International Journal of Heat and Mass Transfer 126 (2018) 442–451

Contents lists available at ScienceDirect

International Journal of Heat and Mass Transfer

journal homepage: www.elsevier.com/locate/ijhmt

Freezing delay, frost accumulation and droplets condensation properties of micro- or hierarchically-structured silicon surfaces

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ARTICLE INFO

Article history:
Received 11 January 2018
Received in revised form 27 April 2018
Accepted 29 April 2018

Keywords:
Micro-pillared
Hierarchically-structured
Freezing-delay
Frost thickness
ESEM

ABSTRACT

The processes of single droplet freezing, frost accumulation and droplets flooding during condensation were analyzed and compared for surfaces of different morphologies. Four samples were first fabricated – a micro-cubes silicon, a micro-cylinders silicon, a hierarchically-structured silicon and a smooth silicon. After measuring the static contact angles and roll angles, the sessile water droplet freezing and frost accumulation under a series of surface temperature were studied. The droplets condensation processes were for the first time compared through environmental scanning electron microscopy (ESEM). The results showed that the droplet freezing time on hierarchically-structured silicon was 4 times longer than that of uncoated silicon. Micro-pillared and hierarchically-structured silicons could minimize the frost thickness only in the initial stage of frost formation and had quite different frost growth patterns. The ESEM images can facilitate explain the different patterns of freezing and frosting on micro-pillared and hierarchically-structured samples.

1. Introduction

In nature, water freezing or frost formation on cold surfaces is ubiquitous and poses negative impact to many applications, such as the aerospace, railway, telecommunication and power engineering industries [1,2]. Therefore, the issue of avoiding or minimizing frost accumulation has arosed great attention. So far, the applied measures for the mitigation of frost formation can be divided into two categories: active and passive methods. The active method including substrate heating and using de-icing chemical agents, is both cost-intensive and environmentally problematic [3]. However, the passive method can effectively remove ice from the surfaces, in which there is less energy consumption because of the lower ice adhesion strength. Recently, the use of hydrophobic surfaces, one of passive methods, has been widely investigated in anti-icing/frosting applications.

Many papers about the sessile droplet freezing [4–8] and frost depostion [9–11] processes of hydrophobic surfaces were discussed. The results showed that hydrophobic surfaces may delay droplets freezing time, prevent frost accumulation in the initial stage of frosting process, reduce ice adhesion strength and make defrosting easier. Despite hydrophobic surfaces being identified by many researchers potential candidates for anti-icing surfaces, contradictory findings were being reported that a hydrophobic surface is not always icephobic [12–15]. They demonstrated that wettability is not the sole parameter determining the anti-icing property of a solid surface. Instead, it is more rational to consider the effects of both wetting behaviors and surface topography when designing an icephobic surface. In anti-icing fields, fabrication of micro-/nanostructures or hierarchical structures and modification of surface energy level by chemical substance are two major methods used to design an ice-repellent surface. In the past, a lot of studies have focused on effects of various coatings with low surface energy on anti-icing property [16–18]. However, how micro-structures surface without any modification influence the droplet freezing or frosting process is seldom disscussed. Moreover, the existing researches almost focus on the micro-cubes surfaces when investigating the microstructures effect on anti-icing. There is a lack of comparison of droplets freezing and frosting processes on different micro-pillars or hierarchical structures.

Recently, Tan [19] has concluded the process of droplet condensation on micro-pillared hydrophobic surface into three main stages, (i) drops nucleated at the bottom of the substrate, (ii) tiny drops grew larger and merged, (iii) the Wenzel droplets were developed with metastable state [20–22]. For superhydrophobic surfaces, the condensation droplets would grow to a Cassie state droplet when micro-structure arrays on the substrate were small [23,24] or the surface was with multi-tier structure [25]. In addition to the condensation process, Oberli et al. [5] also studied the

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https://doi.org/10.1016/j.ijheatmasstransfer.2018.04.165
0017-9310/

freezing mechanism of three different micro-pillared surfaces. They found that the pillars dimension and distribution can greatly affect the wetting state, the coalescence pattern of condensed water micro-droplets and the formation of dendritic ice crystals. The dendritic ice crystals are responsible for the spreading of a frost front [26], thereby affecting the freezing delay. Therefore, the structure itself and its density can affect the freezing of droplets. Further investigations should be focused more on dynamic condensation and freezing of water on different surface structures, not just on improving the contact angles studies. ESEM [27,28], as an advanced SEM system, can help provide clear images of detailed dynamic motions of droplet condensation on micro-structures. However, the few existing droplets condensation/frosting researches via ESEM only took one micro-structure surface [29–31] or micro-cubes of different parameters [32] as the target. Until now, there is no literature that provides a clear comparison of how droplets perform on different micro-structures through ESEM during condensation and freezing.

In this study, a micro-cubes silicon, a micro-cylinders silicon and a hierarchical-structured silicon were fabricated. A polished silicon wafer was used as a reference. The dymamic processes of single droplet freezing, frost formation under a series of surface temperature, and droplets condensation were studied. The freezing processes were mainly characterized by the droplet profile and the freezing-delay time. The frosting-delay, frost growth pattern and frost thickness were discussed. The droplets condensation process were recorded through ESEM.

2. Experimental details

2.1. Fabrication of textured samples

Three kinds of samples including micro-pillared or hierarchical structures were fabricated. Sample 1 and Sample 2 with micro-pillar arrays were fabricated via reactive ion etching technology, the micro-pillars structures of which are cube and cylinder respectively. Fabricating a periodical micro cubes or cylinders silicon with certain parameters, the width and space could be controlled by photolithography mask design, and the height could be controlled through adjusting etching/passivation circle time. Sample 3 is a new type of hierarchical structures named two-stage black silicon which has been introduced in the previous study [33]. The fabrication of two-stage black silicon mainly contained two steps: (1) fabricate the micro cylinders silicon surface using reactive ion etching method; (2) fabricate black silicon particles on the microstructure. Micro-pillared silicon was cleaned and etched using Inductively Coupled Plasma technique, whose structures were controlled by adjusting the passivation-to-etching ratio. Therefore, sample 3 consists of periodical micro-cylinders and aperiodic black silicon particles distributed on or between the micro-cylinders. Fig. 1 shows the schematic diagram of two-stage black silicon (Sample 3). Sample 4 was a smooth silicon wafer. All samples have the same size of 10 mm × 10 mm × 0.5 mm. (The specific fabrication process was provided by Suzhou Promisense Electronic Technology Co. Ltd. in China.)

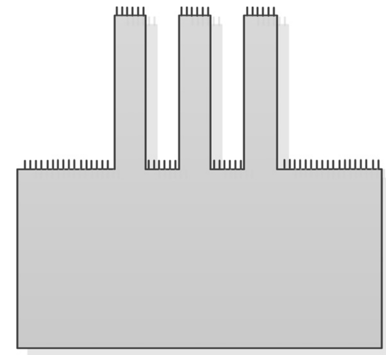

Fig. 1. Schematic diagram of two-stage black silicon (Sample 3).

2.2. Sample characterization

The surface morphologies were observed using a scan electron microscopic system (SEM, JSM-6390, Japan). SEM micrographs at different magnifications of samples were examined in the micro-scale uniformity of the sample surfaces. With the use of the video-based contact angle meter (XG-CAMC) and the angle adjustment platform (Kruss, DSA100), the water contact angle (CA) and rolling angle (RA) were measured separately. The CAs were measured on three different surface positions for each sample by dispensing 10 μL droplets of de-ionized water with 99.99% purity. To measure the RAs, 10 μL droplets were deposited onto the sample. After the initial droplet shape was equilibrated, manipulation with the precise angle adjustment platform allowed us to change the surface tilt in a controllable manner and detect the RA by averaging over three times measurements on each surface. Both static contact angles and dynamic rolling angles were measured at an ambient temperature of 23 ± 1 °C and relative humidity 60 ± 2%

Fig. 2 shows the typical SEM images of samples 1–3. Table 1 gives the geometric parameters and wettability of all test samples. As seen in Fig. 2 (a,d) and (b,e), both the cuboid and cylindrical micro-pillars are complete and well-ordered. Table 1 shows that sample 1 and sample 2 have the same structure parameters with 24 μm pillar width (a, pillar width refers to the diameter for cylinders), 30 μm pillar edge spacing (b) and 50 μm pillar height (h). Sample 3 is a two-stage silicon. Its first-stage structures are micro-cylinders (see Fig. 2(c,f)) whose parameters are same with sample 2 (see Table 1). Howerer, sample 3 has the second stage structures composed of black silicon particles that distributed on and between the first-stage micro-pillars. The shape of the black silicon is like the tiny branch. Its average height is between 5 and 10 μm, behaving a smaller scale compared with the parameters of cylindrical micro-pillars. The combination of smaller black silicon particles and micro-pillars can act as a similar micro-nano strutcure which may behave a better hydrophobicity and freezing-delay characteristics discussed in the previous researches [34–37]. Therefore, sample 1 and sample 2 have different shapes of micro-pillars with same parameters. Sample 3 has same micro-pillars and parameters with sample 2 but adds to a second-stage structure.

CA and RA are the typical parameters used to characterize the wetting behavior of a solid surface. As seen in Table 1, the CA of smooth silicon surface (sample 4) was 75.6 ± 0.3°, showing apparent hydrophilicity. It changed to 139.9 ± 0.6° (sample 1) and 142.9 ± 0.6° (sample 2) after fabricating micro-pillars on the surfaces. Sample 3 had the largest CA (146.4 ± 0.7°) of all samples. Moreover, RAs of samples 1–3 were 19.8 ± 0.1°, 23.4 ± 0.9°and 8.9 ± 1.2° respectively. It proved that water on the micro-cubes surface had a better rolling property than micro-cylinders. Hierarchical structures greatly changed the dynamic wetting characteristic of micro-pillars. Since a better hydrophobic surface refers to one with a larger CA and smaller RA, therefore, in this study, sample 3 had the best water-repellency property.

2.3. Experimental apparatus

As seen in Fig. 3, the experimental system mainly contains a CCD camera (Guppy PRO F-032B/C), a thermostaic bath, cold base,