ARTICLE IN PRESS

Corrosion Science xxx (xxxx) xxx-xxx



Contents lists available at ScienceDirect

Corrosion Science



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

Fabrication of polydimethylsiloxane-derived superhydrophobic surface on aluminium via chemical vapour deposition technique for corrosion protection

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A R T I C L E I N F O

Keywords: A. Aluminium A. Organic coatings B. EIS C. Pitting corrosion

ABSTRACT

In this paper, a facile method based on the combination of chemical-etching and chemical vapour deposition is developed for the fabrication of superhydrophobic polydimethylsiloxane-derived film on aluminium substrate. It is revealed that the fabricated superhydrophobic aluminium has a static water contact angle as high as 158.7°, a good self-cleaning property and a persistent high corrosion resistance to 3.5 wt.% NaCl solutions. The presented method provides a scalable route to endow metals and alloys with multiple functions, making them suitable for the applications that need waterproof, dustproof and corrosion resistance properties.

1. Introduction

Superhydrophobicity phenomenon, known as "lotus effect", exists widely in nature, such as on the surface of plant leaves (lotus leaf, Pistia stratiotes), on the bodies of some animals and insects (Stenocara beetle, water skipper) [1–3]. Inspired by this phenomenon, great efforts have been made to artificially prepare superhydrophobic surfaces, as well as explore their applications [4-8]. Up till now, various methods have been used to create superhydrophobic surfaces, including chemical vapour deposition (CVD) [9,10], etching [11-14], sol-gel techniques [15,16], hydrothermal synthesis [17], layer-by-layer self-assembly [18,19], electrochemical methods [20–25], etc. Among these methods, CVD holds a great promise of preparing nano-thick or even single-atomthick hydrophobic films on metal substrates [25,26]. However, the existing researches on CVD-based superhydrophobic surfaces mainly focus on their wetting-related properties such as self-cleaning, antiicing, waterproof, oil-separation, ect., few of them have reported the corrosion protection performance of the hydrophobic films themselves.

For the corrosion protection of superhydrophobic surfaces, it has been revealed that the air film existing at the solid/liquid interface plays an important role in preventing metal substrates from being corroded by aggressive electrolyte [24,27,28]. Generally, the trapped air acts as an effective barrier film to water during short-term immersion (Cassie wetting state). However, it has been reported that ultrafine electrolyte particles generated in salt spray tests and corrosive vapours formed during long-term immersion may easily penetrate the hydrophobic film to directly contact the metal substrates, resulting in the disappearance of the strapped air films (Wenzel wetting state) and a rapid deterioration of corrosion protection [29,30]. Therefore, it is inferred that if the hydrophobic film of a superhydrophobic surface has an extremely dense structure, it would provide a long-term dual protection for the metal substrates even in Wenzel wetting state. Up to now, dense and hydrophobic polymeric films (e.g. modified epoxy, polyanline, polydimethylsiloxane, etc.) with rough surface have been reported to be a promising material that provides a durable corrosion protection for metal substrates, as well as endowing the metal substrates with selfcleaning, dustproof and waterproof properties [31-33]. However, to our best knowledge, it is still challenging to fabricate dense and longterm hydrophobic or superhydrophobic films on a commercial scale on arbitrarily shaped metal substrates for corrosion protection applications.

In this work, a facile and scalable two-step method for the fabrication of dense and ultra-thin superhydrophobic polydimethylsiloxane (PDMS) film on Al substrates is demonstrated. The presented method involves a chemical etching procedure of Al substrates in HCl solution and a procedure of CVD at low temperature. The PDMS-derived film with rough micro/nano-scale structure and high contact angle (CA) endows the Al substrates with desirable corrosion resistance and selfcleaning properties. This work would shed light on the preparation of ultra-thin coating on aluminium substrates with enhanced corrosion

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http://dx.doi.org/10.1016/j.corsci.2017.09.005

Received 25 November 2016; Received in revised form 5 September 2017; Accepted 7 September 2017 0010-938X/ @ 2017 Elsevier Ltd. All rights reserved.

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resistance and lotus leaf-like function (e.g. waterproof, dustproof, selfcleaning, etc.), while without obvious change of some properties at the metal surface (e.g. thermal conductivity, optical properties, etc.).

2. Experimental

2.1. Chemical etching of aluminium substrates

Industrial Al sheets (99.99% purity) were cut into 20 mm \times 60 mm rectangular foils with a thickness of 0.4 mm. Firstly, the Al foils were ground with 240 mesh SiC paper for \sim 5 s to damage the oxide film rapidly. Then, then foil was successively ground with 800 and 1200 mesh SiC paper for \sim 10 s to further remove the oxide film. Subsequently, the Al foils were cleaned ultrasonically with acetone and deionized water for 5 min, respectively. The cleaned samples were then immersed in 1 M NaOH solution for 1 min at room temperature to completely remove the oxide film on their surface. After this immersion, the samples were rinsed with deionized water and dried in the air at room temperature. The pretreated Al foils were then chemically etched in 2.6 M HCl solution at 20 °C for 5 min and finally rinsed with deionized water for three times. And the resultant Al sample is denoted as chemical-etched Al (CE-Al).

2.2. Preparation of superhydrophobic aluminium

An open crucible pot containing 5 mL polydimethylsiloxane (PDMS) was placed in a stainless steel chamber. A 400-mesh porous stainless steel screen was placed on the crucible pot, and the CE-Al substrate was placed on the stainless steel mesh. The stainless steel screen was used to suspend the aluminium foil in PDMS vapour so that PDMS can grow uniformly on the aluminium surface. The stainless steel chamber was sealed and then heating at 300 °C for 5 h in a muffle furnace. When heated in air, decomposition (Reaction 1, D is cyclosiloxanes) and oxidization reactions (Reaction 2) will occur to the PDMS. In addition, the produced cyclosiloxanes can be further oxidized to form hydrophobic organic SiO₂ nanoparticles adsorbing onto the CE-Al surface. These reactions occurred during the calcination processes. As calcination proceeds in an enclosed space, the PDMS-derived SiO₂ nanoparticles can connect to each other directionally to form film structures on CE-Al surface gradually. As a result, surfaces with low surface energy and rough structures were obtained, which led to the formation of superhydrophobic surfaces [34]. The resultant Al sample is denoted as superhydrophobic Al or PDMS-coated chemical-etched Al (PDMS-CE-Al). The experimental scheme is presented in Fig. 1. Similarly, PDMS-coated pristine Al was also prepared in this work (denoted as PDMS-Al).



Fig. 1. Experimental scheme of the preparation of superhydrophoic Al.



2.3. Electrochemical tests

The electrochemical measurements were carried out in a conventional three-electrode electrochemical cell employing 3.5 wt.% NaCl solution as the electrolyte. A saturated calomel electrode (SCE) was used as the reference electrode, and a platinum electrode was employed as the counter electrode. The working electrode was the as-prepared aluminium sample. Before each electrochemical measurement, open circuit potential (E_{OCP}) of the samples was monitored for ~30 min to obtain a stable value. Electrochemical impedance spectroscopy (EIS) was performed on a CHI 660 B electrochemical workstation (Chenhua, Shanghai) at the obtained E_{OCP} with applied sinusoidal perturbations of 10 mV over a frequency range of 0.01 Hz-100 kHz. The Tafel polarization was performed on a CS300 (CorrTest, Wuhan) and scanned from cathodic to the anodic direction (from E_{OCP} -300 mV to E_{OCP} + 300 mV) with a scan rate of 10 mV min⁻¹. Each experiment in this study was performed at least three times in this study to confirm reproducibility of the data.

2.4. Apparatus and characterization

The morphology of the Al samples were characterized by scanning electron microscopy (SEM, JEOL-5600LV, Japan). The PDMS-derived film on Al was confirmed by Fourier transform infrared (FT-IR) spectra, and the spectra were recorded on an AVTAR360 Nicolet FT-IR spectrometer using a KBr pellet. The static water CAs of all the Al samples were measured from a 5 μ L deionized water droplet by an automated goniometer (Model 200). The static water CAs were measured at three different areas for each sample.

3. Results

Fig. 2a shows the FT-IR spectra of pristine PDMS and superhydrophobic Al (PDMS-CE-Al), respectively. The FT–IR spectrum of superhydrophobic Al shows three characteristic peaks appearing at 2964, 2902 and 1260 cm⁻¹, and they are ascribed to the asymmetric Al). C–H stretching of –CH₃, the symmetric C–H stretching of –CH₃ and the symmetric deformation of –CH₃ [34], respectively. The three peaks agree well with that of the PDMS elastomer, which indicates the for- D_{x+1} (x=2, 3, 4...) mation of PDMS-derived film on Al substrate. Elemental mapping was also used to characterize the formation of such a PDMS-derived film on (1) Al substrate. The images of elemental mapping reveal a uniform distribution of Al, C, O and Si element on the Al substrate which was treated by PDMS vapours, verifying that the Al has been completely coated by a PDMS-derived film (Fig. 2b–e).

Fig. 3a–e present the SEM images of different Al samples. It is observed that ground Al exhibits grinding cracks, and its surface is relatively rough (Fig. 3a). After 1 min of immersion in 1 M NaOH solution, the Al becomes more rough (Fig. 3b). Whereas the Al substrate shows a typical porous morphology with numerous micro-scale cavities and islands dispersed across its surface after HCl-etching (Fig. 3c). High-magnification SEM study further reveals that the HCl-etched Al has a rectangle topography, with an average width of ~1 μ m and an average length of ~3–10 μ m. Fig. 3d shows that the surface morphology of HCl-etched Al does not change obviously after growing PDMS-derived film on its surface via CVD. The inset image presented at the bottom right of Fig. 3d is the cross-section SEM image of the resultant PDMS-CE-Al surface, and it reveals the roughness nature of PDMS-CE-Al surface.

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