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# Preparation of double linked waterborne epoxy resin coating using Titanium curing agent and aminopropyltriethoxysilane and its anticorrosive properties

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To obtain a waterborne coating with excellent anticorrosion performance, a titanium ion-containing curing agent and polysiloxane were used separately and together to cure epoxy resin coatings. Fourier transform infrared (FTIR) spectroscopy was employed to investigate the curing process. An immersion test in NaCl solution was performed, and then, the morphology changes of different coatings were observed by field emission scanning electron microscopy (FESEM). Electrochemical impedance spectroscopy (EIS) and copper-accelerated acetic acid salt spray (CASS) testing were conducted to characterize the corrosion resistance of the coatings. The FTIR spectra showed that when both agents were used, the curing process of the resin involved three stages, which resulted in much denser crosslinks than when using a single agent. After immersion, numerous cracks were observed on coatings cured with a single agent, whereas the double-agent-cured coating remained integrated. The impedance of the double-agent-cured coating reached  $8.5 \times 10^8 \Omega$  cm<sup>2</sup> and remained as high as  $1.7 \times 10^8 \Omega$  cm<sup>2</sup>, even after 40 days of immersion.

Keywords: epoxy coating, titanium ion curing agent, polysiloxane, anticorrosion

## **1. INTRODUCTION**

Metal is the most widely used structural material used in various fields; nevertheless, energy and material loss due to severe corrosion in harsh environments has remained a perennial problem [1–4]. To protect metals from corrosion, many effective techniques have been developed, and coating is one of the most commonly used approaches [5, 6]. To reduce adverse impacts on the environment, massive research has been devoted to waterborne coatings in recent years [7, 8]. However, the

protective effect of waterborne coatings heavily depends on their microstructure, which is always inferior to that of solvent-based coatings. For instance, despite wide study and application, the performance of epoxy resin-based waterborne coatings still needs continuous improvement [9,10].

Hydrophobicity and crosslinking density are two important factors that determine the anticorrosion properties of waterborne epoxy resin coatings [11–15]. A hydrophobic coating will reduce water adsorption from air and solution and thus suppress hydrolysis that would degrade the coating within a relatively short time [16]. On the other hand, it is also necessary to increase the coating's crosslink density to obstruct the invasion of corrosive medium as much as possible [17]. The addition of a curing agent is a proven approach to improve the quality of waterborne coatings in both aspects [18-22]. Lu et al used silane-modified triethylene tetramine as a curing agent and found that the hardness, water resistance and adhesion of the cured epoxy film were improved significantly [20]. Hu et al reduced the water absorption of bisphenol-A epoxy resin using a novel biphenyl-containing amine as a curing agent [23]. Dai et al cured epoxy with both dopamine and phenethylamine and effectively enhanced the adhesion of the coating [24]. The selection and synthesis of novel curing agents has become an indispensable research topic in the field of waterborne coatings [25, 26].

Amino group-containing chemicals, such as siloxane, have been widely used as curing agents for epoxy resin since they can open epoxy rings and facilitate polymerization. In addition, siloxane tends to self-polymerize in coatings and thus can increase the crosslinking density and adhesion of the coating [27, 28]. However, in the epoxy resin coating dominated by a ring-opening polymerization mechanism, numerous -OH groups remained, which endowed the coating with good hydrophilicity and reduced the anticorrosion ability. Tetra-n-butyl titanate readily undergoes reaction with -OH groups and is an excellent crosslinking agent in the chemical industry. To eliminate the -OH groups, tetra-n-butyl titanate was used as a waterborne epoxy curing agent in a previous study; however, the agent was prone to hydrolysis and thus impaired the stability of the coating in aqueous solution [29].

In this work, we modified tetra-n-butyl titanate with ethylene glycol and obtained a stable titanium ion curing agent. Next, this titanium ion curing agent and aminopropyltriethoxysilane were used together to form a double-crosslinked waterborne epoxy resin coating. Electrochemical evaluation, immersion testing and copper-accelerated acetate acid salt spray (CASS) testing were carried out to evaluate the corrosion resistance of the coatings.

#### 2. EXPERIMENT

#### 2.1 Coating preparation

Analytically pure tetra-n-butyl titanate (Sinopharm Group) was stirred at 1500 rpm in a distillation unit with flowing argon at 70°C for 30 min and then ethylene glycol (Sigma-Aldrich) was supplied at double the amount of tetra-n-butyl titanate. The mixture was further stirred at 85°C for 3 hour to obtain stabilized titanium ion agent.

Four kind of waterborne paints were prepared based on epoxy resins (KITO Chemical). For the first kind, additive was not supplied, and it was coded as pure Epoxy for short; for the second kind, 3 wt% (vs. resin) titanium ion curing agent was added, coded as ET; for the third kind, 5 wt%

aminopropyltriethoxysilane was added, coded as ES; for the fourth kind, 3 wt% titanium ion curing agent and 5 wt% aminopropyltriethoxysilane were added simultaneously, coded as ETS. After short stirring, 300 wt% (vs. resin) distilled water was added to each kind of paint followed by a constant stirring for 10 min at room temperature.

Uniform-sized mild steel sheets polished by sandpaper up to 1200# were used as substrate. They were coated well with the aforementioned paints via brushing method and thickness of the coating was real-time detected by a detector to ensure an average value of  $65\pm5$  µm. Finally, all the coatings were dried in oven at 80°C for 5 hours.

### 2.2 Coating characterizations and corrosion tests

Fourier Transform Infrared (FTIR) measurements were performed in transmission mode to determine chemical changes in the curing process of resin. A ATR-Nicolet iS10 Spectrometer (Thermo Fisher Scientific) was used and the data was collected in the range of 400-4000 wavenumber/cm. Specimens were immersed in 3.5% NaCl solution for 40 days. After thorough resining, the surface morphology was observed using a Merlin Compact field emission scanning electron microscope (FESEM, ZEISS, Germany).

Electrochemical impedance spectroscopy (EIS) test lasting for 40 days was conducted on an electrochemical workstation (Autolab, Metrohm). 3.5 wt% NaCl solution was used as corrosive medium. A standard three-electrode configuration was employed, containing saturated calomel electrode (SCE), platinum electrode (PE) and coated specimen as working electrode. The impedance was measured when open circuit potential become stable. The frequency range is 105 Hz-0.1 Hz and the amplitude of sine signal was 10 mV (vs SCE). Copper-accelerated acetic acid salt spray (CASS) test was conducted on a salt spraying tester (Ningbo Oulinte Machinery) for 120 hours according to the ISO 3770-1976, and then the appearance of specimens were photographed.

#### **3. RESULTS AND DISCUSSION**

#### 3.1 Curing of coating

Fig. 1 shows the FTIR spectra of the pure epoxy resin, ET, ES and ETS coatings. In the spectrum of pure epoxy resin, the band at 1409 cm<sup>-1</sup> corresponds to bending vibrations of O-H, and the absorption peak at 913 cm<sup>-1</sup> indicates an epoxy ring [17, 21, 22]. In the spectrum of the ET coating, the peak at 1409 cm<sup>-1</sup> disappeared, indicating the elimination of O-H bonds due to crosslinking with Ti-O groups in the titanium ion agent. In the spectrum of the ES coating, the signal of the epoxy ring could no longer be observed due to ring-opening polymerization; moreover, peaks at 1090 cm<sup>-1</sup> and 2924 cm<sup>-1</sup> appeared that corresponded to stretching of the Si-O-Si band and C-H stretching in Si-O-CH<sub>3</sub> [30]. In the spectrum of ETS, both peaks at 1409 cm<sup>-1</sup> and 913 cm<sup>-1</sup> disappeared, which meant that the abovementioned crosslinking reactions occurred independently without mutual interference.



Figure 1. FTIR spectra of pure epoxy resin, ET, ES and ETS coatings



Notes: -•-•- - stands for epoxy resin, stands for titanium ion curing agent, stands for aminopropyltriethoxysilane, represents the self-crosslinking reaction of siloxane, while the epoxy group and hydroxyl represented by and respectively.

Figure 2. Schematics of pure coating(a), ET(b), ES(c) and ETS(d) coatings

Schematics of the curing process of epoxy coatings based on the FTIR analysis results are illustrated in Fig. 3. Large amounts of epoxy monolayers are deposited on the mild steel surface when the epoxy coating does not include a curing agent (Fig. 3 a). However, curing agents can connect each epoxy molecule into a network structure (Fig. 3 b, c). The two curing agents worked in different ways, and simultaneously, they cured the epoxy resin to form a more compact spatial network structure when they were added to the epoxy together (Fig. 3 d).

The FTIR results confirmed double crosslinking processes in the ETS coating, which definitely

increased the crosslinking density. Generally, the corrosion resistance of the coating would be improved since the short polymeric chains could covalently interconnect with crosslink nodes and thus form a chemically inert barrier that prevents aggressive penetration of species that initiate corrosive processes [32, 33].

## 3.2. Surface topography of coatings after immersion

As seen from the FESEM image, the substrate was carefully polished, and scratches and corrosion pits were not observed. After drying, all the paints applied on the steel plate formed uniform coatings with homogeneous topography. Nevertheless, epoxy coatings without curing agents showed corrugation all over the surface due to fast water evaporation and slow polymerization [34], while coatings with curing agents had much smoother surfaces.

After 40 days of immersion, the surface of the bare steel plate was fully covered by granular rust. Plenty of rust was also found on the coating without a curing agent, and fatal large cracks formed in the coating, indicating that the coating had been severely destroyed. The ET coating and ES coating demonstrated better anticorrosion properties because the destruction of coatings was greatly suppressed; nevertheless, shallow cracks and rust could be found. In sharp contrast, the ETS coating appeared to be least affected, as it remained compact and complete without significant accumulation of corrosion products. These observations suggested that the use of a curing agent could significantly enhance the stability of the waterborne epoxy coating, especially when two agents were added at the same time.



**Figure 3.** FESEM images of mild steel, pure epoxy, ET, ES and ETS coating before (a, b, c, d, e) and after (f, g, h, i) 40 days of immersion in 3.5% NaCl solution

## 3.3 EIS tests

After immersion in NaCl solution for 10 days, semicircles were noted in the Nyquist plots of all the coatings, which meant that at this time point, all the coatings showed capacitive features, indicating

structural integrity and good barring performance, as shown in Fig. 4. The data were fitted well by the equivalent circuit in Fig. 6 (a), which was also used by other research groups [35-39]. In this circuit, R<sub>s</sub> refers to the solution resistance,  $CPE_c$  is a constant phase element representing the coating capacitance, and  $R_c$  represents the resistance of the coating. The parameters obtained from the fitting are shown in Table 1. It is generally believed that organic coatings can provide protection for metal substrates when their impedance is greater than  $10^6 \ \Omega \ \text{cm}^2$  [40]. The addition of a curing agent greatly increased the impedance of the waterborne epoxy coating by approximately two orders of magnitude. In addition, it is remarkable that the coating simultaneously crosslinked with two kinds of agents had the highest impedance among all coatings.



Figure 4. Nyquist plots of pure epoxy (a) and ET, ES, ETS (b) coatings after 10 days of mmersion

**Table 1.** The parameters of the equivalent circuit from Fig. 6 (a) after fitting the impedance spectra of 10 days.

Coating system	$R_{\rm c} (\Omega {\rm cm}^2)$	$CPE_{c}(F)$
Pure epoxy	$4.4 \times 10^{6}$	$2.08 \times 10^{-9}$
ET	$2.5 \times 10^{8}$	$1.3 \times 10^{-9}$
ES	$3.2 \times 10^{8}$	$1.2 \times 10^{-9}$
ETS	$8.5 \times 10^{8}$	$1.2 \times 10^{-9}$

After 40 days of immersion, the Nyquist plot of the coating without curing agent showed more than one time constant, as illustrated in Fig. 5 (a). Taking into account the FESEM observations, it was believed that the NaCl solution diffused through the coating and that corrosion started underneath the layer. For the ET, ES and ETS coatings, the Nyquist plots remained as perfect semicircles, indicating an intact structure and good waterproof ability as suggested earlier. Accordingly, another equivalent circuit was proposed for the pure epoxy coating, as shown in Fig. 6 (b), in which  $CPE_{dl}$  is the constant phase element of the double-layer capacitance and  $R_r$  represents the resistance of rust [44]. However, the ET, ES and ETS coating data were continuously fitted with the circuit in Fig. 6 (a). As seen from Table 3, the  $R_c$  of pure epoxy decreased obviously, while the newly formed rust could hardly provide any protection. On the other hand, although the  $R_c$  values of the ET, ES and ETS coatings also



decreased, their protection ability was not severely impaired with prolonged time.

Figure 4. Nyquist plots of pure epoxy (a) and ET, ES, ETS (b) coatings after 40 days of mmersion

**Table 1.** The parameters of the equivalent circuit from Fig. 6 (a) after fitting the impedance spectra of 40 days.

Coating system	$R_{\rm c} (\Omega {\rm cm}^2)$	$CPE_{c}(F)$	$R_{\rm r}  (\Omega  {\rm cm}^2)$	$CPE_{dl}(F)$
Neat epoxy	$2.0 \times 10^{5}$	1.3×10 <sup>-9</sup>	40	$1.4 \times 10^{-6}$
ET	$5.5 \times 10^{7}$	2.7×10 <sup>-9</sup>	-	-
ES	$8.3 \times 10^{7}$	$1.5 \times 10^{-9}$	-	-
ETS	$1.7 \times 10^{8}$	1.3×10 <sup>-9</sup>	-	-



Figure 6. Equivalent circuits used for fitting the EIS plots

## 3.4 Optical images of the samples

Fig. 7 shows photographs of the pure epoxy, ET, ES, and ETS coatings before and after immersion in 3.5 wt.% NaCl solution for 40 days. All the paints formed uniform coatings on the steel plate after brushing and drying. It is obvious that the addition of curing agents improved the appearance of the coating, making it smoother and glossier. Incompletely cured epoxy coatings tended

to contain internal structural defects, and their adhesion and self-leveling were also poor; therefore, a matte surface was observed.

After 40 days of immersion, part of the pure epoxy coating was destroyed and replaced by a red rust layer under attack of water and Cl- [45]. The coating thoroughly lost its barrier ability in most areas. For the ET and ES coatings, although they remained integrated, white cloudy zones could be observed, indicating obvious water absorption. For the ETS coating, a significant change in appearance was not observed. The results clearly showed that the use of a curing agent, particularly the use of double agents, endows the coating with more improved anticorrosion performance due to a higher curing density [46].



Figure 7. Optical images of pure epoxy (a, e), ET (b, f), ES (c, g) and ETS (d, h) coatings before and after 40 days of immersion in 3.5wt.% NaCl solution

# 3.5 CAAS test

Salt spray is an effective and visualized testing method to verify the anticorrosion ability of organic coatings [39, 47]. The appearance of coatings before and after the 120-hour CASS test is shown in Fig. 9. All the coatings were uniform and glossy before the CASS test; however, after 120 hours of exposure, the differences in appearance were quite obvious. The different appearances observed by the naked eye lead to the following conclusions: the anticorrosion ability of coatings could be ranked in the order of ETS>  $ET \approx ES$ > pure epoxy.

The pure epoxy coating lost its protective function almost entirely, and peelings far from the scribed lines were observed; for the ET and ES coatings, corrosion happened at the scribed lines and the spread was limited; meanwhile, corrosion was not found even along the scribed lines in the ETS coating. Based on such results, a 6-grade rating method was adopted to discriminate the protection performance of the coatings [29,48], and the results are shown in Table 3. The CASS results show that the anticorrosion performance of the coating was greatly improved with the addition of the curing agents, which was consistent with the results from the immersion and EIS tests.



**Figure 8.** Surface topographies of neat epoxy (a), ET (b), ES (c) and ETS (d) coatings before and after 120 hours of CAAS (e), (f), (g) and (h).

Table 3. Individual levels of the CASS results for each coating after 120 hours of exposure

Coatings	grades
Neat epoxy	2
ET	5
ES	5
ETS	6

## 4. CONCLUSIONS

A double crosslinking mechanism was proposed to solidify epoxy resin by simultaneously using a titanium ion curing agent and siloxane. The use of a curing agent improved the workability of the paints, making the coating surface smoother and glossier. After adding the curing agent, especially when two kinds of agents were added at the same time, the resistance of the coating increased significantly to more than 108  $\Omega$  cm2, and the stability in the corrosive solution was also improved. The 40-day immersion test results and CASS test results also showed that the use of a double crosslinking agent could significantly improve the protection ability and stability of the coating.

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