Fabrication of super-hydrophobic surface on aluminium substrate and a study of surface frosting behaviours

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\textbf{Abstract.} Frosting is common found natural phenomenon in late autumn or early spring, which sometimes brings unwanted effects or even disaster to human. With the development of science and technology, frosting may to some extend be mitigated by hydrophobic surfaces. Micro-Nanostructure was gained via chemical etching on aluminium surface. The surface was further modified by low surface energy polymer coating to form a very thin nano-layer on the surface. A kind of fluoroalkylsilane is used as the coating polymer. After fluorination treating, the aluminium surface become super hydrophobic with water contact angles more than 150°. The surface was observed to be configured with plateaus and caves of micro-Nanoscale. In subcooled environments, frosting experiment was carried out on the surface of aluminium rods and aluminium sheets using humidifier to supply the water droplets. The comparison of the quantity of frost formed on the super-hydrophobic and untreated aluminium surfaces was conducted in the present work. And the quantity of frost formed on aluminium surfaces of different contact angles was compared. The results show that super-hydrophobic aluminium surfaces have obvious advantages in inhibiting frost. Besides, the contact angle has evident influences on restraining frost. Therefore, super-hydrophobic surfaces may serve as an effective means in decreasing the accumulation of frost on outside solid surfaces.

1. Introduction
Wettability is one of fundamental properties of solid surface. Surfaces with strong water repellency were studied with great effort in the past two decades, such as the self-clean lotus leaves [1], the legs of water striders which walk and jump freely on water [2]. Researchers found hydrophobic surfaces may find great perspective utilizations in many fields [3-6]. Frosting is common found natural phenomena in late autumn or early spring, which sometimes bring unwanted effects or even disaster to human. Frost is commonly formed on home refrigerators resulting in further consideration for the convenience uses. Industry heat exchangers for cooling also face with the problem of frost layer on the cooling surface [7-9], which significantly decreases the heat transfer efficiency [10,11]. With the development of science and technology, frosting may to some extend be mitigated by hydrophobic surfaces owing to the strong water repellency of the surface.

In recent years, many works were done on the fabrication on water repellent surfaces [12-15] among which the method of chemical etching followed by polymer coating may be the feasible means for industry application. Ice inhibition was studied on hydrophobic surfaces [15-18] and ice forming process was analyzed [19,20]. Relatively few work was done on the mitigation of frost on hydrophobic surfaces [21,22]. Since the process of ice formation and frost formation are complex, further studies are necessary for the development for ice or frost resistant surfaces.
In present work, super hydrophobic surfaces (SHS) with micro-nano hierarchical roughness were fabricated via the two steps of chemical etching and organic polymer coating. The hydrophobicity of the surface was examined by means of water contact angle (WCA) measurement. Frosting experiments were carried out to study the anti-frosting effect of SHS.

2. Preparation of SHSs on aluminium substrate

The preparation of SHSs in the present work includes two procedures: chemical etching and surface fluorination.

2.1. Chemical etching

Hydrochloric acid is adopted as the etchant. Specimen cuts were immersed in hydrochloric acid of different concentrations from 1 to 5 M. Etching time was changed from 4 to 70 min. The specimens were immediately rinsed ultrasonically with water after etching, and dried at 50°C in air. A kind of hierarchical roughness of micro-nano scale was formed on the surface of the samples as shown in figure 1. In large micrometer caves stand nano-sized plateaus.

2.2. Surface coating

Naked aluminum surface is hydrophilic. When etched properly, it becomes super hydrophilic. The surface can be turned super hydrophobic further by low energy material coating. In present work, 1% (wt) tridecafluorocetyltriethoxysilane (FAS) ethanol solution was used as the coating agent. The etched aluminium substrates were put into the FAS solution for 1 hour, and then kept at 130°C heat treatment for about an hour. A thin film with low surface energy of was formed via the following chemical reaction:

2.3. Characterizations

The wettability of a solid surface is often characterized by water contact angle measurement. In the present study, WCA were measured with a CA meter. The Equilibrium contact angle values are averages.
of 5 measurements made on different sites of the surfaces. After fluorination, the surface exhibit super hydrophobicity with WCA up to 158°. Water droplet on the surface looks as a perfect sphere as shown in figure 2.

![Image](image1.jpg)

(a)  

![Image](image2.jpg)

(b)  

**Figure 2.** The image of a water droplet on SHS. (a) photograph and (b) WCA measurement image with WCA about 158°.

3. Frosting experiment

If the surface temperature of a solid material is below both the dew point of humid air and the freezing point of water, water vapour in humid air will form a frost layer on the surface. Frosting experiments were conducted in a closed cabinet in which temperature can be automatically controlled and kept constant with a refrigeration system. An ultrasonic humidifier was used to generate fog in the air. For comparison, untreated and super hydrophobic samples of aluminium in the shape of rods and sheets were tested for frosting behaviours which are depicted both visually and by means of mass increases of the tested samples. Figure 3 shows a typical visual comparison of the frost formed on an untreated aluminium surface and on an aluminium SHS.

![Image](image3.jpg)

(a)  

![Image](image4.jpg)

(b)  

**Figure 3.** visual comparison of the frost formed on an untreated aluminium surface and on an aluminium SHS (subcooling: 25 K, time: 30 min). (a) untreated aluminium surface and (b) aluminium SHS.

It can be seen from figure 3 that the frost on SHS is obviously less than on untreated surface. It can retard the starting time of frosting evidently. For longer frosting time, frost can also cover the whole area of SHS, being no sharp visual difference from that of untreated surface, but the amounts of frost (in grams) formed on the two kinds of surface are different as shown in figure 4.

The tested specimens can be etched under different conditions, say, different etchant concentrations and different etching time, resulting in different water repellence with different WCAs. The influence of WCA on frost formation is shown in figure 5, illustrating the frost mitigation effect of the hydrophobic surfaces.
4. Conclusion
Micro-Nanostructure was gained via chemical etching on aluminium surface. With fluorine treatment, the surface shows ultra-hydrophobic properties with a WCA of more than 150°. In the subcooled environments, frosting experiment was carried out on the surface of aluminium rods and aluminium sheets. The results show that aluminium SHSSs have obvious advantages in inhibiting frost. Therefore, SHSSs may serve as an effective means in decreasing the accumulation of frost on outside solid surfaces.

Acknowledgment
The authors are grateful for the financial support from the National Natural Science Foundation of China (grant No.: 20476014, 51376030).

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