Ice adhesion Measurements: Assessing the effect of icing conditions

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Abstract— **Minimizing the effect of icing on numerous structures such as airplanes, helicopters, wind turbines, bridges, pylons, and even solar panels remains relevant in present time. Icephobic coating design relies mainly on preventing ice from accumulating or reducing its adhesion. When the ice adhesion is sufficiently reduced, the shedding occurs more easily. Thermal and mechanical methods, centrifugal force, natural vibrations, gravity and wind may then be sufficient to remove ice. However, to assess the icephobic coating behaviour, the evaluation should not be limited to only one type of icing but rather to study the various scenarios that can be encountered during the winter period for both in-flight and ground icing. Ice formed in clouds versus on the ground does not have the same physical and mechanical properties so the study of their adhesion under different conditions is of interest.**

The objective of this paper is to present a comparative analysis of the accreted ice adhesion measurements under artificial icing conditions. In this study, three types of icing were investigated; hard rime obtained under a freezing drizzle precipitation in a cold room (10 to 15 g/h) at -8°C, without wind conditions and a median volumetric diameter (MVD) of 327 μm (CATZL), glaze obtained in an icing wind tunnel at -7.5°C, with a wind speed of 55 m/s, with an MVD between 20-30 μm and an LWC of 0.9 ± 0.1 g/m3 (CATWT-Glaze) and rime obtained in an icing wind tunnel at a temperature of -15°C, with a wind of 50 m/s, with an MVD between 20-30 μm and an LWC of 0.3 ± 0.1 g/m3 (CATWT-Rime). Thirteen (13) different candidate coatings were evaluated in this study. All candidates were evaluated using the Centrifuge Ice Adhesion Test (CAT). A total of 45 CAT series were performed; fifteen (15) in CATZL, fifteen (15) in CATWT Glaze and fifteen (15) in CATWT Rime. This represents a total of 540 individual centrifuge tests. The ice adhesion measurements show higher values for the ice accumulated in the cold room than in the wind tunnel. In light of these results, in-flight icing in the wind tunnel with smaller droplet diameter and higher impact velocity shows a significant decrease in ice adhesion for both LWC conditions tested. On the other hand, a well performing coating under cold room conditions does not necessarily translate to good performances in the wind tunnel. The results obtained in this study indicate that coatings are not optimal under all conditions and that the design of a coating must be based on its intended application and the conditions it may encounter.

Keywords— ***Ice Adhesion, Coating, Rime, Glaze, Hard Rime, Atmospheric Icing, Wind Tunnel, Centrifuge, Climatic Cold Room***

I. Introduction

The protection of structures in winter is a concern for all designers, manufacturers, and users in the world of transportation as well as in fixed installations. Whether it's helicopters, airplanes, ships, railroad tracks, wind turbines, bridge structures, solar panels, or telecommunication towers and pylons, they all have their problems related to icing. In order to improve the protection of the structures, active or passive solutions are investigated [1, 2]. The active solutions can be mechanical [3], thermal [4] or chemical [5], while the passive solutions are mainly in the form of protective coating or surface texturing [4, 6-8]. While the passive solution can minimize the impact that ice can generate on those different structures [9], without any energy consumption, to improve those solutions, it is still important to deepen the knowledge and understanding of ice adhesion.

Several studies have been conducted to present different results of ice adhesion [6, 9-24]. It is important to remember that ice adhesion can be affected by multiple parameters. These parameters could be divided into three main categories: the substrate, the type of icing, and the measurement technique. The substrate can be described by its roughness, wettability, heat transfer, a possible phase change and hardness. As for the type of icing, (i.e. ground icing, in-flight icing, etc), the principal parameters are the air and water droplets temperature, the impact speed of the droplets, their diameter, the accretion rate, and the temperature variations during precipitation to consider. Finally, the measurement techniques for ice adhesion should also be considered [7, 17, 25], whether in-situ or ex-situ, the presence of normal, shear or mixed stress, bending, compression, tension or rotation methods, and adhesive versus cohesive fracture analysis. All these parameters can influence the results obtained.

This work presents a screening campaign aiming at comparing the adhesion shear stress of a wide variety of products in order to find the best way forward in obtaining a low adhesion product for ice protection. This test campaign is run for three different types of icing; a typical ground icing precipitation and the other two defined as in-flight conditions. A centrifugal force adhesion measurement technique, where the stress is pure shear, and fracture adhesive, is used. The results of this test campaign are presented according to the type of application method used to apply the coatings on the substrates. This paper stems from an industrial request and the test samples other than those based on polyurethane were provided by the manufacturer of the coatings and are identified by number to preserve confidentiality. Those results will allow to identify what products are most promising in order to pursue investigating coating as an ideal ice protection system. In addition, these results will allow to analyze the presence or not of a clear trend between different icing conditions for the same candidate coating.

II. Material

In this study, thirteen different coatings were applied on two different types of substrates:

A. *Aluminum*

The tests are performed using 32 mm wide x 6.4 mm thick aluminum 6061-T6 flat bars cut to a 340 mm length (Fig. 1). The Roughness and Contact Angle of the aluminium are 0.6 ± 0.2 µm and 75 ± 5°.

Fig. 1 Conventional Test Sample.

B. *Titanium*

Test coupons were fixed on the aluminum samples as shown at pictures (Fig. 2). The roughness and contact angle of the titanium surface are 0.4 ± 0.2 µm and 87 ± 7 °. The titanium substrate (T6AV) was used when the product could not be applied on an aluminium substrate, mainly due to the application method and surface preparation.

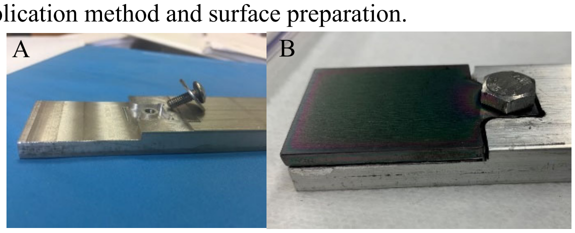

Fig. 2 CAT Sample (A) and Embedded Test Coupon (T6AV) (B).

III. Application Methods

In order to evaluate the variation in the results, six samples per candidate products were selected. Coatings are subdivided according to their method of application.

Depending on the base of the coating, there are several ways to apply the coatings. Each method of coating is designed to fully coat the substrate in the protective coating material. However, everything from the substrate's size to its complexity impacts what coating application method is the right fit.

In this study, six organics (four polyurethane based, one polymer based, and one DLC-F based), and seven inorganics-based coatings were used which were applied by four various methods to evaluate the effect of icing conditions on ice adhesion. Organic coatings are often liquid or powder coatings that require several layers before a thick enough coating to protect substrate. These coatings are based on carbon containing materials normally derived from refined and/or modified petroleum products, as well as different solvents/water, pigments, additives, and fillers. Below are four organic coatings commonly used in industrial applications: **polyurethane, epoxy, alkyd etc.** Inorganic coatings encompass surface conversion, anodizing, enameling, metallic coatings and more. These coatings are created through a chemical action that changes the surface layer of metal into a metallic oxide film or compound to protect substrate.

A. *Spraying Technique*

Spraying is the most prevalent paints and coatings application technique among industrial manufacturers due to its versatility and economic viability. Conventional spray is a universal method to apply different types of coatings to large area. This method of spraying relies on air for coating atomization. The air spray machine is simple and cheap, whereas the volume, pressure, and flow rate of the air must be appropriate to obtain a good film appearance. Most industrial coating materials can be applied using a conventional spray. Different size of tips can be selected for more viscous, mastic-type coatings. The coating manufacturer often recommends application equipment and will specify tip sizes for optimum application characteristics. Because larger amounts of air are mixed with the coating during application using spray, coating losses from "bounce back" or "overspray material" that miss the substrate can be high, depending on the configuration of the substrate. This bounce back effect makes coating corners and crevices difficult. In terms of air spray coating, the most unfavorable condition is that it cannot be applied thickly with one coat because it needs to be diluted with a solvent to adjust the viscosity of the paint or coating [26].

Eight candidate coatings were applied using a spray technique, with atomized air. In this study, four various Polyurethane based clear coatings with different formulations were used. Due to the fact that PU coatings have a lot of polar groups on the top of their surface, PU based coatings are rather hydrophilic in nature such as PUC-1 (Table I). Therefore, achieving hydrophobic and icephobic PU based coatings remains an important issue for research and development. One of the useful approaches for converting PU coatings to hydrophobic coatings is the use of active additives (siloxane or fluorinated based additives) to modified surface chemistry. PUC-2, PUC-3 and PUC-4 are such example of hydrophobic PU coatings.

The identification of Polyurethane-based coatings is presented in Table I.

TABLE I. Polyurethane based candidate samples

Candidate samples	**Substrate**	**Roughness (µm)**	**Contact Angle**
PUC-1	Aluminum	0.2 ± 0.1	*77° ± 6 (hydrophilic)*
PUC-2	Aluminum	0.2 ± 0.1	*100° ± 1 (hydrophobic)*
PUC-3	Aluminum	0.2 ± 0.1	*99° ± 2 (hydrophobic)*
PUC-4	Aluminum	0.2 ± 0.1	*99° ± 2 (hydrophobic)*

Four other coatings applied by the spraying technique were also supplied by a manufacturer. These required curing in an oven. Three coatings were developed using the Sol-Gel process [27, 28] and one with a silicone based. The composition of the coatings and process parameters are confidential. The identification of candidate samples is presented in Table II and III.

TABLE II. SOL-GEL CANDIDATE SAMPLES

Candidate samples	Substrate	Roughness (μm)	Contact Angle
SGC-1	Aluminum	0.2 ± 0.1	*91° ± 2 (hydrophobic)*
SGC-2	Aluminum	0.5 ± 0.2	*114° ± 1 (hydrophobic)*
SGC-3	Aluminum	0.2 ± 0.0	*63° ± 5 (hydrophilic)*

TABLE III. SILICONE CANDIDATE SAMPLES

Candidate samples	Substrate	Roughness (μm)	Contact Angle
SI-1	Aluminum	0.6 ± 0.4	*107° ± 2 (hydrophobic)*

B. Film Applicator "Bar-coater"

This method is suited to materials with a viscosity range from about 1 to 1000 centipoise, subject to them flowing out after coating. The actual deposit from a wound bar will depend on the absorbency of the substrate and the flow characteristics of the coating material [29].

Only a polymer coating was applied to the two substrates, aluminum and titanium using a bar coater. The identification of polymer coatings is presented in Table IV.

TABLE IV. TEXTURED POLYMER CANDIDATE SAMPLES

Candidate samples	Substrate	Roughness (μm)	Contact Angle
TPC-1	Aluminum	2.6 ± 0.5	*82° ± 7 (hydrophilic)*
TPC-2	Titanium	2.8 ± 0.7	*101° ± 5 (hydrophobic)*

C. Physical Vapor Deposition Process (PVD)

Physical vapor deposition technique (PVD), which uses evaporation, sputtering, and other physical processes to produce vapors of materials instead of chemical processes, is characterized by a process in which the material goes from a condensed phase to a vapor phase and then back to a thin film condensed phase. The PVD is used in the manufacture of items which require thin films for mechanical, optical, chemical or electronic functions. The PVD technique, however, has some drawbacks, such as low deposition rate and low-pressure requirements. Moreover, this process might require subsequent annealing, which could be a drawback [26].

Three candidate coatings were prepared by the PVD process. All of them were deposited by a cathodic arc source. They are presented in Table V. The PVD-1 is a multilayer CrN coating applied only on the Titanium substrate. Regarding the PVD-2 and PVD-3, they were applied on titanium and aluminum substrates, both are aluminum-titanium nitride coating (AlTiN).

TABLE V. CANDIDATE SAMPLES

Candidate samples	Substrate	Roughness (μm)	Contact Angle
PVD-1	Titanium	0.4 ± 0.1	*92° ± 2 (hydrophobic)*
PVD-2	Titanium	0.5 ± 0.1	*98° ± 5 (hydrophobic)*
PVD-3	Aluminum	1.0 ± 0.3	*49° ± 1 (hydrophilic)*

D. Plasma-Assisted Chemical Vapor Deposition Process (PACVD)

PACVD is a widely used technique to obtain device quality thin films at low substrate temperatures. PACVD is a chemical vapor deposition process used to deposit thin films from a gas state (vapor) to a solid state on a substrate. Chemical reactions are involved in the process, which occur after creation of a plasma of the reacting gases. In PACVD, source gases are decomposed in plasma by the collisions between energetic electrons and gas molecules. This process has been developed for the semiconductor industry and is extensively used in microelectronics applications. This process has been adapted to the solar industry and it is a key deposition technique used in the manufacture of industrial silicon solar cells [26].

This coating is applied by Plasma assisted chemical vapor deposition. The composition of this coating is Fluorinated Diamond-Like Carbon (F-DLC) films deposited on Titanium substrate. The identification of this coating is presented in Table VI. The incorporation of F content can enhance the hydrophobicity of the coating [30].

TABLE VI. CANDIDATE SAMPLES

Candidate samples	Substrate	Roughness (μm)	Contact Angle
PACVD-1	Titanium	0.3 ± 0.1	*96° ± 2 (hydrophobic)*

IV. MEASUREMENTS METHODS

A. Wetting Characterization

The water contact angle (WCA) of a liquid drop on a solid depends on the chemical affinity between them and the surrounding gaz. When the chemical affinity is high, it will tend to flatten on the solid, to wet it. A material easily wettable by water is qualified as hydrophilic (WCA < 90°) while a hardly wettable material is qualified as hydrophobic (WCA > 90°). The higher the contact angle, the higher the surface is hydrophobic, while above 150° it is called a super hydrophobic surface. The wetting properties of a coating are evaluated by measuring static water contact angles WCA. The contact angle is measured using a goniometer at five positions on each substrate using a 5 μL deionized water drop at ambient temperature accurate to ± 2°.

B. Surface Profilometry

The calibrated apparatus used for roughness measurement is a Surtronic 25 profilometer. The average roughness, expressed as Ra, has been measured on the coated sample at three different directions.

C. Centrifuge Ice Adhesion Testing

In order to quantify, in detail, the surface's icephobic behavior, the centrifugal force measurement method (CAT) commonly used at the laboratory to measure ice adhesion have been carried out.

The Centrifuge Ice Adhesion Test (CAT) consists of a two-step procedure where the extremities of samples are iced, and then rotated in a centrifuge developed at the Anti-Icing Material International Laboratory (AMIL) [31] to evaluate the ice adhesion on the substrate.

The ice adhesion strength is the centrifugal shear stress exerted on the ice sample at the time of the detachment. A conventional CAT sampling consists of twelve samples; six of the candidate materials compared to six reference samples. The candidate materials are rotated at an acceleration speed of 300 rpm/s until the ice shed under the centrifugal force. The rotational speed is controlled during the acceleration phase by the AMIL-made software. The software records and plots the angular velocity in real time with data from the sensors used during the tests. An accelerometer sensor located on the wall of the centrifuge is used to determine the exact ice detachment time. This time corresponds to a sharp increase in the vibration signal. The centrifugal force is calculated from the ice detachment velocity, the ice mass and the beam length at the center of the ice coupon (Eq. 1).

$$F = mr\omega^2 \qquad \text{(Eq. 1)}$$

where:

F = centrifugal force (N)
m = mass of ice (kg)
r = sample radius (m)
ω = rotation speed (rad/s)

The shear stress (Eq. 2) is obtained by dividing the centrifugal force by the area of ice in contact with the material:

$$\tau = \frac{F}{A} \qquad \text{(Eq. 2)}$$

where:

τ = bulk shear stress (MPa)
F = centrifugal force (N)
A = ice detachment area (mm²) (the ice surface is measured before and after the test).

V. Icing Tests

Three different conditions are performed in this comprehensive test campaign. These conditions were selected since they are key conditions in the different industry standards, for ground or in-flight icing, and used in most studies in the literature.

A. Icing in Wind Tunnel

The samples are installed into the wind tunnel for ice accumulation. Following the icing, the samples are carried (in an isolated box) to the cold room maintained at -the same air temperature of icing where they are kept for one hour before testing.

For rime conditions the parameters are: an air speed of 50.0 ± 1 m/s, an air temperature of -15 ± 0.5°C and a liquid water contents (LWC) of 0.3 ± 0.1 g/m³. For the glaze icing conditions, the parameters are: an air speed of 55 ± 1 m/s, an air temperature of -7.5 ± 0.5°C and a liquid water contents (LWC) of 0.9 ± 0.1 g/m³ (Table VII).

TABLE VII. Icing Parameters for Wind Tunnel (CATWT)

Ice Type	Air Temperature (°C)	Air Speed (m/s)	LWC (g/m³)	MVD (µm)
Glaze	-7.5	55	0.9 ± 0.1	20 - 30
Rime	-15	50	0.3 ± 0.1	20 - 30

A typical ice deposit formed on the samples is shown in Fig. 3 for rime conditions, and is shown in Fig. 4 for glaze conditions.

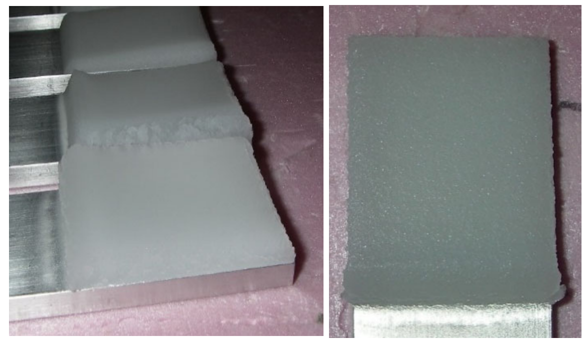

Fig. 3 Typical Ice Deposit in Wind Tunnel at -15°C with Wind Speed set to 50 m/s

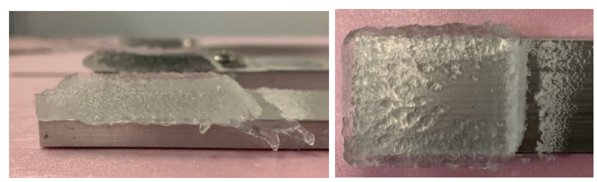

Fig. 4 Typical Ice Deposit in Wind Tunnel at -7.5°C with Wind Speed set to 55 m/s

B. Icing in Cold Room

The extremity of the twelve samples (six coated, six bare samples) are iced simultaneously in the climatic chamber at -8.0 ± 0.2°C with freezing drizzle (ZL) (shown in Table VIII), to obtain a hard rime from water droplets with a median volumetric diameter (MVD) of 327 µm on a 1100 ± 70 mm2 surface and a thickness of around 7 ± 1 mm. Droplet speed corresponds to their free-fall values in the air vertical flow. The samples are iced for about 35 minutes to obtain 5.5 ± 0.5 g of ice. One hour following icing, the iced samples are individually tested in the centrifuge in a climatic chamber at -10.0 ± 0.2°C.

TABLE VIII. Icing Parameters for Cold Room (CATZL)

Ice Type	Air Temperature (°C)	MVD (µm)
Hard Rime	-8 / -10	327

A typical ice deposit formed on the samples as shown in Fig. 5 for hard rime (ZL) conditions.

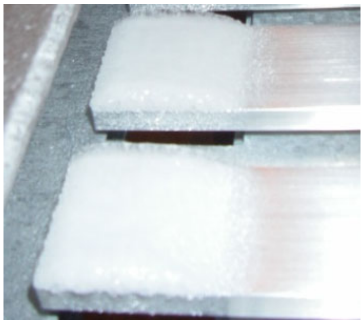

Fig. 5 Typical Ice Deposit in Cold Room at -8°C

VI. Results and Discussion

The average ice adhesion obtained on reference samples (aluminum and titanium) has been calculated using all samples tested for each series of testing. A total of 85 individual centrifuge tests were performed on aluminum uncoated substrates for rime in wind tunnel conditions, 126 for glaze in wind tunnel conditions and 78 for hard rime in cold room. For the titanium uncoated substrate, 7 tests were performed under the Rime ice condition, 10 for the Glaze ice conditions and 4 in the cold room. The fact that the bare substrate was retested at the same time as each of the coating explains these high numbers.

A. Icing Conditions

1) Rime in Wind tunnel

The results obtained on aluminium substrate for the rime ice accreted in the wind tunnel are shown at Fig. 6, presented in descending order of performances. The ice adhesion obtained on the Aluminum substrate for rime conditions was 0.348 ± 0.2 MPa. The best performing coating for this condition is SI-1 with an ice adhesion of only 0.011 ± 0.001 MPa and the worst is PVD-3 with an ice adhesion of 0.416 ± 0.04 MPa.

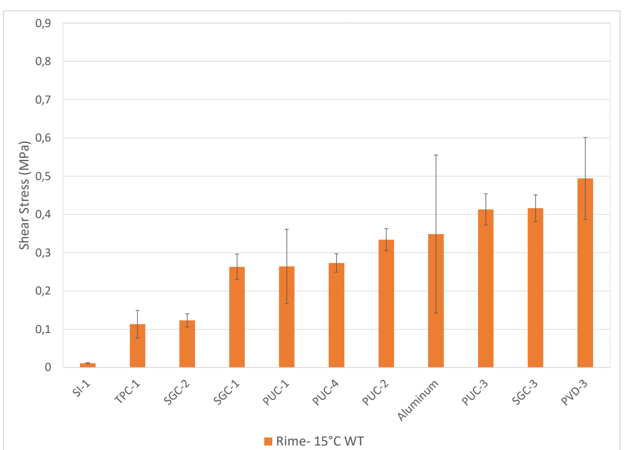

Fig. 6 Shear Stress Comparison obtained under rime in wind tunnel for coating applied on aluminum substrates

The results obtained for the titanium substrate for the rime ice accreted in the wind tunnel are presented at Fig. 7, again in descending order of performances. The ice adhesion obtained on the bare Titanium substrate for the rime condition was 0.291 ± 0.2 MPa. The difference between the results obtained with the four coatings applied on the titanium substrate is not really significant. PACVD-1 has obtained the lowest adhesion with 0.115 ± 0.04 MPa.

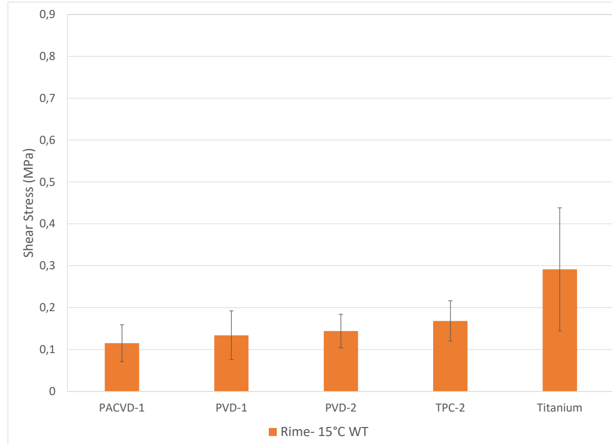

Fig. 7 Shear Stress Comparison obtained under rime in wind tunnel for coating applied on titanium substrates

2) Glaze in Wind Tunnel

For the results obtained on the aluminum substrate with glaze ice accumulated in the icing wind tunnel are presented at Fig. 8. The bulk shear stress obtained with the aluminum uncoated substrate is lower comparatively to the results obtained for the rime by a factor of around 1.3. For this type of icing, the SI-1 coating remains the best and the PVD-3 the worst. SGC-2 and SGC-1 have however switch position with PUC-1 and PUC-4.

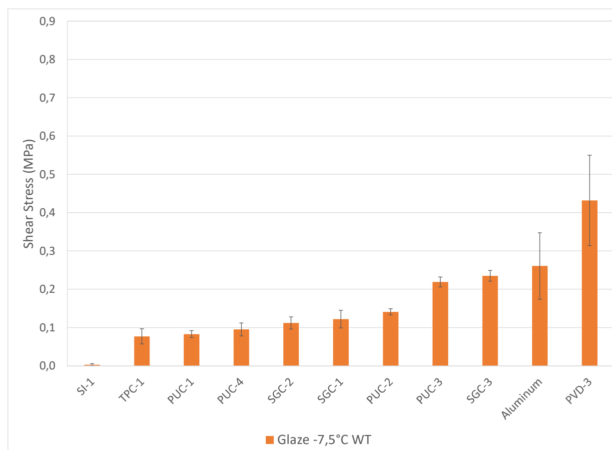

Fig. 8 Shear Stress Comparison obtained under glaze in wind tunnel for coating applied on aluminum substrates

The results obtained on the titanium substrate with glaze ice accumulated in the icing wind tunnel are presented at Fig. 9. The bulk shear stress obtained with the titanium substrate is lower compared to the results obtained for the rime by a factor of around 1.2. In glaze condition, the best coating is TPC-2 instead of PACVD-1. However, as for the rime ice tests, results are very similar for all the products.

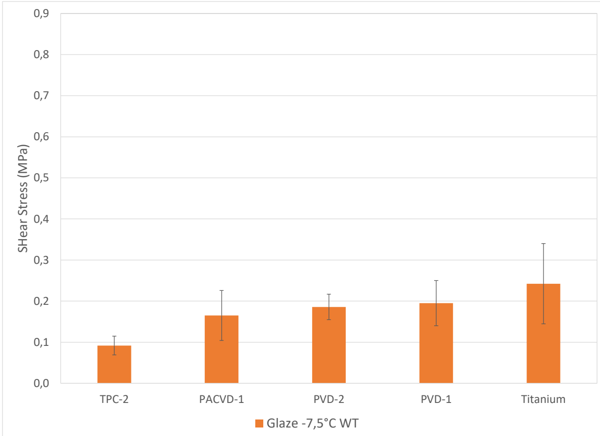

Fig. 9 Shear Stress Comparison obtained under glaze in wind tunnel for coating applied on titanium substrates

3) *Hard Rime in Cold Room*

The ice adhesion on 6061-T6 aluminum obtained in a climatic chamber at a temperature of -8°C, without wind conditions and an MVD of 327 µm, is around 0.615 ± 0.1 MPa. Fig. 10 presents the results obtained on the coatings applied on the aluminum substrate. The best coating is once again SI-1and the worse PVD-3. However, for this condition, many products have switch ranks in performance.

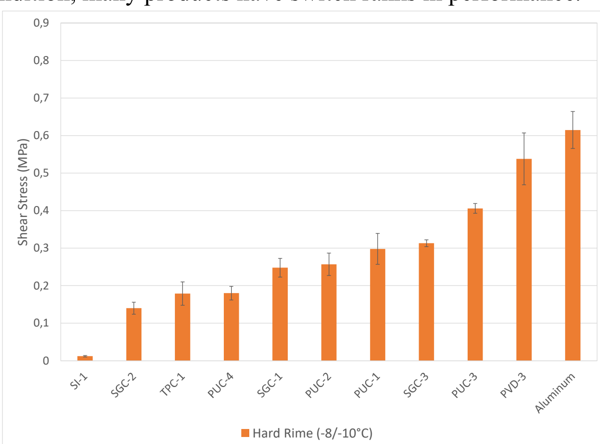

Fig. 10 Shear Stress Comparison obtained under hard rime in Cold Room for coating applied on aluminum substrates

The ice adhesion on titanium obtained in the climatic chamber is around 0.681 ± 0.1 MPa. Fig. 11 presents the results obtained on coating applied on the titanium substrate for that condition. The best coating is TPC-2, as for the glaze condition. Again, the ranking of the coatings in term of performances greatly vary from the other conditions, but this time, also show slightly more variations from coatings to coatings.

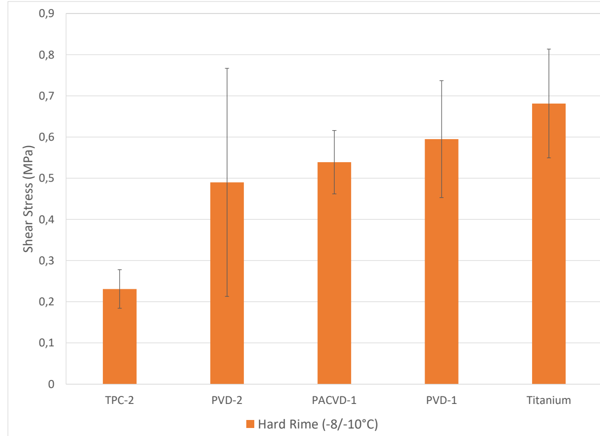

Fig. 11 Shear Stress Comparison obtained under hard rime in Cold Room for coating applied on titanium substrates

B. *Surface Characterization*

Fig. 12 shows the results obtained with the candidate coatings applied on the aluminum substrate for all three icing types while Fig. 13 shows the results for the titanium substrate. The ice adhesion for the glaze condition was always the lowest one no matter the coating with the aluminum substrate. The best coating is clearly SI-1 for all the conditions tested. From the results, no clear trends or conclusions can be drawn from the coating's contact angle and their ice adhesion, the difference between the contact angles is not necessarily significant to observe any conclusions [32].

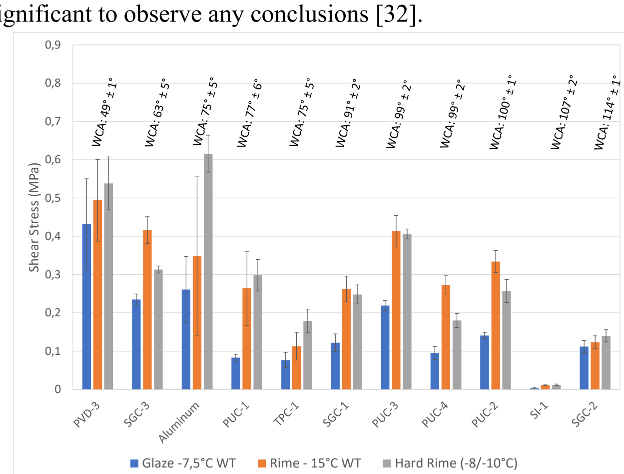

Fig. 12 Shear Stress Comparison Coatings applied on Aluminium substrate classified according to their contact angle

As for the surface wetting properties, no clear relationship was obtained with the surface roughness. The TPC-1 with the highest surface roughness of 2.6 ± 0.5 µm, obtained ice adhesion results in the same order of magnitude as those with a surface roughness between 0.2 and 0.5 µm. In addition, SI-1 has an Ra of 0.6 ± 0.4 micron-m and obtained an excessively low ice adhesion value compared to all those with an Ra of 0.2 µm.

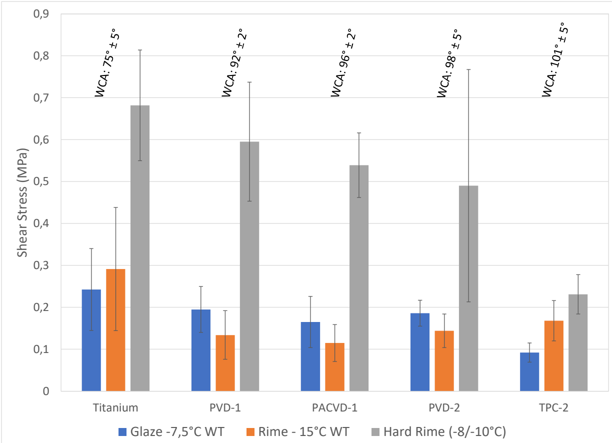

Fig. 13 Shear Stress Comparison Coatings applied on Titanium substrate classified according to their contact angle

VII. Conclusions

A total of 540 centrifuge individual tests were done in this study. The ice adhesion was measured for 13 different products, of different chemical composition and applied with different technique in order to perform a screening campaign. All 13 candidate coatings were evaluated under different conditions, i.e., three different temperatures, three different wind speeds and two different droplet sizes. This was able to demonstrate the dependence of ice adhesion not only on the surrounding temperature but on these two other conditions. Based on the results is it possible to conclude that:

- No matter the test condition, SI-1 is always the best performing product by a significant margin.
- However, results also indicates that coatings are not optimal in every condition and that designing a coating should be emphasized to counter the conditions most encountered for its target application.
- No clear relationship can be established between the contact angle, surface roughness and the ice adhesion, as it was also observed in other studies.
- Adhesion strength is higher for the hard rime ice obtained under a freezing drizzle produced in cold room as opposed to glaze and rime conditions obtained in wind tunnel. Based on other literature, simulated freezing precipitation in a cold room with much larger drops and less impact strength on the surface allows the precipitation to spread over the surface and fill the cavities to create anchors on the surface, thus increasing its adhesion.
- Ice adhesion values obtained in this study greatly vary, between 0.2 and 0.7 MPa on the aluminum substrate, depending on the test condition. This is in agreement with the great variability found in the literature.
- The results do not allow to determine whether one application technique is favorable over another for reducing ice adhesion. It can, however, influence the quality of the application, in terms of its uniformity and repeatability between samples. While this remains a hypothesis, a great variation was observed for coatings that were applied with this technique, specifically for the PVD-2 coating. During the tests carried out in the cold room, a variation of 57% was obtained, with two samples that did not detach at all.

For the next studies, a 3D scanner has been acquired to improve the analysis of the results and thus deepen the knowledge of the ice density of the simulated deposits under different conditions.

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References

[1] W. Yu, X. Yi, M. Guo, and L. Chen, "State of the art and practice of pavement anti-icing and de-icing techniques," *Sci. Cold Arid Reg,* vol. 6, no. 1, pp. 14-21, 2014.

[2] J.-L. Laforte, M. Allaire, and J. Laflamme, "State-of-the-art on power line de-icing," *Atmospheric Research,* vol. 46, no. 1-2, pp. 143-158, 1998.

[3] Y. Wang, Y. Xu, and F. Su, "Damage accumulation model of ice detach behavior in ultrasonic de-icing technology," *Renewable Energy,* vol. 153, pp. 1396-1405, 2020.

[4] C. Antonini, M. Innocenti, T. Horn, M. Marengo, and A. Amirfazli, "Understanding the effect of superhydrophobic coatings on energy reduction in anti-icing systems," *Cold Regions Science and Technology,* vol. 67, no. 1-2, pp. 58-67, 2011.

[5] A. Muthumani, L. Fay, M. Akin, S. Wang, J. Gong, and X. Shi, "Correlating lab and field tests for evaluation of deicing and anti-icing chemicals: A review of potential approaches," *Cold Regions Science and Technology,* vol. 97, pp. 21-32, 2014.

[6] L. Makkonen, "Ice Adhesion - Theory, Measurements and Countermeasures," *Journal of Adhesion Science and Technology,* vol. 26, no. 4-5, pp. 413-445, 2012/03/01 2012, doi: 10.1163/016942411x574583.

[7] S. Rønneberg, J. He, and Z. Zhang, "The need for standards in low ice adhesion surface research: a critical review," *Journal of Adhesion Science and Technology,* vol. 34, no. 3, pp. 319-347, 2020.

[8] B. Subeshan, A. Usta, and R. Asmatulu, "Deicing and self-cleaning of plasma-treated superhydrophobic coatings on the surface of aluminum alloy sheets," *Surfaces and Interfaces,* Article vol. 18, 2020, Art no. 100429, doi: 10.1016/j.surfin.2020.100429.

[9] E. Villeneuve, C. Blackburn, and C. Volat, "Design and Development of an Experimental Setup of Electrically Powered Spinning Rotor Blades in Icing Wind Tunnel and Preliminary Testing with Surface Coatings as Hybrid Protection Solution," *Aerospace,* vol. 8, no. 4, p. 98, 2021.

[10] É. Villeneuve, S. Ghinet, and C. Volat, "Experimental Study of a Piezoelectric De-Icing System Implemented to Rotorcraft Blades," *Applied Sciences,* vol. 11, no. 21, p. 23, 2021.

[11] C. V. Eric Villeneuve, Sebastian Ghinet, "Numerical and experimental investigation of the design of a piezoelectric de-icing system for small rotorcraft part 2/3: Investigation of transient vibration during frequency sweeps and optimal piezoelectric actuator excitation," *Aerospace,* 2020.

[12] C. V. Eric Villeneuve, Sebastian Ghinet, "Numerical and Experimental investigation of the design of a piezoelectric de-icing system for small rotorcraft part 3/3: Numerical model and experimental validation of vibration based de-icing of a flat plate structure," *Aerospace,* 2020.

[13] E. Villeneuve, C. Volat, and S. Ghinet, "Numerical and experimental investigation of the design of a piezoelectric de-icing system for small rotorcraft part 1/3: Development of a flat plate numerical model with experimental validation," *Aerospace,* 2020.

[14] J.-D. Brassard, D. Posteraro, S. Sobhani, M. Ruggi, and G. Momen, "A Multi-Tool Analysis to Assess the Effectiveness of Passive Ice Protection Materials to Assist Rotorcraft Manual De-Icing," *Applied Sciences,* vol. 11, no. 24, p. 11847, 2021.

[15] A. H. Work Jr, "The measurement of the adhesion of glaze ice," 2018.

[16] K. A. Emelyanenko, A. M. Emelyanenko, and L. B. Boinovich, "Water and ice adhesion to solid surfaces: Common and specific,

the impact of temperature and surface wettability," *Coatings,* vol. 10, no. 7, p. 648, 2020.
[17] A. Work and Y. S. Lian, "A critical review of the measurement of ice adhesion to solid substrates," *Progress in Aerospace Sciences,* vol. 98, pp. 1-26, Apr 2018, doi: 10.1016/j.paerosci.2018.03.001.
[18] S. Ronneberg, Y. Z. Zhuo, C. Laforte, J. Y. He, and Z. L. Zhang, "Interlaboratory Study of Ice Adhesion Using Different Techniques," *Coatings,* vol. 9, no. 10, Oct 2019, Art no. 678, doi: 10.3390/coatings9100678.
[19] A. Laroche, D. Bottone, S. Seeger, and E. Bonaccurso, "Silicone Nanofilaments Grown on Aircraft Alloys for Low Ice Adhesion," *Surface and Coatings Technology,* p. 126971, 2021.
[20] G. Fortin, A. Beisswenger, and J. Perron, "Centrifuge Adhesion Test to Evaluate Icephobic Coatings," presented at the 2nd AIAA Atmospheric and Space Environments Conference, Toronto, ON, 2010.
[21] 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 *Proceedings of the International Workshop on Atmospheric Icing of Structures, IWAIS*, 2015.
[22] S. Höhne, C. Hoch, C. Böhm, R. Winkler, E. Bittrich, and P. Uhlmann, "A new measuring system for the determination of the ice adhesion strength on smooth surfaces," *Langmuir,* vol. 36, no. 16, pp. 4465-4476, 2020.
[23] H. Memon, J. Liu, D. S. De Focatiis, K.-s. Choi, and X. Hou, "Intrinsic dependence of ice adhesion strength on surface roughness," *Surface and Coatings Technology,* vol. 385, p. 125382, 2020.
[24] M. Hassan, H. Lee, and S. Lim, "The variation of ice adhesion strength with substrate surface roughness," *Measurement Science and Technology,* vol. 21, no. 7, p. 075701, 2010.
[25] M. L. Pervier, B. G. Lerma, E. P. Moncholi, and D. Hammond, "A new test apparatus to measure the adhesive shear strength of impact ice on titanium 6Al-4V alloy," *Engineering Fracture Mechanics,* vol. 214, pp. 212-222, 2019.
[26] B. Fotovvati, N. Namdari, and A. Dehghanghadikolaei, "On coating techniques for surface protection: A review," *Journal of Manufacturing and Materials processing,* vol. 3, no. 1, p. 28, 2019.
[27] J. Livage, "Sol-gel processes," *Current Opinion in Solid State and Materials Science,* vol. 2, no. 2, pp. 132-138, 1997.
[28] W. N. W. Ismail, "Sol–gel technology for innovative fabric finishing—a review," *J. Sol-Gel Sci. Technol.,* vol. 78, no. 3, pp. 698-707, 2016.
[29] Labomat. "Theory: How does a bar coater work." https://labomat.eu/gb/faq-film-application/790-theory-how-does-a-bar-coater-work-.html
[30] L. Zhang, X. Zong, F. Guo, B. He, and X. Yuan, "Effect of Fluorine Incorporation on DLC Films Deposited by Pulsed Cathodic Arc Deposition on Nitrile Butadiene Rubber and Polyurethane Rubber Substrates," *Coatings,* vol. 10, no. 9, p. 878, 2020.
[31] G. Fortin, A. Beisswenger, and J. Perron, "Centrifuge Adhesion Test to Evaluate Icephobic Coatings," presented at the AIAA Atmospheric and Space Environments Conference, 2-5 August 2010, Toronto, Ontario, 2010.
[32] S. Rønneberg, S. Xiao, J. He, and Z. Zhang, "Nanoscale correlations of ice adhesion strength and water contact angle," *Coatings,* vol. 10, no. 4, p. 379, 2020.