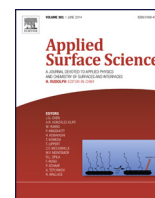




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Superhydrophobic membranes on metal substrate and their corrosion protection in different corrosive media

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ABSTRACT

Superhydrophobic membranes with different surface morphologies and chemical compositions were prepared by sol–gel method. The superhydrophobicity on the surface with high roughness is ascribed to the biomimetic structure of the lotus, while the wetting behavior observed on the relatively smooth surface is attributed to the air bubbles trapped in the asperities and the discontinuous contact line. Moreover, it has been demonstrated that the humid air test is required to assess the tendency occurrence of electrolyte film and that the Tafel plots and the water repellency are not adequate to evaluate the corrosion resistance in the practical environment. Compared with the surface chemical compositions, the surface morphology plays more important role in anti-corrosion ability in humid air.

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Introduction

Wettability is one of the most significant properties for interfaces in academic research due to its great potential applications [1]. Inspired by the special wetting phenomena in nature, such as lotus [2–4], water-strider [5,6], gecko's feet [7,8] and rose petals [9–11], a growing number of artificial surfaces with self-cleaning [12], anti-icing [13,14] and drag reduction [15,16] properties have been developed. Using superhydrophobic membranes to enhance the corrosion resistance of metal has been an upsurge of interest in corrosion protection [17–20]. The mechanism of corrosion resistance is to restrain the contact between the corrosive solution and the metal substrate using the water repellent nature of the superhydrophobic membranes.

It is widely accepted that the influence of superhydrophobic membranes on the corrosion resistance of metal is evaluated by the potentiodynamic polarization curve and the surface wettability by the following two aspects: on one hand, it is generally believed that the lower corrosion current (I_{corr}) value denotes lower corrosion dynamic rate and positive corrosion potentials (E_{corr}) represents lower corrosion; on the other hand, the higher static contact angle and lower tilt angle are believed to provide better corrosion resistance due to the mechanism of anti-corrosion property of superhydrophobicity as described above. However, the corrosion

resistance of superhydrophobic membranes in humid atmosphere is relatively recent and has not been sufficiently investigated.

In this paper, superhydrophobic membranes with different surface roughness and chemical compositions were prepared by sol–gel method. A deep investigation for how superhydrophobic membranes influence the corrosion resistance of metal was carried out in 3.5 wt.% NaCl aqueous solution and humid air with special consideration given to the surface morphology and chemical compositions. Furthermore, using only the surface wettability (i.e., contact angle and tilt angle) and Tafel plots to evaluate the effect of superhydrophobic membranes on the corrosion resistance of metal is not objective, humidity test is required to assess the tendency occurrence of electrolyte film.

Experimental

Materials

Tetraethyl orthosilicate (TEOS), trimethylethoxysilane (TMES), methyltrimethoxysilane (MTMS), hexadecyltrimethoxysilane (HDTMS), ammonium hydroxide (25%) and all solvents used were purchased from Sigma-Aldrich (U.S.A.). All chemicals were used as received. 080M46 steel was used as the substrate.

Pre-treatment of steel substrate

To obtain uniform membranes, the substrates need to get pre-treatment. The specimens were embedded in an epoxy resin and

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the cross sections of specimens were grounded with waterproof abrasive paper sheets up to no. 2500 and then polished with diamond paste. The polished substrates were rinsed with deionized water and ultrasonically degreased in ethanol for 20 min to remove the surface contamination and dust.

Preparation of superhydrophobic membranes

T1 and T3 membranes

First, 3 mL of ammonium hydroxide (25%) was added to 40 mL of ethanol. The solution was stirred vigorously at 60 °C for 30 min. Then 2 mL of TEOS was added to the solution dropwise. The reaction mixture was further stirred for 1.5 h and left for aging at room temperature over night. Finally, the prepared silica sol was coated on the pre-treated substrate by spray-deposition. The prepared membrane was desiccated in an oven at 50 °C for 1 h to remove the residual solvent followed by soaking in 3 wt.% MTMS and 5 wt.% HDTMS in hexane at 35 °C for 6 h, respectively. The membrane modified with MTMS is abridged as T1, while the membrane modified with HDTMS is abridged as T3.

T2 membrane

The preparation of T2 has already been pointed out in our previous work [21]. In the first step, 1 mL of ammonium hydroxide (25%) was added to 40 mL of ethanol. The solution was stirred vigorously at 60 °C for 30 min. Then 1 mL of TEOS and 0.8 mL of TMES was added to the solution dropwise. The reaction mixture was further stirred for 2.5 h and left for aging at room conditions for 30 days. Finally, the pre-treated substrate was soaked into the colloids and then withdrawn with a speed of 1.5 mm/s. The membrane was desiccated in an oven at 50 °C for 1 h to remove the residual solvent.

Characterization

The morphology of the prepared films was characterized by field-emission scanning electron microscopy (FE-SEM, Hitachi S-4800 II). Atomic force microscopy (AFM, Bruker Multi Mode 8) was used in ScanAsyst mode for roughness measurements. The contact angles (CAs) and tilt angles (TAs) were carried out with a contact angle measurement instrument (JC2000D2, Shanghai Zhongchen Digital Technology Apparatus Co., Ltd). Fourier transform infrared (FTIR, Nicolet IS-10) spectroscopic studies of the films were carried out using spectroscopy in 1200–3600 cm^{-1} range. The corrosion resistance of the membranes in the corrosive solution medium was evaluated by a potentiodynamic polarization curve measured in 3.5 wt.% NaCl aqueous solution with a saturated calomel reference electrode (SCE) by the Electrochemical workstation (VersaSCAN system, Princeton). The anti-corrosion ability of the membranes in the humid air was studied in a humidity chamber (RGDS-500, Surui Instruments Co., Ltd).

Results and discussion

FT-IR studies

The chemical composition of the membranes was investigated by the FT-IR spectroscopy. Fig. 1 shows the FT-IR spectra of T1, T2 and T3. The peaks between 3100 and 3500 cm^{-1} are due to the stretching vibration of –OH groups and the peaks at around 1700 cm^{-1} are due to the physically adsorbed water [22]. The peaks observed at 2950 and 1400 cm^{-1} are due to the stretching and bending of C–H bond [23]. The C–H peaks observed on T1 and T3 are attributed to the modification of MTMS and HDTMS by self-assemble method. While the C–H bonds observed on T2 are due to the addition of TMES, which acts as the co-precursor. It is clearly evident that all the prepared membranes are hydrophobic, which

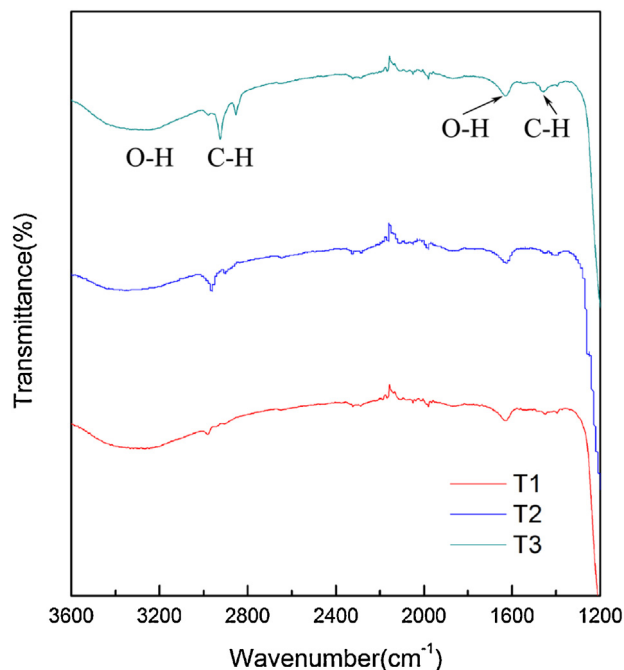


Fig. 1. FTIR spectra of T1, T2 and T3 membranes.

would influence the wetting behavior as described in the following section. On the other hand, the intensity of C–H bond of T3 is more as compared to those of T1 and T2. This could be attributed to the long chain of HDTMS, clearly indicating that the particles of T3 are more hydrophobic. The –OH groups can be seen in all the FT-IR spectra, indicating that even for the superhydrophobic surface, the –OH groups exist.

Surface morphology

To characterize the surface features of various samples, the surface morphologies were carried out with FE-SEM and AFM. Fig. 2 shows the FE-SEM images. For the sake of clarity and ease of comparison, all pictures have the same magnification. It can be seen that the particle size of T2 is much smaller, which would lead to the better water repellency and is advantageous to the anti-corrosion ability of the steel in the humid air as described in the following section. This is because with the addition of TMES, more –CH₃ groups are introduced to the surface of the silica, which in turn retards the condensation reactions and the size of the particles is limited. Meanwhile, the scale of the silica clusters has been limited and relatively flat surface is obtained [24].

In order to further study the surface morphology, AFM images are presented in Fig. 3. The images were recorded at 3 × 3 μm^2 planar in ScanAsyst mode. R_q is the root-mean-square roughness. The topographies show a striking contrast: the surfaces of T1 and T3 exhibit noticeable high roughness with R_q of 373 nm yet the surface of T2 is relatively smooth with R_q of 44 nm. This is because the scale of the silica clusters of T2 has been limited due to which that the addition of TMES restrains the condensation reaction. On the other hand, micro-scale structures could be observed in Fig. 3a, indicating the hierarchical structures of T1 and T3, that is, with nano-scale details superimposed on the micro-scale details.

Wettability

Fig. 4a shows the static contact angles (CAs) and tilt angles (TAs) of prepared membranes. The volume of the individual DI water droplet is 5 μL . Generally speaking, on macro level, all

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