese sulfide (MnS) often provides initiation sites for pitting corrosion and aluminium oxide (Al₂O₃) has also been considered to cause destructive effects on corrosion resistance of steels [12–14]. The addition of RE has been reported to capture sulfur to form RE inclusions and inhibits the formation of MnS [11,15,16]. In addition, RE element is active and probably enriched

at the interfaces such as grain boundaries or surfaces due to the big difference of atomic radius between RE and iron [17–19]. The microstructure and corrosion properties are changed because of the concentration of RE elements at grain boundaries, which affects the diffusion of other elements and restrain the nucleation and growth of new phases [20,21]. RE also segregates towards interfaces, which is considered as the micro-alloying effect, though it differs from what is commonly thought as solid solution effect.

Although many investigators have shown effects of RE elements on microstructure and inclusions in steels, few studies have focused on the influence of RE alloying such as segregation of RE atoms in steels under wet-dry cycle immersion conditions. Two common construction steels including Q235B and Q355B steels were chosen to be the experimental steels. This paper elucidates how RE increased the corrosion performance under wet-dry cycle immersion tests. Scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS), electron probe microanalysis (EPMA) and auger electron spectroscopy (AES) were conducted to investigate the mechanism of improving corrosion resistance by the method of RE alloying.

2. Experimental

Two kinds of steels were selected as experimental samples marked as Q235B and Q355B. A certain amount of RE elements (Ce and La) were added into these two modified steels and were marked as Q235BM and Q355BM. The alloys were melted into ingots and then hot rolled into steel plates (300 mm × 1000 mm). The average chemical composition of final experimental steels is listed in Table 1.

Table 1. Chemical composition of experimental steels (wt%).

Sample	C	Si	Mn	P	S	Cr	Al	Cu	Ce	La
Q235B	0.12	0.09	0.34	0.019	0.009	0.024	0.022	0.023	-	-
Q235BM	0.12	0.08	0.34	0.020	0.010	0.023	0.022	0.023	0.018	0.0065
Q355B	0.14	0.23	1.15	0.020	0.007	0.023	0.039	0.020	-	-
Q355BM	0.13	0.23	1.15	0.020	0.007	0.023	0.039	0.020	0.011	0.0041

Three parallel samples (45 mm × 55 mm × 3 mm) were prepared for calculating the rate of corrosion weight loss under wet-dry cycle immersion tests. Immersion tests were undertaken at 45 °C in a solution of 0.01 mol/L NaHSO₃. One immersion cycle included 15 min of infiltration and 45 min of exposure. The test durations were determined as 24 h, 48 h, 96 h and 144 h. Before immersion tests were conducted, the specimens were cleaned ultrasonically and weighed in a dry environment. Part of rust was scraped off for X-ray diffractometer (XRD) analysis. After corrosion products were removed, the specimens were weighed to determine the mass loss. The rest rust on the surface were chemically moved in a specific solution (500 mL HCl + 500 mL distilled water + 3.5 g hexamethylenetetramine). After corrosion products were removed, the specimens were weighed to determine the mass loss.

Metallographic images were observed by optical microscopy (OM). The morphology of inclusions was observed using a thermal field scanning electron microscopy (SEM) and combined with energy dispersive X-ray spectroscopy (EDS). The surface micro-corrosion morphology of samples after 144 h of immersion tests was observed. Cross sections of the specimens were mounted in epoxy resin and sprayed with gold. Electron Probe Microanalysis (EPMA) was determined to quantitatively measure chemical composition of phases and element distribution of rust layers. The impact specimens of Q235BM and Q355BM were subjected to an impact test to obtain a brittle fracture at the temperature of −20 °C. The in-situ scanning Auger Electron Spectroscopy (AES) was used to analyze the change of RE concentration at different sputtering depths from the fracture surface. The ion beam with the energy of 2 keV covered an area of 2 mm × 2 mm and sputtered at intervals of 0.5 min.

3. Results and Discussion

Figure 1 displays the change of surfaces morphology during immersion tests. It is observed that double rust layers are formed on the surfaces of these steels. The outer rust layer is a porous rufous or tawny solid, while the inner layer is a dense black solid. It is found that more pores and local exfoliating of outer rust layer appear on the sample surface in Q235B and Q355B steels with the extension of test time. Observed from the sides of sample, the rust layer in Q235B and Q355B steels turns thicker obviously. Whereas, the rust layers of Q235BM and Q355BM steels are much thinner than those of Q235B and Q355B steels and are mainly composed of compact dark solid with a little loose tawny solid on the surface.

Figure 1. Surface morphology of sample rust layers after immersion tests (24–144 h).

Corrosion rates obtained in wet-dry cycle immersion tests are shown in Figure 2. The corrosion rates ($\text{g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$) of these four steels are calculated from the following Equation (1) [22,23]:

$$W = \frac{G_0 - G_1}{S \times t} \quad (1)$$

where W means corrosion rate ($\text{g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$), G_0 means initial weight of specimens before immersion test (g), G_1 means weight of corroded specimens after removing surface corrosion products (g), S means initial total surface area of specimens (m^2), t means test time (h). At the beginning of corrosion tests, corrosion rates of these two steels are high and tend to decrease with further corrosion due to a certain

protective roles of rust layers formed on the surface [24]. Rust layers can effectively prevent the corrosive ions in the atmosphere from contacting the matrix. Corrosion rate of steels after RE alloying is lower than that before RE alloying. Furthermore, corrosion rate of Q355BM decreased faster than that of Q235BM, indicating RE alloying has a more obvious effect on low-alloy steels than low-carbon steels.

Figure 2. Corrosion rates obtained in wet-dry cycle immersion tests.

The morphology of oxide products on the surface of rust layers has been observed by SEM, as shown in Figure 3. The oxide particles on the surface in the samples of Q235B and Q355B steels are large and rough before RE alloying. In particular, the surface of rust layer in Q355B steel is leaky and some deep cracks are unevenly distributed on the surface. After the addition of Ce and La, the surface oxidation products turn finer and are distributed uniformly, while the number of cracks and pores is obviously reduced. However, the rust layer is not easy to crack after RE alloying and has a strong ability to protect the matrix.

Figure 4 presents the XRD analysis of corrosion products. The main corrosion phases are all composed of γ -FeOOH, α -FeOOH, Fe_3O_4 and γ - Fe_2O_3 . According to previous study, γ -FeOOH is firstly formed and then transforms to α -FeOOH and Fe_3O_4 during the atmospheric corrosion of steels [7,23,25,26]. α -FeOOH is the most stable hydroxy iron oxide and the main phase in protective rust layers. γ -FeOOH is an electrochemically active phase and has negative influence on the protection of rust layers in the initial stage of corrosion. It is difficult to determine accurately whether Fe_3O_4 and γ - Fe_2O_3 is a mixture or one compound due to their similarity of XRD patterns. The relative content of corrosion products is shown in Table 2. The proportion of α -FeOOH is enhanced obviously, while the content ratio of γ -FeOOH is decreased after RE alloying. The content of Fe_3O_4 and γ - Fe_2O_3 is also improved. The corrosion products of rust layers gradually turn to stable products and protective effect on the matrix is strengthened after RE alloying.

Figure 3. Oxide products on the surface of rusty samples after 144 h immersion tests (a) Q235B (b) Q235BM (c) Q355B (d) Q355BM.

Figure 4. XRD analysis of corrosion products.

Table 2. The relative content of corrosion products of steel samples.

Sample	α -FeOOH	γ -FeOOH	Fe ₃ O ₄ or/and γ -Fe ₂ O ₃
Q235B	28%	53%	19%
Q235BM	35%	34%	31%
Q355B	17%	63%	20%
Q355BM	44%	23%	33%

In order to clarify the existence of RE element, Figures 5 and 6 respectively show the cross-section morphology and elemental distribution in rust layers after 144 h immersion tests. The cross-sections are selected from the center of the sides of specimens where the rust layer is the thickest. Due to many cracks and defects existing in rust layers, some epoxy entered into rust layer during sample processing and brings in fluctuation of element obviously. The width of the thickest rust layer in Q235B and Q355B steels is about 150 μ m, while the width is less than 100 μ m in Q235BM and Q355BM steels. In all steels, the content of Al and Cr in the loose surface rust layer and outer rust layer is low, while enriched peaks are found in inner rust layers. In Q235BM and Q355BM steels, the contents of Ce and La are much higher in matrix than in rust layers and obvious rising peaks are found near rust layers shown by red arrows. No RE inclusions is observed to gather towards the interface. Therefore, RE element probably has an obvious aggregation towards the interface in the form of atoms during corrosion process, forming a RE atom layer between rust layers and substrate.

Many researchers believe that RE atoms tend to segregate towards grain boundaries or interfaces such as surfaces or grain boundaries due to their large radius [18,23]. Whether segregation of RE elements towards interfaces occurs or not, an AES experiment has been performed for the specimens with RE addition to test this finding. A completely intergranular fracture surface covered by grain boundaries has been produced and the change of Ce content in each layer under different ion sputtering time is analyzed as shown in Figure 7. According to previous study, the kinetic energy range of Ce is chosen from 52 eV to 115 eV including three typical Auger peaks of Ce at 68 eV, 87 eV and 104 eV, respectively [23]. Although the spectra lines have a certain deviation from the standard map, the tested Auger peaks of Ce in Q235BM steel are at 68 eV, 87 eV and 103 eV, which are consistent with typical peaks in Auger Electron Spectroscopy Reference Manual [27]. For Q355BM samples, the Auger peaks are detected at 69 eV, 89 eV and 104 eV. It is certainly confirmed that Ce atoms gather towards fracture surface. After sputtering for 0.5 min, the peak values of three characteristic peaks turn less obvious, which indicates that the content of Ce is decreasing. When sputtering at 1.0 min, the peaks almost disappear because the content of Ce is too low to be detected. AES results show that the content of Ce elements on surfaces covered with grain boundaries is the highest and then decreases with the depth into grains. Whereas, no obvious Auger peaks of La are found by AES, although La atoms should have the similar Auger peaks as Ce atoms in theory. Previous study has explained this phenomenon due to the minimum level to be effectively detected and the difficulty to be distinguished from Ce [23]. Although the peaks of La are not obvious as Ce, La atoms are still believed to segregate towards grain boundaries for its similar chemical properties to Ce atoms.

Figure 5. Element distribution in rust layers after 144 h immersion tests (a) Q235B (b) Q355B.

Figure 6. Element distribution in rust layers after 144 h immersion tests (a) Q235BM (b) Q355BM.

From the results of AES spectrum in Figure 7, it is certain that the phenomenon of RE segregation tends to occur in steels. Since the auger sputtering depth is limited to less than 2 nm under the surface, which is equivalent to several atomic layers, interfacial segregation of RE elements occupies only a few atomic layers [28]. The characteristics of RE segregation towards interfaces is beneficial to improve corrosion resistance of steels. In a cyclic corrosive environment, the corrosive ions can easily contact the matrix through rust layers to accelerate the corrosion of steels. Due to the differential dissolution of RE elements in matrix and in rust layer, RE elements begin to play a role in the formation and transformation process of rust layers. RE atoms will be preferentially enriched in the pores and defects at the interface under the action of micro current, segregating and forming a RE segregation region between the rust layer and the matrix. The formation of RE atom layer is beneficial to accelerate the healing of defects and block the channel between the corrosive medium and the matrix. In addition, the segregated RE atoms may also become the crystallization cores of the rusting phase, making the rust ions grow up slowly and promoting the density of rust layers [9,29]. Furthermore, the RE segregation region can also inhibit the aggregation of S in the rust layer due to the strong affinity between RE and S, which improves the external environment and facilitates the rapid formation of protective rust layers [10,30,31].

Figure 7. Intergranular impact fracture morphology (red plus sign marks the location where AES test is conducted) and AES spectrum of Ce in Q235BM (a,b) and in Q355BM (c,d).

Based on above analyses, the corrosion resistance schematic of RE segregation in steels is proposed in Figure 8. RE atoms migrate towards the interface to fill in the pores and defects between rust layer and matrix. A protective RE atom region is formed during the process of immersion tests. Before RE alloying in steels, corrosive ions can contact the matrix through pores and cracks in rust layers to continuously corrode the matrix as shown in Figure 8a. However, the segregated RE atoms fill in the pores and cracks, thus cutting the access and preventing the corrosive ions from further entering the matrix after RE alloying, as indicated in Figure 8b. It is beneficial to improve the compactness and adhesion of the rust layer and slow down the corrosion rate of the matrix by RE-alloying method. Nevertheless, further work should be conducted to determine if RE atom layer is stable or dissolved later and how RE atoms spread into rust layers with the increasing corrosion time.

Figure 8. Schematic of RE atom layer preventing from corrosion (a) before RE alloying (b) after RE alloying.

Besides, the increased corrosion properties after RE alloying is also closely related to microstructure refinement. Microstructure before and after RE alloying are shown in Figure 9. The matrix is composed of ferrite and pearlite. The average grain size has been estimated to be 23 μm in Q235B steel and 15 μm in Q235BM steel, while the average size is about 11 μm in Q355B steel and 7 μm in Q355BM steel. Effects of RE on grain refinement are firstly due to the increase of subcooling degree by RE addition, which improves the nucleation rate of austenite and refine grains [14–16]. In addition, RE oxysulfide can refine grains as smaller heterogeneous nucleation [15,17]. The segregation of RE can also prevent grain boundary from migrating and inhibit grain growth. Grain refinement is reported to decrease the number of metastable pits [32–34]. The reduction of grain size results in a decrease in the ratio of grain areas involved in the cathode reaction to the total areas of corrosion zone. The corrosion uniformity in refined grains is better and the probability of forming deeper cracks and cavities is smaller after RE alloying. Grain refinement can increase the density of rust layers, thus improving its resistance to atmospheric corrosion.

Figure 9. Microstructure of as-rolled steels (a) Q235B (b) Q235BM (c) Q355B (d) Q355BM.

Additionally, modified RE inclusions can also increase corrosion resistance. The inclusions of MnS and Al_2O_3 are commonly observed in traditional low-carbon steels and low-alloy steels, as shown in Figure 10. The inclusion in Q235B steel is mainly composed of long-strip MnS with a size of more than 20 μm (Figure 10a), while the main inclusions in Q355B steel are composite inclusions in chains formed by Al_2O_3 and MnS with a size of more than 10 μm (Figure 10b). The addition of RE elements into steels can modify the inclusions to form spherical RE-sulfides and (RE, Al)-oxy-sulfides with a smaller size, as shown in the Figure 11. In traditional steels, non-metallic inclusions especially MnS often provide initiation sites for pitting corrosion [21]. Because MnS often acts as a cathode and the matrix acts as an anode in a corrosion system, the oxide film between MnS and the substrate is easily destroyed by aggressive ions. The activated matrix preferentially dissolves to produce Fe^{2+} around MnS and then hydrolyzed to produce H^+ . It is tended to cause local acidification at the interface and promotes the dissolution of MnS, which produces S^{2-} and HS^- to promote further pitting corrosion [35]. RE can suppress the formation of MnS and control the size of inclusions, owing to the lower standard

formation energy of RE inclusions [30]. The interface area between RE inclusions and the matrix is far less than that between MnS and the matrix, so the active area is reduced and weakens micro galvanic corrosion. Results above have also shown that the diameter of inclusion in steels alloyed by RE elements has been decreased to less than 5 μm and the shapes are transformed from irregular to spheroid shapes. The dispersed small inclusions can become activated points of corrosion reaction, which is beneficial to the formation of continuous uniform rust layer.

Figure 10. SEM images and EDS mapping of inclusions in (a) Q235B and (b) Q355B.

Figure 11. SEM images and EPMA mapping of inclusions in RE-modified (a) Q235BM (b) Q355BM.

4. Conclusions

The effects of RE elements on corrosion properties of Q235B and Q355B steels under wet-dry cycle immersion tests are analyzed. RE elements play an important role in enhancing corrosion performance. Firstly, microstructure is greatly refined by RE alloying, which is beneficial to increase the density of rust layers and thus improve the corrosion resistance. The beneficial effect of RE in optimizing inclusions is to form smaller spherical RE-oxy-sulfides and reduces the occurrence of pitting. Furthermore, segregation of RE towards grain boundaries confirms that RE is likely to accumulate at interfaces with high energies. The segregation of RE between rust layers and the matrix can effectively improve the adhesion of the rust layers and prevents corrosive ions from contacting the matrix. Therefore, low-carbon and low-alloy steels combined with high corrosion resistance via RE alloying can extend the life of steels, showing a promising application as economic structural materials.

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