

Energy, Resources and Environmental Technology

Fabrication of superhydrophilic surface on copper substrate by electrochemical deposition and sintering process[☆]

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ARTICLE INFO

Article history:

Received 29 April 2014

Received in revised form 17 November 2014

Accepted 23 November 2014

Available online 15 April 2015

Keywords:

Superhydrophilic

Superhydrophobic

Copper surface

Electrochemical deposition

Sintering process

ABSTRACT

Superhydrophilic surfaces were fabricated on copper substrates by an electrochemical deposition and sintering process. Superhydrophobic surfaces were prepared by constructing micro/nano-structure on copper substrates through an electrochemical deposition method. Conversion from superhydrophobic to superhydrophilic was obtained via a suitable sintering process. After reduction sintering, the contact angle of the superhydrophilic surfaces changed from 155° to 0°. The scanning electron microscope (SEM) images show that the morphology of superhydrophobic and superhydrophilic surfaces looks like corals and cells respectively. The chemical composition and crystal structure of these surfaces were examined using energy dispersive spectrometry (EDS) and X-ray diffraction (XRD). The results show that the main components on superhydrophobic surfaces are Cu, Cu₂O and CuO, while the superhydrophilic surfaces are composed of Cu merely. The crystal structure is more inerratic and the grain size becomes bigger after the sintering. The interfacial strength of the superhydrophilic surfaces was investigated, showing that the interfacial strength between superhydrophilic layer and copper substrate is considerably high.

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1. Introduction

In recent years, wettability of solid surfaces has aroused a great deal of concern. Especially, super-hydrophobic surfaces, with water contact angle (CA) greater than 150° and superhydrophilic surfaces (with water CA below 5°) have attracted great interest for their special functions and potential applications [1–3]. Superhydrophobic surfaces have many practical applications, such as corrosion resistance, friction drag reduction, self-cleaning and anti-icing [4–7]. Similarly, superhydrophilic surfaces can be applied in heat transfer enhancement, biological medicine, self-cleaning, friction drag reduction, and so on [8–13]. Generally, chemical component and rough surface structure have great influence on the wettability of a solid surface. There are two basic methods for superhydrophilic surface fabrication: one is photo-induced superhydrophilic (PIH), for example TiO₂, ZnO and WO₃ will show superhydrophilic after irradiating by UV or visible light [14,15]. The other method is constructing rough structure on hydrophilic surfaces. In the last decade, numerous techniques for artificial superhydrophilic surface fabrication have been developed based on

the above methods. Liu *et al.* [16] prepared superhydrophilic surface by constructing strawberry-like microstructure on the molybdenum substrate via a hydrothermal method. Behners *et al.* [17] fabricated superhydrophilic surfaces on polymer film by photo-induced micro-folding. Superhydrophilic TiO₂–SiO₂ composite surfaces were prepared through a sol–gel route by Houmar *et al.* [18]. Additionally, there are plasma vapor deposition process [19], self-assembly approach [20] and electrochemical method [21], *etc.* However, a lot of artificial methods have certain limitations, such as complicated process, expensive equipment and low interfacial strength. Therefore, further efforts should be made to develop more simple and economical techniques for fabricating superhydrophilic surfaces with high interfacial strength.

Copper is a kind of important engineering material which has been widely used in lots of industrial applications. Because of the special functions, superhydrophobic and superhydrophilic copper surfaces have great potential applications. Superhydrophobic surfaces have been prepared on copper substrates by various processes, such as the solution-immersion method [22] and electrochemical approach [23]. However, preparation of superhydrophilic copper surfaces with high interfacial strength has not been reported. In this work a novel route for fabricating superhydrophilic copper surfaces with high interfacial strength is provided. Micro/nano-structure was first built on copper substrates via an electrochemical deposition method. The next step is a sintering heat treatment for the prepared copper surfaces. This flexible and controllable method does not require any chemical modification. Furthermore, the interfacial strength between

[☆] Supported by the National Natural Science Foundation of China (51275180), the Fundamental Research Funds for the Central Universities (2013ZM0003) and the Doctorate Dissertation Funds of Guangdong Province (sybzzxm 201213).

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superhydrophilic layer and copper substrate is sufficiently high for practical application.

2. Experimental

2.1. Materials and sample preparation

After polished by 1500[#] silicon carbide paper, the copper plates with a size of 20 mm × 20 mm × 2 mm (99.9%, Dongguan Tongzhuang Metal Materials Co., China) were ultrasonically cleaned in acetone (99.5%, AR) and deionized water for 3–5 min, respectively, then dried in air at ambient temperature. The electrolyte with 1 mol·L⁻¹ H⁺ and 0.02 mol·L⁻¹ Cu²⁺ was prepared using HCl (36%–38%, AR) and CuCl₂·2H₂O (99.0%, AR). The prepared copper plates used as anode and cathode were immersed into the electrolyte at ambient temperature while a DC regulated power supply (QJ21005X, Ningbo QJE Electronic Co., China) was utilized. The space between the cathode and the anode was 10 cm and both electrodes were placed vertically. The current densities varied from 10 to 50 mA·cm⁻² and the deposition time ranged from 5 to 20 min. After the electrochemical deposition, the cathode copper plate was cleaned with deionized water and dried at 100 °C in a thermostatic oven for 30 min, then cooled in air to ambient temperature. The next step was a sintering process. After heating for 80 min in a vacuum sintering furnace (ZSJ-45/45/60, ACEM, China) at an initial temperature of 30 °C, the temperature reached 400 °C and then thermal insulation for 20 min. To further heat for 30 min, the temperature reached 500 °C and then thermal insulation for 60 min. After that heating was stopped to allow the furnace temperature to drop to 200 °C, then the samples were taken out of the furnace and placed in air to cool further. In this sintering process, there was always protective gas in the furnace until the samples were taken out. The protective gas was nitrogen when

the furnace temperature was lower than 400 °C and it was hydrogen when the furnace temperature was above 400 °C.

2.2. Surface characterization

The morphology of the obtained surfaces was characterized by SEM (S-3700N, Hitachi, Japan) at 20 kV, and the chemical composition was analyzed using EDS (Quantax, Bruker, Germany) at 20 kV. The crystal structure of the prepared surfaces was determined by XRD (D8 ADVANCE, Bruker, Germany) with a Cu K_α radiation (λ = 0.15418 nm) at 40 kV and 40 mA. Contact angle measurements were performed on a contact angle system (JC2000D, Powereach, China) at ambient temperature. The accuracy is ± 1°. Water droplets (5 μl) were delivered onto five different sample spots for each specimen and the average of five contact angles was used for analysis.

3. Results and Discussion

3.1. Morphological analysis

SEM images of the non-sintered and sintered surfaces with different current densities and deposition times are depicted in Figs. 1 and 2, respectively. It is obvious that current density and deposition time have a great effect on the density of copper sediments, but little impact on the morphology of the obtained surfaces. Fig. 1(e1), (e2) and (e3) shows the morphology of the non-sintered surface under different magnifications. The global morphology in Fig. 1(e1) looks like clusters of stamens whose average size is around 20 μm. In a larger magnification [Fig. 1(e3)], the coralloid structure with a size of around 200 nm is given to exhibit the morphology of the fine scale structure. Under different magnifications, one can see that a fine scale structure exists in the coarse scale structure, i.e., the structure on the non-sintered surfaces is

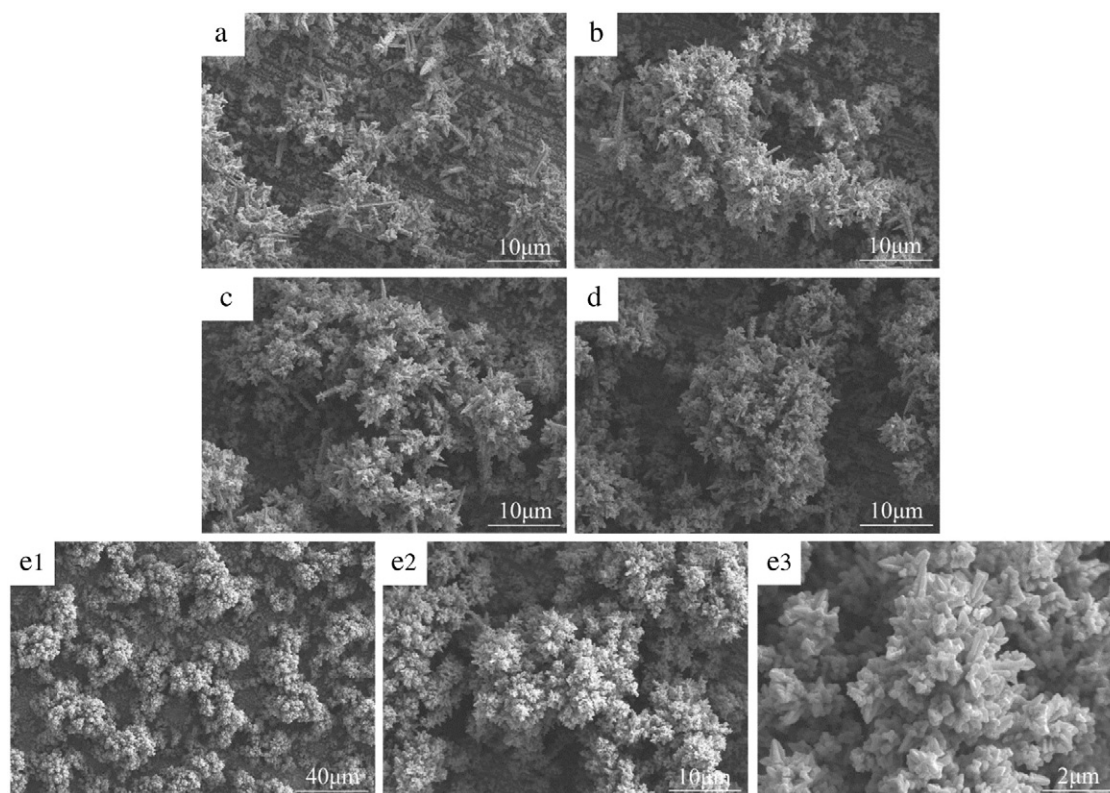


Fig. 1. SEM images of the non-sintered surfaces with different current densities and deposition times: (a) 10 mA·cm⁻², 20 min; (b) 20 mA·cm⁻², 15 min; (c) 30 mA·cm⁻², 15 min; (d) 40 mA·cm⁻², 12 min; and (e1)–(e3) 50 mA·cm⁻², 12 min.

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