



A facile method for fabrication of superhydrophobic surface with controllable water adhesion and its applications



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ABSTRACT

In this work, a simple alkaline etching method was presented to fabricate superhydrophobic surfaces. X-Ray Diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS) and atomic force microscope (AFM) were utilized to characterize the surfaces. The morphology of superhydrophobic surfaces shows hierarchical structures and the corresponding water contact angles (WCA) are more than 155°. The adhesion force between the water droplets and the surfaces were discussed in detail, the specimen etching for 40 min in NaOH solution shows the lowest adhesion. The applications of these superhydrophobic surfaces were exploited. For instance, the superhydrophobic surface with high adhesion can be used in drug delivery. In addition, the self-cleaning property of the superhydrophobic surface with low adhesion was also tested by applying nano-silica powder as dust on the surfaces. Importantly, the superhydrophobic surfaces show excellent anti-corrosion performance with an extremely low corrosion current density of 2.748×10^{-9} A/cm², and the anti-corrosion mechanism was discussed comprehensively. The present method can also be used to fabricate large-scaled superhydrophobic surfaces and expand applications of metal materials.

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1. Introduction

Inspired by natural surfaces, such as lotus leaves, nepenthes and gecko's feet [1–4], which show unusual properties due to their special structures. Researchers have been made great efforts to fabricate surfaces with special structures to achieve excellent performances [5–9]. Among these properties, superhydrophobicity is the most mentioned property due to its wide applications in self-cleaning [5], anti-corrosion [6], anti-icing [7], microfluidic transmission [8] and oil-water separation [9]. Researches reveal that both rough surface and low surface energy account for the superhydrophobicity [10]. In general, surface energy can be easily decreased by modification with some organic compounds. Thus, main efforts have been devoted to constructing a significant rough structure on substrate to obtain superhydrophobicity.

In recent years, various methods have been developed to fabricate superhydrophobic surfaces, such as electrochemical

deposition [11], chemical vapor deposition [12], laser etching [13], anodic oxidation [14], electrospinning [15], hydrothermal synthesis [16] and chemical etching [17]. However, most of these methods require expensive equipment or complicated process. As an exception, chemical etching is a simple and low-cost technique. Liu et al. [18] fabricated a superhydrophobic Cu surface via a facile solution-phase etching route by the H₂O₂ and HCl etchants, it exhibited a water contact angle of 170° and a sliding angle of ~2.8° after hydrophobization. Wang et al. [19] prepared a superhydrophobic surface on steel via the synergetic corrosion of H₂O₂ and H₂SO₄ and followed by modification of silanes, the as-prepared superhydrophobic surface exhibited excellent anti-icing ability in extremely condensing condition. Boinovich et al. [20] developed superhydrophobic surface by immersing nanoparticles and hydrophobic polymer on etched stainless steel, it showed durable icephobicity.

However, substrate would be damaged by chemical etching and could not be protected from corrosion. Generally, a coating can be deposited as a protective layer to prevent corrosion occurs on substrate surface. Thus, a deposited layer can be fabricated on substrate to serve as the surface which is applied to chemical etching. Up to now, the report of superhydrophobic surface

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fabrication with combination of electrochemical deposition and chemical etching method is still scarce. In this work, a Zn-Ni coating was firstly deposited on mild steel, and then a superhydrophobic surface was developed on Zn-Ni coating via the corrosion of NaOH solution and with modification of myristic acid. The morphology and composition of the as-prepared specimens were characterized, and the wettability and water adhesion of specimens were systematically studied. Moreover, the surfaces' self-cleaning and corrosion resistance were also discussed. The applications of as-prepared specimens are mainly focus on its corresponding properties.

2. Experimental

2.1. Materials and chemicals

Mild steel (composition in wt. %: C \leq 0.1, Mn \leq 0.5, P \leq 0.035, S \leq 0.035 and Fe balance) were purchased from Shanghai Guangzong Industrial Co., Ltd., China, and then cut into 50 mm \times 20 mm \times 1 mm as substrates in this work. A titanium mesh coated with ruthenium oxide was used as anode plate. The composition of plating bath was shown in Table 1. Myristic acid (CH₃(CH₂)₁₂COOH, 95%) was used as modification agent which purchased from Aldrich Chemical Inc., USA. All other chemicals with analytical grade were obtained from Nanjing Chemical Reagent Co., Ltd., China. Deionized water (DI water) was self-prepared. All the chemicals were used as received without any further treatment.

2.2. Fabrication of specimens

Before electrochemical deposition, mild steel was first degreased in the solution of 35 g/L Na₂CO₃, 25 g/L NaOH and 35 g/L Na₃PO₄ at 75 °C for 20 min, then immersed into 125 ml/L HCl solution to remove the possible oxide layer on the surface. Lastly, the substrate was activated in the solution of 30 ml/L HCl. Substrate need to be rinsed by deionized water between each step. After that, substrate was deposited in the above plating bath for 20 min. Then the specimens were immersed into 1 mol/L NaOH solution at 30 °C for 10 min, 20 min, 30 min, 40 min, 50 min, respectively, and dried under atmospheric condition. The dried specimens were then immersed into 0.1 mol/L myristic acid to modify the surfaces with low energy component. In order to distinguish the specimens which were immersed into NaOH solution for 10–50 min simply, the specimens before and after modification with myristic acid were noted as S1, S2, S3, S4, S5 and M1, M2, M3, M4, M5, respectively.

2.3. Characterization and tests

The crystal structure of specimen was analyzed by X-ray diffraction (XRD, D8 Advance, Bruker, Germany) at a scanning speed of 2°/min from 10° to 90° of 2 θ , using a filtered Cu K α

(0.15406 nm) as a radiation source. The morphologies and compositions of specimens were characterized by a field emission scanning electron microscope (FESEM, LEO1550, ZEISS, Germany) equipped with an X-act electron microprobe for energy dispersive X-ray spectrum (EDS), and an atomic force microscope (AFM, Dimension Edge, Bruker, Germany) was used for visualization of the surfaces. The water contact angles (CA) were tested by a contact angle measuring system (DSA100, Kruss Corporation, Germany), the average CA values were obtained by measuring five different parts with a 6 μ L water droplet for the same specimen. The water bouncing behaviors were observed by a high speed camera.

The corrosion resistance of specimens was measured in 3.5 wt % NaCl solution (pH = 5.9) for potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS) by electrochemical workstation (CHI 750C, Shanghai Chenhua Instrument Corporation, China) at room temperature. A standard three-electrode system was employed in this electrochemical test. Specimen with an exposed area of 1 cm² was acted as the working electrode, a platinum (Pt) sheet and a saturated calomel electrode (SCE) were used as the counter electrode and the reference electrode, respectively. Before the electrochemical tests begin, specimens were immersed into the NaCl solution for 6 h to obtain a stable open circuit potential (OCP). The potentiodynamic polarization curves were measured with a scanning rate of 5 mV/s, and the EIS curves were operated at the OCP of specimens in the frequency ranges between 10⁵ Hz and 10⁻² Hz with a sinusoidal perturbation signal of 5 mV.

3. Results and discussion

3.1. XRD patterns

In order to identify the crystal structure and the preferred orientation of the electrochemical deposited coating, XRD measurement was used to analyze it. As shown in Fig. 1, it can be observed that the coating consist of several peaks in 2 θ of 42.8°, 44.7°, 62.3°, 65.0°, 78.7° and 82.3°. The peaks in 42.8°, 62.3° and 78.7° are corresponding to that in the JCPDS card (No. 06-0653), which show crystal orientation of (330), (600) and (552) plane respectively. Indicating the coating has only a single cubic γ -phase structure of γ -Zn₅Ni₂₁ [21,22]. The Fe peaks in this XRD pattern

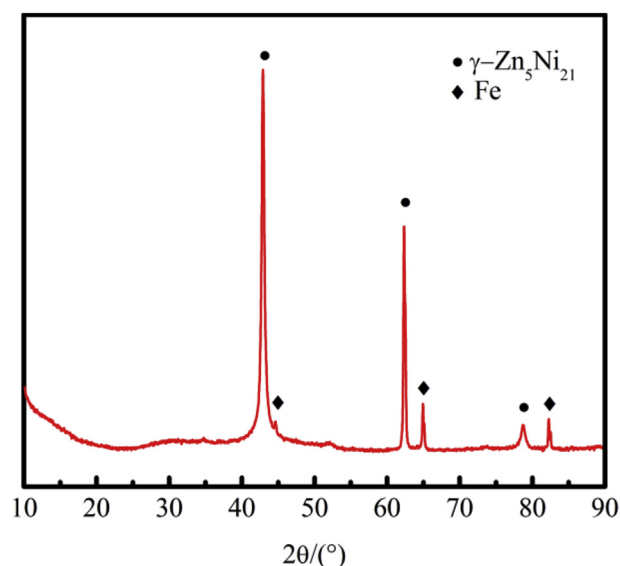


Fig. 1. XRD pattern of Zn-Ni coating.

Table 1
The composition of plating bath and parameters.

Plating bath compositions	Concentration (g/L)	Bath parameters
H ₃ BO ₃	35	pH 5.0
ZnCl ₂	75	Temperature 35 °C
NiCl ₂ ·6H ₂ O	110	Planting time 20 min
NH ₄ Cl	35	Stirring speed 100 rpm
KCl	200	Current density 4 A/dm ²
K ₃ C ₆ H ₅ O ₇ ·H ₂ O	20	
C ₁₈ H ₂₉ NaO ₃ S	0.1	

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