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An Experimental Study to Evaluate the Droplet Impinging Erosion Characteristics of an Icephobic, Elastic Soft Surface

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Abstract

Elastic soft material/surface, such as Polydimethylsiloxane (PDMS), is a perspective, useful and low-cost hydrophobic and icephobic coating. While it has been reported to have good mechanical durability, its erosion durability under the high impacting of water droplets pertinent to aircraft inflight icing phenomena has not been explored. In this study, the droplet imping erosion characteristics of an icephobic PDMS surface/material is evaluated systematically upon the dynamic impinging of water droplets at different impact velocities (~ up to 75m/s), in comparison with other state-of-the-art icephobic materials/surfaces, such as superhydrophobic surface (SHS) and slippery liquid-infused porous surface (SLIPS). Surprisingly, the contact angle (CA) of the elastic PDMS is shown to have an over 20° increase (from 105° to 128°), which represents better hydrophobicity, after the erosion test which is mainly contributed to the higher roughness of the eroded PDMS surface. As for the icephobicity evaluation, intact PDMS was found to has ultra-low ice adhesion (~8 kPa), in comparison with SHS (i.e., ~100kPa) and SLIPS (i.e., ~35kPa). PDMS also shows outstandingly stable ice adhesion during the erosion test (i.e., fluctuation only within ~4kPa) as a result of the growth of cracks on the PDMS surface and the increased surface energy.

Introduction

Polydimethylsiloxane (PDMS) is a silicon-based polymer and has been found for several decades but its excellent icephobicity is just realized in the last few years. It has some unique mechanical and optical properties such as viscoelasticity, hydrophobicity, optically transparency in the UV-vis regions and biocompatibility. Due to these special features, PDMS has been widely applied to the manufacture of MEMS[1], biomedical model[2] and anti-icing coating[3]. Liu et al. introduced the influence of heating temperature on a mechanical characteristic of PDMS and found that it does not suffer any degradation with low temperature but will have reduced mechanical strength until temperature reaches 200 °C[4]. Khanafer et al. tried a different mixing ratio to get the best PDMS with proper elastic modulus for the simulation of blood flow in arteries[2]. Generally speaking, PDMS is a hydrophobic material with low surface energy but Bhattacharya et al. made a hydrophilic PDMS surface by using Oxygen-Plasma surface treatment. It is probably contributed by the generation of a thin layer of oxide on the surface of PDMS[5]. Although PDMS has been commonly and deeply studied, icephobicity of it is just investigated from these decades. Icephobic coating is usually superhydrophobic surface (SHS) or slippery liquid-infused porous surface (SLIPS). SHS can be applied for anti-icing because it has intrinsic low surface energy and reduces the contact area by its nanoscale and rough pattern[6–10]. The icephobicity of SLIPS is mainly contributed by the lubricating liquid which can steadily adhere within the substrate membrane but can strongly repel water or any unwanted liquids[11–14]. However, some undesired drawbacks impede the real application of SHS and SLIPS for anti-icing. Firstly, condensing water will come into the small asperities on the SHS where wetting state transfers from Cassie-Baxter state to Wenzel state where the ice adhesion will increase dramatically and sometimes even higher than the surface without the asperities[3]. In another respect, the lifetime of SHS is also a limitation. Zhang et al. studied the durability of a superhydrophobic coating and found the hydrophobicity of it will suffer a quick degradation and the ice adhesion of it increases dramatically to 0.4 MPa within 1-hour water erosion test[9]. SLIPS can have more stable and ice repellent performance than SHS but the depletion of lubricant liquid is a serious challenge for the durability of it[14–16]. PDMS is a kind of durable icephobic material offering ultra-low ice adhesion strength. It provides an option to avoid the disadvantages of SHS and SLIPS. Beemer et al. used a PDMS, with very low adhesion to ice (~ 5kPa), to conduct a mechanical durability test and they found that the ice adhesion does not have an observable change (< 2kPa) within 1000 sandpaper abrasion cycles[17]. They also explored the mechanism of separation of ice from the PDMS that trapping air cavity at the interface contribute significantly to the ultra-low ice adhesion strength. Liu et al. conducted the droplet erosion test by spraying high-pressure water onto the coating[18]. Their results show few degradations of the wettability and morphology of PDMS but they did not evaluate the icephobicity of PDMS. He et al. presented a facile preparation of PDMS by low-cost raw material and evaluate the durability of PDMS by conducting icing/de-icing cyclic tests which shows PDMS can be used in a long-term because ice adhesion almost keeps a constant within 25 cycles[19].

Aircraft icing severely threatens aviation safety every year, and over 700 aircraft accidents are reported during 1998-2007[20]. The icing on the wind can generate unwanted effects on the aerodynamic performance, and also, can increase the weight at the same time. Applying icephobic coating on the aircraft is a useful and prospective solution for the aircraft icing. Meanwhile, compared with other anti-icing technique, coating merely affects the aerodynamic performance. Liu et al. firstly applied PDMS for aircraft icing mitigation and discovered that the amount of ice on airfoil can have a remarkable shrink[3]. The result shows PDMS is an effective anti-icing coating for the aircraft but a further thought is how long time is PDMS effective for aircraft anti-icing. Most of those ice-related problems happened as airplane encountered with super-cooled droplets cloud or plenty of ice crystals. Droplet impingement with such high speed can generate undesirable damage to the coating icephobic characteristics or even totally remove the coating. Therefore, it is necessary to study the degradation process of PDMS with high-speed spray erosion and properly evaluate the durability of it.

In the present study, the droplet erosion durability of a PDMS is completely discussed. The PDMS coating is made from vinyl-terminated PDMS (v-PDMS), hydride-terminated PDMS (h-PDMS) and trimethyl-terminated PDMS (t-PDMS). The stiffness of PDMS can be varied by adding a different amount of t-PDMS and the material containing more t-PDMS will become softer with lower shear modulus. Erosion test is conducted by an open wind tunnel with different wind speed, liquid water content (LWC) and duration. The hydrophobicity and icephobicity of the eroded coating are characterized in terms of static contact angle and ice adhesion strength at -10°C. This study also gives a comparison of ice adhesion of the different icephobic coating.

Experimental details

Fabrication of PDMS gels

The PDMS coating is fabricated via hydrosilylation of v-PDMS with h-PDMS. The stiffness of PDMS is tuned by adding t-PDMS with different mixing ratio to the hydrosilylation mixture. More t-PDMS or higher mixing ratio will lead the mixture to be softer with lower shear modulus because the t-PDMS can weaken the cross-link and result in non-robust cross-linked PDMS gels and also, softer PDMS will own lower ice adhesion stress[17]. The concentration of t-PDMS in the selected PDMS is 80% which is the maximum mixing ratio because the hydrosilylation mixture will remain liquid-like and cannot form a gel-like coating which can have good mechanical durability. The 80% PDMS can have the minimum ice adhesion strength but the mechanical durability of it is probably the worst compared with other PDMS coatings with low t-PDMS concentration. Therefore, it is meaningful to study the droplet erosion durability of 80% PDMS. The thickness of the PDMS also has an effect on the adhesion of ice but it will not be touched in this study because all coatings have the same thickness. The PDMS gels are prepared by spin coating the mixture on a microscope glass slide.

Droplet erosion test

The durability test is conducted by the open wind tunnel shown in figure 1 which can accelerate the spray droplets driven by a high thrust electric ducted fan (EDF, JP Hobby). The wind speed at the wind tunnel outlet is measured by a Pitot tube which can vary from 30m/s to 95m/s and distance from wind tunnel outlet to the test plate is 50mm. A nozzle (BIMV-11002 nozzle) is implemented in the middle section of the wind tunnel, which can spray water droplets in the flow direction. This nozzle is designed to mix compressed air and water inside the nozzle that air flows into the center of the nozzle, while water goes along its circumference so that droplets are sprayed forward without swirling. External high-pressure air and water can control the size of spray droplets and otherwise, LWC at the outlet. The maximum water and air pressure of the nozzle are 60psi and its minimum working pressure is 30psi. Deionized (DI) water is used in this study to eliminate the corrosion to the nozzle and the PDMS coating. This wind tunnel can provide a flow with LWC up to 13g/m3 at the outlet with a wind velocity of 75m/s and such high LWC can accelerate the degradation of SHS. This wind velocity is used for erosion test in this study. Droplets will move nearly 1 meter in the wind tunnel before impacting on the target plate where the PDMS is uniformly coated, which the size of the droplets may significantly change. Hence, it is necessary to measure the droplet size at the outlet. The size of the droplets is measured by shadowgraph technique which uses a high-speed CCD camera (FastCam Photron) to record the shadow of droplets with the bright light background provided by a powerful LED lamp (MiniConstellation). High light intensity can increase the signal-noise ratio and meanwhile, reduce the measurement error. Finally, over 8000 single droplets are analyzed by ParticleMaster to get the distribution of droplets size impacting on the PDMS. Figure 1(b) reveals the velocity profile near the outlet measured by particle image velocimetry (PIV) with a free stream velocity of 65m/s and a test plate put at the downstream 33mm from the outlet. A highly uniform flow field can be seen near the outlet in figure 1(b) as well as flow would slow down as approaching the test plate due to increasing pressure drag. Figure 1(c) gives the droplet size distribution at the wind tunnel outlet with 40psi water and air pressure which is the working condition of the nozzle in this study. The mean diameter of droplets is 17μm and the Sauter mean diameter (SMD) is approximately 30μm.

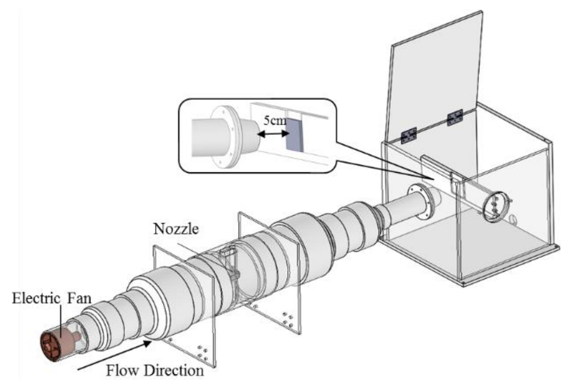


Figure 1: Droplet erosion wind tunnel setup.

Hydrophobicity and icephobicity measurement

Measurement setup of contact angle (CA) and hysteresis consists of a high-speed camera (PCO 1200hs) and a syringe pump (Genie Touch). The shape of the droplet is recorded by the camera and will be analyzed by ImageJ to get the angle at the contact line. A syringe pump (Genie Touch syringe pump) is used to squeeze or release a specific volume of water in a specific time. 10 μL water is squeezed for contact angle measurement and 50μL water will be squeezed and retracted in 10 seconds for hysteresis measurement. The average of CA at both sides of the droplet is used as the CA. The measurement will be repeated at least three times for each experimental case to eliminate the random error.

Figure 2 shows the ice adhesion strength measurement apparatus. Meuler et al. used a similar apparatus to measure ice adhesion for different metal and composite materials and get quantitatively meaningful results[21]. The test plate will be put into a climate chamber where housed with low-humidity CO2 provided by dry ice to prevent frost over the test surface. The temperature in the climate chamber is controlled by a Peltier cooler (TETech CP-061) which is controlled by an external digital thermal controller (TETech TC-48-20) and can achieve -20°C under ambient room temperature. A 3-D printed hollow cylinder sample made with the ViewWhite plastic material whose diameter is 11mm is used as an ice sample holder. At least two cylinder samples are put in the impingement area and 0.5ml

DI water is infused into each of them. In this paper, after infusing water to the sample holder, the temperature will go down to -10°C and stabilize for 15 minutes allowing the water to be fully frozen under the determined temperature. A force gauge (Mark-10 series 4) is implemented on a linear actuator (Newport CONEX-LTA-HS) which moves at a rate of 0.5mm/s until the cylinder sample is detached from PDMS. The sample rate of force gauge is 22000 samples/s to get the maximum force which can be converted into ice adhesion by equation (1) where A is the cross-sectional area of ice holder and F_{max} is the recorded maximum force. The probe locates less than 2 mm above the surface to minimize the cohesion stress as detachment happening.

$$\tau = \frac{F_{max}}{A} \qquad (1)$$

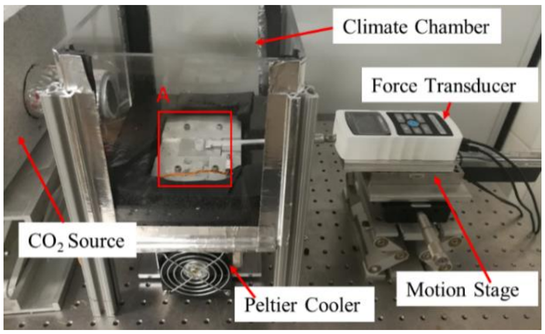


Figure 2: Ice adhesion strength measurement setup.

Results and discussions

Hydrophobicity and surface topology test

The hydrophobicity of the coating is characterized in terms of CA and receding angle (RA) in Fig. 3. Ma et al. found the durability of SLIPS is much better than the SHS and as a result, this experiment is designed according to the SLIPS study but slightly elongates the experiment duration to get a more convincing comparison. The CA of SHS has a noticeable decrease after erosion test, which is mostly attributed to the smaller surface roughness on SHS. The CA of SLIPS does not have noticeable change in the whole period but the most lubricant oil is actually removed. The initial CA of the PDMS is 105° ± 3° indicating the PDMS is a hydrophobic material which agrees with the previous results[3,22]. Surprisingly, CA of PDMS has an over 20° increase. This trend is unexpected because it means the hydrophobicity of PDMS becomes better after erosion test. It is interesting to study the reason why CA of PDMS has a reversed trend to the other two coatings. CA is mainly determined by the liquid's surface tension and coating's surface chemistry given by Young's equation but for the hydrophobic surface, this parameter can also be influenced by surface roughness according to the equation of Wenzel state and Cassie-Baxter state[23]. Generally, higher roughness on a hydrophobic surface such as PDMS can probably contribute to the higher CA which probably is a reason for the increase of CA on the PDMS. Fig 5. Shows the surface topology of intact PDMS and eroded PDMS. It is very clear that after long-term erosion, pits and grooves appear on original smooth surface. In another word, the surface become rougher after erosion test. Therefore, the increase of CA on PDMS is highly contributed by the higher surface roughness.

In figure 5, small cavities are generated on surface firstly before pits and grooves appearing. Single high-speed droplet impingement cannot generate small cavities in figure 5(b), since millions of droplet has impinged on PDMS at that time. One reason for this evolution features is the surface fatigue. However, this reason only can be properly applied for the speed less than 100m/s because higher velocity could probably lead to severe plastic deformation on PDMS. In the real application, like for aircraft (velocity > 200m/s), PDMS probably will be totally removed due to the huge impact pressure. Therefore, PDMS should enhance the yield strength of it for the application to aircraft. Current results indicate that PDMS is an excellent durable icephobic material for wind turbine and UAV where wind speed is around 100m/s.

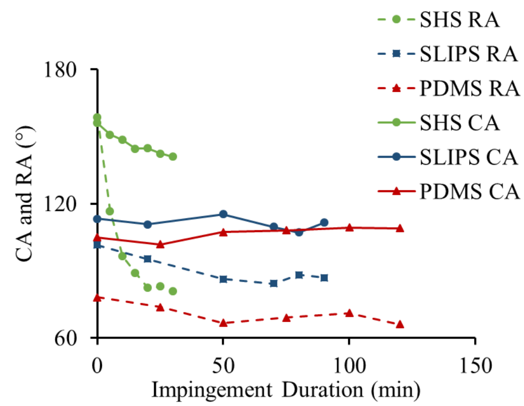


Figure 3: Evolution of CA of the PDMS at impact velocities V=75m/s as a function of impingement duration and times.

Ice adhesion test

The ice adhesion results of the SHS (triangle symbol), SLIPS (rectangular symbol) and PDMS (cycle symbol) are shown in figure 4. The ice adhesion of intact PDMS (8 ± 5 kPa) is the lowest among these three materials which agree with the result of Beemer et al[17]. SLIPS has a relatively high ice adhesion (35 ± 3 kPa) and the initial ice adhesion of SHS is 100 ±5 kPa which is in the same order with the bare aluminum or the steel (500-1000 kPa). SHS has high ice adhesion because SHS will transfer to Wenzel state in the icing process because condensed water will go into the small asperities which greatly increase the ice adhesion. SLIPS can resist water or ice by its hydrophobic lubricant oil and ice would like to have a cohesive failure which is much weaker than the adhesive failure on the surface. PDMS can achieve ultra-low ice adhesion by trapping air cavities in that the interface which remarkably reduces the contact area but ice is still attached to the surface by Vander Waals force. As for the time evolution of the ice adhesion, the SHS can be shown a tremendous increase to nearly 400 kPa within a small impingement time (0.8×10^5) and the SLIPS has some observable fluctuation but basically keeps stable. It is very exciting to show that PDMS is a very durable icephobic material because the ice adhesion of it has a very tiny change (8 kPa to 12 kPa) after suffering a long-term erosion test. Considering the high LWC in the experiment, in the real fog weather ($LWC=0.05g/m^3$), PDMS probably could have the same performance in several days or even longer. This result is also very surprising because in figure 5, surface topology has a considerable modification (generation of grooves and pits) after long-term erosion and the hydrophobicity of eroded PDMS also has a visible change. Intrinsically, a noticeable higher ice adhesion should exist on the eroded PDMS because the PDMS has been severely damaged. Besides, theoretically, the relationship of ice adhesion and hydrophobicity, given by equation (3), tells us that ice adhesion also should increase because RA has a noticeable change in figure 3 [21]. The right term of equation (2) reveals the surface energy of materials. This equation predict the ice adhesion of PDMS should have a 10% (0.8kPa) increase but due to the accuracy limitation of test facility, such tiny change is hardly to be observed. Actually, fluctuated ice adhesion is an indirect evidence of the correctness of this theory. In addition to hydrophobicity degradation, cracks on PDMS is beneficial to the icephobicity enhancement. Pits and grooves on the PDMS lead to the increase of the number of crack initiator, which technically decrease the ice adhesion. Lower ice adhesion which is also observed by He et al when they measured the ice adhesion of the PDMS with regular microscale pattern [24]. In another word, the erosion test can enhance the icephobicity of the PDMS by generating cracks on it. Therefore, the stable ice adhesion of PDMS is based on two mechanisms: one is erosion increase the surface energy and another is erosion generates more cracks. This two phenomenon have opposite effects on the ice adhesion of PDMS and offset each other. This discussion gives us the inspiration to design erosion resistant material that initial microscale or nanoscale patterns on PDMS is not preferred because erosion will damage patterns and probably increase the ice adhesion.

In this study, we freeze bulk water on the sample to measure the ice adhesion and evaluate the icephobicity of PDMS. One of the benefit is that we can get perfect ice-PDMS interface, without bubbles or any defects, and accurate ice adhesion data but impact ice mostly happens in reality. Technically, the ice adhesion of impact ice should be lower than the ice adhesion of bulk frozen water, since interface of impact ice is more defective. However, regardless of impact ice or bulk frozen ice, the durable icephobicity of PDMS is incontrovertible, and next probable work should be increase the yield strength of PDMS to meet the requirement of aviation system.

$$\tau \propto \sqrt{1+\cos(RA)} \qquad (2)$$

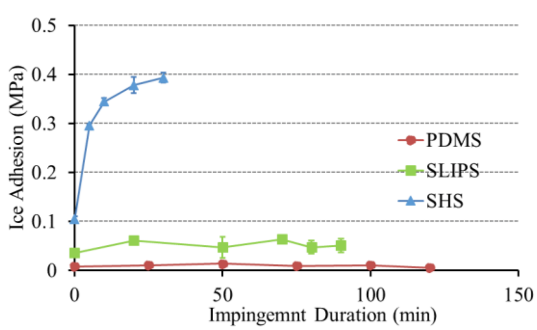
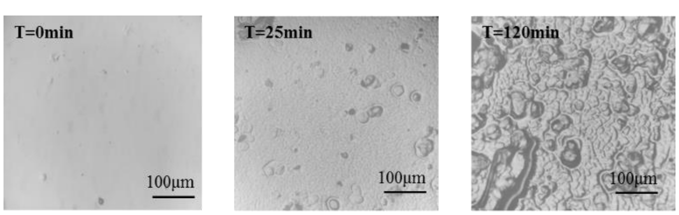


Figure 4: Ice adhesion stress of SLIPS and SHS with an impinging velocity of 75m/s and the PDMS with different impinging velocities as a function of impingement times.



(a) (b) (c)

Figure 5: PDMS surface topology with an impinging velocity of 75m/s.

Conclusions

The droplet erosion durability of a PDMS is studied by recording the change of hydrophobicity, icephobicity and surface topology. It was found that after the erosion test, the CA always have an increase. The growth of CA is surprising as it means the enhancement of the hydrophobicity of PDMS and the elevation is mainly due to higher surface roughness. PDMS is a durable icephobic coating because, after a long time erosion test, its ice adhesion still keeps at the ultra-low level (8kPa). The stability of the icephobicity of PDMS is probably contributed by higher surface energy and higher surface roughness.

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Definitions/Abbreviations

CA	contact angle
RA	receding angle
SHS	superhydrophobic surface
SLIPS	slippery liquid-infused porous surface
PDMS	polydimethylsiloxane
LWC	liquid water content