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Stochastic approach to the anti-freezing behaviors of superhydrophobic surfaces

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ABSTRACT

The anti-freezing (or freezing delay) characteristics of sessile water droplets placed on bare and superhydrophobic surfaces were experimentally investigated. The change of the droplet temperature during the cooling and freezing processes was monitored under a constant surface temperature. The freezing delay time was calculated for a statistically large ensemble of droplets, which showed a random distribution. The minimum and maximum freezing delay times of droplets on the bare and superhydrophobic surfaces were analyzed by using a cumulative distribution function. The maximum freezing delay time of the superhydrophobic surfaces was more than three times longer than that of the bare surfaces. Moreover, about 7% of the water droplets on the superhydrophobic surface could be frozen earlier than or similarly with those on the bare surface.

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1. Introduction

Delaying the freezing of water droplets has significant impacts on many important industrial applications. For example, icing (or frost formation) on the heat exchanger of air conditioning systems degrades the thermal performance of the exchanger [1,2] and the energy efficiency of the systems [3,4], and icing on aircraft wings decreases the aerodynamic efficiency [5,6]. Icing issues are not only limited to those applications, but also detrimental to several other industries, including automobiles, buildings, and wind power plants.

Accordingly, many efforts have been driven to investigate novel methods of delaying or preventing ice formation. Among various anti-freezing techniques, superhydrophobic coatings are regarded as an effective and viable approach for retarding the freezing of water droplets without using external power sources [7,8]. The freezing delay characteristics of superhydrophobic surfaces can be rationalized using classical nucleation theory [9,10]. A Gibbs free energy barrier (i.e., nucleation barrier) is required to initiate the phase transition from liquid water to ice. The Gibbs energy barrier is higher for water droplets suspended in air because, in this case, there are no preferential nucleation sites (homogeneous nucleation). On the contrary, water droplets placed on a surface typically exhibit a lower Gibbs energy barrier, compared to suspended drops, and this barrier increases as the static contact angle

increases (heterogeneous nucleation). Consequently, the surfaces with relatively high static contact angles like superhydrophobic surfaces can be used to suppress the phase change of water droplets due to their larger Gibbs energy barrier. Many experimental demonstrations have supported the anti-freezing characteristics of superhydrophobic surfaces. Kim et al. [11] analyzed the early-stage of frosting process based on measuring the reflectivity of test surfaces, which showed that the seed growth period increased as the static contact angle of water droplets on the surfaces increased. Huang et al. [12] and Liu et al. [13] carried out the frosting experiments with on the bare and the superhydrophobic copper surfaces, and revealed that the freezing of droplets on the superhydrophobic surface was retarded, compared to a bare surface, based on in-situ observation results.

The anti-freezing properties of superhydrophobic surfaces have been previously characterized by measuring the freezing delay time. Huang et al. [14] and Tourkine et al. [15] measured the freezing delay time by observing the transparency of water droplets. Zhang et al. [16] analyzed the change of the interface between supercooled water and ice phases using image processing, thereby measuring the freezing delay time. Furthermore, Heydari et al. [17] measured the temperature of water droplet to calculate the freezing delay time. However, the rigorous comparison of the results between the studies is difficult. It is not only due to the differently defined freezing delay time in those studies, but also because of the reported freezing delay times representing the arithmetic means of several repeated measurements. In this study, we suggest the method to characterize the freezing delay time based on

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Nomenclature

f	function
ΔG^*	Gibbs energy barrier, erg
n	number
N	total number
ΔS	entropy of fusion, $\text{erg}\cdot\text{cm}^{-3}\cdot\text{C}^{-1}$
T	temperature, $^{\circ}\text{C}$
ΔT	temperature difference, $^{\circ}\text{C}$
t	time, s

Subscripts

<i>drop</i>	droplet
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<i>het</i>	heterogeneous
<i>m</i>	melting
<i>s</i>	static

Greek symbols

Φ	cumulative distribution function
θ	contact angle, deg
σ	surface energy, $\text{erg}\cdot\text{cm}^{-1}$

thermodynamic concepts, representing the stochastic modeling of the random distribution of freezing events. The freezing delay times of bare and superhydrophobic surfaces are described using the newly developed method, which enables the rigorous comparison of the freezing delay times according to the surface properties.

2. Experiments

2.1. Experimental setup

Fig. 1a and b show representative optical images of sessile water droplets (a volume of $\sim 10 \mu\text{l}$) placed on bare and superhydrophobic surfaces at an ambient air temperature of 25°C . Static droplet contact angles were obtained by analyzing the droplet profile via axisymmetric drop shape analysis (solid blue curves in Fig. 1) [18]. The static contact angles on bare and superhydrophobic surfaces were $75 \pm 1^{\circ}$ and $153 \pm 2^{\circ}$, respectively. The bare surface was composed of aluminum 6061 (Alcoa), and the superhydrophobic surface was obtained by etching the bare aluminum 6061 surface. This process consisted of three steps. Initially, the aluminum oxide film was dissolved by immersion in the 1 M sodium hydroxide solution (Daejung Chemicals and

Metals). Subsequently, the surface was etched by dipping it into the 33.3 vol% hydrochloric acid solution (Daejung Chemicals and Metals). The etched aluminum was coated with 2H-perfluorodecyl trichlorosilane (Alfa Aesar), which imparted durable superhydrophobic characteristics to the surface.

Fig. 1c and d show the scanning electron microscopy (SEM) images of bare and superhydrophobic surfaces. Compared to the smooth surface of the bare aluminum sample, the nano- and micro-scale structures were formed on the superhydrophobic surface.

Fig. 2 schematically describes our experimental setup. The cooling apparatus consisted of a thermoelectric element (TEC). A heat sink and fan were attached at the bottom of the TEC, and an aluminum plate was placed on the TEC to ensure that the temperature distribution was uniform. A type-T thermocouple (Omega, TT-T-36-SLE) was embedded in the aluminum plate and used to measure the surface temperature. The constant surface temperature was controlled by using a proportional integral derivative (PID) controller (Temcoline, T34) and a power supply (DHECN, LS-150B-12). The aluminum plate and test surface (i.e., bare or superhydrophobic surface) were grounded in order to minimize the effects of static surface charges. A camera (Nikon, D880e with Micro ED 105 mm lens) mounted beside the cooling stage was used for visual capture of the droplet freezing process.

The surfaces were cooled down to $-10 \pm 0.2^{\circ}\text{C}$ starting from an ambient temperature (25°C) at a constant rate of $10^{\circ}\text{C}/\text{min}$. In each measurement, four sessile water droplets (a volume of $\sim 50 \mu\text{l}$) were dropped and frozen on the test surface. This process was repeated 25 times, yielding a total of 100 freezing delay data points for each test surface. The droplet temperature was measured by immersing a small, type-T thermocouple (36 gauge) into the sessile water droplet. During the course of experiments, the temperature and relative humidity of the ambient air were held constant at $25 \pm 1^{\circ}\text{C}$ and $30 \pm 3\%$, respectively, without forced air flow around the test surface.

2.2. Experimental procedure

The TEC and data acquisition board were operated after the four water droplets were placed on the test surface with thermocouples immersed in each of the droplet. Once all the droplets were frozen, all the devices were turned off. Then the frozen droplets and the condensates were thoroughly removed by using a hot air blower with the setting of the air temperature as 50°C . Then, the test surface was cooled by the ambient air until the temperature of the test surface reached to $25 \pm 1^{\circ}\text{C}$. Accordingly, the identical procedure was repeated for 25 times on each surface.

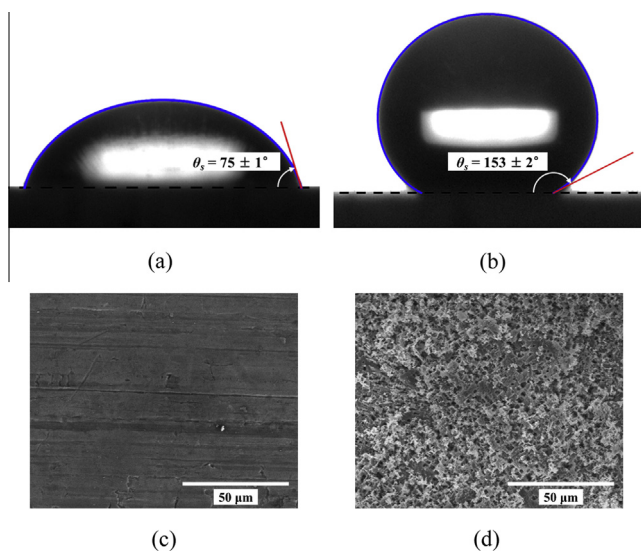


Fig. 1. Static contact angles of sessile water droplets (volume $\sim 10 \mu\text{l}$) on (a) bare and (b) superhydrophobic surfaces, and scanning electron microscopy (SEM) images of (c) bare and (d) superhydrophobic surfaces. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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