

Short Communication

Preparation of Ni-W/PTFE Composite Coatings on Q345 Pipeline Steel and their Corrosion Resistance in Simulated Oilfield Produced Water

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Ni-W/PTFE composite coatings were prepared on the surface of Q345 pipeline steel by electrodeposition technology, and the effect of the concentration of PTFE particles added to the plating solution on the composition, surface morphology, surface roughness, and corrosion resistance in simulated oilfield produced water was investigated. The results show that with the increase of PTFE particles concentration from 2 g/L to 12 g/L, the content of PTFE particles in the composite coating first increases and then decreases, while the crystal cell size and surface roughness first decrease and then increase, playing a big role on the corrosion resistance in simulated oilfield produced water. The Ni-W/PTFE composite coating prepared by adding 8 g/L PTFE particles is compact and dense with only 0.372 μm surface roughness, and the charge transfer resistance and coating resistance are about 2.4 times higher than that of Q345 pipeline steel. The Ni-W/PTFE composite coating has a protection efficiency of 70.6%, which can significantly improve the corrosion resistance of Q345 pipeline steel in simulated oilfield produced water to prolong its service life.

Keywords: Q345 pipeline steel; Simulated oilfield produced water; Ni-W/PTFE composite coating; Corrosion resistance

1. INTRODUCTION

The whole process of oilfield development has the problem of corrosion. The oilfield produced water contains sodium chloride, acetic acid, sodium bicarbonate and other substances, which seriously corroded the steel pipeline [1-7]. Therefore, how to improve the corrosion resistance of steel pipeline to prolong its service life has become the focus and direction of efforts. As a result, the focus and direction of the work has shifted to how to increase the corrosion resistance of steel pipeline in order to extend its service life. In recent years, surface coating technology has been proved to significantly

improve the corrosion resistance of steel pipeline.

Many surface coating technologies can be used to improve the corrosion resistance of steel pipeline, such as electrodeposition, surface passivation, phosphorization, electroless deposition and so on [8-12]. Meanwhile, electrodeposition is a kind of common surface coating technology, which has the advantages of stabilization and controllability. It has been discovered that nickel based alloy prepared by electrodeposition has the best corrosion resistance and mechanical properties. Moreover, some nonmetal and metal particles doping could further improve the chemical and physical properties of nickel based alloy coating reported in some papers [13-17]. PTFE (Polytetrafluoroethylene) is a kind of polymer materials with higher impedance and lower friction coefficient, which has better corrosion resistance in the environment containing sodium chloride and acid solution [18-19]. Therefore, nickel based alloy coating doping with PTFE coating is suitable to protect steel pipeline for oil transportation.

In this paper, Q345 pipeline steel is selected as the research object and simulated oilfield produced water is taken as the corrosion medium. Ni-W/PTFE composite coatings were electrodeposited by introducing PTFE particles based on the principle of codeposition mechanism. The aim is to improve the corrosion resistance of Q345 pipeline steel in simulated oilfield produced water by using the characteristics of Ni-W/PTFE composite coating.

2. EXPERIMENTAL

2.1 Pretreatment and materials

The chemical composition of Q345 pipeline steel is 1.70%Mn, 0.20%C, 0.55%Si, 0.02~0.15%V, 0.03%P, 0.03%S and residual Fe. The samples were polished, washed by alkali solution (45 g/L sodium hydroxide+12 g/L sodium carbonate, soaked at 60°C for 8 min), dipped into acid solution (10% hydrochloric acid, soaked at room temperature for 1 min), cleaned and dried with distilled water in sequence.

2.2 Component of simulated oilfield produced water

The component of simulated oilfield produced water is listed in Table 1. The reagents used are all analytically pure grades, and distilled water is added to a constant volume of 1000 mL. Carbon dioxide gas is pumped into the simulated oilfield produced water for 2 h to remove oxygen.

Table 1. Component of simulated oilfield produced water

Reagents	NaCl	CH ₃ COOH	CaCl ₂	Na ₂ SO ₄	NaHCO ₃	KCl
Concentration/ (g·L ⁻¹)	16.6	2.0	1.8	1.3	3.2	0.4

2.3 Preparation of Ni-W/PTFE composite coatings

Ni-W/PTFE composite coatings were prepared on the surface of Q345 pipeline steel by electrodeposition technology. The plating solution was composed of analytical pure nickel sulfate, sodium tungstate, boric acid, ammonium citrate dibasic and sodium dodecyl sulfate shown in Table 2. The bath heating keeps the bath temperature at $(60\pm 0.5)^{\circ}\text{C}$. KPS3040D DC regulated power supply is adopted and the current density is set to 2 A/dm^2 . During the electrodeposition process, the plating solution was continuously stirred at stirring rate of 300 r/min to ensure that the PTFE particles were fully wetted and evenly dispersed. After electrodeposition for 60 minutes, the sample was taken out and dried.

Table 2. Component of plating solution for Ni-W/PTFE composite coatings

Reagents	Concentration/ ($\text{g}\cdot\text{L}^{-1}$)
$\text{NiSO}_4\cdot 6\text{H}_2\text{O}$	24
$\text{Na}_2\text{WO}_4\cdot 2\text{H}_2\text{O}$	45
H_3BO_3	30
$\text{C}_6\text{H}_{14}\text{N}_2\text{O}_7$	100
$\text{C}_{12}\text{H}_{25}\text{SO}_4\text{Na}$	0.06
PTFE particles	2, 5, 8, 12

2.4 Characterization and testing of Ni-W/PTFE composite Coatings

2.4.1 Surface morphology and composition

The surface morphology of Ni-W/PTFE composite coating was characterized by Supra55 scanning electron microscope, and the composition of Ni-W/PTFE composite coating was analyzed by X-Max energy dispersive spectrometer, and the mass fraction of each element was obtained. According to the relative molecular weight relationship between F element and PTFE, the content of PTFE particles in the composite coating was calculated. In addition, a Micro Phase 3D microscope camera was used to characterize the three-dimensional morphology of Ni-W/PTFE composite coating and measure the surface roughness.

2.4.2 Corrosion resistance testing

Q345 pipeline steel electrodeposited with Ni-W/PTFE composite coating was used as the working electrode, platinum sheet as the counter electrode and saturated calomel electrode as the reference electrode. The three electrode system was immersed in simulated oilfield produced water for a period of time, and the electrochemical impedance spectrum was tested after the open circuit potential was stabilized. 5 mV sine wave excitation signal was applied to scan from 100 kHz high frequency to 10 mHz low frequency. The test data were imported into ZSimpWin software for fitting,

and the charge transfer resistance, coating resistance and low frequency impedance values were obtained. According to the charge transfer resistance, the protective efficiency of different Ni-W/PTFE composite coatings on Q345 pipeline steel was estimated. A full immersion corrosion experiment was carried out in simulated oilfield produced water with Q345 pipeline steel sample and Ni-W/PTFE composite coating as the research objects. After 240 h, the sample was taken out, the corrosion products on the surface were cleaned with rust remover, and the corrosion weight loss was calculated with FA2204N electronic balance. The corrosion protection effect of Ni-W/PTFE composite coating on Q345 pipeline steel was studied using charge transfer resistance, coating resistance, low frequency impedance, protection efficiency, and corrosion weight loss.

3. RESULTS AND DISCUSSION

3.1 The composition of Ni-W/PTFE composite coatings

Table 3 lists the composition of different Ni-W/PTFE composite coatings. Table 3 shows that different composite coatings are composed of Ni, W, C and F elements, among which F element comes from PTFE, which can confirm that PTFE particles are doped in the composite coatings along with the deposition process. When 2 g/L PTFE particles are added, the mass fraction of F element is relatively low, about 1.47%, and the content of PTFE particles in the composite coating is estimated to be about 1.93%, as shown in Figure 1. As the concentration of PTFE particles increased, the mass fraction of F element increased first and then decreased, and the content of PTFE particles in the corresponding composite coating increased from 1.93% to 3.87% and then decreased to 2.96%, as shown in Figure 1. Analysis shows that the increase of the concentration of PTFE particles increases the number of uniformly dispersed particles in the plating solution, so the particles attached to the deposition surface and doped in the composite coating with the deposition process increase under the liquid flow disturbance. However, when the concentration of PTFE particles added is too high, the agglomeration effect of particles in the bath resulting in fewer particles attached to the deposition surface, so the content of PTFE particles in the composite coating decreases along with the deposition process. When the concentration of PTFE particles introduced is too high, the agglomeration effect of the particles in the bath causes fewer particles to adhere to the deposition surface, resulting in a drop in the content of PTFE particles in the composite coating during the deposition process. The agglomeration effect and mechanism of nano particles are found and reported by some people [20-22].

Table 3. Composition of different Ni-W/PTFE composite coatings

concentration of PTFE particles/ (g·L ⁻¹)	mass fraction of Ni/ %	mass fraction of W %	mass fraction of C %	mass fraction of F %
2	67.45	21.03	10.05	1.47
5	67.11	20.32	10.09	2.48
8	66.94	20.15	9.97	2.94
12	67.03	20.27	10.45	2.25

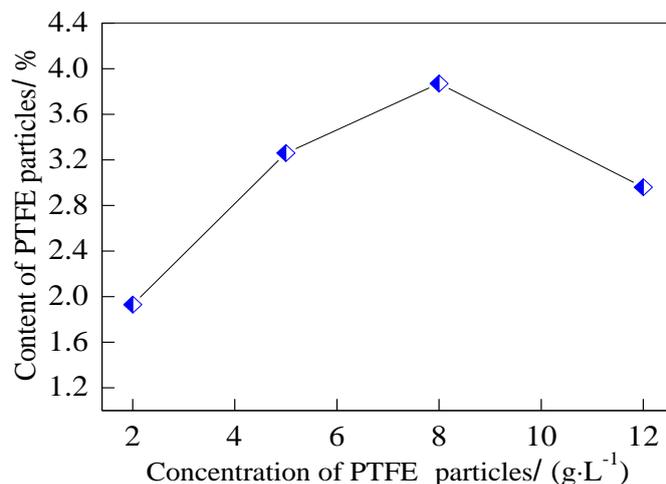


Figure 1. Content of PTFE particles in Ni-W/PTFE composite coatings;

3.2 The surface morphology of Ni-W/PTFE composite coatings

Figure 2 shows the surface morphology of Q345 pipeline steel and different Ni-W/PTFE composite coatings. By comparison, Ni-W/PTFE composite coating was successfully prepared on the surface of Q345 pipeline steel. It can be seen from Figure 2(b) that when 2 g/L PTFE particle is added, the composite coating is in the shape of cauliflower, and uneven and approximately spherical crystal cells are distributed on the surface, but the crystal cells are not compact. With the increase of PTFE particles concentration, the crystal cell diameter of the composite coating decreases.

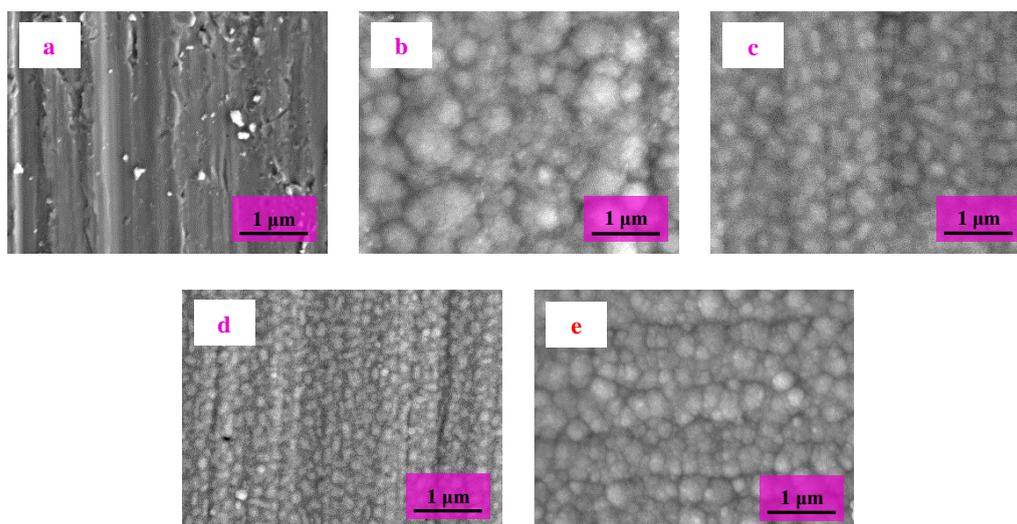


Figure 2. Surface morphology of Q345 pipeline steel and different Ni-W/PTFE composite coatings; a-Q345 pipeline steel; b-concentration of PTFE particles 2 g/L; c-concentration of PTFE particles 5 g/L; d-concentration of PTFE particles 8 g/L; e-concentration of PTFE particles 12 g/L; automatic focusing mode and image contrast adjustment, acceleration voltage 15 kV;

This is due to the fact that the particles doped in the composite coating increase in number as

the deposition process progresses, occupying the cell growth site and preventing cell growth [23-25]. The doping particles or metals are beneficial to inhibit cell growth resulting in dense structure which is also reported by many researchers [26-33]. As shown in Figure 2(d), the surface of Ni-W/PTFE composite coating prepared by adding 8 g/L PTFE particles is uniform and density with smaller crystal cell. But when PTFE particles concentration is too high, particle aggregation effect makes it difficult to effectively promote crystallization nucleation which resulted in increase of crystal cell size and decrease of composite coating density, as shown in Figure 2 (e). Figure 3 shows the three-dimensional morphology of different Ni-W/PTFE composite coatings. With the increase of PTFE particles concentration from 2 g/L to 8 g/L, the surface roughness of composite coating decreases from 0.784 μm to 0.372 μm , as shown in Figure 4. It is confirmed that appropriate addition of PTFE particles concentration can improve the surface smoothness of composite coating and improve its compactness. However, with the increase of PTFE particles concentration from 8 g/L to 12 g/L, the surface roughness of the composite coating increases to 0.578 μm , which proves that the high concentration of PTFE particles is not beneficial to improve the surface flatness of the composite coating, but also reduces its compactness. The surface roughness of metal alloy films and effect of PTFE on surface structure are also investigated in some literatures [34-38].

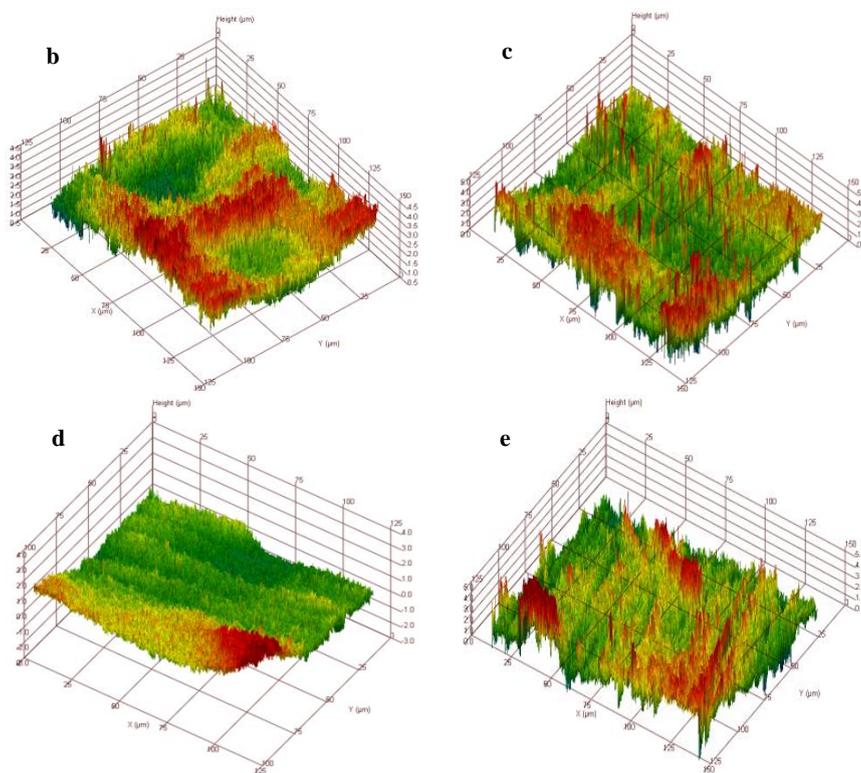


Figure 3. Three-dimension morphology of different Ni-W/PTFE composite coatings; b-concentration of PTFE particles 2 g/L; c-concentration of PTFE particles 5 g/L; d-concentration of PTFE particles 8 g/L; e-concentration of PTFE particles 12 g/L; The sample size is 10 mm×10 mm, the resolution is 752×480 pixels, and the repetition accuracy was 1 nm;

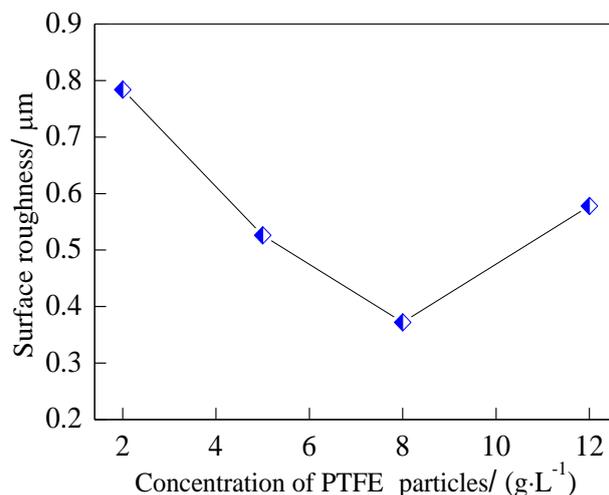


Figure 4. Surface roughness of different Ni-W/PTFE composite coatings; The sample size is 10 mm×10 mm, scanning rate 10 μm/s, scanning length 4000 μm.

3.3 Corrosion resistance of Ni-W/PTFE composite coating

3.3.1 Electrochemical impedance spectroscopy

Figure 5 shows the electrochemical impedance spectra of Q345 pipeline steel and different Ni-W/PTFE composite coatings. Table 4 lists the fitting results of electrochemical impedance spectra. It can be seen from Figure 5(a) that compared with Q345 pipeline steel, the impedance spectrum radius of different Ni-W/PTFE composite coatings all increases first and then decreases with the increase of PTFE particles concentration from 2 g/L to 12 g/L. According to Table 4, when 2 g/L PTFE particles are added, the composite coating is weak in blocking the erosion of the corrosive medium in simulated oilfield produced water due to its large crystal cells and roughness surface. Charge transfer process is easy to occur on the surface of the composite coating. The charge transfer resistance is relatively low. In addition, the corrosion coating formed on the surface of the composite coating prepared by adding 2 g/L PTFE particles is not too dense in the electrochemical corrosion process, which is difficult to effectively inhibit the further development of electrochemical corrosion, and also shows that the resistance of the coating layer is relatively low. As the concentration of added PTFE particles increases, the crystal cells of the composite coating are refined and the surface flatness and compactness are gradually improved resulting in gradual increase of the charge transfer resistance. The Ni-W/PTFE composite coating prepared by adding 8 g/L PTFE particles has the highest charge transfer resistance and coating resistance, which are 6578.4 Ω·cm² and 877.1 Ω·cm², respectively. Compared with Q345 pipeline steel, the charge transfer resistance and coating resistance are increased about 2.4 times. The results show that the composite coating has good corrosion resistance and can effectively inhibit the electrochemical corrosion of Q345 pipeline steel.

It can be seen from Figure 5(b) that compared with Q345 pipeline steel, the low frequency impedance values of Ni-W/PTFE composite coatings all increase first and then decrease with the increase of PTFE particles concentration from 2 g/L to 12 g/L. The low frequency impedance value

can reflect the blocking effect of the composite coating on the corrosive medium in simulated oilfield produced water, and is also an evaluation index of the corrosion resistance of the composite coating. The Ni-W/PTFE composite coating prepared by adding 8 g/L PTFE particles has the highest low frequency impedance, reaching $1.35 \times 10^4 \Omega \cdot \text{cm}^2$, which is nearly 3 times higher than that of Q345 pipeline steel. It also shows that the composite coating can block the corrosive medium in the simulated oilfield produced water well, thus delaying the corrosion of Q345 pipeline steel.

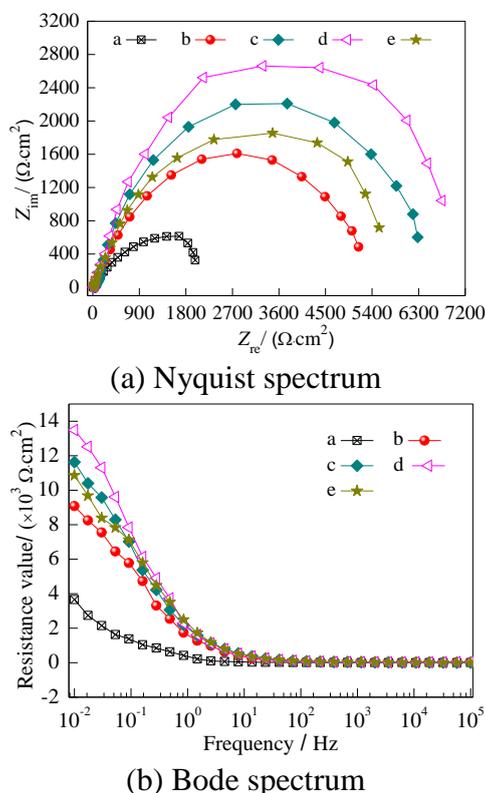


Figure 5. Electrochemical impedance spectra of Q345 pipeline steel and different Ni-W/PTFE composite coatings; a-Q345 pipeline steel; b-concentration of PTFE particles 2 g/L; c-concentration of PTFE particles 5 g/L; d-concentration of PTFE particles 8 g/L; e-concentration of PTFE particles 12 g/L; simulated oilfield produced water as the corrosive medium; Ni-W/PTFE composite coating was used as the working electrode, platinum sheet as the counter electrode and saturated calomel electrode as the reference electrode; 5 mV sine wave excitation signal was applied to scan from 100 kHz high frequency to 10 mHz low frequency;

Table 4. Fitting results of electrochemical impedance spectra

Samples	Charge transfer resistance / ($\Omega \cdot \text{cm}^2$)	Coating resistance / ($\Omega \cdot \text{cm}^2$)	Low frequency impedance/ ($\Omega \cdot \text{cm}^2$)
a	1932.6	257.7	3.66×10^3
b	5082.3	677.6	9.08×10^3
c	6220.5	829.4	1.16×10^4
d	6578.4	877.1	1.35×10^4
e	5479.0	730.5	1.09×10^4

Figure 6 shows the protective efficiency of different Ni-W/PTFE composite coatings on Q345 pipeline steel. As shown in Figure 6, with the increase of PTFE particles concentration from 2 g/L to 12 g/L, the protection efficiency of the composite coating on Q345 pipeline steel increased from 62.0% to 70.6% and then decreased to 64.7%. The composite coating prepared by adding 8 g/L PTFE particles has the highest protection efficiency of 70.6% on Q345 pipeline steel. The composite coating can play a good corrosion protection effect and effectively inhibit the corrosive medium in the simulated oilfield produced water, thus delaying the corrosion of Q345 pipeline steel. The corrosion resistance of Ni-W alloy coating was reported by some investigators [39-41]. Compare with Ni-W alloy coatings, the Ni-W/PTFE prepared in the paper possesses better corrosion resistance.

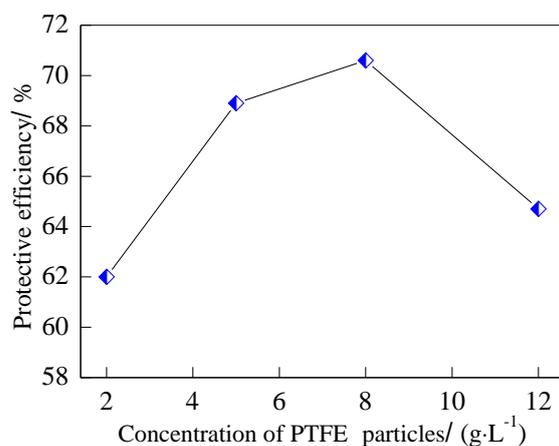


Figure 6. Protective efficiency of different Ni-W/PTFE composite coatings on Q345 pipeline steel; protective efficiency was estimated according to the charge transfer resistance;

3.3.2 Immersion corrosion testing

The corrosion weight loss of Q345 pipeline steel and different Ni-W/PTFE composite coatings are listed in Table 5. Compared with Q345 pipeline steel, the corrosion weight loss of different Ni-W/PTFE composite coatings decreases first and then increases with the increase of PTFE particle concentration from 2 g/L to 12 g/L.

Table 5. Corrosion weight loss of Q345 pipeline steel and different Ni-W/PTFE composite coatings; simulated oilfield produced water as the corrosive medium, immersion time 240 h

Samples	Corrosion weight loss/ (mg·dm ⁻²)
a	231.09
b	99.45
c	82.46
d	73.08
e	93.50

As the concentration of added PTFE particles increases, the surface flatness and compactness of the composite coating gradually improve, and the increased content of PTFE particles enhances corrosion resistance of coatings in the corrosive medium, so the corrosion weight loss decreases. The optimal corrosion resistance of PTFE particles was verified by many works [42-44]. However, when the concentration of PTFE particles added is too high, due to particle agglomeration effect, the content of PTFE particles doped in the composite coating is small with larger crystal cell and rougher surface. Therefore, the degree of corrosion becomes more serious, showing an increase in corrosion weight loss. The corrosion weight loss of Ni-W/PTFE composite coating prepared by adding 8 g/L PTFE particles is 73.08 mg/dm², which is about 68% lower than that of Q345 pipeline steel.

4. CONCLUSIONS

(1) Ni-W/PTFE composite coating was successfully prepared on the surface of Q345 pipeline steel which is mainly composed of Ni, W, C and F elements with spherical crystal cells morphology. PTFE particles were doped in the composite coating along with the deposition process, which prevented the corrosion of corrosive medium in the simulated oilfield produced water and inhibited the electrochemical corrosion of Q345 pipeline steel.

(2) The concentration of PTFE particles added in the plating solution has a certain effect on the composition, compactness, surface roughness, corrosion resistance of Ni-W/PTFE composite coatings. The Ni-W/PTFE composite coating prepared by adding 8 g/L PTFE particles has small crystal cell structure, smooth and compact surface. Compared with Q345 pipeline steel, the charge transfer resistance and coating resistance of Ni-W/PTFE composite coating is about 2.4 times higher and the low frequency impedance value is nearly 3 times higher. In addition, the protection efficiency of Ni-W/PTFE composite coating can reach 70.6% and its corrosion weight loss is about 68% lower than Q345 pipeline steel. The composite coating can significantly improve the corrosion resistance of Q345 pipeline steel in simulated oilfield produced water.

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