Electrochemical Corrosion Performance of Low Alloy Steel in Acid Sodium Chloride Solution

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ABSTRACT

The corrosion behavior of low alloy steel for cargo oil tanks (COT) is investigated by thermo mechanical control process (TMCP) at various cooling rates. The corrosion rate is estimated by accelerated corrosion test in strong acid sodium chloride solution. The corrosion mechanism is studied by potentiodynamic polarization curve and electrochemical impedance spectroscopy (EIS) measurements. It indicates that the acicular ferrite is refined as the cooling rate increases, the CR15 steel with higher cooling rate exhibits good corrosion resistance with a less weight loss, lower corrosion current density and bigger impedance modulus than steels CR10 and CR12.

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Keywords : Low Alloy Steel, Strong Acid Solution, Accelerated Corrosion Test, Polarization

1. Introduction

The materials of low alloy steel for cargo oil tanks (COT) not only require good mechanical properties, but also have superior corrosion resistance. There is a certain amount of chloride ion, hydrogen ion and sulphide when the tank is filled with crude oil, the special environment determines the life of COT steel, even leading to some accidents. So the corrosion problem of low alloy steel for COT has been focused more and more people's attention. At present, the protective measures mainly include: the anti-corrosion coating, corrosion inhibitor and developing corrosion resistant steel. The coating and inhibitor play a strong role on protecting corrosion, but the long-term investment is very expensive. So it is very necessary to develop the corrosion resistant steel for providing the long protection.

Now, the corrosion resistant properties of alloy steel have been studied, obtaining some achievements, major consideration with respect to atmospheric corrosion,^{1–4} marine corrosion^{5–8} and the corrosion for some special environments.^{9–12} However, very few studies on the corrosion behavior of COT steel have been reported.^{13–15} The evaluation methods of corrosion resistance have been widely concerned. Weight loss measurement is the most direct and reliable method but time consuming and information limited, therefore the corrosion resistance has been investigated by simulated and accelerated corrosion tests. In addition, electrochemical measurement is a powerful tool to analyze corrosion mechanism, obtaining good results by equivalent circuit method,^{16–20} and the research about COT steel also has achieved results using this method.^{14,15} To our knowledge, no studies have been reported in the literature concerning the corrosion behavior of COT steel by analysis microstructure, so it is particularly important to develop a new type of corrosion resistant steel for COT and study the influence of microstructure on the corrosion performance.

In this paper, the electrochemical and corrosion behavior for a new type of low alloy steel for COT are studied in acid 10 wt% NaCl solution (pH = 0.85). Firstly, the microstructures are observed by optical microscope (OM) and transmission electron microscope (TEM). Then, the weight loss is used to calculate corrosion rate. The polarization curve and electrochemical impedance spectroscopy

(EIS) are obtained to analyze the corrosion behavior, and using Tafel fitting and equivalent circuit method, respectively. Finally, the relative residual error is used to evaluate the fitting effect for EIS dates, determine the circuit model is effective.

2. Experimental Material and Procedures

2.1 Materials and solution

The chemical composition (wt%, mass fraction) of a new type of corrosion resistant steel for COT is C = 0.085, Si = 0.24, Mn = 1.36, P = 0.006, S = 0.002, Al = 0.04, Cr = 0.23, Cu = 0.16, W = 0.21, Mo = 0.21, Ni = 0.11, Nb = 0.012, and Fe to balance. The thermal simulation experiments are performed using the multi-function thermal simulator. The acicular ferrite is appeared with cooling rate from 9°C/s and 18°C/s based on the dynamic continuous cooling transformation. Based on above analysis, three kinds of samples with various cooling rate of 10.4°C/s, 12.8°C/s and 15.9°C/s are obtained, which are marked as CR10, CR12 and CR15, respectively. The mechanical properties are shown in Table 1. It can be clearly seen that the yield strength is between 420 MPa and 450 MPa, and the tensile strength is between 600 MPa and 620 MPa, the elongation is greater than or equal to 20%, the yield ratio is lower than 0.8. The impact energy range is from 220 Joules to 280 Joules at –20°C, the impact energy is between 180 Joules and 280 Joules at –40°C. The mechanical properties for corrosion resistant steel can meet the DH36 steel requirement, which indicate the TMCP process is feasible.

According to IMO MSC 87/26/Add 1 Annex 3 MSC.289 (87),²¹ the samples for immersion tests are cut into the rectangular shape for 60 mm × 25 mm × 5 mm, and then drilled hole (Φ3 mm) for suspension. The samples for electrochemical tests are cut into the rectangular shape for 10 mm × 10 mm × 3 mm, and the exposed surface is 10 mm × 10 mm, with an area of 100 mm², all other surfaces are isolated from the environment by epoxy resin. Prior to testing, the samples are ground with a series of SiC waterproof abrasive papers (100, 600, 800, 1200 and 1500).

The corrosive solution of all experiments in this study is 10 wt% NaCl solution at 30°C, pH value is 0.85, which is adjusted using concentrated hydrochloric acid.

Table 1. The mechanical characteristic parameters.

Sample	Tensile strength				Impact	
	Yield strength /MPa	Tensile strength /MPa	Elongation/%	Yield ratio	Impact toughness/J –20°C	Impact toughness/J –40°C
CR10	428	603	20.7	0.71	276	274
CR12	442	610	21.6	0.72	226	213
CR15	437	612	20.0	0.71	224	180

2.2 Accelerated corrosion tests

The immersion test is performed according to IMO MSC 87/26/Add 1 Annex 3 MSC.289 (87), the performance standard of corrosion protection for COT in order to simulate corrosion environment of inner bottom. The coupons surface preparation is degreased with acetone, and washed with distilled water and dried with filter paper. After weighting (W_a), the tested samples are suspended in 10 wt% NaCl solution with pH value of 0.85 at 30°C for 72 hours. Subsequently, the test samples are removed and rinsed with distilled water, and dried in the nitrogen atmosphere, and weighed (W_b). The weight loss ΔW can be obtained from $W_a - W_b$. Three parallel samples are used to perform each immersion experiments, and the mean value is reported, and the corrosion rate is calculated.

2.3 Electrochemical measurements

The electrochemical measurements are performed using an electrochemical workstation (PATSTAT2273). The experiments are carried out in a conventional three-electrode cell, the platinum electrode as the auxiliary electrode, the saturated calomel electrode (SCE) as the reference electrode, which is connected to the NaCl solution via a Luggin capillary and a salt bridge, and the sample as the working electrode. All polarization curves are recorded potentiodynamic with a scan rate of 1 mV/s and the potential range from 100 mV to –100 mV, and a CorrView software is used to analyze the polarization curves. The electrochemical impedance spectroscopy experiments are conducted at an open circuit voltage with 8 mV perturbation and a frequency range is from 10^5 to 10^{-1} Hz, and Zsimpwin software is used to analyze the EIS results based on an equivalent circuit method. Finally, the relative residual error is used to evaluate the fitting effect for EIS dates, determined the circuit model is effective.

2.4 Microstructure observation

The microstructures are observed by optical microscope (OM). The high-resolution transmission electron microscope (TEM) is used to reveal the microstructure in detail. The electron back scattered diffraction (EBSD) are performed on FEI/Philips quanta 600 to investigate crystallographic characteristics maps and grain size.

3. Results and Discussion

3.1 Microstructures

The microstructures of the experimental steel are reflected by OM. The results are shown in Fig. 1. As can be seen from these pictures, the type of microstructures for steels CR10, CR12 and CR15 is all acicular ferrite (AF), i.e., quasi-polygonal ferrite (QF), granular ferrite (GF), and a little bainite ferrite (BF).²²

TEM are used to reveal the microstructure in detail in Fig. 2, it also display that the microstructure is acicular ferrite, which is consistent with OM observations. Also the QF grains have irregular and jagged boundaries, including high density of dislocations and subboundaries.

Figure 3 shows that the area fraction of grain size. It is seen that the fine ferrite grain size (grain diameter <7 microns) will become

Figure 1. The OM images for the experimental steel with varying cooling rate. **Figure 2.** The TEM graphs for the sample steel CR12.

more and more with cooling rate increases. Figure 4 shows the crystallographic characteristics maps of the experimental steel analyzed by EBSD. Image quality maps with grain boundary misorientation distribution show the grains size and different types of grain boundaries, the red lines indicate the low misorientation boundaries of 2–15° and the blue lines stand for the high misorientation boundaries of greater than 15°. It has been accepted

Figure 3. (Color online) The EBSD micrographs for the experimental steel with varying cooling rate.

Figure 4. (Color online) The EBSD analysis of the experimental steel with varying cooling rate.

Figure 5. (Color online) Potentiodynamic polarization curves of the experimental steel in 10 wt% NaCl acid solution. (a) bare steel, (b) 72h pre-corrosion immersion.

that the grain boundary character plays a key role on the corrosion behavior, the low angle grain boundaries are usually more stable than high angle grain boundaries.²³ As the cooling rates increase, the length fraction of low misorientation boundaries goes up, which is 0.103, 0.119 and 0.181 for steels CR10, CR12 and CR15, respectively. The percentage increase of low angle grain boundary is beneficial to the improvement of the corrosion resistance.

3.2 Accelerated corrosion tests

The corrosion rate is calculated by the following equation.¹³

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

The samples is the rectangular shape for 60 mm × 25 mm × 5 mm. where v is the corrosion rate, mm/year; ΔW is the weight loss, g; S is the total surface area of the sample, cm²; ρ is the metal density (8.036 g/cm³), t is the immersion time (72 h).

The average corrosion rates are 0.253, 0.245 and 0.213 mm/year for steels CR10, CR12 and CR15, respectively. It demonstrates that the corrosion rate is in the following order: $v_{\text{CR10}} > v_{\text{CR12}} > v_{\text{CR15}}$, and the experimental steel has well corrosion resistance. The corrosion rate for CR15 steel is lower than other steels. It shows the corrosion rate depends on the steel microstructure and grain size, which may be result from the different cooling rates.

3.3 Polarization behaviors

Potentiodynamic polarisation measurement is conducted on the samples with bare steel and 72 h pre-corrosion immersion. The polarization curves experimentally measured for the steel samples in acid 10 wt% NaCl solution are shown in Fig. 5. It can be seen that the anodic current density increases continuously with increasing corrosion potential, which indicate that all samples show active dissolution behaviors. The anodic and cathodic branches show Tafel behavior. So Tafel extrapolation can be applied to calculate the corrosion kinetics parameters. The corresponding anodic Tafel constants (b_a), cathodic Tafel constants (b_c), corrosion potential (E_{corr}), and corrosion current density (I_{corr}) are given in Table 2. The anodic and cathodic Tafel constants do not obviously change for bare steel samples, which show that the similar mechanism for corrosion reaction. The anodic Tafel constants of the bare steel are close to the theory value 60 mV/dec at 30°C ($2.303RT/F = 60$, mV/dec, R , T , and F represents universal gas constant, absolute temperature and Faraday constant, respectively). With the cooling rates increase, the corrosion potential shift towards the positive direction, and the corrosion current density will decrease, which indicate the corrosion resistance of study steel will be better and better. For pre-corrosion immersion 72 h, it is found that the corrosion potential (E_{corr}) shift to a negative value and the corrosion current density (I_{corr}) increase with increasing of immersion time,

Table 2. Potentiodynamic polarization parameters from a curve-fitting approach.

Immersion time/hour	sample	b_a (mV/dec)	b_c (mV/dec)	E_{corr} (mV vs. SCE)	I_{corr} (mA cm ⁻²)
0	CR10	60	-153	-495	0.0864
	CR12	59	-152	-475	0.0769
	CR15	60	-164	-473	0.0588
72	CR10	97	-187	-499	0.1451
	CR12	141	-104	-542	0.1239
	CR15	124	-96	-533	0.0610

which shows that the corrosion resistance is becoming more and more poor. We can see that the corrosion current density of CR15 steel is smaller than other steel, and the corrosion current density (I_{corr}) is from 0.0588 mA to 0.0610 mA, increasing about 0.0022 mA. However the changes of the corrosion current density of steels CR10 and CR12 are relatively fast, the increase values are about 0.0587 mA and 0.047 mA, respectively. So CR15 steel shows the good corrosion characteristics, indicating the smaller corrosion current density. This result verifies that the more fine grain and low angle grain boundary exhibit a suppressing effect on corrosion resistance.

In addition, corrosion rate v is calculated by substituting corrosion current density taken from Tafel plot for the following equation.²⁴

$$v = 0.00327 \frac{ai}{nD} \quad (2)$$

where a is molar mass of steel (g/mol), i is corrosion current density ($\mu\text{A}/\text{cm}^2$), n is valency, D is steel density. Results of the corrosion rate are 1.68, 1.44 and 0.71, respectively. Although the corrosion rates in the potentiodynamic polarisation measurement would not be the same as that obtained from accelerated corrosion test, it indicates that the corrosion rate of CR15 steel is much less than that of steels CR10 and CR12, which means that the corrosion resistance of CR 15 steel is enhanced greatly.

3.4 Electrochemical impedance spectroscopy (EIS)

Electrochemical method is known to be an effective technology for measurement of corrosion behavior and corrosion mechanism. In order to confirm the results of the potentiodynamic polarization test, the electrochemical impedance spectroscopy (EIS) measurement is performed under the same experimental conditions as the polarization curves. Figure 6 shows the typical Nyquist plots for bare steel samples, the low frequency part of Nyquist plot, below 0.409Hz, appearing a large degree of scattering, obviously without the definition of a clear relaxation process, and for this reason these results have not been considered. We can see that each spectrum includes an incomplete semicircle, the semicircle diameters of CR15 steel is larger than steels CR10 and CR12, which will indicate that the corrosion resistance of steel CR 15 is higher than steels CR10 and CR12.

Bode diagrams are shown in Fig. 7. It is seen that the impedance modulus $|Z|$ of experimental steels will gradually bigger with the increase of cooling rates in the low frequency range, and the impedance modulus $|Z|$ is biggest for CR15 steel, and the smallest one is for CR10 steel. As shown in Fig. 7(b), the broad-phase angle peak for the experimental steel can show at least two time constants. So the electrochemical behavior can be simulated by a simple equivalent circuit by the ZSimpWin software in Fig. 8. The dates are obtained from the equivalent circuits, which are listed in Table 3. R_s is the electrolyte resistance, which is resistance of the solution between the work electrode and Luggin capillary. The point where the Nyquist plot intersects the real axis at high frequency yields

Figure 6. (Color online) Nyquist plots of the bare steel samples in 10 wt% NaCl acid solution.

Figure 7. (Color online) Bode diagrams of the bare steel samples in 10 wt% NaCl acid solution. (a) The amplitude-frequency characteristic curve, (b) The phase-frequency characteristic curve.

Table 3. The Parameters of equivalent circuit for bare steel.

Samples	R_s ($\Omega \text{ cm}^2$)	Y_f ($\text{S}\cdot\text{sec}^n/\text{cm}^2$)	n_1	R_f ($\Omega \text{ cm}^2$)	Y_{dl} ($\text{S}\cdot\text{sec}^n/\text{cm}^2$)	n_2	R_{ct} ($\Omega \text{ cm}^2$)
CR10	9.407	0.0005196	0.6876	30.61	0.008544	0.4841	183.2
CR12	8.515	0.0004634	0.7034	40.53	0.005835	0.4597	197.6
CR15	11.42	0.0007729	0.7247	54.99	0.005281	0.5734	263.8

Figure 8. Equivalent circuits with two time constants. **Figure 9.** (Color online) Nyquist plots of the experimental steel samples after 72 h pre-corrosion in 10 wt% NaCl acid solution.

solution resistance. R_f is the resistance of corrosion product film. R_{ct} is the charge transfer resistance, which is used to represent the difficulty of the electrochemical reaction. CPE is the constant phase element, which is expressed as follows.

$$Z_{CPE} = 1/Y_0(j\omega)^n \quad (3)$$

where Y_0 is a general admittance function, j is the complex operator with $j = (-1)^{1/2}$, $\omega = 2\pi f$ is the angular frequency, n is a power, which is an adjustable parameter that lies between 0 and 1. CPE_f represents total surface electrode capacitance after electrode surface dispersion, n_1 ($0 < n_1 < 1$) is dispersion effect index of CPE_f , when $n_1 = 1$, CPE_f is equivalent to the ideal capacitance, when $n_1 = 0$, CPE_f is equivalent to the ideal resistance. CPE_{dl} represents the capacitance for solution/metal surface in the corrosion hole, n_2 ($0 < n_2 < 1$) is dispersion effect index of CPE_{dl} . The total impedance Z can be rewritten as

$$Z = R_s + \frac{1}{\frac{1}{R_f + \frac{1}{\frac{1}{Z_{CPE_{dl}}} + \frac{1}{R_{ct}}}} + \frac{1}{Z_{CPE_f}}} \quad (4)$$

In order to clarify the corrosion process, the typical Nyquist plots and Bode diagrams of the experimental steel after 72 h pre-corrosion are displayed in Fig. 9 and Fig. 10, respectively. From the Fig. 9,

Figure 10. (Color online) Bode diagrams of the experimental steel samples after 72 h pre-corrosion in 10 wt% NaCl acid solution. (a) The amplitude-frequency characteristic curve, (b) The phase-frequency characteristic curve.

the curve of CR10 steel appears the arc contraction phenomenon, which may result from the serious corrosion and even pitting corrosion. Also, the semicircle diameter of CR15 steel is larger than steels CR10 and CR12. With the increase cooling rate, the impedance modulus of the experimental steel will gradually increase in low frequency range from Fig. 10(a). It is seen that the impedance modulus of CR15 is biggest, which show that corrosion resistance of CR15 steel are better than other steels. The curve of the experimental steel consists of a deformed semicircle with two time constants. The equivalent circuit mode is the same as that shown in Fig. 8. The dates are obtained from the equivalent circuits, which are listed in Table 4. Table 4 shows that fitting results for test steels after 72 pre-corrosion steel in 10 wt% NaCl acid solution.

Based on the above analysis from Table 3 and Table 4, R_s changes a little, the average value is about $10 \Omega \text{ cm}^2$, and the electrolyte resistance is almost the same, which will explain each test system is in a state of relative stability. With the increasing of immersion time, Y_{dl} will fluctuate, it may be that the corrosion products leave the surface of the electrode, and get into the solution.

Table 4. The Parameters of equivalent circuit for 72 pre-corrosion steel.

Samples	R_s ($\Omega \text{ cm}^2$)	Y_f ($\text{S}\cdot\text{sec}^n/\text{cm}^2$)	n_1	R_f ($\Omega \text{ cm}^2$)	Y_{dl} ($\text{S}\cdot\text{sec}^n/\text{cm}^2$)	n_2	R_{ct} ($\Omega \text{ cm}^2$)
CR10	8.914	0.00004425	0.8921	1.198	0.004051	0.5085	165.4
CR12	9.681	0.00006825	0.9016	1.177	0.004049	0.4943	179.2
CR15	12.18	0.00002569	0.9234	1.209	0.004034	0.4732	300.6

The more obvious the fluctuation of Y_{dl} , and the more serious the samples surfaces are corrode. The Y_{dl} are generally used to account for the deviation caused by the surface roughness due to corrosion, which will results in surface inhomogeneity on the microscopic level, such as porosity.²⁵ The Y_{dl} is smallest for steel CR15, it may caused by high cooling rates. Y_f and R_f appear fluctuation phenomena, the R_f value is very small compared with other literatures, because the corrosion medium in this study is strong acid solution, the surface of the sample will not be able to form a stable protective film, even no appear layer on sample surface.

The polarization resistance from EIS can be expressed as $R_p = R_{ct} + R_f$, which has been widely used to account for the kinetics of electrochemical corrosion.²⁶ R_p is related to the corrosion current density i through the Tafel slopes b_a and b_c according to Stern-Geary equation.²⁷

$$i_{\text{corr}} = \frac{1}{2.303R_p \left(\frac{1}{b_a} + \frac{1}{|b_c|} \right)} \quad (5)$$

The corrosion current density for steels CR10, CR12 and CR15 is 0.173 mA, 0.145 mA and 0.080 mA, respectively. With the increase cooling rates, the R_p values will increase, the largest value is 300.6 $\Omega \text{ cm}^2$ for steel CR15, which showing that the CR15 steel has high corrosion resistance with lower corrosion current density. The obtained result by the EIS is consistent with those obtained by weight loss potentiodynamic polarization measurements.

To further verify the effectiveness of the fitting dates to an equivalent circuit, the relative residual errors for the real and imaginary components of the impedance (η_{re} and η_{im}) can be calculated using Eqs. (6) and (7), respectively. The residual errors can give a method for understanding the quality of the fit obtained.

$$\eta_{re} = \left(\frac{Z_{re,exp} - Z_{re,fit}}{Z_{re,exp}} \right) \times 100\% \quad (6)$$

$$\eta_{im} = \left(\frac{Z_{im,exp} - Z_{im,fit}}{Z_{im,exp}} \right) \times 100\% \quad (7)$$

where $Z_{re,exp}$ and $Z_{im,exp}$ are the real and imaginary impedances measured from the EIS experimental results, respectively. $Z_{re,fit}$ and $Z_{im,fit}$ are the real and imaginary impedances obtained from the equivalent circuit. Figures 11 and 12 indicate the relative residual errors curves of steels CR10, CR12 and CR15 for bare steel and 72 pre-corrosion steel in acid 10 wt% NaCl solution, respectively. The similar curves are obtained and the errors with the exception of the highest frequency point, are within 7%, which shows the quality of fits is very good. Residual errors are most frequently used to determine the fitting effect when determining the Kramers-Kronig compliance of the EIS data using the measurement model method.²⁸ In this case, the residual errors of 7% have indicated a good fitting, which also have shown a reasonable for the choice of the equivalent circuit.

4. Conclusions

The microstructure and the corrosion resistance of low alloy steel for COT with varying cooling rates have been investigated. The conclusions are as follows:

Figure 11. (Color online) Relative residual errors resulting from the equivalent circuits for bare steel CR10 (a), CR12 (b) and CR15 (c) in 10 wt% NaCl acid solution.

- 1) The microstructure of the experimental steel is primarily AF, i.e., QF,GF and a little BF. With the increasing cooling rates, the fine ferrite grain size will become more and more, and the percentage of low angle grain boundary will also increase.

Figure 12. (Color online) Relative residual errors resulting from the equivalent circuits for 72 pre-corrosion steel CR10 (a), CR12 (b) and CR15 (c) in 10 wt% NaCl acid solution.

- 2) The CR15 steel with higher cooling rate exhibits good corrosion resistance with a less weight loss, lower corrosion current density and bigger impedance modulus than steels CR10 and CR12 with lower cooling rate.
- 3) The relative residual error method can generate greater confidence in the results obtained from fitting an equivalent circuit model to EIS data.

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