Icephobic Performance of Different Surface Designs and Materials

Henna Niemelä-Anttonen, Jarkko Kiilakoski, Petri Vuoristo, Heli Koivuluoto

Materials Science and Environmental Engineering,
Tampere University, Finland

henna.niemela-anttonen@tuni.fi, heli.koivuluoto@tuni.fi

Abstract— **We compared seven different surface designs and materials, tested with the same setup for their icephobic performance. In order to gain reliable, comparable and reproducible results, the test parameters should be similar for evaluation purposes. All the presented samples were tested in Tampere University in the icing wind tunnel (IWIT). The ice was accreted from supercooled microdroplets and the gained ice type was mixed glaze. After the accretion, all the samples were handled similarly and given the same amount of resting period for the ice to set and freeze thoroughly. The ice adhesion values, here ranging from 340 to 5 kPa, were obtained by using a centrifugal ice adhesion tester (CAT) situated in the same cold room. We demonstrate comparable data for these seven different surface designs and materials, and suggest important aspects in ice accretion and adhesion measurement processes.**

Keywords— ***icephobicity, ice accretion, ice adhesion, icing wind tunnel, mixed glaze ice, icephobic surfaces, coatings, testing***

I. INTRODUCTION

Icing and ice adhesion has remained within the focus of many research fields and researchers for many decades. The icing phenomenon itself is a complex and multifold event, and when it occurs on different surfaces, the complexity grows with increasing material and surface variables.

Although researchers use the phrase icephobic to refer to surfaces with low ice adhesion[1], [2], ice itself is rarely exactly the same. Even the slightest climatological changes can alter the ice or the ice accretion process – both in nature and in laboratory scale. However, different applications or application fields, such as aviation, off-shore operators, transportation or energy industry, may encounter wet ice or dry ice more frequently. In these cases, the icing research should take the right ice type into consideration when testing icephobicity from an application-related perspective.

Depending on the application, its purpose and the level of desired icephobic performance, the surface must withstand altering conditions for a specific period of time. Currently, the main challenge lies in incorporating durability to low ice adhesion. Luckily, some applications might need an icephobic solution that possesses the phobicity extremely well for only a short period of time. The need of icephobic performance can vary between permanent and seasonal – from few hours to some months.

The icephobic performance of a surface can be enhanced by understanding the phenomena behind icing, ice formation and ice adhesion – or alternatively approaching the topic from an application-related perspective. In the latter approach, it is of utmost importance to understand the needs and the requirements of the application and carry out the research in a climatically correct environment with the proper ice type, which simulates the outdoor icing event most accurately.

The purpose of the ice-related research dictates the scale of the ice related test to be performed. Field tests give valuable information from real outdoor environments[3], [4]. However, ensuring repeatability can be challenging even though it can be considered as the characteristic nature of the test. Macroscale tests can be performed in cold laboratories by producing ice with more control of the environmental variables[5]–[7]. The same ice type can be formed consistently and, thus, the results are more comparable between separate tests. In microscale tests, microliter droplets are frozen on top of the surface and the freezing or the droplet adhesion can be examined. This is commonly performed with a peltier cooling stage and a droplet shape analyser[8], [9]. These tests utilize a relatively small test area but can have less variables and parameters than larger scale tests. In addition, the icing can be examined in nanoscale with equipment such as a scanning electron microscope (SEM)[10], which can increase understanding of the actual phenomena related to icing and ice formation on surfaces. The test scales are described in Figure 1 with some characteristic features.

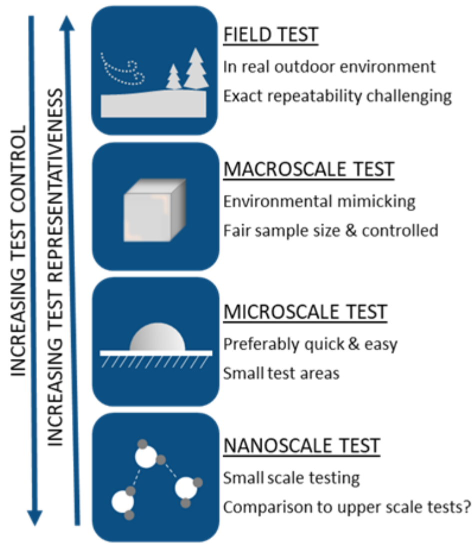

Fig. 1 Ice-related test scales. Depending on the application, it is important to choose the proper testing model and scale. In the smaller scale tests, the control can be enhanced by narrowing down the variables. However, the outdoor field tests with their complexity and multiple, unexpected variables, depict the real circumstances in the nature.

In all cases, the reproducibility, the comparability and representativeness of the results are key issues. In this paper, we demonstrate the icephobic performance of seven different surface designs/materials and evaluate their comparability. We also discuss some important aspects to consider when examining the ice adhesion values in the laboratory scale with accreted ice.

II. ACCRETING ICE AND TESTING ICE ADHESION

As in the nature, ice can be formed in several ways. Nature-mimicking methods are commonly used in laboratory scale ice studies, e.g., by moulding the ice or by accreting it from supercooled microdroplets.

Depending on the chosen method, the properties of the ice and its contact with the surface vary greatly. When the ice is accreted from supercooled microdroplets it mimics precipitation icing[11], [12]. If the ice is created from more wet particles, the accreted glaze ice contains more liquid water. Due to this, it makes a greater and more even contact with the surface on which it builds upon. In another situation, dryer and more porous rime ice does not make a similar, constant contact. The ice particles in rime will freeze before hitting the surface leading to lower liquid water content and thus, different mechanical properties of the ice.[11], [13] In our studies[5], [7], [14], [15], we have been using mixed glaze type of ice. This ice type has glaze-like features without icicles or runback ice formation. It also forms a clean-cut rectangular structure on top of iced surface. Figure 2 shows different ice types, glaze, mixed glaze and rime, which were accreted in the icing wind tunnel (IWIT) in Tampere University by altering the icing parameters.

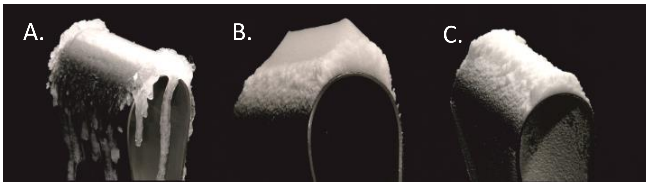

Fig. 2 Different ice types, accreted from supercooled microdroplets in the icing wind tunnel (IWIT) at Tampere University. A.) Glaze, B.) mixed glaze, and C.) rime ice.

Apart from the ice accretion, the ice adhesion test method is another crucial variable to consider in the research of icephobicity. These methods consist of using centrifugal, pendulum, torsional or pulling/pushing energy and motion in order to detach and measure the ice adhesion strength. Thus, the adhesion can be evaluated by many methods and the key point is acknowledging the division of the shear stress between the ice and the sample. The method of measuring the ice adhesion is also somewhat connected to the ice type, since in some situations the test method might force the ice to break cohesively instead of the desired adhesive failure between the ice block and the tested surface.

In the IWIT, the supercooled microdroplets are sprayed from the nozzles and directed towards the test surface with specific wind speeds. For the IWIT testing, several parameters need to be considered in order to gain the desired icing event and thus the proper ice type. These parameters rely on the climate of the cold room, such as the ambient temperature, the humidity and the wind speed – and their variations during the icing. Equally important are the settings of the IWIT system, such as the flow and pressure of water and air, but also the used spraying system, e.g., the nozzle geometry and the spraying distance. All of these affect the parameters, the mean droplet volume, the liquid water content of the ice and thus, the resulting ice type. The overall cleanliness and purity also play an important role since the water grade, the surface cleanliness and the impurities circulating in the cold room can influence the icing and the ice accretion processes. The deviation between each separately tested sample does not only arise from the setup and its variations, but also from the sample itself. Noteworthily, there must be a sufficient number of parallel samples with representative and uniform quality for obtaining as accurate data as possible.

Testing the ice adhesion with the centrifugal ice adhesion tester (CAT) can yield valuable information about their icephobic performance[7], [16], [17]. By examining the ice adhesion values, the surfaces can be ranked for a specific application to meet the desired criteria for the surface. Additionally, a more general characterization of the surface designs and the materials can be made, which in turn increases the fundamental understanding of the icephobicity of various material possibilities.

The CAT analysis should always be performed for multiple parallel samples and a well-known reference sample should be examined within each icing event to support the analysis. Visual inspection of the iced samples plays a major role before performing the CAT – depending on the icing, ice can have unexpected stresses which lead to cracks or fractures. The tested samples and the accreted ice should always be representable, comparable and reproducible examples of themselves. Moreover, the numerical ice adhesion value should not be evaluated as a single indication for icephobic performance. The post-examination, such as examination of a change in wettability as well as, profilometry or microscopy analyses can reveal important information on the effect of the icing but also about the ice detachment and its impact on the surface.

III. COMPARING THE RESULTS

The most important factor for the reproducibility of the results is to understand all the aspects influencing the icing and the ice adhesion testing. In addition, it is important to consider what is actually evaluated. For example, it might not be useful to compare superhydrophobic surfaces (SHS) to slippery liquid infused porous surfaces (SLIPS) since their functionality is based on completely different physical/chemical features[5]. When comparing materials or surface designs with each other, it is important to understand that they all have different wettability, surface free energy, surface morphology, liquid-solid interaction, surface chemistry, heterogeneity, and liquid absorption/retention etc., properties that make them unique. Even within the same material group, variations can be found. For example, in our tests polymeric films had mean ice adhesion strengths ranging from 40 to 170 kPa, as seen in Figure 3 and Table 1. The data has been measured in Tampere University by using the same test setups, the IWIT and the CAT, with mixed glaze ice type.

Figure 3 illustrates how different samples within the same surface design or material group can have altering ice adhesion values. Bulk metals, such as aluminum and stainless steel, have high ice adhesion values, around 340 and 270 kPa, respectively. On the other end of the scale, SLIPS can possess extremely low ice adhesion values, reaching as low as 5 kPa. The remaining groups retain moderate ice adhesion with values close to 100 kPa. With many surface designs, low ice adhesion values, i.e. values lower than 50 kPa, can be reached. However, many applications would benefit from surfaces with extremely low ice adhesion, under 10 kPa, a value that can be obtained with SLIPS surface design which incorporates a porous solid material and a lubricating oil.

We also calculated the standard deviation for each surface design and material group. For both, bulk metal and bulk polymer samples, the standard deviation was 8 %, depicting representative icing and homogeneous sample quality. For the paint group, the standard deviation percentage was 12 %, and in SLIPS, there was a 15 % deviation within the slippery surfaces presented here.

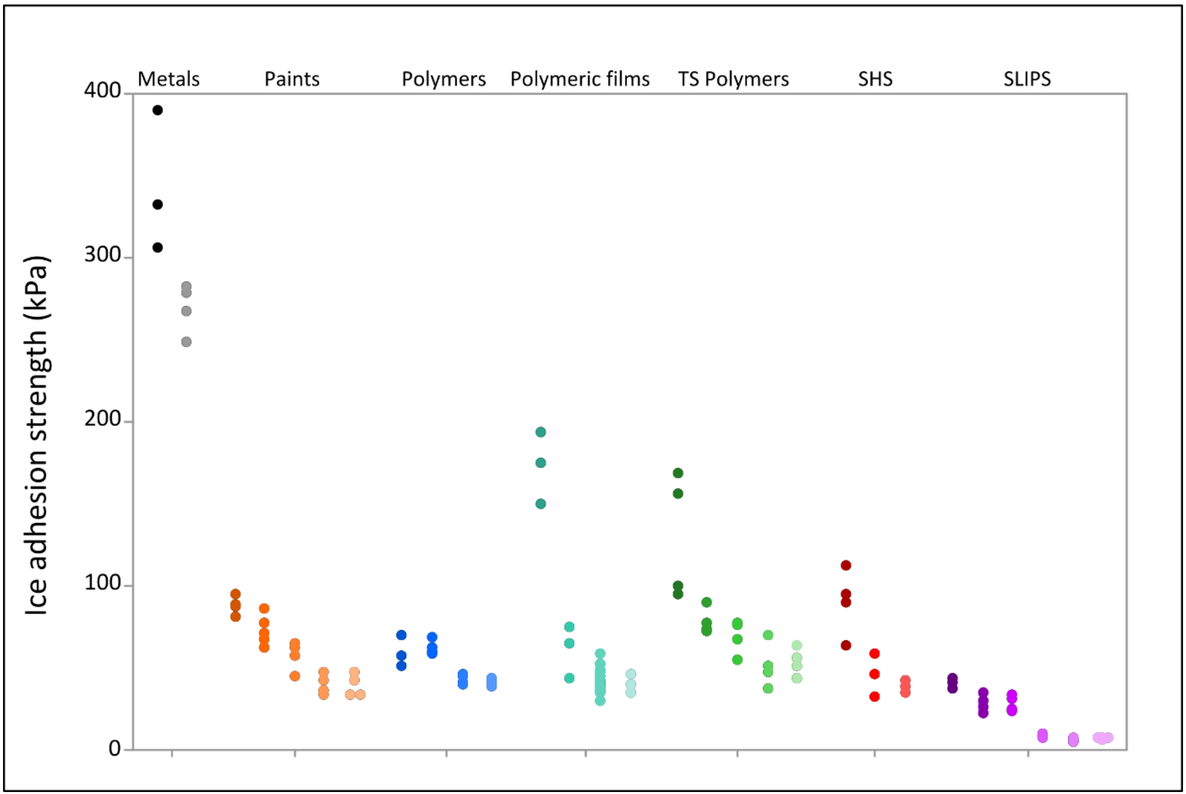

Fig. 3 The ice adhesion strengths for seven different surface design or material groups. The ice type, mixed glaze, was accreted in IWIT and the ice adhesion was tested with CAT in Tampere University. The color depicts a specific group and the different color shades illustrate different samples within the group.

Additionally, the polymeric films, the thermally sprayed (TS) polymer coatings and the SHS group had a fair standard deviation in between 15 to 17 %.

The ice adhesion is not an exact value and cannot be interpreted as such, but more an estimation of the value scale. The gained values can alter based on various factors within the material, the manufacturing process or in the testing. More precisely, not only the testing event but the samples and their uniformity/heterogeneity, handling, history, surface quality, roughness, chemistry, thermal properties, and purity, might influence the obtained values.

The comparison of results obtained with different ice accretion and adhesion measurements setups, should be done with extreme caution. The effect of altering test setups is clear in the research community, thus in ice adhesion values this is seen as a great variation e.g., in the case aluminum. The ice adhesion values for aluminum surfaces varies in different tests between 360 and 1570 kPa[18]–[20]. However, if a similar ice adhesion test method is used, the variation of values is more modest: for aluminum measured with a centrifugal ice adhesion tester, the ice adhesion is around 370 kPa[15], [20], [21]. Well-planned round-robin tests are needed to gain more knowledge on the ice adhesion and its comparability between different setups and also in similar setups located in different laboratories.

A. The studied surface designs and materials

The mixed glaze ice type was accreted in IWIT and the adhesion was examined with CAT, both located in the same cold room at Tampere University. All samples were 60 x 30 mm in size, with the studied and accreted area being 30 x 30 mm. The samples were let to cool down in the cold room for minimum of 30 min before the ice accretion process.

The bulk metal surfaces, aluminum (Hakudo, YH75) and stainless steel (EN 1.4301/2B), were mirror polished prior to testing. The studied paints were Blade Rep LEP 9 (Alexit®, Mankiewicz Gebr. & Co.), wind turbine paint (Carboline Ltd.), and Nanomyte®, (NEI Co.) which was classified as an anti-ice coating. The other paint samples consisted of paint-like silicone-based hybrids with and without ceramic additives (MilliDyne Oy).

The bulk polymer group consisted of polyethylene (PE-HWU, Simona AG), polypropylene (PP-DWU AlphaPlus, Simona AG), polytetrafluoroethylene (PTFE G400, Guarniflon) and ultrahigh molecular weight polyethylene (UHMW-PE, Tivar® 1000, Quadrant Group). The polymeric films were PTFE tape (PTFE Extruded Film Tape, 5490, 3M™), PE tape (Transparent PE-Repairing Tape, 4668, Tesa), wind protection tape (Wind Blade Protection Tape, W8607, 3M™), and polyetheretherketone (0.1 µm poresize, PEEK, Novamem Ltd). TS-polymer coatings were prepared as described in our previous study[15] from polymeric powders.

The SHS consisted of Ultra Ever Dry® coating (UltraTech International, Inc.), fluorine containing coating (MilliDyne Oy), and PTFE membrane (0.2 µm pore size, Sterlitech Inc.). SLIPS surfaces were prepared as described in our earlier publication[5]. The membranes were obtained from Sterlitech Inc. and the used lubricants were perfluorinated oil (Krytox® 103, DuPont™), silicone oil (50 cSt, Sigma-Aldrich, Merck KGaA), rapeseed oil and sunflower oil (Vita D'Oro).

These various surface designs and materials possess different properties and could be utilized in altering application fields where specific icephobic performance is combined with other desired surface properties, e.g., durability, hydrophobicity, and/or optical transparency.

Table I. The surface designs and materials with their ice adhesion strength and standard deviation in kPa. The mixed glaze ice was accreted in the IWIT and the ice adhesion was tested with the CAT at -10 ℃.

Surface	Sample and ice adhesion (kPa)
Metals, bulk	Aluminum (343 ± 35) Stainless steel (269 ± 13)
Paints	Blade Rep 9 (88 ± 5) Wind turbine paint (57 ± 8) Nanomyte (40 ± 5) Silicone-based hybrid paint (73 ± 8) Silicone+ceramic -based hybrid paint (40 ± 6)
Polymers, bulk	PE (43 ± 3) UHMWPE (62 ± 4) PP (60 ± 8) PTFE (41 ± 2)
Polymeric films	PTFE tape (44 ± 7) PE tape (40 ± 5) Wind protection tape (173 ± 18) PEEK (61 ± 13)
Thermally sprayed polymers	PE polished (54 ± 7) PE as-sprayed (70 ± 9) PE+FEP polished (52 ± 12) PE+FEP as-sprayed (79 ± 7) UHMWPE (130 ± 33)
Superhydro phobic surfaces	Ultra Ever Dry (40 ± 3) Fluorine containing coating (46 ± 11) PTFE porous membrane (90 ± 18)
Slippery surfaces, SLIPS	PTFE + perfluorinated oil (8 ± 1) PC + silicone oil (5 ± 1) PP + silicone oil (28 ± 5) PP + sunflower oil (41 ± 3) PP + rapeseed oil (29 ± 5)

IV. CONCLUSIONS

This paper demonstrates ice adhesion strengths for seven different surface designs or materials, in total for 28 different samples. The measured values can be considered as comparable since they were all tested in the same setup with the same ice type and all the samples had similar handling throughout the testing procedures.

The standard deviation value was calculated for each group and it varied between 8 to 17%. The lowest deviations were obtained for bulk metal and polymer surfaces, whereas the highest were in SHS group. Till some extent, the variations arise from the ice accretion and adhesion measurement processes, but also from the natural heterogeneity of the samples. We have observed that even the smallest dents, scratches and imperfections on the sample can elevate the ice adhesion values and so alter the standard deviation. Additionally, since the ice accretion is a delicate process, the ice cannot be considered as a constant, neither in the nature nor in the laboratory scale. However, the obtained values present good reproducibility and they can be compared between the samples to some extent.

It might be challenging to compare these surface designs/materials to one another due to their inherent differences material wise. All the materials have their characteristic properties and their exploitation is based on the overall performance needed for the application. Depending on the needs, the icephobic performance needs to be combined with other relevant properties, such as durability, optical transparency, hydrophobicity and so on. In addition, finding a connection between the icephobic performance and another surface related variable might be difficult to establish. Some connections might be found within a specific surface design/material group, e.g., in SLIPS, where a low sliding angle might promote low ice adhesion[5].

We underline that the results obtained with different ice accretion and moulding methods are not comparable as such due to different ice type and ice properties. Another aspect is the ice adhesion measurement method. These methods rely in different shear or tensile stresses and thus, the measurement processes differ from another. More studies are needed to standardize the ice accretion and the adhesion measurements. Collaboration between different research institutes is needed as round-robin tests would provide important knowledge about testing and evaluation of icephobic performance.

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REFERENCES

[1] V. Hejazi, K. Sobolev, and M. Nosonovsky, "From superhydrophobicity to icephobicity: forces and interaction analysis.," *Sci. Rep.*, vol. 3, p. 2194, Jan. 2013.

[2] H. Sojoudi, M. Wang, N. D. Boscher, G. H. McKinley, and K. K. Gleason, "Durable and scalable icephobic surfaces: Similarities and distinctions from superhydrophobic surfaces," *Soft Matter*, vol. 12, no. 7. Royal Society of Chemistry, pp. 1938–1963, 10-Feb-2016.

[3] K. F. Jones and K. J. Claffey, "Observations and modeling of sea splash icing," in *IWAIS XV.*, 2015.

[4] Á. J. Elíasson, H. Ágústsson, G. Hannesson, and E. Thorsteins, "Comparison of measured and simulated icing in 28 test spans during a severe icing episode," in *IWAIS XV.*, 2015.

[5] H. Niemelä-Anttonen *et al.*, "Icephobicity of Slippery Liquid Infused Porous Surfaces under Multiple Freeze–Thaw and Ice Accretion–Detachment Cycles," *Adv. Mater. Interfaces*, vol. 5, no. 20, p. 1800828, Oct. 2018.

[6] C. Laforte, J.-L. Laforte, and J.-C. Carrière, "How a solid coating can reduce the adhesion of ice on a structure," in *IWAIS X.*, 2002.

[7] H. Koivuluoto, C. Stenroos, R. Ruohomaa, G. Bolelli, L. Lusvarghi, and P. Vuoristo, "Research on icing behavior and ice adhesion testing of icephobic surfaces," in *Iwais 2015*, 2015, pp. 1–14.

[8] L. Boinovich and A. M. Emelyanenko, "Role of Water Vapor Desublimation in the Adhesion of an Iced Droplet to a Superhydrophobic Surface," *Langmuir*, vol. 30, no. 42, pp. 12596–12601, Oct. 2014.

[9] S. Jung, M. K. Tiwari, N. Vuong Doan, and D. Poulikakos, "Mechanism of supercooled droplet freezing on surfaces," *Nat. Commun.*, vol. 3, 2012.

[10] K. Rykaczewski, S. Anand, S. B. Subramanyam, and K. K. Varanasi, "Mechanism of frost formation on lubricant-impregnated surfaces," *Langmuir*, vol. 29, no. 17, pp. 5230–5238, Apr. 2013.

[11] M. Farzaneh, *Atmospheric icing of power networks*. Netherlands: Springer Science & Business Media, 2008.

[12] G. Fortin, J. Perron, and A. Ilinca, "Behaviour and Modelling of Cup Anemometers under Icing Conditions," in *IWAIS XI*, 2005.

[13] L. Makkonen, "Models for the growth of rime, glaze, icicles and wet snow on structures," *Philos. Trans. R. Soc. London. Ser. A Math. Phys. Eng. Sci.*, vol. 358, no. 1776, pp. 2913–2939, Nov. 2000.

[14] H. Niemelä-Anttonen, H. Koivuluoto, M. Kylmälahti, J. Laakso, and P. Vuoristo, "Thermally Sprayed Slippery and Icephobic Surfaces," in *ITSC*, 2018.

[15] H. Koivuluoto, C. Stenroos, M. Kylmälahti, M. Apostol, J. Kiilakoski, and P. Vuoristo, "Anti-icing Behavior of Thermally Sprayed Polymer Coatings," *J. Therm. Spray Technol.*, vol. 26, no. 1–2, pp. 150–160, Jan. 2017.

[16] C. Laforte and A. Beisswenger, "Icephobic Material Centrifuge Adhesion Test," *Proc. Int. Work. Atmos. Icing Struct. (IWAIS XI)*, no. June, pp. 1–5, 2005.

[17] C. Laforte, C. Blackburn, and J. Perron, "A Review of Icephobic Coating Performances over the Last Decade," 2015.

[18] M. Susoff, K. Siegmann, C. Pfaffenroth, and M. Hirayama, "Evaluation of icephobic coatings—Screening of different coatings and influence of roughness," *Appl. Surf. Sci.*, vol. 282, pp. 870–879, Oct. 2013.

[19] T. Bharathidasan, S. V. Kumar, M. S. S. Bobji, R. P. S. P. S. Chakradhar, and B. J. Basu, "Effect of wettability and surface roughness on ice-adhesion strength of hydrophilic, hydrophobic and superhydrophobic surfaces," *Appl. Surf. Sci.*, vol. 314, pp. 241–250, Sep. 2014.

[20] S. Farhadi, M. Farzaneh, and S. A. Kulinich, "Anti-icing performance of superhydrophobic surfaces," *Appl. Surf. Sci.*, vol. 257, no. 14, pp. 6264–6269, May 2011.

[21] S. A. Kulinich, S. Farhadi, K. Nose, and X. W. Du, "Superhydrophobic surfaces: are they really ice-repellent?," *Langmuir*, vol. 27, no. 1, pp. 25–29, Jan. 2010.