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Study on Epoxy-Phosphate Conversion Coating

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Abstract:

E44 epoxy resin was reacted with phosphoric acid to prepare epoxy-phosphate conversion coating. The coating was applied on the surface of rusty Q235 steel to form rust conversion coating. The effect of rust conversion coating was evaluated by photo recording, scanning electron microscopy and energy dispersive spectrometry. The performance of rust conversion coating was characterized by adhesion test and salt spray test. The results show that the coating with the molar ratio of E44 / phosphoric acid of 1:3 has the best adhesion, 3.4 MPa; the coating with the molar ratio of E44 / phosphoric acid of 1:2 has the longest salt spray test time, 500 h.

Keywords: *Epoxy phosphate, Rust conversion coating, Carbon steel, Corrosion and protection.*

I. INTRODUCTION

As the most effective, economical anti-corrosion methods, organic anti-corrosion coating has been widely used in metal base industries[1]. Generally, the surface of steel has caused a certain degree of corrosion during storage and transportation. It is required that the corrosion substances must be completely removed before coating to ensure that the coating has good adhesion and protection ability[2]. Therefore, in the general coating process, the cost of surface treatment (rust removal, oil removal, etc.) based on sand blasting accounts for more than 30% of the whole project[3,4]. However, in some harsh field conditions, it can't reach the standard of sand blasting Sa2.5 or above, so the new requirements of low surface treatment, anti-corrosion coatings with capabilities such as rust and moisture on the surface of steel are put forward[5,6].

Since 1970's, rusty coatings appeared in China, it has developed rapidly in recent decades. The most obvious advantage of rusty coating is that it can be directly applied on the metal surface with rust or incomplete rust removal, and has good adhesion. On the whole, in addition to the performance of general anti rust coating, the rusty coating must also have two special abilities: one is that it must have enough penetration ability to the rust layer, that is, it must fully infiltrate and penetrate the entire loose and porous rust layer, so that the rust can be bonded into a continuous closed coating to seal the entire rust layer and the steel surface; Second, it must have enough reactivity to the rust layer, that is, it can make the active and harmful ferrites in the rust layer passivate and transform into stable harmless fillers. Only with these two conditions can it adapt to the special surface such as rust layer.

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Rust converter can directly produce a black protective film on rusty metal[1], the film has good adhesion, matching with primer, and can significantly improve the corrosion resistance of primer. Water-based phosphoric acid-potassium ferrous cyanide conversion fluid rust conversion liquid can enhance the rust-proof effect of steel. An organic protective film with good adhesion formed by the reaction of phosphoric acid-tannin conversion fluid and rust[5].

In this paper, E44 epoxy resin and phosphoric acid were used to prepare an epoxy- phosphate based rust conversion coating. An organic-inorganic hybrid rust conversion layer was obtained on the surface of Q235 steel corroded for 3 h in salt spray. The effect and comprehensive performance of the rust conversion layer were evaluated.

II. EXPERIMENT

2.1 Materials

The base metal is Q235 steel, and its chemical composition is shown in Table I.

Table I Chemical composition of Q235 steel (wt.%)

C	Mn	Si	S	P	Cu	Fe
0.14-0.22	0.3-0.65	≤0.30	≤0.05	≤0.045	≤0.045	residual

Before use, the steel specimens were sandblasted with 120 mesh glass beads to remove the oxide film, floating rust and other pollutants on the surface. After ultrasonic wash for 30 minutes with acetone, the sandblasted steel specimens were quickly dried by electric hair dryer and put into the dryer for standby.

2.2 Test Medium

The epoxy resin is diglycidyl ether bisphenol A epoxy resin, its model is E-44, and the epoxy value is 0.41 ~ 0.47mol/100g, which is produced by Shanghai resin factory. The curing agent is a modified polyamine, type 5772, amine value 660 ~ 850mgkoh / g, active hydrogen equivalent 48. Shanghai resin factory.

Rust destroyer (No. D anticorrosive coating) produced by app company of the United States was used as the contrast sample.

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Table II. Chemicals used in the experiment

Chemicals	Grade	Manufacturer
Phosphoric acid	Analytically pure	Sinopharm Chemical Reagent Co., Ltd
Xylene	Analytically pure	Sinopharm Chemical Reagent Co., Ltd
Acetone	Analytically pure	Sinopharm Chemical Reagent Co., Ltd
N-butanol	Industrial grade	Sinopharm Chemical Reagent Co., Ltd
Rapid curing AB glue		American 3M company

Preparation of epoxy-phosphate esters: When the molar ratio of epoxy resin to phosphoric acid is less than 1.0, the rust conversion ability of the coating is very weak, it is impossible to turn the rust layer into the composition of the coating, the coating is not attached to the metal surface after curing, there is a rust layer between the coating and the metal. When the molar ratio of epoxy resin to phosphoric acid is more than 1.5, the coating appears gel phenomenon, can not be properly coated. So the synthesis methods are as follows. Weigh 22.85g E44 epoxy resin into a 100ml conical flask, add 40ml acetone, and add phosphoric acid according to the molar ratio of E44: H_3PO_4 = 1:2, 1:3, 1:4, 1:5. The conical flask was placed on a magnetic stirrer to mix E44 with phosphoric acid evenly, then heated to 65 ℃ for 1 h, and then heated to 85 ℃ for refluxing reaction for 1 h. Remove the reflux device and react at 85 ℃ for 0.5-1 h to make the excess acetone solvent volatilize and take away the water in the system in the process of acetone volatilization. When the viscosity of the system is appropriate, stop heating and cool to room temperature to obtain four epoxy-phosphate esters. Reaction mechanism of preparation of epoxy-phosphate as shown in Figure 1.

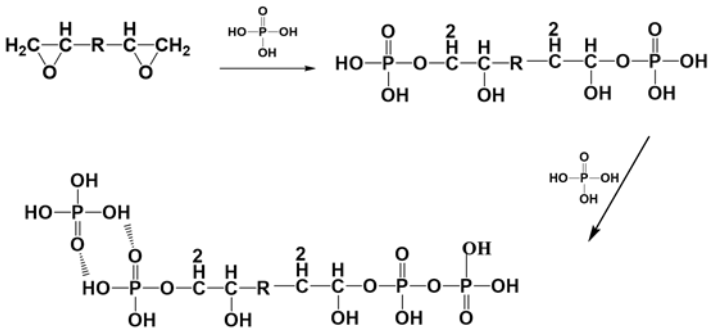

Fig 1: Reaction mechanism of epoxy-phosphate preparation

Preparation of rust conversion anticorrosive coating: Xylene and n-butanol were used to adjust the four kinds of epoxy phosphate to the appropriate concentration, and brush each kind of epoxy phosphate conversion solution on the corrosion surface of Q235 steel was exposed to salt spray test for 3 h, and then it was placed at room temperature for 6-12 h. When the decomposition and blackening of corrosion

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products were obviously observed, and the surface of conversion solution was dry at room temperature for one week.

III. RESULTS AND DISCUSSION

3.1 Macro Analysis of Rust Transformation Effect about Coating

The results show that the epoxy-phosphate tbased conversion solution can transform the corrosion on Q235 steel surface, and the higher ratio of phosphoric acid in the epoxy-phosphate esters, the better of the conversion effect.

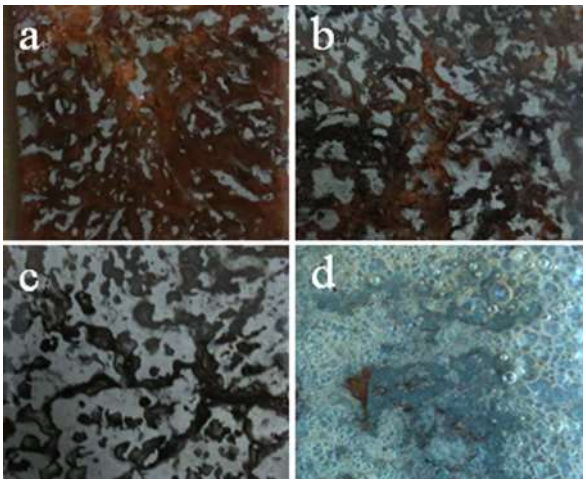

Fig 2: Morphology of the rust conversion anticorrosive coating (E44: H_3PO_4= a: 1:2, b: 1:3, c: 1:4, d: 1:5)

3.2 Micro Analysis of Rust Transformation Effect about Coating

When the E44: H_3PO_4 molar ratio is 1:4, the conversion effect is the best, the conversion layer has uniform composition, the inorganic phase particles are small, the organic-inorganic phase has good compatibility, as shown in Figure 3.

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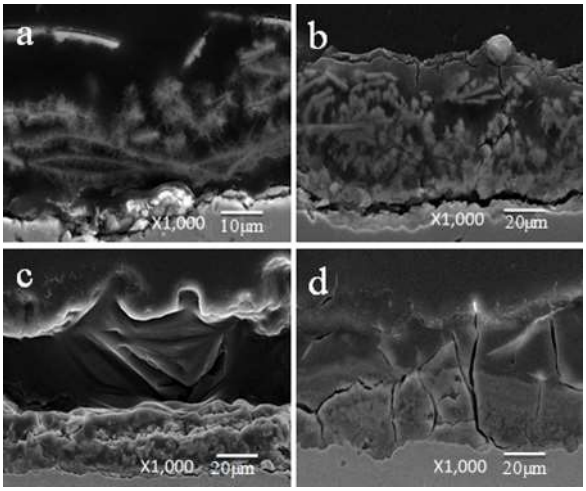

Fig 3: SEM analysis of coating section (E44: H_3PO_4= a: 1:2, b: 1:3, c: 1:4, d: 1:5)

When the molar ratio of E44:H_3PO_4 is 1:4, the conversion coating contains O, P and Fe. It is proved that the layer is composed of conversion product and epoxy phosphate ester, as shown in Figure 4.

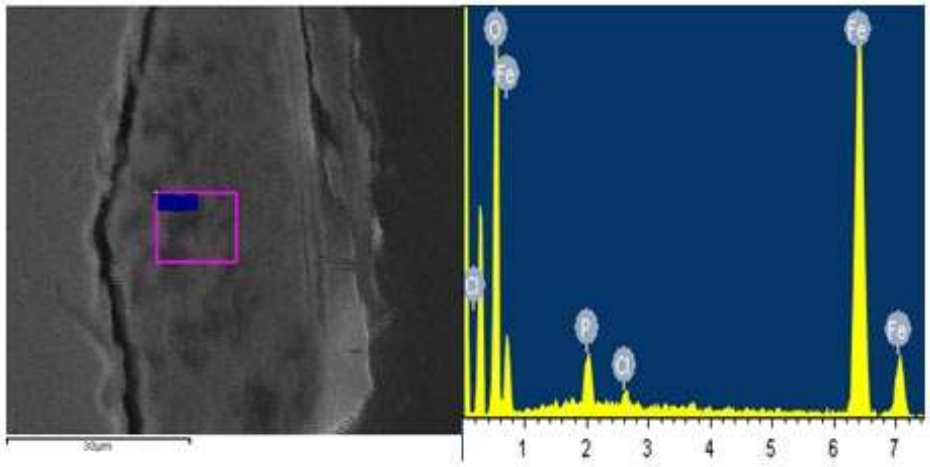

Fig 4: SEM / EDS analysis of coating section (E44: H_3PO_4=1:4)

The adhesion performance of the four rust conversion is shown in Figure 5. The adhesion value of E44: H_3PO_4 with 1:3 molar ratio was 3.4Mpa, and that of E44: H_3PO_4 with 1:2 molar ratio was 2.4Mpa, E44: H_3PO_4 molar ratio of 1:4 group and app company No. D were 2.8MPa and 2.7Mpa respectively.

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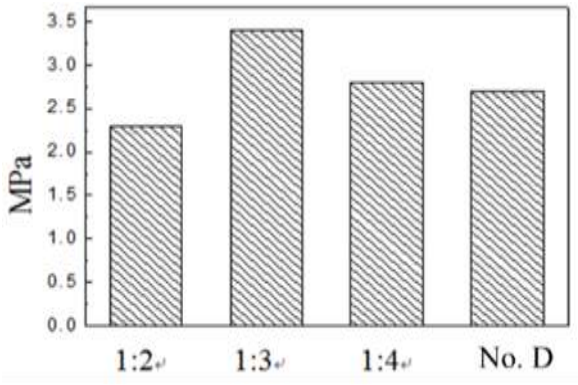

Fig 5: Adhesion of coating with different rust conversion (E44: H_3PO_4 = 1:2, 1:3, 1:4 and No. D)

Salt spray resistance test of epoxy phosphate rust conversion coating: The salt spray resistance time of E44: H_3PO_4 with 1:2 molar ratio was 500 h, and that of E44: H_3PO_4 with 1:3 molar ratio was 380 h, and that of E44: H_3PO_4 with 1:4 molar ratio was 290 h, and that of app company No.D was 260 h.

IV. CONCLUSION

(1) Different mole ratios of epoxy-phosphate based rust conversion coating were prepared. The experimental results showed that epoxy phosphate type rust conversion solution had conversion effect on corrosion products of Q235 steel under salt spray condition.

(2) When the molar ratio of E44: H_3PO_4 is 1:3 and 1:4, the conversion effect of rust conversion coating is better, and the formed conversion layer has better adhesion to the metal matrix.

(3) The salt spray resistance of the composite anti-corrosion coating decreases with the increase of phosphoric acid content in the epoxy phosphate conversion coating. When the molar ratio of E44: H_3PO_4 is 1:2, the salt spray resistance time of the coating is the longest, and the best value is 500 h.

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