Video Article

Preparation and High-temperature Anti-adhesion Behavior of a Slippery Surface on Stainless Steel

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Abstract

Anti-adhesion surfaces with high-temperature resistance have a wide application potential in electrosurgical instruments, engines, and pipelines. A typical anti-wetting superhydrophobic surface easily fails when exposed to a high-temperature liquid. Recently, *Nepenthes*-inspired slippery surfaces demonstrated a new way to solve the adhesion problem. A lubricant layer on the slippery surface can act as a barrier between the repelled materials and the surface structure. However, the slippery surfaces in previous studies rarely showed high-temperature resistance. Here, we describe a protocol for the preparation of slippery surfaces with high-temperature resistance. A photolithography-assisted method was used to fabricate pillar structures on stainless steel. By functionalizing the surface with saline, a slippery surface was prepared by adding silicone oil. The prepared slippery surface maintained the anti-wetting property for water, even when the surface was heated to 300 °C. Also, the slippery surface exhibited great anti-adhesion effects on soft tissues at high temperatures. This type of slippery surface on stainless steel has applications in medical devices, mechanical equipment, *etc*.

Video Link

The video component of this article can be found at https://www.jove.com/video/55888/

Introduction

Anti-adhesion surfaces at high temperatures for use with liquids and soft tissues have received considerable interest because of their extensive application potential in electrosurgical instruments, engines, pipelines *etc.*[1,2,3,4]. Bioinspired surfaces, particularly superhydrophobic surfaces, are considered the ideal choice because of their excellent anti-wetting abilities and self-cleaning properties[5]. In superhydrophobic surfaces, the anti-wetting ability should be ascribed to the locked air in the surface structure. However, the superhydrophobic state is unstable because it is in the Cassie-Baxter state[6,7]. Also, at high temperatures, the anti-wetting for liquid droplets can fail because of the wetting state transition from the Cassie-Baxter to the Wenzel state[8]. This wetting transition is induced by small liquid droplet wetting in the structures, which results in the failure to lock the air in place.

Recently, inspired by the slippery properties of the peritome of the pitcher plant, *Nepenthes*, Wong *et al.* reported a concept to construct slippery surfaces by infusing a lubricant into the surface structures[9,10,11]. Due to capillary force, the structures can firmly hold the lubricant in place, just as in the locked air pocket on superhydrophobic surfaces. Thus, the lubricant and surface structures can form a stable solid/liquid surface. When the lubricant has a preferential affinity for the surface structure, the liquid droplet on the composite surface can slide easily, with only a very low contact angle hysteresis (*e.g.,* ~2°)[12]. This lubricant layer also enables the surface to have remarkable anti-wetting capabilities[13], demonstrating great potential for medical devices[14,15]. However, previous studies on slippery surfaces mainly focused on the preparation for application at room temperature or low temperatures. There are very few studies on the preparation of slippery surfaces with high-temperature resistance. For example, Zhang *et al.* showed that the rapid evaporation of lubricant rapidly causes the failure of the slippery property at even slightly high temperatures[16].

Slippery surfaces with high-temperature resistance can widen the application potential; for example, they can be used as liquid barriers to decrease soft tissue adhesion to electrosurgical instrument tips. During the surgical operation, severe soft tissue adhesion occurs because of the high temperature of the electrosurgical instrument tips. The soft tissue can be charred, causing it to adhere to the instrument tip, which then tears the soft tissue around the tip[17,18,19]. The adhered soft tissue on the electrosurgical instrument tip negatively influences the operation and also may induce the failure of hemostasis[19,20]. These effects significantly harm people's health and economic interest. Therefore, solving the issue of soft tissue adhesion to electrosurgical instruments is very urgent. In fact, slippery surfaces offer an opportunity to solve this problem.

Here, we present a protocol to fabricate slippery surfaces available at high temperatures. Stainless steel was selected as the surface material because of its high-temperature resistance. The stainless steel was roughened by photolithography-assisted chemical etching. Then, the surface was functionalized with a biocompatible material, saline octadecyltrichlorosilane (OTS)[21,22,23,24]. A slippery surface was prepared by adding silicone oil. These materials enabled the slippery surface to achieve high-temperature resistance. The anti-wetting property at high temperatures and the anti-adhesion effects on soft tissue were investigated. The results show the potential of using slippery surfaces to solve the anti-adhesion problem at high temperatures.

Protocol

1. Photolithography on Stainless Steel

1. Design the photomask using a mechanical drawing software and fabricate the design by submitting it to a photomask printer[4].
2. Wash the stainless steel (316 SS; lengthx width: 4 cm x 4 cm, thickness: 1 mm) by rinsing it in alkaline solutions (50 g/L NaOH and 40 g/L Na_2CO_3) at room temperature for 15 min to remove oil contaminants.
3. Thoroughly clean the stainless steel by performing ultrasonic cleaning in an ultrasonic cleaning machine (working frequency: 40 KHz, ultrasonic power: 500 W). Rinse it sequentially with deionized water, n-hexane, acetone, and ethanol for 10 min each.
4. Dry the stainless steel by placing it on a hot plate at 150 °C for 30 min. Protect the stainless steel by covering it with a sheet of aluminum (Al) foil.
5. Place the stainless steel on the center of a spin coater. Use a dropper to deposit positive photoresist (about 1 mL) onto the stainless steel, from the center to the edge, until the photoresist completely covers the stainless steel. Avoid bubble formation in the photoresist.
    1. Perform spin-coating, first with a speed of 700 rpm/min for 6 s, to start the spin cycle, and then with a speed of 1,500 rpm/min for 15 s, to evenly spread the photoresist.
6. Release the vacuum valve and retrieve the stainless steel using a pair of tweezers. Place the stainless steel on a hot plate at 120 °C for 2 min to bake the photoresist.
7. Place the stainless steel on the vacuum valve of a photolithography machine. Set the exposure time of the photolithography machine to 25 s.
NOTE: Here, the photolithography machine is a contact aligner with an ultraviolet (UV) light wavelength of 254 nm and a light intensity of 13 mW/cm^2.
8. Release the stainless steel and place it in the developer solution for 1 min to remove the photoresist without exposing it to the UV light. Remove the stainless steel from the developer solution, wash it with deionized water, and dry it under N_2 gas.
9. Place the stainless steel on a hot plate to bake at 120 °C for 2 min.
10. Use an upright microscope with a magnification of 100x to observe the surface of the stainless steel to inspect the obtained photoresist texture.

2. Chemical Etching of Stainless Steel

1. Prepare a chemical etching solution with a volume of 200 mL (400 g/L $FeCl_3$, 20 g/L phosphoric acid, and 100 g/L hydrochloric acid) in a 500-mL beaker.
2. Place the stainless steel with photoresist texture in the chemical solution for 10 min. Do not allow the stainless-steel pieces to contact each other. Place a maximum of four stainless steel pieces at one time.
3. Take out of the chemically etched stainless steel using tweezers, wash the pieces with deionized water for 1 min, and dry them with N_2 gas.
4. Remove the photoresist texture by submerging the stainless steel in acetone for ultrasonic cleaning for 5 min. Then, dry the chemically etched stainless steel with N_2 gas.

3. OTS Self-assembly on Chemically Etched Stainless Steel

1. Clean the chemically etched stainless steel with a steady stream of deionized water, dry it with N_2 gas, and place it on a hot plate at 100 °C for 30 min to completely dry the surface.
2. Hydroxylate the chemically etched stainless steel with an O_2 plasma treatment in an RF plasma machine, with an RF power of 100 W for 10 min, a system pressure of 100 mbar, and a flow rate of 20 sccm.
3. Prepare 1 mM OTS solution in anhydrous toluene in a beaker. Dry the beaker thoroughly before solution preparation.
4. Rinse the chemically etched stainless steel with the OTS solution for 4 h at room temperature. Place the beaker in a sealed bag. Do not allow the stainless steel pieces to contact each other.
5. Remove the stainless steel, clean it with anhydrous toluene by performing ultrasonic cleaning for 10 min, and dry it with N_2 gas.

4. Slippery Surface Preparation

1. Deposit approximately 10 mL/cm^2 silicone oil (viscosity: 350 cst; surface tension: 21.1 mN/m) onto the OTS-coated, chemically etched stainless steel using a dropper.
2. Use an optical stereomicroscope to observe the wetting process of the silicone oil on the stainless-steel surface (magnification of 10x).
3. Remove the excess silicone oil by placing the stainless steel in a vertical position for 1 h.

5. Investigation of Water Sliding Behavior on Slippery Surfaces

1. Deposit a 4-μL water droplet on the slippery surface. Place the stainless steel under an optical microscope and tilt the substrate by ~2°.
2. Visualize the water droplet sliding on the slippery surface at a low magnification (50x) to check that the slippery surface has the easy sliding property.

6. Analysis of Anti-wetting on the Slippery Surface at High Temperatures

1. Place the stainless steel with a slippery surface on a hot plate using tweezers. Set the hot plate at different high temperatures (*i.e.,* 200 °C, 250 °C, and 300 °C) to analyze the anti-wetting behaviors at different temperatures.
   NOTE: Do not directly touch the high-temperature stainless steel with hands.
2. Use a micro-syringe to deposit a 10-µL water droplet on the slippery surface.
   NOTE: Before dropping the water droplet, the temperature of the slippery surface should reach equilibrium.
3. Use a high-speed camera to record the water droplet movement at a frame rate of 500 Hz.
   1. Fix the camera to a tripod and direct the lens of the camera toward the stainless steel. Adjust the focus of the camera to obtain a clear water droplet image. Record the movement of the water droplet on the stainless-steel surface by pushing the start button of the camera. Push the end button of the camera when the water droplet slides off the stainless steel to complete the recording.

7. Analysis of the Anti-adhesion Effects of the Slippery Surface on Soft Tissue

1. Use a manipulator, a dynamometer, a hot plate, and a stationary fixture to set up an adhesion force measurement platform[4], as shown in **Figure 3a**.
2. Place the test surface on the hot plate. Use a clamp to fix the stainless steel on the plate. Heat the test surface to a certain high temperature (*e.g.,* 300 °C).
   NOTE: The test surface should closely contact the hot plate to ensure efficient heat transport to the slippery surface.
3. Fix the dynamometer to the manipulator. Connect a cylinder table (diameter: 2 cm) with a force head to act as a soft tissue fixed platform.
4. Fix the soft tissue (*e.g.,* chicken breast; length: 5 cm, width: 2 cm, thickness: 3 mm) onto the cylinder table using a thin wire. Ensure that the soft tissue surface is approximately even.
5. Load the soft tissue onto the test surface at a speed of 1 mm/s until the dynamometer reaches a certain maximum force (*e.g.,* 4.5 N) by rotating the motion button of the manipulator. Then, unload the soft tissue at the same speed.
6. Connect a computer to the dynamometer using a data transmission line and record the real-time force between the soft tissue and the test surface.

Representative Results

The slippery surface was prepared by adding silicone oil to OTS-coated, chemically etched stainless steel. Due to their similar chemical properties, the surface was completely wetted by silicone oil. The wetting process is shown in **Figure 1a**. The red dotted line marks the wetting line. After the wetting, a visible oil layer could be distinguished from the dry surface. The slippery property of the prepared slippery surface was investigated by depositing a water droplet on the slippery surface with an angle of approximately 2°. **Figure 1b** shows the *in situ* water droplet movement over the slippery surface. The yellow dotted line marks the contact line, and the results show the water droplet floating and sliding on the slippery surface.

The anti-wetting behaviors of the prepared slippery surface on a water droplet at high temperatures was investigated. The slippery surface was heated to different temperatures and water droplets were deposited on the surface. At 200 °C (**Figure 2a**), the water droplet first firmly contacted the surface, and then the contact area between the droplet and the surface decreased. After about 6,200 ms, the water droplet began to slide off the surface. At 250 °C (**Figure 2b**), the water droplet had a very small initial contact area with the surface. After about 800 ms, the water droplet began to slide off the surface. At 300 °C (**Figure 2c**), the water droplet had unstable contact immediately after being deposited and rapidly slid off the slippery surface after just 250 ms.

The anti-adhesion effect of the slippery surface on a soft tissue was evaluated by measuring the adhesion force. We set up an adhesion force measurement platform by combining the heating and manipulation systems (**Figure 3a**). The soft tissue was fixed onto the dynamometer, which was connected to the manipulator, and the test surface was fixed on a hot plate. Chicken breast was selected as the representative because of its pure tissue. After loading the soft tissue on the test surface at a pressure of 4.5 N, the unloading process generated an adhesion force between the soft tissue and test surface. The results are shown in **Figure 3b**. The adhesion forces were 0.80 ± 0.18 N and 0.04 ± 0.02 N on the smooth stainless-steel and slippery surfaces, respectively. The adhesion force decreased by an order of magnitude on the slippery surface compared to that on the smooth stainless-steel surface.

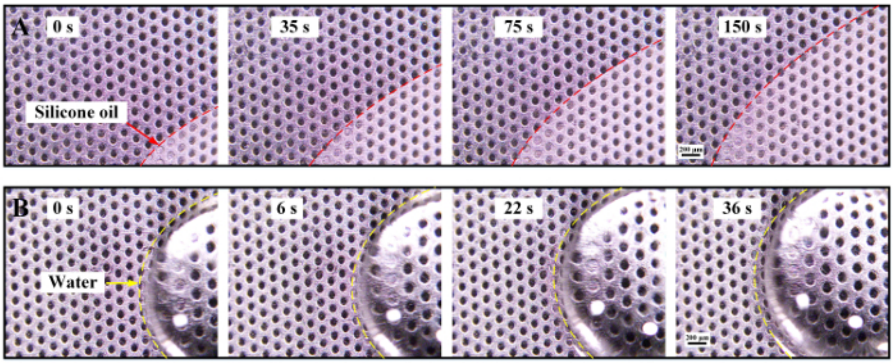

Figure 1. Formation process of the slippery surface and its slippery property. (**A**) Wetting process of silicone oil on the OTS-coated, chemically etched stainless steel. The surface can be completely wetted by the silicone oil due to similar chemical properties between the OTS molecular layer and the silicone oil. (**B**) Water droplet floating on silicone oil and showing its easy-sliding property. The stainless steel has a tilt angle of approximately 2°. Please click here to view a larger version of this figure.

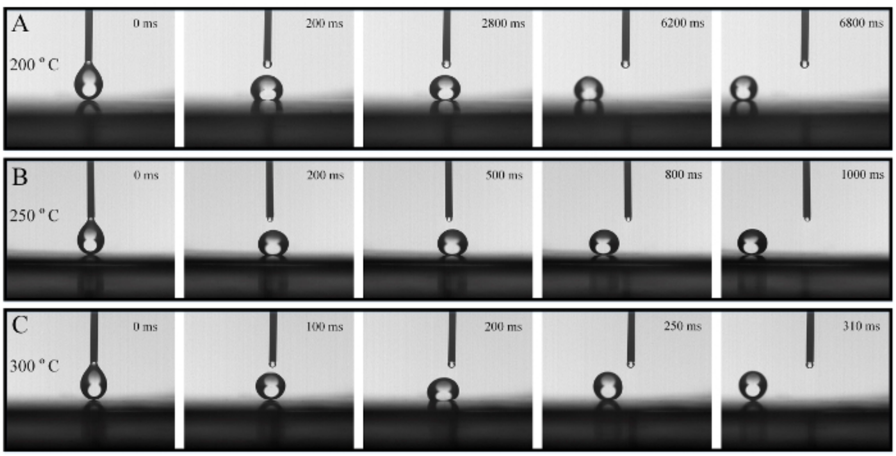

Figure 2. Anti-wetting behavior of the slippery surface with a water droplet at high temperatures. Water droplet movement after being deposited on a horizontal slippery surface at different high temperatures: (**A**) 200 °C, (**B**) 250 °C, and (**C**) 300 °C. All the water droplets slid off the slippery surface after a certain time, and the necessary time for the water droplet to slide away decreased with increasing surface temperature. Please click here to view a larger version of this figure.

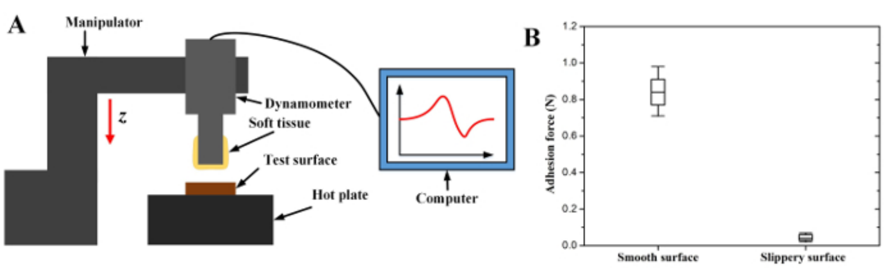

Figure 3. Anti-adhesion evaluation of the slippery surface with a soft tissue at high temperatures. (**A**) Schematic of the adhesion force measurement platform. Soft tissue was loaded on the test surface using a manipulator connected to a dynamometer. The adhesion force was transmitted to the computer. (**B**) Adhesion force between the soft tissue and the test surface. Soft tissue was loaded on the test surface at a surface temperature of 300 °C. The adhesion force on the slippery surface was decreased by about an order of magnitude compared to that on the smooth stainless-steel surface. The error bars shown are the average standard deviations (SD). Please click here to view a larger version of this figure.

Discussion

This manuscript details protocols for fabricating a slippery surface with high-temperature resistance. The slippery property of our prepared surface was demonstrated by observing the easy-sliding behavior of a water droplet. Then, the anti-wetting of the prepared slippery surface at different high temperatures was investigated by depositing a water droplet on the hot surface. The results show that the prepared slippery surface maintained its slippery property even when it was heated to above 300 °C. We also determined the anti-adhesion effects of the slippery surface on soft tissue.

Unlike the superhydrophobic surface, the surface structures on the slippery surface act as the holding structures for the infused lubricant. According to a previous study[25], because of the preferential affinity of the OTS-coated surface for the silicone oil, a water droplet would float on the silicone oil-infused surface structure, as demonstrated in **Figure 1B**. Besides, this liquid/liquid/solid interface gives the surface a very low contact angle hysteresis for liquid droplets immiscible with silicone oil. Therefore, the water droplet could easily slide on the as-prepared slippery surface.

Because of the excellent high-temperature resistance of the stainless-steel substrate, functionalized layer OTS, and infused silicone oil, the prepared slippery surface can maintain its slippery property at very high temperatures. However, at high temperatures, the water droplet does not slide on the surface, but it can roll on the surface. The results can be ascribed to the Leidenfrost effect[26]. At high temperatures, the silicone oil and water evaporate, and the vapor can form a vapor layer between the water droplet and the silicone oil layer. In fact, evaporation of the silicone oil and water droplet increase with increasing temperature. Therefore, the air layer at higher temperatures has an improved ability to prevent direct contact between the water droplet and the silicone oil. Similar to the water droplet rolling on the surface at 300 °C (**Figure 2C**), the water droplet almost floated on the air layer. The contact was very unstable, and thus it rapidly slid off the surface.

The lubricant layer can also act as an anti-adhesion barrier for soft tissue. An adhesion force measurement platform was set up to investigate the anti-adhesion effect of the slippery surface on soft tissue. Due to the pure tissue, the chicken breast was selected as the experimental soft tissue. The soft tissue was loaded on the smooth stainless-steel surface and on the slippery surface. The results demonstrate a significant decrease of adhesion force on the slippery surface (*i.e.,* from 0.80 ± 0.18 N on the smooth surface to 0.04 ± 0.02 N on the slippery surface). This concept offers new insights into solving the soft tissue adhesion problem on electrosurgical instruments. Because silicone oil and the OTS are biocompatible[22,27], our method can be applied to electrosurgical instruments, including the monopolar scalpel and ultrasonic scalpel.

In addition, our method is very simple, and it can be simplified further. The pillar structure enables the surface to hold more silicone oil, and more silicone oil can efficiently act as a barrier for soft tissue. However, if there is no need for so much silicone oil, such as when it used for the anti-wetting of a water droplet, the stainless steel can be directly roughened by chemical etching. The simplified method is simpler and can be applied to different surface types, including a curved surface. It should be noted that silicone oil will evaporate when the surface is heated to a high temperature, and the slippery property will finally fail after a certain time. But by adding silicone oil to the surface, it will regain the slippery property. The critical step of our protocol is the preparation of OTS coatings on the surface structures, which determines the final slippery property of the slippery surface. Thus, the OTS assembly step should be carefully performed.

Slippery surfaces are an emerging functional surface to achieve self-cleaning, anti-adhesion, anti-icing, *etc.* It has many advantages, including easy fabrication, robust repellence for different liquids, good pressure stability, and self-healing. Our straightforward method offers a way to construct a slippery surface with high-temperature resistance. We believe that the proposed method will enable slippery surface application in medical devices, engines, hot-water pipelines *etc.*

Disclosures

The authors have nothing to disclose.

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