Comparative study on the influence of surface characteristics on de-icing evaluation

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ABSTRACT

A comparative study of de-icing evaluation methods was conducted in this work, and their variations in response to surface characteristics were investigated. The mechanical de-icing measurements include centrifugal, push, and tensile methods. The centrifugal and the horizontal push (shear) methods suggested a linear relationship of ice adhesion strength with surface roughness, whereas the tensile (normal) method indicated an inverse curvilinear relationship with contact angle hysteresis. A partial correlation of contact angle hysteresis on the shear-based methods was also indicated over a specified range of surface roughness. Further attempts were also made on 1H,1H,2H,2H-perfluorooctyltriethoxysilane-coated surfaces, and the ice adhesion indicated a clear reduction in the normal de-icing method, whereas the shear-based methods did not show a considerable change in ice adhesion, highlighting their mechanical forces-centric response. Lastly, a further evaluation using a hybrid de-icing method was conducted, to verify the influence of surface characteristics on ice removal involving heating, which demonstrated a partial correlation of energy consumption with the ice adhesion strength over a specified range of surface roughness. The results obtained in this study provide crucial information on the influence of surface characteristics on ice adhesion and offer material-dependent correlations of the popular de-icing evaluation methods. The conclusions could be applied to define an appropriate testing method for the evaluation of ice-phobic surfaces and coatings.

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GRAPHICAL ABSTRACT

Introduction

Ice accretion is a major multibillion-dollar problem. Multiple aspects of ice problems were widely studied, but ice detachment (de-icing) or a delay in ice formation (anti-icing) remains the primary indicator in passive icephobic studies. Over the last 90 years, different de-icing evaluation methods were used to evaluate icephobic performance. The non-existence of a standardised de-icing evaluation method means that each research group publishes their measured data and the comparability of data is difficult due to the variations in icing sample preparation, measured magnitude, applied force, and interfacial contact areas. For example, the ice adhesion measured using existing de-icing methods varies by orders of magnitude [1] and the stress distributions across an interface area are not uniform and are uneven [2].

A majority of these de-icing tests consists of mechanical tests (such as push method) [3–5], shear method (such as shear lap joint test) [6–8], centrifuge rotating test [9–11], and tensile method [12–14]. One of the most widely used methods to measure ice adhesion strength is horizontal/vertical push method (HPM) owing to its simplistic and economical

design. The ice adhesion strength measured on the push method is greatly affected by the contact location of a force probe. In this case, the stress distribution may not be completely uniform [1, 15]. The centrifugal method (CAT) on the other hand is the most repeatable ice adhesion test and is ideal for large components [1, 15]. However, this method is complicated to set and unable to produce stress-strain curves [16]. Apart from the operational and equipment limitations, intrinsic shear or nominal adhesion force measured by the de-icing methods is also dependent on interfacial contact area and the true contact area may vary with the change in surface roughness and/or wettability model. For example, a change in the wetting model or an alteration in ice formation with different surface characteristics may have a serious impact on the ice fracture mechanics. From a fracture mechanics point of view, different shear and peel stresses are exerted by the de-icing methods on crack tips and the crack formation, and the propagation entirely depends on the load case and orientation [17].

Promising strategies to initiate the standardisation of de-icing evaluation methods may include generating comprehensive reviews of existing techniques, highlighting similarities and challenges the methods

possess, and to forge collaborations between research laboratories to compare ice adhesion results using different test rigs to cross-validate the evaluation methods and results. Interlaboratory examinations have been initiated in the last few years [2, 18], and reviews on existing de-icing evaluation techniques are reported [1, 15, 16]. However, the influence on ice adhesion related to the surface characteristics has not been studied. Work et al. [1] reviewed de-icing techniques and suggested that the testing parameters, such as temperature, surface roughness, strain rate, and impact velocity, play crucial roles in ice adhesion measurements. Additionally, they indicated that most of the studies did not indicate the influence of stress concentrations during de-icing tests, which may have caused significant variations in the results. Laroche et al. [19] found that the major factors that affect the adhesion of ice are the purity of ice, surface feature (wettability), surface texture (roughness), and testing temperature. Overall, there is a need to initially quantify the de-icing evaluation methods, while keeping the testing parameters, such as temperature, surface roughness, wettability, and interfacial contact area, unchanged. These results can prompt further understanding on the influence of surface characteristics, which may involve a systematic alternation in surface characteristics, on de-icing fracture mechanics, and the generation of stress concentrations in the ice/surface interfaces.

In this paper, we conducted a comparative study of three de-icing methods and focused on the influence of surface characteristics in de-icing evaluation. Firstly, ice adhesion strength was measured using the centrifugal, horizontal push, and tensile methods, ensuring the same interfacial contact area and testing temperature for all the tests. These methods were further debated in terms of fracture mechanics at ice/solid interfaces, possible stress concentrations, thermodynamics, and the influence of surface wettability. Secondly, the effect of heating at the ice/surface interfaces via a hybrid de-icing method was also examined. Additionally, the comparison results were analysed to study the variations of response with surface characteristics of the samples, together with the de-icing methods. These results could fill a knowledge gap on the influence of surface characteristics on ice adhesion strength measured using different de-icing methods. Finally, the conclusions could be applied to define an appropriate

testing method for the evaluation of icephobic surfaces and coatings.

Experimental

Four types of materials/coatings were used in this study, and each surface was characterised and compared in terms of surface roughness, wettability, and ice adhesion strength. The material types and coatings are summarised in Table 1, and their properties are detailed in supplementary Table S1.

Substrates and raw materials

The stainless steel 303 (SS 303) and aluminium 2024 (Al 2024-T4) plates were used as references with the size of 50 mm × 20 mm × 3 mm and 50 mm × 20 mm × 1 mm, respectively. 1H,1H,2H,2H-Perfluorooctyltriethoxysilane (POTS) was procured from Fluorochem Ltd. Micro-polishing cloth and colloidal silica suspension were procured from Struers and MetPrep, respectively. N, N-Dimethylformamide (DMF) was purchased from Sigma-Aldrich.

Thermoplastic polyurethane matrix Estane 54610 (TPU) in the form of pellets was purchased from Lubrizol, USA. Hexagonal Boron Nitride nanoparticles with a size of 70 nm (hBN NPs) were procured from Lowerfriction lubricants, Canada. All the materials were used as received.

Preparation of coatings

The Al-AR/SS-AR samples/substrates were washed with ethanol and deionised water thrice and the samples were then dried using compressed air. The aluminium and stainless steel plate samples were smoothened using grinding and polishing techniques with a series of steps employing sandpapers having grits sizes of 220, 320, 400, and 600, 1 µm polishing cloths, and 0.25 µm (chemically induced) polishing cloths using Metprep colloidal silica suspension particles, respectively. The grinding and polishing techniques are detailed in the reference [20, 21]. The Al-SB/SS-SB samples were roughened in a Guyson F1200 sandblaster system using Guyson 180–220 µm alumina particles. The Al-fSB/SS-fSB samples were subsequently functionalised using POTS by employing a chemical vapour deposition (CVD) method, which had been reported elsewhere [22].

Table 1 Summary of the sample types and their composition

Sample types	Sample codes	Substrates	Surface treatment/coating	Description
Aluminium plates	Al-AR	Al2024 T4	–	–
	Al-SB		Sandblasted	–
	Al-fSB			POTS Functionalised
	Al-S1		Grinding	Grinded (600 grit)
	Al-S2		Polished	1 µm polishing cloth
	Al-S3			0.25 µm Silica particles
Stainless steel plates	SS-AR	SS 303	–	–
	SS-SB		Sandblasted	–
	SS-fSB			POTS Functionalised
	SS-S1		Grinding	Grinded (600 grit)
	SS-S2		Polished	1 µm polishing cloth
	SS-S3			0.25 µm Silica particles
Polymeric nanocomposite coatings	TPU	Al2024 T4	TPU-hBN	–
	TPU-5			hBN (5 wt%)
	TPU-f5			POTS Functionalised hBN (5 wt%)
	TPU-10			hBN (10 wt%)
	TPU-40			hBN (40 wt%)
	TPU-60			hBN (60 wt%)
	TPU-80			hBN (80 wt%)

The TPU-based coatings were developed, and the nanoparticles were functionalised using methods mentioned reported previously [20, 21]. All the coating solutions were magnetically stirred in a vial for 60 min, followed by 30 min of ultrasonic mixing. All TPU-hBN coatings were fabricated using the dip-coating method. The resultant coatings were dried in the oven at 80 °C for 4 h and followed by a further treatment at 150 °C for 8 h.

Surface characterisation

A Zeta-20 non-contact optical profiler was used to evaluate the surface roughness. The roughness values reported were the average of a minimum of 30 measurements, and R_a was measured over a line stretching across the observed surface. The measurement method has been described elsewhere [23]. The system was also used to examine topographical changes on the observed surfaces. An FEI Quanta 650 ESEM (environmental scanning electron microscope) was used to take microstructural images. The same system was used to observe morphological changes on the coatings examined in this study. Supplementary Table S1 provides the complete data of surface roughness measurements.

Evaluation of hydrophobicity

The sessile drop technique was used to measure water contact angles (WCAs) using an FTÅ200 goniometer and 5 µl of a controlled volume of water drop was analysed. The tests were conducted at room temperature, and the measurement method is described elsewhere [24, 25]. The static and dynamic water contact angles (WCAs), including advancing WCAs (AWCAs), receding WCAs (RWCAs), and contact angle hysteresis (CAH), are summarised in supplementary Table S1.

Evaluation methods of de-icing tests

Ice adhesion strength measurements using different methods are summarised in supplementary Table S1. The ice adhesion strength τ_{ice} (kPa) can be calculated by,

$$\tau_{ice} = F/A \quad (1)$$

where A (m²) is the substrate/ice projected contact area and F (N) is the force associated with the ice detachment. The ice adhesion tests were conducted at the surface/ambient temperature of −10 °C. It is imperative to mention that the CAT was carried out under ambient sub-zero conditions in a climate

chamber, whereas the HPM and NTM were carried out on a Peltier water-cooled plate. The shape of ice moulds was customised to adapt to the requirements of the tests, while the contact area of the ice moulds used in all three tests was kept at 3.1 cm^2 .

Centrifugal method

A MOOG G403-2053A servo motor was used to measure the ice shear strength tests via a centrifugal method, and the test was performed in an environmental chamber (ALPHA 1550-40H) to simulate the freezing conditions. The measurement method is further detailed in our previous work [26].

The shear force F (N) on the samples can be calculated by,

$$F = mr\omega^2 \quad (2)$$

where ω (rad/s) is the rotational speed at the ice removal, r (m) is the full rotor length, and m (kg) is the mass of ice. Bulk ice was grown on the sample inside the environmental chamber to avoid introducing a thermal and mechanical history to each sample.

Horizontal (transverse) push method

Ice shear strength was measured using a horizontal push method in a custom-built setup at the surface temperature of -10°C . More details on the custom-built setup are detailed in our previous work [27]. A square-shaped mould of volume 5.48 cm^3 was used to form ice on a closed-loop water-cooled Peltier plate. The height of the force probe in HPM was kept at a minimal distance from the surface to eliminate the discrepancies in the adhesion measurements. The adhesion strength was measured by employing a digital force gauge, pushed at $100 \mu\text{m/s}$, and the force gauge had a capacity of measuring up to 500 N at a resolution of 0.1 N .

Normal tensile method

Unlike the centrifugal and push methods which are based on the shear strength (Mode II fracture), the tensile de-icing method measured the normal adhesion strength (Mode I fracture). However, due to the application of transverse motion on one side of the bulk ice, the shear-based de-icing methods often produce a combination of Mode I and Mode II

fracture [15, 19], as shown in Fig. 5c and explained in detail in “[Fracture mechanism at ice/solid interfaces](#)” Section. The normal adhesion force on the tensile method was measured via a custom-made ice mould, which was connected to a force gauge and was pulled using the threads connected on the opposite ends of the ice mould. The shape and geometry of the ice mould were optimised to achieve a uniform stress distribution across the contact area. More details on the tensile method and its construction were reported previously [28]. A digital force gauge of a similar configuration as that of HPM was used. The surface temperature was maintained at -10°C during the ice formation and throughout the testing duration.

Electro-thermal de-icing evaluation

For electro-thermal de-icing tests, the bulk ice was formed over the surface using silicone moulds against gravity and the ice was formed overnight. The electro-thermal de-icing evaluation was carried out by the introduction of heating at ice/surface interfaces, and no external mechanical forces were introduced during the ice melting process and the ice was allowed to detach under the gravitational pull. It is important to mention that the samples were held perpendicularly and a shear force could be generated at the ice/solid interfaces under the influence of gravity. Thus, the electro-thermal de-icing method is different from other de-icing methods used in this study which mainly utilise external mechanical forces to de-ice. The electro-thermal de-icing was conducted in a custom-built de-icing rig, constituting a sandwiched heating element which was connected to a 12 V , 0.3 A power source to heat the sample. The samples were mounted on the rig and were allowed to cool down to -10°C . The test was initiated at a time when the surface temperature reached -10°C . The de-icing method is described in detail in our previous work [29]. The temperature was monitored using a rod thermostat which was inserted in the rig, located between the heating element and the sample. In all tests, a fan was operational to ensure temperature consistency in the chamber and an ambient temperature of -10°C was also maintained.

Results and discussion

Microstructural analysis

An alternation in surface roughness was introduced using grinding, polishing, or sandblasting of the metallic substrates and via the incorporation of nanoparticles in the polyurethane matrix. The surface roughness (R_a) on sandblasted SS-SB samples was $1 \pm 0.02 \mu\text{m}$ and was brought down to $0.03 \pm 0.01 \mu\text{m}$ on the smoothened SS-S3 samples. The microstructures of SS-SB, SS-AR, and SS-S3 (from right to left) are shown in Fig. 1a.

Similarly, the surface roughness (R_a) was reduced from $1.2 \pm 0.02 \mu\text{m}$ on Al-SB samples to $0.05 \pm 0.01 \mu\text{m}$ on Al-S3 samples and from $0.6 \pm 0.01 \mu\text{m}$ on TPU-80BN to $0.01 \pm 0.01 \mu\text{m}$ on TPU-5BN coatings. The microstructural images of the aluminium substrates (Al-SB, Al-AR, and Al-S3 from right to left) and TPU-hBN coatings (TPU-5, TPU-10, TPU-40, and TPU-80 from right to left) are shown in Fig. 1b and c, respectively.

Comparison of de-icing methods for the evaluation of ice adhesion strength

The ice adhesion measurements acquired using the HPM, the CAT, and the NTM are illustrated in Fig. 2a, b, and c, respectively. The results obtained on the CAT and HPM methods suggest a linear relationship of ice adhesion strength on surface roughness. Firstly, all the methods were sensitive to surface roughness, whereas the CAT method showed greater sensitivity. For example, on aluminium substrates, ~ 12 -fold increase in ice adhesion was observed from Al-SB to Al-S3 samples using the CAT method, whereas $\sim$ twofold increase was observed on the HPM method. The microscopically rough surface not only provides large cavities for possible ice anchoring points and substantial mechanical interlocking but also extends the contact area of the ice, and thus the true interfacial contact area is enlarged. Similarly, the smoothening of surfaces reduces the number of cavities and subsequently, the overall true interfacial area is shrunk, and the mechanical interlocking is minimised. Thus, the interfacial area and mechanical interlocking of ice could be used to explain the higher ice adhesion on the microscopically rough surface. Secondly, the CAT and the NTM demonstrated a greater sensitivity

among the studied samples. For example, the dotted squares in Fig. 2a, b, and c show that the polymeric samples indicated a higher ice adhesion as compared to the metallic samples at similar surface roughness using the CAT and NTM methods. A $\sim$ two-fold increase in magnitude was observed between the polymeric and metallic samples on both the CAT and NTM methods, compared to a ~ 1.2 -fold increase using the HPM method. The adhesive ice failure was reported on a majority of the tested samples in all three de-icing methods, whereas the ice on rougher surfaces, especially on sandblasted samples, might have detached cohesively. Thus, the definitive comparisons drawn in this section, in terms of ice detachment magnitudes, were only based on the adhesion failures.

Few observations can be drawn on the ice adhesion results measured with the NTM setup. Firstly, the magnitude of ice adhesion measured on the NTM setup is in the range of 200–1500 kPa, as compared to 200–800 kPa and 10–160 kPa using the HPM and CAT methods, respectively. The comparison of the ice adhesion strength does not necessarily suggest a direct relationship between the studied de-icing methods, but the differences in magnitude measured by different de-icing methods could be reflected. A direct comparison of ice adhesion magnitude may not be suitable as the NTM measures the ice fracture in Mode I fracture, whereas the shear methods detach the ice in a combination of Mode I and Mode II fracture. Secondly, the ice adhesion measured using the NTM method demonstrated a non-linear relationship with surface roughness, rather a non-monotonic curvilinear relationship. Surprisingly, a higher ice adhesion strength was recorded on the smoother samples using the NTM method, whereas the ice adhesion strength on similar samples decreased with the lower surface roughness using the CAT and HPM methods. Thus, this observation suggests that there are other influencing factors in the NTM methods, and the results are further debated in terms of surface wettability in the next section.

Influence of contact angle hysteresis on the ice adhesion

The previous section indicated a strong sensitivity of the de-icing methods on surface roughness, and the lower surface roughness resulted in reduced ice adhesion strength on most samples. However, a higher ice adhesion was obtained on the smoothened

Figure 1 The microstructure of **a** aluminium, **b** stainless steel, and **c** TPU-BN samples, from the roughest sample on the left side to the smoothest sample on the right. All images have the same scale bar.

samples from the NTM test. To understand ice adhesion behaviour in the NTM method, the ice adhesion results were plotted in a single figure with contact angle hysteresis (CAH) values as shown in Fig. 3a, b, and c. Two conclusions can be drawn from Fig. 3. Firstly, the CAH demonstrated inverse proportionality with ice adhesion strength on the SS samples. Similar correlations of ice adhesion strength with CAH were also observed on the aluminium samples and TPU-BN coatings. It is imperative to

mention that the ice adhesion, obtained using the CAT and the HPM methods, did not correlate with CAH values completely. A partial correlation of CAHs was observed on surface roughness (R_a) below $0.5\text{ }\mu\text{m}$ on the studied surfaces and the receding water contact angles also followed a similar trend, as shown in supplementary figures S1 and S2. Similar findings were also reported in our previous work [20, 21]. The partial correlation of CAHs on ice adhesion suggests a dominant role of surface

Figure 2 Ice adhesion strength vs. surface roughness using **a** the horizontal push method, **b** the centrifugal method, and **c** the tensile method.

wettability, whereas the mechanical forces may add up on surfaces roughness above $0.5 \mu\text{m}$ due to a higher interfacial contact with the surface.

The surface roughness (R_a) of $0.5 \mu\text{m}$ is not a rigid value to evaluate the relationship of CAHs with surface roughness, rather an indication of a probable change in ice fracture mechanics. Bishoy et al. [30] indicated a modest dependence of ice adhesion strength in the surface roughness (RMS) range of $< 0.5 \mu\text{m}$ and reported a several-fold increase in adhesion strength with a larger surface roughness ($1\text{--}5 \mu\text{m}$). This critical surface roughness range ($< 0.5 \mu\text{m}$) might be linked to the detailed microstructures. Similar observations were also reported in other relevant work [31, 32]. Thus, the ice adhesion obtained on a surface with roughness below the critical range could be explained by CAHs, whereas the rapid increase in ice adhesion beyond the critical range may be attributed to the mechanical forces generated in the enhanced interfacial contact area.

Secondly, the ice adhesion correlated inversely with CAHs on tensile ice adhesion strength, contrary to many studies that suggest a direct relationship using shear ice adhesion methods [33, 34]. However, the tensile ice adhesion demonstrated a partial correlation with receding water contact angles over a certain range of surface roughness, with an exception of the sandblasted samples, as shown in supplementary figure S2 (g-i). It is imperative to mention that the ice adhesion increased systemically with an increase in surface roughness in the shear-based de-icing methods, whereas in the normal-based tensile method, the ice adhesion decreased with an increase in surface roughness at first and then the adhesion strength increased over a certain range of higher surface roughness. The roughening of surfaces may result in a change of spatial parameter (e.g. auto-correlation length), which links to the spacing of surface features. Davis et al. [35] reported similar findings on an air-pressurised tensile system and indicated that the ice adhesion correlated directly

Figure 3 Ice adhesion strength and CAH vs. surface roughness on **a** aluminium substrates, **b** SS substrates, and **c** TPU-BN coatings obtained using the NTM method.

with the spatial parameter, rather than a partial correlation with the arithmetic roughness.

The relationship of tensile ice adhesion with CAHs could be further explained in terms of the interfacial contact area and the subsequent change in surface wettability. The work required to break the ice interface or the work of adhesion on a tensile system is the product of surface energy and the interfacial contact area [16]. The CAH is an indicative measurement for surface energy, and Gao et al. [36] explained CAH from the perspective of kinematics. They reported the CAH values as a reflection of the activation energy and can be quantitatively expressed in terms of changes in interfacial free energy. However, this assumption is only valid for liquids on a perfectly smooth surface and may differ from a frozen liquid as solid adhesives. Thus, the smoothing of samples may alter the probability and/or intensity of surface wetting, which are heavily influenced by surface energy. Thus, the relationship between $F(x)$

(force required to break an ice/surface interface) and the work of adhesion W_a (the practical work calculated based on surface wettability) is not straightforward, but they are intrinsically linked to one another [37].

Ice adhesion strength of POTS coated surface

The idea of measuring ice adhesion on the samples having similar surface roughness but varying surface wettability was explored to understand the influence of the POTS-coated surfaces on the studied de-icing methods. Figure 4a shows the ice adhesion measured on the aluminium samples before and after the hydrophobic functionalisation. An increase in CAHs after the functionalisation of the metallic surfaces could be attributed to a higher interfacial contact and the surfaces are expected to be wetted in a Wenzel (or mixed with Cassie-Baxter) regime [38]. The results obtained on the aluminium samples indicate a

Figure 4 Ice adhesion before and after the surface functionalisation of **a** the SS-SB samples **b** Al-SB samples, and **c** TPU-5 coatings (Before Func.: incorporated with pristine hBNs, After Func.: incorporated with POTS functionalised hBNs).

reduction in ice adhesion across the de-icing methods. The ice adhesion obtained on the SS samples and TPU-BN coatings is summarised in Fig. 4b and c, respectively. The TPU-BN coatings were incorporated with either pristine hBNs particles or POTS functionalised hBNs particles for comparison purposes.

The results confirmed that the NTM method demonstrated a greater sensitivity to the surface functionalisation as compared to other de-icing methods, and indicated a significant reduction in ice adhesion strength after surface functionalisation. A dominant role of surface wettability (CAHs) on normal ice adhesion strength could be reflected. The HPM method was relatively insensitive to the change of the POTS coated surfaces. The ice adhesion increased after the surface functionalisation of hBNs nanoparticles incorporated in TPU-5 coatings. The tests indicated a clear dominance of mechanical forces over the influence of wetting behaviours in the cases of the HPM and the CAT methods and these methods might have only evaluated the mechanical

adhesion of ice. The dominance of mechanical forces might come from the stress concentrations that could have been generated due to the uneven distribution of shear and peeling forces as depicted in Fig. 5a. This may form the rationale behind the significant reduction in ice adhesion after hydrophobic functionalisation using the NTM method (as the mechanical interlocking of ice would have much less effect on perpendicular pull-up forces), whereas the mechanical forces-dominant systems, such as the CAT and the HPM methods, were less sensitive to wettability.

Fracture mechanism at ice/solid interfaces

Intrinsic shear strength or the nominal adhesional force per unit area is dependent on the true interfacial contact area and can be calculated by dividing the shear/normal force by the interfacial ice contact area. However, the loading on an interfacial contact area may not be uniformly distributed in different

Figure 5 **a** The mechanical forces around the surface asperities. **b** The forces acting on the ice rotating in a centrifuge. The mechanism and force schematics of de-icing methods: **c** horizontal

push method, **d** centrifugal method, **e** normal tensile method. (The figure was drawn using biorender.com).

evaluation methods and could jeopardise the accuracy of ice adhesion measurements. For example, an increase of 1.5-fold ice adhesion strength was reported on the push method as compared to that of the centrifugal method, with an uncertainty of 36% on an aluminium substrate at $-10\text{ }^{\circ}\text{C}$ temperature [2]. Additionally, in the case of microscopically rough surfaces, the chances of cohesive fracture are high due to the mechanical interlocking of ice on the surface asperities and the probability of a pure adhesional fracture is lowered [16]. Ice may rupture in two forms: an adhesive (mode I) or a cohesive (mode II) fracture. The fracture of ice depends on the magnitude of the applied load, the rate of loading, testing temperatures, and the type of ice formed/accumulated [39]. The interfacial stresses can be categorised into two distinctive types: the outward force or the peeling stress and the shear stress along the interface. The centrifugal and push methods develop the peeling stress at the edges of the ice sample, whereas the rest of the interfacial contact area is mainly dominated by the shear stresses [40, 41] as shown in Fig. 5, thus inducing a fracture in a combination of Mode I and Mode II failures. The stress concentration at the edges of ice/surface interfaces is severe in the case of the push methods as compared to the

centrifugal methods, as shown in Fig. 5c and d. The FEA analysis on the push method indicated strong stress concentrations at the interface and edges [17, 42]. However, stress concentrations at the interfaces alone do not provide a plausible explanation for the higher magnitude of ice adhesion strength measured on HPM methods. To the best of the authors' knowledge, researchers have so far not considered the effect of aerodynamic drag and vibrations on ice adhesion strength using CAT methods in a laboratory setup. The drag on the ice sample is depicted in Fig. 5b. The drag and vibrational forces generated while spinning in a centrifuge may explain the lowered ice adhesion strength measured using CAT methods, as it may aid an early release of ice. An attempt to minimise the vibrational forces was made in the experiments by placing a counterweight on the spinning arm in the CAT method.

In an NTM method, a uniform distribution of shear stresses might be approached on a tensile method [28] as the tensile method mainly involves the peeling stresses, the mechanism is illustrated in Fig. 5e. In addition to the fracture mechanism, the presence of liquid-like layers (LLL) at the ice/surface interface may further complicate the adhesive release of ice. LLL is known to aid the easy removal of ice by

inducing a slippery behaviour at the ice/solid interface [43]; however, the ice adhesion was mainly evaluated under a shear-based method. Goertz et al. [44] experimentally measured the adhesive response of LLL on the ice surfaces using a normal force mechanism, and they formed a bulk ice cube on a Peltier cooling system (similar to the system used in this study). They reported that at the temperature of $-10\text{ }^{\circ}\text{C}$, the thickness and adhesive force were maximised; however, the values of both properties decreased with the decreasing temperature (down to $-30\text{ }^{\circ}\text{C}$). Thus, there is a possibility of the LLL layers (the viscous and meniscus forces) working as an adhesive between the ice/solid interfaces, leading to an impact on the ice adhesion under a normal force mechanism, in addition to the increased affinity with higher wettability or increased interfacial contact due to a higher spatial parameter over a range of higher surface roughnesses [35].

The ice shearing methods, such as the CAT and HPM methods, produce a combination of shear and peeling forces, whereas the NTM method predominantly generates the peeling forces. A recent study indicated the compatibility between the HPM and the CAT methods [2]. However, the studies conducted by Schulz et al. [17] on the push method demonstrated irregular stress distributions at the ice/surface interfaces. Their FEA model suggested strong peeling forces and stress concentrations at the edges of ice bulk and uneven stress forces across the contact area, as depicted in Fig. 5a, whereas the interfacial stress on centrifugal methods were evenly distributed and could reach near-constant values provided the substrate would be an ideal rigid surface [17]. They also suggested that the influence of shear stresses was higher than that of the peel stresses under the FEA model assumptions, thus indicating the mechanical forces-centric nature of the shear methods. The phenomena discussed in this section could be used to explain the greater response of the CAT methods on surface roughness as compared to the HPM methods and might attribute to the even distribution of interfacial shear stresses on the CAT methods.

To conclude, an ideal de-icing evaluation test would involve a singular force that must promote the adhesional fracture. However, the complete isolation of de-icing forces is not realistic. Instead, the methods may involve a loading case where several stress components exist, but one dominates. The NTM method is one of the analytically straightforward

stress states and offers a dominant component of peeling forces. However, the NTM method is likely to induce a cohesive failure, influenced by the wetting conditions, and subsequently, the possible influence of LLL layers. Furthermore, the selection of a shear method would be practical for de-icing evaluation as the shear forces are useful for mimicking forces present in applications requiring de-icing. For example, if the ice is removed by aerodynamic force alone, then the largest stress component for such a loading would be in shear [19]. In terms of shear methods, HPM method is simple and cost-effective. However, the transverse shear method is not designed to create uniform stress at the interface and a constant force on one side may not create a single uniform force at the interface. The method may also induce strong stress concentrations in the interface region, and produce uneven and/or ununiformed distribution of interfacial shear forces [15]. The results obtained in this study also indicate that the shear-based methods are sensitive to roughness asperities, and the ice anchoring or mechanical interlocking of ice seems to be the main factors to reflect the influence of wettability on ice adhesion over a certain range of surface roughness. The HPM and NTM methods may also not be suitable for impact or sprayed de-icing evaluation. The use of the CAT method makes a better case for de-icing methods as they are appropriately sensitive to both the mechanical and wettability conditions. The CAT is also the most repeatable ice adhesion test, has a higher probability of adhesive failure, and is ideal for large components and different types of ice formations, such as spray, accumulated, and bulk formations [1, 15]. Furthermore, Work et al. [1] indicated an average variation of 14.7%, 18.7%, and 24.1% for CAT, HPM, and NTM on aluminium samples, respectively. Thus, the CAT method offers a small variation in results as compared to that of the HPM and NTM methods. The CAT method also offers a dominant component of shear forces than the peeling forces and the shear stress are almost constant in the interface [17]. However, it is imperative to mention that the CAT method is complicated to evaluate the relationship of strain rate and adhesion strength and is expensive to set, and the influence of aerodynamic drag and vibrational forces is yet unknown [1].

Effect of heating at the ice/solid interfaces

Several researchers have also examined the use of active support in combination with a passive system to aid the de-icing process [45, 46]. The idea is also an active field of research in aerospace and wind energy industries as the energy-saving alternatives to current energy-intensive de-icing methods. In this section, the studied samples, which demonstrated considerable sensitivity to surface roughness and/or wettability using the mechanical de-icing methods, were examined to understand the influence of heating potential at the ice/surface interfacial contact area under the gravitational pull, but without the influence of a detaching mechanical shear/normal force. Theoretically, a shear force could be generated at the ice/surface interfaces by gravity as the sample was held perpendicularly to the surface. Preliminary tests were conducted to investigate if the sample surface characteristics have any impact on the duration of ice heating melt tests. It is imperative to mention that the substrates' or coatings' thermal and electrical conductance might influence the test readings. The coating thickness in the case of polymeric coatings may also produce a thermal gradient across the coating types. Thus, the tests were purely inductive and direct comparisons could not be drawn to compare the heating de-icing mechanism to passive de-icing methods.

Schaaf et al. [47] conducted various tensile ice adhesion measurements on samples having different surface roughness and the interfacial contact area was heated with an induction heating method. They indicated that the smoother surfaces needed a less debonding energy under a similar tension loading at a temperature of -6°C . In this paper, the constant heating potential was supplied at the interface and the ambient temperature was controlled throughout the tests. No external force was exerted on the ice cube, and the heating potential was continued until the ice cube dropped under the influence of gravity. The heating time (time to detach the ice cube), alongside ice adhesion measured on the centrifugal method, on the SS samples is shown in Fig. 6a. The results obtained on the aluminium samples and TPU-hBN coatings are summarised in Fig. 6b and c, respectively. These figures indicate a partial correlation of the heating time with surface roughness (R_a) below $0.5\text{ }\mu\text{m}$. Furthermore, the time to detach the ice

cube on all the studied samples were in the range of 530–700 s.

Variation in heating time suggest that the total initial ice adhesion force F on ice/surface interface may have changed and the residual adhesion force F_o can be calculated as [48]:

$$F_o = (F - F_m) \cdot K \quad (3)$$

where F_m is the ice adhesion deducted at the melting part and K is the modification coefficient. The value of K is dependent on the surface material, surface morphology, ice uniformity, and the adhesion state. The heat energy Q at the interfacial area can be mathematically calculated as [48]:

$$Q = \frac{U^2}{R} t \quad (4)$$

where t is the heating time, R is the resistance value of the heating material, and U is the power supply voltage. The ice heating melt tests conducted in this study, R and U were constant, thus $Q \propto t$ can be assumed.

The results of ice heating melt tests could be explained using Eqs. 3 and 4. The results indicated a partial but direct correlation with surface roughness, which could be explained using the modification coefficient K as the coefficient is dependent on surface morphology, whereas the heat energy required to melt the ice interface could be approximated using the heating time. Thus, the heating period required to detach the ice could be up to 530 s, whereas further delay (up to 700 s) in the separation of ice bulk might come from the influence of surface characteristics.

Conclusions

One of the promising strategies to initiate the standardisation of de-icing evaluation methods includes interlaboratory collaborations that offer to cross-validate the results measured using different test rigs. An interlaboratory examination was carried in this study, focusing on the influence of surface characteristics in the de-icing evaluation. The evaluation methods included the horizontal shear-based method (HPM), centrifugal shear-based method (CAT), tensile normal-based method (NTM), and electro-thermal de-icing evaluation (without external forces). The paper highlights significant discrepancies and/or differences present among the icephobicity

Figure 6 Ice heating melt tests on **a** the SS substrates, **b** the aluminium substrates, and **c** TPU-BN coatings.

evaluation methods; however, the work also offers several correlations, based on surface characteristics, which could be exploited for the meaningful development of standardised de-icing evaluation methods.

In terms of the mechanical de-icing methods, the ice adhesion obtained on HPM and CAT suggests a linear relationship with surface roughness, whereas NTM and the electro-thermal de-icing evaluation demonstrated a correlation over a certain range of surface roughness. Furthermore, the NTM method indicated an inverse curvilinear relationship with contact angle hysteresis. The surface functionalisation of rougher metallic substrates demonstrated that the shear-based methods were appropriately insensitive to a change in surface wettability, highlighting the mechanical forces-centric nature of shear-based methods.

The results obtained in this study indicate the CAT method makes a better case for de-icing evaluation as they are appropriately sensitive to both the mechanical and wetting conditions. At last, the variations in ice adhesion response with the surface characteristics

investigated in this study offer crucial information on the pre-selection of testing mechanisms.

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Declarations

Conflict of interest There are no conflicts to declare.

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