Robust Electrical Uni-directional De-icing Surface with Liquid Metal (Ga₉₀In₁₀) and ZnO Nano-Petal Composite Coatings

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Abstract: A novel superhydrophobic electro-conductive coating with liquid metal (Ga₉₀In₁₀) and ZnO nano-petals is fabricated by integrating the methods of spraying, crystal growth and low surface energy modification. The results indicate that the paper-based surface could be superhydrophobic in cold environment and successfully realize uni-directional ice driving with the help of thermal gradient. We anticipate that the combination of excellent superhydrophobic performance of the composite structure with inherent conductive advantage of liquid metal will find a vast application prospect.

Keywords: de-icing, liquid metal, superhydrophobic, electrothermal effect.

1. Introduction

In recent years, extraordinary superhydrophobic material with multilevel topography has received a keen attention for its commercial potentials related to anti-fogging [1, 2] and anti-icing coatings [3-5]. However, in cold environment, water freeze and ice accumulation at the interface often cause many potential damages to the micro-/nanostructure of the superhydrophobic surfaces and shorten the service life of devices [6-8]. Especially on the rigid surfaces, continuous impact of supercooled droplets or icing/de-icing cycle may cause equipment performances failure [9, 10]. Great efforts have been made to develop anti-icing and

de-icing materials based on superhydrophobic performance of micro-/nanoscale composite structure [11-15]. As for the superhydrophobic surface, the pocketed air between the interfaces of liquid and substrates results in a larger contact angle and lower hysteresis than that of smooth surface. The droplets on the superhydrophobic surface keeps in Cassie state. It is considered that a feasible way to prevent the surface from wetting for retarding freeze. However, there are still a great deal of technical problems that need to be addressed before wide application in fields of icephobicity, superhydrophobicity, wettability, etc. Firstly, there is no one-fit-all scheme which could successfully solving poor adhesion strength of nano-structure to various kinds of substrates such as rigid silicon wafer, soft PVC, cloth, paper, etc. [16-19]. Therefore a new enhanced cushioning material is needed. Secondly, some complicated device systems such as space aircraft and spaceship need much more protection to satisfy the multi-functions demands. The accumulated electrons on the substrates can lead the coatings to shed off and threaten the system safety. So, the special electric conductive coating which can avoid the electrostatic hazards resulting from electrons accumulation is particularly important. Combining the above two points, designing a conductive and robust adhesive cushioning layer between the nanostructures and substrate materials (organic, inorganic) is critical in a wide range of applications from icephobic/de-icing performances to anti-static fields. Liquid metal, which has characteristics of flexibility, high conductivity and high viscosity at room temperature is suitable for modifying most of the inorganic or organic substrate materials except some kinds of metals [20-23].

In this paper, liquid metal ($\text{Ga}_{90}\text{In}_{10}$, mass ratio, LM) which acts as cushioning layer is introduced to modify some substrate materials such as paper, polyvinyl chloride (PVC), cloth and rigid silicon wafer. These substrate materials are all non-corrosive to the liquid metal. Subsequently, ZnO nano-petals are planted on the liquid metal cushioning layer by enhanced crystal growth method. After being modified by (Heptadecafluoro-1, 1, 2, 2-tetradecyl) trimethoxysilane (FAS-17) which is a low surface energy material, the surface acts some

excellent properties such as superhydrophobicity, icephobicity, high conductivity, electrical uni-directional de-icing. This finding reveals a joint of inorganic and organic materials, which has wide potential application in interfacial field.

2. Experimental Section

2.1. Preparation of paper-based LM-ZnO composite coating

Liquid metal ($Ga_{90}In_{10}$) cushioning layer: Firstly, 90 g of Gallium and 10 g of Indium were mixed together and then stirred with a magnetic stirrer in a glass beaker at 150°C for 1 h to obtain a uniformly mixing metal liquid. Then, after cooling down to room temperature, the liquid metal was sprayed onto the different substrates respectively under pressure of 5 MPa for 2.5 s. The substrates could be paper, PVC plate, Si wafer, which all have robust resistance to the corrosion of liquid metal.

Preparation of ZnO nanostructure: The substrates with $Ga_{90}In_{10}$ cushioning layer were rinsed by deionized water for 5 min and flushed with nitrogen. The mother liquid of crystal growth was prepared as follows: 0.42 g hexamethylene tetramine and 0.95 g $Zn(NO_3)_2 \cdot 6H_2O$ (purchased from Beijing Lanyi Chemical Co., Ltd., China) were mixed together into 100 mL deionized water and gently stirred with magnetic stirrer until completely dissolved. Placed the prefabricated LM modified substrates and mother liquid into the Teflon reactor at 95°C for 12 hours. After the temperature of reactor cooled down to room temperature, removed the substrates out and washed them with deionized water and ethanol, respectively. Subsequently, put the substrates in the oven at 60°C to dry them for 1 hour. Finally, the superhydrophobic surface was obtained by the vapour deposition of FAS-17 (purchased from Shanghai Sinofluoro Chemicals Co., Ltd., China) for 5 h at 80°C under vacuum.

2.2. Measurement and characterization

Scanning Electron Microscopy: The morphology of the surfaces were obtained by Environmental Scanning Electron Microscopy (ESEM, Quanta FEG 250, FEI, USA) under a voltage of 5-15 kV.

Measurement of the wettability: Water contact angles (CAs), advancing contact angles (ACAs), receding contact angles (RCAs) and sliding-off angles on the paper-based composite surface were tested with optical contact angle meter system (Dataphysics SCA40, Germany). The temperature of the cooling stage could be controlled from -10 to 30°C, and the samples were fixed on the cooling stage using copper foil double-sided conductive tape. Droplets (5 μ L in volume, deionized water) were placed onto the film surface from syringe gently through mechanical vibration.

Energy Dispersive X-ray Spectroscopy: The chemical elements composition of the surfaces were obtained by Scanning Electron Microscopy (SEM) equipped with Energy Dispersive X-ray Spectroscopy (JSM-6500F, JEOL).

2.3. Icing and electrothermal de-icing test of the droplet on paper-based surface.

The paper-based composite surface was placed on the cooling sample stage of the contact angle system at the temperature of -10°C and the humidity of 80%. And two aluminium foils were employed as contact electrodes at both sides of the sample. After ten minutes, one droplet with the volume of 10 μ L placed onto the surfaces using the microsyringe. After the droplet froze, an electric circuit was set up on the both sides of the surface with 1 V and 1 A. And the test process was recorded simultaneously using a CCD camera of the contact angle system and a thermal infrared imaging camera (FLIR A665sc, America).

2.4. Impacting and bouncing test.

The paper-based composite surfaces bent with different curvature radii were placed on the cooling sample stage of the contact angle system at the temperature of -10°C and the humidity of 80%, respectively. After ten minutes, one droplet with the volume of 10 μ L impacted onto

the surface using the microsyringe from the height of 5 cm. And the test process was recorded by a high-speed camera (Phantom v9.1, Vision Research, America) at 2000 fps.

3. Results and discussion

The topography of the paper-based LM coating surface are shown in **Figure 1**. The surface is composed of clusters of nano-petals (**Figure 1a-b, Figure S1**) with thickness of 100 nm (**Figure 1c**). To reveal the bond state of $\text{Ga}_{90}\text{In}_{10}$ cushioning layer and nanostructure, we have observed the cross section of sample (**Figure d-f**). The sample consists of three layers, including paper (substrate), LM (middle, cushioning layer) and ZnO nano-petals (top). LM layer acts as a cushioning material to adhere inorganic ZnO nano-petals onto the organic/inorganic substrates. It is noteworthy that the cushioning layer plays a significant role not only in enhancing the affinity of different materials but also in electrical conductivity of materials. From the side view of the multi-layers, we find that the ZnO is characterized as height of 6 μm with a hexagon shape (inset of **Figure 1f**). The colour of paper changed from metallic colour to white colour after modifying ZnO nano-petals on it (**Figure 1g**).

Figure 1. Topographies of the paper-based surface modified with LM and ZnO nano-petals. **a)** SEM image of the paper-based composite structure surface which is composed of clusters of nano-petals **b-c)** Magnified images of nano-petals with thickness of 100 nm. **d)** The cross section image of the paper-based composite structure. **e)** Magnified image of (d). The sample consists of three layers, including paper (substrate), LM (middle, cushioning layer) and ZnO nano-petals (top). **f)** The side view of the sample. The nano-petal with height of 6 μm and hexagon shape (inset) is observed. The thickness of LM cover is about 6 μm . **g)** The macro-photo of paper (covered with liquid metal) before and after being modified by ZnO nano-petal.

To explore the universality and significance of this new scheme, three substrates including silicon wafer (rigid inorganic material), PVC and cloth (soft organic materials) are investigated. The SEM images and Energy Dispersive X-ray Spectroscopy (EDS) show that all three samples are coated with LM cushioning layer and ZnO nano-petal layer (as shown in **Figure 2**).

Figure 2. Topography and Energy Dispersive X-ray Spectroscopy (EDS) analysis of the surfaces coated on PVC (a), cloth (b) and Silicon wafer (c).

Liquid metal characterized as high electrical conductivity, good heat conductivity, flexibility, is mainly applied in thermal transfer field. According to ohm law, the resistance of an electric conductor is inversely proportional to its cross-sectional area. Thus, the conductive layer of LM with thickness of $\sim 6 \mu\text{m}$ shows large resistance. The surface generates a large amount of heat with high speed (**Figure 3a**, **Figure S2**) when the direct current passes along the direction of the sample from one end to the other, and forms a thermal gradient on the

surface of sample (The sample is placed on a cooling stage with the temperature of -10°C). The temperature of the surface closed to electrode rises rapidly from -10°C to 30°C in 2.1 s.

When an electric current flow in a certain direction steadily, the collision between electrons and materials atoms is responsible for the temperature raise. This is Joule effect, which can be described by the following **Equation (1)** [24]:

$$Q = I^2 R t = IVt \quad (1)$$

where Q indicates the heat, I is the current intensity, R is the resistance, V is the applied voltage and t is the time of current flows. When the applied voltage V is constant, the greater the electronic conductivity of the LM layer, the more heat will be generated. The temperature distribution change over time was observed by far-infrared thermograph imaging system (FLIR SC620, FLIR Systems Inc, USA) from top to bottom under the condition of constant current of 1 A and voltage of 1 V. The composite surface is covered with ZnO nano-petals, whose emissivity is 0.673 [25]. And the result indicates that the temperature gradient on the surface was generated (**Figure 3b**). As we known that, there are three forms of heat transfer including conduction, convection, and radiation. Due to the relative low temperature, the main form of heat transfer is conduction, and the heat radiation and convection can be ignored. Herein, we only consider the heat conduction in the process. According to the thermal resistance theory, when the current passes through LM layer from top to bottom, the total thermal resistance R can be described as $R = \frac{\delta}{\lambda A}$, where, δ represents the horizontal distance from the electrode (from top to bottom), λ indicates the thermal conductivity of LM and A represents the sectional area, respectively. The heat flux q through LM layer is available as in **Equation (2)** [26-27]:

$$q = \frac{T_1 - T_2}{R} A = \frac{T_1 - T_2}{\delta} \lambda \cdot A^2 = k \cdot \lambda \cdot A^2 \quad (2)$$

where T_1 and T_2 represent the temperature of two points along the horizontal direction respectively, k is the slope of the curve in **Figure 3b**. The thermal conductivity (λ) and the

sectional area (A) are constants. Obviously, the temperature gradient is determined by the horizontal distance δ between two points. Under the condition of different current from 0-2.5 A, the temperature distribution reached a steady state in 10 s (**Figure 3c**). The excellent electrothermal property of LM could lead to a rapid raise of the temperature under the driving of the electricity, which forms shear force and wettability gradient along the sample surface (**Figure S3-S4**).

Figure 3. The heat transfer process on the sample (with LM and ZnO coating) recorded by far-infrared thermograph imaging system. **a)** The analysis of heat transfer on paper with constant current (1 A) and voltage (1 V). The dotted white square highlights the connection of direct current. **b)** The temperature distribution from top to bottom versus time under constant current (1 A) and constant voltage (1 V). The curve valley is induced by touch voltage. **c)** The thermal distribution from top to bottom of the sample under different current (1-2.5 A). The constant voltage is 1 V and the relative time is 10 s.

The paper-based LM-ZnO composite coating surface shows not only a good electrical conductivity, but also an excellent superhydrophobic performance. After being modified by FAS-17, the surfaces (paper-based) were endowed with the performances such as superhydrophobicity and icephobicity, which can even realize directional ice driven under the electrothermal effect. This new type of ice driving is induced by thermal gradient which results in a surface tension gradient on the two sides of ice particle. In the test, we employed droplets with a series of volumes from 5 μl to 10 μl . After the direct current is switched on, all the ice particle began to melt with the increase of the surface temperatures (**Figure 4a, b**). Afterwards, a water film was formed owing to the heat gradient and the ice particle on the surface ran from the higher temperature region to the lower region with the help of the water film lubrication. The sketches of top and side observation of the droplet on the paper surface was shown as **Figure 4c**. Even if, in some cases, the ice particle was pinned on the surface, it would melt and evaporate in a short time once the current connection is established (**Figure S5**). The unbalanced Yong's force (dF) induced by temperature is given by **Equation (3)** [28-30]:

$$\begin{aligned} dF_Y &= [(\gamma_{SV} - \gamma_{SL})_1 - (\gamma_{SV} - \gamma_{SL})_2]dx \\ &= [(\gamma_{SV} - \gamma_{SL})_{T_1} - (\gamma_{SV} - \gamma_{SL})_{T_2}]/k \cdot dT \end{aligned} \quad (3)$$

where γ_{SV} and γ_{SL} indicate the surface free energies of the solid-vapour and solid-liquid interfaces, respectively, dx represents the distance of region 1 and 2, T_1 and T_2 are the temperature of region 1 and 2, and k is the ratio of temperature to distance, respectively. When the temperature of region 1 is higher than that of region 2, the unbalanced force caused by the increase of the thermal gradient becomes stronger than the shear force at the same temperature, the droplet/ice will move from the higher temperature region to the lower region. Thus uni-directional driving of the droplet/ice is realized. The results of the gradient contact angles (CAs) on different regions of the surface (as shown in **Figure 4d**) indicate that the droplets on the

lower temperature region show much more wettability. The larger the CAs gradient at both ends of the droplet is, the easier the droplet driving realizes [31].

Figure 4. Uni-directional ice particle driving under the heat gradient on surface. **a)** The thermal infrared sequential images of the uni-directional ice particle driving. The ice particle was driven from the higher temperature region to lower temperature region. **b)** The optical images in the dynamic driven process of the partially melted ice particle. **c)** The sketch of the partially melted ice particle driven process. **d)** The contact angles (CAs) on both sides of the droplet on different regions of paper (temperature gradient) at 10 s (1 V, 1 A).

Figure 5. The time-lapse photographs of water droplet bouncing on the surfaces with different curvature radii. a-c) The droplets with volume of 10 μL impacted onto the curved surfaces and detached in a short time. **d)** The dynamic motion of droplet on the flat surface.

For the ordinary superhydrophobic surface, some mechanical behaviours such as impact, bend and friction will damage its surface nanostructure, and undermine its excellent performance. In order to demonstrate the robust adhesion of the coating onto the substrate surface, we carried out the droplet impact experiments on the surfaces bent with different curvature radii. The results are shown in **Figure 5**. All the droplets with volume of 10 μL impacted onto the surfaces from height of 5 cm above and retracted into a pancake shape in 2.5 ms. Finally, the droplets rebounded and left completely from the surfaces in 7 ms without wetting the surface (**Figure 5a, b, c** and **Figure S6, Movie 1** and **2**). After more than 1 hour under droplet impact test, the surface still keep in superhydrophobic state. This indicates that

the surfaces all kept their excellent superhydrophobic property after being bent with different curvature radii (bended SEM test figure is shown in Figure S7), which means a robust adhesion between the coatings and the substrates. In addition, the flexible performance of paper substrate transferred the impact force of the droplets into an elastic energy and released it in the rebound process, which effectively reduced the contact time [32-35]. On the flat paper surface, the contact time was longer than 7.5 ms (**Figure 5d**).

4. Conclusion

In conclusion, we fabricated a novel superhydrophobic electro-conductive coating with liquid metal ($\text{Ga}_{90}\text{In}_{10}$) and ZnO nano-petals on various substrates such as paper, PVC plate, cloth and silicon wafer, by integrating the methods of spraying, crystal growth and low surface energy modification. It showed not only an excellent superhydrophobic performance, but also a good electrical conductivity in cold environment. And the uni-directional ice driving behavior was successfully realized with the help of thermal gradient. It is bound to promote the progress of the anti-/de-icing materials in many applicable processes such as aerospace devices for electrostatic eliminating and anti-icing in future.

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Graphical Abstract

The water droplet is uni-directionally driven off the surface with the help of the temperature gradient.