Thursday Morning, May 23, 2019

Topical Symposia
Room Golden West - Session TS2-ThM
Icephobic Surface Engineering

Moderators: Alina Agüero Bruna, Instituto Nacional de Técnica Aeroespacial (INTA), **Jolanta-Ewa Klemberg-Sapieha**, École Polytechnique de Montréal, Canada

8:20am **TS2-ThM-2 Synthesis And Characterization Of Amphiphobic Hybrid Coatings For Industrial Applications***, **Giulio Boveri**, M Raimondo, F Veronesi,* Institute of Science and Technology for Ceramics, Italy

In the coming few years, the control of materials repellence against liquids is one of the biggest challenge to make innovation in many industrial fields. Gathering on the same material the ability of repelling liquids with physical properties (typically surface tension γ) in a wide range of values represents the topic of many scientific efforts. Materials wetting strictly depends on surface chemistry and reactivity and on structural features at nanoscale as well.

This work is based on the surface modification of aluminum substrates by deposition of thin layers of ceramic oxide nanoparticles, in particular Al_2O_3 and SiO_2 synthesized by sol-gel routes, in order to introduce nano features which, coupled with the organic modification of the surface chemistry, lead to an almost complete repellence against water (*superhydrophobicity*, contact angles approaching 180° and contact angle hysteresis <5°) and liquids with γ as low as 25 mN/m (*superhydrophobicity* plus *oleophobicity* = *amphiphobicity*). *Amphiphobic* materials are drawing much interest in different industrial sectors, such as naval, aerospace, energy and automotive, so that great efforts have been making to improve their durability and wearing resistance simulating their application in real environments.

Two different approaches were typically used to produce non-wetting surfaces: the so-called *Lotus Leaf* (*LF*) and the *Slippery Liquid Infused Porous Surfaces* (SLIPS) ones, both introducing textural and chemical modifications at surface level that, in turn, is working in a solid-liquid-air (LF) or liquid-liquid-air three phasic environment. Self-cleaning, anti-icing and anti-soiling behavior was assessed on these surfaces and the detected performances correlated to the nature of the coatings, other than to the physical state of the working interfaces.

Keywords: Amphiphobicity, hybrid coatings, ceramic nanoparticles, surface modifications.

8:40am **TS2-ThM-3 *In situ* Ice Growth Kinetics on Water-repellent Coatings in Atmospheric Icing Conditions***, **Jacques Lengaigne**, P Xing, É Bousser,* École Polytechnique de Montréal, Canada*; A Dolatabadi,* Concordia University, Canada*; L Martinu, J Klemberg-Sapieha,* École Polytechnique de Montréal, Canada

Ice accretion on the surface of airplanes during flight and in other similar situations presents significant safety concerns and economic loss. Icing occurs when micrometric Supercooled Water Droplets (SWD) impact at high speed and freeze on exposed components. Current de-icing systems (chemical agents, heaters or inflatable boots) are energy intensive and prone to failure. To address these shortcomings, research has focused on the development of so-called icephobic surfaces or coatings. These surfaces could provide an efficient passive protection: inhibiting ice nucleation, reducing ice growth or improving the efficiency of current de-icing technology. At the forefront of these new coatings, superhydrophobic surfaces, combining materials with low surface energy and high multiscale roughness, are regarded as one of the most promising avenues. In this work, we investigated the performance of water-repellent coatings against SWD ice accretion.

To replicate realistic SWD icing conditions, we used a small-scale icing wind-tunnel to generate microdroplets with diameters ranging from 10 to 90 µm, and an air speed of 10 m/s impinging at a temperature of -17 °C. The test section was equipped with a sample holder incorporating a thermoelectric module for heating/cooling the sample, a side camera to visually record the ice formation, and an *in situ* ice growth monitoring system. This latter module was developed to measure ice thickness during the testing cycle. It functions by following the displacement of a laser beam using a dedicated digital camera. Moreover, the laser spot spreading offers insights into the evolution of the roughness of the ice layer.

Three types of surfaces with different wettability and roughness R_q values were studied in this experiment: hydrophilic mirror-polished Ti-6Al-4V alloy (θ_c=74±1°, R_q= 51±4nm), hydrophobic spray coating (θ_c=107±1°, R_q= 0.35±0.04 µm) and superhydrophobic hierarchical spray coating (θ_c=173±3°, R_q= 12±2 µm).

The ice growth cycle follows a similar behavior on all samples: first the ice nucleation occurs followed by an incubation period before the continuous ice growth. Once the ice has formed a continuous layer, the growth rate is linear. It was found that the incubation period is longer when the surface is more hydrophobic. In fact, the superhydrophobic coating showed twice longer incubation time compared to the substrate. However, ice growth rate increased on the water-repellent coatings (up 34% faster on the superhydrophobic surface) compared to the pristine titanium alloy. Finally, de-icing of each sample showed that the conductive substrate is de-iced faster compared to both water-repellent coatings.

9:00am **TS2-ThM-4 Icephobic Elastomeric Surfaces?***, **Pablo Francisco Ibáñez**, F Montes Ruiz-Cabello, M Rodríguez Valverde, M Cabrerizo Vílchez,* Universidad de Granada, Spain

It is well-accepted that superhydrophobic surfaces may lead to interesting properties such as anti-fogging, self-cleaning, or anti-icing. However, some studies have reported that superhydrophobicity does not assure anti-icing/de-icing performance. Icephobic surfaces should hold three requirements: subcooled-water repellency, freezing delay and low ice-adhesion energy. Superhydrophobic surfaces are rough and ice adhesion is mostly increased by interlocking. Lubricant-Impregnated Surfaces (LIS) are also proposed to repel water. Moreover, there are evidences of that these surfaces also mitigate icing and reduce ice-adhesion. For this reason, we are investigating others approaches to prepare low ice-adhesion surfaces.

In this work, to absorb the surface shear stresses of freezing water, we focus on elastic hydrophobic surfaces such as fluorosilicones. We study the ice-adhesion strength on PDMS surfaces with different elastic modulus and thickness. Further, we study oil-infused elastomeric surfaces. Finally, we evaluate the durability of the prepared surfaces (wear, abrasion).

9:20am **TS2-ThM-5 Design and Fabrication of Superhydrophobic, Ice-phobic Coatings for High Voltage (HV) Power Lines Application***, **Mariarosa Raimondo**, G Boveri, F Veronesi,* ISTEC CNR - Institute of Science and Technology for Ceramics, Italy **INVITED**

The interaction between water under many different forms – dry or wet snow, ice, frost, rime or their combination – and materials is a complex matter to investigate, depending on many parameters, among which composition and surface texturing, outside temperature, wind velocity, etc. The deposition of water, ice, etc. on structural installations, facilities and infrastructures represents a huge problem in many cold regions. To overcome heavy risks on HV lines, such as the overloading and electricity blackout, new effective strategies - other than the mostly used based on the Joule effect, mechanical removal, electro-impulse methods and application of chemical de-icing fluids - need to be adopted.

New frontiers for superhydrophobic (SH) materials lie in their potential ability to hinder the interaction with water under many different forms so that they might be able to reduce snow, ice or even frost formation and accretion on structural installations, facilities and infrastructures under critical conditions (e.g. at temperatures below 0°C). Here, SH surfaces were obtained by deposition of hybrid nanostructured coatings on aluminum alloy cables commonly used for HV power lines. Two different design approaches to water repellence were pursued: the typical Lotus leaf (*LF*) and the one referring to Slippery Liquid-Infused Porous Surfaces (*SLIPS*), both obtained by the initial deposition of Al_2O_3 nanoparticles, followed by chemical hybridization with a fluoropolymer, plus additional infusion with an ultrasmooth, water-immiscible lubricant overlayer for *SLIPS*. Given the well-known role of surface texture on superhydrophobicity, sandblasted substrates with different roughness were used to assess its influence on the wetting-related performances. Indoor experimental test at lab scale revealed that superhydrophobicity involves the decreasing of shear stress on coated cables even if the ice adhesion strength varies in a more complex way depending on the surface roughness. Materials were also exposed outdoor at test facilities located in the west of Italian Alps, at an altitude of 959 m asl. At T of about -2°C under the conditions of dry snow with a low liquid water content (LWC) and spherical snowflakes morphology, *LF*-like designed sandblasted cables showed a significant delay (some hours) in snow deposition if compared to both the untreated and the *SLIPS* ones. This behavior stood out as the most relevant with respect to the other coated cables, whether smooth or sandblasted. However, under different conditions (-2°C < T < 0°C and wet snow with higher LWC), sandblasted *LF* materials seem to partially lose their ability of delaying show and ice accretion.

10:00am **TS2-ThM-7 Energy Saving Strategy for the Development of Icephobic Coating and Surface***, Y Zheng, J Wang, J Liu, K Choi,* ***Xianghui Hou,*** The University of Nottingham, UK

Aircrafts are frequently exposed to cold environments and ice accumulation on aircraft surface may lead to catastrophic failures. An appropriate solution of ice protection is a critical issue in the aerospace industry . In the R&D of icephobic coating, the current coating design target mainly aims on lowering the ice adhesion strength between the ice and the coating surface. However, as a passive ice protection approach, the use of icephobic coating is often combined with an active ice protection solution (e.g. electro-thermal heating and hot air bleeding), especially for the in-flight application where the reliability of ice protection must be ensured. Therefore, ice adhesion strength is no longer the sole criterion to evaluate the icephobic performance of a coating and a surface. It is a need to establish a better strategy for the design of icephobic coating and surface. In this work, an energy saving strategy has been proposed to assess the de-icing performance of the icephobic coating and surface when heating is involved. The energy consumed for the de-icing operation is used as the key criterion for the overall performance of icephobic coating and surface. Successful validation has been clearly obtained in the evaluation of the de-icing performance of selected coatings and surfaces, which demonstrates a new criterion on the R&D of icephobic coating and surface for ice protection.

10:20am **TS2-ThM-8 Anti-Icing Hard Steel Coating Modified With Polymer Particles***, P García,* ***Julio Mora****, A Agüero,* Instituto Nacional de Técnica Aeroespacial (INTA), Spain

Icing is a severe problem, in particular on aircrafts, with important consequences in safety, energy consumption, ecologic impact, and economy. To fight it, ice protection systems have been developed and have evolved over the last decades, from the highly energy consumption active de-icing systems based on heating, and non-eco-friendly use of de-icing fluids on-ground, to the recent boom that have experimented the technology of passive, anti-icing materials, as well as alternative more efficient new active technologies.

Indeed, very attractive passive surface modification systems have been proposed, decreasing ice accretion and adhesion, but until now, these solutions are not durable and capable of withstanding the harsh conditions to which aircraft surfaces are exposed. A practical approximation would be the development of durable passive solutions that contribute to reduce the power required by the active deicing systems saving energy.

Durable anti-icing coatings with high thermal conductivity deposited over metallic aerodynamic surfaces have been developed and applied by HVOF (High velocity oxyfuel) thermal spray. Mixtures of powders of a high hardness steel and polymer particles were sprayed on stainless steel alloy 304L, and dense 50-100 microns coatings were obtained, with improved anti-icing properties in comparison with the untreated substrate. Extensive preliminary testing of the newly developed coatings has been carried out, including ice accretion in an icing wind tunnel (IWT), employing representative in-cloud icing conditions, allowing to obtain different types of ice: glaze, mixed-glaze, mixed-rime and rime ice. The developed systems showed a significant reduction in ice accretion in comparison with the untreated substrates, with some variations depending on the type of ice and the concentration of added polymer.

Moreover, the durability of the coatings was also examined by repeating icing cycles and by testing for sand and rain erosion resistance. The so tested samples maintained the hydrophobicity with a good water droplet mobility, and in general very promising results in terms of durability after repeated icing/deicing cycles in an IWT.

10:40am **TS2-ThM-9 Development of Superhydrophobic and Icephobic Coatings by Suspension Plasma Spraying***,* ***Ali Dolatabadi****, N Sharifi, R Attarzadeh, C Moreau, M Pugh,* Concordia University, Canada **INVITED**

Suspension plasma spraying (SPS) technique has been used to develop microtextured TiO_2coatings with a hierarchical surface roughness to develop superhydrophobic surfaces. Superhydrophobic coatings demonstrate extremely water repellent properties and can be potentially used for applications such as anti-icing, reduced drag and friction, self-cleaning and corrosion resistance purposes. The focus of this presentation is on engineering the hierarchical morphology or so-called "cauliflower" features using a parametric study approach to optimize the wetting properties of the coatings. It is demonstrated that by carefully designing and controlling the process parameters, rather fine and uniform dual-scale (hierarchical) surface textured coatings can be generated. Finally, icephobic performance of superhydrophobic surfaces are assessed both experimentally and also through a detailed numerical modeling of cloud-sized droplet impact and solidification.

11:20am **TS2-ThM-11 Minimum Required Thickness of a Hydrophobic Topcoat to withstand Cycling in an Icing Wind Tunnel***,* ***Stephen Brown,*** Ecole Polytechnique de Montreal, Canada*; J Lengaigne,* Polytechnique Montréal, Canada*; N Sharifi,* Concordia University, Canada*; L Martinu, J Klemberg-Sapieha,* Ecole Polytechnique de Montreal, Canada

Atmospheric icing occurs on aircraft when supercooled water droplets impact exposed surfaces, and quickly freeze in place. This leads to numerous issues such as increased fuel consumption, flight delays, and even crashes. Superhydrophobic surfaces have been shown to reduce icing by allowing droplets to bounce off the surface upon contact, as well as reducing ice adhesion compared to bare metals. A common technique for the creation of a superhydrophobic surface is to develop a surface with hierarchical roughness, and then to coat this surface with a hydrophobic material. If the hydrophobicity comes purely from the topcoat, however, then the durability of the entire system is limited to the durability of this topcoat.

In the present study, we explore the effect of topcoat thickness on sample durability, with the goal of defining a minimum required thickness to withstand repeated icing/deicing cycles. To achieve the desired hierarchical morphology, a ~100 µm TiO2 coating is first deposited on stainless steel by suspension-plasma-spray. Samples are then coated with a fluoropolymer by thermal evaporation, with thicknesses ranging from 7 to 75 nm. These surfaces show contact angles up to 164 ± 5 °, with hysteresis values as low as 5.8 ± 0.9 ° prior to cycling. The durability of the developed surfaces is tested by performing icing/deicing cycles in an icing wind tunnel, exposing the samples to supercooled microdroplets at a temperature of –10°C and a velocity of 43 m/s. Contact angle and contact angle hysteresis measurements are performed every 10 cycles. It is shown that samples with topcoats thinner than 20 nm completely lose their superhydrophobic properties after only 10 cycles, while samples with 75 nm topcoats retain at least some droplet mobility after as many as 100 cycles.

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