Inkjet Printing of Patterned Ultra-Slippery Surfaces for Planar Droplet Manipulation

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Abstract

We propose a facile, inexpensive and scalable inkjet-printing method to fabricate patterned lubricant-infused surfaces as the ultra-slippery platform for surface microfluidics, and demonstrate typical planar droplet manipulations. Micrometer patterns were printed on scotch tapes by a home-use inkjet printer to form a template with patterned adhesion contrast. Closely packed monolayer of microbeads were assembled driven by the water surface tension and selectively adhered on the unprinted tape. Then poly(dimethylsiloxane) (PDMS) was cast against the microbead template to form a bowl-like surface, and ultra-slippery PDMS surface was achieved after spin coating lubricant oil. Its interface characterization, sliding performances and long-time stability which are critical for surface microfluidic applications were evaluated and analyzed. Finally the patterned ultra-slippery PDMS were employed as the surface microfluidic platform, and droplet transportation, mixing, trapping and releasing functions have been enabled by very simple and facile driving methods such as pneumatic and gravity driving, which pave a new way for potential flexible and low-cost applications of surface microfluidics.

Keywords

ultra-slippery surfaces, microfabrication, contact angle hysteresis, droplet manipulation, surface microfluidics

1. Introduction

Surface microfluidics, or planar microfluidics, has recently attracted intensive research interests due to their unique advantages compared with traditional channel-based microfluidics, featured with facile fabrication, direct access and easy integration [1-3]. For the surface microfluidic platform, various droplet manipulation methods have been widely used on typically Si or glass substrate, such as electrowetting on dielectric (EWOD) [4], optoelectrowetting (OEW) [5], liquid dielectrophoresis (L-DEP) [6], surface acoustic wave (SAW) [7], magnetic [8] or thermocapillary actuation [9]. However, for all of the driving methods the strong contact angle hysteresis (CAH) lead to the higher driving energy and has been recognized as the common bottleneck for droplet manipulation on surfaces. Although superhydrophobic or superlyophobic surfaces [10, 11] with micro-/nano- surface structures to minimize actual liquid-solid contact have been proposed to largely reduce CAH and move droplet effectively, the risk of wetting transition from the Cassie-Baxter state to the Wenzel state under pressure hindered their application in surface microfluidics [12-14], not to mention that the micro/nano structures which provide robust superhydrophobic or superlyophobic performance are typically difficult for fabrication.

Since 2011, Aizenberg's group [15, 16] has developed bio-inspired self-repairing ultra-slippy surfaces named SLIPS (Slippery Liquid Infused Porous Surfaces) by locking lubricant oils in porous or micro array of solid substrates, which have been intensively studied for the purpose of anti-dirt, anti-ice, anti-frost, and anti-biofouling [17-19]. Due to the incompressible and defect-free liquid surface of lubricant oil on SLIPS, they can greatly reduce CAH and provide extreme pressure stability, which can be a good candidate for

surface microfluidics. However, so far the application of SLIPS in surface microfluidics remains rarely explored due to several limitations [20-22]. Firstly, uniform and well-controlled micro/nano surfaces which can lock lubricants efficiently are highly preferred for SLIPS [15, 23, 24], however, most SLIPS reported in literature were based on bulky and porous materials [25-28], and it is still difficult to control structure uniformity and hence slippery performances on a large area.

Secondly, special structure patterns are usually necessary in surface microfluidics to precisely guide droplets along a specific trace and achieve more complicated operations. Therefore additional processes are required to obtain specific pattern on SLIPS, yet they are difficult to be applied in existing fabrication methods for SLIPS [29-31].

Thirdly, sufficient oil is required on SLIPS to completely cover the structured solid surfaces to avoid pinning effect [24, 32], nevertheless excess oil can exhibit good ultra-slippery performance temporarily and the excess oil will gradually drain away under gravity, resulting in the degradation of slippery performance. Hence the characterization of the ultra-slippery performance of SLIPS should be unified.

Herein we propose a facile, inexpensive and scalable approach to fabricate patterned ultra-slippery poly(dimethylsiloxane) (PDMS) surfaces using the simple yet effective self-assembly polystyrene (PS) microbeads on scotch tapes with the aid of an inkjet printer, and systematically evaluated their ultra-slippery performances and long-term stability for surface microfluidic applications. Typical droplet manipulations such as droplet transportation, mixing, trapping and releasing were demonstrated on the as-prepared ultra-slippery platform using facile gravity and pneumatic driving. Such patternable

ultra-slippery surfaces provide a new platform for surface microfluidics, and enables simple, inexpensive and programmable driving methods.

2. Materials and methods

Materials

A common scotch tape (Scotch 600, 3M, USA), a commercial ink-jet printer (LBP6018, Canon, Japan) with the original cartridge (Canon CRG-925), PS microbeads (5 μm , 20 μm , 50 μm and 100 μm , KB sphere, China) and PDMS (Sylgard 184, base: curing agent = 10 : 1, Dow Corning, USA) were used for fabricating and patterning ultra-slippery surfaces. Methyltrichlorosilane (~ 99 wt%, Sigma Aldrich, USA) was used for low-surface-energy treatment of PDMS for easy demolding. Toluene (~ 99.8 wt%, Sigma Aldrich, USA) was used to dissolve the residual PS microbeads, and phenyl silicone oils (100 cP, 300 cP, 500 cP and 1000 cP, Sinopharm, China) were used as the lubricant.

Methods

Firstly, as-required micro pattern was drawn in a computer and printed on a scotch tape using the inkjet printer in order to selectively block adhesion area with the resolution better than 500 μm (Figure 1a). Then the tape was immersed with an aqueous solution containing homogeneous PS microbeads (Figure 1b). Multi-layer microbeads were closely packed on the whole scotch tape driven by water surface tension after evaporation or manual extraction of water (Figure 1c). Because the adhesive force of the tape is stronger than adhesion between PS beads to PS beads and ink to PS beads, by applying a moderate pressure to squeeze the

microbeads on the tape and shaking off redundant microbeads without being stuck to the tape, compactly packed monolayer PS microbeads were obtained, as the negative template for the required pattern (Figure 1d). After the low-surface-tension modification of PS with methyltrichlorosilane treatment, PDMS precursor was casted against the template and demolded following the standard soft lithography method [33]. Residual microbeads stuck on PDMS were then dissolved by toluene washing (Figure 1e). Finally, phenyl silicone oil was spin-coated on the bowl-like PDMS microstructures (Figure 1f).

For systematic comparison, different sizes of PS microbeads, as well as oils with different viscosities (100 cP, 300 cP, 500 cP and 1000 cP), were used to evaluate the ultra-slippery performance and durability on PDMS. First, the as-prepared samples were vertically placed for different time intervals and their residual oil weights were measured for quantitative analysis of the oil film thickness. Second, the critical sliding angles of water on these samples were measured by using the contact angle profiler (DSA25, KRUSS, Germany). Finally, the patterned ultra-slippery PDMS surface was used as a surface microfluidic platform to demonstrate typical droplet manipulations, such as droplet transportation, mixing, trapping and releasing using two simple driving methods, gravity and pneumatic driving. The tilting stage used for gravity-based driving was controlled by the home-made open-source hardware (Arduino Uno R3, Seeed, China). Minipumps (KPV36A, KOGE, China) were used for pneumatic driving.

3. Results and discussions

3.1 Characterization of the oil-solid interface

Figure 2 shows the optical microscopic and scanning electronic microscopic (SEM, NanoSEM 450, Nova, USA) pictures of the assembled PS microbeads with different sizes, as well as their negative replication on PDMS. Closely-packed and uniform microstructures have been readily achieved.

For the long-term stability test, PDMS samples with different porous sizes (5 μm , 20 μm , 50 μm and 100 μm) and phenyl silicone oils with different viscosities (100 cP, 300 cP, 500 cP and 1000 cP) were tested (see Figure S1 ESI), yet only data for 100 cP and 1000 cP were shown in Figure 3 for clarity. As shown in Figures 3a-b and Figure S2 (ESI), in the initial 8 hours, oil residue on PDMS decreased rapidly mainly due to the flowing off of oil, and after that the decrement was much slower, indicating that oil was effectively locked by microstructures, and evaporation should play the dominant role. Therefore all experiments for sliding performance characterization were conducted after vertically placing the samples for 8 hours, to eliminate the rapid reduction of oil thickness. These results also showed that lower oil viscosity and smaller porous size led to less oil residual, thinner lubricate layer and better sliding performance. For example, when oil viscosity is 100 cP, the oil residual for samples with 5 μm size reaches a stable level of $\sim 25.6 \mu\text{g}/\text{mm}^2$, while for samples with 100 μm porous size, it is about $40.1 \mu\text{g}/\text{mm}^2$ (Figure 3a). Similar trend occurred for samples with oil viscosity 1000 cP, and the residual oil significantly increased to $36.6 \mu\text{g}/\text{mm}^2$ (for 5 μm size) and $51.8 \mu\text{g}/\text{mm}^2$ (for 100 μm size).

We measured the critical sliding angle (CSA) with different porous sizes, oil viscosities and water droplet volumes. As shown in Figures 3a-b, CSA decreased with smaller porous size, higher oil viscosity and larger water droplet (see Figure S1 in the ESI), therefore the

optimal configuration in our experiments for best sliding performance (CSA $\sim 3^\circ$) is 5 μm porous size, oil viscosity of 1000 cP.

Another simple method to evaluate the oil residual thickness is the transparency test for PDMS samples, considering that oil-solid state can affect the transparency as well as the slippery property [34]. As shown in Figure 3c, transparency became worse for larger porous sizes, and the sample with 5 μm porous size demonstrated the best transparency. Therefore, it is concluded that minimizing the porous size yields better oil locking ability for long-time use despite less residual oil, which is consistent with previous report [34]. This may be explained by the scaling effect of surface to volume ratio (S/V). With reducing porous size, S/V of oil increases and the oil-solid adhesion per area increases, which is preferred for droplet thermodynamics, therefore enhances the oil locking effect.

3.2 Analysis of water-oil interface

The droplet status on SLIPS is essential for droplet manipulation. Several studies showed that droplets can move easily only when droplet floated on the lubricant [24, 32, 35]. D. Quéré proposed a criterion to judge whether a water droplet floats on oil or contacts with solid [36]:

$$\Delta\gamma = \gamma_{\text{oa}} \cos \theta_{\text{os}} - \gamma_{\text{wa}} \cos \theta_{\text{ws}} - \gamma_{\text{wo}} > 0, \quad (1)$$

Where γ represents surface tension, θ represents intrinsic contact angle, and subscript w, o, a, s represents water, oil, air and solid, respectively. When $\Delta\gamma > 0$, water is floating on oil. In our experiments using silicon oil with 1000 cP, γ_{wo} is measured as 16.23 ± 0.64 mN/m, $\gamma_{\text{wa}} = 69.34 \pm 0.62$ mN/m, $\gamma_{\text{oa}} = 21.31 \pm 0.10$ mN/m while θ_{ws} and θ_{os} are calculated as $(106.36 \pm 2.06)^\circ$ and $(13.96 \pm 1.62)^\circ$, respectively (see Table S3 in the ESI for details).

According to Eq. 1, $\Delta\gamma=23.98$ mN/m. Therefore, a water droplet should be floated on the silicone oil without contacting with solid, and therefore allows good sliding performances.

Furthermore, there are two possible states in the oil-water system: water totally cloaked by oil or water floated on oil. For surface microfluidic applications, the former is highly preferred since the existence of oil film can largely prevent water evaporation. The judgment is based on the calculation of spreading coefficient S_{ow} between oil and water, expressed as [32]:

$$S_{ow} \equiv \gamma_{wa} - \gamma_{wo} - \gamma_{oa} > 0, \quad (2)$$

In our configuration $S_{ow}=31.80$ mN/m >0 , so the silicone oil will cloak the water droplet to minimize the surface energy of the whole system. Therefore such SLIPS platform is very advantageous for aqueous droplet manipulation in surface microfluidics by reducing water evaporation.

We also examined the internal flow pattern inside a droplet. According to Smith *et al.*, the criterion for analyzing the internal flow status is written as [32]:

$$v/v_i \propto (1 + \frac{\mu_o}{\mu_w} \frac{h}{t}), \quad (3)$$

where v is the droplet velocity, v_i is the velocity of oil-water interface, μ_o and μ_w respectively represents the dynamic viscosity of oil and water. h is the height of the droplet mass center above the solid surface, and t is the oil film thickness. In our case $h \gg t$ so that $(1 + \mu_o/\mu_w h/t) \gg 1$ and $v/v_i \gg 1$, which suggests a water droplet mainly rolls on SLIPS (see Figure 4), which is beneficial to accelerate droplet mixing.

3.3 Demonstration of droplet manipulation

Based on the above analysis, we fabricated patterned ultra-slippy PDMS (5 μm porous size, 1000 cP silicone oil) to demonstrate their application as a surface microfluidic platform (Figure 5). In Figure 5a, a Y-shape sample and a zigzag-shape sample were obtained by inkjet printing (illustrated in Figure 1a). Figure 5c shows a large-area ultra-slippy PDMS and Figure 5d shows an ultra-slippy ring-shape PDMS with a sliding water droplet on it.

Such surfaces have been used to demonstrate typical droplet manipulations. Figures 6a-c show that droplets were transported, mixed and split in an X-shape ultra-slippy shallow channel defined by the tapes, and Figures 6d-e show the batch movement of a droplet array. Three water droplets (with yellow dye) were dispensed into separate branches, and moved together towards the opposite side by manually tilting the substrate. Programmable driving by gravity was also demonstrated using a simple rotary stage. Figure 6g-l show a PDMS surface with an ultra-slippy zigzag path fixed on a tilt rotary stage. The rotation controlled the gravity component and thus the transport direction. 9 traps were designed to store sequential droplets to form a droplet array (Figure 6g-j), and then droplets were simultaneously released (Figure 6k, l). Note that the blue droplet on the normal PDMS surface did not move in any cases for comparison. These exemplified the potential applications of ultra-slippy surfaces as the novel surface microfluidic platform for facile and low-resistance droplet manipulation based on gravity.

Droplets can also be easily manipulated through other simple driving methods such as blowing. Figure 7 shows that droplets were transported along the designed X-shape slippy paths and mixed at the appointed spot with fast transport speed up to 20 mm/s in a well-defined time sequence, by using low-pressure air blow generated by mini-pumps (see the

video in ESI S4). Such pneumatic driving should be readily programmed to enable the fully automatic control, direct access of individual droplets with low-cost and easy use (see the concept video in ESI S5). Therefore, compared with the traditional driving methods in surface microfluidics, such as electrowetting, magnetic actuation, thermo-capillarity and SAW, non-electrical gravity driving and pneumatic driving methods on SLIPS provide low-cost, high-speed and great flexibility for manipulating individual as well as batch droplets, which may inspire many practical applications of surface microfluidics.

4. Conclusions

In summary, a facile, inexpensive and scalable method using inkjet printer to define the ultra-slippery pattern is proposed to fabricate the bowl-like and lubricant-infused ultra-slippery surfaces as the novel platform for droplet manipulation in surface microfluidics. Common scotch tapes with inkjet-printed patterns were used to selectively stick monolayer PS microbeads onto tapes. We analyzed the lubricant locking ability of each substrate for long time use, and examined effects of microbead sizes, viscosity of lubricant oils and droplet sizes. We found that smaller porous sizes and higher oil viscosity yield better oil locking effect by increasing the oil-solid adhesion, and therefore led to smaller CSA.

We also analyzed the droplet status on the ultra-slippery surfaces. In our experiments we found droplets are floated on the silicone oil, cloaked by oil and will be rolling during transportation, which endows superiority for droplet manipulation to reduce motion resistance and water evaporation, and accelerate mixing.

Finally, we demonstrate the typical droplet manipulation such as droplet transportation, mixing, trapping and releasing on the patterned ultra-slippery PDMS surfaces using gravity as

well as pneumatic driving. Both methods are simple, non-electrical and programmable, and hence suitable for biological/chemical reactions and cell manipulation. These achievements shed new light to surface microfluidics by using patterned lubricant-infused surfaces as the low-cost and versatile platform.

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Figure captions

Figure 1 Schematic illustration of the microfabrication strategy. The scotch tape was printed to obtain the patterned adhesive region(a), then it was fixed on a glass slide and immersed into water (b).PS microbeads were added and water was removed by evaporation or manual extraction (c) to enable closely packed microbead assembly. After removing floating microbeads, a monolayer PS sticking to the tape was obtained (d). PDMS was cast against the PS microbead template (e), and residual microbeads on PDMS were dissolved by toluene. Eventually ultra-slippery PDMS surface was achieved after spin coating lubricant oil (f).

Figure 2 Microscopic pictures of monolayer PS microbeads (5-100 μm). In(a)-(d), left side shows PS microbeads on the adhesive tape, and right side shows SEM images of the reverse porous microstructures on PDMS.

Figure 3 Characterization of stability, sliding performance and transparency of ultra-slippy PDMS. In (1-b), lubricating oil was found to flow down in the initial 8 hours so that residue oil reduced rapidly, and after that oil evaporation played a main role and the residue oil reduced slowly. Samples with smaller porous size and less viscous oil have less oil residue. The critical sliding angle (CSA) for different porous sizes decreases with larger droplet and higher viscosity of silicone oil. In (c), the declining transparency of samples also reflects the decreasing oil thickness. See ESI Figure S1-S2 for more data.

Figure 4 Illustration of a water droplet rolls on the ultra-slippy surface and is cloaked by thin oil film.

Figure 5 As-fabricated ultra-slippy PDMS samples. (a-b) a Y-shape and a zigzag-shape samples obtained by inkjet printing (Figure 1). (c) A large-area (11 cm x 7 cm) sample. (d) A ring-shape sample with a water droplet.

Figure 6 X-shape and zigzag-shape slippy channels for typical droplet operations. (a-c) Droplets transportation, mixing and splitting in the as-fabricated slippy X-shape channel. (d-f) Droplets dispensed to different branches and the droplet array was simultaneously transported. Sample with a zigzag-shape slippy path was fixed on a tilt rotary stage. By controlling the transport direction and speed, droplet array with 9 trapping slots could store sequential droplets (g-j), and then they were simultaneously released (k-l). Blue droplets on the normal PDMS surface (d-i) did not move in any cases for comparison.

Figure 7 Pneumatic driven droplet mixing along an X-Shape ultra slippy path. A sample with an X-Shape slippy path was used to mix droplets at the appointed location by controlling the sequence of droplet mixing. Low-pressure air blow was used to push forward droplets with facile and precise control.

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Figure(s) 1

Figure(s) 2

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Figure(s) 7

Figure(s) S1

Figure(s) S2