Durability of icephobic materials

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Abstract— **The thorough assessment of icephobic coatings is an important prerequisite for their integration in relevant technical applications. The challenges are that surface parameters are still not well understood and tests for icephobic properties are often linked to a high level of uncertainties. This paper gives insights in evaluation strategies for icephobic coatings and future challenges for the development and assessment of these materials.**

Keywords— ***icephobic coatings, degradation, ice adhesion, correlations***

I. Introduction

The idea of creating icephobic surfaces that prevent ice accretion or reduce ice adhesion is of high interest for many technical applications, because icing can adversely affect e.g. aircrafts, ships, cars, and wind turbines. In previous studies it could be shown that especially their combination with active devices such as heating systems results in significantly reduced power consumption as well as improved performance of the ice protection system [1], [2]. There has been a lot of progress with the current challenge to increase the durability of icephobic effects.

This paper summarizes results of a study, conducted with the focus on aircraft specific degradation stressors. Latest material developments of a cooperation partner have been used to evaluate the icephobic performance during different degradation regimes; accompanied by the assessment of surface wettability and roughness. Data are used to further understand degradation mechanisms of icephobic coatings and to conduct correlation assessments for the identification of most relevant surface parameters. The findings will be an important step towards efficient development and evaluation processes for icephobic materials.

II. Materials & Methods

For this study two Polyurethane-based coatings were used, delivered by a cooperation partner as ready-to-use test specimens. Methods for coating degradation and tests are described as follows.

A. *Degradation regimes*

Test samples were exposed to one of the following stressors, known as to be most severe for coatings in aircraft applications:

- UV(B)-light
- Hydraulic fluid “Skydrol” (phosphate-ester)
- Mechanical loads (scratches)

UV light exposure was carried out in a QUV chamber (Q-LAB) in cycles with UV(B) exposure at (60 ±2)°C and water condensation phases at (50 ±2)°C). Samples were removed for characterization and testing after 200h, 500h, and 1000h, respectively.

Samples for chemical degradation tests were immersed into hydraulic fluid for 30d at room temperature prior to testing.

A TQC taber abraser was used to create multiple scratches on coating surfaces with sanding paper (dry, 220), 5 cycles (Trial 1) + 10 cycles (Trial 2). Sanding was conducted perpendicular to the ice removal direction and perthometer measurements.

B. *Surface parameters*

The assessment of surface properties included:

- Surface free energy (SFE)
- Water droplet contact angle (WCA)
- Sliding angle (SA) and contact angle hysteresis (CAH)
- Roughness R_a and R_z

Wettability tests were performed with Drop Shape Analyzer DSA 100 (Krüss, Germany) according to relevant specifications (DIN 55660-2, -6, -7). Surface roughness was measured by using Perthometer M2 (Mahr, Germany).

C. *Ice adhesion testing*

Ice adhesion reduction is an important function of icephobic coatings. There are no standardized test methods available, and many different approaches have been developed to evaluate ice adhesion reduction of icephobic coatings (e.g. [3], [4], [5]). The centrifuge test is conducted in this study. It uses centripetal forces to shear ice from the test surface [6]. Fraunhofer IFAM uses a conventional laboratory centrifuge with a modified rotor to place and fasten the prepared sample in the device. Opposite the sample is a counterweight that can be adjusted according to ice mass. The centrifuge runs in the ice lab that is cooled to -8°C during the tests (Fig. 1).

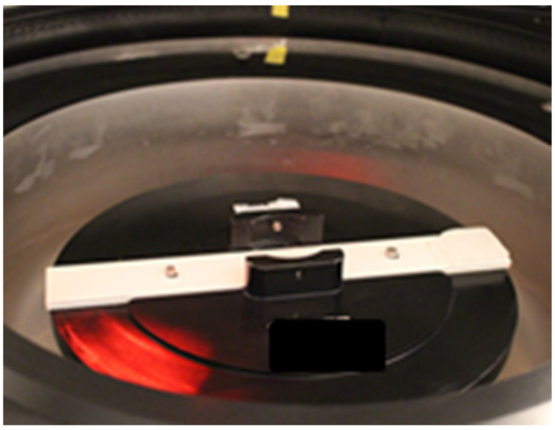

Fig. 1 Ice adhesion test – centrifuge.

Impact ice was created by inserting the coated test samples into the test section of the ice wind tunnel to collect ice, formed from supercooled droplets. Tab. 1 summarizes conditions that were used in this test program.

TABLE I. PARAMETERS FOR CENTRIFUGE ICE ADHESION TEST

Ice formation conditions	
Temperature [°C]	-8
Wind speed [m/sec]	95
Liquid Water Content [g/m^3]	0,6
Median Volume Diameter [µm]	15
Ice mass [g]	3 to 4
Iced area [cm^2]	9
Ice removal conditions	
Sample radius [cm]	9,5
Acceleration [rpm/sec]	300

The prepared sample is fixed in the centrifuge, in which the iced sample is spun at a constantly increasing rate until the ice is sheared off. Separation is detected by a piezoelectric cell when the ice hits the centrifuge wall and correlated to the rotational speed of the centrifuge rotor. This speed (angular velocity ω in rad/s) is used to calculate the shear strength of ice to the surface according to the following equation:

$$\tau = \frac{F}{A} = \frac{m_{ice}\ \omega^2\ r}{A}$$

where m_{ice} is mass of ice [kg], r is the radius of the rotating beam at the mid-length ice position [m], and A is the surface area of the adherent interface [m^2].

III. RESULTS & DISCUSSION

Ice adhesion test results of two Polyurethane-based coatings with icephobic properties are presented in Fig. 2, showing means and standard deviations for three test samples per test run. Three subsequent test runs were conducted.

Initial (fresh) coatings A and B showed ice adhesion values of ~50kPa, which can be classified as low ice adhesion [7]. A trend can be observed that values increase with the increasing number of test runs (fig. 2). This may be linked to a surface degradation during the icing/de-icing cycles.

For coating A a significant increase in ice adhesion due to UV-light exposure (500h, 1000h) and Skydrol immersion could be observed. Coating B shows a different behaviour, ice adhesion values even decrease after 200h UV exposure and degradation is significantly lower compared to coating A.

Again, first test runs on aged surfaces showed lower ice adhesion values compared to the second and third. The results of 1st test runs also indicate a recovery process for both coatings, especially for results after 1000h* UV exposure. Due to a defect of the centrifuge, tests could be conducted only three weeks after completion of UV exposure. During that time potential additives in the bulk material could migrate to the surface, leading to ice adhesion values comparable to fresh coatings (Coating B). These additives are then removed from the surface during the first icing / de-icing cycle.

Generally, coating B showed less performance loss due to the stressors UV or Skydrol than coating A and standard deviations for coating A are higher compared to coating B. This indicates that standard deviations for ice adhesion test results are not only caused by the test design, but also by the materials due to e.g. inhomogeneous surface properties.

High standard deviations could also be observed for tests with multiple scratched surfaces (not displayed). Generally, scratches on coating surfaces resulted in an increased surfaces roughness, accompanied by increased ice adhesion values as it was expected. However, applied sanding process resulted in different surface properties per test sample. Therefore, tests were evaluated per test sample (no mean

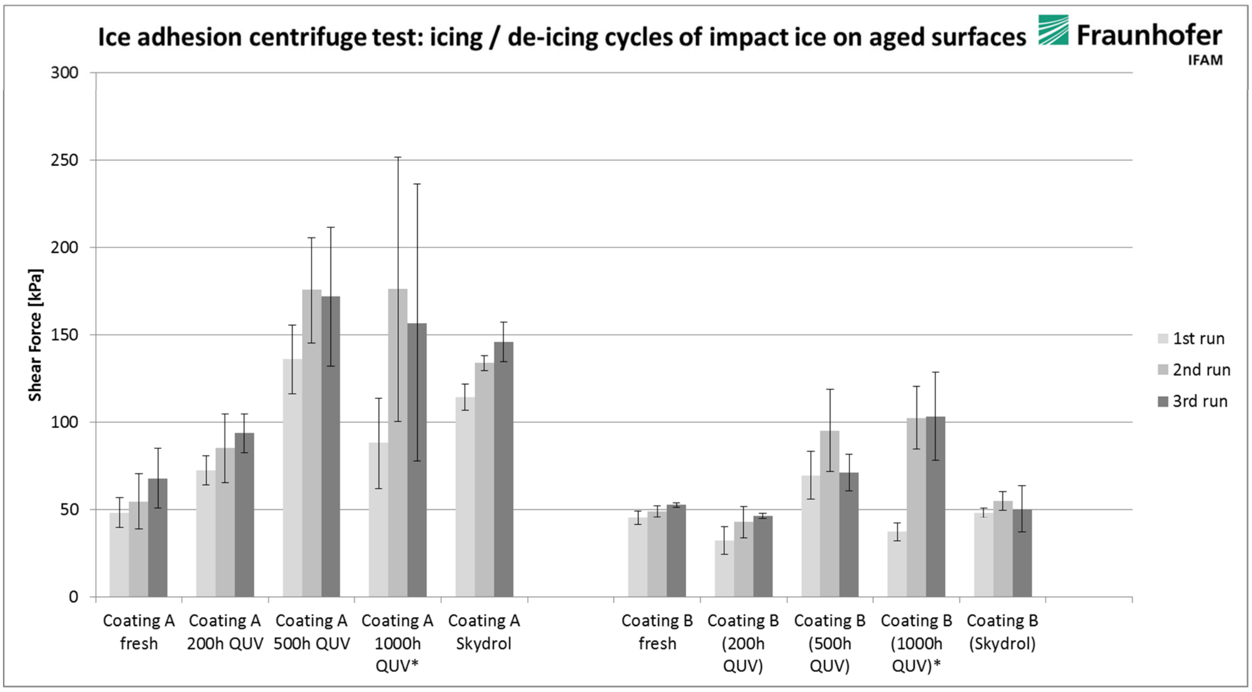

Fig. 2 Results of ice adhesion centrifuge tests.

calculation) for the correlation assessments of surface properties, described as follows (Fig. 3 & Fig. 4).

Various studies have assessed correlations between surface / material properties and icephobic effects (e.g. [8], [9], [10]), but main conclusions are missing due to the complexity of the interrelations. Mainly wetting behaviour (incl. SFE, WCA, CAH, SA), roughness (R_a, R_z, etc.), and elasticity (incl. coating thickness) were related to results from icephobic performance tests. The comparability of the studies is limited because of material specific behaviours as well as the lack of standardized ice-related tests. However, ice adhesion centrifuge tests are widely used and this campaign may be one of the necessary steps to improve the understanding of relevant surface and material properties for effective icepobic coatings.

Correlation assessments in this study were conducted by eliminating first test run results of centrifuge tests (due to the postulated recovery behaviour of the coatings). Results of remaining ice adhesion tests were compared with surface parameters for fresh and aged surfaces. With regard to WCA, CAH and SA, no clear correlations could be identified. Figures 3 & 4 show observed findings for ice adhesion vs. SFE.

Coating A showed lowest ice adhesion values for fresh surfaces with a SFE of 23 mN/m. Both, a further decrease or increase of SFE led to an increase in ice adhesion. This is a first hint that for SFE an optimum can be identified for this coating with regard to low ice adhesion. As of now, the finding is not a clear correlation proof, because surface roughness as important parameter for ice adhesion strength is only indirectly considered (as it influences SFE values). Roughness of all aged coating surfaces increased compared to the fresh coating, but data scattering prevented the identification of clear trends and appropriateness of roughness parameters R_a and R_z remain questionable.

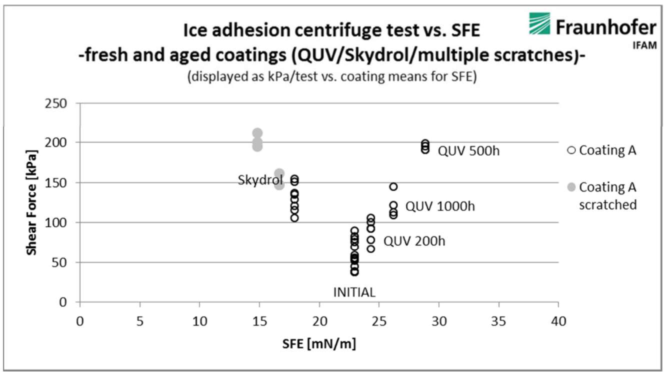

Fig. 3 Correlation ice adhesion centrifuge vs. SFE, coating A.

For coating B lowest ice adhesion strength was assessed for surfaces after 200h UV exposure. The SFE of fresh surfaces increased from 18 mN/m to 20 mN/m after this ageing treatment. This finding is a second hint within this study that there is an optimum for SFE with regard to low adhesion of impact ice.

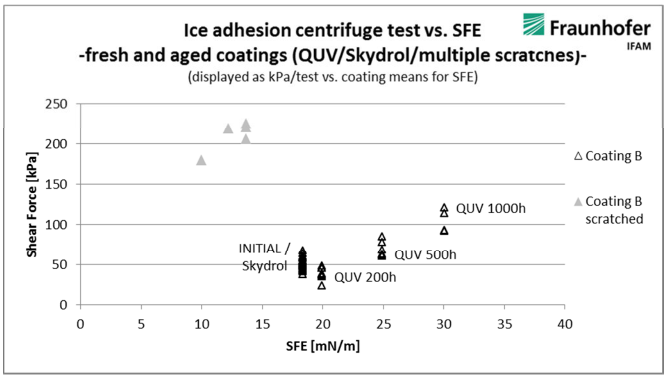

Fig. 4 Correlation ice adhesion centrifuge vs. SFE, coating B.

The correlation between SFE and surface/contaminant adhesion were already described in a completely different field of surface functionality: for foul-release coatings the minimum adhesion occurs between 22 and 24 mN/m and the so-called BAIER curve shows similar trends as it was observed here [11]. This may further support the findings in the current study with regard to ice as contaminant. However, also for foul-release coatings the importance of further parameters were discussed, incl. roughness, elasticity, and coating thickness. These parameters will be subject of further studies at Fraunhofer IFAM.

IV. *CONCLUSIONS*

This study provides important information about the degradation and recovery behaviour of Polyurethane-based icephobic coatings under ageing regimes, relevant for aircraft applications. The findings give indications about the long-term performance of icephobic materials as the main challenge for material developers. It is one thing to create icephobic surfaces and test them in a well-defined laboratory environment; it is another to proof the effectiveness for the relevant technical application. In this regard, the understanding of correlations between surface parameters and icephobic performance is of importance, also in the light of potential monitoring tools for these materials.

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