Standardizing the testing of low ice adhesion surfaces

Sigrid Rønneberg[1], Jianying He[1], Zhiliang Zhang[1]

[1] *Department of Structural Engineering, Norwegian University for Science and Technology (NTNU), NO-7491 Trondheim, Norway*

Sigrid.Ronneberg@ntnu.no, Jianying.He@ntnu.no, Zhiliang.Zhang@ntnu.no

Abstract— **Low ice adhesion surfaces are a promising strategy to develop anti-icing surfaces. At present, however, the reported ice adhesion strengths are not comparable due to a multitude of performed ice adhesion tests and types of accreted ice. Furthermore, the necessary experimental details are often not included in the published studies. In this paper, a literature review for ice adhesion tests is carried out and experiments performed at the AMIL facility for ice types are reported to show the necessity of comparability. In addition, a protocol for future experiments to help standardize the ice adhesion research is presented. This protocol includes both ice adhesion tests, types of accreted ice, environmental conditions and surface parameters. A reference is proposed with standard aluminum surface and bulk water ice as well as horizontal shear ice adhesion test at -10ºC. The experiments might be performed in different facilities to avoid having to build a new, comprehensive infrastructure, but this cooperation requires a common basis of definitions and references.**

Keywords— *Anti-icing, Ice adhesion strength, Ice adhesion test, Ice type, Standardization*

I. Introduction

Ice removal is necessary to avoid both dangerous situations and the unwanted icing of infrastructure [1], [2]. It is essential to remove the ice efficiently, either with traditional de-icing methods such as thermal, mechanical or chemical deicing or with passive anti-icing surfaces. Passive methods do not require additional energy, but utilizes natural forces such as wind, gravity or surface tension to ensure ice-free surfaces [3]. There are three main pathways to achieve passive anti-icing surfaces: removing water before freezing; the delay of ice nucleation; and the reduction of ice adhesion strength [4]. Considering long-term exposure of anti-icing surfaces in the ambient environment in cold region, the most promising strategy for durable anti-icing surfaces is the lowering of ice adhesion strength [5].

Low ice adhesion surfaces are often defined by an ice adhesion strength below 60 kPa [6]. Surfaces with ice adhesion strength below 20 kPa enables the ice to shed due to natural wind [5], and surfaces with ice adhesion below 10 kPa enables one cubic meter of ice to fall off by its own weight [4].

Research on low ice adhesion surfaces has continuously increased over the past 15 years, and there are many promising coatings available [7]. However, the available literature reports ice adhesion strengths that span three orders of magnitude, and there is no general agreement about reference values [8]. Several standard tests have been proposed earlier [9], [10], and the earliest to our knowledge was presented at IWAIS in 1998 [11]. There have also been published several reviews comparing different widely used test methods [8], [12], [13]. However, the proposed standards do not include comparative discussions, and the comparisons between different methods do not include ice types.

In this paper, we summarize our present work on the different types of ice adhesion test methods and ice types, and propose a future protocol for standardizing ice adhesion research.

II. Current Status of Ice Adhesion Tests

Although a multitude of different ice adhesion tests are available, four tests are most widely in use [14]. These are the horizontal shear test, the vertical shear test, the tensile test, and the centrifugal adhesion test, see Figure 1. For all tests, the ice adhesion strength is defined as the ratio of peak removal force to the interface area of ice, such that $\tau = {}^{F}/_{A}$. As there is no standard today, most research groups develop its own testing set-up [15], [16]. As a result, experimental results originating from different research groups are not comparable today.

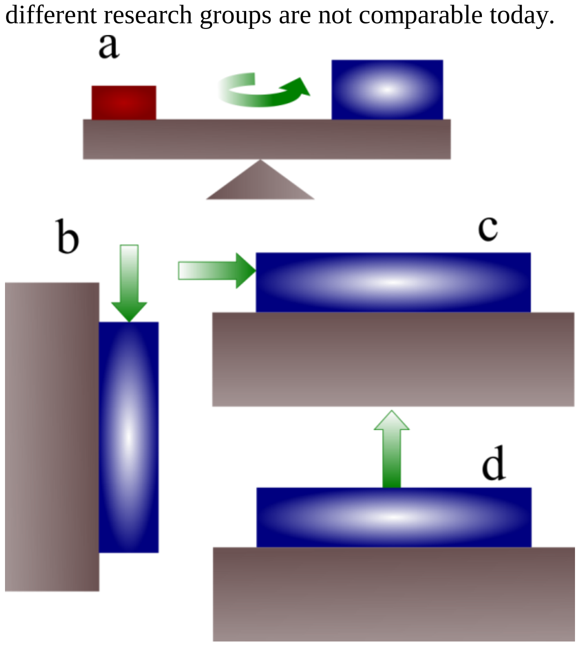

Fig. 1 Schematic illustration of the four most widely used tests methods for ice adhesion strength measurements: a) Centrifugal adhesion test (counterweight is red) b) Vertical shear test c) Horizontal shear test d) Tensile test.

Most publications do not include all their experimental details, such as strain rate and freezing time for ice. In Table I, it can be seen how ice adhesion measurements on a reference aluminum surface differ both within and between the ice adhesion tests. This variation substantiates the great difference between the different tests available, and the importance of a detailed experimental section describing the experiments to ensure reproducibility. An example of the impact of experimental details on the results is the distance between the force probe and the surface for the horizontal shear tests, where a change of 3 mm alters the ice adhesion strength with almost 70% [9]. When several studies do not include this measure in their manuscripts, the results can clearly not be directly compared.

Table I Selection of Reported Ice Adhesion Strengths for a Reference Aluminum Surface.

Test Method	Ice Adhesion Strength [MPa]	Reference
Vertical shear test	0.49	He et al [6]
Horizontal shear test	0.80	Dou et al [17]
Horizontal shear test	0.11	Hejazi et al [18]
Horizontal shear test	0.7-1.0	Lou et al [19]
Centrifugal adhesion test	0.28-0.78	Rønneberg et al [20]
Centrifugal adhesion test	0.19-0.76	Guerin et al [21]
Centrifugal adhesion test	0.32	Laforte and Beisswenger [22]

Experimental details with impact on the same line as probe distance is the probe impact speed for horizontal shear tests [23], temperature [21], ice sample size [24], and stress concentrations [8], [24], among others. In Table II, eight low ice adhesion studies with bulk water ice are shown with their experimental details. As can be seen, the experimental details vary on several accounts, and although the ice adhesion tests were performed with similar ice adhesion tests and with ice frozen in a mold giving bulk water ice, the reported ice adhesion strengths are still not comparable.

Similar to the differences in ice adhesion test methods, the type of accreted ice affects the ice adhesion strength. The properties of ice are highly dependent on the environmental and mechanical conditions, such as temperature, cooling rate, grain size, and crystallization process [26]. The generation process of the ice thus determines the properties of the ice, including ice adhesion strength.

So far, no systematic investigation has been performed to test the effect of different ice adhesion test methods on similar ice types under similar conditions. However, the authors in cooperation with the Anti-icing Materials International Laboratory (AMIL) have performed a comparison of three different accreted ice types at the same temperature.

III. Testing Ice Types

To test the effect of different types of accreted ice on the ice adhesion strength, three types of accreted ice widely used in ice adhesion research were tested at the AMIL facility [22]. More than 120 experiments were performed, and the ice adhesion strength was measured with the centrifugal adhesion test [20], [22]. The centrifugal adhesion test is illustrated in Figure 1d, and utilizes centripetal acceleration as a small ice sample on a beam is rotated with an increasing acceleration. The moment of ice detachment is recorded with piezoelectric cells, and the detachment force is further calculated from the detachment angular velocity. Centrifugal adhesion tests results in more repeatable measurements and has a high probability of adhesive failure, although it can only accommodate one beam shape and can damage surface coatings [8], [14].

The three ice types tested were precipitation ice, in-cloud ice or impact ice, and bulk water ice. Precipitation ice was generated with a freezing drizzle in a cold room, as explained elsewhere [21]. Impact ice was generated in a wind tunnel of wind speed 15 m/s in a standardized procedure at AMIL [22]. Bulk water ice was generated by freezing water in a silicone mold with the aluminum bars placed on top [20]. Bulk water ice is most common on ice adhesion tests, but is not representative for several application such as aircraft icing [8], [15]. All the experiments were performed at -10°C.

The results of the ice adhesion measurements can be seen in Figure 2. The three different ice types clearly differ in their ice adhesion strength even under same environmental conditions and with the same ice adhesion test. Figure 2 clearly indicates that simply stating the ice adhesion strength of different low ice adhesion surfaces without considering the ice type gives inaccurate and flawed comparisons.

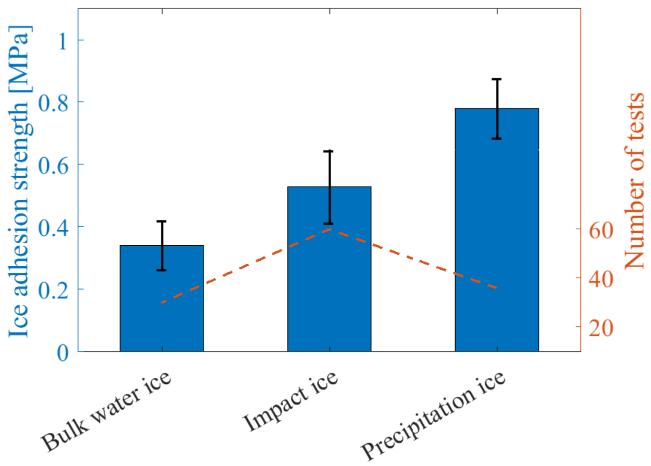

Fig. 2 Results of ice adhesion tests performed at AMIL for three different ice types, together with their standard deviation and number of tests performed [20].

IV. Proposal for Standardizing Protocol

So far, we have shown that reported ice adhesion strengths cannot be directly compared due to differences in both ice generation methods and ice adhesion testing set-ups, even though the environmental conditions are similar. The goal of the low ice adhesion research is to obtain the optimal anti-icing surface with lowest possible ice adhesion strength to mitigate icing on structures. However, before the required comparisons can be made, the research community must agree upon a standard by which to perform ice adhesion strength calculations on different surfaces. Such a standard must be developed by means of international cooperation to ensure that it is applicable for all purposes.

Table II Experimental Conditions for a Selection of Low Ice Adhesion Studies with Bulk Water Ice.

Shear test	**Temperature**	**Freezing time**	**Probe distance**	**Probe impact speed**	**Ice adhesion strength**	**Ref.**
Vertical	-15ºC	3 h	2 mm	0.05 mm/s	50 kPa	Wang et al [23]
Vertical	-15ºC	24 h	3 mm	0.1 mm/s	5-7 kPa	He et al [6]
Horizontal	-10ºC	15 h	2 mm	0.5 mm/s	165-510 kPa	Meuler et al [15]
Horizontal	-20ºC	1 h	1 mm	0.8 mm/s	5-2 kPa	Beemer et al [25]
Horizontal	-25ºC	1 h	3 mm	0.1 mm/s	1 kPa	Irajizad et al [10]
Horizontal	-10ºC	-	1 mm	-	0.15 kPa	Golovin et al [7]
Horizontal	-20ºC	-	-	-	252 kPa	Hejazi et al [18]
Horizontal	-15ºC	5 h	-	-	27 kPa	Dou et al [17]

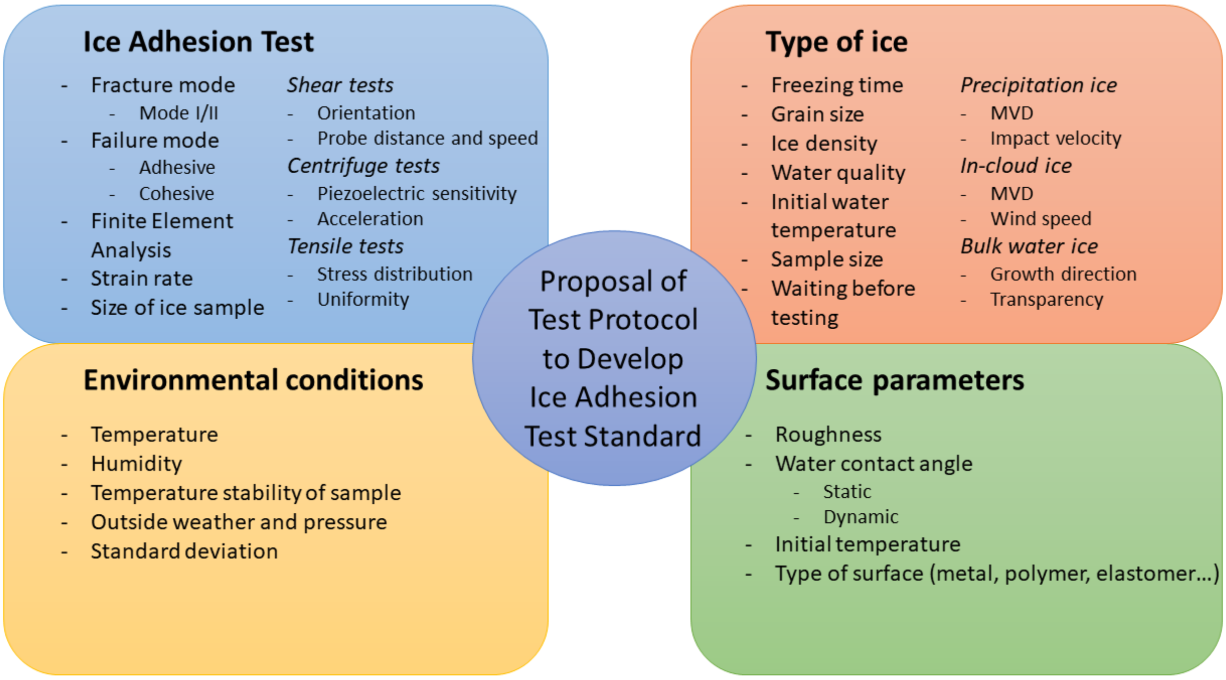

Fig. 3 Selected elements of the proposed experimental protocol to develop an ice adhesion standard.

In this paper, we propose an experimental protocol to help investigate the different parameters that must be accounted for in a standard ice adhesion measurement. Such a standard should be easy to implement, comparable for different types of ice and applicable for many anti-icing applications. Figure 3 shows an example of different parameters that will need to be included in such experiments to determine each of their impact on the ice adhesion strength. To properly explore a fitting ice adhesion standard, each parameter in Figure 3 must be tested and accounted for. The effect of each of the parameters must be tested against a reference. Based on the previous tests performed, the most commonly utilized test and ice type is the horizontal shear test with bulk water ice. This set-up is also easiest to adapt and expand upon in different laboratories and conditions. As a consequence, we propose a reference of standard aluminum surface with bulk water ice of a specific size and horizontal shear ice adhesion test with probe distance 1 mm and probe impact speed 0.5 mm/s. The temperature should be -10ºC during the whole process, and the freezing time should be 2 hours. Based on this reference, all parameters in Figure 3 must be systematically changed and its effect recorded. For statistical accuracy, each set of parameters must be recorded and tested at least 5 times.

For ice adhesion test methods, all available tests should be performed, and all variable parameters must be checked. These parameters will be different for each measurement technique, for instance will the probe behavior be essential to test for shear tests but not applicable for centrifugal tests. However, for all test methods, it is important to investigate stress distribution and strain rate to fully understand the mechanisms of ice detachment, which will be different for various tests [8], [14], [24]. Also the size of the ice sample might impact the ice adhesion strength, especially for the vertical shear test where gravity affects the results. This effect is particularly important for low ice adhesion surfaces [14]. Furthermore, the failure mode is important to distinguish in the different ice adhesion tests. It is vital that failure during ice adhesion tests are adhesive failures instead of cohesive failure, and such a failure mode is more common for some tests, for instance the centrifugal adhesion test, than others such as the tensile test [14].

For types of accreted ice, bulk water ice is chosen as the reference because it is most widely used and it has the fewest controllable parameters [14]. However, bulk water ice does not occur in many realistic applications of low ice adhesion surfaces, such as aircraft or power lines application. As a result, all types of ice must be tested with both horizontal shear test, to compare with reference, and with the other tests as well to see if all ice types are affected in the same way by the different ice adhesion tests. As grain size and ice density are thought to impact the ice adhesion strength [20], different ice generation methods must be specified to create several versions of the same accreted ice. For instance the water median volume drop diameter (MVD) will impact both the density and grain size of the ice, and must be varied. In addition, all ice types generated should be investigated specifically to determine grain size distribution and ice density, to improve our understanding of the ice adhesion mechanism.

For the environmental conditions, the same procedure must be followed of changing one parameter at a time and comparing to the reference test. It is at present unclear what environmental conditions affects the ice adhesion strength. An example can be seen in Figure 2, where the ice adhesion strength within each type of generated ice varies with up to 25 % in spite of the exact similar experimental procedures [20]. At present, it is unclear where this variation comes from, but a hypothesis is that the weather conditions outside impacted the ice generation inside the cold room. As a result, such parameters must also be included in the recording of the experiments.

Last, for the surface parameters, properties such as surface roughness and impurities impacts the ice adhesion strength [21]. Furthermore, different materials and metals display varying ice adhesion strengths, and the effect of such variations should be compared to both the reference tests and the different tests and ices. Low ice adhesion surfaces have their own challenges that must be solved [14], and those challenges might be illuminated by testing multiple surfaces.

No laboratory facility today includes the necessary infrastructure and equipment to perform the required tests and parameter checks included in this proposed protocol. It might be possible to perform the different tests at various facilities, but these scattered experiments must have a common reference basis as well as a common language. In addition to agreeing on the parameters of the experiments, the name and definition of ice types and tests must be homogeneous. At present this agreement is lacking, as can be seen by the many definitions of the term "glaze ice" [27]-[29].

V. Summary

This paper has dealt with the lack of standards within ice adhesion research, both with respect to ice adhesion strength measurements and types of accreted ice tested. Several examples have been described which shows how direct comparison between reported values of ice adhesion strengths is impossible today. Furthermore, a protocol to test the requirements of a new ice adhesion standard has been described. This protocol includes tests on ice adhesion test methods, type of ice tested, environmental conditions and surface parameters. The experiments might be performed in different facilities to avoid having to build a new, comprehensive infrastructure, but this cooperation requires a common basis of definitions and references.

Acknowledgment

The authors would like to thank Caroline Laforte, Christophe Volat and Caroline Blackburn at AMIL for their invaluable contribution in conducting the ice adhesion experiments. The authors gratefully acknowledge the financial support from the Norwegian Research Council FRINATEK project Towards Design of Super-Low Ice Adhesion Surfaces (SLICE, 250990).

References

[1] L. Makkonen, "Ice Adhesion —Theory, Measurements and Countermeasures". *Journal of Adhesion Science and Technology*. vol. 26, nr 4-5, p. 413-445, 2012

[2] M. J. Kreder, J. Alvarenga, P. Kim, and J. Aizenberg., "Design of anti-icing surfaces: smooth, textured or slippery?" *Nature Reviews Materials*, vol. 1, p. 1-15, 2016

[3] J. Brassard, C. Laforte, F. Guerin, and C. Blackburn, *Icephobicity: Definition and Measurement Regarding Atmospheric Icing*, ser. Advances in Polymer Science. Berlin, Heidelberg, Germany: Springer, 2017.

[4] Z. He, Y. Zhuo, J. He and Z. Zhang, "Design and Preparation of Sandwich-Like Polydimethylsiloxane (PDMS) Sponges with Super-Low Ice Adhesion". *Soft Matter*, vol. 14, p. 4846-4851, 2018

[5] J. Chen, J. Liu, M. He, K. Li, D. Cui, Q. Zhang, X. Zheng, Y. Zhang, J. Wang, and Y. Song, "Superhydrophobic surfaces cannot reduce ice adhesion". *Applied Physics Letters*, vol. 101, 2012.

[6] Z. He, E. T. Vågenes, C. Delabahan, J. He, and Z. Zhang, "Room Temperature Characteristics of Polymer-Based Low Ice Adhesion Surfaces". *Scientific Reports*, vol. 7, p. 42181, 2017

[7] K. Golovin, S. P. R. Kobaku, D. H. Lee, E. T. DiLoreto, J. M. Mabry, and A. Tuteja, "Designing durable icephobic surfaces", *Science Advances*, vol. 2, nr. 3, 2016.

[8] A. Work, and Y. Lian, "A critical review of the measurement of ice adhesion to solid substrates", *Progress in Aerospace Sciences*, vol. 98, p. 1-26, 2018.

[9] C. Wang, W. Zhang, A. Siva, D. Tiea, and K. J. Wynne, "Laboratory test for ice adhesion strength using commercial instrumentation", *Langmuir*, vol. 30, nr. 2, p 540-547, 2014.

[10] P. Irajizad, A. Al-Bayati, B. Eslami, T. Shafquat, M. Nazari, P. Jafari, V. Kashyap, A. Masoudi, D. Araya, and H. Ghasemi, "Stress-Localized Durable Icephobic Surfaces", *Materials Horizons*, vol. 6, p. 758-766, 2019.

[11] N. D. Mulherin, R.B. Haehnel, and K.F. Jones, "Toward developing a standard shear test for ice adhesion", in *8th International Workshop on Atmospheric Icing of Structures*, 1998, p. 73-79.

[12] M. R. Kasaai, and M. Farzaneh, "A critical review of evaluation methods of ice adhesion" in *23rd International Conference on Offshore Mechanics and Arctic Engineering*, 2004, paper 3, p. 919-926.

[13] J. M. Sayward, "Seeking low ice adhesion", U.S. Army Cold Regions Research and Engineering Laboratory, Tech. Rep. 00197109, p. 1-88, 1979.

[14] S. Rønneberg, J. He, and Z. Zhang, "The Need for Standards in Ice Adhesion Research: A Critical Review". *Submitted*, 2019.

[15] A. J. Meuler,J. D. Smith, K. K. Varanasi, J. M. Mabry, G. H. McKinley, and R. E. Cohen, "Relationships between Water Wettability and Ice Adhesion", *ACS Applied Materials & Interfaces*, vol. 2, nr. 11, p. 3100-3110, 2010.

[16] 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, p. 1938-1963, 2016.

[17] R. Dou, J. Chen, Y. Zhang, X. Wang, D. Cui, Y. Song, L. Jiang, and J. Wang, "Anti-icing Coating with an Aqueous Lubricating Layer", *ACS Applied Materials & Interfaces*, vol. 6, nr. 10, p. 6998-7003, 2014.

[18] V. Hejazi, K. Sobolev, and M. Nosonovsky, “From superhydrophobicity to icephobicity: forces and interaction analysis”, *Scientific Reports*, vol. 3, 2013.

[19] D. Lou, D. Hammond, and M.-L. Pervier, “Investigation of the Adhesive Properties of the Ice–Aluminum Interface”, *Journal of Aircraft*, vol. 51, nr. 3, p. 1051-1056, 2014.

[20] S. Rønneberg, C. Laforte, C. Volat, J. He, and Z. Zhang, “The effect of ice type on ice adhesion”, *AIP Advances*, in press, 2019.

[21] F. Guerin, C. Laforte, M.-I. Farinas, and J. Perron, “Analytical model based on experimental data of centrifuge ice adhesion tests with different substrates”, *Cold Regions Science and Technology*, vol. 121, p. 93-99, 2016.

[22] C. Laforte, and A. Beisswenger. “Icephobic Material Centrifuge Adhesion Test” in *11th International Workshop on Atmospheric Icing on Structures (IWAIS)*, 2005.

[23] C. Wang, M. C. Gupta, Y. H. Yeong, and K. J. Wynne, C., “Factors affecting the adhesion of ice to polymer substrates”, *Journal of Applied Polymer Science*, vol. 135, nr. 24, 2017.

[24] M. Schulz, and M. Sinapius, “Evaluation of Different Ice Adhesion Tests for Mechanical Deicing Systems”, *SAE International*. 2015.

[25] D. L. Beemer, W. Wang, and A.K. Kota, “Durable gels with ultra-low adhesion to ice”, *Journal of Materials Chemistry A*, vol. 4, p. 18253-18258, 2016.

[26] S. Løset, K. N. Shkhinek, O. T. Gudmestad, and K. V. Høyland, *Actions from Ice on Arctic Offshore and Coastal Structures: Student's Book for Institutes of Higher Education*, St. Petersburg: "LAN", 2006.

[27] G. Fortin, and J. Perron, “Ice Adhesion Models to Predict Shear Stress at Shedding”, *Journal of Adhesion Science and Technology*, vol. 26, nr. 4-5, p. 523-553, 2012.

[28] T. Armstrong, B. Roberts, and C. Swithinbank, *Illustrated Glossary of Snow and Ice*, Cambridge: Scott Polar Research Institute, 1973.

[29] J. Liu, Z. Janjua, M. Roe, F. Xu, B. Turnbull, K.-S. Choi, and X. Hou, “Super-Hydrophobic/Icephobic Coatings Based on Silica Nanoparticles Modified by Self-Assembled Monolayers”, *Nanomaterials*, vol 6. nr. 12, 2016.