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Fabrication of robust conductive and superhydrophobic coating based on carbon nanotubes

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Abstract

This paper reported a simple approach to prepare robust conductive/superhydrophobic coating. The hierarchical structure was obtained through the addition of microscale filler (graphite powder and expanded graphite) and nanoscale filler (carbon nanotube). The self-similar structure was obtained through bonding the fillers using the epoxy matrix. Through the combination of the hierarchical and self-similar structures, the as-prepared superhydrophobic coating demonstrated excellent anti-abrasion property, good conductivity, excellent self-cleaning performance in both oil and water environment, outstanding anticorrosive property, and superior thermal stability simultaneously. Moreover, this superhydrophobic coating was achieved by a simple casting method, which has the potential to be used in large scale production.

1. Introduction

Superhydrophobic surfaces with high water contact angles (>150°) and low contact angle hysteresis (<10°) have attracted considerable attention due to their diverse applications in anti-corrosion [1–3], anti-fouling [4], drag-reduction [5], anti-icing [6–8], oil–water separation [9, 10], and so forth. It has been found that suitable surface roughness and low surface energy are two indispensable parts for constructing superhydrophobicity [11, 12]. Several methods (for instance, spraying-coating, cast coating, layer-by-layer deposition [13], template-based methods [14], lithography [15], sol-gel process [16]) have been adapted for fabricating such surfaces. Among these method, spray/casting method has the advantage of cost efficiency, simplicity, and suitable for industrial applications.

Recently, more and more researches tried to combine the superhydrophobicity with other functionalities such as transparency [17], electrical conductivity [18], and antimicrobial activity [19]. Particularly, superhydrophobic/conductive coating may have bright future in anti-corrosion for the grounding grid application [20]. Many superhydrophobic/conductive coatings have been fabricated by scattering carbon-based fillers in the polymer matrix. For instance, Hejazi et al prepared hair-like superhydrophobic CNT structure by a template method, which achieved superhydrophobicity without any modification [21]. Gu et al fabricated superhydrophobic surface by coating polystyrene onto the carbon nanotube membrane [22]. Wang et al constructed a superhydrophobic coating by mixing the graphene with the polydimethylsiloxane [23]. However, these superhydrophobic/conductive surfaces are prone to be injured by a slight scratch, or even finger contact [24].

Until now, there are three different approaches to enhance the mechanical robustness of the superhydrophobic materials. First, some researchers tried to bonding the hydrophobic particles using the
organic/inorganic adhesive [25], but there is an upper limit in the bonding strength between particles and adhesive. Second, hierarchical structure, which aimed to introduce a second scale nano-roughness on top of and between the first scale micro protrusions, tried to use the relatively strong microstructure to protect the fragile nanostructure [26]. Third, we can design a coating which has self-similar structure [27]. Although the exposed parts of the coating will be abraded, the new exposed part maintain superhydrophobicity because they are similar in texture and functionality with the abrade parts.

In this research, we tried to prepare a robust superhydrophobic conductive coating which combines hierarchical structure and self-similar structure. In order to obtain hierarchical structure, both microscale filler (graphite powder and expanded graphite) and nanoscale filler (multiwall carbon nanotube) were utilized. In order to obtain self-similar structure, the fillers were uniformly dispersed in the epoxy resin. Based on the aforementioned strategy, the as-prepared superhydrophobic surface demonstrated excellent conductivity, good mechanical robustness, and superior self-cleaning performance. Moreover, the superhydrophobic materials were achieved by simply casting the paint-like mixture, which has the potential for large-scale production. Therefore, this superhydrophobic coating may have a bright future for the application of protecting the grounding grid.

2. Experimental procedure

2.1. Materials

The E51 epoxy resin, Q235 steel plate, expanded graphite (EG, 400 mesh) was purchased from local market. The diethylenediamine (DETA) was bought from the Sigma-Aldrich, and used as the hardener. The multi-walled carbon nanotubes (NC7000), which has a mean diameter and length of 9.5 nm and 1.5 μm, were purchased from Nanocyl Co. Ltd, Belgium. 1H,1H,2H,2H-perfluorooctyltriethoxysilane (C8F13H4Si(OCH2CH3)3, FAS) were purchased from Aladdin Reagent Co., Ltd, Shanghai, China. The graphite powder (100 mesh) was purchased from Sinopharm Chemical Reagent Co., Ltd (SCRC, China). All other chemicals were bought from SCRC, and used as received.

2.2. Pretreatment of the substrate

Q235 steel plates with dimensions of 20 mm × 20 mm × 2 mm were utilized as the substrates. Before using, Q235 substrates were ultrasonically cleaned in ethanol and deionized water, respectively, then dried in air for 1 h.

2.3. Preparation of the superhydrophobic coating

The fabrication scheme can be found in figure 1. First, 2 g E51 epoxy resin was added into 3 g tetrahydrofuran (THF), and then stirred for 2 h to form uniform epoxy solution (solution A). Separately, 0.5 g graphite powder, 0.16 g MWCNTs, 0.04 g EG, and 0.1 g FAS were sequentially dispersed into 5 g THF by vigorous magnetic stirring, which was marked as solution B. In this research, the amount of the MWCNTs and EG was changed to optimize the electrical property of the coating. In the next step, the solution A was mixed with solution B by magnetically stirring to obtain a mixture A. Before using, 0.2 g DETA was added into the mixture A and stirred for 30 min, which was marked as mixture B. The final product was obtained by simply casting the mixture B onto the Q235 sample using a disposable Pasteur pipette (1 ml), and cured at room temperature for 24 h. The thickness of the as-prepared samples was measured to be 0.9–1.1 mm.
2.4. Characterization
In this research, the samples composed of 2 g epoxy, 0.5 g graphite powder, 0.16 g MWCNTs, 0.04 g EG, and 0.1 g FAS were utilized for the various characterization and tests without special explanation. The scanning electron microscope (SEM, TESCAN Vega3) was used to evaluate the surface microstructures of the superhydrophobic coating. Prior to SEM measurements, a thin Au layer (ca. 5 nm) was deposited on the specimens by sputtering. A home-made contact angle meter was used to measure the water contact angles (CAs). The sliding angles (SAs) and dynamic bouncing test were investigated through the high speed camera (Revealer 2F04) equipped with Tamron 90 mm zoom lens. We used 5 μl water droplets in the aforementioned test. The electrical conductivity was calculated by a Four-Probe Resistance Meter (HPS2523, China). The polarization curves were measured in 3.5 wt% NaCl aqueous solution through a CHI600D electrochemical workstation (Shanghai CH instruments). We utilized a three electrode system. The sample and a platinum electrode were utilized as the working and counter electrodes, respectively. A saturated calomel electrode (SCE) was used as reference electrode. The scanning rate of polarization curves was 1 mV s⁻¹.

2.5. Sandpaper abrasion test
The superhydrophobic sample was loaded with a weight of 500 g (12.25 KPa) and faced down to the rough surface of 200 grid SiC sandpaper. By the means of external forces, the sample moved along a ruler for 20 cm as one abrasion cycle.

3. Results and discussion
In this research, we tried to construct hierarchical structure through the combination of microscale filler and nanoscale filler. This strategy was confirmed by the SEM observation. As shown in figure 2(a), there are many microscale plates (10–100 μm), which are ascribed to graphite and expanded graphite. From the further enlarged observation (figure 2(b)), it can be found that many MWCNTs adhered to the microplates. As the microplates could offer support to the nanoscale MWCNTs, this structure has the potential to exhibit mechanical robustness. The EDS measurement was carried out at the area of figure 2(b). As shown in figure 2(c), C, O, F and Si components were detected. The other tiny peaks were ignored in this research. Here, the F component was not the main element in the superhydrophobic coating; it only affected the bonding between MWCNTs and the microplates, further improving the mechanical robustness.
component came from the FAS, which is crucial for low surface energy \cite{28, 29}. The Au component came from the Au sputtering before the SEM test.

The hierarchical structure together with the low surface energy contributed to the superhydrophobicity. As shown in figure 2(d), nine round shape water droplets was randomly distributed on the surface of the sample, indicating the excellent superhydrophobicity. In order to quantitatively assess the wetting state, we further measured the water CAs and SAs. The as-prepare sample exhibited a high CA of 162$^\circ$ and a low SA of 5$^\circ$, indicating a Cassie-Baxter state where the water droplets are suspended on the samples \cite{30}.

Mechanical robustness is the key component restricting the widespread application of superhydrophobic surfaces, because the micro/nano structures are prone to be abraded \cite{31–36}. In this research, we performed the sandpaper abrasion test to investigate the mechanical robustness. As shown in movie S1 is available online at stacks.iop.org/MRX/7/055009/mmedia and figure 3(a), the superhydrophobic sample was faced down to the rough surface of 200 grid SiC sandpaper under a pressure of 500 g weight (12.25 KPa). With the help of external forces, the superhydrophobic sample moved along a ruler for 20 cm, which was defined as one cycle. Although some powder was abraded from the sample during the sandpaper abrasion process, the sample maintained superhydrophocity (movie S1).

It was found that the thickness of the superhydrophobic coating decreased from $\sim$1.00 mm to $\sim$0.52 mm after 50 abrasion cycles. Figure 3(b) further demonstrated the change in CAs and SAs after the sample was abraded. The CAs of the superhydrophobic sample were between 159$^\circ$ and 165$^\circ$, and the SAs were between 5$^\circ$ and 8$^\circ$. Thus, it is reasonable to say that the superhydrophobicity of the sample was not destructed by mechanical abrasion, although many powders have been worn. We attribute this to the self-similar structure, which let the undamaged layer of the superhydrophobic coating are similar in functionality and texture with the exposed parts (figure 4(a)). This deduction was further confirmed by the SEM image. From figures 4(b) and (c), the surface of the as-prepared sample retained hierarchical structure even after 50 abrasion cycles.

The conductivity is another important factor because the diverse application of superhydrophobic/conductive coating. In this research, the CNT, graphite powder and EG connected with each other, which led to the formation of conductive network (figure 1). Thus, this superhydrophobic coating is conductive. Here, we tried to alter the amount of the MWCNTs and EG optimize the electrical property. During the optimizing process, the content of the epoxy and graphite powder remained unchanged. The experiment results are summarized in table 1. The detailed abrasion process could be found in the experiment section. Once the sample lost superhydrophocity, the abrasion cycles were recorded and summarized in table 1. It could be found that the electrical conductivity was increased when the amount of conductive filler was increased, but the anti-abrasion property decreased simultaneously. Thus, how to make a balance between the electrical conductivity and anti-abrasion property is a big challenge. An optimized conductive/superhydrophobic coating could reach a conductivity of 90.91 S m$^{-1}$, which achieve a balance between the conductivity and anti-abrasion.

This superhydrophobic coating also demonstrates superior self-cleaning performance. As shown in figure 5(a) and movie S2, we first placed the superhydrophobic sample at a slope angle of $\sim$15$^\circ$, and then dredge a layer of soil onto the sample. In the next step, we dropped the water droplets onto the sample. It can be found from the movie S2 that the water droplets retained the round shape and rolled off easily (figures 5(b) and (c)). Once the water droplet contacted the soil particles, it would bring the soil array. Finally, a completely clean
Figure 4. (a) Schematic illustration of the anti-abrasion strategy. The SEM image of the sample after 50 abrasion cycles with (b) low and (c) high magnification.

Table 1. The experiment results of the superhydrophobic with different content of filler.

| Experiment | MWCNT (g) | EG (g) | Conductivity (S m⁻¹) | Anti-abrasion (cycles) |
|------------|-----------|--------|----------------------|------------------------|
| 1          | 0.06      | 0.013  | 3.33                 | >250                   |
| 2          | 0.1       | 0.025  | 10.00                | 200                    |
| 3          | 0.12      | 0.03   | 33.33                | 80                     |
| 4          | 0.16      | 0.04   | 90.91                | 50                     |
| 5          | 0.2       | 0.05   | 238.10               | 3                      |

Figure 5. Self-cleaning process on the as-prepared superhydrophobic surface: (a) the surface with soil powder as a model of contaminant; (b) and (c) the contaminated surface with water droplets on it; (d) the contaminated surface after the water pouring process.
sample was obtained (Figure 5(d)), indicating that this superhydrophobic coating could effectively prevent the contamination.

Until now, most superhydrophobic surfaces are apt to be contaminated by the oil because the oil has lower surface tension than that of water which would further lead to the oil penetrating. Although some researches have fabricated superamphiphobic surfaces, these surfaces would lose function in some specific situation which requires not only a smooth oil layer but also excellent water repellency, such as lubricating gears. Recently, Lu et al. reported some special superhydrophobic coating could exhibit outstanding self-cleaning performance in both air and oil environment [25]. In this research, a similar dirt-removal test was performed. As shown in movie S3 and figure 6(a), the as-prepared superhydrophobic sample was partly inserted into oil (hexadecane), and then contaminated by the dirt (soil powder). Then the water droplets were drop wisely on the sample (figures 6(b) and 6(c)). It was found that the water droplets would carry the contaminants away once they met them. Finally, a clean surface was obtained after the water pouring process. Thus, it can be deduced that our superhydrophobic surface showed outstanding self-cleaning performance in both oil and water environment. Here, the excellent self-cleaning performance after oil contamination is due to the slippery structure which was inspired by the nepenthes pitcher plant. The slippery structure could lock-in the oil, and act by itself repelling the water droplets [37].

Furthermore, this superhydrophobic demonstrated superior anti-corrosive performance. As is known to all, the steel is widely used in our daily life. However, the carbon steel (especially Q235 steel) is prone to be oxidized. Here, it is found that this superhydrophobic coating could effectively protect the Q235 steel from corrosion using the polarization curves. In this research, the control experiment was performed by investigating the anti-corrosive properties of bare Q235 steel, epoxy coating and superhydrophobic coating simultaneously. The thickness of the epoxy coating and the superhydrophobic coating were set at 1 mm. In this research, 3.5 wt% NaCl aqueous solutions were used as the electrolyte. Before the test, the samples were immersed into the electrolyte for 3 h. From figure 7, the corrosion potential ($E_{corr}$ versus SCE) and the corrosion current density ($I_{corr}$) can be deduced, which are two important parameters for assessing the anti-corrosive performance. Then, the corrosion potentials for bare steel, epoxy coating and superhydrophobic coating were calculated to be $-0.462$, $-0.714$ and $-0.876$ V, respectively. Moreover, the corrosion current density for the bare steel, epoxy coating, and superhydrophobic coating was $6.46 \times 10^{-4}$, $3.47 \times 10^{-5}$, and $3.43 \times 10^{-6}$ A cm$^{-2}$, respectively. It should be noted that the corrosion current density of steel surface with superhydrophobic coating reduced 188-fold compared with bare steel, and reduced 10-fold compared with epoxy coating. We attributed this phenomenon to the mechanism of superhydrophobicity. When the superhydrophobic coating was immersed in

Figure 6. The self-cleaning test of the as-prepared superhydrophobic coating on oil–solid–vapor interfaces: (a) the contaminated sample was partly immersed in the oil. (b) and (c) the contaminated surface with water droplets on it; (d) the contaminated surface after the water pouring process.
the NaCl solution, air will be stuck in the micro/nano structure of the coating. The trapped air will serve as a cushion and prevent the electron transfer between Q235 steel substrate and the corrosive electrolyte [38, 39].

Thermal stability is another important parameter for assessing the durability of the superhydrophobic coating under harsh environment. Here, we first put the samples into the oven with different temperature for 2 h. From figure 8(a), it can be found that this coating maintained superhydrophocity even when the heating temperature was up to 300 °C. Then, we tried to heat the superhydrophobic sample under 250 °C heat treatment for 0–24 h. The CA and SA measurement indicated that the superhydrophocity was maintained (figure 8(b)).

4. Conclusion

In summary, scattering microscale and nanoscale conductive fillers into epoxy matrix endows the composite with robust superhydrophobicity and good conductivity. The as-prepared conductive/superhydrophobic coating exhibits a robust resistance to sandpaper abrasion, high temperature heating, and corrosive solution attack. Moreover, this coating demonstrates outstanding self-cleaning performance in both water and oil environment. Another advantage of our method is the simplicity of the painting process; complicated equipment is not required.

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Conflicts of interest
The authors declare no conflict of interest.

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