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# Effect of Nano Clay on Corrosion Protection of Zinc-rich Epoxy Coatings on Steel 37

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Epoxy zinc rich coatings containing clay nanoparticles were prepared and the effect of clay content on the cathodic protection performance of the coatings was evaluated by electrochemical impedance spectroscopy (EIS) and immersion test. Open circuit potential (OCP) measurements and immersion tests were also carried out to better understand the behavior of zinc rich coating. EIS and OCP measurements showed that addition of 1 wt% clay improved the cathodic protection duration and sacrificial properties of the epoxy zinc rich coating. Transmission electron microscopy (TEM) photographs confirmed that clay nanoparticles were successfully dispersed in the coating matrix loaded with 1 wt% clay. Immersion test results indicated that addition of 1 wt% clay nanoparticles in zinc rich epoxy coatings increased the cathodic protection ability of coatings.

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## 1. Introduction

Steels are the most popular and important structural materials in the world. However, the problem of corrosion of structural steels must be considered during industrial applications. One of the most widely used corrosion protection methods for steel is the application of an organic protective coating<sup>[1,2]</sup>. The barrier, adhesion and inhibiting features have an important role in lifetime of coatings. The strength of coating/metal system is in proportion to the contact time of system with the corrosive medium, changes in adhesion, barrier and inhibiting properties. Aging and diffusion of corrosive agents such as H<sub>2</sub>O, O<sub>2</sub> and H<sup>+</sup> into coating/substrate interface can lead to blistering, reduction in stability of the adhesion bond and degradation of coating and substrate<sup>[3–6]</sup>. As a primary method for corrosion protection of metals, coatings make a barrier by preventing the diffusion of oxygen and water. However, there is no perfect barrier coating, and all coatings eventually fail due to existing defects, such as pin holes or mechanically weakened spots<sup>[7,8]</sup>.

Zinc is a typical metal pigment widely used in anticorrosion coatings and the sacrificial action of the zinc provides both cathodic protection and barrier protection<sup>[9–13]</sup>. Zinc rich paints are widely

used in a variety of severe environments because of their unique property of protecting the metal even after instances of slight mechanical damage to the coating<sup>[14]</sup>. According to the nature of the paint vehicle, zinc rich coatings are classified into two kinds i.e., organic and inorganic coatings<sup>[15]</sup>. The most common organic binders are epoxy/polyamine-amide, vinyl resins, chlorinated rubbers, unsaturated polymers, etc. On the other hand, inorganic binders are based on silicates; they can be classified into inorganic silicates if curing takes place by chemical reaction or thermal treatment of inorganic and organic silicates if self-curing takes place<sup>[16]</sup>.

In order to have sufficient cathodic protection by zinc rich paint, there should be a close contact of zinc particles among themselves and with the metallic substrate to be protected. To this end, pigment volume concentration (PVC) must be in the order of the critical pigment volume concentration (CPVC). Otherwise, the electrical contact is insufficient and consequently the metallic substrate is not suitably protected<sup>[9,16–18]</sup>.

In the past, efforts have been made to improve the protectiveness of zinc rich paints by controlling zinc content, particle size and shape of zinc metal<sup>[9,19]</sup>. Many different types of pigments including micaceous iron oxide and lamellar Zn and Al pigments<sup>[15,18,20]</sup>, nanoparticles including carbon nanotube and nanoparticulate zinc<sup>[1,21]</sup>, have been utilized in order to enhance the protective properties of the zinc rich coating. Exfoliated polyaniline clay (PANiC) nanocomposites were also added to a commercial zinc rich epoxy primer to improve its initial barrier properties<sup>[22]</sup>.

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The aim of this work is to improve the barrier properties of the zinc rich coating and also increase the cathodic protection period by addition of clay nanoparticles into coating matrix. Transmission electron microscopy (TEM) was used to observe the structure of the clay in the coating. Different techniques including electrochemical impedances spectroscopy (EIS) and immersion test were used in order to investigate corrosion protection properties of the zinc rich paints. The sacrificial protection of the coatings was studied by measuring the open circuit potential (OCP) values at different immersion time. The morphology of the zinc rich coatings was also studied by scanning electron microscopy (SEM) technique before and after immersion test.

## 2. Experimental

### 2.1. Materials and sample preparation

The diglycidyl ether of bisphenol A epoxy resin (RL 440) and polyamine hardener (HY 440) were supplied by Pars Composite Co (Tehran, Iran). The solid content and the density of the epoxy resin were 100% and  $1.10 \text{ g cm}^{-3}$ , respectively. The zinc particles with average particle diameters of 2–4  $\mu\text{m}$  (super fine) were supplied from Pars Zinc Dust Co (Tehran, Iran). The clay, which is an organically modified montmorillonite, was received from Southern Clay Products Inc. with the particle size of 0.5–5  $\mu\text{m}$  and layer thickness of 1 nm.

Four types of zinc rich coatings were prepared. The zinc content of all samples was 85% by total weight of coating. The clay contents were 0, 1%, 2% and 3% by the weight of binder and were denoted as coatings A, B, C and D, respectively. The specification of each coating is listed in Table 1.

The mixture of epoxy resin with appropriate amounts of clay was stirred at 1000 rpm for 3 h using a high shear mechanical mixer at ambient temperature. In order to get rid of bubbles, the mixtures were kept in vacuum chamber for 40 min. After that, the mixtures were sonicated for 60 min. Then, zinc powder was added to the mixtures and stirred for 45 min. A stoichiometric amount of hardener was added and the compositions were thoroughly mixed.

The St-37 steel panels with dimensions of 15 cm  $\times$  11 cm were polished with emery papers 120 to 800 in order to remove any traces of surface oxides. The steel substrates were then degreased by acetone. Afterwards, the epoxy zinc rich coatings were applied to the clean steel substrate using a film applicator. The coated samples were kept at room temperature for 7 days. The dry film thickness of  $48 \pm 3 \mu\text{m}$  was measured on the cured samples after 7 days.

### 2.2. Methods

The electrochemical impedance spectroscopy (EIS) measurements were performed at open circuit potential in 3.5 wt% NaCl solution. A sinusoidal voltage signal of 10 mV was applied over a frequency range of 100 kHz to 10 mHz. The experimental device for

EIS measurements was a traditional three electrode configuration consisting of a saturated calomel electrode (SCE) as reference electrode, platinum rod as counter electrode and coated sample as working electrode. The EIS tests were carried out on 3.14 cm<sup>2</sup> area of the samples by PARSTAT 2273, EG&G Princeton Applied Research, which were controlled by PowerSuite software.

Open circuit potential (OCP) was monitored as a function of exposure time while immersing in 3.5 wt% NaCl solution for confirmation of cathodic protection period.

Transmission electron microscopy (TEM) was performed to evaluate the intercalation/exfoliation of clay nanoparticles in epoxy zinc rich coating matrix using Philips CM30.

Immersion test was carried out according to ASTM D6943 in 5 wt% NaCl solution. The test was done on 5 cm  $\times$  5 cm area of the sample having the average coating thickness of 48  $\mu\text{m}$ . The scribed marks were centrally positioned in the test area of each panel and consisted of an "X" comprising the diagonals of 5 cm  $\times$  5 cm. Scanning electron microscope (SEM, TESCAN VEGA3 XMU) studies with energy dispersive X-ray (EDX) were carried out equipped. The scanning electron micrographs were taken of the panels coated with the zinc rich paints before and after immersion test and the surface configurations of these coatings were studied.

## 3. Results and Discussion

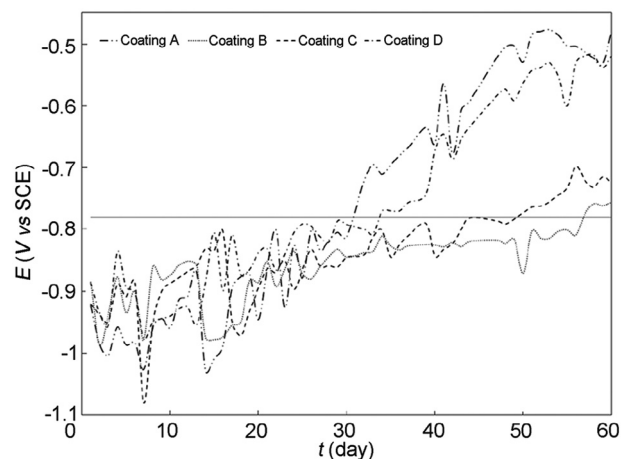
### 3.1. OCP measurements

Electrochemical properties of zinc rich paints can be described by measuring the open circuit potential (OCP) of the coating in contact with the substrate. Fig. 1 presents the evolution of OCP for all epoxy zinc rich coatings with immersion time. According to the commonly accepted criterion to provide cathodic protection, OCP value should remain more negative than  $-0.780 \text{ V vs SCE}$ <sup>[23]</sup>.

As can be seen, the OCP values of all coatings are about  $-0.9 \text{ V vs SCE}$  at the first day of the immersion. This result shows that, there is a good electrical contact between zinc particles among themselves and with steel substrate, which implies that steel substrate is under a good cathodic protection. The increase in immersion time results in the shift of OCP to more positive values<sup>[18]</sup>. The potential of coatings A, B, C and D rises above the protection level after 30, 56, 49 and 33 days, respectively and drifts to more positive values thereafter. The increase in potentials corresponded to zinc particle oxidation and the decrease of the electroactive zinc area, which means the decrease of cathodic protection intensity<sup>[24]</sup>. In other

**Table 1**  
Specification of the coatings with different clay loading

Type of coating	A	B	C	D
Weight ratio of zinc (%)	85	85	85	85
Weight ratio of binder (resin + hardener + clay) (%)	15	15	15	15
Solid content of resin + hardener	9	9	9	9
Weight ratio of clay/binder	0	0.01	0.02	0.03
Volume percent of zinc (%)	–	46.82	46.91	46.97
Volume percent of resin and hardener (%)	–	52.80	53.36	51.92
Volume percent of clay (%)	–	0.38	0.73	1.11
PVC	–	47.19	47.64	48.08
CPVC	–	62.25	62.13	61.88



**Fig. 1.** Variation of open circuit potentials vs. immersion time of the epoxy zinc rich coatings containing different amounts of clay in 3.5 wt% NaCl solution.

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