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Anti-frosting/anti-icing property of nano-ZnO superhydrophobic surface on Al alloy prepared by radio frequency magnetron sputtering

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Abstract

Superhydrophobic (SHP) surfaces had promising application in anti-icing field. In this work, ZnO SHP surfaces were prepared on aluminum alloy by radio frequency (RF) magnetron sputtering. The structure, chemical composition, morphology, wettability of the SHP surfaces were investigated by using corresponding methods. The anti-frosting/anti-icing performance of the ZnO SHP surfaces were investigated. Morphology of surfaces showed that sputtering time affected the surface morphology of the ZnO surfaces. When the sputtering time was 15 min, the rough structure exhibited excellent anti-frosting/anti-icing property due to the exceptional superhydrophobicity and self-transfer behavior at subzero temperature, which composed by densely gathered nanoclusters. The freezing of water droplets was delayed for about 2 h, and frosting was delayed for approximately 5 h at the temperature of −10 °C. The SHP surfaces could efficiently enhance the anti-icing property in glaze ice. Meanwhile, the as-prepared ZnO SHP surfaces displayed a low ice-adhesion strength of 12 ± 4.7 kPa. However, the structure with large undulation and pore space reduced the anti-frosting/anti-icing property of the SHP surface. This work provided a direction for the design and preparation of anti-icing surfaces for power transmission lines.

1. Introduction

The accumulation of ice on cold surfaces could cause many accidents in aircrafts [1], wind turbines [2], high-voltage power transmission lines [3, 4], and so on. In the past couple of decades, there were many accidents in power systems caused by icing, such as collapse of towers, galloping of conductors, and consequent power outages. However, most traditional methods, like thermal, mechanical or electrical techniques, had many disadvantages, such as low efficiency, waste of energy, or even equipment damage [5]. Therefore, research on effective anti-icing method had a great significance. Inspired by the lotus effect, many superhydrophobic (SHP) surfaces have been fabricated by researchers for their potential applications, such as self-cleaning [6], anti-corrosion [7], and anti-frosting [8, 9], and anti-icing [10, 11] technologies.

SHP materials were considered as a promising method in anti-icing because cooled water could roll away from SHP surface. Cao et al [12] claimed that superhydrophobic coatings could prevent ice formation upon impact of super-cooled water. Kulinch et al [13, 14] demonstrated that the SHP surfaces could reduce ice adhesion strength. Liao et al [15] fabricated a SHP coating through the nanoparticle filling method to enhance the anti-icing property of glass insulators. Fochi Wang et al [4] prepared an SHP anti-icing surface for Al alloy transmission lines by simple chemical etching. However, SHP surfaces were not always anti-icing. Scholars have pointed out that the superhydrophobicity would lost when water vapor condensed on many natural and artificial superhydrophobic surfaces at subzero temperature [16]. Frost nucleation might occur on micro-nanostructure of the SHP surfaces [17, 18]. In harsher climate conditions, frosting would form before icing and
accelerate the forming of icing. Therefore, the anti-frosting property was essential for the anti-icing application of SHP surfaces. Guo et al. [19] deposited ZnO nanohairs on a series of ratchet-like micro-structure morphologies through the hydrothermal synthesis method and found that ZnO nanohairs demonstrated excellent anti-frosting performance. He et al. [20] reported that the surfaces with ZnO nanorod arrays were also super-hydrophobic to condensed micro-droplets at temperatures below 0 °C and could effectively retard and prevent frost formation. Zuo et al. [21] showed that ZnO SHP nanostructures with the spontaneous jumping ability of condensed microdrops could effectively retard frosting for over 140 min even at −10 °C.

Research indicated that the microstructures of SHP surfaces were a crucial factor for the property of anti-icing/frosting. It was found that the SHP surface with nanostructure or hierarchical structure exhibited remarkable anti-icing/frosting performance while SHP surfaces with impaled condensates exhibited a poor anti-icing/frosting effect [22]. Mengxi [23] found that the micro-nanostructure had an excellent anti-icing ability compared with nanostructure and microstructure. More obvious spontaneously condensate drop motions were observed on surfaces with a nanostructures possessing sufficiently narrow spacing and higher perpendicularity [24, 25]. The dense nano-texture of the superhydrophobic surface was critical for the repellency of supercooled water droplets, and the presence of micron-scale features was its downfall [26].

The anti-frosting/icing property of ZnO SHP surfaces prepared via RF magnetron sputtering on glass has been researched [27, 28]. However, it was worth to investigate the effect of sputtering time on microstructure of ZnO surfaces and the relationship between the microstructure of surface and anti-icing/frosting properties, which has not been explored. Moreover, the ice adhesion strength was essential for deicing and should be discussed. In this work, RF magnetron sputtering was used to prepare the ZnO nano-structure SHP surfaces on Al alloy. ZnO SHP surfaces with different rough microstructure were obtained by different sputtering time. The anti-frosting/anti-icing property of the as-prepared surfaces were investigated at ≤ −10 °C. Furthermore, the effect of the microstructure on the anti-frosting/anti-icing property of the SHP surfaces were analyzed.

2. Experiments

2.1. Materials
A 30 mm × 30 mm × 5 mm Al alloy (6201) plate was used as a substrate. Zn target was provided by HeZong XinCai Company. 1H,1H,2H,2H-heptadecafluorodecyltrimethysilane (FAS-17) was afforded by Shanghai Aladdin company.

2.2. Preparation of ZnO SHP surface
The Al alloy plate was first sanded using emery paper, washed ultrasonically with deionized water and then by ethanol, and finally dried. The surfaces were prepared through the RF magnetron sputtering of a Zn target. Sputtering was conducted in an Ar plasma with 99.99% purity under a working pressure of 1.5 Pa. The sputtering power was set to 100 W, and the target–substrate distance was a fixed 10 cm. The sputtering time was 5–30 min (denoted as ZnO-X, where X was the sputtering time). Then, the samples were annealed in a muffle furnace at 400 °C for 30 min. The annealed samples were immersed in an ethanol solution of 2 wt% FAS-17 solution, for 30 min and subsequently dried at 90 °C for 1 h.

2.3. Characterization
The crystal structure and chemical composition of the samples were characterized by x-ray diffraction (XRD, Panalytical Empyrea, Netherlands) and Fourier transform infrared spectrophotometry (FTIR, Nicolet i55, USA). The surface microtopography was examined using a scanning electron microscope (SEM, JSM-7800F, Japan) and (SEM, Zeiss Auriga, Germany). The topography of ZnO-15 and ZnO-30 were investigated by a laser scanning confocal microscope (LSCM, LEXT OLS4000, Olympus, Japan). The contact angle (CA) and sliding angle (SA) were measured using a CA meter (Theta, Attension, Sweden). The water volume was 7 µl.

2.4. Anti-icing/anti-frosting test
The anti-icing behavior and static freezing of water drops on the samples were investigated on a Peltier-based cooling platform as shown in figure 1(a). The glaze ice was simulated and the dynamic anti-icing behavior was investigated by the artificial climate laboratory as shown in figure 1(b). Water at 3 °C was sprayed onto the sample surface about 10 L h⁻¹. The diameter of sprayed water was about 100 µm. The inner ambient temperature of chamber was −15 ± 1 °C. The inner ambient humidity was 50 ± 5%. The tilting angle of the sample was about 60°.
2.5. Ice adhesion strength test
The ice adhesion strength of as-prepared ZnO SHP surfaces and bare aluminum were investigated by the self-made experimental device. In detail, the device consisted of a Peltier-based cooling stage, a force transducer, a motion stage and a plastic cuvette. The samples were attached to the Peltier-based cooling stage. The cuvette was placed on the testing samples and filled with 2 ml of water. The cooling stage was maintained at $-20^\circ$C for 3 h. Subsequently, the maximum force required to detach ice cuvette from its substrate was recorded by moving the force transducer perpendicularly to the ice cuvette at a speed of 1 mm s$^{-1}$. The ice adhesion strength of each sample was calculated by dividing the measured maximum force by the cross-section area of the ice-sample interface ($1.13 \text{ cm}^2$).

3. Results and discussion

3.1. Structure and chemical composition
Figure 2(a) showed the XRD patterns of the surface prepared by magnetron sputtering of Zn target after annealing at 400 $^\circ$C. The samples presented the same characteristic peaks. The peaks centered at $2\theta = 31.70^\circ$, $2\theta = 34.36^\circ$, $2\theta = 36.24^\circ$, $2\theta = 47.6^\circ$, $2\theta = 56.58^\circ$, $2\theta = 62.86^\circ$, $2\theta = 66.42^\circ$ and $2\theta = 67.98^\circ$, corresponding to the (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (2 0 0), (1 1 2) and (2 0 1) planes, respectively of hexagonal ZnO (JCPDS Card No.03-0888). The peaks centered at $2\theta = 38.47^\circ$, $2\theta = 44.72^\circ$, $2\theta = 65.10^\circ$, $2\theta = 78.23^\circ$ and $2\theta = 82.44^\circ$ correspond to the (111), (200), (220), (311) and (222) planes, respectively of Al (JCPDS Card No.04-Z0787). XRD results might be suggesting a total oxidation of Zn. Chemical composition was considered to influence the wettability and super-hydrophobic surface commonly prepared by modifying micro-/nano-structured with materials of low surface free energy. Here, to ensure the FAS-17 molecules had successfully combined on ZnO, FTIR was used to analyze the chemical composition of the samples after modification with FAS-17. Figure 2(b) showed that the FTIR spectrum bands of 1240, 1205, 1148, and 1114 cm$^{-1}$ were attributed to the C–F stretching vibration of the $-\text{CF}_2$– and $-\text{CF}_3$ groups. The peak at 1066 cm$^{-1}$ belonged to the framework vibration of Si–O–Si. The FTIR results indicated that FAS-17 was successfully combined on the ZnO surfaces.
3.2. Morphological characterization and wettability

Figure 3 showed the SEM images of the as-prepared ZnO SHP surfaces on the top and side views with different sputtering time. Figures 3(a1)–(a3) showed the morphology of the ZnO-5. The ZnO-5 surface was composed of many nanoparticles. Lots of pore spaces existed between these nanoparticles. There were some micron-sized hole defects as show in figure 3(a1). With the increasing of sputtering time, the size of the nanoparticles grew. The prepared surfaces became more uniform gradually. As shown in figures 3(c1)–(c3), the ZnO-15 surface presented the most uniform micromorphology at all. In addition, the nanoclusters densely gathered and formed much micro-scale mastoid. As shown in figures 3(d1)–(d3), the micromorphology of ZnO-20 had suddenly changed. A certain rougher microstructure appeared. More and larger pores were exhibited in ZnO-20 and ZnO-30. The thickness of the surfaces ranged from 1.2 μm to 12.5 μm. It demonstrated a significant change in thickness of ZnO SHP surfaces with different sputtering time.

Figure 4 showed the CA and SA of the as-prepared SHP surfaces. The as-prepared SHP surfaces exhibited outstanding superhydrophobicity with CA above 160° and SA less than 4°, especially the ZnO-15 and ZnO-30. Water droplets could easily roll off the SHP surface. According to the classic Cassie and Baxter’s equation [29], \[ \cos \theta = f_1 \cos \theta_1 + f_2 \cos \theta_2 - f_1 f_2 \] where \( f_1 \) and \( f_2 \) were the fractions of solid surface and air in composite surface, respectively, and \( f_1 + f_2 = 1 \). \( \theta_1 \) and \( \theta_2 \) were the CA of the rough and flat ZnO surface, respectively. The CA of flat ZnO modified with 2 wt% FAS-17 was 109.72° [21]. According to the above equation, the \( f_1 \) of ZnO-5, ZnO-10, ZnO-15, ZnO-20 and ZnO-30 value was approximately 7.31%, 5.63%, 4.7%, 6.98% and 3.75%, respectively. These results demonstrated that the pore spaces of rough SHP surfaces were helpful to trap a large amount of air and reduce the solid-liquid interface contributing to increase the contact angle of as-prepared SHP surfaces.
3.3. Anti-frosting and anti-icing property of ZnO SHP surface

Anti-frosting behaviors of ZnO SHP surfaces and bare aluminum were investigated by the Peltier-based platform. The temperature of the sample was maintained at −10 °C. The ambient temperature was 15 ± 2 °C, and the corresponding humidity was 50 ± 5%. As shown in figure 5, the bare Al alloy surface was covered completely by frost after 21 min. However, many small, sphere-like water droplets condensed on ZnO SHP surfaces after 30 min, indicating that the as-prepared SHP surfaces were also superhydrophobic to condensed water droplets when the surface temperature were −10 °C. It resulted in the froze process of condensed water droplets much longer than bare aluminum. ZnO-15 showed the outstanding performance in the retardation of frost formation. Large area of the as-prepared SHP ZnO-15 surface remained frost-free within 120 min. Most area of other ZnO surface were covered by frost after 120 min. It took 316 min for all condensed water drops on ZnO-15 to freeze, which was much longer than the time consumed by the drops on other ZnO SHP surfaces, to freeze into frost. The arrow in figure 5 revealed that condensed water droplets on ZnO-15 were easily combined with neighboring water droplets and then rolled away. Thus, condensed water droplets on the movement path were adsorbed and formed large water droplets (white dashed circle) and a dry and clean area (red dashed bordered). It shown that ZnO-15 SHP surface had better superhydrophobicity than other SHP surfaces at low temperature for a long time. Interestingly, although both of ZnO-15 and ZnO-30 exhibited excellent superhydrophobicity, they displayed different anti-frosting property. The outstanding anti-frosting property of the ZnO-15 surface might be attributed to the existence of dense and uniform nano-structure.

In the previous report [30], condensed water microdroplets on the pillar coatings with the smallest pillar dimensions were coalescing on top of the pillars, while condensed micro-droplets on the substrate with the largest pillar dimensions were coalescing at the base of the pillar structure. The nanostructures with sufficiently narrow spacing caused less pinning of the condensate drops and contributed to the enhanced drop mobility [26, 31]. In order to further investigate the microstructure of ZnO-15 and ZnO-30, the morphological was also investigated by the 3D confocal microscopy. As shown in figure 6, ZnO-15 had a dense and uniform ZnO nano-structure. Many mastoids and pits were distributed on ZnO-30, which had a large undulation and pore space. The average roughness (Sa) of ZnO-15 and ZnO-30 were 370 ± 99.6 nm and 441 ± 84 nm, respectively. The schematic illustrations of condensation process on the ZnO-15 and ZnO-30 at low temperatures were shown in figure 7. The surface of ZnO-15 was composed with dense ZnO nanoclusters. The densely gathered nanoclusters might prevent the condensed micro-droplets from entering the valleys of the microstructure and maintain them at the Cassie–Baxter state. Slow heat transfer was caused by the small contact area between the condensed water droplets and the SHP surfaces. In addition, the self-movement of condensed water droplets was also contribute to delay the formation of frost. The self-transfer phenomenon could sweep the as-prepared surface clean and dry, and thus effectively inhibited the spreading of frost by delaying the ice-bridging process [30, 32]. However, frost nucleation occurred gradually on the valleys of the microstructure of ZnO-30 due to the presence of large pore space. A study has reported that if frost formed within the textures of a SHP surface, the air–solid interface changed into a liquid–solid interface, and this phenomenon was identical to that observed on lotus leaves [33]. Thus it led to a Cassie-to-Wenzel transition of the condensed water droplets, which increased the contact area between the water droplets and the surface and contributed to the accelerated freezing of the condensed water droplets. Consequently, the freeze time of the condensed water droplets on ZnO-15 was considerably longer than that consumed by the condensed water droplets on ZnO-30.
The static anti-icing behaviors of water droplets on as-prepared ZnO SHP surfaces and bare aluminum were researched by the Peltier-based platform. Five water droplets of 7 μl were dripped on the surface. The sample temperature was maintained at −10 °C. The ambient temperature was 15 ± 2 °C, and the corresponding humidity was 50 ± 5%. Figure 8 showed the freezing process of the water droplets on the bare Al alloy and ZnO SHP surfaces. All of the water droplets on the bare aluminum alloy surface froze within 3 min. The water droplets on ZnO-15 totally frozen at 120 min, which was much longer than that of other SHP surfaces. This result was in good accordance with the results of the frosting test.
According to Fourier’s law, the heat transfer (Q) between surface and water droplets can be written as

\[ Q = \int_{T_1}^{T_2} K S dT, \]

where \( T_1 \) and \( T_2 \) were the temperatures of water droplets and surface, \( K \) was the heat transfer coefficient, \( S \) was the solid-liquid contact area [20]. Thereby, \( Q \) was proportional to \( S \). The existence of trapped air between the droplets and the rough SHP structures reduced the solid-liquid interface. Less heat will be transferred from water droplets to the SHP surfaces, which led to the frozen process much longer than bare aluminum [34]. Smaller roughness scales were favoured for retaining the Cassie state of a superhydrophobic surfaces during condensation. However, larger roughness scales increased the Cassie to Wenzel state transition of the water droplets and the solid-liquid contact area [35, 36]. At the same time, frost played a critical role in the freezing of droplets. Frost front acted as the seed to induce freezing of water droplets (micro- and macro-) upon intimate contact [36]. Frosting results showed that Zno-15 had better anti-frosting performance than other Zno SHP surface. It was the reason that the frozen process of water droplets on ZnO-15 much longer than other SHP surface.

The glaze ice was the most dangerous type of ice associated with highest probability of high-voltage power transmission lines [3]. In this work, glaze ice was simulated and the dynamic anti-icing behavior was investigated by the artificial climate laboratory. Figure 9 showed that the spraying water was attached to bare Al alloy whose surface was covered with an ice layer within 3 min. However, the spraying water was easily rolled off the SHP surfaces, so the SHP surfaces almost remained dry and clean in 3 min spraying. After 28 min, ZnO-5 and ZnO-
30 were covered with thick ice. Long icicles were formed on the surfaces of ZnO-10 and ZnO-20 after 28 min. A relatively large area of ZnO-10, ZnO-15 and ZnO-20 surface remained ice-free within 28 min. After spraying for 35 min, the amount of icicles on ZnO-15 were less than those on other ZnO SHP surface.

At the beginning of the dynamic icing test, the sprayed cooling water droplets could easily roll away from the SHP surfaces before freezing. Frost nucleation occurred on the SHP surface with the increase of spraying time.
This frost point would act as crystallization nucleus accelerating the sprayed cooling water freezing. The ice point on SHP surfaces became the defect point, which led more water droplets to adhere and subsequently freeze. The ZnO-15 with excellent anti-frosting performance could effectively prevent the defect point, thus help to improve the anti-icing property in glaze ice.

The ice adhesion strength of ZnO SHP surface and bare aluminum were shown in figure 10. The ice adhesion strength of bare Al was as high as 485.1 ± 82.3 kPa. Obviously, the ice adhesion strengths on the ZnO SHP surfaces were much lower than that on the bare Al. The ice adhesion strength of as-prepared ZnO SHP surfaces were all below 20 kPa. Furthermore, the ice adhesion strength of ZnO-15 was as low as 12 ± 4.7 kPa, which was reduced by 97.5% in contrast with the bare Al. Ice adhesion strength below 20 kPa was seen as the benchmark for surfaces that allows passive ice removal by factors such as wind or vibration. In summary, the as-prepared ZnO-15 SHP surface exhibited outstanding anti-icing and anti-frosting property.

4. Conclusion

ZnO SHP surfaces were prepared on an Al alloy by RF magnetron sputtering. It demonstrated that the sputtering time affected the surface morphology of the ZnO surface. Even though all of the as-prepared ZnO SHP surfaces demonstrated excellent superhydrophobicity as defined by the contact angle and roll-off angle, they displayed different anti-frosting and anti-icing property. It was found that ZnO-15 with the densely gathered nanoclusters rough structure showed outstanding anti-frosting/anti-icing property. A structure with large undulation and pore space reduced the anti-frosting/anti-icing property of the SHP surface. Frosting and freezing processes of water drops on ZnO-15 SHP surfaces have been effectively delayed for about 5.5 h and 2 h at -10 °C, respectively. Meanwhile, the as-prepared ZnO-15 SHP surface could efficiently enhance the anti-icing property in glaze ice. The adhesion strength of the as-prepared ZnO-15 SHP surface was greatly reduced by 97.5% in contrast with the bare Al. This work provided guidance for the design and fabrication of anti-icing surfaces for transmission lines.

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