Journal of Industrial and Engineering Chemistry xxx (2016) xxx–xxx

Contents lists available at ScienceDirect

Journal of Industrial and Engineering Chemistry

journal homepage: www.elsevier.com/locate/jiec

Corrosion and cathodic disbondment resistance of epoxy coating on zinc phosphate conversion coating containing Ni^{2+} and Co^{2+}

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ARTICLE INFO

Article history:
Received 8 September 2016
Received in revised form 12 November 2016
Accepted 18 November 2016
Available online xxx

Keywords:
Phosphate coating
Ni^{2+} and Co^{2+} additive
Epoxy coating
EIS
Cathodic disbondment
Adhesion

ABSTRACT

In this study, surface morphology, composition and anti-corrosion properties of zinc phosphate conversion coating with nickel and cobalt additives were investigated. In the second step, the phosphated coating was used as the pretreatment for the epoxy coating. The results showed that the corrosion resistance of phosphated coating containing additives has been increased significantly. The phosphated coating containing mixture of additives formed on the surface, has lower porosity and higher compaction than other samples. Also by adding a mixture of additives to the composition of the phosphated coating, adhesion strength and resistance to the cathodic disbondment of organic coatings were increased.
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Introduction

Carbon steel in high tonnages is used for marine applications, nuclear energy and fossil fuel of power plants, transportation, chemical processing, petrochemical and refineries production, pipelines, mining, construction and metal equipment of the process. Due to the low amounts of additive elements in the composition of this type of steel (lower than 2 wt%), carbon steels are so sensitive to corrosion in aggressive atmospheres and solutions [1]. Epoxy coatings show excellent corrosion resistance in highly corrosive environments, excellent chemical stability and good adhesion to the substrate. According to the environmental and economic benefits and excellent coating properties, powder coatings such as epoxy are used on metal substrates. Powder coating is usually applied on the surface by electrostatic method using a dry paint without solvent [2]. Physical and chemical properties of the coating and the metal substrate affect the adhesion. Adhesion of organic coatings is influenced by the corrosive electrolyte [3–10]. Adhesion Strength of the coating to the substrate is drastically reduced through the electrolyte penetration [4–6]. Water molecules can easily break the hydrogen bonds between the coating and substrate [6,8]. It is known that more adhesion is possible when the coating reacts to the metallic substrate chemically or mechanically. Nevertheless, reaction locations on the substrate that adhesion happens on them are covered by various contaminants like corrosion products, metal oxide or oil. Wettability of metal substrate is dependent to the shape of the cavity, roughness and contact angle. Nowadays, conversion coatings are used to create corrosion protection and improve adhesion resistance of organic coatings to the substrate. By using conversion coatings, preferred places will be increased to improve the adhesion. Also conversion coatings have a significant impact on the roughness and contact angle (wetting properties) of metal substrates. Using conversion coatings, contact angle is decreased and the obtained surface roughness is increased [4,5].

Phosphated conversion coatings are widely used for surface treatment and finishing of ferrous and non-ferrous metals. This type of conversion coating because of the economy, ability of quick apply, relatively good corrosion resistance, abrasion resistance, adhesion properties and lubrication, plays a vital role in automotive and industrial devices [11]. In order to improve the corrosion resistance of phosphated coatings, recent efforts have focused on the pretreatment methods before phosphating and technical processes for phosphating. Zinc phosphating is one of acceptable methods to increase the corrosion resistance of iron and steels [12–14]. Recent studies are performed to develop the temperature and time of immersion of phosphated coatings and increasing the corrosion resistance properties. Reducing the temperature of phosphating bath leads to the reduction of the coating thickness and corrosion resistance. One of the methods to modify the properties of phosphated coating at low temperature is adding

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http://dx.doi.org/10.1016/j.jiec.2016.11.027
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catalysts or additives to the phosphating coating bath. Phosphated coating contains cavities which act as routes to penetrate the corrosive electrolyte to metal substrates; therefore, corrosion resistance of conventional phosphated coatings is not enough. Nowadays additives such as Ni^{2+}, Ca^{2+}, Co^{2+}, Mo^{2+}, Cu^{2+}, Mn^{2+}, Nb^{2+} and fluoride are widely used to create low porosity and uniform phosphated coatings and improving the corrosion and adhesion resistance [15–18]. Sandro et al. have evaluated the effect of adding different types of metal cations on properties of the zinc phosphated coatings. They found that the morphology and anti-corrosion properties are considerably influenced by the additives. Additives can affect the microstructure, thickness and grain size of the conversion coatings [19–21]. Cho et al. have reported that adding Co^{2+} to the bath of chromate conversion coating can increase the anti-corrosion properties. metal ions such as nickel, cobalt and iron to improve the corrosion resistance behavior of chromated coatings [1]. Rosalbino et al. found that adding bivalent ions of cobalt and nickel to the chromating bath substantially enhances the coating properties. They showed that cobalt and nickel by means of electron transfer mechanism convert Cr(III) to Cr(VI) [22]. Akhtar et al. have evaluated the effect of Mn^{2+} and Ni^{2+} on phosphating of 2024 aluminum alloy. They found that additives decrease the size of phosphated crystals, which Mn^{2+} decreases the size of crystal more than Ni^{2+}. Besides, the coating created on the metal surface in a bath containing Mn^{2+} is thicker than the coating created in a bath containing Ni^{2+} [13,23]. Sun et al. studied the properties of phosphated coating in the presence of copper on the surface of aluminum. They realized that adding this type of additive to the phosphate coating solution improves the properties of coating [24]. Banczek et al. investigated the effect of nickel and niobium cations on corrosion resistance properties of zinc phosphate coatings on the surface of carbon steel. They represented better corrosion resistance for the phosphated layer containing Nb^{2+} compared to the phosphated layer containing Ni^{2+} [25]. On the other hand it is shown that surface treatment on steel which is done by zinc phosphate conversion coatings leads to a significant increase in adhesion properties and corrosion resistance of epoxy coatings [26]. Bajat et al. demonstrated that the phosphated coating significantly improved the adhesion strength of epoxy coating on the surface of galvanized steel [4,5]. Harun et al. found that the surface preparation with functionalized silanes on steel has considerably improved the adhesion strength of epoxy and alkyd coating [3]. Ghanbari et al. evaluated the effect of surface pretreatment applied on steel by zirconium conversion coating on adhesion performance and cathodic delamination of epoxy coating. They found that after surface pretreatment, the adhesion strength increased considerably and the cathodic delamination decreased. Totally it is proven that the rate of cathodic delamination and lack of adhesion will be significantly reduced by conversion coatings [27].

In this research, first the effect of adding Ni^{2+} and Co^{2+} to zinc phosphate coating on the surface morphology, composition, crystal size, surface energy and corrosion resistance were investigated by SEM, EDS, XRD, contact angle measurement, DC polarization and EIS respectively. Second, the effect of surface preparation by phosphated conversion coating on the corrosion resistance, adhesion and cathodic disbondment properties of epoxy coatings were studied by EIS and Pull-off test.

Materials and procedure

Materials and sample preparation

Carbon steel plates with dimensions of $7 \times 10 \times 0.5\,cm^3$ are used as the substrate. Samples were abraded with sandpaper 800 and were degreased by ultrasonic for 10 min in a solution of acetone. After that samples were acid washed in a solution of 5% volume H_2SO_4, then washed with distilled water and dried at room temperature. To prepare the phosphate solution, phosphoric acid and nitric acid were purchased from Merk Company. Zinc oxide, sodium nitrate and sodium fluoride also were purchased from Mina Ariya Tajhiz company (Iran). The sodium hydroxide (Merck CO) was used to adjust the pH.

Preparation of the solution of phosphate coating

The combination of phosphate solution is given in Table 1. Surface treatment is performed in pH = 2–2.5. Temperature and time of immersion during surface treatment were 85 °C ± 5 and 10 min, respectively. After adjusting the pH on 4, solution containing the additives was prepared with different amounts of nickel and chromium. After surface treatment samples were dried in the air.

Coating process by electrostatic spray method

To apply epoxy powder (20–50 μm, product of Nikfam Co.) on the surface of phosphated steel parts connected to ground, electrostatic spraying device IRIS model with gun of Corona model was used with voltage source of DC100 kV. After spraying the powder on the surface of parts, samples were placed in the oven for 12 min and curing treatment was performed at 200 °C.

Surface characterization

Scanning electron microscope (SEM) VEGA3 TESCAN model and energy dispersive spectroscopy (EDS) model were employed to evaluate the surface morphology of phosphated coating and composition of the coating, respectively. The fabricated phosphate coating was analyzed by X-ray diffraction (XRD), K_α radiation from cobalt lamps with energy of 40 kV, 2θ from 10° to 90° and scan rate of 10°/min. As well as AFM (model Ara 0101/A) was used for better investigation of surface morphology. The contact angle on the surface of the samples was measured using the contact angle measuring system 15 OCA plus. For this purpose, distilled water with a liquid probe has been used in the temperature and humidity of 25 ± 2 °C and 30 ± 5%, respectively. A small drop of distilled water (2–3 μL) was applied on the surface of samples. Shape of droplets was recorded by the digital camera Dino type after 10 s and the contact angle value was calculated using image analysis of image J.

Evaluation of anti-corrosion performance

Anti-corrosion performance of phosphated coating samples and organic coated samples were evaluated by potentiodynamic polarization and electrochemical impedance spectroscopy (EIS), respectively. Measurement of EIS and polarization on the coated steel samples were performed using a Potentiostat/Galvanostat (EG&G Model A273) equipped with frequency response analyzer

Table 1
The combination of phosphate solution.

Composition	1	2	3	4	5
Phosphoric acid (mL/L)	10	10	10	10	10
Nitric acid (mL/L)	3	3	3	3	3
Zinc oxide (g/L)	5.6	5.6	5.6	5.6	5.6
Sodium nitrite (g/L)	1.2	1.2	1.2	1.2	1.2
Sodium flouride (g/L)	0.3	0.3	0.3	0.3	0.3
Nickel nitrate (g/L)	–	1	–	1	0.5
Cobalt nitarte (g/L)	–	–	1	1	0.5

Please cite this article in press as: M. Golabadi, et al., Corrosion and cathodic disbondment resistance of epoxy coating on zinc phosphate conversion coating containing Ni^{2+} and Co^{2+}, J. Ind. Eng. Chem. (2016), http://dx.doi.org/10.1016/j.jiec.2016.11.027

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