Electrochemical Behavior of Solvent-Free Epoxy Anticorrosive Coatings Under Simulated Seawater Scouring Conditions (Postprint)

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Abstract

The failure behavior of two epoxy powder coatings and one solvent-free epoxy liquid coating in a 3.5 wt% NaCl solution containing 1 wt% quartz sand at 60°C with a flow velocity of 2 m/s was investigated by Electrochemical Impedance Spectroscopy (EIS). The surface morphology of the eroded coatings was observed using Scanning Electron Microscopy (SEM), and the cross-sectional morphology of the coating/metal interface was examined using Confocal Laser Scanning Microscopy (CLSM). The results indicated that, compared with static immersion in the same temperature medium, flowing sand-containing conditions significantly accelerated the failure of the solvent-free coatings. Under erosion conditions, the #1 and #3 solvent-free coatings, due to their relatively dense structure, exhibited a failure process primarily divided into three stages: the transport stage of corrosive medium within the coating, the substrate metal corrosion initiation stage, and the substrate metal corrosion development and coating failure stage. The #2 solvent-free coating, due to numerous internal pores, rapidly formed through-pores, and no intermediate failure stage was observed. The presence of pores within the coating system is an important factor accelerating coating failure.

Full Text

Electrochemical Behavior of Solvent-Free Epoxy Anticorrosive Coatings Under Simulated Seawater Erosion Conditions

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Abstract

The failure behavior of three types of solvent-free epoxy anticorrosive coatings was investigated in simulated flowing seawater at 60°C with a flow velocity of 2 m/s and 1 wt% quartz sand content using electrochemical impedance spectroscopy (EIS). The surface morphology of eroded coatings was examined by scanning electron microscopy (SEM), and the coating/metal cross-sectional morphology was observed using confocal laser scanning microscopy (CLSM). The results demonstrate that, compared with static immersion at the same temperature, flowing sand-containing conditions significantly accelerate the failure of solvent-free coatings. Under erosion conditions, the amine-cured epoxy powder coating and phenolic amine-cured epoxy liquid coating, which exhibit slow curing crosslinking rates and relatively dense structures, undergo a three-stage failure process: (1) transmission of corrosive medium through the coating, (2) initiation of substrate metal corrosion, and (3) development of substrate corrosion accompanied by coating failure. In contrast, the phenolic-cured epoxy powder coating, characterized by rapid curing and numerous internal pores, quickly develops through-thickness channels, resulting in a shortened and less distinct intermediate stage, with the failure process primarily occurring in two stages. These findings indicate that the presence of pores within the coating system is a critical factor accelerating coating failure.

Keywords: solvent-free epoxy anticorrosive coating; erosion; seawater; electrochemical behavior; pores

1. Introduction

Solvent-free epoxy powder coatings and solvent-free epoxy liquid coatings, collectively known as solvent-free epoxy anticorrosive coatings, are widely used for corrosion protection of steel structures and concrete in marine environments due to their excellent barrier properties against corrosive media, good adhesion, and environmental friendliness [1]. Current research on solvent-free epoxy coatings has primarily focused on failure mechanisms under static conditions [2,3]. However, most structural components in marine environments operate under combined effects of flow, sand content, and temperature, such as in splash zones, tidal zones, seawater zones, and seabed erosion zones.

Wei et al. [4] used a custom-built rotating cylinder device to compare the failure behavior of epoxy powder coatings under static immersion and low-velocity flow (0.3 m/s) in 3 wt% NaCl solution without abrasive particles. Le Thu et al. [5] employed EIS to compare the failure behavior of three thin-film solvent-containing organic coating systems under laminar flow conditions. Wang and Bierwagen [6] utilized a simulated pipeline flow apparatus to investigate the effect of flow velocity on organic coating failure under laminar flow, concluding that while increased flow velocity significantly reduces coating barrier properties, the failure mechanism remains unchanged, suggesting that flow conditions can serve as an accelerated testing method for coating degradation. Luo et al. [7-9] investigated the effects of flow velocity, sand content, and particle size on the erosion characteristics of epoxy powder coatings using a custom rotating disk erosion-corrosion tester, establishing relationships between erosion rates and these parameters, but without examining the electrochemical failure mechanisms under erosion conditions. These studies primarily focused on solvent-containing organic coatings under low flow velocity, sand-free, or pipeline flow conditions, with limited research on the electrochemical failure behavior and mechanisms of solvent-free organic coatings under higher flow velocities (2 m/s) and sand-containing erosion conditions.

This study investigates two epoxy powder coatings and one solvent-free epoxy liquid coating, examining the failure behavior of these three thick-film organic coating systems in simulated seawater (3.5 wt% NaCl solution) at a relatively high flow velocity (2 m/s) containing 1 wt% quartz sand (median particle size D50 = 140.7 m) at 60°C using EIS. The failure mechanisms in this environment are discussed, with CLSM employed to observe coating/metal cross-sections and analyze the influence of film formation methods on internal coating structure. SEM observations of post-erosion coating surfaces are used to analyze coating wear mechanisms. This work provides theoretical foundations and experimental basis for developing high-performance epoxy coatings for harsh marine environments.

2. Experimental Methods

Structural steel 16Mn (Q345E) rods were machined into 15 mm × 30 mm × 2 mm specimens. After degreasing with ethanol and acetone, the specimens were sandblasted to Sa 2.5 grade, ultrasonically cleaned, and dried. Three commercial solvent-free epoxy anticorrosive coatings were used: epoxy powder coating A, epoxy powder coating B, and solvent-free epoxy liquid coating C (with solid content >97%). All three coating systems used solid or liquid bisphenol A-type epoxy resin as the binder, with curing systems comprising solid amine (high-temperature slow cure), modified phenolic resin (high-temperature fast cure), and modified liquid phenolic amine (room-temperature slow cure), respectively. Pigments and fillers in all systems consisted of wollastonite, titanium dioxide, and carbon black.

Epoxy powder coatings A and B were applied by electrostatic spray fusion bonding. Specimens were preheated in a furnace at (240±2)°C for 10 min, electrostatically sprayed with epoxy powder, then cured in a furnace at (230±2)°C for 30 min (coating A) or 10 min (coating B), followed by air cooling. Solvent-free epoxy liquid coating C was applied by manual scraping at room temperature under low humidity conditions. After drying, specimens were cured in an oven at (80±2)°C for 24 h. The glass transition temperature (Tg) of the coatings was measured using a Q200 differential scanning calorimeter (DSC) according to SY/T 0315. The Tg values for coatings A, B, and C were 113.1°C, 98.1°C, and 80.6°C, respectively. Coating thickness and holiday detection were performed, and specimens with coating thickness of (280±20) m were selected for testing. Copper wires were soldered to the back of specimens, which were then encapsulated in epoxy resin, yielding a working/test area of approximately 4 cm^2.

Erosion testing was conducted using a custom-built temperature-controlled adjustable-speed rotating erosion apparatus [Figure 1: see original paper] in 3.5% NaCl solution (simulated seawater) at (60±2)°C, 2 m/s flow velocity, and 1% quartz sand content. Static immersion tests were performed in 3.5% NaCl solution at (60±2)°C. Three parallel specimens were used for each test condition. EIS measurements were performed offline using a PAR 273A potentiostat and 5210 lock-in amplifier, with a frequency range of 10 -10 ^{2} Hz. To improve signal-to-noise ratio, the perturbation signal was 50 mV during initial immersion and 20 mV after the coating system stabilized. A three-electrode cell was used with a saturated calomel electrode (SCE) as reference, Pt electrode as counter, and the coated metal specimen as working electrode. PowerSuite software was used for measurement, and ZSimpWin software was employed for data fitting and analysis.

Cross-sectional morphology was examined using an OLYMPUS LEXT OLS4000 confocal laser scanning microscope (CLSM), with six specimens observed for each coating type. Post-erosion surface morphology was observed using an FEI Nano SEM Nova 430 field emission scanning electron microscope (ESEM) after carbon coating.

3. Results and Discussion

3.1 EIS Spectra of Coating Systems

Figures 2 [Figure 2: see original paper] and 3 show Bode plots for epoxy powder coating A immersed in 3.5% NaCl solution at 60°C under erosion and static conditions, respectively. As shown in Figure 2a, the initial low-frequency impedance modulus (|Z| . Hz) of coating A exceeded $1\times10^{11}\ \Omega\cdot cm^2$, indicating excellent protective insulation properties during the initial water saturation stage. Under flowing sand-containing conditions, |Z| . Hz decreased to $1\times10\ \ \Omega\cdot cm^2$ after 12.5 days, then increased above $1\times10\ \ \Omega\cdot cm^2$ after 15 days. This increase in |Z| . Hz is attributed to corrosion reactions at the interface, where initial corrosion products create a reverse osmotic pressure that partially blocks active or inactive transport channels for corrosive species in the thick-film coating, thereby retarding ion penetration. After 22 days, |Z| . Hz dropped below 1×10 $\Omega\cdot cm^2$. Although no blistering was observed on the coating surface at this time, the coating had essentially lost its protective function [10]. In contrast, under static immersion in the same corrosive medium (Figure 3a [Figure 3: see original paper]), |Z| . Hz decreased much more slowly, remaining above 1×10 $\Omega\cdot cm^2$ even after 109 days, demonstrating that the coating maintained good barrier and protective properties after water saturation.

Figures 4 [Figure 4: see original paper] and 5 present Bode plots for epoxy powder coating B under erosion and static immersion conditions, respectively. The initial |Z| . Hz exceeded 1×10^{11} $\Omega\cdot\mathrm{cm}^2$, indicating good coating performance. Under flowing sand-containing conditions, |Z| . Hz dropped below 1×10 $\Omega\cdot\mathrm{cm}^2$ after 15 days, with microscopic blistering observed on the coating surface, indicating basic failure. The decrease then slowed, reaching 2.2×10 $\Omega\cdot\mathrm{cm}^2$ after 22 days and 4.1×10 $\Omega\cdot\mathrm{cm}^2$ after 26.5 days. The anomalous increase in |Z| . Hz at this stage occurs for the same reason as in coating A. Under static immersion (Figure 5a [Figure 5: see original paper]), |Z| . Hz remained essentially unchanged, staying near 1×10^1 $\Omega\cdot\mathrm{cm}^2$ after 109 days with phase angles approaching 90°, indicating excellent barrier properties.

Figures 6 and 7 show Bode plots for solvent-free epoxy liquid coating C under erosion and static immersion conditions, respectively. The initial |Z| . Hz exceeded 1×10^{11} $\Omega\cdot\mathrm{cm}^2$. Under flowing sand-containing conditions, |Z| . Hz reached 1×10 $\Omega\cdot\mathrm{cm}^2$ after 15 days, increased above 1×10 $\Omega\cdot\mathrm{cm}^2$ after 18.5 days (showing the same anomalous behavior), then dropped below 1×10 $\Omega\cdot\mathrm{cm}^2$ after 22 days when blistering was observed. Under static immersion (Figure 7), |Z| . Hz remained essentially unchanged, staying near 1×10^1 $\Omega\cdot\mathrm{cm}^2$ after 109 days with phase angles approaching 90°, indicating good protective properties.

Figures 8 [Figure 8: see original paper]–10 show Nyquist plots for the three solvent-free coatings under erosion conditions at 60°C in 3.5% NaCl solution at various times. Figure 11 [Figure 11: see original paper] presents the equivalent circuits used for EIS fitting. In the early erosion stage (Figures 8a, 9a, and 10a), all three coatings exhibited single capacitive arcs with decreasing radius over immersion time, corresponding to equivalent circuit model A in Figure 11a, where Rs is solution resistance, and Rc and Qc are coating resistance and coating capacitance, respectively. During this stage, corrosive species continuously diffused into the coating, causing Rc to decrease and Qc to increase, representing the initial failure stage. Within the 0–109 day timeframe, Nyquist plots for all three coatings under static immersion at 60°C could be fitted using model A, showing single capacitive arcs with gradually decreasing radius but only one time constant, indicating the coatings remained in the initial failure stage.

With increasing erosion time, Nyquist plots for epoxy powder coating A and solvent-free epoxy liquid coating C in the time ranges of 9 days and 12.5-18.5 days, respectively, could be fitted using model B (Figure 11b), where Qdl and Rct represent double-layer capacitance and charge transfer resistance at the interface. The Nyquist plots transitioned from single to double capacitive arcs. Although coating C (Figure 10b) appeared to show a single capacitive arc characteristic, magnification of the high-frequency region and EIS fitting results revealed two time constants, likely because the interfacial electrochemical reaction was too weak to be clearly distinguished in the impedance spectrum [11-13]. Subsequently, epoxy powder coating A exhibited real-axis contraction in the low-frequency region at 12.5 days (Figure 8c), showing inductive characteristics that were fitted using model C (Figure 11c), where L and RL represent inductance and inductive resistance. This behavior primarily results from non-uniform transport of corrosive species in the coating, causing preferential reactions at surface defects where corrosion active sites induce inductive characteristics. During this period, corrosive species diffused to the coating/metal interface and electrochemical reactions occurred, representing the intermediate failure stage. Epoxy powder coating B did not exhibit a distinct intermediate stage with double capacitive arcs. With further erosion time, Nyquist plots for coating B at 9 days (Figure 9b [Figure 9: see original paper]) showed diffusion arc characteristics in the low-frequency region, fitted using model D (Figure 11d), without undergoing the double capacitive arc process. This is likely because coating B contained numerous pores (or low crosslink density regions) that quickly interconnected under repeated sand and solution impact, forming conductive channels that led to substrate corrosion. The accumulation of corrosion products caused diffusion arc characteristics, necessitating the introduction of Warburg impedance (Zw) in model D. Zhang et al. [12,13] proposed that coatings impede diffusion of corrosion products from the metal/coating interface into the solution, making product diffusion the rate-controlling step for the Faradaic process. With increasing erosion time, impedance spectra for coating A (Figure 9c) and coating C (Figure 10c) also exhibited this behavior, fitted using model E (Figure 11e), where Qdiff and Rdiff represent diffusion capacitance and diffusion resistance. This diffusion behavior is typically not ideal Warburg impedance, with dispersion exponent (n) deviating from 0.5, representing the final failure stage.

Tables 1 -3 present fitted EIS parameters for the three coating systems at various time intervals. For epoxy powder coating A and solvent-free epoxy liquid coating C, Rc clearly shows three stages: slow decrease in the early stage, rapid decrease in the intermediate stage, and stabilization in the final stage. In contrast, coating B' s Rc primarily exhibits two stages: 4.2×10^1 $\Omega\cdot cm^2$ at 4 days, dropping sharply to 8.9×10 $\Omega\cdot cm^2$ at 9 days, rapidly transitioning from early to final stage due to quick penetration of transport channels and localized rapid failure, shortening and obscuring the intermediate stage. CLSM and ESEM observations confirm that these different failure stage progressions result from varying internal porosity. Qc gradually increased in the early stage, approaching saturation and stabilization in the intermediate stage, then changing significantly in the final stage. Rct characterizes the difficulty of corrosion processes at the coating/substrate interface. For coatings A and C, Rct decreased with immersion time, indicating increasing interfacial corrosion rates. For coating B, Rct remained relatively unchanged, suggesting that localized corrosion occurred at interconnected pores, with corrosion products blocking small pores and retarding interfacial corrosion.

3.2 Variation of Coating Capacitance and Low-Frequency Impedance

The coating resistance (Rc) and constant phase element (Qc) obtained from equivalent circuit fitting often show deviations and require substantial effort, making rapid coating performance evaluation difficult [10,14,15]. Since ion penetration is sensitive to coating resistance and coating capacitance typically reflects water absorption, low-frequency impedance values are commonly used for rapid assessment of ion barrier properties, while high-frequency capacitance evaluates water uptake. Mobin et al. [10] proposed that the low-frequency impedance (|Z|f→0) represents the resistive character of protective coatings and effectively characterizes ion shielding performance: |Z|f→0 > 1×10 Ω · cm^2 indicates excellent coating performance; 1×10 Ω · cm^2 < |Z|f→0 < 1×10 Ω · cm^2 indicates degraded but still protective performance; and |Z|f→0 < 1×10 Ω · cm^2 indicates loss of protective function against ion attack. Walter et al. [15] suggested calculating coating capacitance using the imaginary component of impedance (Z”) at high frequency (10 Hz):

$$C_c = \frac{-1}{2\pi f Z"}$$

where f = 10 Hz.

Figures 12 [Figure 12: see original paper]-14 show the variation of Qc and |Z| . Hz with immersion time for epoxy powder coatings A and B and solvent-free epoxy liquid coating C under different conditions. Under static immersion (Figures 12a-14a), all three coatings exhibited two-stage Qc behavior within the experimental timeframe: the first stage involved water absorption to saturation, occurring over a relatively long time, during which lg Qc showed a linear relationship with $t^1/^2$, indicating Fickian diffusion of H O in the coatings [16]; the second stage represented saturated dynamic equilibrium, which persisted for an extended period and was still ongoing at the test conclusion.

Under flowing erosion conditions, all three coatings showed Fickian diffusion behavior in the initial stage. Coatings A and C exhibited three-stage capacitance behavior: rapid water absorption in the first stage, significantly faster than under static immersion, demonstrating that erosion accelerated H O transport; saturation in the second stage, occurring much more quickly than in static immersion; followed by a third stage where Qc increased rapidly, indicating structural changes until failure. Coating B showed primarily two-stage behavior: rapid initial absorption followed by rapid increase in the third stage, with the saturated second stage being relatively indistinct.

As shown in Figures 12b-14b, under static immersion, |Z| . Hz for all three coatings decreased slowly over time, remaining above 1×10 Ω · cm^2 throughout the experimental period, indicating slow ion ingress and good protective performance. Under flowing erosion conditions, coatings A and C showed pronounced three-stage |Z| . Hz behavior: slow initial decrease, rapid decrease in the intermediate stage, and slowing decline in the final stage. Coating B exhibited primarily two-stage behavior, rapidly transitioning from early to final stage with an indistinct intermediate stage. This pattern matches the equivalent circuit fitting results, demonstrating that this method can be used for rapid performance evaluation. The decreasing rate of |Z| . Hz under erosion was significantly greater than under static immersion, confirming that erosion conditions accelerate failure of solvent-free epoxy coatings compared to static immersion.

3.3 Cross-sectional Morphology and Post-Erosion Surface Observations

CLSM cross-sectional observations of the three solvent-free epoxy coatings (Figure 15 [Figure 15: see original paper]) revealed pores of approximately 20–30 m diameter in all systems. In epoxy powder coatings A and B, pores were concentrated primarily at the coating/metal interface, while in solvent-free liquid coating C, pores were located mainly in the coating bulk near the coating/air surface. Coating B contained the highest pore density, followed by coating A, with coating C having the fewest pores. These differences in pore quantity and location result from different film formation and curing methods. Since coatings A and B employed high-temperature curing, residual air and low-melting-point substances in the coating interior did not have sufficient time to migrate to the surface for bubble elimination during the high-temperature electrostatic spraying process. The metal surface reached the highest temperature with the shortest gelation time, resulting in higher pore concentrations at the coating/metal interface. In contrast, coating C cured slowly at room temperature, allowing initial air and small additive molecules sufficient time to migrate to or accumulate near the surface. During later curing stages, as the surface coating approached dryness, residual gas or low-boiling-point substances could only accumulate near the surface, resulting in fewer pores located near the coating surface region.

Although coatings A and B are both epoxy powders cured at high temperature, coating A cured more slowly than coating B, providing better flow and long-range polymer mobility that allowed more time for residual gas or small molecules to migrate out during spraying. Consequently, coating A had lower porosity than coating B. These pores first reduce the effective coating thickness and second create near-bare metal areas at the coating/interface. When corrosive species diffuse to the interface, these exposed regions corrode preferentially. Although coating C showed no pores at the coating/metal interface, pores in the coating bulk still reduced effective thickness. Under erosion conditions, continuous medium impact causes physical cutting and wear, enlarging micropores and connecting new small pores, allowing adjacent pores or low crosslink density regions to interconnect and form conductive pathways. This enables corrosive species to rapidly reach the coating/metal interface, accelerating interfacial corrosion compared to static immersion.

SEM surface morphologies of the eroded coatings after 26.5 days (Figure 16 [Figure 16: see original paper]) show that coatings A and C exhibited similar erosion mechanisms, both characterized by selective erosion [8], where the resin matrix provides erosion resistance. Under sand particle impact, pigments and fillers were removed or fractured. This physical wear effectively reduced coating thickness, allowing corrosive species to reach the coating/metal interface more quickly and accelerating coating failure. The eroded surface of coating B showed holes that significantly shortened the diffusion path for corrosive species to reach the coating/metal interface, reducing effective thickness and creating conductive channels that led to rapid coating failure. This observation is consistent with the cross-sectional pore analysis.

4. Conclusions

(1) Compared with static immersion, erosion conditions accelerate the failure process of solvent-free epoxy coatings and can be used for rapid evaluation of coating anticorrosion performance under flowing and sand-containing conditions.

(2) Under flowing erosion conditions, epoxy powder coating A and solvent-free epoxy liquid coating C, which have relatively dense structures, exhibit a three-stage failure process: (i) initial stage—transport of corrosive media (water, ions, etc.) through the coating, reducing barrier properties; (ii) intermediate stage—corrosive species reach the metal/coating interface and electrochemical corrosion initiates; (iii) final stage—electrochemical reactions progress, corrosion products accumulate, leading to coating failure. In contrast, epoxy powder coating B, due to its high internal pore density that rapidly forms interconnected channels, shows a shortened and indistinct intermediate stage, with failure primarily occurring in two stages.

(3) Internal pores generated during coating film formation are a critical factor accelerating coating failure under flowing erosion conditions. Optimizing film formation methods and curing processes to obtain uniformly dense coating structures will be essential for future development of durable coatings.

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