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## A study on the fabrication of superhydrophobic iron surfaces by chemical etching and galvanic replacement methods and their anti-icing properties

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#### ABSTRACT

Hierarchical structures on iron surfaces were constructed by means of chemical etching by hydrochloric acid (HCl) solution or the galvanic replacement by silver nitrate (AgNO<sub>3</sub>) solution. The superhydrophobic iron surfaces were successfully prepared by subsequent hydrophobic modification with stearic acid. The superhydrophobic iron surfaces were characterized by Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS) and water contact angle (WCA). The effects of reactive concentration and time on the microstructure and the wetting behavior were investigated. In addition, the anti-icing properties of the superhydrophobic iron surfaces were also studied. The FTIR study showed that the stearic acid was chemically bonded onto the iron surface. With the HCl concentration increase from 4 mol/L to 8 mol/L, the iron surface became rougher with a WCA ranging from 127° to 152°. The AgNO<sub>3</sub> concentration had little effect on the wetting behavior, but a high AgNO<sub>3</sub> concentration caused Ag particle aggregates to transform from flower-like formations into dendritic crystals, owing to the preferential growth direction of the Ag particles. Compared with the etching method, the galvanic replacement method on the iron surface more favorably created roughness required for achieving superhydrophobicity. The superhydrophobic iron surface showed excellent anti-icing properties in comparison with the untreated iron. The icing time of water droplets on the superhydrophobic surface was delayed to 500 s, which was longer than that of 295 s for untreated iron. Meanwhile, the superhydrophobic iron surface maintained superhydrophobicity after 10 icing and de-icing cycles in cold conditions.

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#### 1. Introduction

Recently, superhydrophobic surfaces (SHS), imitated from lotus leaf, have drawn much attention due to their excellent water repellency. Water droplets cannot wet the SHS, and the surfaces show large water contact angles (WCAs) of higher than 150° [1–4]. In addition, water droplets easily slide from the SHS while removing dirt, endowing the surface with self-cleaning and other amazing properties, such as those for anti-corrosion [5], anti-icing [6,7] and water/oil separation [8–10]. Current research has developed fabrication methods for SHS based on etching [11], electrospinning [12], phase separation [13], chemical deposition [14], sol–gel processing [15] and template use [16].

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Metals and their alloys are important for human lives and have been widely used in the automotive industry, construction, household appliances, electrical power generation and machinery manufacturing [17,18]. However, the surfaces of metal products are easy to wet and cover with a layer of water film, leading to serious problems including corrosion and icing [19,20]. Therefore, it is wise to design metal products with superhydrophobicity by adjusting the surface chemical compositions and structures [21]. Etching and chemical deposition methods are thought of as promising approaches for the large-area fabrication of SHS on metal products due to their advantages of ease of use, low cost and timesaving characteristics [22,23]. Zhang et al. [24] adopted a simple two-step etching process to prepare superhydrophobic aluminum (Al) with good friction-reducing performance. The textured surface appeared on the Al substrate after immersion in a sodium hydroxide (NaOH) solution at 100 °C for 1 h, and the subsequent deposition of a stearic acid film endowed the surface with high hydrophobicity. Fan et al. [25] fabricated a superhydrophobic film onto the surface







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of a copper wafer by etching and chemical deposition methods. They found that the obtained copper possessed good anti-corrosive properties to a NaOH solution because of the excellent water repellency. Al substrates with SHS also exhibited remarkable anti-icing characteristics, and the icing time could be delayed in comparison with untreated Al [26]. Although many attempts have been made to fabricate SHS on Al, Cu and Mg alloys, less attention has been paid to iron products, and some fundamental research is still required [27].

Ice adhesion and accumulation on metal products may cause traffic accidents, economic losses and threaten human safety [28]. For metal products with a low specific heat capacity, icing problems are more serious than for other materials. It is well known that icing occurs when the water on solid surfaces condenses into ice under 0°C, and the accumulation of ice continues on the iced surface. When this occurs, the water repellent property is particularly important for anti-icing, and icing problems may be inhibited with SHS [29–31]. Cao et al. [32] showed that superhydrophobic surfaces prevented ice formation, and the anti-icing properties depended on the wetting behavior and the microstructure of the surface. However, the superhydrophobic surface showed poor mechanical durability during icing/deicing cycles [33,34]. Kulinich et al. [35,36] found that the ice-repellent properties of superhydrophobic materials deteriorated during icing/deicing cycles due to the gradual damage of the surface asperities, and water was condensed more easily between the surface asperities in a humid atmosphere, leading to high ice adhesion strength. Therefore, further study is needed regarding the anti-icing properties of superhydrophobic materials and how water icing and ice formation on the superhydrophobic surface occurs. The mechanical durability against icing/de-icing should also be examined.

In this study, the irons were chemically etched by an HCl solution or galvanically replaced by an AgNO<sub>3</sub> solution, then the superhydrophobic iron surfaces were fabricated after modification with stearic acid. The superhydrophobic iron surfaces were characterized by Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS) and water contact angle (WCA). The effects of reactive concentration and time on the microstructure and wetting behavior were investigated. Finally, the anti-icing properties of the superhydrophobic iron surfaces were also studied.

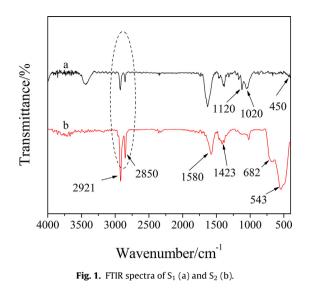
#### 2. Experimental

#### 2.1. Materials

Hydrochloric acid (HCl), silver nitrate (AgNO<sub>3</sub>) and ethanol were obtained from the Guangzhou Chemical Reagent Factory (China). Sandpaper (500<sup>#</sup>) was got from the Guangzhou Qianhui Bose Instrument Company (China). Stearic acid was supplied by Guangzhou Longsun Technology Company Ltd. (China). Iron plate was acquired from Guangzhou Pacific Tinplate Company Ltd. (China). All of the reagents were used as-received.

#### 2.2. Fabrication of superhydrophobic iron surface

First, the iron substrate was mechanically polished by  $500^{\#}$  sandpaper until the surface became mirror flashed, then it was ultrasonically washed in an ethanol-water solution for 15 min. After that, the iron substrate was immersed into the HCl (or AgNO<sub>3</sub>) solution for a predetermined amount of time. The iron substrate was rinsed with distilled water and immersed into an ethanol solution containing 0.01 mol/L stearic acid at 40 °C for 30 min. Finally, the resultant iron was washed by distilled water and transferred to an oven at 80 °C for 30 min. The superhydrophobic



iron surfaces fabricated by chemical etching of the HCl solution and the galvanic replacement of the  $AgNO_3$  solution were labeled as  $S_1$  and  $S_2$ , respectively.

#### 2.3. Anti-icing property

A water droplet of approximately 20  $\mu L$  was placed on the iron surface, and the sample was transferred to a closed refrigerator at  $-10\,^\circ C$  to  $-15\,^\circ C$ , then the icing time of the water droplet was measured.

The superhydrophobic iron was also put into a closed refrigerator at -10 °C to -15 °C for a predetermined amount of time, and then it was moved into the ambient air until the ice was completely melted and the surface retained dry. The WCA of the superhydrophobic iron surface was measured and the entire operation was repeated 10 times.

The icing behavior on the iron surface was studied by placing the iron substrate into a closed refrigerator at -10 °C to -15 °C with a humidity of approximately 80–90%, and digital photos of the iron surface were taken after 8 h, 24 h and 48 h.

#### 2.4. Characterization

The Fourier transform infrared spectroscopy (FTIR) spectra were recorded on a Bruker Tensor 27 spectrometer (Bruker Optics, Germany) at a range from 4000 cm<sup>-1</sup> to 400 cm<sup>-1</sup> using KBr pellet technique. Samples were obtained by scraping the iron surface and collecting the residual powders. The microstructures were observed by a field-emission scanning electron microscope (SEM, Fei Nova Nanosem 430, Netherlands) equipped with an energy dispersive X-ray spectroscopy (EDS) unit under an acceleration voltage of 15 kV. A thin chrome layer was sprayed onto the sample surface prior to testing. The water contact angle (WCA) values were measured at room temperature by a contact angle detector (DSA100, Germany) equipped with a digital camera using 5  $\mu$ L distilled water as an indicator. All of the sample measurements were taken from at least five different locations to obtain the mean values.

#### 3. Results and discussion

#### 3.1. FTIR

Fig. 1 shows the FTIR spectra of  $S_1$  (a) and  $S_2$  (b). In spectra (a) and (b), after stearic acid modification, the absorptions at 2921 cm<sup>-1</sup>

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