



Full Length Article

Effect of pulse frequency on the one-step preparation of superhydrophobic surface by pulse electrodeposition

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ARTICLE INFO

Keywords:

Superhydrophobicity
Wettability
Pulse-electrodeposition
Frequency

ABSTRACT

A union of surface with rough micro/nano structures and low surface free energy is critical for the preparation of superhydrophobic surfaces. In this study, a rapid one-step pulse electrodepositing method was used to prepare superhydrophobic surfaces on Cu substrates. The electrolyte was prepared with ethanol, myristic acid ($\text{CH}_3(\text{CH}_2)_{12}\text{COOH}$) and lanthanum chloride ($\text{LaCl}_3 \cdot 6\text{H}_2\text{O}$). The surface morphology, chemical composition and superhydrophobic property were characterized by SEM, XRD, FTIR, EDX, optical contact angle instrument and high-speed camera. It turned out that the deposited surfaces have micro/nano hierarchical structures mainly being composed of lanthanum myristate. It is found that the optimal water contact angle is approximately 160.3° with a sliding angle of around 5° . The effects of pulse frequency on the surface morphology and wettability were specifically studied and discussed under an equivalent electrolytic time of 10 min. In this way, it can effectively save time and be simply applied to other materials with good conductivity and has a promising wide range application.

1. Introduction

Surfaces and interfaces are the keys to fulfill the functionality of devices and materials, and so the construction of surface micro/nano structures has great significance to the performance of materials [1,2]. Wettability, one of the foremost features of a solid material, has been extensively researched in recent years. Usually, the static water contact angles (WCA) above 150° are identified as superhydrophobic surfaces, while indications about the adhesion of water droplets are given by the hysteresis and the sliding angle (SA) [3]. Such surfaces have attracted enormous research interest in light of their applications in self-cleaning, anti-icing, anti-corrosion, bactericidal surfaces, water-oil separation, and so on [4–10]. The superhydrophobic phenomenon is due to a suitably roughened surface having a particular micro-nanostructures and/or material with low surface energy.

Enlighten by several kind of plants and insects in nature, such as rose petal [11], lotus leaf [12,13], rice leaf [14], cicada wings [8] and mosquito complex eyes [15], numerous ways are sought to prepare surfaces of man-made superhydrophobicity, including electrochemical corrosion [16,17], chemical corrosion [18–20], solution-immersion [21,22], sol-gel processing [23,24], femtosecond laser ablation [25,26], CVD [27], electro-spinning [28], hybrid processes [29–32].

Nevertheless, there are a few disadvantages such as chemical pollution, ultra-clean and high vacuum operating conditions, high-priced equipment (e.g. photolithographic equipment), time-consuming, complicated multi-step machining with most of the methods mentioned above, which restrict their extensive potential applications.

Direct current (DC) electrodeposition [33–39] has aroused great interest among the research staff for the preparation of superhydrophobic surfaces on conductive materials due to its appealing advantages of low cost, facility and high efficiency. Furthermore, no matter how complicated the geometric shape of the substrates is, the feasibility of the mass production in industrial applications is an obvious superiority of cathodic electrodeposition. Besides, the surface morphology of the deposits is able to be easily controlled by changing the processing parameters such as electrical parameters, concentration and composition of electrolyte, machining time and temperature. Rare earth elements (REE) were applied to the preparation of superhydrophobic surfaces owing to its effective role in enhancing thermal stability as well as anti-corrosion of advanced functional materials [7]. Liu et al., prepared cerium myristate superhydrophobic surface with controlled adhesion by electrodeposition [34]. Ishizaki et al., studied the corrosion resistance and durability of superhydrophobic surfaces formed on a magnesium alloy coated with a nanostructured cerium

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Received 21 April 2018; Received in revised form 2 July 2018; Accepted 17 July 2018

Available online 18 July 2018

0169-4332/ © 2018 Published by Elsevier B.V.

oxide film and fluoroalkylsilane molecules [40]. Xiao et al., tuned the wettability of $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ coatings from superhydrophilicity to superhydrophobicity by hierarchical microstructure [41].

Compared with conventional DC electrodeposition in which only the potential or current can be adjusted, pulse electrodeposition can provide better control on the structure of electrodeposits as well as their performance by adjusting duty ratio and frequency. These unique parameters offer the opportunity to perturb the adsorption-desorption phenomenon at the cathode/electrolyte interface (catholyte), which has a considerable impact on the nucleation rate and the growth rate of crystals during the electro-crystallization, and ultimately decide the size and shape of crystals [42–44]. Different from the direct current deposition method, the pulse electrodeposition owns some unique advantages, such as rapid refreshment of adsorptive ions, thin diffusion layer, high deposition efficiency and convenient control of surface morphology. Apparently, the forming mechanisms of pulse electrodeposition are different from that of DC electrodeposition.

Nonetheless, to our knowledge, there are only a few explorations and reports on preparing superhydrophobic surfaces by pulse electrodeposition [38,45–47]. Although, in these studies, a number of superiorities over the DC deposition method have been discussed, seldom systematic study has been conducted to investigate the influence of the electrical parameters of the pulsed current on the morphologies of the deposits as well as their effect on wetting properties.

Taking the above background into account, in this study, a one-step processing method for preparing of superhydrophobic surfaces was presented. The effect of pulse frequency on the surface structure and wettability of the deposits was studied by pulse electrodeposition with an electrolytic solution containing REE. According to the results of experiments, the forming mechanism of the deposited layer and performance of superhydrophobicity are discussed. With the advantages of simplicity, low toxicity, cost-effectiveness and greater control, this presented method could be simply applied to other conducting materials with a promising future for wide applications.

2. Materials and methods

2.1. Materials and sample preparation

Analytically pure myristic acid ($\text{CH}_3(\text{CH}_2)_{12}\text{COOH}$) and absolute ethanol were purchased from Damao Chemical Co., Ltd. Lanthanum chloride ($\text{LaCl}_3 \cdot 6\text{H}_2\text{O}$) was supplied by Kemiou Chemical Reagent Co., Ltd, which can be used without purifying. Copper plates were employed as the substrates for the preparation of deposited layers. Firstly, the copper substrates with the size of $30\text{ mm} \times 30\text{ mm} \times 1\text{ mm}$, were ground with abrasive papers (grade 800–2000), afterward, degreased ultrasonically in absolute ethanol for 10 min, and then ultrasonically cleaned in deionized water for 10 min and finally dried at natural atmosphere.

2.2. Pulse electrodeposition

To prepare electrolytes, 0.1 M $\text{CH}_3(\text{CH}_2)_{12}\text{COOH}$ and 0.04 M $\text{LaCl}_3 \cdot 6\text{H}_2\text{O}$ were solved with 150 ml absolute ethanol under magnetic agitation at room temperature. The anode and cathode were both copper plates and the distance between them was set by 2 cm in the beaker. With voltage of 30 V and a duty ratio of 50%, pulse currents with frequencies ranging from 5 to 3000 Hz were applied by using a pulse power supply (ELGAR SW5250A, USA). Table 1 shows the electrical parameters and the composition of the electrolyte. The waveforms of the pulse current applied in the experiments are exemplified in Fig. 1. The used solution was replaced with fresh electrolyte to avoid pollution and lower the effects of solution concentration after each sample was prepared. All the samples were treated with an equivalent electrolysis time for 10 min at ambient temperature under agitation condition by a magnetic agitation setup with a rotation rate of 200 rpm.

Table 1

Experimental parameters of the pulse electrodeposition.

Current parameters	Value	Electrolyte parameters	Value
Voltage (V)	30	Lanthanum chloride (M)	0.04
Duty ratio	50%	Myristic acid (M)	0.1
Frequency (Hz)	5, 50, 500, 1k, 2k, 3k	Ethanol (ml)	150

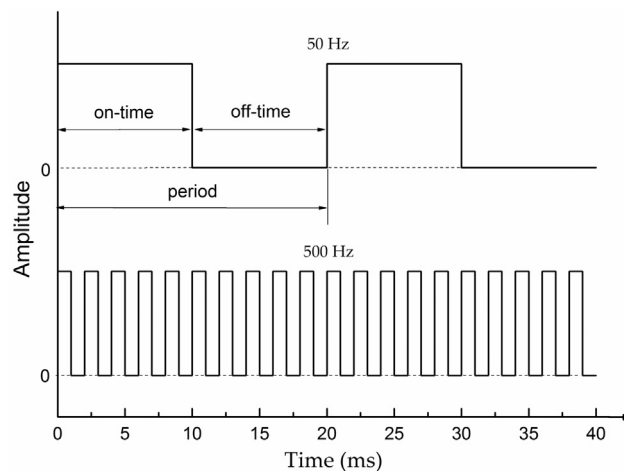


Fig. 1. An example of pulse waveforms.

After that, ethanol and distilled water were used to wash the cathode for several times under atmospheric condition. Ultimately, the superhydrophobic surfaces with hierarchical micro-nanostructures were prepared.

2.3. Sample characterization

The surface morphologies and the corresponding surface elements of the deposits were investigated by a scanning electron microscope (SEM, JEOL JSM 6060LV, Japan) equipped with an energy dispersive spectrometer (EDX, Genesis 60, USA). To investigate the cross-sectional structure of the deposited layer, a focused ion beam field emission scanning electron microscope (FIB-FESEM, LYRA 3 XMU, Tescan, Czech) was operated at 5 kV to observe the surface and its FIB module was used to accurately remove materials from the as-prepared surface and then the SEM was used to observe the fabricated cross-section. The 3D profile and the corresponding surface roughness were measured by a white light interferometer (Bruker Contour GT-X, Germany). The average of six measurements at different locations on the samples was reported. The details of functional group were probed by a Fourier transform infrared spectrophotometer (FTIR, NICOLET 8700, USA) while the crystalline details was examined by an X-ray diffractometer (Bruker D8, Germany) applied with 40 kV and 40 mA and monochromatic $\text{Cu K}\alpha$ radiation ($\lambda = 0.15418\text{ nm}$). Water contact angles were measured by an optical contact angle meter (XG-CAMB1, China) under room temperature for each sample. By vibrating the burette, about $4\ \mu\text{l}$ water droplets were fallen onto the deposition layers from a height of 20 mm. The sliding angles were measured and read from an angle scale which installed on the tilting platform. Five measurements were detected at different locations on the same sample to obtain the average. The water bouncing was photographed with 400 fps by a high-speed photography system (Fastec HiSpec5, USA).

3. Results

3.1. Surface morphology

Morphology is one of the decisive factors of the wettability of

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