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Fabrication of a durable anti-icing composite coating based on polyurethane elastomer and silica nanoparticles

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Abstract

In order to design and fabricate hydrophobic, durable and anti-icing coating for aircraft, a polyurethane elastomer matrix was hydrophobically modified and incorporated with fluorinated SiO$_2$ nanoparticles to prepare a micro/nanostructured coating. The micro/nanostructured coating with low surface energy displayed significantly improved mechanical properties and hydrophobicity, which exhibited the water contact angle of 162$\degree$ as well as the sliding angle of 2$\degree$. The coating is highly stretchable, which could sustain large-scale extension, and exhibits tensile strength and elongation at break up to 16.22 MPa and 385%, respectively. Furthermore, the coating exhibited a remarkably weak ice adhesion strength of 14.33 kPa, on which the accumulative ice is prone to fall off under natural wind and its own weight. The coating sustained long-term superhydrophobic properties and anti-icing performance even after 1000 abrasion cycles. The proposed method for the production of scalable superhydrophobic coating is cost-effective and can be applied in aerospace and automotive anti-icing systems.

1. Introduction

Ice formation and accretion on critical control surface of aircraft during cold weather has a series of detrimental effects of disruption of smooth air flow, reduction of the lift generated by wings and increase of drag, which is believed to be a great threat to the aviation safety [1]. To ensure the aviation safety and long-term operation of aircraft in cold weather conditions, various anti-icing techniques such as mechanical, thermal and chemical anti-icing systems have been developed to reduce ice formation [2–4]. However, these active anti-icing approaches have drawbacks of weight gain and extra energy input. Compared with active anti-icing approaches, passive anti-icing methods like superhydrophobic coatings have attracted extensive research interests due to their advantages of high efficiency and zero-energy input [5–8].

Inspired by lotus-leaf effect, superhydrophobic surfaces with hierarchical micro/nanostructures capture air pockets, reduce actual solid-liquid contact area and exhibit water contact angle (WCA) greater than 150$\degree$ and sliding angle (SA) below 10$\degree$. Moreover, the trapped micro-nano air pockets can cut down thermal transmission between the surface and the water molecules, delay ice icing time at low temperature and reduce the ice adhesion strength by providing stress loci to cause initiation of cracks at the interface between ice and surface [9–12]. Therefore, the ice formed and accreted on superhydrophobic surfaces can be easily eliminated due to its own weight or natural wind [13–15]. With this in mind, it is urgently needed to develop superhydrophobic coatings with ultralow ice adhesion strength for the aircraft anti-icing system. Based on the Wenzel wetting model, the preparation methods for superhydrophobic coatings are mainly categorized on making rough surface from hydrophobic materials [16, 17], modifying rough structures with low surface energy materials [18, 19] and using materials with low surface energy to fabricate integrated rough microstructures [20–23]. However, the superhydrophobic coatings (SHCs) fabricated by the first two approaches are fragile and often destroyed during application as a result of surface flaking. The SHCs prepared by the third method tend to form a self-similar structure and sustained the original micro/nanostructure and composition during the mechanical abrasion
cycles [24, 25]. Therefore, the third method is preferable to be used to prepare robust superhydrophobic coatings. Lu et al. combined commercial adhesives with TiO₂ nanoparticles to create a self-cleaning superhydrophobic coating and improved the coating’s mechanical performance [21]. Peng et al. incorporated polytetrafluoroethylene particles with epoxy resin to prepare SHCs, which displayed good jet impact resistance and excellent adhesion and [22]. However, polymers used in the above-mentioned methods are thermoplastic polymers. Compared to thermoplastic polymers, elastic microstructure will avoid being damaged by generating elastic deformation when it is subjected to shear force and pressure during the abrasion process. Once the load is withdrawn, the elastic deformation will immediately rebound and restore the original microstructure [26–28]. Therefore, elastomers enjoy great advantages in constructing superhydrophobic surfaces with better abrasion resistance.

Polyurethane elastomer (PUE) based on hydrogenated acrylonitrile butadiene rubber (HNBR) displays excellent oil, heat and abrasion resistance combined with superior mechanical properties [29–31]. However, HNBR-based PUE with relatively high surface energy is hydrophilic. The hydrophilicity restricts its applications in the fabrication of superhydrophobic or hydrophobic coatings. Based on the above discussion, in this paper, we used dihydroxydodecyl-terminated polydimethylsiloxane with low surface energy to modify the HNBR-based PUE, and incorporated fluorinated SiO₂ nanoparticles to prepare a SHC with integrated micro/nanostructure and superb mechanical performance. The HNBR-polydimethylsiloxane graft copolymer serves as the matrix and greatly decreases the composite coating’s surface energy. The SiO₂ nanoparticles aggregates densely accumulate and form the micro/nanostructures. The combination of low surface energy and the micro/nano hierarchical structures works together and leads to the composite coating’s superhydrophobicity which can greatly reduce the contact area between the water and coating surface and prevent water from icing and accumulating when the temperature drops below zero centigrade. Besides, the coating exhibits superior elasticity, which can promote the removal of ice by providing stress loci to initiate the ice cracks. Furthermore, we evaluated the micro/nanostructure of the composite coating surface and studied the anti-icing properties and mechanical durability of the coating.

2. Experimental

2.1. Materials

The hydrophobic polyurethane elastomer (PUE) was synthesized by using hydrogenated hydroxy-terminated butadiene–acrylonitrile copolymer (HHTBN) and dihydroxydodecyl-terminated polydimethylsiloxane (HPSO) as the soft segments, isophorone diisocyanate (IPDI) as the hard segments and 1,4-butenediol (BDO) as the chain extender. HHTBN, with a hydroxyl group value of 0.5774 mmol·g⁻¹ and a number average molecular weight of 8700 g·mol⁻¹, was supplied by Qilong Chemical Co., Ltd, the hydrogenation degree of which is nearly 100%. HPSO, with a hydroxyl group value of 0.6667 mmol·g⁻¹ and a number average molecular weight of 7535 g·mol⁻¹, was offered by Institute of Chemistry, Academia Sinica. IPDI was purchased from Shanghai Titan Technology Co., Ltd. Dibutyltin dilaurate (DBTDL) was purchased from Beijing Huawei Ruike Chemical Co., Ltd as the catalyst. BDO was purchased from Aladdin Reagent Co., Ltd. Perfluoreodecyltriethoxysilane (PFDTE) coupling agent was purchased from Shangfluoro Technology Co., Ltd. Ammonium hydroxide (25 wt%), Tetraethoxysilane (TEOS), xylene and ethyl alcohol were obtained from Sinopharm Chemical Reagent Co., Ltd. Aluminum alloy panels (series 6061) were provided by Alfa Aesar Inc. Before being used, the panels were rinsed with deionized water and acetone to remove grease and other contaminants, then followed by phosphate anodization. All chemical reagents used were of analytical grade and without further purification.

2.2. Preparation of fluorinated SiO₂ nanoparticles

The fluorinated SiO₂ nanoparticles (SiO₂-F) were prepared based on the modified stöber method [32]. Firstly, 160 ml ethyl alcohol, 10 ml ammonium hydroxide (25 wt%) and 20 ml TEOS were sequentially added to a round-bottom flask. The mixture was stirred 12 h at room temperature. Secondly, 2.0 g PFDTE was added to the system and the reaction was continued for another 4 h. Finally, the resultant mixture was centrifugated at 10000 rpm for 10 min to obtain the fluorinated SiO₂ nanoparticles. The completely washed SiO₂-F nanoparticles and xylene were ultrasonically dispersed by various weight ratios to prepare various SiO₂-F suspension with different SiO₂ nanoparticles contents. The obtained SiO₂ nanoparticles are spherical with size from 10 to 20 nm.

2.3. Fabrication of the composite coating

The synthetic route of the composite coating is schematically shown in figure 1. Firstly, HHTBN (60.00 g) and xylene (100.00 g) were put in a round-bottom flask and dewatered at 140 °C for 0.5 h. Secondly, when HHTBN was cooled down to 80 °C, HPSO (20.00 g), DBTDL (0.50 g) and IPDI (17.33 g) were added successively into the round-bottom flask. After being stirred for 6 h at 80 °C, isocyanate-terminated PUE’s prepolymer was
synthesized. Thirdly, BDO (4.23 g) was added to the isocyanate-terminated PUE’s prepolymer to obtain the PUE resin solution. Then, 10 ml SiO₂–F suspensions with various concentrations were added to 10 ml of PUE resin solution, followed by the ultrasonic dispersion for 10 min. The obtained PUE/SiO₂–F composite suspension was air-sprayed onto the aluminum alloy substrates surface using a spray-gun, with 0.5 MPa pressure and 10 cm distance. Finally, the resultant coating was kept for 2 h at room temperature to allow the solvent (xylene) to evaporate. The hydrolysis and coupling of the siloxane occurred subsequently. The anti-icing coating was obtained after 8 h crosslinking at 80 °C [33–36]. The thickness of the anti-icing coatings was 20 ± 5 µm.

2.4. Characterization
Fourier-transform infrared spectrometer (Nicolet) was used to study the surface composition of the sample in the range of 500 cm⁻¹ to 4000 cm⁻¹ at 25 °C. SEM images and 3D topography images were obtained with an Nova NanoSEM 450 field-emission scanning electron microscope, a Sneox Sensofar 3D topography instrument and a Bruker MultiMode 8 atomic force microscopy respectively to analyze the coating surface morphology. Tensile properties of the as-prepared coating were carried out on universal testing machine (CMT4104) at the deformation rate of 500 mm min⁻¹ according to ASTM D412. To improve accuracy and reliability, the test was repeated 5 times for each sample and the average result was used for analysis. The JC2000D contact angle meter was used to study the static WCA and SA by the sessile drop method with a deionized water droplet of 4 µl at room temperature. The SA was the tilt angle of the coating sample surface at which the water droplet began to roll off. The average values of WCAs or SAs from four parallel samples were used to analyzed the surface wettability of samples. All WCA and SA experiments were performed at 23 °C ± 2 °C with the relative humidity of 52% ± 6%.

The tape-peeling test and the abrasion test were performed to study the mechanical durability of the composite coating based on ASTM D3330 and ASTM D4886 respectively. As for the abrasion test, the sandpaper with 500 mesh SiC loaded with the weight of 200 g was placed face-down on the sample surface and reciprocating horizontally within 20 cm at the speed of 5 mm s⁻¹. The sample’s WCA and ice adhesion strength were recorded every 100 reciprocation cycles.

The 3M adhesive tape was used to be applied to the surface of the composite coating under the pressure of 30 kPa and peeled off during the tape-peeling test. The tape-peeling test was performed for 200 cycles.

The ice adhesion strength on the bare aluminum panel and the composite coating was carried out based on tensile shear motion, which has been used to study the anti-icing property in the previously published reports [37, 38]. The testing sample was closely applied to the cooling stage with the fixing clamp and 3M thermally conductive tape. The thermocouple was fixed beneath the testing sample to detect the accurate temperature. The cooling stage is capable of holding 9 testing zones, where the cuvettes with internal dimensions of 10 mm × 10 mm × 25 mm was placed vertically. When the test began, the chamber was sealed and the cooling stage worked. Once the sample temperature dropped to −15 °C, the cuvette was injected with 1 ml of ice-water mixture and then maintained for 5 h to ensure that the water inside completely frozen. The XY motion stage horizontally pulled the cuvettes via the connection rod at the speed of 0.5 mm s⁻¹. The force transducer was used to record the maximum force to detach the ice block from the sample surface. The ice adhesion strength (kPa) was defined as the maximum force divided by the cuvette’s internal cross-section area (1 cm²). To improve accuracy and reliability, the test was repeated at 9 random zones for each sample, and the average result was used for analysis. All ice adhesion strength test was carried out at the relative humidity of 50% ± 5%.
3. Results and discussion

3.1. Surface characterization

Figure 2 shows the FT-IR spectrum for the PUE. The absorption peak at 2235 cm$^{-1}$ belongs to stretching vibration of C≡N groups of HHTBN segments. The double peak at 1020 cm$^{-1}$ and 1080 cm$^{-1}$ and the peak at 795 cm$^{-1}$ can be associated with the stretching vibration of Si–O groups and C–Si groups of HPSO segments. The multiple peaks at 1544 cm$^{-1}$, 1685 cm$^{-1}$ and 3365 cm$^{-1}$ can be attributed to N-H in-plane scissoring, C=O stretching vibration and N-H stretching vibration respectively, which demonstrates the appearance of the urethane linkage (–NHCOO–). It is consistent with the vanishing of hydroxyl stretching vibration peak from 3400 cm$^{-1}$ to 3600 cm$^{-1}$ [37]. Therefore, it can be confirmed that HHTBN and HPSO have been successfully introduced into the molecular structure of the PUE.

3.2. Surface hydrophobicity

Low surface energy and high surface roughness are the two critical requirements for superhydrophobic surfaces according to the Wenzel wetting mode [38]. Surface wettability of Si-PUE with various SiO$_2$–F nanoparticles contents was studied through WCA and SA measurements in figure 3. The WCA of the bare aluminum panel is 75°. After the panel is coated with the PUE, the surface exhibited good hydrophobicity with WCA of 108°. As shown in figure 4(a), the PUE coating is very smooth. The results of EDS showed that PUE mainly consisted of C, Si, O and N (figure 4(b)). The content of silicon was relatively high, indicating that the HPSO chains with low
surface energy tends to accumulate on the surface of the coating. Furthermore, the hydrophobicity of Si-PUE was improved greatly with the growth of the SiO$_2$–F nanoparticles content. When the SiO$_2$–F nanoparticles content goes up from 0 to 8 wt%, Si-PUE displays a substantial WCA enhancement from 108° to 162°, demonstrating its excellent superhydrophobic property. SA, as another indicator for hydrophobicity, exhibits the similar trend, which steadily descends to 2°. With SiO$_2$–F nanoparticles content growing from 8 wt% to 10 wt%, the WCA and SA of Si-PUE coatings become stable, which could be ascribed to excess of silica nanoparticles on the PUE. Therefore, the Si-PUE coating with 8 wt% content was used for the following experiments as the resultant superhydrophobic coating. The superhydrophobicity of Si-PUE derives from the collaboration between low surface energy composition and rough micro-nanoscale structure, which is ulteriorly confirmed by surface morphology of Si-PUE (figure 4(e)). The surface roughness (Rq) of the obtained coating measured by AFM is 9.956 nm. As shown in figure 4(c), the Si-PUE coating is completely covered with clusters of silica nanoparticles, piling up to form the rugged microstructure. Because hydrophobic and rough surface is capable of capturing air cushions, the air cavities remarkably reduce the contact area between the coating surface and the water droplet [39].

3.3. Anti-icing properties
The material’s anti-icing ability was evaluated via the ice shear adhesion test, which simulates the ice block movement in practical application. The surface with low ice adhesion presents better anti-icing performance. Figure 5 gives the ice adhesion strength of the bare aluminum, the PUE coating and the Si-PUE coating prepared with 8 wt% silica nanoparticles suspension. The ice adhesion strength for the bare aluminum panel is 235.36 kPa. With the PUE sprayed onto the aluminum panel subsequently, the ice adhesion strength attenuates to 68.04 kPa. In comparison, the Si-PUE coating exhibits the ultra-weak ice adhesion strength of 14.33 kPa, more than an order of magnitude lower than that of the bare aluminum, on which the accumulative ice is prone to fall off under its own weight or natural wind [40–42]. Such result is consistent with WCA and SA, which can be attributed to the coating’s superhydrophobicity. Typically, water sits on the top of the micro/nanostructures of the superhydrophobic surface in the Cassie state so that large amounts of air is captured in the cavities between the ice and the superhydrophobic, which leads the reduction of attachment sites [43, 44].

Figure 4. (a) The SEM image and (b) EDS results of the PUE surface. The SEM images of Si-PUE surface with (c) 8 wt% silica nanoparticles suspension; (d) 10 wt% silica nanoparticles suspension and (e) The AFM image of Si-PUE surface with 8 wt% silica nanoparticles suspension.
3.4. Mechanical durability

Elastomers, such as rubber and polyurethane elastomers, are regarded as the best damping materials when large deformation is required for superior mechanical properties and abrasion resistance \[45-47\]. Tensile strength and elongation are the two critical properties for elastomers which are able to stretch a long distance and still bounce back. Figure 6 displays the tensile stress-strain curve of the Si-PUE coating. The Si-PUE coating is highly stretchable, which could sustain large-scale extension, and exhibits tensile strength and elongation up to 16.22 MPa and 385%, respectively. The externally mechanical energy can be dissipated in the thermal energy state by the mutual friction between the Si-PUE molecules when the elastic coating presents a cyclical movement of stretching and retraction. Finally, it can be expected to achieve the purpose of greatly reducing mechanical vibrations and abrasions \[48, 49\].

The tape-peeling test was firstly performed to study the mechanical durability of the Si-PUE coating. The changes in the WCA and morphology of Si-PUE coating after peeling test were shown in figure 7. The Si-PUE coating WCA after 200 times tape-peeling reduced slightly from 162° to 154° but the surface was still superhydrophobic. Besides, it can be observed that there is no obvious morphological degradation for the Si-PUE coating. It can be explained that the SiO₂ nanoparticles were strongly interlocked with PUE chains.

Moreover, the mechanical durability of the anti-icing properties for the Si-PUE coating was tested by the sandpaper abrasion test. The sandpaper with 500 mesh SiC loaded with the weight of 200 g was placed face-down on the Si-PUE coating surface and reciprocating horizontally within 20 cm at the speed of 5 mm s⁻¹. After
abrasion for 1000 cycles, the Si-PUE coating surface displayed durable superhydrophobicity with the WCA reduction from 162° to 151.9° (figure 8(a)). In addition, there was a slight increase from 14.33 kPa to 40.71 kPa in the ice adhesion strength (figure 8(b)). The value was still much less than that on the bare Aluminum alloy surface (235.35 kPa), which demonstrates that the mechanical durability of anti-icing coating can be improved by the flexible polymer matrix (PUE) [50–53]. The incorporation of flexible polymer in the adhesive matrix has a positive effect on the strong adhesion to the substrate and the energy dissipation by heat generation against mechanical abrasion, scratching friction and kneading [21, 54]. The insets in figure 8 show the 3D topography images of the Si-PUE coating before and after the sandpaper abrasion test. It can be found that the micro/nanostructure based on the SiO2 nanoparticles agglomeration remained similar to that of the original coating surface, which played essential functions in keeping durable superhydrophobicity.

4. Conclusion

In this work, a mechanically durable anti-icing coating was successfully developed based on the combination of siloxane modified polyurethane elastomer and fluorinated SiO2 nanoparticles. The coating was fabricated
through a facile air-spraying process that can be used to prepare large-scale surface. The surface morphology and hydrophobicity of the composite coatings was based on the SiO2 nanoparticles agglomeration, and the composite coating exhibited the optimal WCA of 162°. The composite coating with siloxane modified polyurethane elastomer matrix is highly stretchable, which could sustain large-scale extension, and exhibits tensile strength and elongation up to 16.22 MPa and 385%, respectively. Due to the combination of flexible polymer matrix and SiO2 nanoparticles, the composite coating displayed excellent robust superhydrophobicity against tape peeling and abrasion treatments. Moreover, the superhydrophobic coating also provided an ultralow ice adhesion strength of 14.33 kPa, showing an extraordinary anti-icing property. After abrasion for 1000 cycles, a slight increase in the ice adhesion strength of Si-PUE coating from 14.33 kPa to 40.71 kPa was observed, but much less than that on the bare aluminum alloy surface (235.35 kPa). This work makes a prospective anti-icing coating for a large-scale application in aerospace and automotive industry.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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