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The role of surface morphology in the barrier properties of epoxy coatings in different corrosion environments

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ABSTRACT

Hydrophobic epoxy coatings with particulated surfaces were prepared via soft lithography using different abrasive papers as the templates. With the increasing surface roughness, the hydrophobicity of these surfaces first increased and then decreased and a maximum water contact angle of $\sim 136^\circ$ was obtained by using a P2000 abrasive paper as the template. Electrochemical impedance spectroscopy (EIS) was used to study the role of surface morphology in the coatings' barrier properties under wet–dry cyclic immersion and in salt-spray environments. The results demonstrated that the hydrophobicity induced by the trapped air in the surface microstructures produced stronger barrier properties than that produced by a smooth coating when exposed to wet–dry cyclic immersion. The strongest barrier performance was achieved by the coating templated with the P2000 abrasive paper. However, these microstructured hydrophobic coatings deteriorated faster in the salt-spray tests because of the direct deposition of salt electrolyte particles on the coating surfaces and the increased coating surface area. Under the salt spray condition, the coating obtained from P6000 abrasive paper exhibited the fastest deterioration.

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1. Introduction

Organic coatings are widely used for corrosion protection due to their barrier effects against water, oxygen, and ions species. Nevertheless, during the long-term exposure to corrosion environments, the continuous penetration of water into the coating inevitably decreases its barrier properties and eventually leads to the loss of its resistance to corrosion [1–3].

One common method of enhancing the barrier properties of the coating is by increasing its surface hydrophobicity which can effectively reduce the adhesion between water and the coating surface and delay the water penetration [4,5]. This is often completed by incorporating low-surface-energy coating materials, for instance, fluorine- or silicon-containing polymers [6–8]. For example, by using silane-functional benzoxazine as the curing agent, Zhou et al. prepared a hydrophobic epoxy coating with a water contact angle of over 100° [9]. The charge transfer resistance of the obtained coating

was three times higher than the coatings without silane-functional benzoxazine, indicating an enhanced barrier properties.

While the water contact angles of a smooth surface rarely exceed 120° , the surface hydrophobicity can be further enhanced by introducing rough surface microstructures. This can be achieved by a variety of different methods including self-assembly [10,11], electrodeposition [12], chemical etching [13,14] and anodization [15]. The obtained surfaces often achieve a hydrophobic or even super-hydrophobic state and can exhibit a greatly improved corrosion resistance [16–18]. To date, this field has focused on the direct synthesis of microstructures on metals, and much less work has been reported on corrosion protection using organic coatings with microstructured surfaces [19,20]. To construct these surfaces, a composite coating can be developed whose surface roughness is induced by the incorporation of aggregated micro/nanoscaled fillers [21–23]. However, these fillers simultaneously change the surface and the bulk properties of the coatings. Thus, the effect of surface microstructures on the coating performance cannot be identified.

Using a soft lithography method, Yang et al. casted and cured polydimethylsiloxane (PDMS) onto *Xanthosoma sagittifolium* leaves and transferred their papillary surface microstructures to an epoxy/graphene coating applied on cold-rolled steel. Compared

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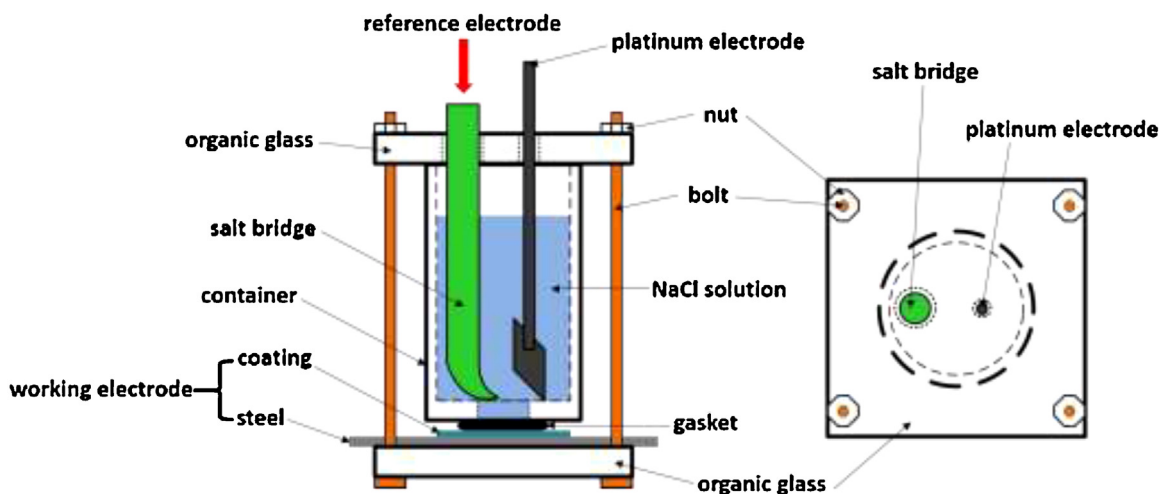


Fig. 1. The set-up configuration of the electrochemical cell.

to the flat coating constructed of the same material, the obtained microstructured coating exhibited a water contact angle of $\sim 120^\circ$ and a superior barrier performance in a 3.5 wt% NaCl solution due to the air trapped within the microstructures [24]. Similarly, the surface microstructures of lotus leaves were successfully replicated to prepare a super-hydrophobic epoxy coating with a water contact angle of over $\sim 150^\circ$ [25]. Electrochemical studies showed that the surface super-hydrophobicity could remarkably enhance the barrier effects of the coating under immersed conditions but failed to protect metal substrates in a salt-spray environment. However, further studies are required for detailed clarifications on the role of surface microstructures in the coating's barrier performance.

In this study, epoxy coatings with particulated surface microstructures were fabricated via soft lithography using abrasive papers as the templates. By changing the grit size of the abrasive papers, the coating's surface microstructures and their induced hydrophobicity can be tuned. The barrier properties of these coatings were evaluated by electrochemical impedance spectroscopy (EIS) performed in wet-dry cyclic immersion and in salt-spray environments. From these tests, the role of the surface morphology in the long-term barrier properties of epoxy coatings is discussed.

2. Experimental

2.1. Materials

Abrasive papers with different grit numbers (P600, P2000 and P6000) were supplied by 3M, and PDMS prepolymer and hardener (Sylgard 184) were purchased from Dow Corning. Bisphenol A diglycidyl ether (BADGE) and Jeffamine D230 hardener were purchased from Sigma-Aldrich. Other solvents and reagents were purchased from Sinopharm.

2.2. Preparation of PDMS templates

Sylgard 184 and its hardener (10: 1, w/w) were mechanically mixed at 25°C for 15 min. The obtained solution was poured onto the abrasive paper fixed in a 20×20 cm mold, followed by degassing in a vacuum oven to eliminate the air bubbles to ensure a high fidelity replication. After curing at 60°C for 12 h, the PDMS template was peeled from the abrasive paper.

2.3. Preparation of epoxy coatings

The Q235 steel panel (10×20 cm²) was ground with P400 abrasive paper, followed by cleaning with acetone under ultrasonication before coating. BAGDE and Jeffamine D230 (3:1, w/w) were mechanically stirred at 40°C and the mixture was applied on the steel substrate using a rod applicator. After curing at 80°C for 2 h, another layer of the mixture was applied on the first layer to minimize coating defects. The PDMS template was subsequently pressed on the mixture which was cured at 80°C for 2 h and 125°C for 3 h. After peeling off the PDMS template, a hydrophobic epoxy coating with an abrasive paper-like surface was obtained. A regular coating with a smooth surface was prepared as the control using a similar procedure. The thickness of all of the coatings was ~ 50 μm .

2.4. Surface characterizations

The surface microstructures on the abrasive papers and the corresponding epoxy coatings were observed using scanning electron microscopy (SEM, FEI Quanta 250) with an accelerating voltage of 10 kV. The surface roughness and surface area of the hydrophobic coatings were measured using confocal laser scanning microscopy (CLSM, KENYENCE VK-X). The corrosion morphologies of the coatings were observed by stereomicroscopy (KENYENCE VHX-P6000). To evaluate the surface hydrophobicity, a sessile water droplet of 4 μL was placed onto the coating surface and the static contact angle was immediately measured using a Dataphysics OCA20 goniometer. The values of the contact angles were calculated based on circular fitting by the instrument software. The contact-angle hysteresis (CAH) was measured through adding and withdrawing water (2 μL) from a sessile water droplet of 4 μL .

2.5. EIS analyses

EIS measurements were conducted to assess the degradation of the barrier properties of the coatings that were exposed to two different corrosion environments. The wet-dry cyclic immersion tests were performed for 30 days inside of a homemade instrument. The coated specimens were exposed to an alternating condition of 30 min immersion in a 3.5 wt% NaCl solution and 30 min of air exposure at 50% RH and RT. The salt-spray tests were also performed for 30 days according to ISO 7253. Prior to both types of tests, the edges and back sides of the coated specimens were covered with water-resistant tape and silicone rubber. EIS measurements were conducted in the 3.5 wt% NaCl solution at $25 \pm 2^\circ\text{C}$ using a

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