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Femtosecond laser-patterned slippery surfaces on PET for liquid patterning and blood resistance

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HIGHLIGHTS

- Different patterned SLIPSes are easily prepared by selective femtosecond laser irradiation.
- Self-arrangement of different complex liquid is realized on the patterned SLIPS.
- The patterned SLIPS is acted as a micro-reactor and carries out different reactions in different parts simultaneously.

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ABSTRACT

Patterned slippery liquid infused porous surfaces (SLIPSes) are highly desirable for their potential applications in biofouling, biomolecule collection, droplet microarrays, and microfluidics. Here, an environmental friendly method to fabricate patterned SLIPS by femtosecond laser processing was reported. This simple method includes three steps, femtosecond laser selective ablation, fluoroalkyl silanes modification, and lubricant infusion. The as-prepared SLIPSes possess excellent repellent property to water and even blood. Benefiting from selective laser ablation, plenty kinds of patterned SLIPSes were prepared, such as rhomb shape, circle shape, triangle shape, leaf shape and pentacle shape. Various kinds of liquids such as blood, yogurt, ink and coffee can be self-patterned after they flowed through the patterned SLIPS. Spatially arrangement of these liquid were realized on the patterned SLIPS. The prepared SLIPS owns an excellent durability and reusability. Patterned micro-reaction of different pH indicators with nitric acid and ammonium hydroxide was realized on the patterned SLIPS. The patterned SLIPS carries significant meanings to field of biological and chemical analysis.

1. Introduction

Micro droplet array has always been a research hotspot because of its broad application in the field of biological and chemical analysis [1–3], including cell-based high throughput screen [4–6], controlled particle deposition [7], biosensor fabrication [8–10], and chemical synthesis in droplet [11]. Up to now, patterned superhydrophobic/superhydrophilic surface is an important way to realize micro droplet array [12–17]. Han et al. fabricated a kind of patterned superhydrophobic surface that can be utilized as a micro droplet transportation tool and test bed for studying the evaporation of micro droplets on the heated surfaces [18]. Garrod et al. prepared a patterned superhydrophobic-superhydrophilic surface by plasma chemical methodology and utilized it to collect water [19]. Elsharkawy et al. produced a patterned superhydrophobic paper which was applied to split, store and analyze water droplets of a given size [20]. However, the patterned superhydrophobic surface still possesses some shortcomings that limit its applications. Firstly, it can only repel liquid with simple ingredients [21]. Secondly, the patterned superhydrophobic surfaces show low stability under conditions of friction, high pressure, and high humidity. This situation is caused by the unstable Cassie state which would easily turn to Wenzel state under the conditions of external impact, high pressure, high humidity or long service time [22–24]. Thirdly, the superhydrophobic surface owns a poor stability that could be easily polluted by external organisms and dirts existing in environment, resulting in the loss of the superhydrophobicity [25–26].

As an alternative, inspired by the Nepenthes, slippery liquid infused porous surface (SLIPS) caught researchers' attention. SLIPS is a type of

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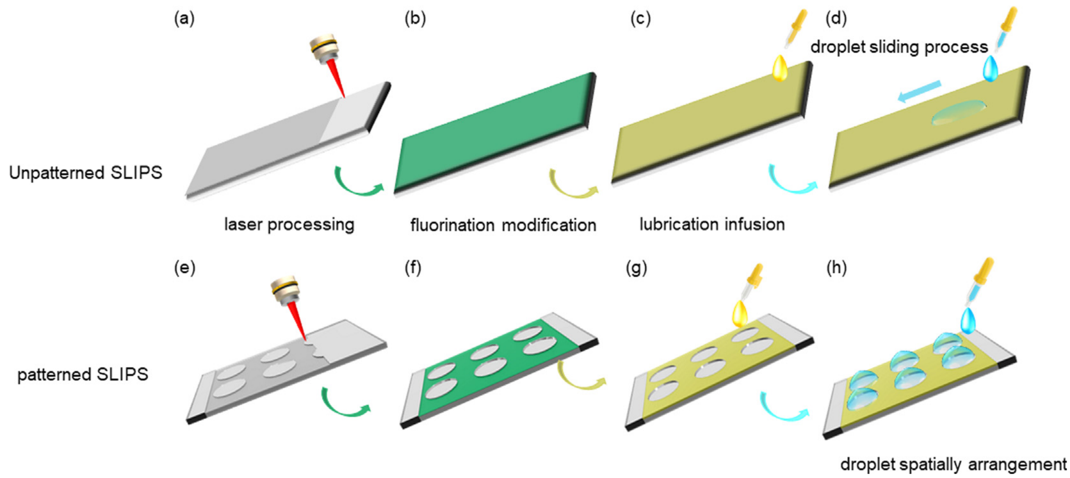

Fig. 1. Schematic diagram of fabrication process of the unpatterned (a-d) and patterned (e-h) SLIPS: (a, e) Laser scanning on PET surface; (b, f) Fluorination modification on the laser-structured area; (c, g) Infusing lubrication; (d) Droplet sliding process; (h) Spatially arrangement on the patterned SLIPS.

solid-liquid complex structure formed by infusing low surface energy liquid into micro-nano pores. It has excellent superomniphobicity, anti-adhesion and self-repairing performance [27]. Wei et al. fabricated a sort of SLIPS surface which exhibits impressive repellency to biological liquid by using the self-assembly method [28]. Yu et al. employed hydrophobic reagent to modify the superhydrophilic filter and endowed it with superhydrophobicity. Then SLIPS surface can be prepared after the filter imbibed the silicone oil [29]. Wang et al. fabricated a SLIPS by sprayed SiO_2-poly (methyl methacrylate) suspension onto substrates and infused perfluorinated lubricant into the rough surface [30]. Nevertheless, the available methods to fabricate SLIPS surfaces still remain some challenges on account of the lack of flexibility, which brought difficulty to the precisely control of the roughness structure's position.

Here, we demonstrated an environmental friendly and precisely controllable method to fabricate patterned SLIPS [31,32]. Femtosecond laser ablation, as an efficient tool in fabricating complex micro-structures [33–39], was used to prepare porous structures on polyethylene terephthalate (PET) surface. Different from other preparing methods which compound a porous film on a substrate material, the femtosecond laser ablation fabricates porous structure on the substrate material directly. Because the porous structure layer and the substrate are actually one, SLIPSes prepared by femtosecond laser ablation are superior on uniformity, mechanical and thermodynamic stability. What's more, during the whole process of the construction of porous structure, there is no chemical waste and other contaminations generated. After fluorination and lubrication, the SLIPS surface can be easily fabricated [40]. The prepared SLIPS showed excellent slippery property to not only water but also blood. Both kinds of liquid can easily slip on the prepared surface. In addition, with the ultra-high flexibility of femtosecond laser processing, patterned porous structure was prepared by selective femtosecond laser ablation. The further prepared patterned SLIPS surface can realize self-patterning of water. Various complex liquid like yogurt, blood and fruit vinegar can also be well patterned on the prepared samples. The prepared sample also possess a good durability and reusability. The following experiment also demonstrated the possibility of our prepared samples acting as a micro-reactor. Different chemical reactions occur on different locations of the patterned SLIPS. Moreover, different patterned droplets would have different response to ammonia and vaporized nitric acid with different pH. This indicated that patterned SLIPS can be used in the field of micro reactor, lab-on-chip and micro sensor.

2. Experimental section

2.1. Femtosecond laser ablation

The PET was chosen as the substrate owing to its good properties. For instance, it shows strong mechanical property, high transparency, and variety liquid resistance [41,42]. A PET sheet with two-millimeter thickness was pre-fixed on a 3-dimentional mobile platform. Through an objective lens (10×, NA = 0.25, Nikon, Japan), the femtosecond laser with the duration pulse of 50 fs was focused onto the PET surface and proceeded the ablation process. A computer software was used to control the movement of the 3-dimentional platform. With the typical line-by-line laser scanning process, micro-porous structure was built on the PET sample. Then the prepared sample was successively cleaned by acetone, alcohol, and deionized water in ultrasonic machine. The femtosecond laser pulses were produced by Ti: sapphire laser system (neutral-usp-he, Coherent, America), with the central wavelength of 800 nm, and frequency of 1 kHz. The average laser power, scanning speed and the interval adjacent were set at 25 mW, 8 mm/s, and 8 μm, respectively. The average diameter of a single crater is 2.95 μm, as shown in figure S1.

2.2. Fabrication of slippery surface

The unpatterned (femtosecond laser ablated the whole PET surface) micro-porous structure was firstly fabricated on the PET surface, as shown in Fig. 1(a). After femtosecond laser ablation, the porous PET was fluorinated and infused lubrication. The porous surface was immersed into 0.5% fluorosilane alcohol solution for 12 h, and put into vacuum drying oven for high temperature curing at about 50 °C for 3 h, as shown in Fig. 1(b). Then the lubrication was infused into porous structure. Here silicone oil was chosen as the lubricating medium on account of its nonvolatile and eco-friendly performance. As shown in Fig. 1(c), the sample was tilted in advance. The lubricant was dripped onto the top part of the sample, allowing it to slip down and immerse into the porous structure by gravity. The micro-porous structure can be easily full-wetted by dimethicone which endow it with the ability to repel various kinds of liquid.

2.3. Fabrication of patterned slippery surface

The procedure of fabricating the patterned SLIPS is similar to the fabrication process of unpatterned SLIPS which also contains four steps. Firstly, the patterned micro-porous structure was fabricated on the PET surface, as shown in Fig. 1(e). Then, utilizing the template method, the

PET surface was partially treated by 0.5% fluorosilane alcohol solution for 12 h, and then put into vacuum drying oven for high temperature curing at 50 °C for about 3 h, as shown in Fig. 1(f). Then the dimethicone was dipped onto the prepared surface. On account of the designed parts are smooth and haven't been treated by fluorosilane, these parts own a weak attraction to the dimethicone. When different kinds of liquid were dropped on the prepared surface, the dimethicone could be easily supplanted and the liquid spatial arrangement was achieved.

2.4. Characterization

The morphology of the laser ablated PET surface was observed by a scanning electron microscope (SEM) (FlexSEM-1000 scanning electron microscope, Hitachi, Japan). The JC2000D contact angle system (PoweReach, China) was used to measure the contact angle and sliding process of blood. The blood was sterile defidrinated sheep blood (CCS30037.01) obtained from MRC Jiangsu. The confocal microscopy (LCSM, Olympics LEXT OLS400) was used to take the confocal photograph.

3. Results and discussion

3.1. Unpatterned SLIPS

Fig. 2(a, b) shows the typical morphology of femtosecond laser-ablated PET. When the femtosecond laser irradiated on the PET surface, the power density would exceed the damage threshold of PET. The laser ablated point of PET surface would absorb the energy of the incident laser through nonlinear effect such as multiphoton and avalanche ionization. High pressure and high temperature plasmas would form because part of the energy was instantly transferred from the electrons to the lattice. The produced heat would melt the area around the focal point and gasification would happen, then plenty of gas would be ejected from the PET surface. The position of the ablated molten state would change with the movement of the femtosecond laser focus point, resulting in the formation of interconnected pores followed by the gas ejecting path. The surface would be occupied by micro/nanoscale rough porous structure with large numbers of protrusions and pores. Some nano fibers, with the diameter ranging from 49 to 160 nm, connected these pores. The diameter of the protrusions ranges from 2 μm to 40 μm and the diameter of the pores ranges from 4 μm to 15 μm. The roughness of the laser ablated PET is 0.351 μm, while the original one is 0.0136 μm, as shown in figure S2. Almost all the micro pores are

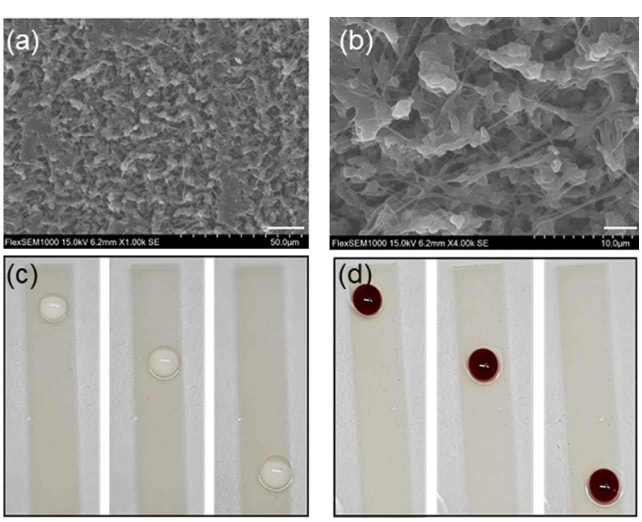

Fig. 2. (a, b) SEM images of unpatterned SLIPS. The scale bars are 150 μm and 40 μm, respectively. Optical images of liquid slipping on unpatterned PET SLIPS: (c) water. (d) blood.

connected to each other, forming a three-dimensional network. After the fluorination modification and lubrication infusion, the prepared porous surface was endowed with the slippery property, as Fig. 2(c) and (d) demonstrated. Both water and blood performed an excellent sliding ability on the prepared PET SLIPS. This phenomenon suggests that PET is an excellent material to fabricate SLIPS. On account of the ultra-high flexibility of the femtosecond laser ablation, the PET surface can be selectively ablated and patterned porous structure could be formed.

3.2. Patterned SLIPS

By running different program to control the area selectively irradiation of the femtosecond laser, different shapes of patterned SLIPSes could be easily fabricated [43]. Fig. 3(a-c) shows the SEM images of the patterned PET surface with the rhomb shape. It can be seen clearly that the surface of patterned non-irradiated area is very smooth, while outside of the patterned area is full of the hierarchical porous microstructure, same as the unpatterned PET surface. In order to obtain a more stable structure, repeated scanning process was designed at the edge. After the fabrication process of each patterned area, the femtosecond laser irradiated the sample along the boundaries of the patterned area to obtain the groove boundary. Between the smooth area and the porous area, microgrooves are existed to divide these two areas clearly. These microgrooves can restrict the experimental liquid and make them exist in the flat area more stably.

After fluorination modification and lubrication infusion, patterned SLIPS was fabricated and droplet self-arrangement can be realized on the prepared surface. Fig. 3(d-g) demonstrated the process of the water self-adhering on the designed area. Firstly, the prepared sample was fixed onto the tilted platform with the tilted angle of 20°. Secondly, the experimental liquid was dipped onto the top part of the sample and slid down from top to the bottom of the sample by gravity. Finally, the experimental liquid was patterned on the certain area of the prepared surface. The raised parts that the arrow pointed in Fig. 3(g) are where the droplets adhered. The pre-designed parts are smooth while the other parts are full of fluorinated micro-porous structure. The porous structure owns a better ability to grab the lubrication that repellent to various kind of liquid. However, because the pre-designed parts were still smooth and were not treated by the fluoroalkyl silane, these parts still remain the original hydrophilicity of the substrate. So the pre-designed parts own a weak attraction to the lubrication, leading to the unstable existence of the lubrication on these parts. The lubrication can be easily replaced by the experimental liquid on these areas, resulting in the liquid self-arrangement.

Patterned SLIPS with circle, rhomb, triangle, leaf, and pentacle shape were easily prepared by a selective femtosecond laser ablation. Blood possess the properties of high propensity for activation of intrinsic hemostatic mechanisms, induction of coagulation, and platelet activation upon contacting with foreign surfaces [44–47]. The reasons mentioned above make blood become a challenging liquid to repel. On general patterned superhydrophobic surfaces, blood always fail to be confined and would wet the entire surface [48]. On account of the repellent property of the SLIPS, blood would not adhered on the areas around patterned parts. Even in the sharp edge of the patterned area, the overflow condition can still be prevented. As shown in Fig. 4(a-d), each shape of patterned area can be full wetted by blood without any overflow condition on our prepared samples. Moreover, other kinds of liquid have also be tested. Except for blood, fruit vinegar, yogurt, ink and coffee were also precisely spatially arranged on the patterned SLIPS surface, as Fig. 4(e-h) demonstrated. This phenomenon illustrated that the fabricated patterned SLIPS can be applied on different kinds of liquid spatially arrangement in various application fields.

This kind of patterned SLIPS surface can be applied as a micro-reactor. Fig. 5 is the reaction process between different patterned droplets with ammonium hydroxide and nitric acid. Firstly, the mixed phenolphthalein solution, litmus solution and water was spatially arranged

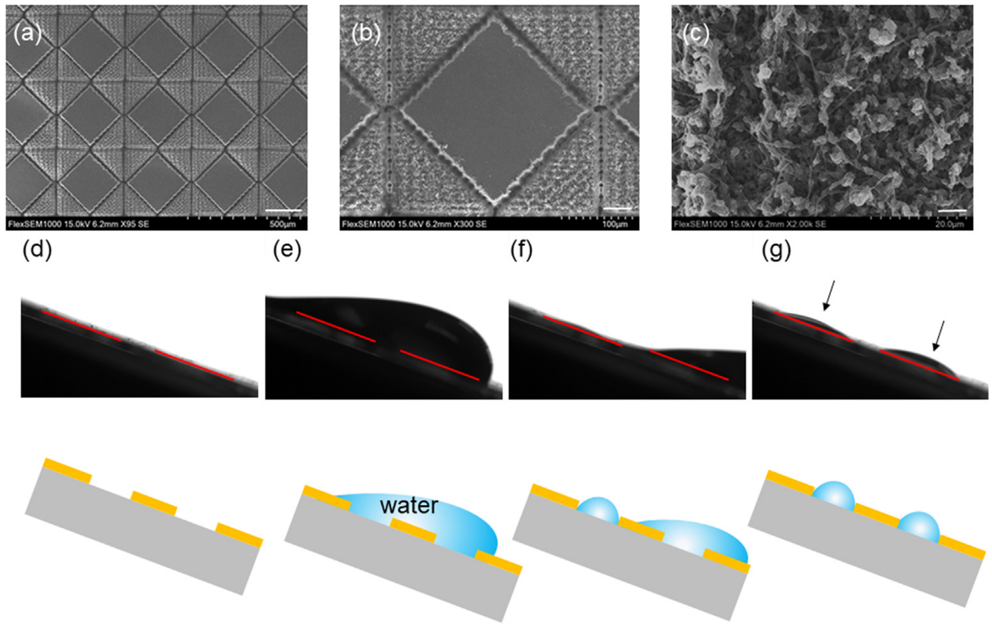

Fig. 3. (a-c) SEM images of the rhomb shaped PET. The scale bars are 1500 μm, 400 μm and 60 μm, respectively. (d-g) water self-arrangement. The red line shows where the patterned area exists. (d) Titled sample. (e) Dropping the experimental liquid onto sample. (f, g) The process of experimental liquid replaced the lubrication on smooth area. The black arrows indicate the adhered droplets. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

on the prepared SLIPS sample as described in foregoing. Then a few drops of nitric acid were dipped on the bottom of a culture dish which was placed subsequently over the sample. On account of its volatility, the nitric acid will react with the phenolphthalein and litmus. The litmus solution gradually turned to red as the reaction proceed while the phenolphthalein still remain transparent, as shown in Fig. 5(a) to (b). Then the culture dish with nitric acid was replaced by another dish with ammonium hydroxide. The phenolphthalein and litmus would continue to react with ammonium, then turns to red and blue, respectively, as Fig. 5(c) and (d) demonstrated. This simple phenomenon proved the feasibility of patterned SLIPS act as a micro-reactor and even a micro-sensor. Compared to traditional superhyphobic/superhydrophilic patterned surface, patterned SLIPS can repel more kinds of liquid with different composition and viscosity, like blood and other biochemical liquid. Patterned SLIPS also inherits the stability of the SLIPS and can maintain its great property under some extreme conditions. Thus, these characteristics allow the patterned SLIPS owns a great potential in chemical and biological field [49–53].

3.3. Durability of the patterned SLIPS

To test the mechanical durability, we carved the sample surface by knife. After been carved, the sample still owns the droplet self-arrangement property, as the Fig. 6(a) and (b) shows. That is because the porous structures of the SLIPS are all connected to each other and the mobility of the silicone oil. Although the carving indeed destroyed the surface structure, the silicone oil could flow over through the porous structure and fill the scratch. Thus, the sample can recover the great property rapidly.

To test the chemical durability of the sample, the samples were immersed into solutions with pH of 1 and 13 for 24 h. After that, they still keep their droplet self-arrangement ability, as Fig. 6(c, d) shows. The reason is that the substrate material is resistant to acid and alkali corrosion, and the micro structure is protected by silicone oil, making the samples less vulnerable to chemical corrosion.

Then we tested the reusability of the patterned SLIPS. We first let the liquid (here we used dyed water) to patterned on the sample and then rinse the sample with water, which is a test circle. The experiment result is that the sample can keep its liquid self-arrangement property even after 20 circles.

4. Conclusions

In this paper, we described the fabrication process to fabricate SLIPS on PET surface, and further established a femtosecond laser ablated method to fabricate patterned SLIPS with excellent durability and reusability. First, patterned porous structure can be obtained by controlling the laser scanning area. Then the prepared surface was partially

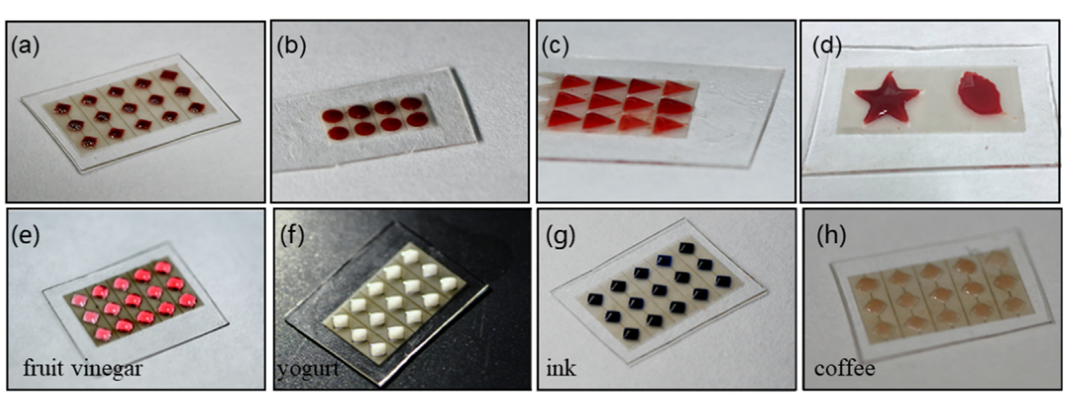

Fig. 4. (a) Rhomb shape with edge length of 2500 μm. (b) Circle shape with diameter of 4000 μm. (c) Triangle shape with edge length of 4300 μm. (d) Pentagram shape and leaf shape. And the microarray of different sorts of liquid (e) fruit vinegar. (f) yogurt. (g) ink. (h) coffee.

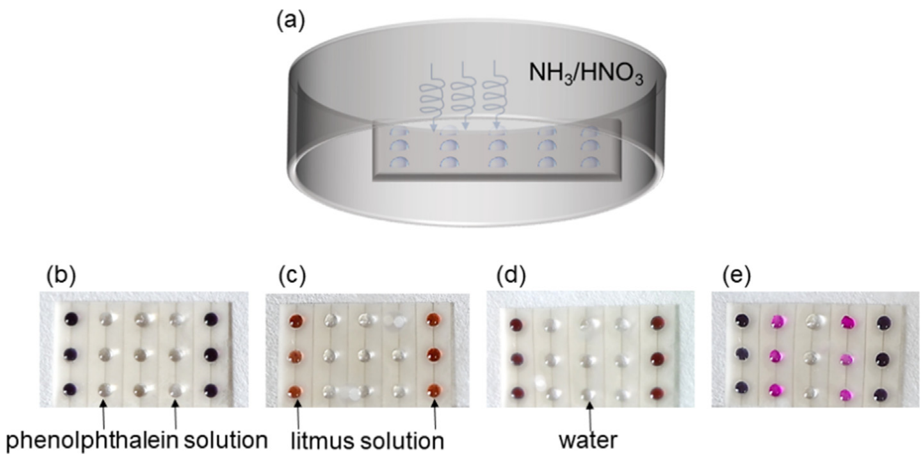

Fig. 5. Patterned micro-reactor: color-change reaction between different micro-droplets (phenolphthalein solution and litmus solution) and nitric acid and then ammonium hydroxide. (a) The illustration of the experiment. (b, c) The litmus solution turns red after reaction with nitric acid. (d, e) The litmus solution turns to blue while the phenolphthalein solution turns to red after reaction with ammonium hydroxide. Water micro-droplets are used as control group. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

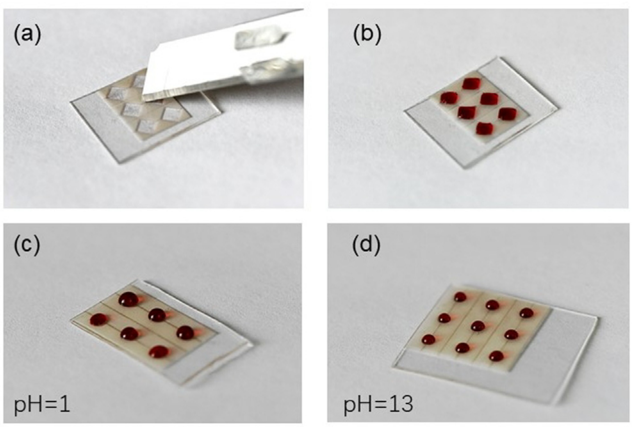

Fig. 6. The durability of the patterned SLIPS. (a) Curving the sample. (b) Droplet self-arrangement of the curved sample. (c, d) The property of the sample after immersed in the solutions with the pH of 1 (c) and 13 (d).

treated with fluorosilane. Finally, lubrication was infused to the microporous structure. After this simple three-step process, different shape of patterned SLIPS can be obtained. We further displayed the self-arrangement of water on the prepared samples and proved that this kind of patterned SLIPS owns excellent spatially arrangement ability to not only blood but also other complex liquid such as fruit vinegar, yogurt, ink and coffee. Moreover, we demonstrated the possibility of this kind of patterned SLIPS to act as a micro-reactor, which indicated that it has a broad potential applications in chemical and biological filed like high-throughput synthesis, analysis, and diagnosis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.optlastec.2020.106469.

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