Highly durable superhydrophobic surfaces based on a protective frame and crosslinked PDMS-candle soot coatings

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Abstract

Superhydrophobic surfaces can be applied to environmental, energy, and healthcare fields. However, the weak durability issue has significantly limited the practical industrial applications. To overcome the readily destroyed interfacial structures and chemical compositions, the superhydrophobic surfaces with high mechanical and chemical durability have been created on a stainless steel mesh (SSM) as a protective frame based on tightly crosslinked polydimethylsiloxane (PDMS)—candle soot (CS) composite coatings through simple mechanical transfer and candle burning processes, which abbreviated to SSM/PDMS—CS and showed a water contact angle (WCA) of 159.4° ± 1.6° and a sliding angle (SA) of 2.3° ± 0.5°. Harsh abrasion examinations based on sandpaper and fiber paper have been conducted by applying 100 g weight and 200 cm sliding distance. And the obtained WCAs and SAs were 155° ± 3.7°, 155.7° ± 2° and 3.7° ± 0.5°, 3.5° ± 0.3°. Scanning electron microscope showed scratches on the surface, but the superhydrophobic property has been well maintained. After 40 kHz ultrasonication treatment for 30 min, WCA and SA were 152° ± 0.9° and 4.1° ± 0.8°. Finally, SSM/PDMS—CS surfaces exhibit good chemical resistance to corrosive solutions even after pH 2, pH 10, 1 M NaCl, and toluene treatments for 15 days.

1. Introduction

In the past decades, the advances in bioinspired superwettable interfacial materials have integrated multidisciplinary research fields and various development stages, involving materials innovation, interfacial construction, mechanism study, applications exploration, practical use, and industrial production, which can apply to environmental protection, biomedical engineering, energy area and so on [1]. Specifically, the functions properties of superwettable interfaces were widely exhibit in self-cleaning, anti-icing, anti-fogging, anti-corrosion, oil-water separation, marine anti-fouling, liquid collection, biosensing, heat transfer, healthcare, nano energy, chemical reaction, and others [1, 2]. Considering air, water, and oil phases, up to 64 unique wetting states have been reported, leading to the overall system with special wettability [1, 3]. Among these interfaces, superhydrophobicity is one of the most classic types, which has gained tremendous interest for long time. The critical theoretical basis of creating superhydrophobicity is introducing the highly rough micro- and nanostructures into a low surface energy surface, which can be interpreted as rough topographical structures and nonpolar chemical surfaces as well [4, 5]. Therefore, a trapped air layer along the interface was formed with decreased contact area, lower surface energy, lower water adhesion, and higher water repellency, leading to a large water contact angle (WCA) and a small sliding angle (SA) of water droplets.
However, the rough structures and surface chemistry of artificial superhydrophobic materials were readily destroyed by harsh ambient environments, which can be mainly divided into mechanical or physical damages, chemical corrosion, biofouling, and environmental impacts [6]. The fact that enhancing mechanical durability of superhydrophobicity has been the prerequisite for moving it toward practical applications has been confirmed, despite other factors must be addressed as well [7]. Therefore, focusing on the relationship among substrate, binding layer, or nonpolar surface, mechanically durable superhydrophobic materials can be created and several preparation strategies were significantly developed. For instance, enhancing passive resistance to external mechanical impact, wear, abrasion, scratch, etc. has been a common and intuitive method for such superhydrophobic surfaces. Specifically, it is very helpful by means of constructing hierarchical architectures with dual micro-/nano-scales [8, 9], creating highly adhesive substrate/coating interfaces [10, 11], crosslinking building blocks based on intramolecular or intermolecular covalent bonding [12, 13], and printing materials as an integrated object or a building block [14, 15]. Moreover, the robust superhydrophobic coatings can be designed in a self-similar manner, which material composition and structure of exposed coatings are similar while suffering from mechanical damages. Thus, mechanically robust all-organic superhydrophobic coatings based on fluorinated epoxy resin, perfluoropolyether, and polytetrafluoroethylene nanoparticles were fabricated [16]. The surface frame as a protective structure can serve as ‘armour’ with a relatively larger microscale to prevent abradants from removing nanostructures for creating fragile superhydrophobic surfaces, which has been an excellent robustness strategy [17]. As a good complementary strategy described as active resistance, healable superhydrophobic surfaces triggered by UV, sunlight, temperature, water, or low surface agent can be realized through transportation, regeneration, or recovery of the surface chemistry and rough structures [18–20]. In the present work, a combination of molecular crosslinking, self-similar coating, and protective frame strategies were applied to creating highly durable superhydrophobic surfaces.

Candle soot (CS) comprising carbon nanoparticles as a traditional unwanted pollutant turned into recycled functional materials [21], extensively applied for biomedical engineering [22], energy research [23], environmental science [24], and other fields [25]. For interfaces with special wettability, a robust superamphiphobic coating has been created based on a CS template [26]. Mediated by polydimethylsiloxane (PDMS) as a binder, CS was simply deposited to form a relatively stable superhydrophobic coating, which lost superhydrophobicity examined by the abrasion test [27]. In a similar manner, the double layered PDMS-CS coating was fabricated on an aluminum alloy substrate for enhancing corrosion resistance, which showed an obviously reduced WCA of about 142° after several friction cycles [28]. In addition, a Janus superhydrophobic/superhydrophilic paper based on a PDMS and CS coating was prepared, whereas the WCA only remained more than 140° after 100 cm sandpaper abrasion [29]. Therefore, simply realizing mechanically durable superhydrophobic PDMS-CS-based surfaces is still highly desired and challenging.

Herein, highly durable superhydrophobic surfaces through a protective stainless steel mesh (SSM) frame and chemically crosslinked PDMS-CS coatings were simply obtained by direct mechanical transfer and candle burning processes. Thanks in large part to a combination of molecular crosslinking, self-similar coating, and protective frame strategies, as-prepared superhydrophobic materials can tolerate harsh mechanical impacts and chemically corrosive environments.

2. Experimental

2.1. Materials

The SSM (635 mesh, 304 model, Guangdong, China), sandpaper (320 grit), PDMS (Sylgard 184, Sigma-Aldrich), paraffin candle, film-casting knife, cellulose-based fiber paper, and ethanol were available commercially and used as received.

2.2. Preparation of durable superhydrophobic SSM/PDMS-CS surfaces

The tailored SSM substrates with a size of 2 cm × 2 cm were immersed in aqueous 50% ethanol with 40 kHz sonication treatment for lasting 10 min, then naturally dried at room temperature. After that, uncured PDMS (weight ratio of prepolymer: curing agent = 10:1) was prepared by mixing and vacuum procedures. The as-prepared SSM was immersed into PDMS for several minutes to fully wetting metal surfaces. Then SSM with uncured PDMS was taken out and excessive PDMS on the surface was removed by an abrasion using a blade. At the same time, a paraffin candle was lighted and remained stable. The flame height was about 2–3 cm. Then, a CS layer was deposited on the glass slide with an approximately 3 cm × 3 cm surface area by holding substrate at the one third position of the flame for 1 min. Then, a CS layer with a microscale thickness was directly transferred onto the resulting uncured SSM/PDMS substrate by compression under a weight of approximately 20 kg. The transferring process aimed at making CS layer contact PDMS well, leading to a superhydrophobic liquid-solid interface with the help of mechanical pressing and capillary action. At last, the obtained SSM/
PDMS-CS substrate was treated with another soot process for about 10 s to directly cure composite coatings and further enhance the superhydrophobicity. Therefore, the highly durable superhydrophobic SSM/PDMS-CS materials were successfully prepared.

2.3. Sample characterization

First, the mechanical durability of as-prepared samples was mainly examined by sandpaper or fiber paper-based abrasion tests and ultrasonication treatments. First, considering a variety of mechanical testing methods with the lack of standardization have been used, the common linear abrasion test was conducted according to a literature [10]. Specifically, the superhydrophobic surface of SSM/PDMS-CS was placed facing down to the rough side of sandpaper or fiber paper, and a loading weight of 100 g was applied to the top position. Subsequently, forward sliding for 5 cm with a constant velocity served as one cycle and backward sliding for 5 cm as the second cycle was carried out. After several sliding cycles, up to 200 cm abrasion distance was conducted. Second, the ultrasonication treatment for deeply testing samples with durable superwettability was conducted at the frequency of 40 kHz. At last, chemical anti-corrosion tests were conducted by immersing samples in pH2, pH 10, 1 M NaCl, and toluene solutions for 15 days, followed by a rinsing process. After various durability tests, surface morphology and wettability can be measured by the field-emission scanning electron microscopy (FE-SEM; Carl Zeiss) and the contact angle goniometer (Smartdrop, Femtobiomed), respectively. The water contact angle (WCA) and the sliding angle (SA) were all measured on three positions using a 4 μl water droplet.

3. Results and discussion

3.1. Preparation of durable superhydrophobic SSM/PDMS-CS surfaces

The present work aimed to creating highly robust superhydrophobic surfaces on a porous stainless steel with a resistance to mechanical abrasion and chemical corrosion. The damage or loss of micro-/nanostructured surface roughness and nonpolar chemistry will be subtly avoided during abrasion and corrosion processes though a combination of molecular crosslinking, self-similar coating, and protective frame strategies mentioned above. As shown in figure 1, the inner pores and surfaces of SSM were filled or covered with uncured PDMS to form SSM/PDMS, which was then mechanically pressed on a glass slide with a CS layer, thereby making CS nanoparticles diffuse into PDMS well based on a mechanical transferring process. Another soot process was conducted to cure SSM/PDMS-CS composites to be more superhydrophobic and durable.

To enhance anti-abrasion and anti-corrosion properties of superhydrophobic SSM/PDMS−CS composites, the PDMS-CS interface and cross-linked PDMS played an important role. Oleophilic CS particles with low surface energy can be readily pressed into uncured hydrophobic PDMS by an external pressure during a transfer process. During the burning process, nonpolar PDMS molecules and CS particles mainly comprising polyaromatic hydrocarbons can bond and interact with each other based on a weak intermolecular force of van der Waals, which is mainly resulted from the dispersion force. Considering flame heating and molecular catalysis, as illustrated in figure 2, PDMS comprising prepolymers and curing agents was rapidly crosslinked based on hydrosilylation reaction between −CH=CH2 and -Si-H. Therefore, the resulting PDMS-CS coatings
were supposed to include three sections, crosslinked PDMS-CS (bottom), crosslinked PDMS-CS (interface), and unconsolidated CS layer (top), which provided a structural basis.

Herein, as shown in figure 3(a), the microscale, porous, and fibrous SSM was selected as a structural frame for protecting superhydrophobic PDMS-CS coatings. Except the protective function, superhydrophobic SSM can also be potentially utilized for various applications in the future. For instance, implantable medical devices, such as metal mesh-based heart stents, covered stents, cardiac occluders, etc. can be developed considering superhydrophobic surfaces have been preliminarily demonstrated with blood-repellent properties for anticoagulation and antithrombus [30–32]. In addition, superhydrophobic SSM has been widely used for metal corrosion protection and oil-water separation [33]. As shown in figure 3(b), CS nanoparticles with a size of about 50 nm aggregated together to form an unconsolidated network through weak interactions [25]. After mechanical transferring and flame burning, robust superhydrophobic SSM/PDMS–CS composites were successfully obtained, exhibiting a unique polygonal shape caused by the pressed SSM frame (figure 3(c)). Accordingly, the CS nanoparticle network in figure 3(d) were shown in a denser state, especially focusing on the boundary region.
3.2. The superhydrophobicity of SSM/PDMS-CS surfaces

The wettability of as-prepared samples was measured by static WCAs and SAs of water droplets. Considering the same chemical composition of CS nanoparticles, the special wettability is mainly determined by surface roughness. Herein, for a comparison, various substrates of glass, bare SSM, and SSM/PDMS were selected for preparing CS-based materials with superhydrophobicity. As illustrated in figure 4(a), CS nanoparticles were deposited on a glass substrate with a porous aggregated structure, which was similar to that on a SSM substrate (figure 4(b)). The measured WCAs were 158.5° ± 0.3° and 159.2° ± 1°, respectively. In addition, figure 4(c) illustrated the material structure and showed the special wettability with a WCA of 159.4° ± 1.6° and a SA of 2.3° ± 0.5°. Although they have similar wettable properties resulted from CS nanoparticles located on the surface, the robustness was completely different due to the difference of substrate structure.

3.3. The mechanical durability test on superhydrophobic SSM/PDMS-CS surfaces

In general, a variety of methods for examining mechanical durability of superhydrophobic surfaces have been employed, such as water jet, sand abrasion, tape peeling, mechanical abrasion, blade scratching, and so on, which the mechanical abrasion test is accessible and reliable by rubbing a rough solid abradant against the superhydrophobic surface under a load [34]. Therefore, the sandpaper-based abrasion test was carried out and the WCA of as-prepared superhydrophobic SSM/PDMS-CS surfaces decreased from 159.4° ± 1.6° to 155° ± 3.7° after an abrasion distance of 200 cm under a 100 g load (figure 5(a)). Accordingly, the SA increased to 3.7° ± 0.5° from original 2.3° ± 0.5° (figure 5(b)). This was resulted from a slight loss of surface roughness caused by erased CS nanoparticles on the SSM/PDMS fame. However, the unique superhydrophobicity was still maintained after a harsh abrasion test, considering a reference standard of 150° for WCA and 5° for SA.

The SSM/PDMS-CS surfaces were composed of convex frame and concave valley, which the overall wettability was controlled by these two parts as well. To analyze the change in surface morphology after an abrasion, FE-SEM images were acquired, which displayed several scratches on the convex frame position of SSM/PDMS-CS surfaces (figure 6(a)). It was noted that morphology of the magnifying concave valley section was protected and unchanged considering the frame protection, which showed aggregated CS nanoparticles (figure 6(b)). First, the crosslinked PDMS-CS coatings can greatly withstand external mechanical abrasion. Moreover, the ultrahigh water-repellency from inlayed PDMS-CS coatings ensured overall superhydrophobicity to some degree, even if PDMS-CS coatings on a SSM frame was partly wiped off.

Compared to a rough sandpaper, the smoother fiber paper was expected to further reduce surface roughness of SSM/PDMS-CS surfaces for a harsh abrasion test. Therefore, a same abrasion distance of 200 cm based on a fiber paper was applied in a similar manner, the WCA gradually decreased to 155.7° ± 2° from original 159.4° ± 1.6° (figure 7(a)) while the SA increased to 3.5° ± 0.3° from 2.3° ± 0.5° (figure 7(b)), which showed a nearly same performance compared to the sandpaper-based abrasion test.

Similarly, as shown in figure 8(a), incompact CS nanoparticles were partly swept away after the fiber paper-based abrasion and PDMS-CS coatings on the frame section became smooth compared to the pristine rough SSM/PDMS-CS surfaces, leading to a reduced WCA. Fortunately, PDMS-CS coatings embedded in concave...
valleys were kept intact, exhibiting rough porous or network structures (figure 8(b)). As a result, abraded SSM/PDMS-CS surfaces remained unique superhydrophobic properties. The present work aimed to prepare highly durable superhydrophobic materials for the practical applications. Therefore, a simple video of abrading SSM/PDMS-CS surfaces against a fiber paper under a random finger pressure much higher than a loading weight of 100 g was recorded, which were processed for corresponding optical images as shown in figure 9. Ultrahigh water-repellency and low water-adhesion can be obviously seen, indicating the excellent superhydrophobicity even after a harsh abrasion test.

To deeply examine the mechanical durability of SSM/PDMS-CS surfaces exhibiting superhydrophobicity, destructive tests were conducted based on an ultrasonication treatment with a stronger impact from a
combination of mechanical stretching, cavitation effect, and thermal effect. Therefore, the ultrasonication with a frequency of 40 kHz was carried out up to 30 min. As shown in figure 10(a), the resultant WCAs were nearly kept unchanged, thereby fluctuating around 155.2° ± 0.9° from a time point of 10 min. Similarly, the SA slightly increased to 4.1° ± 0.8° which was in the standard range of less than 5° for superhydrophobicity (figure 10(b)). It was noted that the superhydrophobic SSM/PDMS–CS surfaces were wetted during sonication and lost the

Figure 8. FE-SEM images of abraded SSM/PDMS–CS surfaces after a distance of 200 cm on a fiber paper. (a) zoom out. (b) zoom in.

Figure 9. Optical images for a finger pressure-based abrasion test on superhydrophobic SSM/PDMS–CS surfaces against a fiber paper.

Figure 10. The relationship between superhydrophobicity and sonication time for SSM/PDMS–CS based on ultrasonication abrasion. (a) WCA variation. (b) SA variation.
trapped air layer along solid-liquid interfaces due to the extruded bubbles, whereas it was reversible and superhydrophobicity was then regained once the surface was dried.

3.4. The chemical durability test on superhydrophobic SSM/PDMS-CS surfaces

During practical applications of superhydrophobic functional materials, chemical durability against harsh corrosive environments (acidic, alkaline, salty, organic, etc.) must be considered, which is another prerequisite to create a long-lived water-repellent surface [6, 16]. Corrosion resistance has been a huge challenge for conventional inorganic materials compared to all-organic building blocks [16]. Therefore, the chemical stability of superhydrophobic SSM/PDMS-CS surfaces needed to be strictly examined to further prove the overall robustness. As shown in figure 11, although superhydrophobic SSM/PDMS-CS surfaces were immersed in various chemically corrosive solutions (pH 2, pH 10, 1 M NaCl, and toluene) for up to 15 days, the values of WCA and SA were nearly constant around 158° and 4°, indicating a high resistance to chemically corrosive environments, which was thanks in large part to the chemically inert and tightly crosslinked PDMS-CS composites.

4. Conclusion

In summary, to overcome readily destroyed micro-/nanostructures and chemical compositions for creating superhydrophobic interfaces, we successfully prepared a highly durable SSM/PDMS-CS surface exhibiting unique anti-abrasion and anti-corrosion properties by means of simple mechanical transfer and candle burning techniques, which were deeply examined by sandpaper-based abrasion, fiber paper-based abrasion, ultrasonication treatment, and chemical corrosion, respectively. This robustness especially for mechanical durability was mainly derived from a protective SSM frame and tightly crosslinked PDMS-CS coatings. After a series of harsh tests, resultant WCAs and SAs fluctuated around 155° and 4°, which satisfied the standards of superhydrophobicity. The as-prepared extremely robust SSM/PDMS-CS surfaces can be further used for various practical applications in the future.

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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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