Icephobic Material Centrifuge Adhesion Test

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***Abstract*— Ice accumulation is a transportation problem for roads, boats, airplanes transmission lines, etc. An icephobic coating applied to exposed surfaces appears to be an interesting solution to prevent ice build-up; however, no coating is perfectly icephobic as ice sticks to everything. Still, some materials that reduce ice adhesion have been developed from which the ice can be more easily shed, possibly even with existing forces such as wind, gravity and vibrations. A method was therefore investigated to measure the ice adhesion and the reduction thereof. The method must be able to measure the force required to separate the ice from the substrate, i.e. adhesive failure and be a comparative method. Existing tests use traction or compressive forces, however, for such, the ice can only be made by freezing water as a block which leads to highly variable results: up to 300% error. To solve these limitations the Anti-icing Material International Laboratory (AMIL) developed the Centrifuge Adhesion Test. The test involves icing the extremity of small beams under supercooled precipitation. After icing, the beams are balanced in a centrifuge and rotated at an accelerating speed until the ice detaches. The ice detachment is picked up by piezoelectric cells located in the vat wall. The ice adhesion shear stress is then calculated from the ice detachment rotation speed. The results are then reported as an Adhesion Reduction Factor, which is the ratio of the adhesion shear stress of the beams with a candidate coating with respect to a simultaneously iced uncoated beam. Therefore, a bare beam would have a factor of one, the higher the factor the more icephobic the coating and values below one indicate and increase in adhesion. The advantages of this method include: the fact that the ice is made from supercooled precipitation and is representative# of atmospheric icing; the small ice samples allow for a homogeneous ice coupon which improves the repeatability of the results; the flat ice surface allows for modifications to the shape of the surface (e.g. cylinders and airfoils) and exposure of the surface to environmental elements.**

I. Introduction

Ice accumulation is well known to be a transportation problem for roads, boats, airplanes and transmission lines. Ideally, a coating would exist that would prevent ice from adhering to structures; unfortunately, ice sticks to everything. Still, some materials that reduce ice adhesion have been developed from which the ice can be more easily shed, by wind, gravity and vibrations. In order to measure the effectiveness of such a coating a method is needed to measure the force to separate the ice from the substrate, the adhesive failure, and the method should be repeatable, and comparative, to be able to evaluate the different coatings.

A number of methods of measuring ice adhesion and evaluating coatings exist and are commercially offered. Most adherence tests consist of applying a force, either in tension or compression, producing a shear stress on the ice sandwiched between two substrates. The ice sample may be cylindrical [1,2], or rectangular [3]. Less commonly the stress may be induced by torsion [4], flexion [5], peeling [6] and centrifugal [7] forces.

These methods are limited by the fact that the ice is made from large samples of freezer made frozen water, and consequently an inhomogeneous glaze ice, not accreted ice. Not only is this ice unrepresentative of atmospheric ice, it can lead to highly variable results, with up to 300% variation.

To solve these limitations accreted ice in the form of freezing precipitation, under highly controlled conditions is required. Small ice coupons for a more homogenous would also improve the repeatability. Any test would also be comparative, where the ice adhesion, or reduction thereof, would be only evaluated on coated and uncoated surfaces simultaneously iced, since small variations in the ice cannot be entirely eliminated.

II. The Centrifuge Adhesion Test (CAT)

The Centrifuge Adhesion Test (CAT) was developed at the Anit-icing Materials Laboratory (AMIL) of the Université du Québec à Chicoutimi (UQAC) to address these limitations. Roughly, the tests consists of artificially icing the extremity of small beams and spinning them in a centrifuge to determine the speed at which the ice detaches.

A. CAT Beams

The tests are normally conducted on 3.2 wide, 0.6 mm thick, aluminum flat bar which is then cut to a 340 mm length (Figure 1). The small aluminum beams are either solid for the reference bare beams and for a coating that is applied overtop, or with a hollowed-out area for a coating which would be embedded (Figure 2).

A counter weight is fixed to the other extremity of the beam with about the same weight as the ice to balance the beam in the centrifuge.

Figure 1 – Test beam

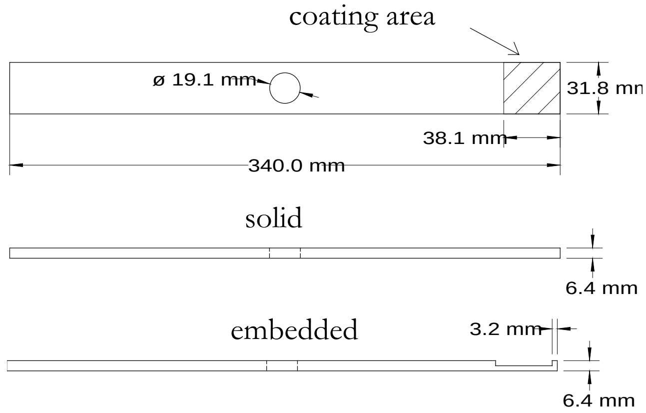

Figure 2 – Test beam dimensions

B. CAT icing

Prior to icing, the beams are weighed and a thermocouple fixed to the base of one beam. Seven beams are simultaneously iced, three of which are bare, three coated with a candidate icephobic coating, and one is used for ice density measurements (Figure 3).

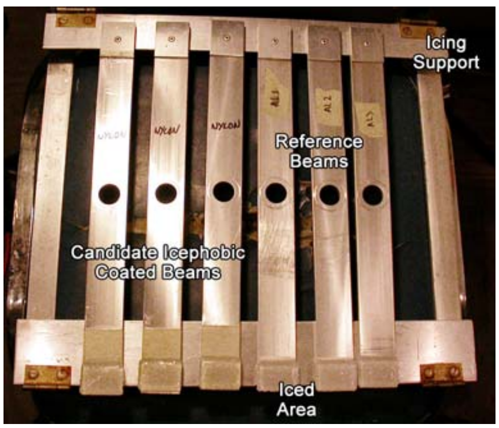

Figure 3 – Six beams simultaneously iced

The beams are placed on a support in a cold room (Figure 4) at –8.0 °C ± 0.1. When the beams have reached the test temperature, they are exposed to freezing drizzle with a mean volumetric diameter, MVD, of about 200 µm. An ice thickness of about 1 cm is accreted, with a density of 0.87 g/cm³ ± 0.02. The beams are weighed following icing and ice density measurements are made on the seventh beam.

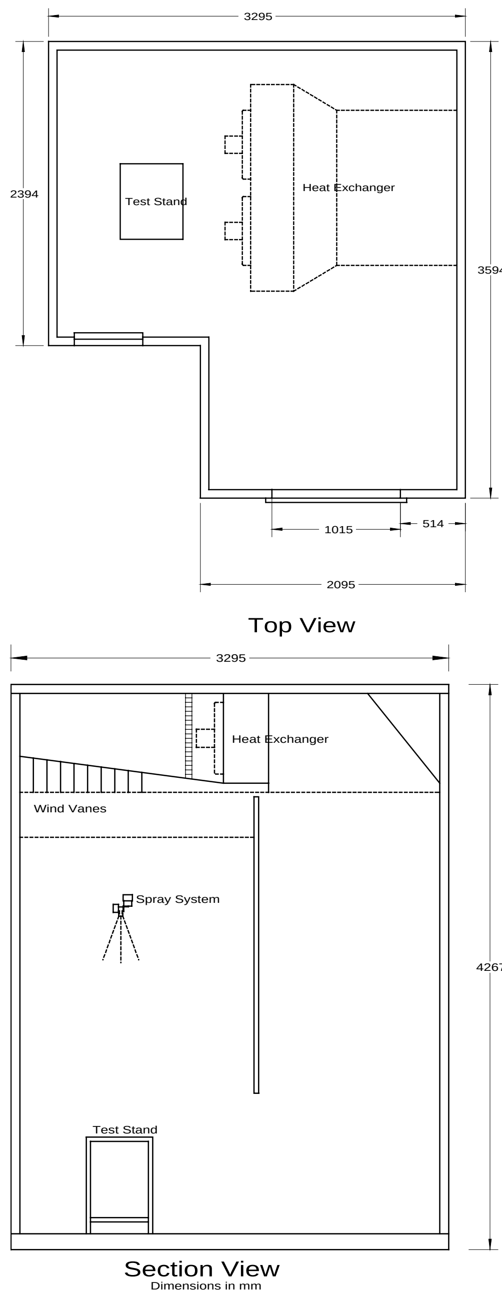

Figure 4 – AMIL's Freezing Drizzle Chamber

C. CAT Centrifuge

The iced beams (Figure 5) are individually tested in the centrifuge vat (Figure 6) placed in a climatic chamber at –10.0 °C ± 0.1. There, the beams are rotated at increasing speeds from 0 to the detachment speed at a constant and controlled acceleration of 300 rpm/s equivalent to a strain rate about 10^{-6} s^{-1}. The whole set-up is computer controlled (Figure 7) .

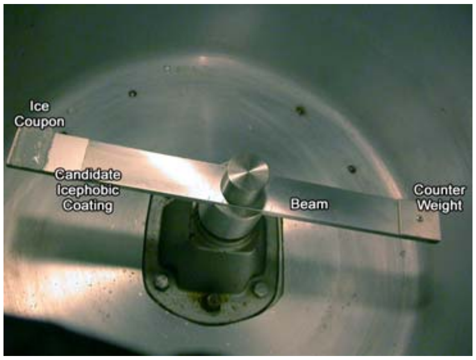

Figure 5 – Test beam in centrifuge

Figure 6 – Centrifuge test set-up, contained in the cold room of Figure 4.

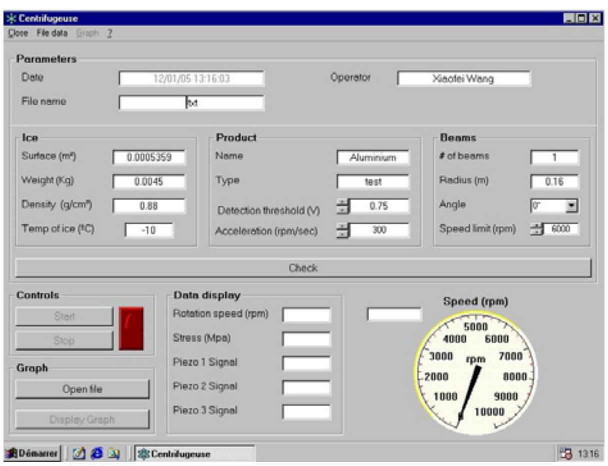

Figure 7 – Centrifuge Adhesion Test (CAT) computer program interface

The centrifugal force resulting from the rotation tends to detach the ice layer. When this force reaches that of the adherence of the ice, the ice detaches. The detachment of the ice is picked up by two piezoelectric cells sensitive to vibrations fixed to the sides of centrifuge vat which relay their signal in real time to a computer (Figure 8). With this signal, the rotation speed reached at the detachment is determined. The tests usually run from 2 to 20 seconds depending on coating. If a rare cohesive break occurs, the test automatically stops as the first piece of ice touches the vat wall and causes vibrations. The test is then rejected and repeated.

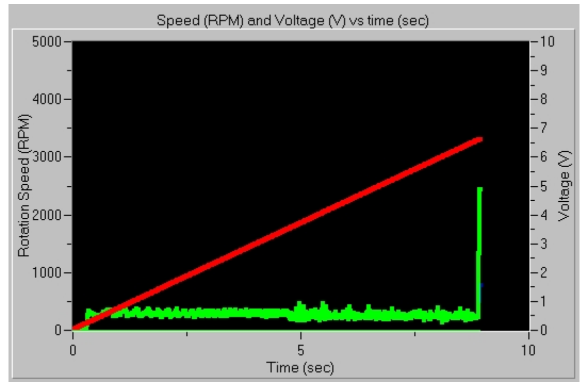

Figure 8 – Data recorded during centrifuge tests, in red is the rotation speed which increases linearly, in green and blue are the piezoelectric cells which pick up the ice detachment and stop the centrifuge after about 8 sec and 3500 rpm for this example.

D. *Calculation of Shear Stress*

Using the speed of rotation at the ice detachment, the adherence force is calculated using the ice mass and beam length as follows:

$$F = mr\omega^2 \qquad [1]$$

where:
F = centrifugal force [N]
m = mass of ice [kg]
r = radius of the beam [m]
ω = speed of rotation [rad/s]

From the centrifugal force, the shear stress is determined:

$$\tau = \frac{F}{A} \qquad [2]$$

where:
A = Area iced [m²]
τ = shear stress [Pa]

The Adhesion Reduction Factor is then calculated using the average shear stress measured on the three coated beams compared to the average stress measured on the three bare beams which were, all six, simultaneously iced:

$$\text{Adhesion Reduction Factor} = \frac{\text{average shear stress measured on the three bare beams}}{\text{average shear stress measured on the coated beams}}$$

i.e.

$$ARF = \frac{\tau_{bare}}{\tau_{coated}} \qquad [3]$$

where:
ARF = Adhesion Reduction Factor
τ_{bare} = average shear stress measured on 3 simultaneously iced bare beams [Pa]
τ_{coated} = average shear stress measured on 3 simultaneously iced beams with candidate icephobic coating [Pa]

As such, a bare aluminum beam would have an Adhesion Reduction Factor of 1; a number less than one indicates an increase in adhesion with respect to bare aluminum. The higher the number, the more icephobic the coating.

III. Results and Discussion

To evaluate the performance and validity of the instrument, many tests were performed on aluminum. The absolute ice adherence on aluminum is hard to compare between authors since it depends on many parameters such as temperature, deformation rate and ice type. Measured under the conditions of this test the ice adhesion on 6061 T6 polished aluminum is in the order of 0.35 Mpa. This is comparable to the results of of Jellinek. [1], who measured 0.32 MPa under similar test conditions (temperature and strain rate) but with frozen water (not accreted). The failure of the ice most often observed was adhesive (Figure 9).

Figure 9 – Photograph of the aluminum beam test surface following a centrifuge test. Note, the ice block is entirely removed indicating an adhesive failure

Tests were performed to evaluate the sensitivity of the machine to different coatings. As such, some commercially available coatings were evaluated with the current CAT test protocol and are presented in Figure 10.

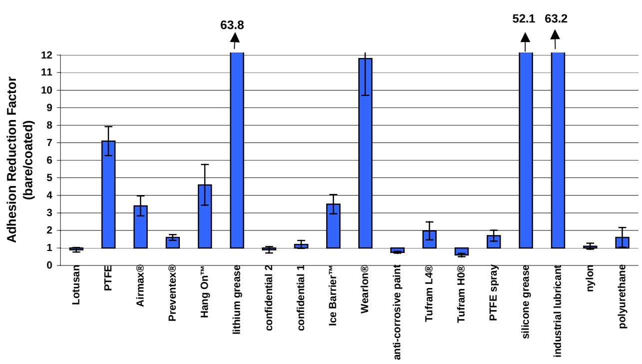

Figure 10 – Adhesion Reduction Factors of some commercial and developmental coatings. An ARF of 1 indicates no reduction in ice adhesion of the coating, the higher the value, the more icephobic; values less than 1 indicate an increase in ice adhesion as compared to bare aluminum.

The spread of the different Adhesion Reduction Factors show that the set-up is sensitive to the different coatings studied. For example, the results show that the greases and non-permanent coatings are considerably more effective than the solid coatings, which confirm the results of Laforte *et al.* [10]. The average standard deviation is about 18%.

IV. Conclusions

The Centrifuge adhesion test can measure the reduction of ice adhesion of candidate icephobic materials. Its principle advantages include:

- Simple test: it can be performed inexpensively and timely
- Repeatable: standard deviation of about 18%
- Accreted ice: more representative of atmospheric ice
- More homogeneous ice because of the small ice coupons
- Real time data collection
- Easily adaptable for other shapes, such as cylinders and airfoils
- Flat surface allows for exposure to environmental elements and repeat testing

V. Future Studies

Future studies include more evaluations and comparisons of candidate icephobic coatings. Also the set-up will be modified for different beam shapes and specific applications, e.g. airfoils and cylinders.

VI. Acknowledgements

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