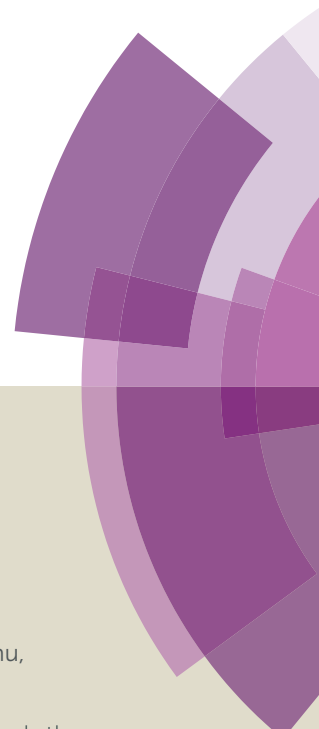


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Flexible and Mechanically Robust Superhydrophobic Silicone Surfaces with Stable

Cassie-Baxter State

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Abstract

Durable non-wetting surfaces require high surface roughness on the nano- or micrometer scale, which is inherently fragile and easily removed by an external force. Elastic materials have potential advantages for constructing superhydrophobic surfaces with abrasion resistance since after friction or force deformation they often rebound to their original structure rather than undergoing degradation. Here we present a large-scale fabrication of free-standing silicone monoliths with a stable Cassie-Baxter state under mechanical stress cycles. The obtained elastic silicone retains excellent mechanical durability with constant super liquid-repellent after high external pressure, knife-scratch, and abrasion cycles with sandpaper. Furthermore the obtained silicone demonstrates high tolerance to continuous contact with extremely corrosive solutions, and also shows self-cleaning properties in air or under oil.

1. Introduction

Lotus leaves in nature are superhydrophobic with water contact angles greater than 150° and roll-off angles below 10° , and this is achieved by a combination of low-energy surfaces and roughness on the nano- and micrometer scales.¹ Mimicking the lotus leaves surface morphology has led to the development of a number of artificial superhydrophobic surfaces, opening-up many applications such as oil-water separation,^{2, 3} self-cleaning paints and windows,⁴ non-wetting fabrics,⁵⁻⁷ buoyancy aids,⁸ anti-fogging⁹ and anti-icing surface.¹⁰ Currently, a major problem of superhydrophobic surfaces is the mechanical fragility of the nanostructures, which results in the degradation of the superhydrophobicity in common environments (e.g., long-term air-exposure, mechanical abrasion, external pressure, contamination with pollutants, acid rain, etc.).¹¹⁻¹⁶ In order to improve the stability of superhydrophobicity, one promising method is to produce hierarchical roughness on a surface as that it resists mechanical wear.¹⁷ The key idea is the protection of the fragile fine-scale surface topographies by using larger scale sacrificial micropillars.¹⁸ However, the mechanical durability of such surfaces is still far from the level that is required once the micro-pillars have been worn away.¹⁹ Another method is to fabricate shape memory materials with reversible recovery of surface topographies. Unfortunately, the shape memory material is unlikely to recover from damage that arises from a significant material loss on its surface. In addition, the self-healing materials are expensive and often toxic, and no healing occurs under ambient conditions. As a consequence, their applicability has remained limited.^{20,21}

Elastic surfaces are regarded as good candidates for designing damage-tolerant superhydrophobic surfaces. Under external pressure and abrasion cycles, the surface elastic microstructures will be compressed by deformation to avoid being broken. Typically after the removal of external load the surface deformations tend to rebound to their original surface structure.²² This elastic deformation will help to stabilize the necessary air cushions trapped in the microstructures that are required to maintain the durable superhydrophobic surfaces with a Cassie-Baxter state. Silicones are important elastic high-performance polymers because of their excellent flexibility, durability, high thermal-stability and excellent biocompatibility,²³ which enable them to be widely used in the area of microfluidic devices,

soft-lithography and biomedical applications etc. In this work, we created superhydrophobic elastic silicone with superior mechanical durability, Cassie-Baxter state stability and outstanding resistance after long-term exposure to extremely corrosive liquids. This was achieved by creating elastomeric surfaces with dual scale roughness from a polymer process. Furthermore if the robust silicone is made to lose its superhydrophobicity (something that is difficult to achieve), it can be simply healed by removing the uppermost layer by sandpaper abrasion or by limited heat treatment. In addition to forming three-dimensional monoliths, the elastic silicone surface can also be fabricated on different substrates as a coating through a brush-painting process in a large scale.

2. Results and Discussions

2.1 Liquid repellency of the obtained elastic silicone

The elastic silicone monoliths were fabricated by mixing two different sizes of surface functionalized hydrophobic SiO₂ particles with polydimethylsiloxane (PDMS, commonly called silicone) followed by pre-cure at 70°C for 2 h then at 210°C for 2 h. The characterization of the two different sizes of surface functional SiO₂ particles is shown in Supporting Information (Figure S1). Figure 1a and b shows the water droplets on the monolithic silicone surface, and they have a static water contact angle (CA) of $156 \pm 1^\circ$ as well as a sliding angle (SA) $4 \pm 0.5^\circ$. The elastic silicone can be readily made into a superhydrophobic thin film which can be easily folded many times and returns to its original shape without cracks or loss of integrity (Figure 1c). The elastic silicone is also stretchable and a typical stress-strain curve shows a large plastic deformation with a tensile strength of 4.7 MPa (Figure S2, Supporting Information). Since coating is an essential step in adjusting the surface properties of materials, a simple brushing method was used to apply the elastic water-repellent coatings to a large-scale fabrication (Figure 1d). The silicone was shown to be highly repellent to many liquids including juice, coffee, milk and blood (Figure S3, Supporting Information). When immersed in animal blood, the silicone surface facilitated no detectable blood adhesion (Figure 1e, f and Movie 1 in Supporting Information), indicating the high repellence.

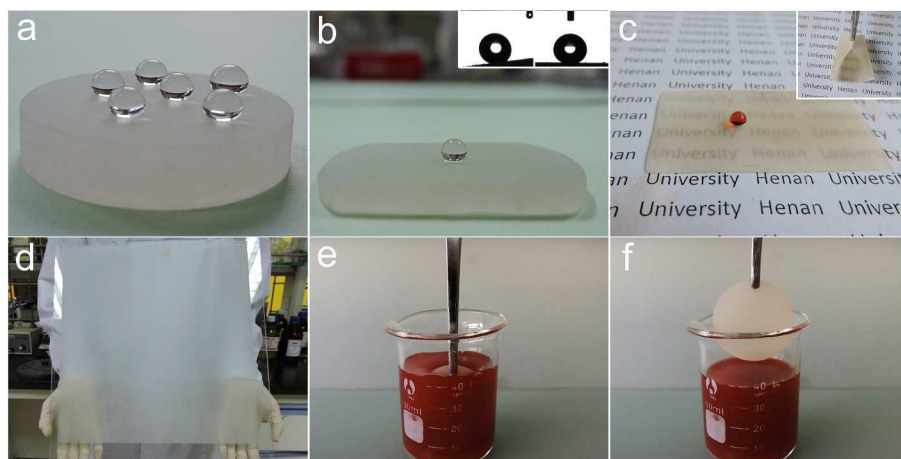


Fig. 1 Liquid repellency of the obtained elastic silicone. (a-b) Water droplet sitting on monolithic silicone surface, (c) on silicone film surface. (The inset in (c) is a photograph of the bent film, and the water droplet was dyed red for easy observation). (d) The silicone coating on glass substrate. (e, f) No adhesion after dipping in blood.

2.2 Stability of Cassie-Baxter state

The mobility of a water droplet on a superhydrophobic surface has long been associated with its wetting state. When water drops are sitting on the top of the solid textures and air is trapped underneath, they are in the Cassie-Baxter state. However, the Cassie-Baxter state is metastable, as the air layer underneath the droplets can be disrupted when subjected to high pressure, high temperature, or when encountering liquids with impurities.²⁴ It is extremely challenging to prevent or delay the transition from the slippery Cassie-Baxter state to the sticky Wenzel state. Squeezing water droplets between two surfaces is often used to investigate the stability of the Cassie-Baxter state of the superhydrophobic coatings against external pressure. The sequence photographs in Figure 2a-e and Movie 2 in Supporting Information demonstrate the behavior of a water droplet on the silicone surface after heat treatment. It is clear that the water droplet turns flat under external pressure, however, the compressed water droplet does not wet the surface instead it slips away easily from the two identical silicone surfaces, suggesting the stability of the Cassie-Baxter state. It is believed that the elastic deformation will stabilize the air cushion and therefore result in a stable Cassie-Baxter state. Figure 2f-j reveals the behavior of a droplet falling onto the surface of the silicone surface. The droplet initially

deformed and flattened into a pancake shape, but then retracted and finally rebounded off the surface without penetrating the nanostructure. After several more bounces, the droplet eventually bounced off the surface without ever coming to rest (Movie 3 and 4, Supporting Information). Note that not all superhydrophobic surfaces reported in the literature achieved this behavior especially when the surface lacked a sufficiently low surface free energy or possessed geometrical spacing between the nanoscale structures.²⁵ Owing to the low adhesion to water of the silicone surface after flame heat treatment, it was very difficult to deposit water drops because they immediately rolled off, and the dynamic contact angle measurement also demonstrated the high repellency and extremely low adhesion to water (Figure 2k-o, Movie 5 and 6, Supporting Information). For details of the flame heat, see Movie 7 in Supporting Information.

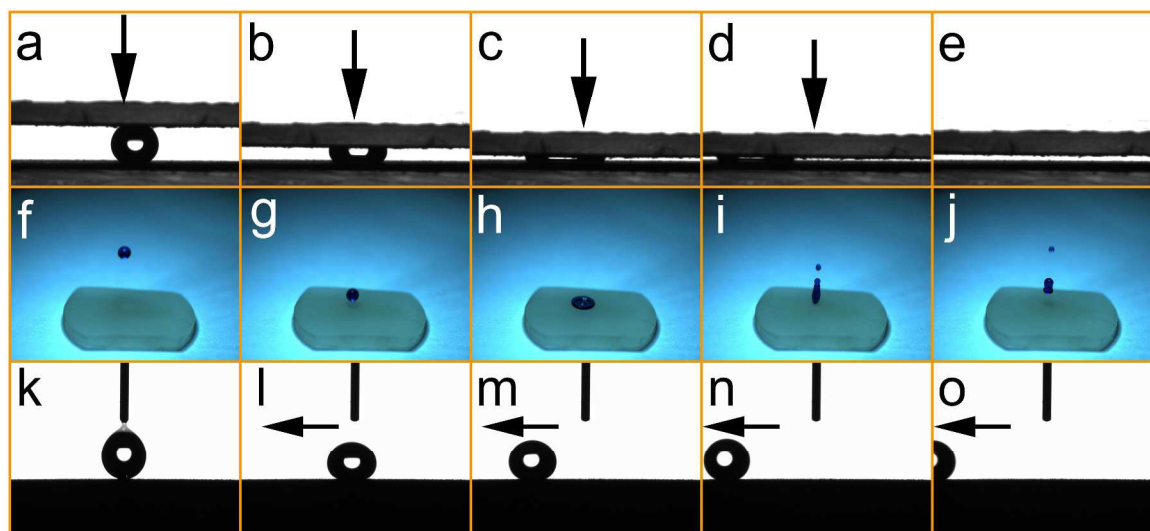


Fig. 2 Stability of Cassie-Baxter state. (a) Behavior of a 5 μL water droplet squeezed between two superhydrophobic silicone. (f-j) Time-lapse photographs of water drop bouncing. (k-o) Water droplet sliding on the silicone surface with sliding angle about 0° .

2.3 Durability of the superhydrophobic silicone in harsh environment

The obtained silicone can also tolerate external mechanical pressure including the continuous impact of water droplets. As shown in Figure 3a and b, the silicone is exposed to two smooth steel plates under a pressure of 1500 N. After the pressure is released, the silicone still retains its superhydrophobicity with a water CA of $154 \pm 1^\circ$ (Figure 3c). Because of the elastic deformation the

surface also retains its superhydrophobicity even after the impact of many thousands of water droplets (Figure 3d and Movie 8, Supporting Information). Under pressure, elastic micro-protuberances will be compressed and collapsed by elastic deformation to avoid being broken, and the deformation will rebound to renew the original surface structure when the load is withdrawn, thereby keeping the superhydrophobicity constant (Figure 3e). This result can be verified from the SEM images. At present, the assessment of mechanical durability of superhydrophobic surfaces has been hampered by the lack of a single, standardized test method.²⁶ No single measure has been used for characterizing the effect of wear. In this work, different methods were employed to evaluate the mechanical resistance of the superhydrophobic elastic silicone. As shown in Figure 3f-i, the elastic silicone or coating exhibits robust superhydrophobicity under various harsh conditions such as the tape-peel test, knife-scratch and finger-wipe (Movie 9, Supporting Information). The silicone monolith can sustain its superhydrophobicity after mechanical abrasion cycles because of the low surface energy and microstructures extending throughout its whole volume. When the uppermost layer is removed upon scrape abrasion, the newly exposed surface is also water-repellent, thereby making the superhydrophobicity permanent (Figure S4 and Movie 10, Supporting Information). In addition, the silicone could withstand extremely low temperatures even cooling in liquid nitrogen and warming to room temperature produced no frost cracks. After being taken from the liquid nitrogen, the silicone was still at very low temperature and immediately became covered with ice because of the condensation of water vapor from the air. However, the superhydrophobic property of the silicone was restored after the ice melted and disappeared (Figure S5, Supporting Information). It is well known that most materials do not have permanent superhydrophobic properties. If our silicone surface is disabled (for example, infused with lubricant), it can be easily healed to recover its superhydrophobicity by applying heat to the worn areas or just by removal of the uppermost layer using sandpaper.

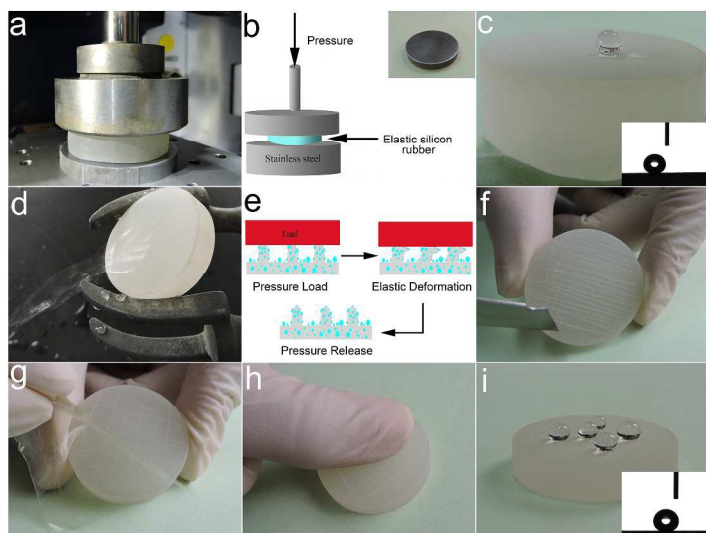


Fig. 3 Wetting stability of silicone in harsh environment. (a) Compressed under a load of 1500 N, (b) schematic for compression test (inset is a photograph of the smooth steel), (c) the silicone sustains the superhydrophobicity after the pressure is released. (d) Water droplet impact. (e) Schematic of elastic deformation. (f) Scratched by a knife, (g) adhered by a tape, (h) touched by a finger, (i) silicone still retains superhydrophobicity after damage.

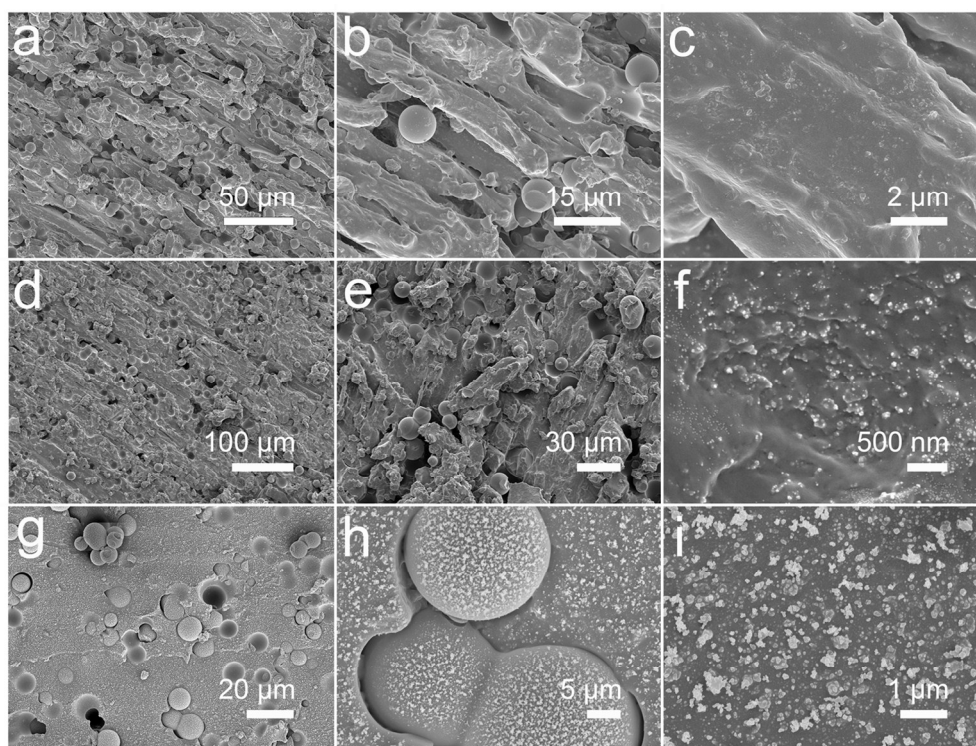


Fig. 4 SEM images of the silicone surface. (a-c) Abrasion with sandpaper, (d-f) after two smooth steel compressed, (g-i) after heated over the flame.

SEM observations were carried out to investigate the variation of the surface morphology after different damage. Figure 4(a-c) show the SEM images of the silicone surface after sandpaper abrasion. It is clear that the surface contains micro and nano-scale roughness features from the different size of hydrophobic SiO₂ particles. Compared with the morphology before sandpaper abrasion (shown in Figure S6 in Supporting Information), the micro-nanoscale structures do not have obvious change and that's why the obtained material surfaces possesses better abrasion resistance after being scratched repeatedly until completely worn out. After being compressed by two smooth steel surfaces under a pressure of 1500 N, the coated surface also has similar micro-nanoscale structures before and after the pressure test (Figure 4d-f), it is believed that the elastic deformation will keep the surface structures unchanged when facing the smooth steel surface under load. Figure 4g-i shows the surface microstructure after being heated over the flame and the morphology was found to have an apparent change. The surface is composed of assembled microsized particles studded with nanoparticles, forming a micro-nanoscale binary material with dual scale rough features. The roughness of the silicone surface increases and this effect enhances the extreme water-repellency. Surface chemical composition is another factor seen in the literature that causes variation in wettability. Here XPS analysis was employed to investigate the chemical composition of the sample before and after being heated over the flame (Figure S7, Supporting Information). Only Si, O, C, and Au are detected indicating the purity of the silicone. Note that the Au signal comes from the Au coating used to aid imaging of the sample. After being heated over the flame, the Si content increases slightly from 29.85 to 30.13 %, which may indicate that more SiO₂ nanoparticles appear on the surface. From the SEM and XPS analysis, it is believed that SiO₂ particles can migrate to repair the degraded area and restore the superhydrophobicity upon application of heat treatment.

When studying the durability of a superhydrophobic surface, the evolution of the static water contact angle in time after exposure to continuous contact with corrosive liquids should be considered. The heat treatment of the polymer film was found to increase the materials corrosion resistance. The degradation of superhydrophobicity for the obtained silicone was investigated in strong acid and alkali

solution. The water contact angle as a function of immersion time in strong alkaline (pH = 14) and acidic solution (pH = 1) was measured, as shown in Figure 5 a and b, respectively. There is no significant degradation of the superhydrophobicity after being attacked in strong acidic or alkali solution. Note the sliding angle was not influenced and was maintained at about 0° for the whole experiment. However, it is believed that the immersion method will not completely reflect the details of superhydrophobic degradation since the mechanism of degradation associated with hydration of surface active sites tends to create hydrogen bonds.²⁷ In the first method, we measured the water CA value of the silicone after being dried at 80°C, which may result in the sample restoring its superhydrophobic property. Hence, it needs to consider the evolution of water contact angle in time (dynamics of wetting) in the conditions of continuous contact with corrosive water droplet. As is shown in Figure 6a and b, the water droplet keeps a near-spherical shape and becomes smaller because of water evaporation. After staying on the superhydrophobic silicone surface for a certain time, the corrosive water droplet detaches from the surface spontaneously and sticks to the hydrophilic needle, which indicates that the superhydrophobic silicone has excellent chemical stability and low adhesion to water.

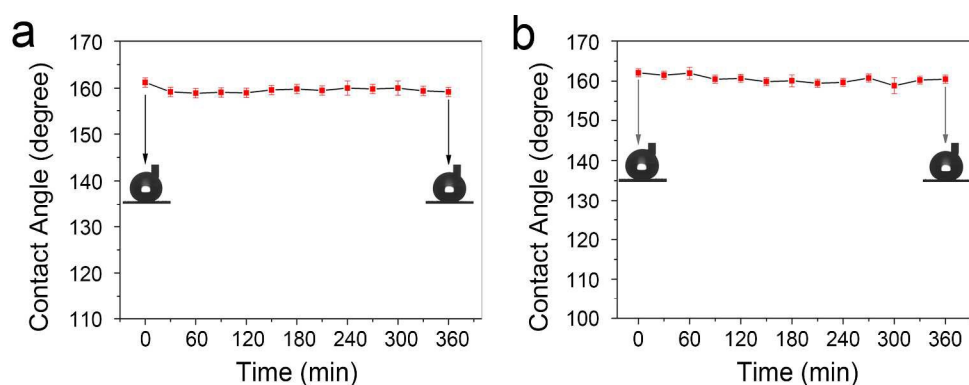


Fig. 5 Contact angle of superhydrophobic silicone as a function of immersion time in corrosive solution. (a) Alkali solution, (b) acidic solution.

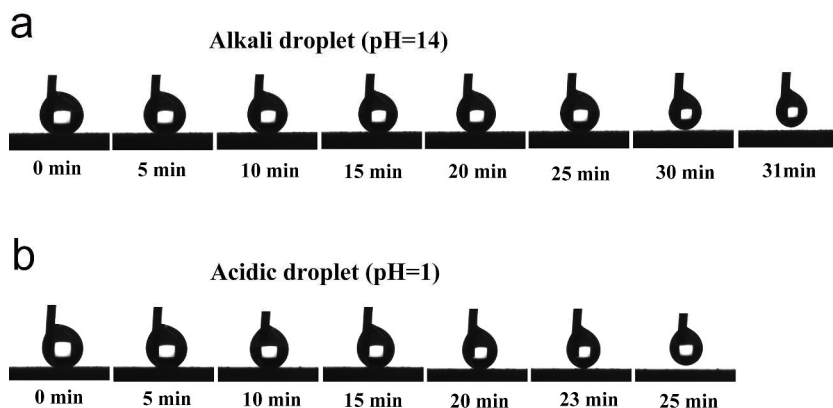


Fig. 6 Time evolution of contact angle for corrosive water at continuous contact with silicone surface. (a) Alkali droplet, (b) acidic droplet.

2.4 Self-cleaning properties of the superhydrophobic silicone

It is well known that superhydrophobic surfaces have good self-cleaning properties in air. When a water droplet falls on a superhydrophobic surface, it promptly rolls off the surface collecting dirt along the way. In this work, the obtained silicone also demonstrated good self-cleaning properties in air as shown in Supporting Information Movie 11. Very few reports have shown any self-cleaning tests in oil because the surface tension of the oil is lower than that of water, resulting in the oil penetrating into the surfaces and removing the trapped air layer.²⁸ Herein, the obtained silicone shows self-cleaning properties under oil. After being immersed in oil, the sample shows superhydrophobicity and spoils can be easily picked up from the silicone surface by the falling water droplets (Movie 12, Supporting Information). As shown in Figure 7, water droplets spread and wet the hydrophilic glass surface (right part in Figure 7 and Movie 13, Supporting Information). While water droplets sitting on the superhydrophobic silicone surface demonstrate spherical shapes with CA value of about 169° because of the support by both oil and surface structures. In this condition, the dirt-removal property in oil is similar to that in air.

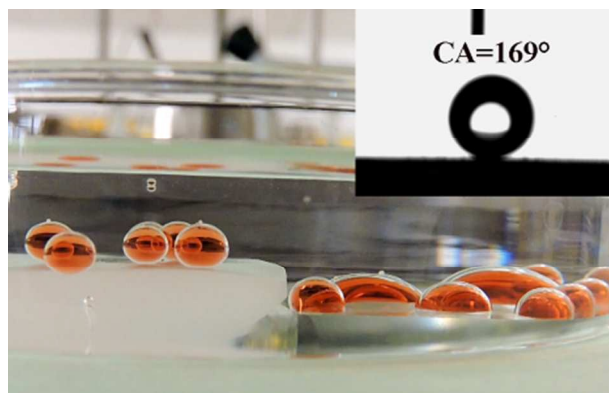


Fig. 7 Water droplets sitting on the superhydrophobic silicone surface under oil (left part) with CA value about 169° (inset) and on the areas of a glass slide (right part). Water droplets are dyed red for easy observation.

3. Conclusions

In conclusion, we have demonstrated a simple approach toward a bottom-up construction of three-dimensional silicone with flexible and mechanically robust superhydrophobicity. The elastic silicone shows a remarkable robustness to harsh environments such as strong corrosion, extremely low temperature, various external damages, especially high mechanical stress. The obtained superhydrophobic surface resists adhesion by blood and shows self-cleaning function when exposed to either air or oil. This large-scale fabrication of non-fluorinated super liquid-repellent silicone with a stable Cassie-Baxter state could yield a prospective candidate for various practical applications in real world.

4. Experimental Section

Hydrophobic SiO_2 nanoparticles and microparticles modified were synthesized in our lab. Laboratory solvents were purchased from Sinopharm Chemical Regent Co. Ltd. (Shanghai China). All chemicals were analytical grade reagents and were used as received.

Hydrophobic SiO_2 nanoparticles were prepared through a hydrolysis reaction. Briefly, 19.4 g of $\text{NaSiO}_3 \cdot 5\text{H}_2\text{O}$ was dissolved in 300 ml of deionized water, followed by the addition of 100 ml of 0.24 M NH_4Cl solution. The mixture solution was stirred for 3 h at 80°C . After that, the white product was filtered, washed with deionized water. The obtained white product and 0.05 mol of chlorotrimethylsilane (TMCS) were dissolved in 200 ml of alcohol. The resultant solution was heated

to 80°C and stirred for 3 h. After reaction, the resulting product was filtered and dried in vacuum at room temperature for 24 h to get the TMCS-functionalized SiO₂ nanoparticles.

Hydrophobic SiO₂ microparticles were also prepared through a hydrolysis reaction. 0.80 g of hexadecyltrimethylammonium bromide and 5.0 g of urea were dissolved in 15 ml distilled water. Then 3.0 g of trifunctional, 2.0 g of difunctional alkoxy silanes and 45 µl acetic acid were added and stirred at ambient temperature until the solution were homogeneous. The solution was heated at 80 °C for about 24 h for completely gelation and aging. The product was washed and dried to get the SiO₂ microparticles.

Fabrication of Robust superhydrophobic silicone monoliths and coatings: in a typical synthesis, 8 g polydimethylsiloxane (PDMS, Sylgard 184 Kit, Dow Corning) and a curing agent were mixed in a 10:1 (w/w) ratio. Then 1.5 g hydrophobic SiO₂ nanoparticles and 1.7 g SiO₂ microparticles was added to the PDMS mixture and stirred manually to get a uniform precursor. The precursor was pumped to get rid of the bubbles and pre-cured at 70°C for 2 h then at 210°C for 2 h. After curing, the sample was removed the uppermost layer by sandpaper and the superhydrophobic silicone monoliths was obtained. To fabricate silicone coating, brush-painting method was employed. A general paintbrush made of nylon fibrils was used to paint the silicone precursor on the substrates (Figure S8, Supporting Information), and the following experimental process was the same as that of silicone monoliths.

Chemical characterization: Surface morphology of the sample was examined by using a JSM-6701F field-emission scanning electron microscope (FESEM, JEOL, Japan). The chemical composition of the as-prepared sample was investigated using X-ray photoelectron spectroscopy (XPS), which was conducted on a PHI-5702 electron microscope. The static water contact angle (CA) and sliding angle (SA) were measured by a DSA100 contact angle instrument (Germany). The average CA and SA values were obtained by measuring the same sample in at least five different positions, and images were captured with a digital camera (Canon).

Pressure resistance characterization: in order to measure the pressure resistance of the elastic superhydrophobic surfaces, the sample was exposed between two smooth steel plates under a load. The load increased gradually from 0 to 1500 N within 5 min and maintains 1500 N for about 5 min. When the mechanical strain is released, the elastic silicone surface keep superhydrophobicity with a water CA of $154 \pm 1^\circ$ and SA of $7 \pm 1^\circ$.

Corrosion resistant characterization: two independent methods were used to study if the samples superhydrophobicity has degradation because of the exposure to strong acid or strong alkali. In the first method, the sample was soaked in strong acidic (pH = 1) and alkali (pH = 14) solution for a certain time, respectively. After a definite time of immersion, the sample was taken off the corrosive liquids, dried with a filter and retained at 80°C for 5 min. After that, a water droplet was dropped on the surface to investigate the influence of the corrosive solution on the wettability. In the second method, an acidic (pH = 1) or alkali (pH = 14) liquid droplet was dropped on the sample surface and the evolution of contact angle was studied as a function of time of corrosive droplet contact with the silicone. For adjusting pH value of the aqueous solutions, HCl and NaOH were used for acidic and alkali solutions, respectively.

Water droplet impact characterization: the stability of the superhydrophobicity was investigated through the long-term exposure to the falling of water droplets. The sample surface was tilted at 40° and the water droplets with weight of 0.38 g fell on the sample surface with a velocity of 1.4 m/s. After 6 h of falling impact, the sample still sustained its superhydrophobicity and no water droplets adhered to the sample surface.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

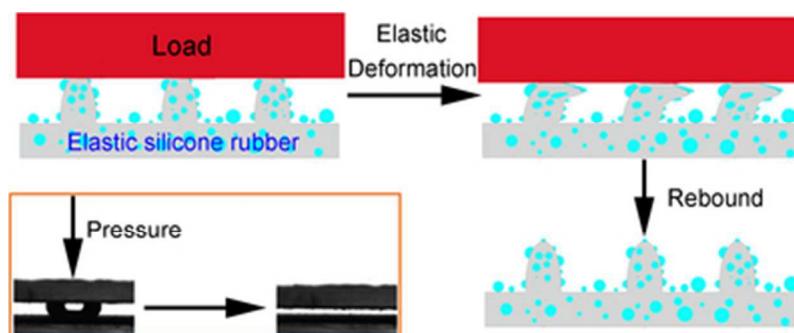
Acknowledgements

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Table of Contents

Here the authors report the use of elastic deformation to stabilize the necessary air cushions that are required to maintain durable superhydrophobic surfaces with a Cassie-Baxter state. The fabricated silicone surfaces displayed permanent superhydrophobicity in various harsh environments.