



Influence of process parameters on microstructure and corrosion properties of hopeite coating on stainless steel



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ABSTRACT

The effects of process parameters on hopeite coating formation and corrosion resistance on stainless steel surface are researched. Higher temperature and pH value favor the formation of hopeite coating. The coatings formed at different temperatures and pH values have similar phase and crystal shape. The electrochemical analysis reveals that the corrosion resistance of the coatings can be improved by temperature and pH value. Furthermore, the optimum process parameters for the formation of hopeite coating is observed at 75 °C with bath pH 2.75, at this condition the interfacial shear strength between the coating and substrate is 13.05 ± 2.2 MPa.

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1. Introduction

Stainless steels are widely used in various fields owing to their low cost, sufficient corrosion resistance, workability and excellent mechanical performances [1,2]. Notwithstanding, stainless steels undergo corrosion when in contact with the aggressive environment, although the passive oxide coating exists on their surfaces [1]. For that, many studies have been conducted to improve the corrosion resistance of the stainless steel [3–6]. When used as biomedical implants, the corrosion resistance of stainless steels is a significant factor, because the aggressive ions in human body will lead to the corrosion of implant surface and the release of the harmful ions [7–10]. It is clearly that surface modification is essential to endow the stainless steels with biocompatible and protective coatings. Nowadays, numerous surface modifications have been reported to enhance the protection of the stainless steels [2,7,11–16].

Phosphate chemical conversion (PCC) has been widely used in industry as a metal pretreatment process for the surface treatment and further painting, due to its low cost, rapid coating formation, suitability for treatment of irregular surface and highly adherent to the underlying metal [17–19]. Based on the composition of the bath, PCC treatment is classified as zinc, manganese, iron and

calcium baths [17]. Among them, zinc phosphate bath is widely used in engineering for surface modification, the main composition of which is hopeite [17–20]. It is reported that the hopeite coating efficiently facilitate re-growth of hydroxyapatite (HA) in the simulated body fluid or in alkaline medium containing saturated with Ca^{2+} [21,22]. More importantly, hopeite possesses many advantages such as good adhesion, good biocompatibility and the enhancement of bone growth [23,24]. In view of this, the hopeite conversion coating would improve protective and biocompatible performance of metal implants. The corrosion resistance of the PCC coating is affected by several parameters, e.g. coating mass, coating thickness, porosity and structure homogeneity [17,25]. For that, the uniform coating with high coating mass and low porosity will provide satisfactory corrosion resistance, which is the aim of the surface modification.

Normally, it is difficult to form a phosphate coating on stainless steels by a traditional chemical conversion method because of the passive oxide coating on their surface, even after treated at 150 °C for 30 days by hydrothermal treatment [15]. For that, several methods including electrophosphating, hot-dip zinc primary coating and hydrothermal treatments, are adopted to fabricated PCC coatings on the surface of stainless steels [12,15,26]. The electrochemical or mechanical pretreatment are also adopted for the PCC process [12,27]. As reported in the literatures, the rate of phosphating depends to a great extent on constitution of Fe^{2+} on the substrate/solution interface [17,18]. In this paper, we focus on the fabrication of uniform and fine hopeite coatings on a stainless steel 304 (304 SS) by a traditional chemical conversion method in

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the medium temperature phosphating solution with the addition of Fe²⁺ by curing process. Based on previous work [18], which main focus on the preparation of the coating, the influences of temperature and pH value on coating mass, thickness, phase, morphology and the polarization curves are explored in this paper. The aim of this study is to develop the optimum temperature and pH value for formation of hopeite coating.

2. Materials and method

2.1. Chemical conversion process

Commercially available 304 SS specimens, having the composition (wt.%) Cr: 18.57, Ni: 8.20, Mn: 1.22, Si: 0.33, P: 0.021, C: 0.054, S: 0.015 and Fe: balance, were used as substrates with the size of 10 mm × 10 mm × 1 mm. The specimens were abraded using 240-grit emery paper, followed by degreasing in 80 g/L sodium hydroxide solution at 60 °C for 15 min. Then, pickling was performed on the specimens in a solution containing 7%V/V of phosphoric acid at room temperature for 10 min. Afterward, activation was performed in a solution of 3 g/L Ti colloids (Na₄TiO(PO₄)₂, commercially obtained) at room temperature for 30 s.

The samples were then immersed in a bath with the composition of zinc oxide: 25 g/L, phosphoric acid (85%): 10 ml/L, nitric acid: 30 ml/L, sodium chlorate: 0.2 g/L, calcium nitrate: 5 g/L and citric acid: 5 g/L at 25 °C to 85 °C for duration times of 30 min. The PCC treatments were carried out at different pH values ranged from 1.5 to 3.0, which were adjusted to the desired pH by addition of phosphoric acid (7% V/V) or sodium hydroxide (7 wt.%).

Before immersion process, the bath solution was cured with 5 g/L pure iron powder (AR, 98%) at room temperature for 24 h. The aim of the curing process was to dissolve part of iron powder and increase the concentration of Fe²⁺ of PCC bath. After curing, the remained pure iron powder was removed. Finally, the samples were washed with distilled water and dried by blowing air at room temperature. The flow chart for preparing hopeite coatings is given in Fig. 1.

2.2. Physical and microstructural characterization

A digital scale with an accuracy of ±0.1 mg was used for the determination of the weights of the samples.

The coating mass (*M*, g/m²) was calculated according to Eq. (1):

$$M = \frac{(m_1 - m_2)}{A} \tag{1}$$

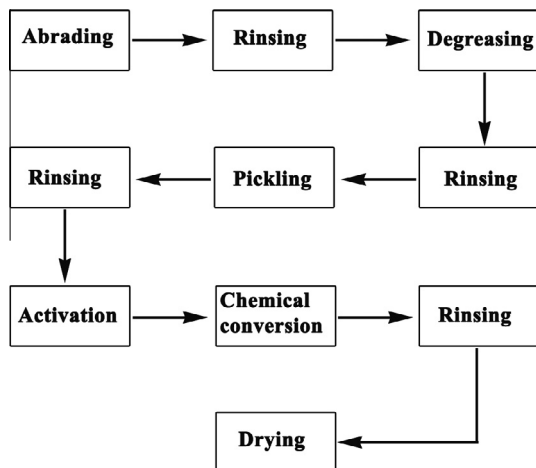


Fig. 1. The flow chart of the fabrication hopeite coating by PCC method.

where *m*₁ and *m*₂ are the weights (g) of the samples before and after the coatings were stripped, respectively, *A* is the areas of the substrates (m²). The stripping was performed in a solution containing 50 g/L of chromium trioxide at 70 °C for 10 min. An eddy current thickness gauge was used to determine the thickness of the coating. The results of coating mass and thickness were both the respective average values of five different measurements.

The sludge precipitated during the PCC treatment was used to determine the efficiency of the bath [25,28]. The bath efficiency (*E*) was calculated according to Eq. (2) [25]:

$$E = \frac{T}{W} \tag{2}$$

where *E* is the bath efficiency, *T* is the thickness of the coating, *W* is the weight of the sludge. The sludge was separated from the PCC solution using filter paper and dried at 110 °C for 20 h. Then the sludge weights were evaluated using a digital scale with an accuracy of ±0.1 mg.

The microstructures of the coatings were studied using a SU-70 field emission SEM (FE-SEM). The phase analysis of the coatings were carried out using a Rigaku D/max-γB X-ray diffractometer (XRD), with a scan speed of 4 deg/min between 5° and 80° 2θ angle and Cu Kα radiation, operated at 40 kV and 100 mA.

2.3. Electrochemical evaluation

The corrosion resistance of the samples was evaluated by electrochemical measurements, which were carried out using a classical three electrodes cell with platinum as counter electrode, saturated calomel electrode (SCE) as the reference electrode and the uncoated or coated samples with an exposed area of 1 cm² as working electrode. The corrosive medium of 0.9 wt.% sodium chloride was used. The potentiodynamic curves were obtained using a Parstat potentiostat model 2273 at constant voltage scan rate of 1 mV/s in the potential range of *E*_{op} ± 250 mV.

The polarization resistance (*R*_{*p*}), which represents the corrosion properties of specimens, was calculated using Eq. (3) [18,29,30]:

$$R_p = \frac{\beta_a \cdot |\beta_c|}{(2.303 I_{corr}(\beta_a + |\beta_c|))} \tag{3}$$

where *R*_{*p*} is the polarization resistance, β_{*a*} is anodic Tafel slope and β_{*c*} is cathode Tafel slope. The porosity percentage of PCC coating was calculated according to Eq. (4) [31]:

$$P = \frac{R_{p,s}}{R_p} \times 100\% \tag{4}$$

where *P* is the total coating porosity percentage, *R*_{*p,s*} is the polarization resistance of bare substrate, *R*_{*p*} is the polarization resistance of coated substrate. Finally, the corrosion protection efficiency was calculated using Eq. (5) [25,32]:

$$P_e \% = \left(1 - \frac{I_{corr}}{I_{corr}^0} \right) \times 100 \tag{5}$$

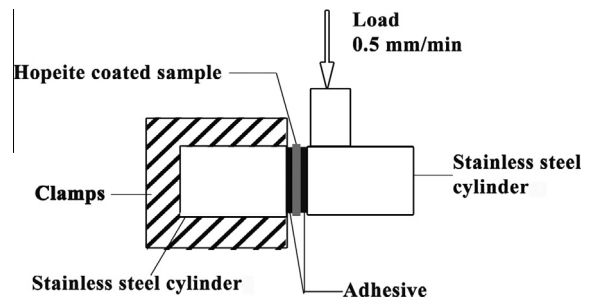


Fig. 2. The schematic diagram of the shear test.

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