



# Surface characterization and corrosion resistance of fluoferrite conversion coating on carbon steel



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## ABSTRACT

Fluoferrite conversion coating has been studied for the corrosion protection of carbon steel by using the conversion solution mainly containing potassium fluoride, ammonium persulfate and other compositions. As a summary, this paper gave the detailed properties of the conversion coating prepared with hydrogen peroxide as the best composition. The morphology, composition and corrosion resistance of the coating were investigated by scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), electron probe micro-analyzer (EPMA) and linear polarization, electrochemical impedance spectroscopy (EIS) and the salt spray test (SST). The results showed that the coating, mainly consisting of  $K_3FeF_6$ , exhibited good corrosion resistance (up to 72 h salt spray time) due to its close-packed particle structure and the great thickness (73  $\mu\text{m}$ ).

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## 1. Introduction

Carbon steel is used in large tonnages in marine applications, nuclear power and fossil fuel power plants, transportation, chemical processing, petroleum production and refining, pipelines, mining, construction and metal-processing equipment [1]. But carbon steel is easily corroded in aggressive solutions and atmospheres. Phosphating is one of the most commonly used treatment methods to improve the adhesion of paint to the underlying steel substrate and to prevent the substrate from corrosion [2–4]. However, phosphate discharges from the concentrated phosphate baths have a detrimental effect on groundwater sources because phosphorous is the most common cause of eutrophication in freshwater lakes and reservoirs [5–6]. Chromate treatment is a process that resembles phosphating and is often used to passivate galvanizing steel owing to high corrosion resistance, good paint adhesion and cost-effectiveness. However, cyanide used in the galvanizing process is highly toxic, and treatments based on hexavalent chromium have been banned to use since the RoHS regulation was enacted [7]. Thus, it is necessary to develop new non-phosphorus and non-chromate conversion treatment technologies on carbon steel surface.

In the last decade, research on new surface treatments had been performed on carbon steel. Fedrizzi et al. [8] reported that corrosion performance was better than phosphate coating for zirconia coating on low carbon steel deposited via the sol–gel method. A hexafluorozirconic acid based conversion coating was also obtained through immersion method on steel substrate [5,9–10]. The results revealed that the hexafluorozirconic acid pretreatment improved the corrosion

resistance and adhesive performance of carbon steel. Other surface treatments have emerged as a possible and valuable alternative. The nanocomposite superhydrophobic coatings were prepared on low-carbon steel and the peculiarities of the corrosion processes on the coatings in NaCl solution were studied by the electrochemical methods and the analysis of wetting [11].

In 2009, our research group first prepared fluoferrite conversion coating on carbon steel with the conversion solution mainly containing potassium fluoride and ammonium persulfate [12–14]. Then, the effect of different compositions on the corrosion resistance of the coating had been studied [15–18]. Results indicated that fluoferrite conversion coating prepared from the solution consisted of potassium fluoride, ammonium persulfate and hydrogen peroxide showed the optimal corrosion protective performance. As a summary, this paper aims to study the detailed information regarding the surface morphology, composition and corrosion resistance of the fluoferrite conversion coating prepared under the optimal condition, which was screened in the previous works. The corrosion resistance of this coating was compared with that of traditional phosphate coating.

## 2. Material and methods

### 2.1. Reagents and materials

All chemicals were commercial materials of the high available purity (analytical grade), and were used without receiving any further purification. Solutions were prepared with deionized water. The sample was carbon steel, with the size of 4 cm × 4 cm × 0.1 cm. Its composition was 0.12% C, 0.05% S, 0.5% Mn, 0.3% Si, 0.045% P and balanced Fe (wt%).

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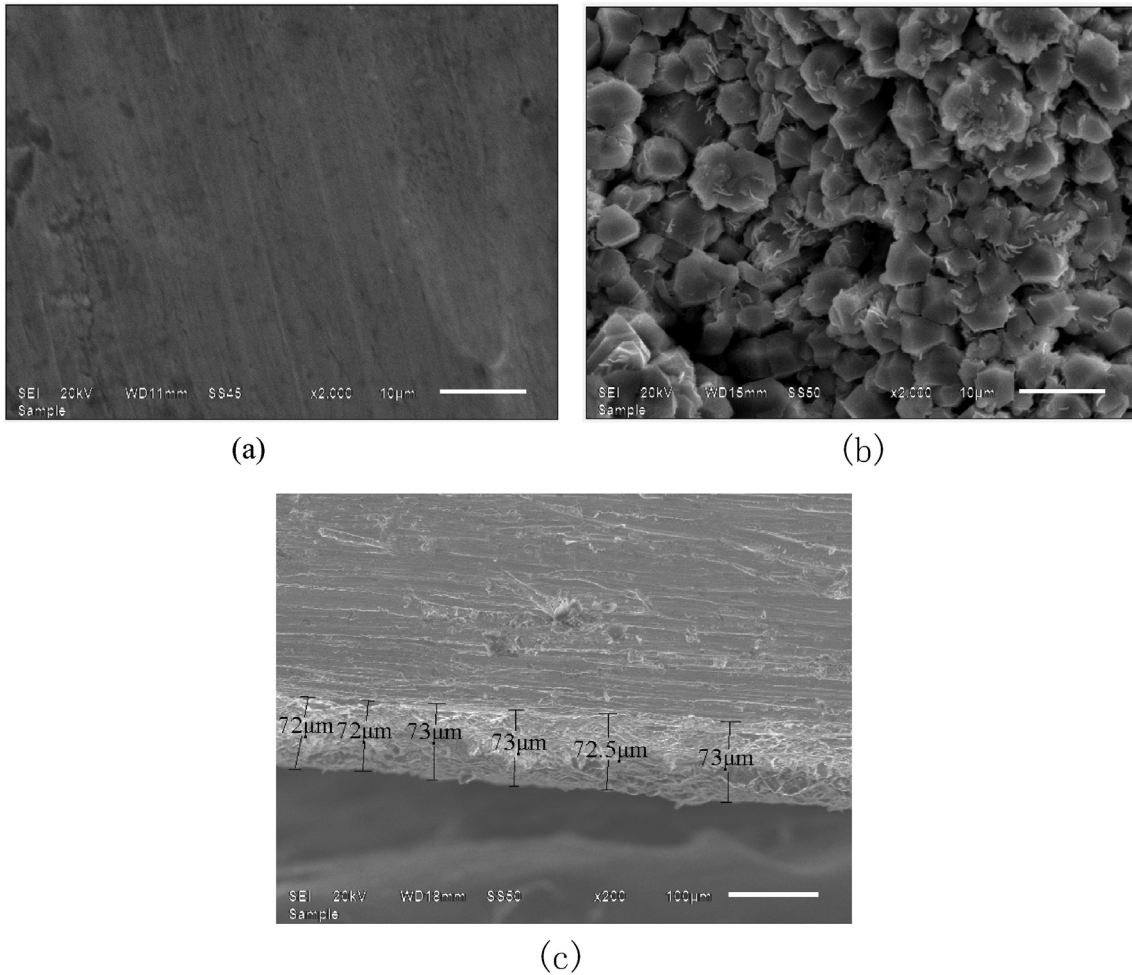


Fig. 1. SEM micrograph of different samples: a) bare sample (2000×); b) fluoferrite conversion coating (2000×); c) the cross-section profile of the fluoferrite coated sample.

2.2. Sample preparation

The sample was firstly degreased with an alkaline solution (35 g/L NaOH, 35 g/L Na<sub>2</sub>CO<sub>3</sub>, 35 g/L Na<sub>3</sub>PO<sub>4</sub>, 5 g/L Na<sub>2</sub>SiO<sub>3</sub>) at 85 °C for 5 min, then derusted with 20 wt% HCl containing 54 g/L hexamethylene tetramine at room temperature for 5–10 min, finally, cleaned with deionized water for the following conversion reaction.

The cleaned sample was immersed in an optimal solution of 2.2 M potassium fluoride, 0.03 M ammonium persulfate and 85–105 mL/L hydrogen peroxide (30%, mass fraction). The optimal reaction condition

was 50 °C, 2 h and pH 2 [15]. The pH was adjusted with HNO<sub>3</sub>. After the treatment, the coated sample was rinsed with deionized water and dried by blowing hot air. Finally, the coated sample was tested and analyzed.

The traditional phosphate treatment [19] was carried out for comparison. The composition of the traditional phosphate coating treatment bath was phosphate ions 19.8 g/L (from addition of 85% phosphoric acid); zinc ions 14.7 g/L (from addition of zinc oxide); nitric ions 23.5 g/L (from addition of nitric acid). The solution's pH was adjusted to 2.5 using sodium hydroxide. Treatment was performed at 60 °C and the immersion time was 15 min.

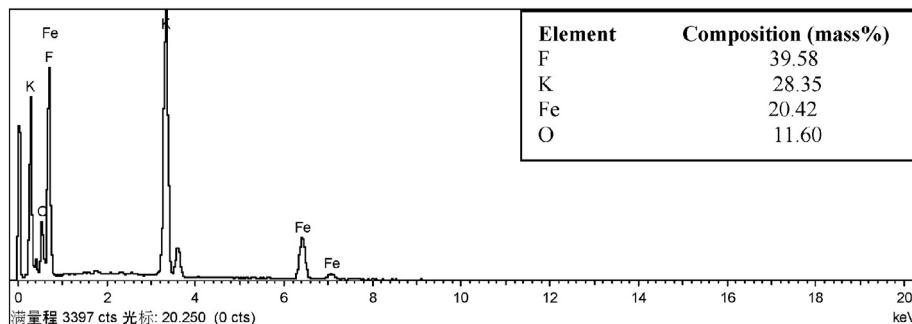


Fig. 2. EPMA analysis of the fluoferrite coating on carbon steel.

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