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Effect of current density on wettability and corrosion resistance of superhydrophobic nickel coating deposited on low carbon steel

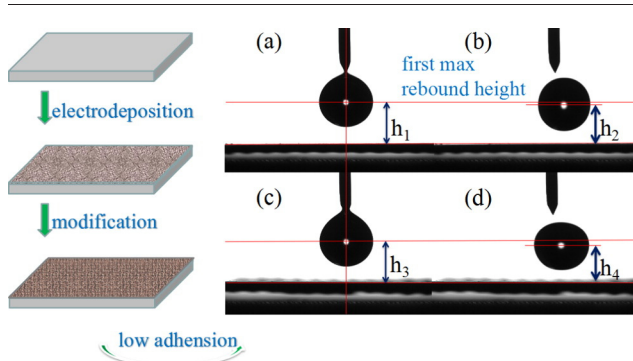
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HIGHLIGHTS

- Superhydrophobic surfaces were fabricated on low carbon steel by a facile method.
- The effect of current density on morphology and wettability of surfaces were researched.
- The superhydrophobic sample shows a well abrasive resistance.
- Equivalent circuit models were applied to illuminate the corrosion behavior of samples.

GRAPHICAL ABSTRACT



Superhydrophobic surface was successfully fabricated on low carbon steel via electrodeposition method and afterward modified by myristic acid. Samples show well abrasive resistance with interesting bouncing behaviors.

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ABSTRACT

In this study, nickel coating was electrodeposited on low carbon steel (LCS) surface. The effect of current density on surface morphology, crystal orientation and wettability of nickel coatings were comparatively studied. The abrasive resistance of coatings was carried out by self-made equipment. Moreover, the corrosion resistance of the surfaces was investigated by potentiodynamic polarization curves as well as electrochemical impedance spectroscopy (EIS) in the corrosive medium of 3.5 wt.% NaCl solution. The results showed that the surfaces present a micro-nano structure at the current density of 6 A/dm² and 8 A/dm², and exhibit superhydrophobicity after modification with myristic acid. The crystal orientation changed with the variation of current densities. It is worth noting that the surface deposited at 6 A/dm² showed excellent mechanical stability after abraded by sandpaper for 10 times with interesting bouncing behavior. Moreover, electrochemical measurements showed that the corrosion potential (E_{corr}) of superhydrophobic surface was ascended from -0.447 V to -0.186 V, and the EIS value was up to $1.04 \times 10^5 \Omega \text{ cm}^2$, indicating excellent corrosion resistance. The corrosion mechanism is illuminated via equivalent circuits.

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1. Introduction

As one of the basic materials, steel is extensively used in the world because of its abundant reserves, excellent physical, mechanical and process performance [1,2]. Especially for low carbon steel (LCS), it has been widely applied in building, transportation, machine manufacturing and so on [3,4]. Hence, both the stability and corrosion resistance

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of LCS under various conditions become the major concerns. However, steel is prone to corrosion and contamination in humid environment, which can greatly hinder its applications. Therefore, a surface coating with corrosion and contamination resistance on LCS is desirable. Inspired by lotus leaves, we can transform the LCS to be superhydrophobic to achieve the goal.

Over the past decades, superhydrophobic surface has attracted considerable interests in both academic and industrial research based on its unique properties, such as water repellency [5], anti-corrosion [6], anti-icing [7], low flow resistance [8]. In general, superhydrophobic surface can be fabricated via regulating both chemical compositions and morphological structure of its surface [9]. Nevertheless, the water contact angle (WCA) is no $>120^\circ$ on a flat surface even with an extremely low surface energy [10]. Thus, a rough structure with appropriate size is critical to superhydrophobic surface construction. Till now, large numbers of methods have been employed to construct suitable structures, such as chemical vapor deposition [11], electrochemical deposition [12], electroless replacement deposition [13], chemical etching [14], sol-gel [15], anodic oxidation [16] and laser etching [17]. Boinovich et al. fabricated superhydrophobic nanocomposite coatings on the surface of LCS with oxide layers by plasma electrolytic oxidation [18]. Li et al. created superhydrophobic stainless steel surfaces by acid treatments and hydrophobic films deposition [19]. Wu et al. created superhydrophobic surface by microstructuring of stainless steel using a femtosecond laser [20]. However, these methods are cost consuming, process complicating, and need sophisticated instruments. On the contrary, electrochemical deposition shows a lot of advantages, including low cost, simple process and easy to control. Liu et al. successfully prepared a superhydrophobic surface on AZ91D magnesium alloy by deposited a nickel coating [21]. Huang et al. reported a superhydrophobic thin films on aluminum alloy substrates by electrophoretic deposition [22]. Moreover, Tam et al. presented an excellent summary of research on superhydrophobic electrodeposits, in addition, the recently developed strategy to improve the durability during the application of superhydrophobic electrodeposits were also discussed [23]. Although the superhydrophobic surfaces have been researched for decades, there is limited investigation about the effect of current density on the wettability and mechanical stability as well as corrosion resistance of superhydrophobic nickel coating deposited on LCS.

In this study, special structures were deposited on LCS surfaces with different current densities, which show a variety of WCAs. After modification with myristic acid, the coatings show superhydrophobicity when the applied current densities are 6 A/dm^2 and 8 A/dm^2 , respectively. The surface morphology and crystal orientation were investigated by a field emission scanning electron microscope (FESEM) and X-ray diffraction (XRD). In addition, the corrosion resistance of surfaces was tested by electrochemical measurement and the results were excellent. Moreover, the surfaces exhibit excellent mechanical stability.

2. Experimental

2.1. Materials

Low-carbon steels (composition: $\text{C} \leq 0.1\text{ wt.}\%$, $\text{Mn} \leq 0.5\text{ wt.}\%$, $\text{P} \leq 0.035\text{ wt.}\%$, $\text{S} \leq 0.035\text{ wt.}\%$, with the rest being Fe) were purchased from Shanghai Guangzong Industrial Co., Ltd., China, and then cut into $50\text{ mm} \times 20\text{ mm} \times 2\text{ mm}$ as samples in this study. Sodium hydroxide (NaOH), sodium carbonate (Na_2CO_3), sodium phosphate (Na_3PO_4), hydrochloric acid (HCl), hexamethylenetetramine ($\text{C}_6\text{H}_{12}\text{N}_4$), nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$), boracic acid (H_3BO_3), potassium chloride (KCl), sodium dodecyl benzene sulfonate (SDBS) and absolute ethanol ($\text{C}_2\text{H}_5\text{OH}$) were analytical pure grade and purchased from Nanjing Chemical Reagent Co., Ltd., China. Myristic acid ($\text{CH}_3(\text{CH}_2)_{12}\text{COOH}$, 95%) was purchased from Aldrich Chemical Inc., USA. All chemical reagents were used as received without any purification, and self-made deionized water was used for preparation of all aqueous solutions.

2.2. Pretreatment of substrates

Firstly, the LCS substrates were polished with silicon carbide paper of successively finer grit down to 2000 grit, and then washed with deionized water in sequence. Secondly, the substrates were immersed in an alkaline solution containing 25 g/L NaOH , $35\text{ g/L Na}_2\text{CO}_3$ and $35\text{ g/L Na}_3\text{PO}_4$ at 80°C for 10 min to eliminate grease. And then, they were immersed into an acid solution with 125 mL/L HCl and $1\text{ g/L C}_6\text{H}_{12}\text{N}_4$ at room temperature for 2 min to remove the possible oxide layer on the surfaces. Finally, these substrates were activated in a solution of 30 mL/L HCl at room temperature for 30 s before drying in air. The substrates should be thoroughly rinsed with deionized water after each treatment step.

2.3. Fabrication of samples

The plating solution was composed of $200\text{ g/L NiCl}_2 \cdot 6\text{H}_2\text{O}$ as ions source, $30\text{ g/L H}_3\text{BO}_3$ as pH buffer, 30 g/L KCl as anode activator and 0.1 g/L SDBS as anti-pitting additive with the pH value of 4.5–5.0. The pretreated LCS substrate was taken as the cathode while a pure nickel plate was used as the anode with magnetic stirring in a water bath kettle at 65°C . The distance between the cathode and anode is 3 cm. To investigate the effect of current density on plating property, the substrates were electrodeposited with different current densities of 2 A/dm^2 , 4 A/dm^2 , 6 A/dm^2 , 8 A/dm^2 by a direct current power supply for 10 min and then dried in air after washed by deionized water, the current efficiency was measured by copper coulometer with results of 94.6%, 92.2%, 89.7% and 84.5% respectively [24,25]. Continually, the samples were immersed into 0.1 mol/L ethanol solution of myristic acid containing at room temperature for 1 h. At last, the samples were dried at 60°C for 2 h in the oven. To well identify these samples, the pure nickel coatings deposited at the current densities of 2 A/dm^2 , 4 A/dm^2 , 6 A/dm^2 , 8 A/dm^2 were labeled as PNC2, PNC4, PNC6 and PNC8, and after modified by myristic acid they were labeled as MNC2, MNC4, MNC6 and MNC8, respectively.

2.4. Measurements and characterization

To measure the WCAs, a contact angle measuring system (DSA100, Kruss Corporation, Germany) was used at room temperature. The volume of the water droplets is $4\text{ }\mu\text{L}$. All of the WCAs were measured at five different points and then averaged. The surface morphologies of these samples were obtained by a field emission scanning electron microscope (FESEM, LEO1550, ZEISS, Germany). The phase identification and growth direction of the Ni coating were detected by X-ray diffraction (XRD, D8 Advance, Bruker, Germany) using filtered $\text{Cu K}\alpha$ as a radiation source at a scanning rate of $5^\circ/\text{min}$ from 10° to 80° of 2θ . The corrosion resistance were measured by potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS) in a $3.5\text{ wt.}\%$ NaCl solution ($\text{pH} = 5.9$) at room temperature via an electrochemical workstation (CHI 750C, Shanghai Chenhua Instrument Corporation, China). Measurements were carried out in a three-electrode cell with a saturated calomel electrode (SCE) as the reference electrode, a platinum electrode as the counter electrode, and the sample with an explore area of $1\text{ cm} \times 1\text{ cm}$ as the work electrode. Prior to the electrochemical measurements, the sample was immersed in NaCl solution until it was able to obtain a stable open circuit potential (OCP). The potentiodynamic polarization curves were measured at a scan rate of 5 mV/s , and the EIS curves were performed by applying a sinusoidal perturbation signal with 5 mV amplitude at open circuit potential by a frequency range from 10^{-2} Hz to 10^5 Hz .

3. Results and discussion

3.1. Surface morphology

The surface morphology of samples deposited at different current densities is shown in Fig. 1. Liang et al. [26] have reported that at low

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