EXPERIMENTAL STUDY OF THE INFLUENCE OF THE TYPE OF MATERIAL, ROUGHNESS AND TEMPERATURE ON ICE ADHESION

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Excessive ice accumulation on outdoor structures such as power networks, communication systems, aircrafts and wind turbines can affect their integrity, with consequent public safety issues and loss of economic activity. To overcome this problem, de-icing methods, including mechanical and thermal techniques, have been developed. However, these techniques are often limited in their application and are generally expensive and time consuming. Recently, many researchers from industries and universities have focused on the development and application of icephobic coatings such as superhydrophobic materials in order to drastically reduce ice adhesion force on exposed equipments. A number of studies have been carried out to better understand the properties of the ice-material interface. However, the fundamental physics of ice adhesion is not well understood yet. This cannot be completed without a thorough knowledge of the mechanisms of ice adhesion of various materials, including the forces involved at the ice-material interface, various factors such as the impact velocity of water droplets, air temperature, water droplet dimension, liquid water content, thickness of accumulated ice as well as coating roughness or porosity. In this study, three factors influencing ice adhesion are investigated: type of material, surface roughness and temperature. Aluminium alloy was chosen for this study since most high voltage conductors are made from it. Titanium, zirconium and zinc oxides were also selected in order to evaluate the influence of the dielectric constant on ice adhesion strength. Surface roughness is also a critical parameter to be taken into account when measuring ice adhesion strength since two phenomena can be observed when ice is formed on such surfaces: the presence of air bubbles within the rough spots and/or pores on the surface and mechanical anchoring of ice. For this purpose, surfaces of different materials were polished in order to study the influence of type of material on the ice adhesion strength. Furthermore, the influence of air temperature on the measurement of the ice adhesion strength on aluminium surface was also studied. Thus, after ice accumulation on the surface of different materials, ice adhesion strengths were evaluated using the centrifuge adhesion test method. Results showed that the strength of ice adhesion depends on the type of the material and its roughness. The higher roughness is (*R*q), the higher ice adhesion strength or shear stress will be (Fig. 1). In addition, a strong correlation between the adhesion strength of ice and its temperature was observed. When temperature decreases, ice adhesion strength increases.

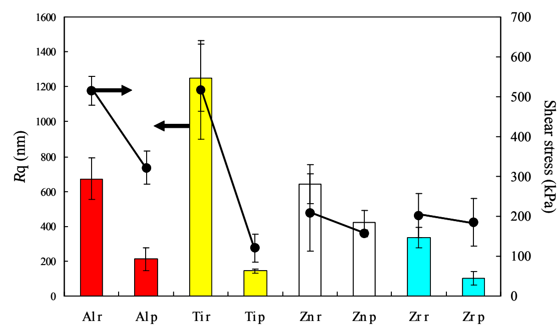

Figure 1. Roughness and ice shear stress vs various materials. M r = raw material and M p = polished material.

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Abstract

Excessive ice accumulation on outdoor structures such as power networks, communication systems, aircrafts and wind turbines can affect their integrity, with consequent public safety issues and loss of economic activity. To overcome this problem, de-icing methods, including mechanical and thermal techniques, have been developed. However, these techniques are often limited in their application and are generally expensive and time consuming. Recently, many researchers from industries and universities have focused on the development and application of icephobic coatings such as superhydrophobic materials in order to drastically reduce ice adhesion force on exposed equipments. A number of studies have been carried out to better understand the properties of the ice-material interface. However, the fundamental physics of ice adhesion is not well understood yet. This cannot be completed without a thorough knowledge of the mechanisms of ice adhesion of various materials, including the forces involved at the ice-material interface, various factors such as the impact velocity of water droplets, air temperature, water droplet dimension, liquid water content, thickness of accumulated ice as well as coating roughness or porosity. In this study, three factors influencing ice adhesion are investigated: type of material, surface roughness and temperature. Aluminium alloy was chosen for this study since most high voltage conductors are made from it. Titanium, zirconium and zinc oxides were also selected in order to evaluate the influence of the dielectric constant on ice adhesion strength. Surface roughness is also a critical parameter to be taken into account when measuring ice adhesion strength since two phenomena can be observed when ice is formed on such surfaces: the presence of air bubbles within the rough spots and/or pores on the surface and mechanical anchoring of ice. For this purpose, surfaces of different materials were polished in order to study the influence of type of material on the ice adhesion strength. Furthermore, the influence of air temperature on the measurement of the ice adhesion strength on aluminium surface was also studied. Thus, after ice accumulation on the surface of different materials, ice adhesion strengths were evaluated using the centrifuge adhesion test method. Results showed that the strength of ice adhesion depends on the type of the material and its roughness. The higher roughness is, the higher ice adhesion strength or shear stress will be. In addition, a strong correlation between the adhesion strength of ice and its temperature was observed. When temperature decreases, ice adhesion strength increases.

Keywords: Ice adhesion; material; ice microstructure; surface roughness; temperature; anodization; porosity;

I. INTRODUCTION

Excessive ice accumulation on outdoor structures such as power networks, communication systems, aircrafts and wind turbines can affect their integrity leading to potential disastrous consequences as far as public safety and economic activity are concerned. To overcome such situations, de-icing techniques such as mechanical and thermal ones have been developed [1]. However, these techniques are often limited in their application and are generally expensive and time consuming. Recently, many researchers from industries and universities have focused on the development and application of icephobic coatings such as superhydrophobic materials to drastically reduce ice adhesion force on exposed equipments [2-9].

A number of studies have been carried out to better understand the properties of the ice-material interface. However, the fundamental physics of ice adhesion is not yet understood. This cannot be completed without a thorough knowledge of the mechanisms of ice adhesion of various materials, including the forces involved at the ice-material interface, various factors such as the impact velocity of water droplets, air temperature, water droplet dimension, liquid water content, thickness of accumulated ice as well as coating roughness or porosity. Therefore, the aim of this paper is to study the influence of the type of materials, roughness and temperature on ice adhesion.

Several studies were dedicated to ice adhesion measurements on different materials (raw or commercial) using various techniques [10-15]. In the present study, four metallic or alloys compounds and their oxides were chosen. The aluminum alloy 6061 was chosen because of its use for high voltage overhead conductors. Titanium, zirconium and zinc oxides were also selected in order to evaluate the influence of the dielectric constant of their oxide surface layers upon ice adhesion strength.

Surface roughness is also a critical parameter to be taken into account when measuring ice adhesion strength since two phenomena can be observed: the presence of air bubbles within rough areas and/or pores on the surface and mechanical anchoring of ice [4, 7, 8, 15 and 16]. For this purpose, different degrees of surface roughness (or porosity) were prepared and the corresponding ice adhesion strengths measured.

II. EXPERIMENTAL PROCEDURE FOR MEASURING ICE ADHESION STRENGTH

A. Surface preparation

Surfaces of raw samples (3 x 5 cm) were cleaned in an ultrasound bath using acetone and ethanol, rinsed in deionized water. They were then blow within a nitrogen flow to eliminate all residues and dusts. The polished samples were obtained using specific procedures from the Struers Company. Between each polishing steps, samples were cleaned in an ultrasound bath using acetone, rinsed in deionized water then dried in a nitrogen flow.

B. Ice accumulation

Samples were attached on support beams and the assemblies were placed in a wind tunnel maintained at a temperature of -10°C. When the tunnel reached the targeted temperature, a 30 minute waiting time was necessary to make sure that the beams reach the same temperature of the tunnel before beginning ice accumulation. After the desired ice thickness was obtained, the beams were kept at -10°C for 30 minutes before being subjected to the adhesive strength (or shear stress) measurements. The thickness of ice is measured by an electronic caliper and the weight of the ice was also evaluated. The environmental parameters used for ice accumulation are summarized in Table 1.

TABLE 1. Parameters used during the ice accumulation

Parameters	***Value***
icing temperature (°C)	-10 ± 0.2
wind speed (m s^{-1})	10 ± 0.2
water liquid content (g m^{-2})	2.2 ± 0.3
Water droplet medium volume diameter (µm)	69 ± 4.3

C. Ice adhesion force measurements

Ice adhesion forces were measured using centrifuge adhesion tests (CAT). The principle of which consists of rotating a beam at a variable and increasing speed until the ice detaches from the coupons fixed at one end of the beam. The shear stress is then calculated from equation (1) [4, 6-8 and 13]:

$$\tau = \frac{mr\omega^2}{A} \qquad [1]$$

Where τ is the shear strength (Pa), m is the ice mass (kg), r is the beam radius (m), ω is the rotation speed (rad s^{-1}) and A is the surface area (m^2).

III. RESULT AND DISCUSSION

A. Surface roughness and ice adhesion force

Sample roughnesses were evaluated using an optical profilometer (average of five to six measurements). The value of the roughness is estimated from the quadratic average, *R*q, given in nanometer. The ice shear stresses measured using the CAT system together with the *R*q values vs the type of materials are shown in Fig. 1. For materials taken separately (rough vs polished), it is clear that higher was the roughness, higher was the ice adhesion strength. Ice adheres strongly to rough surfaces because water penetrates into the porous zones and occupies more volume by solidifying leading to mechanical anchoring.

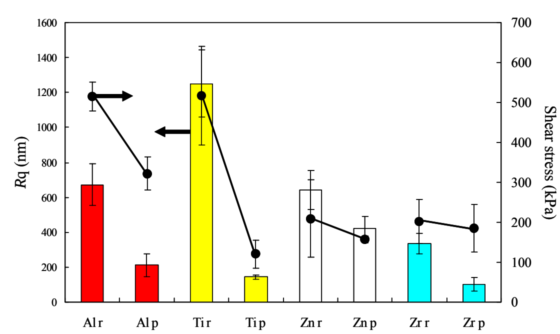

Figure 1. Roughness and ice shear stress vs various materials.
M r = raw material and M p = polished material.

However, comparisons between the nature of the materials (Al, Ti, Zn and Zr) and shear stresses are trickier since these samples have different roughness factors. Secondly, it should be noted that each of these metals have natural protective oxide layers (Al_2O_3, TiO_2, ZnO and ZrO_2) and the corresponding dielectric constants are not the same: ε ≈ 10, 60, 8 and 25 respectively [17]. It is well known that lower is the dielectric constant lower is the ice adhesion force. Therefore possible correlations between ε and shear stress (F_M) values are only possible between materials having similar *R*q values. By analyzing the data from Fig. 1, only three comparisons could be made:

$$F_{\mathrm{Al\,r}}\ (\varepsilon \approx 10) > F_{\mathrm{Zn\,r}}\ (\varepsilon \approx 8)$$
$$F_{\mathrm{Zr\,r}}\ (\varepsilon \approx 25) > F_{\mathrm{Zn\,p}}\ (\varepsilon \approx 8)$$
$$F_{\mathrm{Zr\,p}}\ (\varepsilon \approx 25) > F_{\mathrm{Ti\,p}}\ (\varepsilon \approx 60)$$

The first and second comparisons are in accordance with the fact that lower is the dielectric constant, lower is F_M. However, when comparing Zr p and Ti p, it was found that the above statement was not valid. Further investigations will be needed to explain this fact such as measuring the thickness of different oxide layers as well as evaluating the dielectric constants of the chosen materials using impedance spectroscopy techniques.

B. Influence of ice structure

The adhesion strength of ice formed at -20 °C on a polished aluminum surface was found to be 390 ± 35 kPa, which is higher than that what was obtained at -10 °C (322 ± 32 kPa). This variation is attributed to the microstructure of the accumulated atmospheric ice and depends on the air temperature, size of water droplets, wind speed and the liquid water content [18]. In fact at -10°C, the average ice grain size was less than 0.5 mm, see Fig. 2A, and these values drastically decreased as the temperature falls to T = - 20 ^{o}C, see Fig. 2B [18].

The study also underlined that the primary ice layer accumulated at -20 °C on the aluminum surface contained fine grains compared to those observed at – 10 ^{o}C. This was due to the fast heat transfer between the aluminum surface and water droplets that led to high ice adhesion strength [18].

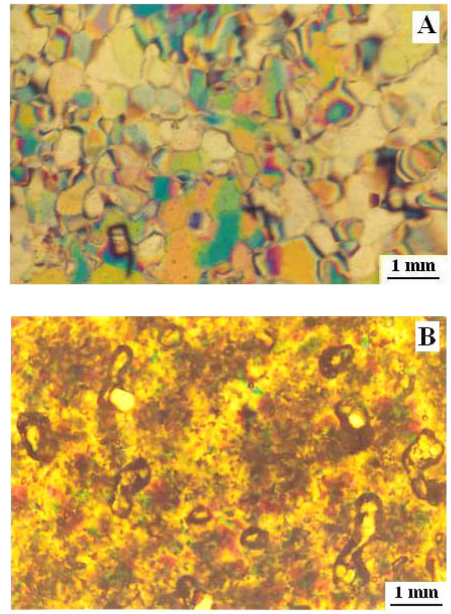

Figure 2. Ice microsturcture accumulated at -10 (A) and -20 (B) °C (from ref. [18])

C. Surface roughness Influence

To further understand the influence of surface roughness on ice adhesion strength, Al 6061 samples were anodized in different conditions. Al coupons were either used as received, grinded or polished. The anodizing procedure was: H_3PO_4 10 % w/w, T = 18 °C and 50 V applied voltage [6]. The only variable parameter was the electrolysis time. The anodized samples were cleaned with hexane in an ultrasonic bath, rinsed with acetone and deionized water, then dried in a nitrogen stream. Using scanning electron microscopy, the surface morphology of the samples exhibited a porous structure made of Al_2O_3 that is commonly observed in such cases. In fact, when aluminum is anodized in an acid solution a compact barrier forms initially followed by growth of porous film, see Fig. 3 [19].

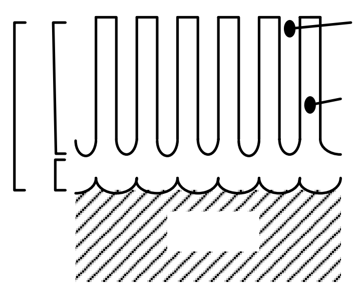

Figure 3. Anodized aluminum layer structure

The images taken by scanning electron microscopy, see Fig. 4, show that pores and walls were formed. Pore diameter was estimated to be of the order of 50-60 nm for 20 minutes, 70-80 nm for 32 min, 80-90 nm for 60 min and 80-140 nm for an anodisation time of 90 min. Regarding the thickness of the anodized layer, it was also measured by scanning electron microscopy on cross-sectionned samples prepared by metallography. The average thickness was 1 µm for 20 min, 1.35 µm for 32 min, 2 µm for 60 min and 5 µm for samples anodized during 90 min.

To assess the hydrophobicity of the different samples, static contact angle (CA) measurements were performed (Table 2). It was found that the longer the anodisation time is, the higher is the hydrophilicity of the surface. It was also clear that higher the roughness (without anodisation) or the porosity (after anodisation) is, higher is the hydrophilicity. Consequently, for high surface energy materials such as metals or oxides it is recommended to polish smoothly the material's surface to enhance hydrophobicity and lower ice adhesion strength.

As far as anodized samples were concerned, a drastic diminution of the CA was observed when the anodisation time was increased. In the same time, both porosity and oxide thickness increased. Deposited water droplets tend to penetrate easily at high porosity and thickness leading to hydrophilic properties. To obtain hydrophobic or superhydrophobic properties with such Al_2O_3 surface layers, subsequent impregnation with hydrophobe polymers is necessary [6].

TABLE 2. Contact angle of different aluminum surfaces

Al 6061	Anodization time (min)	CA (°)
Raw	0	75 ± 3.3
Polished	0	88 ± 3.2
Grinded	20	61 ± 3.7
Grinded	32	37 ± 6.6
Grinded	60	24 ± 2.2
Grinded	90	17 ± 1.3

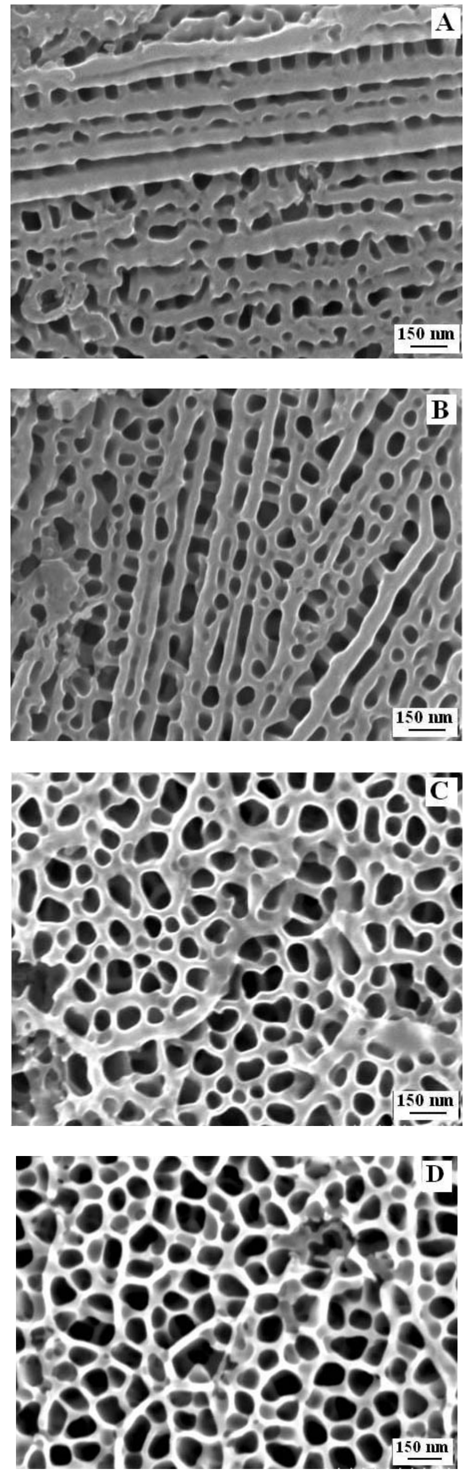

Figure 4. Anodized aluminum surface: (A) 20, (B) 32, (C) 60 and (D) 90 minutes

IV. CONCLUSIONS

Ice adhesion strength depends on the type of the metal or alloy material and particularly on its oxide layer as well as its surface roughness. It was found that the higher the roughness is, the higher ice adhesion strength will be. The results show that a correlation exists between the type of the metallic natural oxide layers (Al_2O_3, ZnO and ZrO_2) and their corresponding dielectric constants. The higher the latter is, the higher is the ice shear stress. However, this statement was not true for TiO_2 and further investigations will be needed. As far as Al 6061 alloy was concerned, several porous Al_2O_3 porous layers were produced by anodisation. The hydrophilic characteristics of these layers were enhanced by increasing their porosity and thickness. Additionally, a strong correlation between the ice adhesion strength and temperature was observed, as temperature decreases when ice adhesion strength increases.

Future works will bear on ice adhesion strength measurements on different thick oxide layers (> 2 μm) on Al, Ti, Zn and Zr vs temperature, dielectric constant values and morphology. Measurements will be also performed on oxide porous structures impregnated with hydrophobic materials such as Teflon®.

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