

Mammalian Cornea Inspired Anti-Fogging Silica Glass Surface Achieved by Femtosecond Laser

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Fog generation can severely damage optical systems by degrading the light absorption rate and imaging quality of optical components. Furthermore, fog can reduce the light flux and transmittance of the optical system, resulting in poor imaging clarity and contrast. Studies have focused on minimizing fog formation and effects. Drawbacks such as high energy consumption and waste pollution severely limit the application of conventional methods. However, achieving high fog resistance of optical components remains a challenge. A novel method of fabricating anti-fogging slippery surfaces (inspired by the anti-fog mechanism of the mammalian cornea) on silica glass by using femtosecond lasers is proposed to achieve durable and environment-friendly optical devices that can achieve anti-fogging in real time. The femtosecond laser wet etching method is used to fabricate the inside cabin of glass. The cabin filled with graphene spontaneously heats the sample under sunlight to prevent fog formation. In addition to exhibiting excellent anti-fogging characteristics, the prepared sample achieves high optical transmittance, high durability, and excellent self-repair capability. Thus, the proposed method exhibits considerable potential for application in numerous domains.

windshields, rearview mirrors, and other equipment can result in catastrophic traffic accidents that can endanger human safety.^[5,6] Furthermore, fog formation impairs the light absorption rate and imaging quality of optical components^[7,8] and generates microscopic droplets on the surface of optical materials, magnifying the reflection of their surfaces and diminishing their clarity drastically. In chilly and humid conditions, fog can freeze into frost and completely block light from radiating onto the surface.^[9–11] Such conditions not only cause glare and ghosting, which adversely affects the normal operations of lenses, lasers, displays, and other optical components, but also reduces light flux in the optical system and its transmittance, which can limit imaging clarity and contrast.^[12–14] Fogging always occurs when the surface temperature of the substrate is lower than the dew point of ambient water vapor. Thus, water vapor condenses

1. Introduction

Pervasive fogging and frosting considerably decrease the light transmission of transparent materials and severely degrade the performance of optical devices.^[1–3] For example, fogging on laparoscopes, laryngeal lenses, or medical protective apparel eyewear interferes with the operation of optical instruments. Fogging on solar panels and agricultural greenhouse films reduces the efficiency of light energy use, which causes considerable economic losses.^[4] The fogging of aircrafts and car

into tiny droplets on the surface of the substrate when reaching saturation or supersaturation in air.^[15–17]

Substrate surface fogging can be attributed to the following two factors. The first factor is environmental factors. When the temperature of the solid substrate surface is lower than the dew point of the water vapor under a given humidity, water vapor in air condenses into tiny water droplets, known as fog droplets, because of external water vapor and temperature variations. The second factor is the inherent nature of the material; the surface tension between the gas, liquid, and solid phases can determine vapor formation.^[18] The surface tension of the matrix material is primarily determined by the intermolecular force of the surface. The greater the intermolecular force is, the higher the surface tension of the matrix is.

The eyes of warm-blooded animals hardly fog even under extreme environments. For example, in human eyes, the cornea is an optical organ that performs observation and imaging activities. Human eyes do not fog even under some extreme circumstances because of two factors. The first cause is that eyes always maintain a steady temperature of $\approx 37^\circ\text{C}$, which renders eyes warmer than the surrounding temperature. Therefore, the temperature difference does not reach atomization, and the condensation of water droplets is inhibited. Furthermore, tears constantly wet the cornea from time to time, which prevents the generation of fog droplets.

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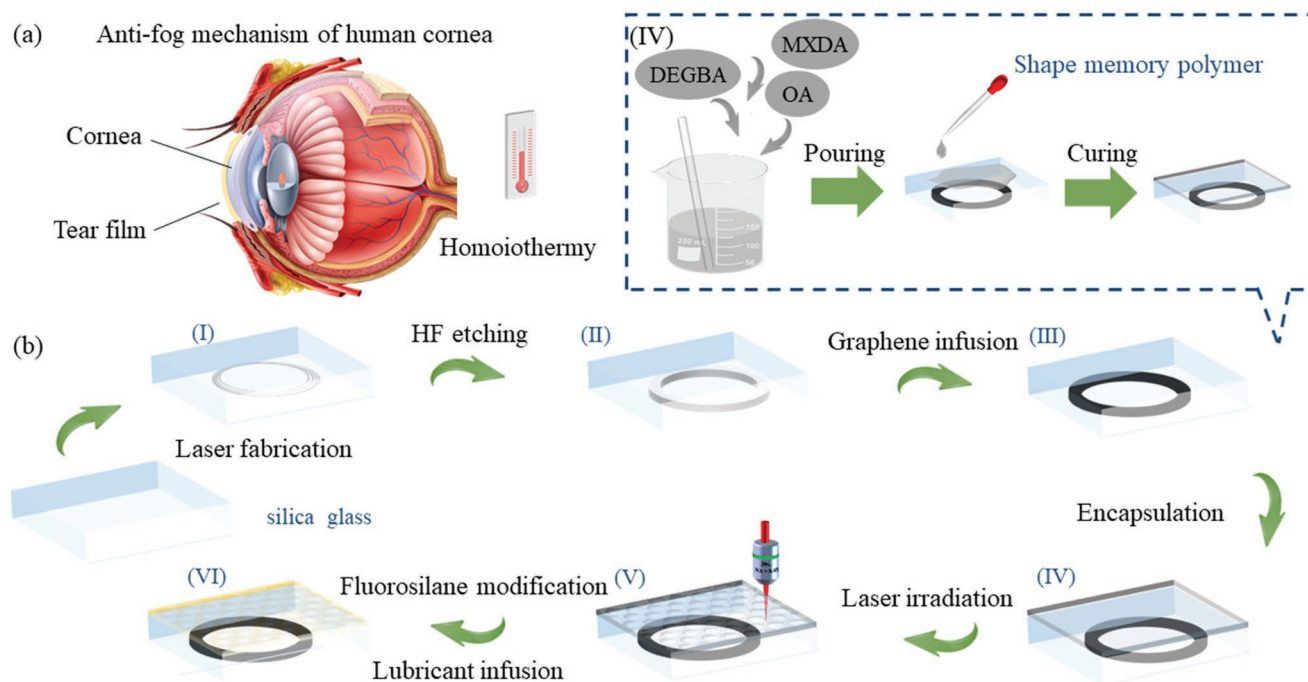


Figure 1. a) Anti-fog principle of the cornea. b) Fabrication process of the anti-fogging slippery surface (SLIPS).

Compared with the anti-fogging mechanism of mammalian eyes, existing anti-fogging methods are limited to some extent. Current anti-fog strategies are either physical or chemical. In physical anti-fog methods, ambient conditions are altered to increase the surface temperature of the material above the dew point of water vapor, which eliminates surface fog. However, these techniques are expensive and difficult to implement for mass manufacturing.^[26] Chemical anti-fog strategies comprise external anti-fog coating and interior anti-fog agent addition. Typically, the surface tension of water is reduced through hydrophilic functional groups. When the contact angle is $<40\text{--}50^\circ$, the surface can achieve a distinct anti-fog effect. When the static liquid contact angle on the surface of the solid material is $<5^\circ$, the surface exhibits a superhydrophilic state, and the liquid spreads on the surface of the substrate material to form a water film layer, which prevents light refraction and scattering to achieve the anti-fog effect.^[19–25] Guo et al. grafted poly(*N*-vinylpyrrolidone-*co*-maleic anhydride) (poly(NVP-*co*-MA)) copolymer on glass slides to develop a multifunctional coating with excellent anti-fogging, self-cleaning, and antibacterial properties.^[14] Ezzat et al. used surface-initiated atom transfer radical polymerization to create polymer brushes coatings based on two zwitterionic analogues, namely poly(sulfobetaine methacrylate) (pSBMA) and poly(sulfobetaine vinylimidazole) (pSBVI) (SI-ATRP).^[12] Jiang et al. proposed a novel and effective organic–inorganic hybrid hydrophilic anti-fog coating. Polyethylene glycol with two molecular weights was used to modify the si-lane coupling agent and was blended with hydrophilic copolymers to generate coating formulations. Polycarbonate (PC) substrates coated with these formulas function as the measurement sample.^[23] Such hydrophilic approaches achieve basic anti-fogging capability. However, in these methods, contaminants can reduce the service life of surfaces.

To solve existing problems, a novel anti-fogging slippery surface (SLIPS) that imitates mammalian cornea was manufactured. A femtosecond laser wet etching method was proposed to fabricate the cabin inside the glass samples to store reduced graphene (RGO), which endows the surface with self-heating capabilities under sunlight. Shape memory polymer (SMP) was used as the encapsulating material. Slippery surfaces were processed on SMP. The prepared transparent anti-fog samples could maintain a temperature of $35\text{ to }50^\circ\text{C}$ under one solar light intensity. This constant temperature ensured that the temperature of the sample remained consistently higher than that of the room temperature, which prevented fog formation. Thus, SMP slippery surfaces provide self-healing, durability, and pollution avoidance properties.^[27–32] Damaged to the surface is self-repaired when heating the sample to a certain temperature. After the high- and low-temperature cycle experiments and the chemical reagent soaking test, the sample maintained excellent slippery performance. The sample retained the transparency and transmittance of quartz glass. Thus, the proposed method can be widely used in optical components typically used in medicine, energy, and optical fields.

2. Results and Discussions

2.1. Design and Fabrication of the Anti-Fogging Slippery Surface

The eyes of warm-blooded animals do not fog even under the extreme environments. The anti-fogging mechanism is displayed in **Figure 1a**. Homoiothermy ensures that the cornea is at a higher temperature than that of the surrounding environment, and tears constantly wet the cornea. These phenomena impede fog formation. Inspired by this anti-fogging capability

of the cornea of warm-blooded animals, an anti-fogging slippery surface was fabricated. Silica glass was used as the substrate for preparing the anti-fog slippery surface because of its excellent heat resistance, high spectral projection, and high hardness. The femtosecond laser wet etching method was proposed to fabricate the cabin inside silica glass. Silica glass was infused with GO to achieve self-heating ability. Next, SMP was used as the encapsulation material, which added self-healing to the surface. Next, the femtosecond laser was used to construct porous structures on the surface. The anti-fogging slippery surface inspired by mammalian cornea was designed following fluorosilane modification and lubricant infusion.

2.2. Transmittance of the Anti-Fog Slippery Surface

Transparency is an essential characteristic of fused silica glass. However, micronano porous structures created by laser processing on the surface cause light scattering and refraction, which considerably decreases the transparency and transmittance of the sample. The infused lubrication fills into the porous structure and smoothens the surface, which reduces scattered light and improves transmittance, as displayed in Figure 2c.^[33] However, the promotion effect of the lubricant

on transmittance is limited. Only samples with certain specifications achieve a superior transmittance after lubricant infusion. The transmittance of samples with various processing parameters was measured before and after lubricant infusion, and the results are displayed in Figures 2a,b. The results revealed that the sample fabricated at a faster speed had a higher transmittance. Transmittance could reach up to 90% after lubricant infusion. The transmittance of the sample manufactured at 2000 m s^{-1} was essentially unchanged after lubricant infusion. This phenomenon could be attributed to the slow scanning speed producing considerable ablation on the sample surface, which could blacken the surface. The lubricant could not change the ultralow optical transmittance of the black area because it was caused by the light absorbent. Figure 2d–f displays the surface morphology after fabrication by 2000, 6000, and $10\,000 \text{ } \mu\text{m s}^{-1}$. The samples were filled with a porous structure. The water contact angle before (135°) and after (67°) lubricant infusion of the sample fabricated with $10\,000 \text{ } \mu\text{m s}^{-1}$ were measured, as displayed in Figure 2g. The sliding property is displayed in Figure 2h. Finally, for its outstanding optical transmittance, excellent porous structure, and slippery feature, a speed of $10\,000 \text{ } \mu\text{m s}^{-1}$ was optimal for manufacturing the sample.

Figure 3 displays that the transparency of the sample was at each step with fused silica as a reference sample. Because no

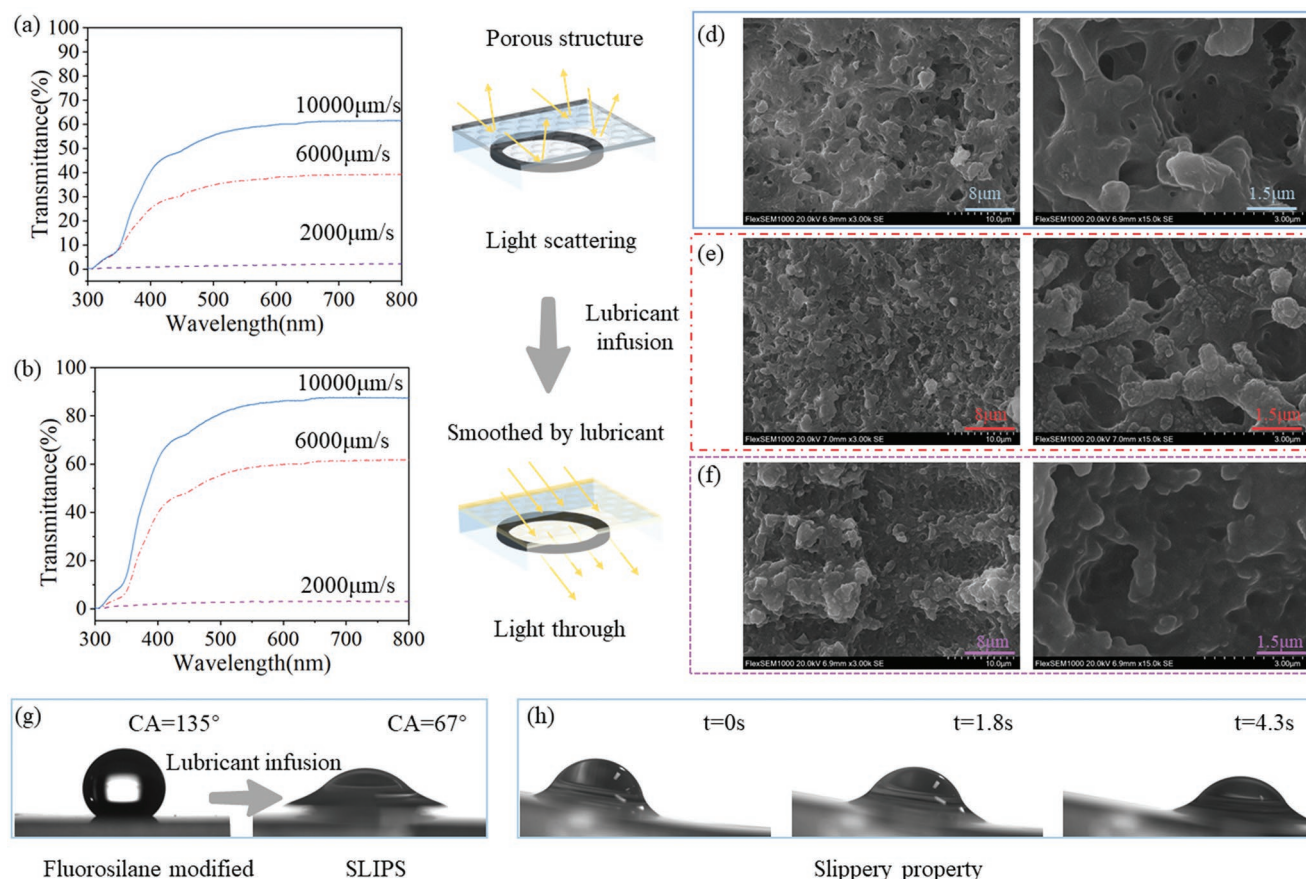


Figure 2. a) Transmittance of samples manufactured by various parameters before lubricant infusion. b) Transmittance of samples produced by various parameters after lubricant infusion. c) Illustration of variations in transmittance after lubricant infusion. d–f) Scanning electron microscopy images of the samples fabricated at 2000, 6000, and $10\,000 \text{ } \mu\text{m s}^{-1}$. g) Changes in the water contact angle on the sample fabricated by $10\,000 \text{ } \mu\text{m s}^{-1}$ after lubricant infusion. h) Water sliding behavior on the sample fabricated by $10\,000 \text{ } \mu\text{m s}^{-1}$ after lubricant infusion.

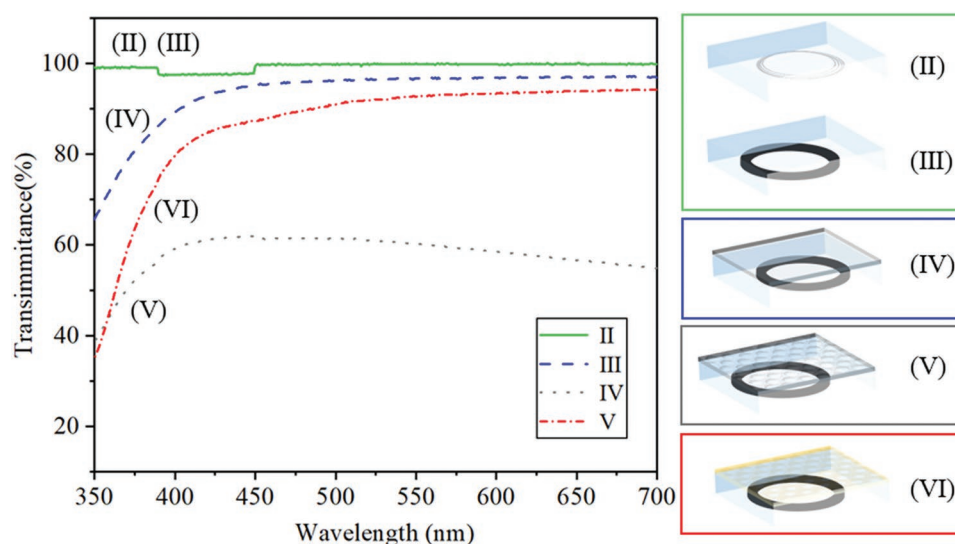


Figure 3. Transmittance of the sample at each stage.

structure was observed on the center of the sample, the transmittance of the center of the sample barely changed after the storage cabin was constructed (Stage I). The SMP covered on the surface (Stage IV) exhibited a negligible effect on transmittance because of SMP transparency. However, the porous structure on the SMP surface (Stage IV) could considerably affect transmittance. Under these conditions, light is scattered and refracted by the micronano structure, which drastically reduces the amount of light that is available. Consequently, transmittance is reduced considerably. Because the refraction index of the lubricant and the fused silica is almost identical to that of air and fused silica, the decreased transmittance could recover after lubricant infusion. Similar refraction indices result in low scattering and refraction, which improves transmittance and eventually reaches 90%. The final sample exhibits excellent transparency and transmittance, rendering the sample suitable for use in numerous optical components.

2.3. Photothermal Characteristics and Thermal Mechanical Property

Mammalian eyes do not fog under cold or hot environment because of several reasons. The temperature remaining constant at $\approx 37^\circ\text{C}$ is the primary cause of homeiothermy. This characteristic prevents eyes from reaching the fogging criteria because they remain at a higher temperature than surrounding air. The prepared anti-fog SLIPS mimicked photothermal response capabilities of mammalian homeiothermy. Graphene is a popular photothermal material because of its exceptional light-absorbing and heat-conducting properties. Here, graphene was infused into the anti-fog cabin (with the width of 2 mm) of SLIPS after mixing with silicon oil in various weight ratios. **Figures 4a** illustrates the temperature change of the bare glass and the samples infused with various densities of graphene. Bare glass and the anti-fog SLIPS with various RGO densities were then placed under a xenon lamp and exposed

to simulated solar light at a light intensity of $\approx 100\text{ mW cm}^{-2}$ to simulate the intensity of 1 sun. The surface temperature of the samples was observed to steadily increase when the irradiation time was increased from 0 to 180 s. The maximum temperature increased from room temperature (23.5°C) to 38.2 , 41.3 , 43.5 , 45.1 , 49.2 , and 53.3°C for samples with the RGO content of 0.1, 0.3, 0.5, 0.7, 1.0, and 2.0 wt%, respectively. By contrast, under the same irradiation duration, the temperature of the bare glass could only increase to $\approx 27.4^\circ\text{C}$. A higher density at the same irradiation rates will result in a greater increase in the temperature. Additionally, compared with the contrast sample, the temperature of prepared samples increased at a faster rate. This phenomenon indicated that the response speed to sunlight irradiation was considerably improved by RGO addition. During the subsequent experiment, the sample with 2.0 wt% RGO/silicon oil in the cabin was selected. **Figure 4c** displays the infrared thermal images of sample with 2 wt% RGO and the bare glass at 10 and 120 s under the xenon lamp. These images could be a direct reflection of the two significant temperature differences of the two samples. The prepared sample could be heated not only under sunlight but also heated under near-infrared (NIR) light. The infrared thermal image of a sample irradiated by the NIR light is displayed in **Figure S3**, Supporting Information. On this basis, the capacity to sustain the heated temperature in a dark environment was examined. As displayed in **Figure 4b**, the sample with 2 wt% RGO and bare glass were exposed to xenon lamp radiation at a 100 mW cm^{-2} for 300 s, after which the lamp was turned off and the temperature was monitored until the temperatures of the sample returned to room temperature. The results revealed that the sample containing RGO achieved a superior insulating property, which reflected a slower decline speed, and could ensure the temperature remained above 30°C for $\approx 200\text{ s}$ without irradiation. The prepared sample could constantly maintain a higher or similar temperature to the surrounding environment because of the superior heating and insulating performance, which effectively prohibited fog formation. The influence of the cabin size of the

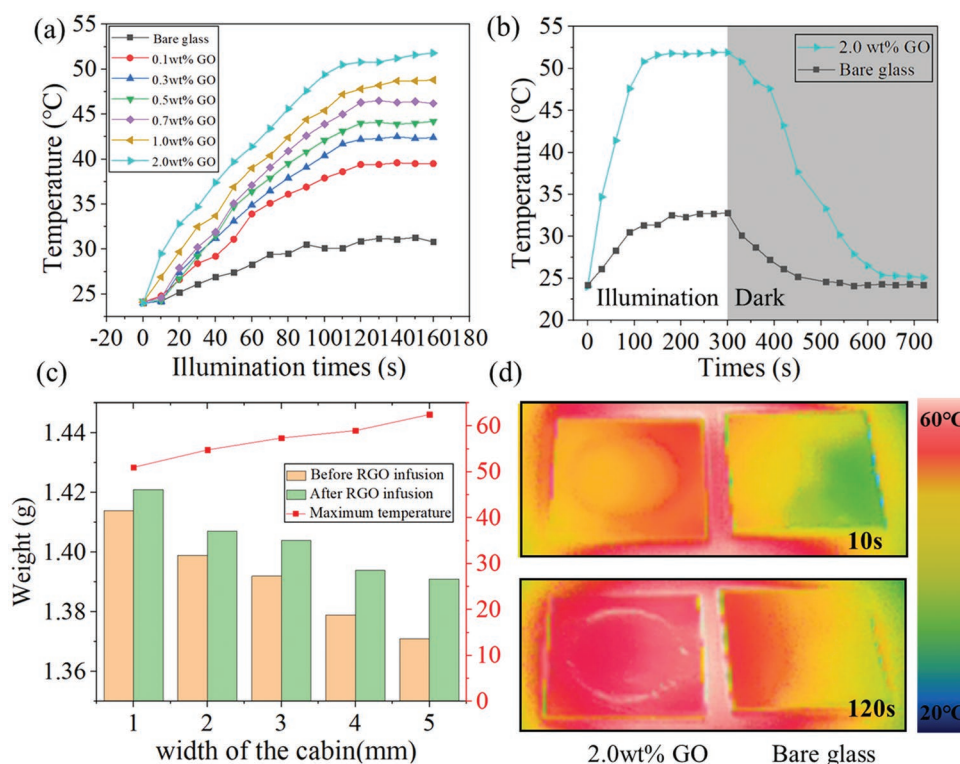


Figure 4. a) Variation in the temperature of bare glass and the samples infused with various density of GO/silicon oil liquid under xenon lamp of $\approx 100 \text{ mW cm}^{-2}$. b) Temperature change of bare glass and prepared sample with 2.0 wt% GO/silicon oil liquid under illumination and in the dark environment. c) Weight change of samples with various cabins before and after RGO infusion. Maximum temperature of these samples under sunlight irradiation. d) Infrared thermography images of the finally used sample.

temperature was calculated, as displayed in Figure 4c. Cabins with widths ranging from 1 to 5 mm were prepared. The weight change of these samples before and after RGO infusion was tested, and the results revealed that wider widths allow more RGO storage. Thus, this phenomenon could lead to a higher maximum temperature under the same sunlight irradiation. However, the difference in the maximum temperature is only 5 °C, and the sample with a small width exhibits a larger view window. Therefore, the sample with the width of 2 mm was used as the tested sample.

2.4. Anti-Fog Property

The anti-fogging ability of the sample was evaluated experimentally. The fabricated anti-fog SLIPS and bare glass, used as the comparison sample, were exposed to xenon light for $\approx 2 \text{ min}$ at an intensity of nearly 100 mW cm^{-2} . Next, as displayed in Figure 5a, both samples were positioned 10 cm above a beaker of boiling water. Figure 5b displays the fogging conditions of two samples. Negligible fog was created on the prepared anti-fog SLIPS (sample A), and the letters “xjtu” were plainly visible. Additionally, no change was observed in the transmittance of the anti-fog SLIPS, as displayed in Figure 5d. By contrast, fog immediately covered bare glass (sample B), and letters “xjtu” were scarcely discernible. Furthermore, a sharp reduction in transmittance, which decreased to less than 60% was

observed. A simple light path was constructed to discern the fogging situation of the two samples directly, as displayed in Figure 5c. A 5 mW laser pen with a wavelength of 532 nm was used to generate a laser beam, which was diffracted into circular spots after passing through an aperture. The circular spots would then pass across samples A and B. The final spots and the initial spot are displayed in Figure 5c. The circular spot after passing sample A exhibited minimal shift, and no fog was observed on the surface of sample A. When the light spot passed through sample B, its surface was completely covered with fog and condensed water droplets. These droplets and fog scatter and refract light, which result in spot deformation.

2.5. Slippery Property and Durability

With the exception of the significant anti-fog property, the prepared sample also exhibited a slippery characteristic. As displayed in Figure 6, various liquids, including milk, juice, hydrochloric acid (HCl, PH = 1), sodium hydroxide (NaOH) solution (PH = 13), and sodium chloride (NaCl) solution (3 wt%) could slide down off the surface rather than adhering to the sample.

Because of the SMP layer on the surface, the fabricated anti-fog SLIPS exhibited a remarkable self-repair ability, as displayed in Figure 7a. The sliding behavior of the droplet before and after the recovery of the surface is displayed in Figure 7a.

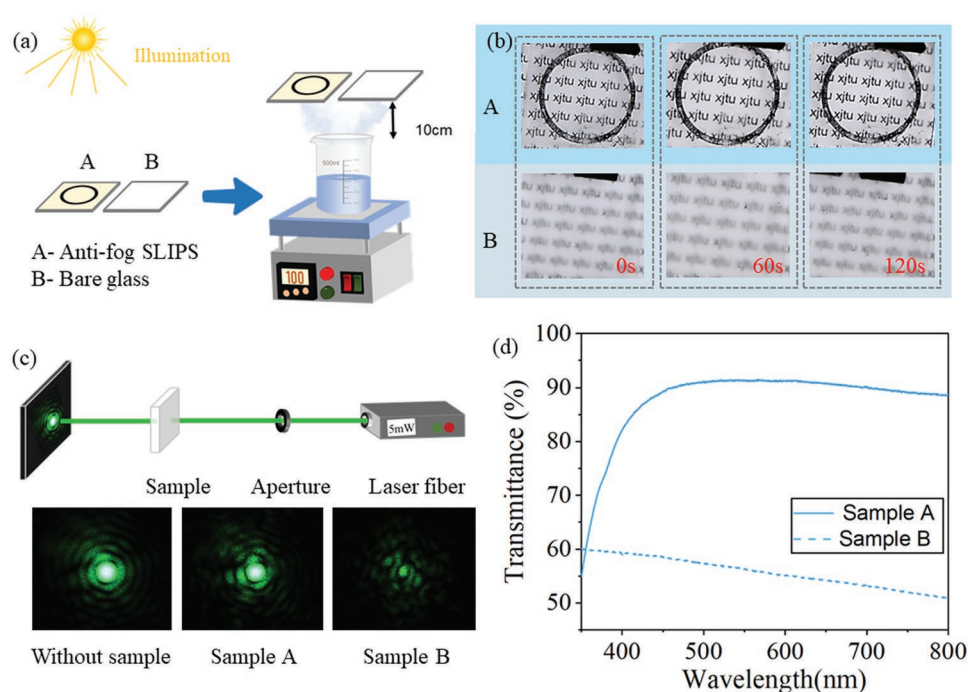


Figure 5. a) Anti-fog experiment. b) Comparison of anti-fog properties of the contrast and prepared samples. The temperature of samples A and B were 49.4 and 33.2 °C, respectively. c) Transformation of the form of the spot after passing through samples exposed under water vapor for 120 s. d) Sample transmission after exposure to vapor for 120 s.

The sample was softened by heating the sample. Next, the soft sample was pressed by the rim of a beaker to form an indentation damage. The composite was then cooled under the pressure to fix the deformed shape of indentation. Figure S5, Supporting Information, displays the scanning electron microscopy image of the damaged sample. The droplet initially slid on the surface till the damaged location. The droplet was stuck at the damaged location and could not move. After illumination under xenon light with a 200 mW cm^{-2} light density, the surface gradually recovered and the droplet continued to slide. Figure 7b displays the molecular chain conformation change of the SMP surface. The GO in the cabin produces heat when the xenon light was turned on, and the SMP on the surface could be heated. The permanent network transitioned from the reversible phase into the activated reversible phase when it reached the transformation temperature and slowly returned to the initial state (Figure 7b). This process revealed considerable self-recovery of the damaged area. Additionally, the sample

also exhibited a strong chemical resistance resulting from the SMP layer. The sample was submerged in solutions of sodium hydroxide (NaOH), hydrogen chloride (HCl), and sodium chloride (NaCl) for 14 days, and the sliding speed of the droplet was measured daily. After the soaking experiment, the sample was removed from the solution and infused with the lubricant again, and slide speed was measured, as displayed in Figure 7c. The image visually revealed the change of speed from day 1 to 14. The results revealed that the slide speed exhibited only a negligible change, which indicated that the prepared sample exhibited excellent chemical durability. The durability under extreme conditions of the prepared sample was tested, as displayed Figure 7e. The temperature cycle test was performed on the prepared sample. The sample was subjected to temperatures of -20 and 150 $^{\circ}\text{C}$ for 2 h, which was considered a cycle. After ten cycles, the prepared sample still exhibited the sliding property. Thus, the sample exhibited excellent temperature durability.

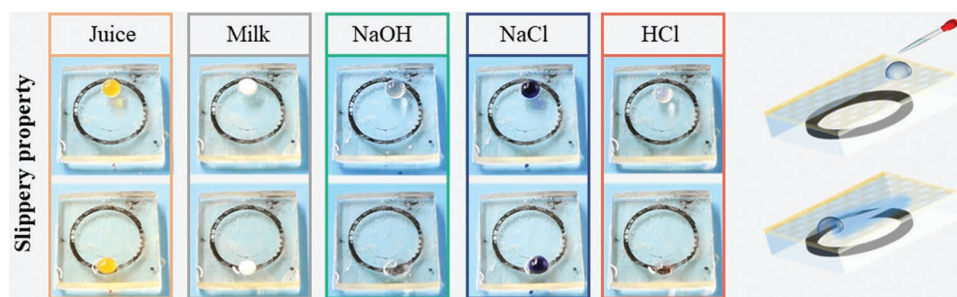


Figure 6. Slippery property of the prepared sample.

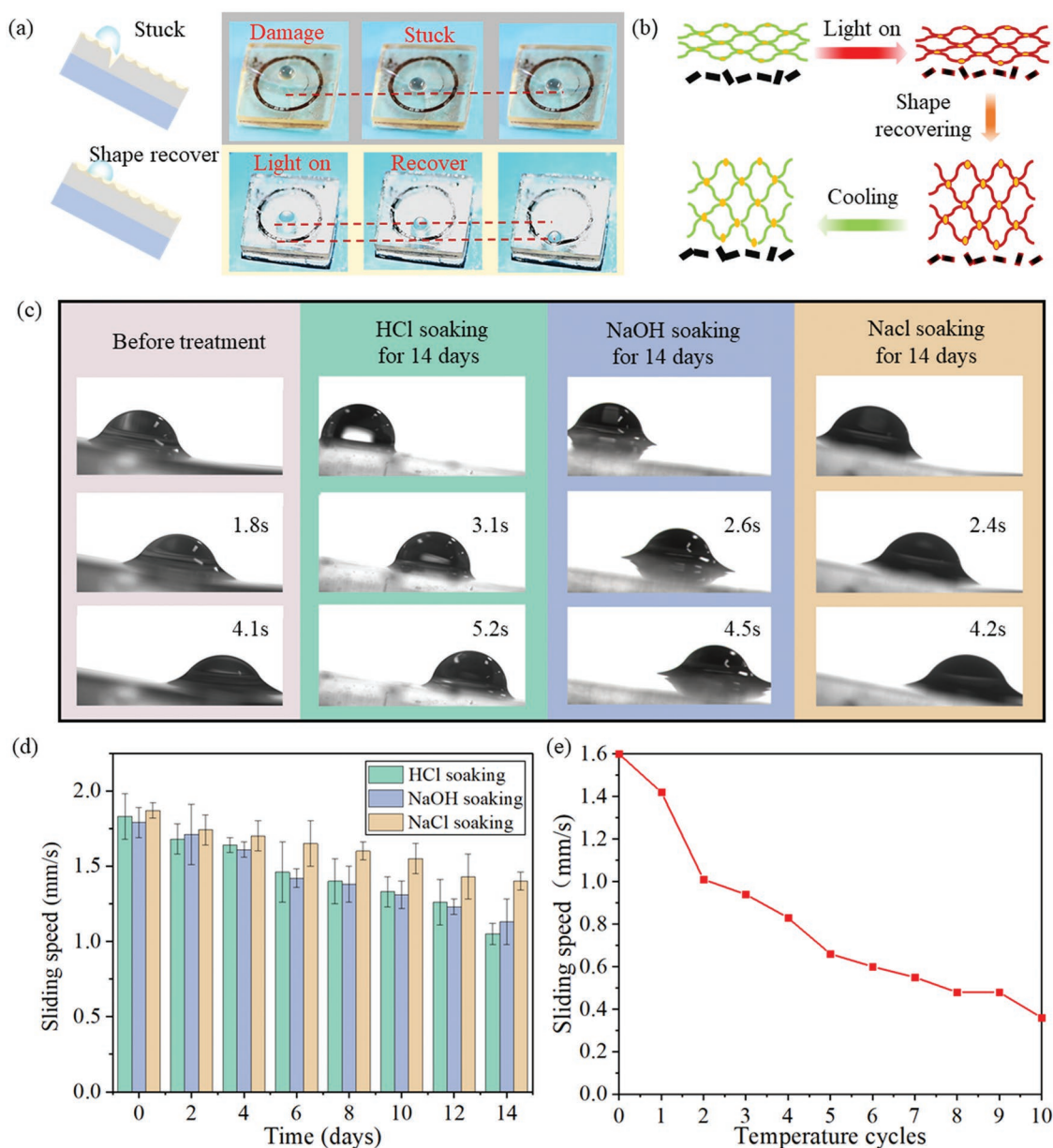


Figure 7. a) Self-repair property of the prepared sample. b) Molecular chain conformation change of the surface. c) Slippery capability of samples after chemical durability test for 14 days. d) Sliding speed after each days' chemical reagent soaking test. e) Slippery speed of the sample after the temperature test.

3. Conclusion

An anti-fog SLIPS inspired by mammalian cornea was fabricated by using the femtosecond laser wet etching method. Graphene in the inner cabin could emit heat spontaneously under the sunlight and heat the sample. This feature ensured that the temperature of the sample remained higher than that of the ambient environment and hindered fog production. The sample exhibited excellent slippery property and self-repair ability because of the SMP SLIPS surface layer. The excellent optical transmittance, anti-fog, and fog-removal property, and self-repair ability ensured that the prepared sample maintained

excellent optical performance even in extreme environments. The proposed processing method can be used in numerous domains.

4. Experimental Section

Materials and Characteristics: Solar light was simulated by using a xenon lamp (Solar-500 W, Beijing NBeT Technology Co., Ltd.), and the power of the simulated solar light was measured by using a power meter (FZ400). The temperature of the surface sample was captured by using a thermal infrared camera (VarioCAM). The surface morphology was imaged by using a scanning electron microscope (FlexSEM-1000,

Hitachi, Japan). The contact angle (CA) and sliding angle were calculated by using a CA measurement device (JC2000D, PoweReach) with a water volume of 7 μL .

The RGO with an average diameter of 0.5–3 μm and a purity larger than 98% was supplied by the Chengdu Organic Chemicals Co. Ltd. Fused silica glass was purchased from WuHan Optics Valley, China. The ethanol (FuYU Chemical Regent), hydrofluoric acid (GR, 40%, Aladdin) and dimethylsilicon oil with various densities (25 $^{\circ}\text{C}$, Macklin) were used without any treatment. The diglycidyl ether of bisphenol A (DGEBA, SMP prepolymer, Nantong Xingchen Synthetic Material Co., Ltd) was mixed with *n*-octylamine (OA, J&K Scientific Ltd) and *m*-xylylenediamine (MXDA, J&K Scientific Ltd). Sodium hydroxide (NaOH), hydrogen chloride (HCl), and sodium chloride (NaCl) were purchased from Aladdin Industrial Corporation.

Fabrication of the RGO Storage Cabin: Step I in Figure 1b is the storage cabin fabrication procedure. The fused silica glass, with a diameter of 2.0 cm $\times$ 2.0 cm $\times$ 1 mm, was cleaned with deionized water and ethanol before fixing on a computer-controlled maglev 3D moving platform. A fiber laser (FemtoYL-40, YSLP, Wuhan, China) was used to generate the femtosecond laser pulse (pulse width = 400 fs, central wavelength = 1030 nm, repetition rate = 2.5 MHz). The signal generator was used to set the output repetition rate to 100 kHz. A 100 $\times$ objective lens ($NA = 0.85$) was used to focus the laser beam onto the surface of fused silica glass, and the channel was written by controlling the movement of the maglev 3D platform along the projected pattern path at a speed of 1000 $\mu\text{m s}^{-1}$. By adjusting the output current, the laser power was set to 400 mW. The computer program was used to control the movement of the 3D platform, and to control the channel shape, height, and helix pitch circle diameter. The prepared sample was subsequently immersed in an ultrasound-assisted solution of 8% hydrofluoric acid for 240 m to fabricate the storage cabin. Next, RGO/silicon oil dispersion was infused into the cabin, as detailed in steps II and III.

Surface Encapsulation: As displayed in Figure 1 (IV), the diglycidyl ether of bisphenol A was mixed with *n*-octylamine and *m*-xylylenediamine in a molar ratio of 5:1:2. Next, the obtained mixture was degassed by a vacuum desiccator to remove bubbles. Subsequently, the mixture was poured onto the samples prepared in the previous process. After curing at 60 $^{\circ}\text{C}$ for 2 h and 100 $^{\circ}\text{C}$ for 1 h, the sample could be successfully encapsulated by SMP, as step IV demonstrated.

Fabrication of the SLIPS on the Surface: As illustrated in step V, the encapsulated sample was pre-fixed on a 3D mobile platform. The femtosecond laser beam (50 fs, 800 nm, 1 kHz) was generated from a regenerative amplified Ti:sapphire laser system (Libra-usp-he, Coherent, USA) and was focused on the sample surface by using an objective lens ($\times 10$, $NA = 0.15$, Nikon, Japan). The average laser power was set at 25 mW. Scanning speeds of 2, 6, and 10 mm s^{-1} and interval adjacents of 2, 6, and 10 μm were tested. After ablation by the typical line-by-line laser scanning process, the micro-porous structure was developed on the sample surface. Next, the prepared sample was successively cleaned by using acetone, alcohol, and deionized water in an ultrasonic cleaning machine.

As presented in step VI, the prepared sample was immersed in 0.5% fluorosilane alcohol solution for ≈ 12 h after the ablation process. Next, the sample was cured in a vacuum drying oven at 50 $^{\circ}\text{C}$ for ≈ 3 h. The lubricant was then infused into the porous structure, as demonstrated in step VII. Dimethicone was selected as the lubricant because of its nonvolatile and environmentally friendly properties.

Supporting Information

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

Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

anti-fogging, optothermal response, slippery surfaces, wettability

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