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Review

Superhydrophobic Coating Solutions for Deicing Control in Aircraft

Michele Ferrari * and Francesca Cirisano

CNR-ICMATE National Research Council, Institute of Condensed Matter Chemistry and Technologies for Energy, Via De Marini, 6, 16149 Genova, Italy; francesca.cirisano@ge.icmate.cnr.it
* Correspondence: michele.ferrari@ge.icmate.cnr.it; Tel.: +39-0106475723; Fax: +39-0106475700

Abstract: The risk of accidents caused by ice adhesion on critical aircraft surfaces is a significant concern. To combat this, active ice protection systems (AIPS) are installed on aircraft, which, while effective, also increase fuel consumption and add complexity to the aircraft systems. Replacing AIPS with Passive Ice Protection Systems (PIPS) or reducing the energy consumption of AIPS could significantly decrease aircraft fuel consumption. Superhydrophobic (SH) coatings have been developed to reduce water adherence to surfaces and have the potential to reduce ice adhesion, commonly referred to as icephobic coatings. The question remains whether such coatings could reduce the cost associated with AIPS and provide durability and performance through suitable tests. In this paper, we then review current knowledge of superhydrophobic and icephobic coatings as potential passive solutions to be utilized alternatively in combination with active systems. We can identify physical parameters, coating composition, structure, roughness, and morphology, durability as properties not to be neglected in the design and development of reliable protection systems in aircraft maintenance.

Keywords: superhydrophobic; icephobic; active ice protection systems (AIPS); passive ice protection systems (PIPS)

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1. Introduction

The term deicing involves actions and procedures aimed to remove or clear ice after formation on critical structures in the environment, while anti-icing systems are instead intended to prevent ice growth and deposition. The aviation industry is strongly affected by this phenomenon reflected in both technological and safety issues.

Before aircraft take off, ice formation prevention procedures usually imply that anti and deicing fluids are commonly administered, even if their effectiveness diminishes rapidly as the aircraft accelerates [1] in addition to releasing harmful fluid components to the environment while, during flight, aircraft can employ heaters and inflatable guards to shield against ice.

Physical parameters like wettability, temperature or pressure describe the direct response of materials to environmental conditions.

The development of an anti-icing surface on a specific industrial coating patch or object has posed a persistent challenge for various industries, including aviation and wind power. To address this challenge, it is essential to perform surface modifications to incorporate the icephobic property into existing commercial coatings for practical applications.

Additionally, permanent hydrophobic coatings can decrease the ability of water to adhere to the aluminum surface, preventing freezing. However, it has been demonstrated in [2] that these coatings can also compromise the effectiveness of anti-icing fluids. According to a recent examination carried out by scientists from Canada, there is a possibility that the application of anti-icing fluid alongside hydrophobic coatings may hinder the formation of a protective film. Researchers at Skoltech [3] also conducted experiments to assess the ability of aluminum surfaces used in aircraft skins to repel water and ascertain whether this has any impact on the effectiveness of these fluids.

Nevertheless, we can regard SH or icephobic coatings (PIPS) as an adjuvant in the inhibition of ice formation during the take-off, in fact eliminating or strongly reducing the use of fluids or chemicals which, in turn, could result as harmful to the environment.

The endurance of three distinct fluid types, all of which met the rigorous aerospace standards of SAE AMS1424 or AMS1428, was examined. These fluid types exhibited varying durations of endurance. To prepare for testing, aluminum plates underwent a process of sanding, polishing, and coating with a hydrophilic acrylic varnish, which yielded a transparent finish that could be either glossy or matte.

The results of the Canadian team's study differ from those of the researchers, as they found that the wetting ability of plates did not affect the effectiveness of anti-icing fluids. However, the researchers attribute this divergence to the surface tension and viscosity of the fluids. Additionally, the researchers acknowledged that surface roughness (Sa) may be a factor, as it takes longer for ice to accumulate on rough surfaces.

Also, temperature and pressure have been demonstrated to affect the shape of the supercooled water on the new icephobic coating. Mechanical tests like cutting, tape tests, pull-off tests and nanoindentation were carried out in [4] to assess the high durability of the icephobic coating. To claim that an icephobic surface is suitable for aeronautical application, it should be tested under flight conditions. This test is possible in an icing wind tunnel (IWT) that can mimic the icing or frosting process in the natural environment by spraying supercooled water droplets onto the substrates. Unfortunately, this facility is rather rare due to the high cost of the setup.

In [5] the effects on roughness at the micro/nanoscale by the pressure have been evidenced due to the impinging of supercooled water droplets, in fact, the pressures at water droplet impact can reach up to 10^5 Pa with the strong mechanical wearing of the coating during its application. Moreover, the Cassie–Baxter state is a condition to be maintained to keep the icephobic properties through the Euler stability of the roughness, the surface asperities resistance to buckling upon water drop impacts.

The huge work on structures and morphology including roughness on hierarchical scales is probably the most innovative approach in this field, considering the high level of manipulation at the micro-nano scale available.

In the last 20 years high water-repellent surfaces have attracted the scientific community due to their wide potential applications in various research and technological fields [6,7] (Figure 1). Lotus-leaf-inspired superhydrophobic surfaces (SHS) and Pitcher-plant-inspired slippery liquid-infused porous surfaces (SLIPS) are compared in this review work [8] providing the state-of-the-art bio-inspired icephobic coatings/surfaces aimed at aircraft icing mitigation Experiments [9] were carried out at Icing Research Tunnel of Iowa State University (i.e., ISU-IRT) facility aiming to evaluate SHS and SLIPS coatings effectiveness in decreasing or eliminating ice accretion and its impact over the surfaces of typical airfoil/wing models. Ice accretion was found to be hindered by both SHS and SLIPS where strong aerodynamic forces were present, while where these forces found a minimum like close to the airfoil stagnation line, ice formation was still found.

This method for preventing ice buildup on aerodynamic surfaces and wings has been successfully demonstrated by combining icephobes with reduced surface heating in the leading edge vicinity. This experimental study assessed the durability of icicle coatings when exposed to rainfall, and the potential applications for preventing aircraft icing. Additionally, it was re-evaluated the impact of ice on erosion and its associated consequences by analyzing variations in ice adhesion forces and surface morphology of eroded surfaces coated with SHS and SLIPS. The findings of this research provide valuable insight into the fundamental physics behind developing anti-/deicing strategies and robust solutions for mitigating aircraft icing.

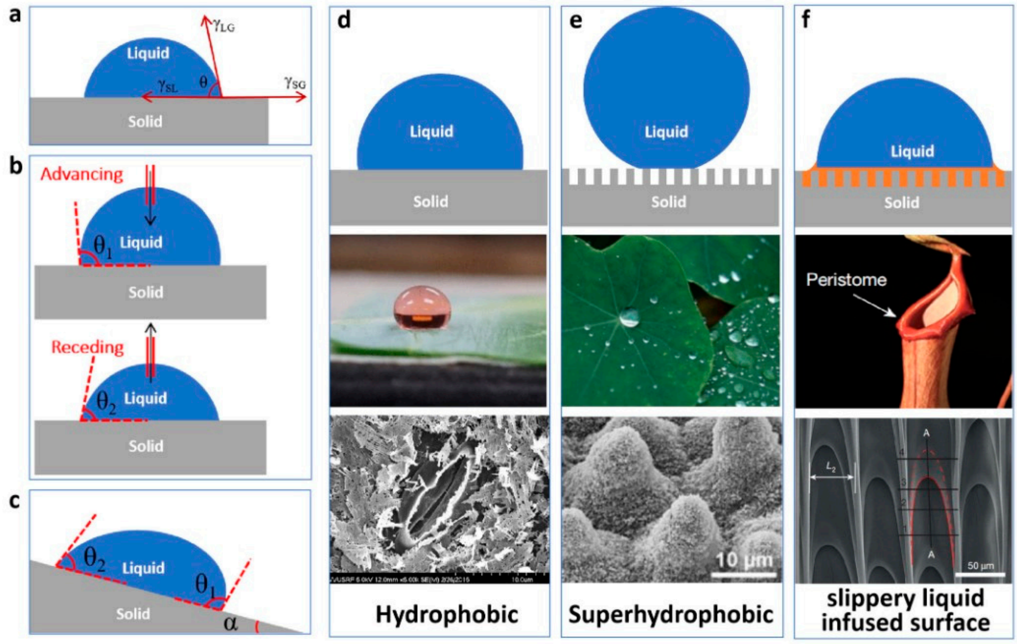

Figure 1. (**a**) Schematic diagram of contact angle and Young's equation. (**b**) Schematic diagram of dynamic contact angle measurement. (**c**) Schematic diagram of rolling angle measurement. (**d**) Hydrophobic model and its prototype in nature and electron micrograph. (**e**) Superhydrophobic model and its prototype in nature and electron mi-crograph. (**f**) Slipperyliquid-infused porous surfaces (SLIPS) model and its prototype in nature and electron micrograph. Reprinted with permission under the terms of the Creative Commons CC BY license from Li, Z.; Wang, X.; Bai, H.; Cao, M. Advances in Bioinspired Superhydrophobic Surfaces Made from Silicones: Fabrication and Application. Polymers 2023, 15, 543. https://doi.org/10.3390/polym15030543 [10].

Nevertheless, the impact of surface coatings on the performances and behavior of the fluids should be thoroughly tested before their use in the industry. Once introduced, new materials for coatings developed for passive ice protection systems undergo for testing with deicing fluids in facilities like the Anti-icing Materials International Laboratory (AMIL) where, more than 30 years, interactions with the ground deicing/anti-icing fluids have been tested before approval for aeronautic application. In this work, current test methods, like the Water Spray Endurance Test (WSET) and Aerodynamic Acceptance Test (AAT), have been carried out on different, commercial and not commercial, surface coatings, with ground deicing/anti-icing fluids: The application of the coating resulted in a decreased spreading, wetting and the endurance time of the commercial fluids. Moreover, the superhydrophobic coating could also avoid aerodynamic drawbacks coming with the reference fluid [2]. The conclusions and methodology of this study were used in the development of sections of the SAE AIR6232 (Society of Automotive Engineers, Aerospace Information Report n°6232) Aircraft Surface Coating Interaction with the Aircraft Deicing/Anti-Icing Fluids standard.

Coating composition is related to its chemistry, to low energy materials, but also aimed to avoid the constraints given by environmental issues, raised by the dispersion of solids and fluids in the air and ground, hardly recoverable and unpredictable long-term accumulation effects.

Among new materials, the ice-repelling properties of superhydrophobic silicone rubber nanocomposite surfaces were created in [11] through either spin coating or spray-coating methods and examined through contact angle hysteresis (CAH), surface roughness, and icing conditions. Both the spin and spray-coated samples displayed a high contact angle (CA) (>150°), a low contact angle hysteresis (<6°), and a roll-off property. While the spin-coated sample had a significantly reduced ice adhesion strength, the ice adhesion strength on the spray-coated sample was surprisingly similar to that of the uncoated sample. This study highlights that a surface's icephobic properties may not directly correspond to its superhydrophobicity, and further investigations, such as considering the effects of icing conditions, are necessary. Additionally, the ice-repelling behavior of the spray-coated sample was found to improve at lower levels of liquid water content (LWC) and under icing conditions characterized by smaller water droplet size.

The role of surface topology on superhydrophobic/icephobic properties has been also investigated by the authors in [12] where, despite high water-repellence with low heat transfer in superhydrophobic surfaces having been widely found for anti-icing purposes, at low temperatures, condensation phenomena occur with the result of more ice adhesion by wetting of the micro- and nanostructures. To address this issue, researchers have developed five different superhydrophobic coatings at the microscale by adjusting the weight ratio of surface-modified nanoparticles to unmodified ones. The strength of ice adhesion and the temperature at which ice nucleation occurs were examined, along with the impact of moisture condensation on ice adhesion. The mechanism and condition of ice strength and formation do not seem to be related to merely morphological aspects of the coating, evidencing the key but not unique role of the roughness in determining the surface features. The results address a more careful surface design underlying the top superhydrophobic coating.

Various studies [13–15] have designed strategies to create an anti-icing surface for specific industrial coating patches or objects carrying out surface modifications with the icephobic characteristic embedded into currently existing commercial coatings for real-world uses. The wide use of polyurethane-based products in the paint and coating industry suggested by a study [16] about the icephobicity of a micron-scaled hydrophobic heterogeneous treatment on Polyurethane Aerospace Coating., an icephobic coating (PPG Industries), evidencing the role of coating in postponing the formation of frost while reducing ice adhesion strength. A copolymer composed of hard and soft sections, poly(methyl methacrylate) and poly(lauryl methacrylate-2-hydroxy-3-(1-amino dodecyl)propyl methacrylate), respectively, a hydrophobic heterogeneity was obtained at the micron-scale at segregation level, resulting in the maintenance of the icephobic properties. The presence of distinct segments with opposite features provides a particular characteristic appearing in a wrinkled design of the coated layer (Figure 2).

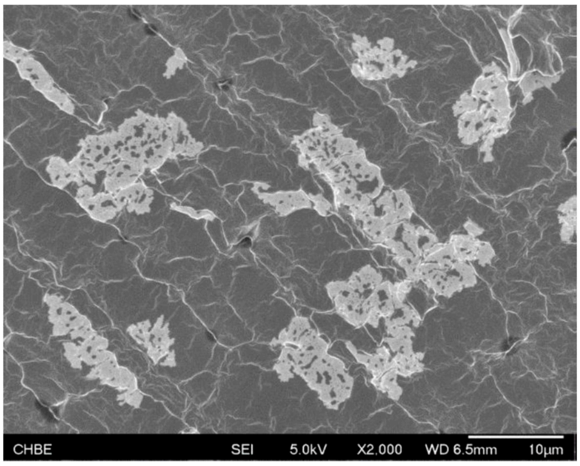

Figure 2. Scanning electron micrograph of the hydrophobic coating showing soft micro-domains embedded on the wrinkled surface. Adapted with permission from Formation of Icephobic Surface with Micron-Scaled Hydrophobic Heterogeneity on Polyurethane Aerospace Coating, Yeap-Hung Ng, Siok-Wei Tay, and Liang Hong, ACS Applied Materials & Interfaces 2018 10 (43), 37517–37528, DOI: 10.1021/acsami.8b13403. Copyright 2018 American Chemical Society [16].

The integration of icephobic solutions with active systems and their testing represents the transition period in which newly achieved coating systems begin to couple existing efficient, but energy-consuming devices.

The effectiveness of a series of coatings composed of amphiphilic silicone polyurethane (AmSiPU) was tested for its anti-icing capabilities [17,18]. While these types of coatings have been previously researched for their antifouling potential, their use for anti-icing purposes remains relatively unexplored. A range of amphiphilic polyurethane-based coatings were produced, containing both hydrophilic polyethylene glycol (PEG) and hydrophobic polydimethylsiloxane (PDMS) with variations in both molecular weight and composition. The surface of these coatings was analyzed, revealing the presence of both PDMS and PEG moieties and confirming their amphiphilic properties. The coatings were then tested for their ability to prevent ice adhesion, with particular attention given to the relationship between their anti-icing performance and water absorption and barrier properties.

The role of innovation aimed to limit accidents caused by ice adhesion on aircraft surfaces poses a significant weight, highlighting the need for AIPS on board. Nevertheless, the downside of AIPS is its added complexity and increased fuel consumption. A potential solution to this is to use PIPS or seek ways to decrease the energy consumption of the AIPS. Superhydrophobic coatings are one such innovative development that could help reduce water adherence on surfaces and subsequently, fuel consumption. The cost associated with AIPS could potentially be decreased by utilizing superhydrophobic or icephobic coatings. Such coatings can effectively decrease ice adhesion and are thus commonly used. The question remains whether these coatings will prove to be a solution for the cost issue at hand.

In these commentaries [19], the author makes a wide comparison among different ways to face ice adhesion-related problems and solutions. Superhydrophobic and icephobic coatings are currently being studied to determine their level of hydrophobicity. Various methods are being developed to adapt existing experimental data to be applied to aircraft. However, it is important to consider the durability of these coatings and, even though some of them have the potential to reduce AIPS power consumption, many do not possess adequate erosion resistance for practical use. To aid manufacturers, guidelines for aircraft erosion tests have been devised. Using superhydrophobic coatings, the extent to which AIPS power consumption can be reduced is dependent on the level of hydrophobicity utilized. Developing coatings that can resist erosion is the primary obstacle faced by manufacturers and designers and the demand for coatings that are superhydrophobic and icephobic arises from this need.

Critical components like leading edges, slats, or vertical tails are strongly affected by ice formation decreasing flight reliability and safety. This work [5] mainly proposed two aims focused on coating design with icephobic properties and, on the other side, developing an innovative tool for wettability assessment. While in the first case, the study addresses coating multifunctional properties including livery effect and resistance to adhesion and abrasion, in the second one the authors attempt to reproduce the thermodynamic conditions like pressure and temperature present at flight altitudes.

In these papers [20,21], the authors evaluated the performance of SHS in preventing ice formation as a function of their durability in terms of erosion coupled with active deicers (Figure 3).

Coupling icephobic coating with electromechanical deicers is one solution for facing ice growth on the surfaces of aircraft with long-recognized issues concerning the security, effectiveness and operational costs. Current dynamic ant-ice strategies, such as electromechanical de-icers, are regularly based on dissolving or breaking ice layers despite these dynamic approaches requiring significant energy for their operation. As passive solutions, SHS can show icephobic properties preventing ice accretion, and, even if their prevention is not completely found, they can be the basis for designing hybrid systems efficient in lowering energy demand for active electromechanical deicers.

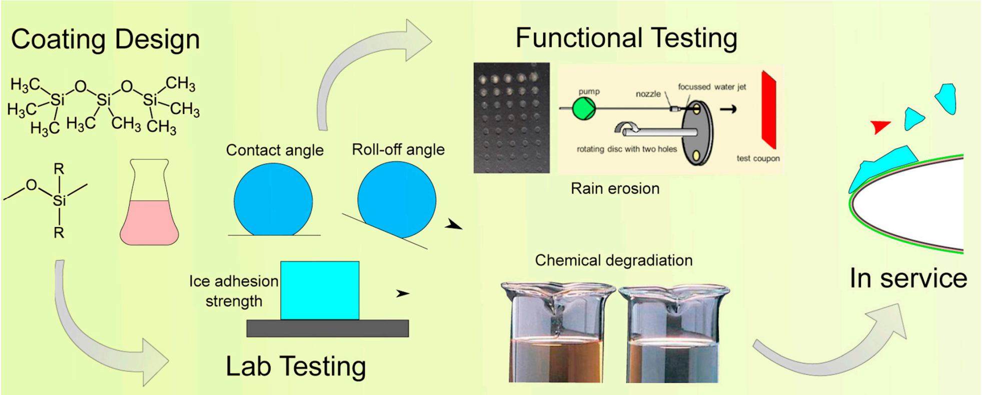

Figure 3. From synthesis to tests, a passive icephobic solution design path to hybrid systems. Reprinted from Progress in Aerospace Sciences, 105, Huang, X.; Tepylo, N.; Pommier-Budinger, V.; Budinger, M.; Bonaccurso, E.; Villedieu, P.; Bennani, L, A survey of icephobic coatings and their potential use in a hybrid coating/active ice protection system for aerospace applications, 74–97, Copyright (2019), with permission from Elsevier [21].

2. Superhydrophobic Surface with Icephobic Behaviour for General Purpose

In recent years, the research on the prevention of ice and frost has significantly grown since this problem affects numerous fields. Superhydrophobic surfaces, due to their low water wettability, are regarded as the most promising materials with anti-icing properties. As we shall see, this statement is partly erroneous because much depends on the actual conditions under which the coating will be exercised (aerospace, marine environment, energy systems). Much research that tries to find a solution to this problem provides results that need to be investigated through specific tests for the different industrial applications.

Chen et al. [22] prepared a macroscopic Al honeycomb structure by electrospray on an SHS with high mechanical strength and anti-icing behaviors. The coating on the honeycomb structure has CA = 161.1 and roll of angle (RoA) = 5.6°. The particular honeycomb structure combined with the low wettability can explain the results from the icing test, in fact, it was observed that at different temperatures below 0 °C the complete freezing time increased for SHS and was better for the honeycomb structure. Also, the frost formation on the sample surface tilted at 60° and at T = −20 °C was less on the SH honeycomb surface both compared to the sample taken as a reference and especially compared to the superhydrophobic on a smooth surface.

Another example is the work of Yu et al. [23] in which the authors affirm preparing an SHS with icephobic behavior. The titanium SHS was prepared via chemical immersion in copper solution and a successively annealing after that the surface showed a CA of about 158°. Icephobicity was tested just by observing the behavior of the water droplet on SH and reference surfaces cooled at −16 °C or observing ice melting on the two surfaces. The authors observed that on SHS, water needs more time to freeze compared to reference and ice, once melted, formed a spherical water droplet able to roll off the surface (Figure 4).

Cheng et al. [24] reported the creation of a superhydrophobic surface with ultra-low ice adhesion without a specific application. For this reason, in this paper, the anti-icing tests did not follow standard protocol. The authors prepared an SHS based on a sprayed PDMS microsphere and OTS@SiO_2 np (OTS is octadecyl trichloro silane), on fluorocarbon resin (PDMS/SiO_2 based surface). SHS show CA = 171° and Sa = 0.277 µm. The Sa was ten times lower with respect to the PDMS coating and in the same order of magnitude coating with only OTS@SiO_2. From static and dynamic anti-icing tests with the surface at −10 °C, it was observed that the PDMS/SiO_2-based surface had good anti-icing ability as it was able to prolong the freezing time probably due to a greater CAH (not reported). Ice adhesion was further evaluated by freezing an ice block on the different surfaces and measuring the shear force to detach ice. The test produced a result in line with previous observations confirming the anti-ice behavior of the prepared SHS. Also, the surface maintains its wettability after 100 mechanical abrasion cycles.

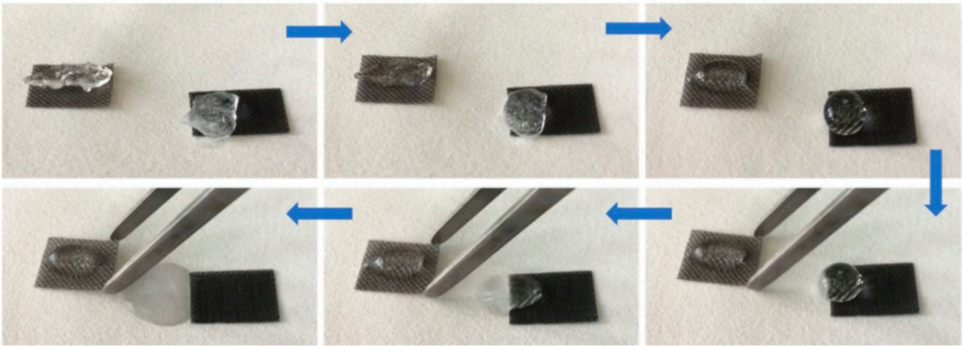

Figure 4. Successive snapshots of ice melting process for adhesion test, the original Ti mesh is on the left and the SSTM is on the right. Reprinted from Applied Surface Science, 476, T. Yu, S. Lu, W. Xu, R. Boukherroub, Preparation of superhydrophobic/superoleophilic copper coated titanium mesh with excellent ice-phobic and water-oil separation performance, 353–362, Copyright (2019), with permission from Elsevier [23].

These are just a few examples where we can see how surfaces are deemed icephobic but in a general context. In addition, the used tests to assess it are nonspecific and, in some cases, exclusively qualitative rather than quantitative.

In the next sections studies on coatings at different degrees of hydrophobicity will be reviewed also according to the test procedures they underwent including the use and the impact of deicing fluids on the stability and durability of their icephobic performances.

3. Superhydrophobic Surface with Icephobic Behaviour for Aircraft Applications

3.1. SHS Not Tested in Flight Conditions

In the literature, some papers aimed to explore the application of superhydrophobic coating in aeronautical fields conducting resistance and anti-icing tests in non-standard conditions. These works could be considered as preliminary and some of them, are indeed a first step of works that applied more stringent and adherence tests to what are the real conditions of use.

Piscitelli et al. presented two works [25,26] in which an SH coating was characterized on a lab scale. The SHS, in both papers, was prepared following the procedure in [27] and the SH coating was applied on different aeronautical substrates, in terms of material and roughness, and the icephobicity was tested by observing the shape of water-frozen droplets (−27 °C) (Figure 5) and then the surface wettability. Cutting and tape tests according to ASTM procedure were performed to evaluate mechanical abrasion resistance. Furthermore, the work of adhesion and surface free energy of the prepared surface was calculated and correlated with sample surface roughness. The authors observed that the SH treatment was necessary to reach high CA and the roughness, regardless of the material, decreased after surface modification, and different roughness can allow the same CA. The works conclude by stating that prepared surfaces can reduce the contact area between ice and substate and that adhesion work, obtained from surface free energy value, was reduced by more than 80%.

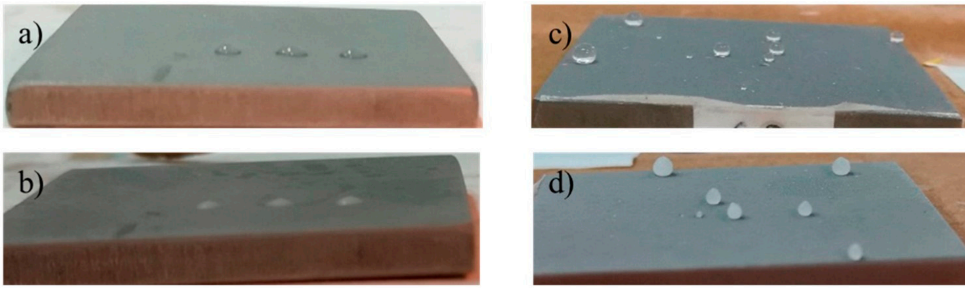

Figure 5. Pictures of samples acquired at −27 °C. Reference with water droplets before (**a**) and after (**b**) the icing at −27 °C; coated sample with water droplets before (**c**) and after (**d**) the icing at −27 °C. Reprinted with permission under the terms of the Creative Commons CC BY license from F. Piscitelli, A. Chiariello, D. Dabkowski, G. Corraro, F. Marra, L. Di Palma, Superhydrophobic coatings as anti-icing systems for small aircraft, Aerospace. 7 (2020) [26].

Liu et al. [28] developed and tested different PDMS-based icephobic coatings for aircraft applications. In this study PDMS coating was fluorinated (F/PDMS) to increase the wettability (CA = 124°) and, to enhance the CA up to 157°, fumed silica was added (F-PDMS/silica). An initial test used to study icephobicity was to deposit a water drop on a surface at a temperature of −10 °C, from this test it was observed that the greater the CA, the smaller the contact area between ice and surface. On the other hand, a lower value was observed for the hydrophobic sample. This result was justified by the different roughness between the samples, SH F-PDMS/silica surface showed an average surface roughness of 7.3 µm while hydrophobic F-PDMS 1.6 µm, with high values meaning high interlocking of the ice within the surface. To investigate operating conditions similar to those of a future application, wettability measurements were performed at a pressure of 0.5 bar and temperature of −12 °C. In such conditions, the CA of the SHS decreases to 104° (reduction of air pockets). From these works, it can be observed that, although the purpose of use of the investigated surfaces is for aeronautical applications, the conducted tests do not follow standard specifications and are rather subjective. Comparing these works, it is also observed that surface roughness at macroscopic magnitudes is important to define ice adhesion but is not sufficient to define a surface as icephobic.

3.2. SHS Tested in Flight Conditions

Some works devoted to ice prevention employing superhydrophobic surfaces for aerospace applications draw incomplete or preliminary conclusions because the tests to which the materials are subjected are not in line with real application conditions. To date, the tests that most closely simulate real-world conditions are those involving the use of an Icing Wind Tunnel (IWT) in which is possible to reach the relevant droplet's impact velocity (50 m/s or higher) at low temperatures (up to −30 °C) generating both rime and glaze ice or specific aging test according to standards. Fortunately, to date, the research employing these tests is increasing and this improves knowledge and the chances, through careful study, of finding a solution to the long-standing problem of ice accretion during flight.

Among the earliest works devoted to the study of the properties of superhydrophobic coatings for aeronautics applications using tests conducted under standard conditions (IWT and others) are those by Antonini et al. [29] and Tarquini et al. [30]. In [29] different surfaces, from hydrophilic to superhydrophobic, were analyzed in IWT (air speed 28 m/s, T = −17 °C) varying icing conditions by acting on the nozzle for the liquid water clouds generation following Icing certification (FAR29, Appendix C—Icing Certification for icing condition requirements). The SH coating (CA = 161° ± 2) was obtained by depositing a thin layer of Teflon by spray coating technique on the wing surface (etched aluminum). To define how a surface was effective in reducing ice adhesion, values of heating power required to avoid ice formation were taken as quantitative parameters. They found that the use of SH coating for aluminum reference provides a reduction in heating power up to 80% to keep the edge of the wing ice-free and a significant reduction of runback ice in high LWC and complete prevention in low LWC conditions. Furthermore, it was observed that the grown ice consists of isolated structures, features that facilitate their breakage and removal. Unfortunately, the present study did not provide data regarding the characteristics of the coating after the test. In [30] the authors tested two SH commercial materials (surface roughness 1.1 and 2.42 μm) and two common metals (baseline) (surface roughness 0.5 μm) to evaluate the icephobic properties in helicopter blades employing an Adverse Environment Rotor Test Stand facility (T up to −25 °C and about 60 m/s) following the Federal Aviation Administration (FAA) advisor circular. The authors found that at high LWC on SH-FAS material, the ice-shedding capability could be slightly enhanced compared to the other samples. However, the authors observed that the adhesion reduction for the two SH samples did not depend on wettability because similar CA show differently in rime ice conditions. Nevertheless, it was observed that SHS exhibited a reduced thickness of the grown ice (Figure 6) but on the other hand, it was pointed out that adhesion stress increases as roughness increases, and the SH samples started to deteriorate after four shedding tests.

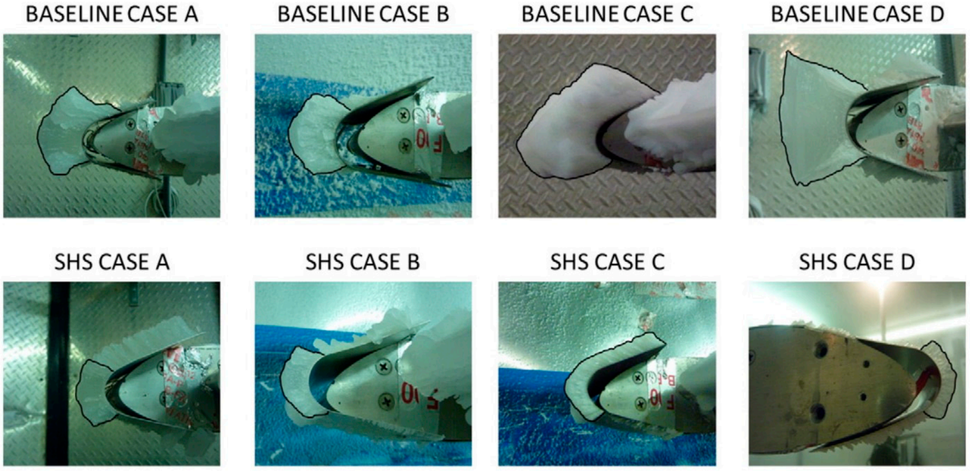

Figure 6. Ice shape pictures for both metallic and SHS materials, taken immediately before the shedding event. Case nomenclature is A, D glaze ide, B, mix condition and C rime. Ice shape profile has been enhanced to ease visual comparisons. SHS case A seems to have a lot of ice on the top surface not enhanced, but this is correct because that ice section is made only by light ice feathers, which would have been shed earlier and separately from the leading edge ice. Reprinted from Cold Regions Science and Technology, 100, S. Tarquini, C. Antonini, A. Amirfazli, M. Marengo, J. Palacios, Pages 50–58, Copyright (2014), with permission from Elsevier [30].

Also, Fortin et al. [31] tested different commercial hydrophobic and superhydrophobic (CA = 152°) coatings under glaze and rime conditions in IWT. Different temperatures were settled to simulate different ice conditions at a constant speed of 21 m/s. From the test, it was observed that the SHS allowed to save energy equal to 33% for glaze ice and about 13% for rime concerning the anti-icing system without coating (reference). Also, the hydrophobic coating allowed for saving energy but in smaller quantities than SHS. From this preliminary work, the increase in hydrophobicity allowed reducing energy consumption for ice protection but no tests about the durability and resistance of SHS were conducted.

Piscitelli et al., over the years, proposed different studies in which they analyzed the yield of SH materials under real application conditions. In these works, [27,32] the SH coating was prepared by spray coating technique where several layers of nanometric hydrophobic silica dispersed in Tetrahydrofuran were deposited on different substrates with or without a polyurethane primer layer. In [27] the authors found that higher CA was reached when the silica dispersion was prepared by ultrasonication, in fact, superhydrophobic behavior was reached only after this step. Reference and SH-coated samples

(Ra = 0.3 μm) were subjected to an aging test according to the MIL standard in which real flight conditions at 16,000 m in altitude were simulated (variation of humidity and temperature) for about 16 h, 10 times. After the aging test, the SH coating maintained its wettability in particular when a primer PU (Polyurethane) layer was applied and measurement of CA at −12 °C and 0.5 bar showed a decrease in wettability losing superhydrophobicity (<130°). An evolution of the work is found in [32] where an SH coating, produced following the procedure in [33], was applied on the NACA0015 (symmetrical airfoil with a 15% thickness to chord ratio developed by the National Advisory Committee for Aeronautics) wing profile has been subjected to an IWT test, the temperature ranged from −3 to −23 °C at an altitude of 3000 m, LWC 0.3 g/m^3 and two-speed tests 50 and 95 m/s. Superhydrophobicity was maintained after the IWT test campaign but the effectiveness of the SH coating on ice prevention/reduction depended on the tested conditions (glace or rime and exposure time) and, in general, for long exposure time it could be considered as a support to an active system and not for use only as an anti-icing system.

Morita et al. [34] conducted validation tests in IWT on a hybrid anti-icing system (CA = 150° RoA = 8°) prepared by combining icephobic polytetrafluoroethylene (PTFE) coating and electrothermal heating (ICE-WIPS). The IWT test chosen conditions appeared in Federal regulation and they selected wind speed of 75 m/s, T = −10/−8 °C and different LWC. The experiments provided raw data concerning the reduction in power consumption and it was observed that, in relationship to the existing heating system, the ICE-WIPS reduced power consumption by 30 to 70% depending on icing conditions. The work, however, did not provide data regarding the durability and the maintenance of properties thus resulting partly deficient for actual applications.

Brown et al. [35] recently developed an SH duplex coating system devoting a great deal of attention to its environmental durability. This material was obtained on stainless steel by depositing first TiO_2 powder by suspension plasma spray and secondly, a thin coating of diamond-like carbon network with SiO_2 by plasma enhanced chemical vapor deposition. The prepared coating showed CA = 159°, CAH = 3.8° and Sa = 10.3 μm. The SH coating was exposed to icing/deicing cycling (IWT), rain erosion, and accelerated aging test, and the results were compared with those from tests performed on SH TiO_2/stearic acid and commercial hydrophobic fluoropolymer. The IWT test conditions were T = −10 °C with air speed of 43 m/s and LWC of 0.5 g/m^3. At the end of the study, it was possible to affirm that the prepared SH coating was resistant to UV exposure, and could resist up to 6000 water droplets impact at 165 m/s and especially started to degrade after 170 icing/deicing cycles resulting in the coating with the best performance. However, the authors pointed out that, although the test in IWT was under severe conditions, deicing by melting and removal of ice through this method was less damaging than removing it by shearing force and that, if this situation could arise, the coating would degrade much faster. As above noted, the works just reported all apply specific test conditions that are very close to real ones even if they are often different from each other. In general, it is observed that SHS decreases the energy required to melt ice in AIPS and that the greater or lesser effectiveness depends on the type of formed ice.

Until now, we revised articles where wettability studies did not take into account the reported roughness values. In the following papers, however, this relationship has been carefully analyzed to draw more accurate conclusions after IWT testing.

Belaud et al. [36] investigated by IWT tests hydrophobic and superhydrophobic surfaces created on an aluminum alloy commonly used for aerospace components. The different wettability and roughness were obtained working on the Al alloy anodization in different media, sulphuric (SA) or oxalic acid (OA), and subsequently, each sample was functionalized by spraying a commercial hydrophobizing agent. OA samples were hydrophobic (max CA = 146°, RoA = 81°) with max surface roughness of 0.32 μm, SA samples were superhydrophobic with CA of 162° and 170°, RoA of 32 and 4°, respectively, and surface roughness one order of magnitude higher than OA samples and smooth Al alloy reference (roughness like OA). IWT tests were performed following FAA specifications creating four different icing conditions (rime, mixed/rime, mixed/glaze, glaze) after each of them, ice adhesion strength was measured. Except for the glaze condition in which the adhesion strength remained unchanged for all surfaces (approx. 45 kPa), in the other three conditions, only the hydrophobic OA sample showed adhesion strength lower, 2–3 times, than the reference (Figure 7). This result was explained considering the different surface roughness between the samples supporting the theory that wettability alone is not synonymous with anti-ice.

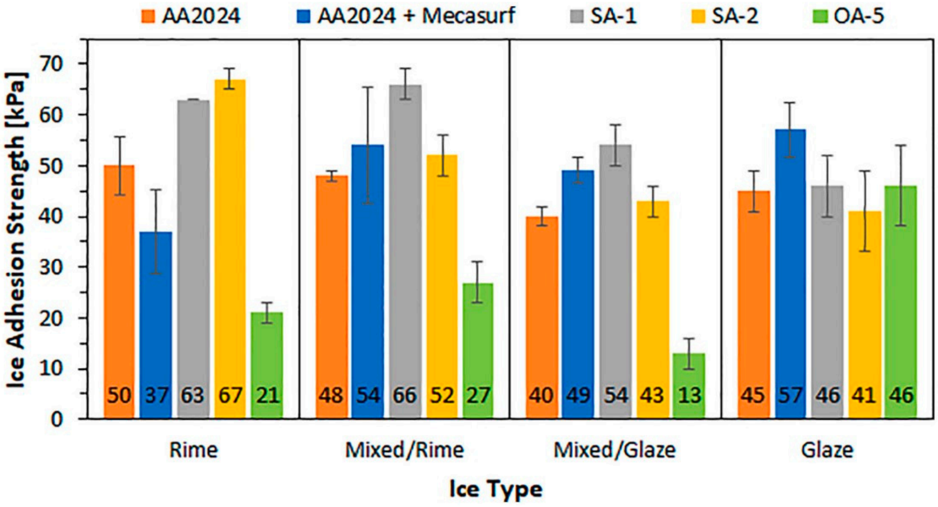

Figure 7. Ice adhesion strength of the anodized samples in rime ice, mixed/rime ice, mixed/glaze and glaze ice conditions with respect to the reference bare AA2024 surface. Reprinted from Surface & Coatings Technology journal, 405, C. Belaud, V. Vercillo, M. Kolb, E. Bonaccurso, Development of nanostructured icephobic aluminum oxide surfaces for aeronautic applications, 126652, Copyright (2021), with permission from Elsevier [36].

Another work supporting this theory is [11] in which the authors prepared, by different techniques (spin and spray coating) but the same mixture, two SHS with different surface roughness. Spin coated sample showed CA = 156°, CAH = 5° and Ra = 500 nm, spray coated sample showed CA = 165°, CAH = 4° and Ra = 8 µm. After the IWT test (T = −10 °C, wind speed 10 m/s, different LWC conditions and size of microdroplets) it was observed that although the samples had similar wettability and CAH, different ice adhesion strength values were registered. The spray-coated sample showed higher values, similar to the reference, compared to the spin-coated one evidencing the dominant effect of roughness on icephobicity. Furthermore, less stable wettability of spin coated sample with respect to spray coated one was observed, and icephobicity decreased after each icing/deicing cycle.

Continuing the search for a performance coating with icephobic behaviors for aerostructure components, Mora et al. [37] proposed the use of quasicrystals (QCs) metallic materials. They are applied by means of high-velocity oxyfuel thermal spray Al-based QCs on common aeronautic materials. In particular, the work aimed to compare the QCs coating with PTFE, PU-based commercial paints and bare metal considering also the surface roughness. To do that, two QC materials were chosen and tested both in an "as deposited" state and after grinding with SiC paper to allow surface roughness similar to the reference. As deposited QCs materials showed different CA, for QC1 it was 158° with a CAH of 58°, for QC2 CA = 114° and CAH = 60°. All other reference materials showed contact angles less than 101° (PTFE). Ground QCs lost their wettability in fact CA for QC1 decreases to 57° and QC2 to 72°. IWT tests were performed both in glaze and rime ice conditions and ice accretion was evaluated. Under rime and glaze conditions the mass of ice accreted on all QCs coating was less than ice on standard metals. A different behavior was observed for the ice adhesion test, in this case, roughness played a critical role, the best results (low shear stress) were observed for the low roughness sample (PTFE and PU reference), and lower values than as-deposited QCs were observed for ground QCs samples even lower than the value obtained for the aeronautic paint. The results thus lead to the statement that for ice adhesion, roughness played a role more important than wettability which seemed to be important in the mass of accreted ice, and finally that QCs materials are promising in terms of durability. Comparing these works, it can be affirmed that roughness is a key factor in the ice adhesion to the surface. Each work concludes by noting that samples with higher roughness, even if SH, show higher shear stress than the unmodified surfaces used as reference. It should be noted, however, that in all these cases the produced SHS shows a macroroughness on the order of microns, while in the only case where an SHS shows performant results, the roughness is below the micron (500 nm, CA = 156°). In conclusion, we can affirm that superhydrophobicity plays a key role only if it is accompanied by nano roughness that does not allow strong ice interlocking.

3.3. Hydrophobic and SLIP Surfaces with Icephobic Purpose Tested in Flight Conplaysditions

It was observed from the literature that it is difficult to obtain SH surfaces with anti-ice behavior and durability; some authors directed their research toward other types of surfaces.

Rivero et al. [38] in the present study proposed three hydrophobic coatings, prepared by different techniques and materials, with anti-icing behavior to deepen the use of coatings for passive solutions in airplanes. The three coatings were tested in IWT (air speed 50 m/s, T = −8 °C, LWC 0.36 g/cm^3) evaluating ice accretion and durability after some icing-deicing cycles. From these tests, it was observed, considering also reference material such as PTFE and Al substrate, that the anti-icing behavior was a combination of surface roughness and surface energy. Materials with similar roughness (coating 1 and PTFE) showed different ice adhesion and high roughness was more important with respect to low wettability. The best coating was that showing CA = 88° and Ra 0.19 µm but the results are far from a possible aeronautical application due to its low mechanical resistance.

SLIPS are a category of materials recently investigated for applications in aeronautical applications. Veronesi et al. [39] prepared and tested them in IWT to evaluate the ice accretion in rime and glaze conditions. Two different SLIPS were prepared, one was Al_2O_3-based and the other was SiO_2-based and both were infused in perfluoropolyether lubricant oils (two liquids that differ in terms of viscosity). IWT were conducted in different conditions but same velocity, 50 m/s, to obtain glaze and rime regimes. Al_2O_3-based and SiO_2-based SLIPS showed similar CA around 120° depending on the used lubricant oil and not on the substrate material. An important difference was the morphology of the samples: Al_2O_3-based coatings showed a flower-like structure, and SiO_2-based showed nanoparticles agglomerate with larger pores compared to alumina coatings. Both SLIPS displayed a decrease in ice accretion with respect to the uncoated surface, in particular, silica-based showed a reduction of up to 45% in glaze conditions. In rime, ice conditions not much improvement was observed compared with the reference.

Also, Vicente et al. [40] studied SLIPS as an anti-icing solution comparing them with self-prepared SHS. Both surfaces were formed by PVDF deposited by electrospinning technique and silicon oil was used as liquid to fill the porous PVDF structure and obtain the SLIPS. The prepared SH surfaces showed a linear correlation between CA and Sa, the highest value of CA (about 162°) was reached for the roughest sample (Sa = 700 nm). The SLIPS obviously showed low CA (106°) but the value of CAH (SHS CAH = 30°, SLIPS CAH = 19°) denoted how droplet mobility was better on this surface. Anti-icing behavior, on the best SHS and on SPLIPS, was tested in two conditions in IWT (air speed 70 m/s, T = −5/−15 °C, LWC 0.5 g/cm^3) in order to produce glaze and rime ice, and adhesion test was performed using a centrifuge. PTFE and Al were used as a reference and in both glaze and rime conditions, as shown in Figure 8, SHS showed higher ice adhesion (especially in glaze conditions) than PTFE, while SLIPS achieved low ice adhesion in both conditions (four and seven times less of PTFE). SHS sample lost its property after only one cycle of icing/deicing instead of SLIPS that resisted up to four cycles. The surface was still not resistant considering that Al and PTFE did not show degradation after the test.

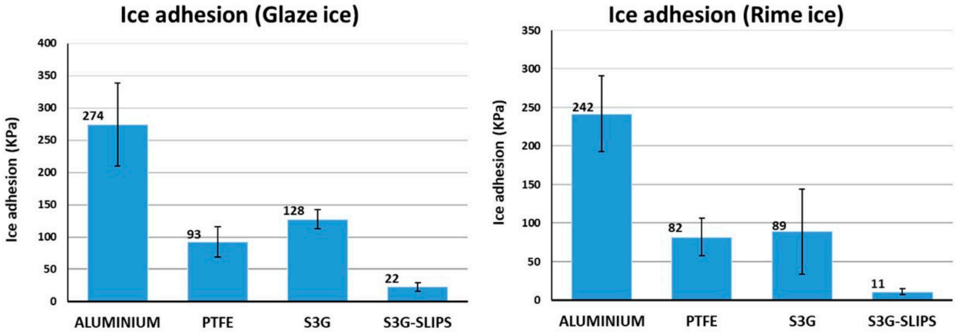

Figure 8. Graphs of ice adhesion tests after glaze and rime icing procedures in IWT. Reprinted with permission under the terms of the Creative Commons CC BY license from Vicente, A.; Rivero, P.J.;García, P.; Mora, J.; Carreño, F.;Palacio, J.F.; Rodríguez, R. Icephobic and Anticorrosion Coatings Deposited by Electrospinning on Aluminum Alloys for Aerospace Applications. Polymers 2021, 13, 4164 [40].

From these studies, it is possible to observe that SLIP surfaces are more performant compared to SHS due to the presence of lubricant in the microstructure that avoids water penetration and ice interlocking with the surface. Unfortunately, SLIPS seems to be not resistant to icing/deicing tests indicating that to date this solution is not applicable.

4. Deicing Fluids

Regardless of whether innovative coatings investigated as potential passive ice protection systems were hydrophobic, superhydrophobic, or SLIPS, they must resist the effects due to the application of deicing fluids and simultaneously that the fluids do not lose effectiveness when applied to surfaces. The problem with deicing/anti-icing fluids regards their composition because they are mainly composed of water, glycols, additives, and surfactants with potential adsorption to the surface and consequential loss of their initial highly hydrophobic properties. Villeneuve et al. [2] performed two current test methods used to qualify the ground deicing/anti-icing fluids on five different surface coatings, commercial and under development, and the results were used to develop a section of the SAE AUR6232 about fluids endurance time. They found that, depending on used deicing/anti-icing fluids, some surfaces could reduce the fluid endurance time that was regulated by law (Figure 9). The authors found no direct correlation between the CA and the endurance time and even when a positive result was observed, it was possible that the surface affected the fluid flow off establishing possible negative effects on aerodynamics. A later study setting out to explore this problem is more accurately described in [3]. In this paper, as opposed to [2], the authors provided detailed information about commercial anti-ice fluids (density, viscosity and surface tension) and employed surfaces (Sa, advancing and receding CA). Superhydrophilic and hydrophobic surfaces were used to test the ice protection of three commercially anti-icing fluids during the water spray endurance test. It was observed that surface wettability had no effect on the anti-icing fluid endurance time and that the results of ice accretion and durability were correlated with fluid viscosity and surface tension. The authors in particular stressed how surface roughness was important and must be considered for comparative studies since rough surfaces took longer to accumulate ice due to the presence of anti-ice fluid in the surface texture.

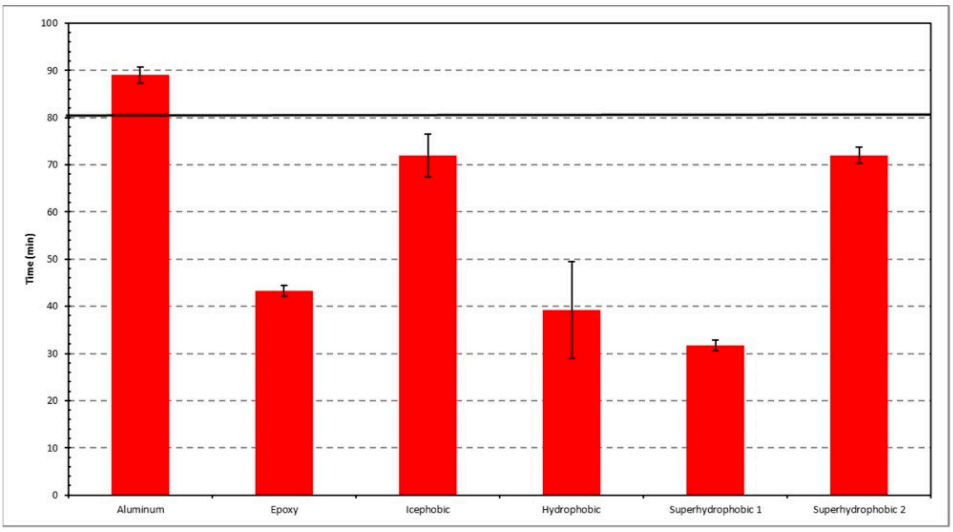

Figure 9. Endurance time for the different surfaces for Fluid B. Bold black line at 80 min is the minimum requirement as per AMS1428. Reprinted with permission under the terms of the Creative Commons CC BY license from Villeneuve, E.; Brassard, J.-D.; Volat, C. Effect of Various Surface Coatings on De-Icing/Anti-Icing Fluids Aerodynamic and Endurance Time Performances. *Aerospace* 2019, *6*, 114. https://doi.org/10.3390/aerospace6100114 [2].

On the other hand, Zhang et al. [41] investigated the negative effects of deicing fluids on coating performance. Commercial SHS and PTFE-based surfaces were chosen as representative icephobic materials and they were applied on test plates. Enamel coating was used as a reference material. Icephobic plates were immersed in two different commercial deicing fluids to simulate a situation where liquids remained on the airframe surfaces for different amounts of time before take-off. The authors observed that the two liquids had different impacts on the surface's wettability. Type-I deicing fluid showed low or no effect on the performance of SH and PTFE coating, on the other hand, Type-IV deicing fluid was capable of strongly contaminating surfaces by drastically reducing their icephobicity and consequently increasing their ice adhesion strength.

5. Conclusions

In this work, recent and fundamental literature has been revised on topics related to strategies to inhibit ice formation in aircraft as passive solutions or in combination with active systems. In the conclusion remarks, we can underline the high potential of SHS, which can be efficiently combined to reduce power consumption in an anti-icing active system. From the literature, some improvements should be addressed among physicochemical properties studies, where it can be observed that icephobicity is correlated with surface roughness, while wettability is not a comprehensive parameter to speculate about icephobic behavior. Moreover, studies addressing erosion resistance and ultraviolet degradation will be conducted, as they significantly affect the durability and overall stability of the treatment, potentially compromising the safety of the aircraft functions. Lack of methodology has been observed in studies on characterization and assessment of the icephobic coating performance, found not following the same protocols (methodology, shape form airfoils, how ice was introduced/grown on material) producing results that are not easily comparable and, in some cases, the test conditions are not reported. Furthermore, in some papers, details about surfaces such as roughness and CAH are not listed making comparison impossible. Future perspectives can be suggested considering the real application on aircraft with more insight into uniforming protocols and tests with de-icing/anti-icing fluids.

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resources, investigation, writing—review and editing; F.C.: image processing. All authors have read and agreed to the published version of the manuscript.

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