



# Microstructural evolution and corrosion resistance of super-hydrophobic electrodeposited nickel films



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## ABSTRACT

Superhydrophobic coatings have become a hot research topic in recent years due to their excellent properties and wide practical applications. In the present work, hierarchical nickel films having intrinsic super-hydrophilic property were fabricated on copper substrate by two-step electrodeposition process. The surface structure and composition were characterized by means of scanning electron microscopy (SEM), X-ray diffraction pattern (XRD) and atomic force microscopy (AFM). The contact angle of water droplets on the hierarchical structure of nickel films increased over time, eventually becoming large enough to classify the surface as super-hydrophobic. The surface morphology of nickel film at micro/nano scale was characterized at different deposition current densities ( $10\text{--}70\text{ mA cm}^{-2}$ ). Results showed that the value of roughness and the size of micro/nano cones were decreased by increasing the current density. The nickel film deposited at  $20\text{ mA cm}^{-2}$  displayed the highest superhydrophobicity with water contact angle of  $155^\circ$ , which could be attributed to its pine cone-like structure. Electrochemical measurements and long-term immersion test showed that the superhydrophobic nickel films greatly enhanced the corrosion resistance of copper substrate in neutral 3.5 wt.% NaCl solution. The corrosion protection of superhydrophobic films was attributed to small area of real contact with the aggressive solution.

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## 1. Introduction

Wettability, as an important aspect of a surface chemistry, is defined by a contact angle of a droplet on a solid surface [1,2]. In recent years, superhydrophobic surface has received much attention due to their unique properties, such as anti-snow adhesion [3], corrosion protection [4] and self-cleaning [5]. It can also be employed for low flow resistance coatings in micro fluidic systems [6]. It has been found that the high roughness with low surface energy of a solid surface leads to apparent water contact angles higher than  $150^\circ$  [7,8]. In fact, the low surface energy is the major factor and the roughness just serves to enhance water contact angle. By increasing the roughness, the surface area of the solid is increased and thus the surface energy is decreased [9].

Techniques to make superhydrophobic surfaces can be simply divided into two categories: making a rough surface from a low surface energy material or modifying a rough surface with a material of low surface energy. Many methods have been developed to produce rough surfaces, including plasma etching [10], anodic oxidization [11], chemical vapor deposition [12], sol-gel method [13], phase separation [14] and electrodeposition [15]. Most of these methods involved multi-step procedures and harsh conditions, or require specialized reagents and

equipment [8,16]. In contrast, electrodeposition has been used as a one step, simple and economic method to fabricate superhydrophobic surface on different substrates [6,8].

Copper has properties such as low cost, high thermal and electrical conductivity, but it is corroded easily. So, creating a superhydrophobic surface is one of the methods used for modifying its surface [17]. Xi et al. [15] prepared a superhydrophobic surface on hydrophilic copper substrate via electroplating at large current density to produce various degrees of roughness without chemical modifications. Xu et al. [18] improved the corrosion resistance of copper by fabricating a superhydrophobic composite coating. Up to now, a few papers have been published on superhydrophobic nickel films produced on copper substrate by electrodeposition without applying low surface energy materials. These researches have usually studied the morphology and wetting behavior of the coatings. However, there is no systematic study on the effect of deposition current density on physical properties, wetting behavior and corrosion resistance of super-hydrophobic nickel films.

In the present work, fabrication of superhydrophobic nickel film with micro-nano cone array on copper substrate was done only by electrodeposition without applying any low surface energy materials. Surface morphology, super-hydrophobicity characteristics and corrosion resistance of nickel films electrodeposited at different current densities were investigated by scanning electron microscopy, water contact angle measurement and the electrochemical technique.

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**Table 1**  
Operating conditions for electrodeposition of nickel films.

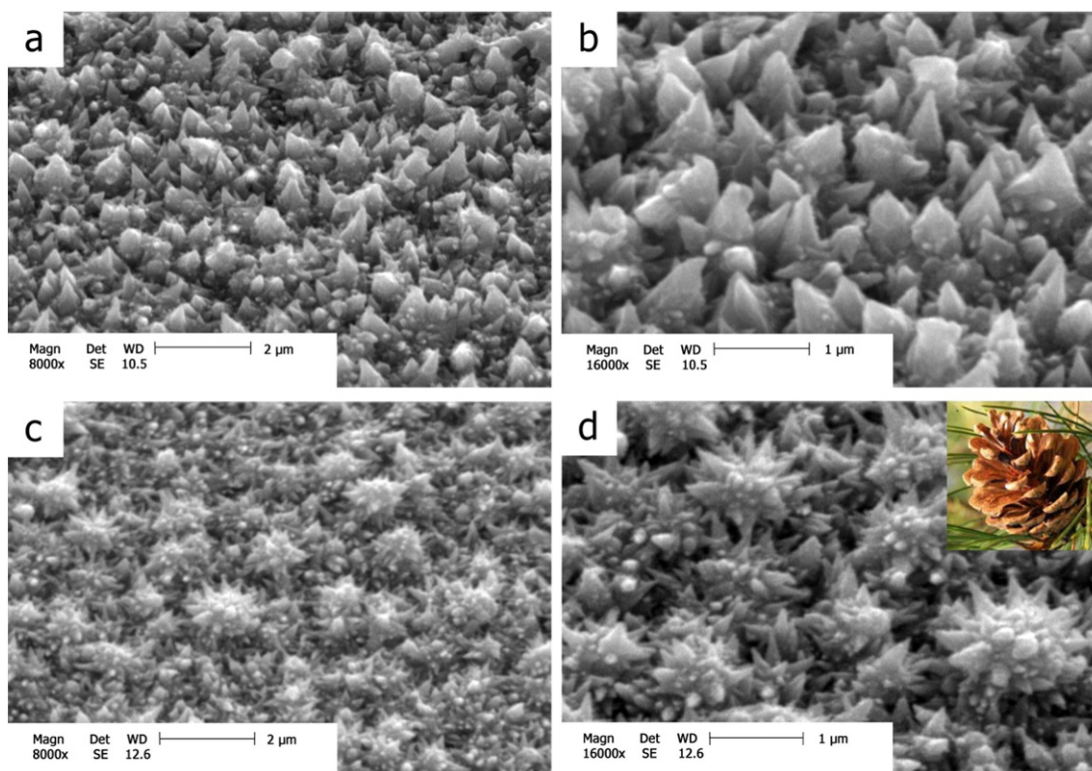
Specimen		Miro-nano nickel film			
Micro-nickel film		Deposition– step 1		Deposition– step 2	
Current density (mA cm <sup>-2</sup> )	Time (s)	Current density (mA cm <sup>-2</sup> )	Time (s)	Current density (mA cm <sup>-2</sup> )	Time (s)
10	1200	10	1200	50	60
20	600	20	600	50	60
30	400	30	400	50	60
50	240	50	240	50	60
70	171	70	171	50	60

## 2. Experimental procedure

Nickel coatings were prepared by electrodeposition on the copper substrate. Before plating, copper substrates in disk shape with a surface area of 1.53 cm<sup>2</sup> were mechanically polished down to 2400 grit size using abrasive SiC papers and then polished with 0.05 μm alumina. The specimens were cleaned ultrasonically in ethanol for 30 min, electropolished at 20 mA cm<sup>-2</sup> for 1 min in a solution containing 70 g L<sup>-1</sup> Na<sub>2</sub>CO<sub>3</sub>, 10 g L<sup>-1</sup> KOH and 10 g L<sup>-1</sup> sodium dodecyl sulfate (C<sub>12</sub>H<sub>25</sub>NaO<sub>4</sub>S) and then activated in 20% HCl for 20 s. After that, they were washed with deionized water and immediately placed in an electrodeposition bath. The bath consisted of NiCl<sub>2</sub>·6H<sub>2</sub>O (1 mol L<sup>-1</sup>) as ion source, H<sub>3</sub>BO<sub>3</sub> (0.5 mol L<sup>-1</sup>) as pH buffer and ethylenediammonium dichloride; C<sub>2</sub>H<sub>10</sub>Cl<sub>2</sub>N<sub>2</sub> (1.5 mol L<sup>-1</sup>), as a crystal modifier, was dissolved in deionized water. The solution temperature was kept constant at 60 °C and pH 4 (adjusted by NH<sub>4</sub>OH). A digital coulometer (model BHP 2050) was used to produce a micro-nano hierarchical structure. The electrodeposition process was carried out in two steps. In the first step, electroplating process was performed at different current densities

and deposition times. The optimized operating conditions including current density and time are given in Table 1. The constant current density of 50 mA cm<sup>-2</sup> was applied for 60 s in the second step of deposition. For convenience in identifying the specimens, labels FNF10, FNF20, FNF30, FNF50 and FNF70 were selected for fresh Ni films (superhydrophilic) deposited at the current densities of 10, 20, 30, 50 and 70 mA cm<sup>-2</sup> and at deposition times of 1200, 600, 400, 240 and 171 s, respectively. Labels SNF10, SNF20, SNF30, SNF50 and SNF70 were also selected for Ni films exposed in air (superhydrophobic) at their corresponding conditions. The counter electrode was a platinum wire (as the anode electrode). The electrochemical cell was connected to an EG&G (model 263A) computer-controlled potentiostat/galvanostat. An EG&G ac responder (model 1025) was coupled with the mentioned potentiostat/galvanostat to read the ac impedance data. The reference electrode was an Ag/AgCl saturated in KCl (SSE). The test solution for the corrosion investigations was 3.5 wt.% NaCl solution at room temperature. Potentiodynamic polarization curves were recorded after 30 min of immersion in the test solution. The potential range for these measurements was fixed from –250 mV, below the open circuit (OCP) value in the cathodic regime, to 500 mV, above OCP value in the anodic regime, at a scan rate of 1 mV s<sup>-1</sup>. The corrosion current density (*i*<sub>corr</sub>) for the specimens was determined by extrapolating the anodic and cathodic branches. Electrochemical impedance spectroscopy (EIS) measurements were performed from an initial frequency of 100 kHz to a final frequency of 10 mHz, using an AC sine wave with the amplitude of 10 mV. The impedance spectra for specimen produced at the current density of 20 mA cm<sup>-2</sup> were gathered after different immersion times (1, 3, 6, 9 and 15 days).

The surface morphology of Ni films was studied using a scanning electron microscopy (SEM) (model Philips XL30). The SEM images were then utilized for measuring the cones size on films surface by processing the images using Image J software. Water contact angle of the films was measured by a 4 μL distilled water droplet at ambient temperature using Theta Attension optical tensiometer (KSV instrument) with



**Fig. 1.** SEM images of nickel films deposited at current density of 20 mA cm<sup>-2</sup> at different magnifications, (a, b) micro nickel film, and (c, d) micro-nano nickel film.

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