Relationships between surface wettability, roughness parameters and ice nucleation time

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Abstract— **The design of icephobic surfaces is of great importance as ice accretion on the surface can lead irreversible losses and terrible economic compensations. In this work, we conducted some experimental tests using five different non-ordered, microstructured silicone rubber surfaces having various wettability properties (hydrophobic to superhydrophobic) and roughness parameters (i.e., arithmetic average, root mean squared, ten-point height, maximum height of the profile, and skewness) to evaluate how surface features, including surface wettability, surface roughness parameters, surface temperature and droplet volume affect the ice nucleation process. The results demonstrate that with decreasing substrate temperature and increasing the droplet volume, the freezing delay time decreased for all five samples. It is also observed that higher values of the contact angle, arithmetic average, root mean squared, ten-point height, maximum height of the profile, along with a Gaussian roughness distribution (skewness near 0) and lower contact angle hysteresis leads to higher ice nucleation time.**

Keywords— ***Icephobicity, Superhydrophobic surfaces, Roughness parameters, Wettability, Ice nucleation time,***

I. INTRODUCTION

Ice formation on different surfaces (either in nature or industries) is a common-faced phenomenon which mostly accounts for a safety problem which endangers the proper performance of the majority of systems and can potentially lead to irreversible losses and deliver terrible economic outcomes [1]. Therefore, effective measures would be taken to remedy these possible problems and remove ice or prevent its formation. These strategies can be divided into active and passive methods. Applying external energy in active approaches makes this method time consuming, uneconomic, and environmentally unfriendly; however, the passive approaches without any external energy focus on delaying ice formation or reducing ice adhesion in order to reduce energy consumption and improve the efficiency of systems [2].

As a remedy to face icing issues, there are a lot of research works regarding success incorporation of superhydrophobic surfaces with the contact angle >150º and contact angle hysteresis<10º using low surface energy materials and surface roughness (inspired by lotus leaves and fabricated by making micro/nanostructures that contain low surface energy chemicals and trapped air inside the structures) as icephobic surfaces in terms of increasing delay in freezing time and reducing ice adhesion. Several studies have reported a correlation between superhydrophobicity and both a reduction in ice adhesion and a delay in ice accretion[3], [4]. Superhydrophobic surfaces can potentially prevent ice formation on a solid surface by decreasing both contact time and contact area as well as increasing droplet roll-off. Contrary to these reports, some investigations, have raised questions concerning the use of superhydrophobic surfaces for icephobic applications especially in condensation frosting condition[5].

Substantial understanding of nucleation would be considered as the initial step to design an efficient and sustainable icephobic surface[6]. Ice nucleation process can be mentioned as a common stage in all ice formation process mainly occurs in two different modes: homogeneous and heterogeneous nucleation that are characterized using absence and present of any external agents, respectively[7], [8]. It is affected by several factors like surface characteristics, droplet size and degree of supercooling. Hence, facing underlying theories and published research experiments in this area requires careful treatment. Two main strategies to delay freezing would be summarized to: (1) modifying the substrate using low surface energy material to change the surface energy and wettability. (2) Changing the surface roughness and topography by physical and chemical methods. Consequently, the effects of surface characteristics such as surface wettability and topography on the ice nucleation process should be considered[9]–[11].

In order to understand how the surface conditions affect icing, it is necessary to understand the surface characteristics and how these may be modified to prevent and delay ice formation. Therefore, adjusting the surface characteristics through surface modifications, changing the surface energy and roughness, affects surface wettability, the energy barrier for nucleation and hence the nucleation rate. These techniques can be employed to delay the onset of ice nucleation and formation. Surface roughness is a measurement of surface texture that cannot be accurately characterized by using a single parameter[12]. Instead, a set of surface roughness parameters is defined. Two of the most commonly used standard surface roughness parameters for an evaluation of the surface roughness are S_a (average surface roughness) and S_q (root mean square roughness). Unfortunately, these two parameters do not describe the contact surfaces sufficiently well. Surface roughness can be accurately assessed by examining a set of surface roughness parameters like ten-point height (S_z), maximum height of the profile (S_t), skewness (S_{sk}), kurtosis (S_{ku}), autocorrelation length (S_{al})[13]. It was shown how two completely different surfaces can show similar, or even the same, values of the standard roughness parameters and vice versa—similar surfaces have much different standard roughness parameters [14]. The influence of roughness parameters on wettability and icing was studied by several researchers, with some focusing on superhydrophobicity [15], [16] and others on icing [17]–[19].

An analysis of the literature thus indicates that there is no available comprehensive study that analyses the influence of the surface wettability, surface roughness (S_a, S_q, S_z, S_t, and S_{sk}), droplet size and surface temperature on ice nucleation time. Therefore, the main objective of this research is offering the optimum surface characteristics to enhance anti-icing properties of silicone rubber surfaces ranging from hydrophobic to superhydrophobic.

II. MATERIAL AND METHODS

A. *Sample Preparation*

High-temperature vulcanized (HTV) silicone rubber was used as the process material. Microstructured aluminum (A6061) templates were produced via a chemical-etching method using a 15 wt.% hydrochloric acid solution and immersion of the aluminum templates in this solution for 2 h. A micro-compression molding machine (Carver Inc. USA) having two temperature-adjustable platens was used to mold the rubber samples. The hydraulic press system can precisely control an applied force of 3 to 194 kN[24]. Three-piece flat molds, all having a right rectangular prism cavity of 25 × 25 × 6 mm3 cast the rubber materials. The template was placed on the lower part of the mold into the cavity, and the rubber material was placed onto the template. The top of the mold was then closed. The mold was set in the press machine to begin the process. After the process, the mold was opened, and the cured silicone rubber was detached from the aluminum template (Fig. 1).

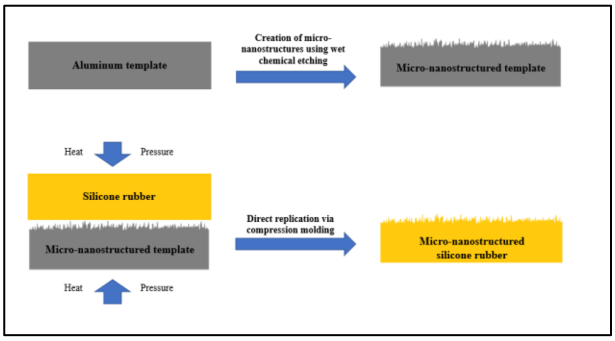


Fig. 1 Schematic of the fabrication of micro-nanostructured silicone rubber surfaces via a microcompression molding technique [21]

B. *Characterization Procedures*

The water CA was determined using a Kruss™ DSA100 goniometer at 25 °C ± 0.5 °C with a 4-µL deionized water droplet, based on the Young-Laplace approximation. The CAH equalled the difference of advancing and receding contact angles when the water droplet moved on the surface. To ensure the accuracy and reproducibility of our results, all wettability measurements were conducted at five different points on each sample; the average and standard deviation for each sample were reported. A confocal laser microscopy profiler (Profil3D, Filmetrics, USA) was used to obtain surface roughness parameters, and a scanning electron microscope (JSM-6480 LV by JEOL Japan) was used to observe the morphology of the fabricated surfaces.

C. *Experimental Setup*

A self-built setup, schematically shown in Fig. 2, was used for icing experiments of sessile droplets. This system contains a thermally insulating and optically transparent chamber, high speed camera, thermostatic bath, cold base, drop injection system, test samples, data acquisition system, temperature sensor, humidity sensor and a vibration-free table.

Before the experiments, the temperature and humidity of the environment and chamber are recorded using temperature sensor and humidity sensor. Then, the temperature of the cold base decreased to different values such as −10 and −20 °C and the sample is places on the cold plate. As the temperature of the samples is stable at the adjusting temperature, different volumes of droplets are placed on the cold surface and its freezing process monitored by a high-speed camera. In each case, there are two steps in freezing of droplet. The first step is regarded as the time when droplet is placed on the surface until the rapid recalescent stage and defined as the nucleation time. This stage can be easily monitored using the high-speed camera (MotionBLITZ, MIKROTRON, EoSens Cube 7, Germany) at 1000 Frame per second. This is followed by a complete freezing. The reported nucleation times are averages of 15 times.

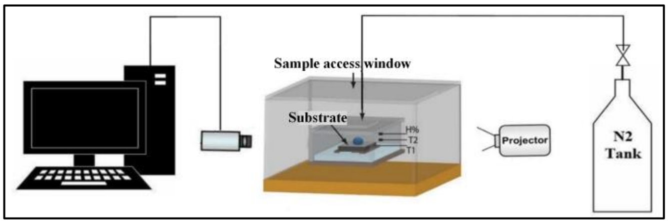


Fig. 2 Schematic of the experimental setup for the ice nucleation study

III. RESULTS AND DISCUSSION

D. *Surface Characterization*

As shown in Figure 3, the surface structures of various samples are illustrated with SEM images and 3D profiles while their wettability properties and surface roughness parameters are summarized in Fig.4 and Fig.5. Generally, surface roughness is evaluated by using surface roughness parameters, such as S_a and S_q; however, these two values do not provide adequate descriptions of surface roughness because they only relate to vertical height, only describing vertical features. In order to detect which parameters best describe silicone rubber surfaces' icephobicity, we selected commonly used roughness parameters (S_a and S_q) along with some less common parameters S_z, S_t, and S_{sk}.

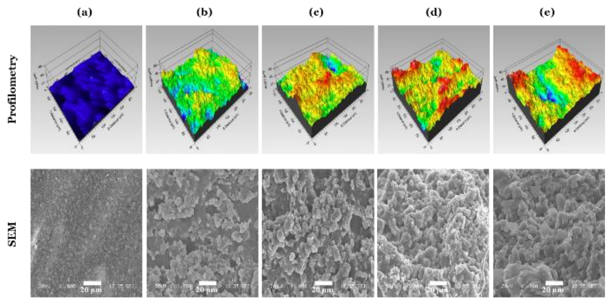


Fig. 3 The 3D surface profiles and SEM images of samples (a) R1, (b) R2, (c) R3, (d) R4, and (e) R5 [21]

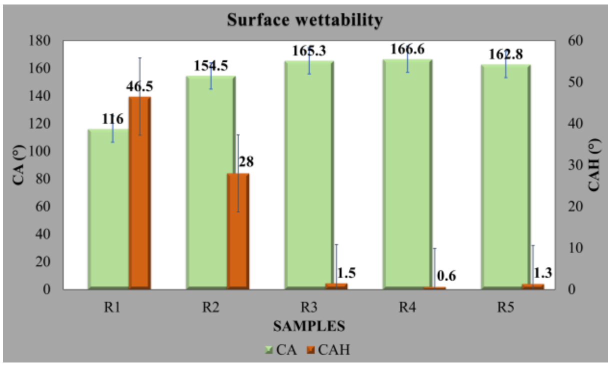
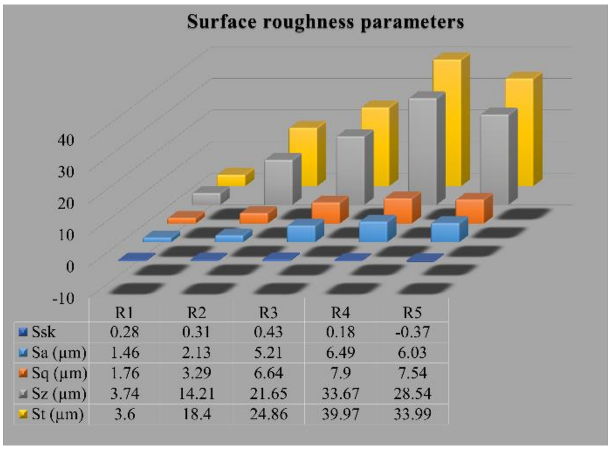


Fig. 4 Water contact angle (CA) and contact angle hysteresis (CAH) values of the samples.

As Sample R1 was replicated on a smooth aluminum template, it proved to be a hydrophobic surface (due to its low surface energy), whereas the other samples exhibited superhydrophobicity (since they were replicated on structured aluminum templates produced with various acid concentrations). The fabrication of superhydrophobic surfaces was therefore accomplished through the selection of a low-surface-energy material with intrinsically hydrophobic properties, combined with a suitable level of surface roughness. In the Sample R2, R3, R4, and R5 samples, S_q increased by 1.87, 3.77, 4.49, and 4.28 respectively compared to sample R1. Besides S_q, S_z and S_t also provide evidence that the created surface profiles are characterized by high peaks and deep valleys. It is also worth noting that the skewness value (a measure of the asymmetry of the profile about the mean plane) varied between −0.37 and 0.43.



	R1	R2	R3	R4	R5
Ssk	0.28	0.31	0.43	0.18	-0.37
Sa (µm)	1.46	2.13	5.21	6.49	6.03
Sq (µm)	1.76	3.29	6.64	7.9	7.54
Sz (µm)	3.74	14.21	21.65	33.67	28.54
St (µm)	3.6	18.4	24.86	39.97	33.99

Fig. 5 The roughness parameters of samples; the average surface roughness (S_a), the root mean square roughness (S_q), ten-point height (S_z), maximum height of the profile (S_t), skewness (S_{sk}).

E. *The Effect of Surface Wettability and Roughness*

Up to now, establishment of relationships between surface roughness and wettability on the ice nucleation process represents a problem for design of icephobic surfaces. To find these complex correlations among surfaces features and ice nucleation time, a set of experiments of ice nucleation process of sessile droplets on cold surfaces with various wettability and roughness were investigated. The shape of water droplets at various times during freezing on the five surfaces is shown in Fig. 6. The results of ice nucleation time for these five different substrates during the freezing process are shown in Fig.7.

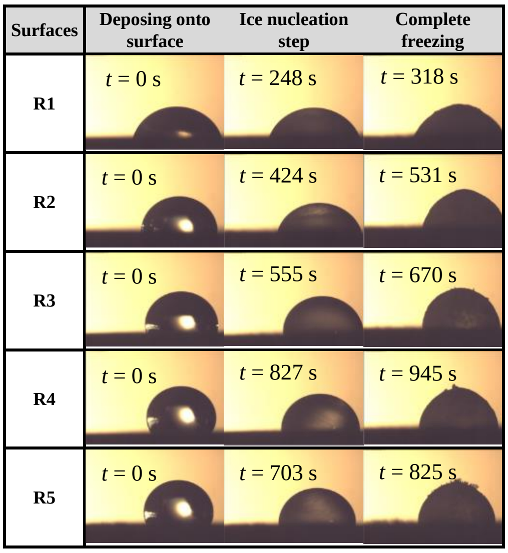
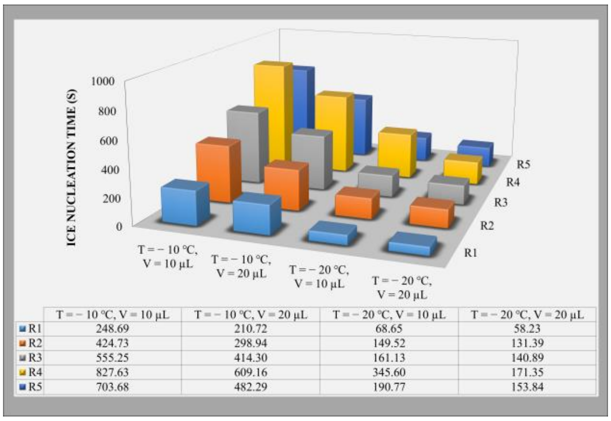


Fig. 6 Images of the freezing of a 10 µL droplet on different solid surfaces at −10 °C

Considering the classical nucleation theory[25], [26], there are two main parameters that affect the critical energy barrier of heterogeneous nucleation: Δμ related to degree of supercooling and f that is the interfacial correlation factor and this factor is a function of the roughness radius of curvature (R) and the contact angle between ice nucleus and the surface structure (Eq.1). It reveals that when the radius of curvature of solid particle or surface textures is much larger than the critical radius of an embryo, f depends only on wetting characteristics and the effect of surface structure in not important[27]. In this study, the surface roughness is in the range of 1.76 to 7.9 µm, which is relatively larger than critical radius of an embryo (i.e., r*= 4.85 nm at −10 °C and 2.19 nm at −20 °C [10]).

$$\Delta G^{*}_{WI} = \frac{16\pi {\sigma_{WI}}^{3} \vartheta_{I}^{2}}{3\left[\Delta h_{WI}\left(\Delta T_m / T_m\right)\right]^{2}} f(R, \theta_{iw}) \,, \tag{1}$$

where Δh_{WI}, T_m, σ_{WI} and ϑ_I are enthalpy of melting, melting point temperature, the interfacial tension ice-water and molar volume of solid phase, respectively.



	T = − 10 °C, V = 10 µL	T = − 10 °C, V = 20 µL	T = − 20 °C, V = 10 µL	T = − 20 °C, V = 20 µL
R1	248.69	210.72	68.65	58.23
R2	424.73	298.94	149.52	131.39
R3	555.25	414.30	161.13	140.89
R4	827.63	609.16	345.60	171.35
R5	703.68	482.29	190.77	153.84

Fig. 7 Ice nucleation times of water droplets of 10 µL and 20 µL on silicone rubber surfaces at −10 °C and −20 °C.

The results of freezing delay time (see Fig. 7) on these various surfaces demonstrate an obvious and remarkable difference. In comparison with ice nucleation time on the original silicon rubber (surface R1), a longer freezing delay time of the water droplets on substrate R2 can be achieved. This time increases significantly when the contact angle reaches to the maximum amount to 166.6 º and contact angle hysteresis reduces to 0.6º. Considering a slightly decrease in CA and roughness and increase in CAH, the ice nucleation time is deceased.

As shown in Fig.7, the same trends can be observed in different temperatures and droplet volumes. According to the results, a remarkable rougher surface (substrate R4) offers the longest time of nucleation because the more air that is trapped at the interface between rougher surfaces and water droplets, the better the insulating effect and the less heat transfer between the droplet and solid substrate. Regarding the surface wettability, a significant increase in ice nucleation time with increasing CA and decreasing CAH is obtained because of reduced contact area and presence of air pockets. Additionally based on classical nucleation theory, Gibbs energy barrier for heterogeneous nucleation in our case is dominated by wetting characterises, means that superhydrophobic surfaces had the greater free energy barrier and longer freezing delay time. It can be said in this research, the longer contact angle, the higher Gibbs free energy barrier.

To investigate the effect of surface roughness parameters, it is shown that a marked microstructure height prevented the droplet from touching the base of the microstructure based on the clear relationship between ice nucleation time and CA as well as S_a, S_q, S_z and S_t. The original roughness parameters of the pristine surface are S_a=1.46 µm, S_q =1.76 µm, S_z=3.74 µm, and S_t=3.6 µm. As a result of increased S_a, S_q, S_z, and S_t, CA and ice nucleation time for superhydrophobic surfaces (Samples R2 to R5) increased. It can be concluded that the height parameter significantly affects the surface wettability. The number of grooves underneath the droplets increased as S_a, S_q, S_z, and S_t of these silicone rubber surfaces increased, lead to smaller liquid–solid contact area and higher CA and longer ice nucleation time.

Based on the skewness shown in Fig. 5, it is evident that all the surfaces have highly symmetric height distributions, as the values for surface skewness range between −0.5 and 0.5. For a surface with S_{sk} close to zero, best performance was obtained in terms of long ice nucleation times and high CA. In other words, a Gaussian distribution of the roughness height is more desirable to achieve super hydrophobic surfaces.

F. The Effect of Surface Temperature

Critical Energy barrier of nucleation is inversely proportional with the square of supercooling ($\Delta {T_m}^2$) and therefore decreases with increase in the degree of supercooling (decrease in governing temperature, T) (Eq.1). It reveals that at earlier stages of temperature reduction (higher temperatures), reduction in critical energy barrier is more than low temperatures. In other words, the rate of decrease in critical energy barrier decreases with decrease in temperature. For example, based on average values of ice nucleation time (Fig. 7), when surface temperature was reduced from -10 to -20 °C, the shortest ice nucleation time was recorded 80.38 s for a 10 L water droplet on Sample R1, while the longest nucleation time was recorded 345.60 s on Sample R4.

Rahimi et al. [10] measured the freezing delay of 6µL water droplet on precooled aluminum substrate modified chemically to obtain different hydrophobic and hydrophilic surfaces without modification of surface topography at different temperatures from −5 to −25 °C. In their experiment, a slightly hydrophilic substrate modified by (3-aminopropyle) triethoxyilance showed longer freezing delays than both more hydrophilic and hydrophobic surfaces because of the surface chemistry and suggested that the ice nucleation kinetics depends on surface wettability and chemistry. For all samples, it is observed that the freezing delay decreases with decreasing substrate temperature.

G. The Effect of Droplet Size

In this part, icing on surfaces with difference surface structures (ranging from hydrophobic to superhydrophobic) focusing on the effect of droplet volume at two different temperatures were tested (Fig. 7). The results show that with increasing the droplet volume, there is a significant decrease in the ice nucleation time. Classical nucleation theory holds that ice nucleation occurs at the nucleation site that is above the critical radius. The probability of nucleation seems to be smaller for smaller drops due to their smaller volume. It seems that smaller drops have a lower probability of nucleating due to their smaller volume[28].

Moreover, the possibility of freezing could be estimated through the nucleation rate of a single embryo, which is inversely proportional to the delay time of ice nucleation and is based on the kinetic theory. The nucleation rate of ice embryos (I) can be estimated as Becker and Doring proposed the following equation to estimate the rate of ice nucleation per unit time and surface area that is related to the free energy barrier of nucleation of critical embryo[29]:

$$I \approx I_0 \exp\left(\frac{-\Delta G^*}{k_B T}\right), \tag{2}$$

where I is the embryo formation rate, I_0 is the kinetic constant, k_B is the Boltzmann constant, and ΔG* is the critical energy barrier according to Eq. 1.

$$I = 1/(t_{nucleation} V), \tag{3}$$

where V is the volume of the system.

It is indicated from this equation that the larger droplet leads to smaller freezing delay time at a constant nucleation rate.

IV. Conclusions

The goal of the presented research was to investigate the optimal surface characteristics to enhance ice nucleation time of water droplet on hydrophobic and superhydrophobic surfaces. We detailed the relationships between surface wettability, roughness parameters, surface temperature, water droplet size, and ice nucleation time. As substrate temperature decreased and droplet volume increased, the freezing delay was reduced for all five surfaces. Thus, S_a, S_q, S_z, S_t, and S_{sk} parameters can be used to determine surface texturing, where higher S_a, S_q, S_z, and St values and a S_{sk} value close to 0 (i.e., a Gaussian distribution for roughness height) lead to superhydrophobic surfaces having a CAH <10º and longer ice nucleation time.

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