Corrosion Mechanism of Corrosion-Resistant Steel Developed for Bottom Plate of Cargo Oil Tanks

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A new type of corrosion-resistant steel consisting of ferrite and bainite phases was developed for cargo oil tanks of crude oil tankers. The corrosion rate of this new steel was 0.22 mm/a, which was equivalent to ca. 1/5 of the criterion (≤ 1 mm/a) for corrosion-resistant steels. The composition and element distribution of the corrosion products were investigated by micro-Raman spectrometry and energy dispersive spectrometer. The results demonstrated that the corrosion product was composed of α -FeOOH, Fe₃O₄ and a continuous Cu enrichment layer. This kind of corrosion product was protective to the steel matrix and accounted for the enhancement of the corrosion resistance of the new developed steel.

KEY WORDS: Alloying elements; Corrosion; Low alloy steel; Raman spectroscopy; SEM

1. Introduction

Oil tanker accidents, which occurred frequently in the last decades, had led to massive oil leakage. These oil leakage disasters not only endangered the natural life, but also caused an enormous economic loss. It is usually related to material degradation caused or influenced by undetected corrosion^[1]. Among which, the corrosion problem of the cargo oil tanks (COT) of crude oil tankers is quite severe. At the COT, the inner bottom plate is covered by oil layer containing sludge, drain water including highly concentrated Cl[−] and H₂S originating from the crude oil. In general, this kind of oil layer could decrease the corrosion rate of the bottom plate. However, local defects would occur in the oil layer by crude oil washing. Then the inner bottom plate is exposed to the severe corrosion environment with concentrated Cl[−] and H₂S. As a

result, pits would originate from these defects and grow by forming galvanic cell between the defect (anode) and steel surface beneath the oil layer around defect (cathode)^[2–4]. Intensive investigations have been carried out on pitting corrosion phenomena on inner bottom plates of COT of Very Large Crude Carrier (VLCC). Based on above studies, some key facts for inner bottom plate corrosion are summarized^[2,5–9]: (1) The solution is neutral brine (not sea water), which contains 10 wt.% NaCl; (2) In pits, pH value of the solution is lower than 1.5; (3) The majority of pits at the bottom plate are of bowl shape; (4) In some cases, the pitting corrosion rate reaches 4 mm/a; (5) The pit growth stops at dock inspection.

Coating is one of the well-known corrosion prevention methods, which isolates the steel surface from corrosion environments. If the coating was applied completely, the isolation effect would be perfect. However, intact coating working without defect would be very difficult from initial surface preparation to the final coating. In general, very tiny defects existed in the coating layer. Moreover, once the coating was removed from conventional steel, the substrate would

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be exposed to corrosion environment. The growth of pitting corrosion would start at high rate, and the risk of perforation of the bottom plate is high, that would lead to crude oil leakage. It was investigated that a lot of bowl-shaped pits 4 mm or more in depth occurred on the inner bottom of COT^[2], which needed inspection and repair of every dock with high economic and labor costs. If the pitting was not detected and repaired, the bottom plate will be confronted with high risk of perforation. Therefore, the development of corrosion-resistant steel which lowered risk of perforation of the inner bottom plates is important. In this respect, such work had been started by Japanese scholars from 2002^[4,6–9], in which micro-alloying was used as the manufacturing approach. These steels had high corrosion resistance compared with conventional steels, with 1/5–1/2 corrosion rate of conventional steels. In recent years, Chinese scholars began to research and develop corrosion-resistant steel for COT. They investigated the effects of composition and microstructure on corrosion properties of corrosion-resistant steel and welded joint^[10–12]. However, the corrosion mechanism of micro-alloying steel is far from clear and needs further research.

The current work is devoted to investigating the effect of alloy elements on the corrosion properties of the self-developed corrosion-resistant steel for COT. Its microstructure and corrosion behavior were studied by SEM, potentiodynamic polarization curves and EIS techniques. And then the composition of the rust layer was analyzed mainly using micro-Raman spectrometry and energy dispersive spectrometer (EDS), which could give us insight into corrosion mechanism of the self-developed steel. Furthermore, the understanding of the corrosion behavior and mechanism of micro-alloying steel will be improved, which is helpful for the development of the new corrosion-resistant steel for COT of crude oil tankers.

2. Experimental Procedures

2.1 Preparation of specimens and solution

The specimens were made of A36 ship plate steel selected as conventional steel according to IMO MSC 87/26/Add. 1 Annex 3 MSC. 289 (87)^[13], and the self-developed corrosion-resistant steel for COT. The compositions of A36 and the self-developed steels are listed in Table 1. It was shown that the self-developed steel had lower content of C and P and higher content of alloying elements, such as Cu, Ni and Mo, in comparison to A36 steel. The self-developed steel was manufactured by Thermo-Mechanical Control Processing (TMCP) at a cooling rate of 15 °C/s. Its yield strength σ_s and tensile strength σ_b were 485 MPa

and 545 MPa, respectively. The impact energy A_K at –40 °C reached 298 J. The samples for immersion tests were cut into 60 mm×25 mm×5 mm, and then drilled hole with ϕ 3 mm for suspension. The longitudinal direction of samples was parallel to the rolling direction of steels. The samples for electrochemical tests were cut into 10 mm×10 mm×5 mm, and then embedded with epoxy resin leaving a working area of 1 cm². Prior to experiments, the specimens were abraded with waterproof abrasive paper down to 2000 grit and finally polished with 1.5 μ m Al₂O₃ powder.

All experiments were performed in 10 wt.% NaCl solution with pH value of 0.85 at (30±2) °C. The solutions were prepared with analytical pure reagents and distilled water. The pH value of the solution was adjusted using concentrated hydrochloric acid.

2.2 Immersion experiments

The immersion experiments were carried out according to IMO MSC 87/26/Add. 1 Annex 3 MSC. 289 (87), the performance standards for alternative means of corrosion protection for cargo oil tanks of crude oil tankers, in order to simulate corrosion environment of ship plate steels equipped at flat inner bottom of COT^[13]. Five parallel specimens were used to perform each immersion experiment, which was carried out for 72 h. The corrosion mass loss rate was used to evaluate corrosion resistance of specimens.

The decaling liquid was hydrochloric acid solution (50 vol.%) + 3.5 g/L (CH₂)₆N₄.

2.3 Electrochemical experiments

Electrochemical experiments were carried out in a three-electrode cell. The counter and reference electrode were Pt sheet and saturated calomel electrode (SCE), respectively. The working electrode was made of A36 steel or the self-developed steel.

The potentiodynamic polarization measurements were performed using an electrochemical workstation (PARSTAT 2273) with a potential scan rate of 0.333 mV/s. The EIS measurements were also carried out on the workstation too. The scanning frequency ranged from 100 kHz to 10 mHz and the perturbing AC amplitude was 5 mV. The reproducibility of the presented data was double checked by multiple measurements and typical results were reported.

2.4 Microstructure observation

The microstructures of A36 and self-developed steels were observed by SEM (FEI Quanta 250) after etching in 4% Nital. The composition of the rust layer on samples after immersion tests was detected

Table 1 Composition (wt.%) of A36 and the self-developed steels

Specimen	C	Si	Mn	P	S	Al	Cu	Ni	Mo	Fe
A36 steel	0.16	0.20	1.17	0.022	0.008	0.030	0.006	0.02	0.008	Bal.
Self-developed steel	0.08	0.30	1.46	≤ 0.010	≤ 0.010	0.030	0.50	0.10	0.02	Bal.

by EDS and micro-Raman spectrometry. Raman spectroscopy observation was undertaken using a JY-HR800 Confocal Microprobe Raman Spectrometer with an exciting wavelength of 532 nm from the light source of an argon ion laser. Low laser power was used for the reference spectra to minimize the risks of spectral changes due to sample degradation^[14,15]. Excitation laser power on the samples was filtered to 0.1 mW to avoid thermal effects on the sensitive iron oxides and oxy-hydroxides.

3. Results

3.1 Microstructure analysis

The metallographic structure of A36 and self-developed steels are shown in Fig. 1. It demonstrates that the microstructure of A36 steel consists of ferrite phase and pearlite (F/P). The grain size is about 10–30 μm measured by a line intercept method. The microstructure of self-developed steel is composed of ferrite phase and bainite (F/B). The grain size is about 5–18 μm . The average grain size of self-developed steel is smaller than that of A36 steel, which can be attributed to its faster cooling rate (15 $^{\circ}\text{C/s}$). The existence of pearlite reduces the corrosion resistance of A36 steel due to the lamellar structure of ferrite and cementite in pearlite^[16].

3.2 Corrosion rate analysis

The corrosion mass loss rate can be calculated by the following equation^[13]:

$$v = \frac{8.76 \times 10^4 \times W}{S \times t \times \rho} \quad (1)$$

where v is the corrosion mass loss rate (mm/a); W is the weight loss (g); S is the sample surface area (cm^2); ρ is the metal density (g/cm^3) and t is the immersion time (h).

The average corrosion mass loss rates of A36 and self-developed steels are 1.31 and 0.22 mm/a, respectively. The corrosion mass loss rate of self-developed steel is merely about 1/6 that of A36 steel. The

accepted criterion for corrosion-resistant steel of COT is $v \leq 1$ mm/a, according to IMO MSC 87/26/Add. 1 Annex 3 MSC. 289 (87). The corrosion mass loss rate of self-developed steels is ca. 1/5 of the accepted criterion. Moreover, this corrosion mass loss rate is lower than that of NSGP-1 as reported in literature [17]. Therefore, the self-developed steel can not only meet the requirement of COT on corrosion-resistant steel, but also has superior corrosion resistance compared with its counterpart.

Based on the obtained corrosion mass loss rate, the corrosion current density i_{corr} can be calculated according to Faraday's law^[18]:

$$i_{\text{corr}} = \frac{v \times z \times F \times \rho}{3.16 \times 10^8 \times M} \quad (2)$$

where z is the number of electrons transferred per metal atom; F is the Faraday's constant (C/mol); M is the molar mass of the metal (g/mol).

Fig. 2 shows potentiodynamic polarization curves of A36 and the self-developed steels measured in 10 wt.% NaCl solution at (30 ± 2) $^{\circ}\text{C}$. Typical active dissolution behavior could be recognized on both curves. The anodic and cathodic branches of the polarization curves of A36 and the self-developed specimens display Tafel behavior. Thus, Tafel extrapolation can be applied to calculate corrosion current density i_{corr} , anodic Tafel slope β_a and cathodic Tafel slope β_c . The results are displayed in Table 2.

Fig. 3 shows EIS results of A36 and the self-developed steels measured in 10 wt.% NaCl solution at (30 ± 2) $^{\circ}\text{C}$, which are presented as Nyquist and Bode plots. It shows that one semicircle exists over the measured frequency range in EIS of A36 specimens. While two semicircles can be observed over the measured frequency range in EIS of the self-developed specimens: a high-frequency capacitive semicircle and a middle-frequency capacitive loop.

The equivalent electrical circuits shown in Fig. 4(a) and Fig. 4(b) were used to fit the impedance data of A36 and the self-developed specimens, respectively. Where R_s is the solution resistance, CPE_{dl} is the constant phase element which expresses the double

Fig. 1 SEM images of metallographic structure of A36 (a) and self-developed (b) steels

layer capacitance and R_t expresses the charge transfer resistance of steel, CPE_f is the constant phase element which related to corrosion product film and R_f is corrosion product film resistance, respectively^[19–21]. The R_t and R_f of the specimens can be obtained through the fitting with the equivalent electrical circuit, which are listed in Table 2. Since $R_P = (Z_F)_{\omega=0}$, the polarization resistance R_P of

A36 and the self-developed steels can be expressed as $R_P = R_t$ and $R_P = R_t + R_f$ respectively. R_P is related to the corrosion current density i_{corr} through the Tafel slopes β_a and β_c according to Stern-Geary equation^[22,23]:

$$i_{\text{corr}} = \frac{1}{2.303R_P(\frac{1}{\beta_a} + \frac{1}{|\beta_c|})} \tag{3}$$

The corrosion current densities i_{corr} obtained by corrosion mass loss, Tafel extrapolation method and Stern-Geary equation are summarized in Fig. 5. It indicates that i_{corr} obtained by immersion test, potentiodynamic polarization and EIS measurements are correlated with each other. The i_{corr} of the self-developed specimen is much less than that of A36 specimen, which means that the corrosion resistance of the self-developed steel was enhanced greatly.

3.3 Composition of corrosion product

Fig. 6 and Fig. 7 show the SEM images and EDS spectra of corrosion products of A36 and self-developed steels after 240 h immersion test. It is demonstrated that there are non-metallic inclusions

Fig. 2 Potentiodynamic polarization curves of A36 and self-developed steels in 10 wt.% NaCl solution at $(30\pm2)^\circ\text{C}$

Table 2 Fitting results of potentiodynamic polarization curves and EIS of A36 and the self-developed steels

specimen	E_{corr} (V)	i_{corr} (10^{-5} A/cm^2)	β_a	β_c	R_t ($\Omega\cdot\text{cm}^2$)	R_f ($\Omega\cdot\text{cm}^2$)
A36 steel	-0.506	15.3	0.078	-0.136	159.0	-
Self-developed steel	-0.478	4.9	0.042	-0.106	644.1	9.3

Fig. 3 The Nyquist plots (a) and Bode plots (b) of EIS of A36 and self-developed steels in 10 wt.% NaCl solution at $(30\pm2)^\circ\text{C}$

Fig. 4 The equivalent electrical circuit for EIS of A36 (a) and self-developed (b) steels in 10 wt.% NaCl solution at $(30\pm2)^\circ\text{C}$

in the research steels. The chemical compositions of corrosion product matrix and inclusions are shown in Table 3. Many kinds of metal elements (such as Fe, Cu and Mn) and non-metallic elements (such as O and Cl) exist in the corrosion products of A36 steel; while no Cl element is found in the corrosion products of the self-developed steel. This indicates that chloride ions might not participate in the formation of corrosion products of the self-developed steel. The

content of Cu in the corrosion products of the self-developed steel is 23 at.%, which is higher than that (3.8 at.%) of A36. It also indicated that the better corrosion resistance of the self-developed steel might be attributed to the formation of copper oxide in the corrosion product.

Fig. 8 and Fig. 9 show SEM image and X-ray elemental maps of corrosion product of A36 and the self-developed steel after 240 h immersion test in 10 wt.% NaCl solution at $(30\pm 2)^\circ\text{C}$, respectively. It can be seen that the inclusions in both steels are composed of O, Al and Mg, which could be assigned to $\text{MgO}\cdot\text{Al}_2\text{O}_3$. The element of Cu in the corrosion product matrix of A36 steel only enriches in local area; while it was uniformly distributed in the corrosion product matrix of the self-developed steel. Therefore, the protection of corrosion product generated on the self-developed steel is better than that on A36 steel.

Fig. 10 shows Raman spectra of corrosion product of A36 and the self-developed steel after 240 h immersion test. There are peaks at 225, 293, 412, 500, 612 and 1320 cm^{-1} in Raman spectra of A36 steel, which are in accordance with the typical Raman spectrum of hematite $\alpha\text{-Fe}_2\text{O}_3$. The bands at 300, 395, 515, 645 and 670 cm^{-1} are associated with maghemite $\gamma\text{-Fe}_2\text{O}_3$.

Fig. 5 Comparison of corrosion current density i_{corr} according to mass loss test, potentiodynamic polarization and EIS measurements

Fig. 6 SEM image of corrosion products of A36 steel after 240 h immersion test in 10 wt.% NaCl solution at $(30\pm 2)^\circ\text{C}$ (a) and EDS spectrums of matrix (b) and inclusion (c)

Fig. 7 SEM image of corrosion products of self-developed steel after 240 h immersion test in 10 wt.% NaCl solution at $(30\pm 2)^\circ\text{C}$ (a) and EDS spectrums of matrix (b) and inclusion (c)

Table 3 Compositions (wt.%) of matrix, corrosion product and inclusion

Specimen	Location	O	Mg	Al	Cl	Mn	Fe	Cu
A36 steel	Matrix	6.7	-	-	1.3	1.4	86.8	3.8
	Inclusion	17.2	6.9	15.8	0.9	0.9	55.7	2.5
Self-developed steel	Matrix	6.6	-	-	-	-	70.4	23.0
	Inclusion	11.2	2.9	6.3	-	1.0	55.1	23.5

Fig. 8 SEM image and X-ray elemental maps of corrosion product of A36 steel after 240 h immersion test in 10 wt.% NaCl solution at $(30\pm 2)^\circ\text{C}$

Fig. 9 SEM image and X-ray elemental maps of corrosion product of self-developed steel after 240 h immersion test in 10 wt.% NaCl solution at $(30\pm 2)^\circ\text{C}$

Fe_2O_3 ^[19,24]. This demonstrates that corrosion product of A36 steel is composed of $\alpha\text{-Fe}_2\text{O}_3$ and $\gamma\text{-Fe}_2\text{O}_3$. There are peaks at 338, 385, 412, 480, and 685 cm^{-1} in Raman spectra of the self-developed steel, which are typical Raman spectra of goethite $\alpha\text{-FeOOH}$. The bands at 320, 560, 613 and 680 cm^{-1} are assigned to magnetite Fe_3O_4 ^[25]. This demonstrates that corrosion product of the self-developed steel is composed

of $\alpha\text{-FeOOH}$ and Fe_3O_4 .

4. Discussion

4.1 Transformation of Fe

A mechanism for the iron dissolution reaction in acidic solutions containing halide ions was proposed^[26]. This mechanism suggests that iron

Fig. 10 Raman spectra of corrosion products formed on A36 and self-developed steel after 240 h immersion test in 10 wt.% NaCl solution at $(30 \pm 2)^\circ\text{C}$

dissolves through two dissolution paths in the presence of Cl^- : (1) reaction path through a FeOH adsorbed intermediate; (2) reaction path through a FeCl adsorbed intermediate. The adsorption of OH^- may be assumed to take place via the following steps^[27]:

where the subscript ads. indicate adsorbed state, the subscript sol. indicate the dissolved state.

Chloride ions are adsorbed on the iron surface during iron dissolution in the presence of Cl^- . The following reactions are proposed^[26]:

The competitive adsorption of Cl^- and OH^- is evident. The EDS results indicate that Cl exists in the corrosion products of A36 steel. However, there is no Cl in the corrosion products of the self-developed steel (Fig. 6 and Fig. 7). Therefore, it can be assumed that there is a strong interaction between Cl^- and Fe adatoms for A36 steel. On the contrary, the adsorption of OH^- prevails for the self-developed steel.

The diffusion of oxygen at the interface transforms $\text{Fe}(\text{OH})_2$ into FeOOH as follows:

Many researchers in the past have proposed that $\gamma\text{-FeOOH}$ is initially formed, which transforms to Fe_3O_4 , $\alpha\text{-FeOOH}$ and $\gamma\text{-Fe}_2\text{O}_3$ ^[25,28]. It is declared that $\gamma\text{-FeOOH}$ transforms to $\alpha\text{-FeOOH}$ by dissolution and reprecipitation according to Misawa^[29]. However, the mechanism proposed by Misawa is not the only

path for the transformation of $\gamma\text{-FeOOH}$ to a more stable $\delta\text{-}$ or $\alpha\text{-FeOOH}$. It has been deduced by Raman and Kuban^[28] that $\gamma\text{-Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ has a hexagonal arrangement of oxygen-hydroxyl layers, which is similar to many of the stable rust phases, such as α , β and $\delta\text{-FeOOH}$. Thus, $\gamma\text{-FeOOH}$ with a cubic stacking arrangement of layers containing oxygen-hydroxyl ions transforms topologically (requiring only slight readjustment of atoms, not requiring long-range diffusion-dependent nucleation and growth processes) to $\gamma\text{-Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ with a hexagonal stacking arrangement of these layers, which in turn transforms easily to $\delta\text{-FeOOH}$ or $\alpha\text{-FeOOH}$. The Raman spectra results has revealed that corrosion product of self-developed steel is composed of $\alpha\text{-FeOOH}$ and Fe_3O_4 . The formation of such stable and protective rust layer will be in favor of improvement of corrosion resistance.

4.2 Effect of Cu

It has been declared that copper ions dissolved during corrosion reaction could be re-precipitated on the surface of steel and served as an efficient cathodic site. This mechanism is applicable when pH value of the local electrolyte is quite low; especially in the presence of chloride ions^[25]. In this work, corrosion electrolyte is 10 wt.% NaCl solutions with pH value of 0.85. And it has been found that Cu exist in rust layer of A36 and the self-developed steels, as shown in Fig. 6 and Fig. 7 and Table 3. It can be seen that there are 3.8 at.% Cu in rust layer of A36 steel and 23.0 at.% Cu in rust layer of the self-developed steel. The content of Cu in rust layer of both steels is much higher than that in steel matrix. This demonstrates that a Cu enriched layer forms on the surface of steels, which can be explained by the preferential dissolution and redeposition process of copper. In an acid solution, both Fe and Cu dissolve, however the dissolved Cu re-deposits with the form of re-crystallized Cu and Cu oxides^[30,31]. The mechanisms of the selective dissolution and re-deposition processes of Cu in low alloy steel can be outlined as follows:

(1) At the initial stage of corrosion, the steel surface is mainly composed of Fe with a small amount of Cu near the solution/metal interface.

(2) When Fe dissolves, Cu may subsequently be away from the metal surface as steel does not have a continuous Cu network. Therefore, the preferential dissolution of Cu occurs along the continuous paths of less noble atoms^[32].

(3) As soon as Cu is separated from the surrounding Fe, Cu dissolves to Cu^{2+} coupling with reduction of the dissolved oxygen.

(4) Cu^{2+} acts as an oxidant in Fe, H_2O and O_2 system. Therefore, a proportion of Cu^{2+} will reduce (with consumption of the electrons produced by oxidation of the Fe) and then re-deposit onto the substrate, which is called Cu plating. And the amount of Cu plating should increases as the Cu^{2+} concentration

increases^[33].

(5) The re-deposited Cu will be precipitated as re-crystallized Cu and Cu oxides. The selective dissolution and redeposition process will repeat until the Cu enriched layer is stabilized^[31].

Because the Cu composition (23.0 at.%) in rust layer of the self-developed steel is much more than that of A36 steel (3.8 at.%), the distribution of Cu in rust layer of the self-developed steel is more homogeneous and continuous, as shown in Fig. 8 and Fig. 9. The continuous formation of Cu enrichment layer may give birth to a higher number of sites which are in favor of the adsorption reaction of the protective corrosion products but have an adverse effect on the overall charge transfer process. The accumulation of copper would diminish the iron dissolution by blocking the active points in the lattice. Therefore, the copper enrichment layer decreases the anodic dissolution rate by a blocking mechanism and a screening mechanism^[33].

5. Conclusions

The self-developed steel has superior corrosion resistance and can be directly applied to COT of crude oil tankers without the protection of coating. The average corrosion mass loss rate of the self-developed steel is only 0.22 mm/a, which is 1/5 of the accepted criterion ($v \leq 1$ mm/a) for corrosion resistance steels, according to IMO MSC 87/26/Add. 1 Annex 3 MSC. 289 (87).

The excellent corrosion resistance of the self-developed steel could be attributed to its low carbon content (0.08 wt.%), high alloy element content (Cu) and fine microstructure (F/B). Consequently, a protective rust layer grows on the surface of the self-developed steel, consisting of α -FeOOH, Fe_3O_4 and a continuous Cu enrichment layer.

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