Fabrication and testing of surface ratchets primed with hydrophobic parylene and hexamethyldisilazane for transporting droplets

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Abstract. We demonstrate a way to transport droplets by an arrowed micropillar array surface with hydrophobic parylene. The lightly hydrophilic parylene surface could be changed to hydrophobic one by treating with fluorine-based plasma (CF_4 or SF_6). The droplet on this hydrophobic parylene surface with arrowed ratchets could be transported by a speaker, and the average measured velocity was 29 mm/s. Moreover, the authors compared driving performance of the parylene surface ratchets with the ones modified by the hexamethyldisilazane (HMDS) vapor. Both the results of using hydrophobic parylene and HMDS were better than the previous work. [DOI: 10.1117/1.3280261]

Subject terms: plasma; parylene; hydrophobic; hydrophilic; surface ratchets.

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1 Introduction

Surface tension–driven devices for microfluidics have attracted much attention in recent years due to their intrinsic advantages of power saving and their concise configuration without moving parts. For example, the transporting of droplets was accomplished by electro-wetting on dielectrics (EWOD)[1] or capillary pumping.[2]

Recently, room temperature-deposited parylene is a promising microelectromechanical systems (MEMS) material and process, and there are many applications[2–11] for using it. It is complemented by chemical vapor deposition (CVD) and has the characteristics of conformal coating (good step coverage). There are many advantages in parylene, such as its flexibility, biocompatibility, and transparency.[12] If one could adjust the wetting behavior of the parylene surface, it is probable to achieve fluid-driven behavior by its wetting phenomenon.

Surface modification processes were developed for changing the surface properties of the material by many researchers, and the methods of surface modification included the following five types:

1. Ion implantation—for instance, implanting silver ions (Ag^+) in polystyrene to increase its electric conductivity.[13]
2. Surface modification of the material by acidic solution or alkaline solution treatments—for example, using H_2SO_4 or H_3PO_4 solutions to slightly react with the parylene surface, resulting in the surface changes

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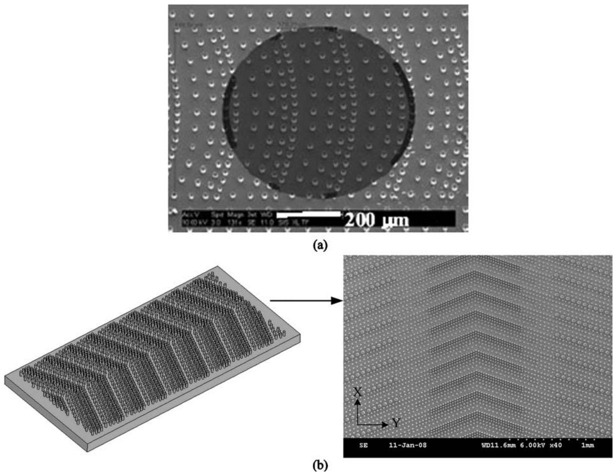

Fig. 1 (a) Pattern of Shastry et al. (Ref. 28), (b) pattern of the design in this study.

in both the advancing and receding contact angles.[14]

3. Using cold plasma to bombard the material surface and to modify the surface tension.[15–18] On the access of plasma surface treatments, the surface property of the polydimethylsioxane (PDMS) is popularly changed by oxygen plasma,[19] and the surface property of the PET (polyethylene terephthalate) and cotton could be likely changed by fluoride-based plasmas (SF_6).[15]
4. Using the self-assembly of a functional group monolayer that directly changes the surface properties of the material.[20–22]
5. Making the biomimetic surface micro- or nanostructure on the target polymer surface.[23–25]

The preceding research had already basically established the surface modification process in the different polymer materials. The surface properties of materials were a function of the surface modification conditions. For instance, when the flow rate, power, and time of the process were increased, the contact angle of the water droplet and the surface roughness of the surface would increase.[26,27] From the results of the previous research, the authors assumed that it would be possible to change the slightly hydrophilic surface of parylene to a hydrophobic surface by using fluoride-based plasma.

A paradigm for transporting droplets on superhydrophobic surfaces was presented,[28] as shown in Fig. 1(a). The surface ratchets were treated with hydrophobic fluorinated-octyl-trichloro-silane (FOTS), and the droplets on surface ratchets were transported by a speaker and a wave form generator. However, one possible drawback of the previous work might be the intrinsic toxicity of FOTS, which restricts the biomedical application of this droplet-driving device. Parylene, on the contrary, is a biocompatible substance, and its conformal coating could make many working substrates easily wrapped by parylene. After the parylene surface is fluorinated by fluoride-based plasma, the lower fluoride ion release has a lower cytotoxicity to avoid damage to the cell. In addition, hydrophobic parylene is less expansive than Teflon, is a simpler process, and has lower process temperature.

To illustrate the motivation for this research, the "Teflon-like" hydrophobic property was exhibited in hydrophobic parylene, so it should be able to replace FOTS[29] or even Teflon.[30] If one followed to design a regular rough surface like a fakir carpet in sub-micron scale by using a plasma-etching technique,[31] this device surface modified with hydrophobic parylene might also be applied to self-cleaning, higher chemical stability, advanced physical characterization. Another emerging application might be the lithographically induced self-assembly via controlling the periodic polymer pillar arrays.[32]

Restated, we would like to design a new micro-structure for transporting water droplets on surface ratchets. Based on the experience of the former optimum gaps between pillars and the optimum height of pillars,[33] the diameter and height of each cylindrical pillar in the ratchet pattern of the design used here were 20 and 30 μm, respectively, as shown in Fig. 1(b).

2 Experiment

2.1 *Plasma Surface Modification*

In this work, four different methods of surface modification treatments will be tested, and those methods have been discussed in the previous section. A reactive ion etching system (Samco, RIE-1C) was used, and its maximum power was 150 W.

2.1.1 O_2 *plasma*

Parylene-C is lightly hydrophilic, and the contact angle of deionized (DI) water on the parylene-C surface is around 74 deg, as shown in Fig. 2(a). The contact angle measurement is performed by an FTA-125 machine. Accordingly, the parylene surface (grown on silicon by SCS PDS-2010 coater) was treated with O_2 plasma (Samco RIE-10N, 10 sccm, 50 W) in 10 s, and the parylene surface was transformed into hyperhydrophilic (the droplet contact angle was 0 deg to 5 deg), as shown in Fig. 2(b). Then, these surface properties could be maintained for at least 2 h. For the consideration of real applications, e.g., O_2 plasma pretreatment on the inner walls of microchannels for capillary suction, the 2-h maintaining time for the hydrophilic surface was still too short. Nevertheless, this experiment was only to demonstrate that the lightly hydrophilic parylene surface could be temporarily adjusted to a hyperhydrophilic one, like the well-known PDMS.

2.1.2 CF_4 *or* SF_6 *plasma*

An interesting phenomenon was found, that the lightly hydrophilic parylene could also be transformed into the hydrophobic surface, induced by fluorine-based plasma treatment. When the surface of the parylene was treated with CF_4 or SF_6 plasma in 10 s, the results were different from case to case. The water droplet contact angle of parylene (of DI water) was around 123 deg after treatment with CF_4 plasma in 150 W, 50 sccm after 10 s, as shown in Fig. 3(a). The water droplet contact angle of parylene (of DI water) was around 117 deg after treatment with SF_6 plasma in 150 W, 50 sccm after 10 s, as shown in Fig. 3(b). From the

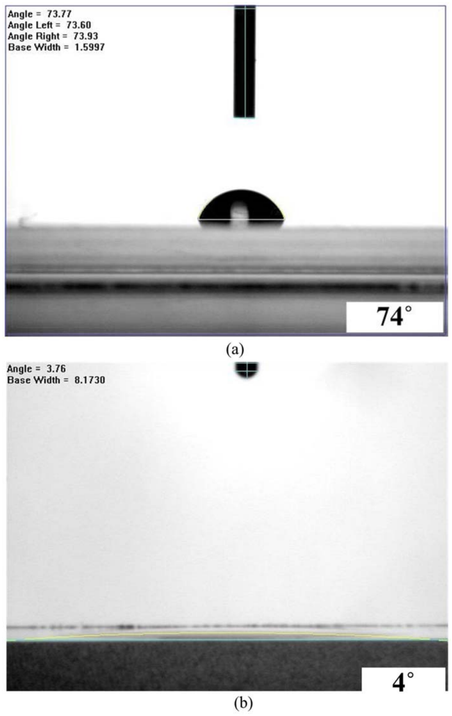

Fig. 2 (a) The original wetting behavior of parylene (RH=37%); (b) the hydrophilic wetting behavior of parylene after treatment by O_2 plasma (50 W, 10 sccm, 10 s).

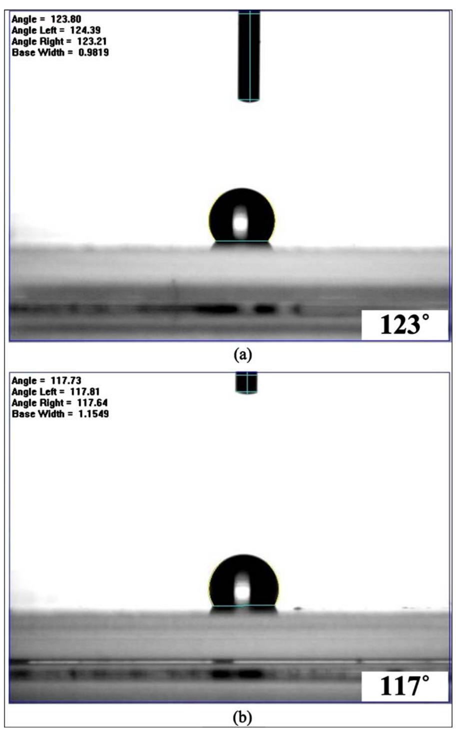

Fig. 3 (a) The wetting behavior of hydrophobic parylene treated by CF_4 plasma of 150 W, 50 sccm after 10 s; (b) the wetting behavior of hydrophobic parylene treated by SF_6 plasma of 150 W, 50 sccm after 10 s.

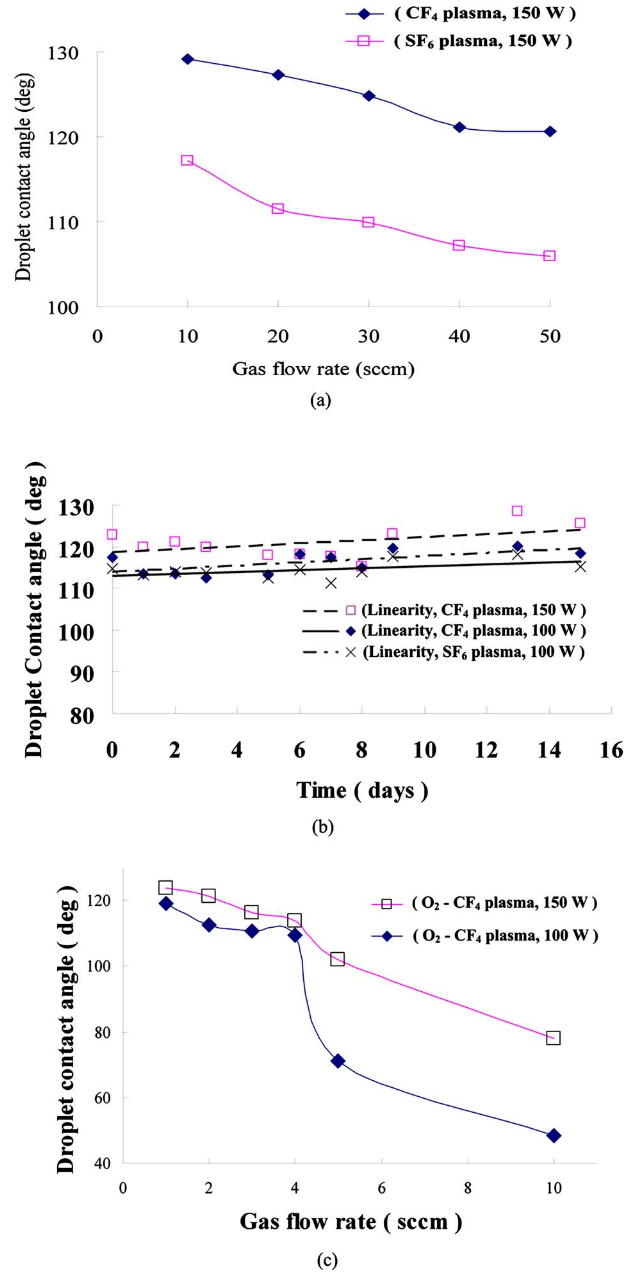

Fig. 4 (a) Comparison of the contact angle in different flow rate of CF_4 and SF_6 plasma; (b) the relation of the contact angle and time in hydrophobic parylene treated by different plasma; (c) the relation of the contact angle and flow rate of 20% O_2 and 80% CF_4 plasma mixture in treated parylene surface.

plasma gas flow rates in 20 to 50 sccm, as shown in Fig. 4(a), the water droplet contact angle of the parylene treated with SF_6 plasma was smaller than that treated with CF_4 plasma. Furthermore, subject to the power limitation of 150 W in the plasma system herein (Samco, RIE-1C), the higher the power of the plasma, the larger the water droplet contact angle would be. (The optimum conditions mentioned in Ref. 18 cannot be verified in this study.) The water droplet contact angle with the preceding treatment was monitored for two weeks. We investigated whether the hydrophobic characterization of the fluoride-based plasma-treated parylene could be maintained for 15 days at least.

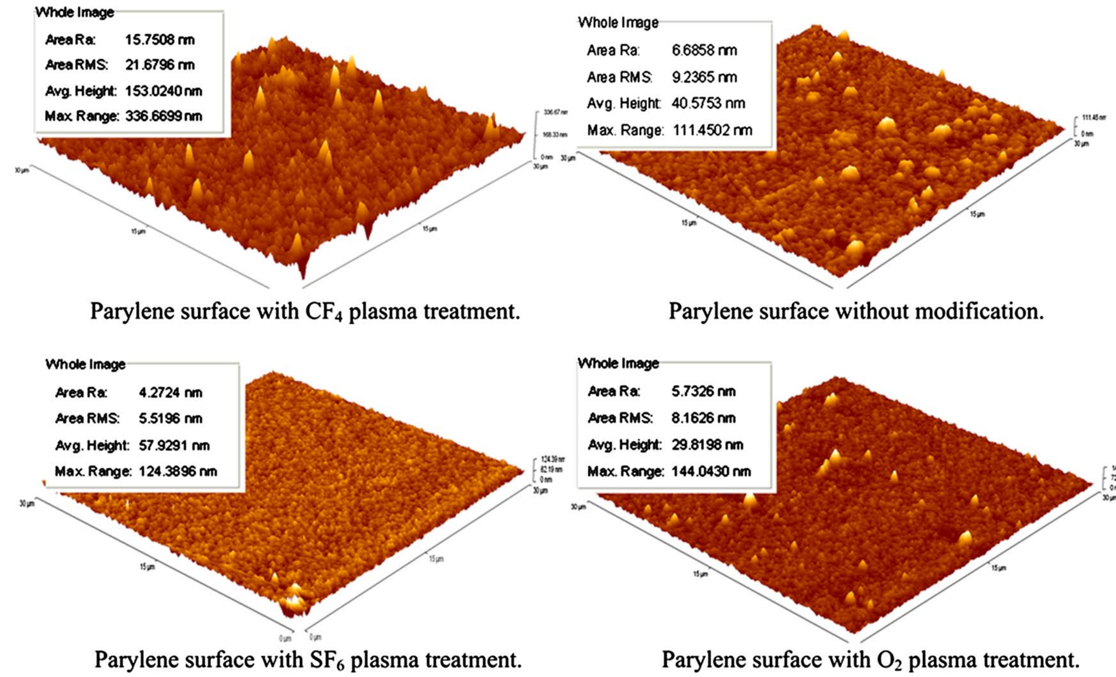

Fig. 5 Roughness of the parylene surface at different conditions from AFM (150 W, 50 sccm at treatment).

This phenomenon is depicted in Fig. 4(b). (The droplet contact angles during 9 to 15 days moreover increase by an amount of 5 to 10 deg, although the reason is not clear.)

2.1.3 *Mixture of* O_2 *plasma and fluorine-based plasma*

In order to figure out the effect of the plasma mixture on the wetting of the parylene surface, the parylene was treated (10 s) with the O_2 (20%) and CF_4 (80%) plasma mixture, and the results are depicted in Fig. 4(c). The results showed that the droplet contact angle decreases with the plasma mixture gas flow rate. There was a big decrease for the 100-W case as the flow rate adjusted from 4 sccm to 5 sccm. Learning from the hydrophilic effect of oxygen plasma in the previous section, the authors reasonably infer that the ionized oxygen dominated over the fluoride-based plasma on the wetting situation of parylene surface as the plasma density reaching a threshold level. Meanwhile, it was still hydrophobic in most of the cases of 150 W except the gas flow rates that went beyond 5 sccm. This result showed that the surface hydrophobic property increased as the RF power increased. The average surface roughness of the parylene surface without modification was 6.68 nm, as shown in Fig. 5. The surface roughness of the parylene surface treated with CF_4 plasma at 10 and 50 sccm was 9.258 and 15.75 nm, respectively, as shown in Fig. 6. It was obvious that the variable surface roughness increased as the RF power increased. Also, the average surface roughness of the parylene surface treated with O_2 plasma was 5.73 nm—this difference was quiet small. The average surface roughness of the parylene surface treated with SF_6 plasma was 4.27 nm, as shown in Fig. 5, so the difference of the roughness before and after SF_6 plasma treatment was not large. The average surface roughness of the parylene surface treated with CF_4 plasma was larger than that treated with SF_6 plasma. This is why the contact angle of the parylene surface treated with CF_4 plasma will be larger than that treated with SF_6 plasma. Being induced by fluorine-based plasma treatment, the lightly hydrophilic parylene could also be transformed into the hydrophobic surface. When the parylene surface was treated with O_2

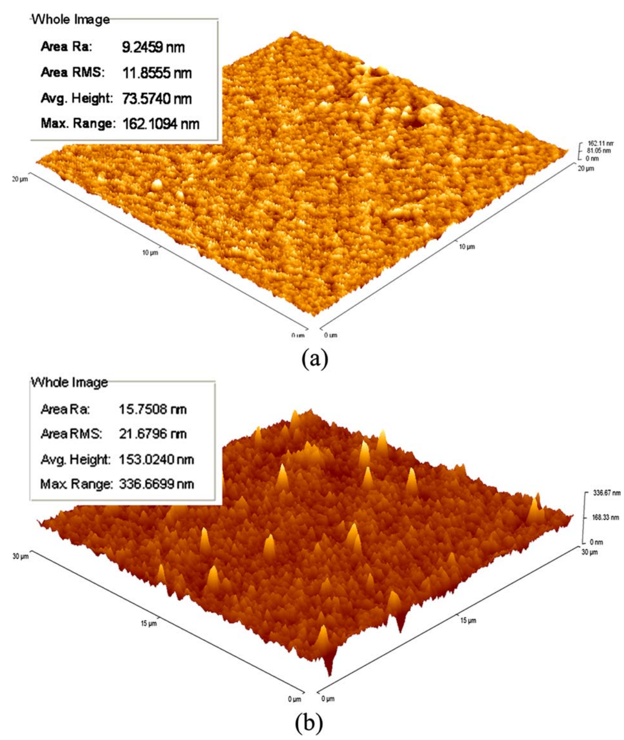

Fig. 6 Roughness of the parylene surface treated by CF_4 plasma at (a) 10 sccm and (b) 50 sccm.

plasma, however, the parylene surface was transformed into hyperhydrophilic. When the surface was treated in the mixed plasma, the RF power increased the hydrophobic property and offset the effect caused by hydrophilic functional groups. The etching effect of the RF power of 150 W was greater than that of 100 W, and it made the shifting of the hydrophilic effect of 150 W poorer than that of 100 W.

From the preceding discussion on the selection of plasma treatment on parylene surfaces with a long-lasting characteristic of wetting behavior, the CF_4 plasma with conditions of 150 W, 10 sccm, 10 s was chosen as the standard processing parameters in the following experiments.

2.2 *Arrowed Surface Ratchets*

The ideal hydrophobic surface is the micro- or nano-scaled rough surface of hydrophobic materials excelling in the well-known "lotus effect."[34,35] So the design of arrowed surface ratchets was additionally used in this research to enhance the lotus effect, and the hydrophobic parylene (primary choice) or hexamethyldisilazane (HMDS) vapor (secondary choice) were chosen as the hydrophobic material or surfactant. The reason for using HMDS primer additionally is as a surfactant comparison to the FOTS.

2.2.1 *Design of the arrowed surface ratchets*

The authors have made fakir surface ratchets similar to the previous work[28] in advance and followed the experimental setup in Sec. 2.3 to investigate the driving speed. But the configuration of the surface ratchets could not guarantee the moving direction of water droplets. The authors modified the fakir design to the arrowed surface ratchet shown in Fig. 7(a) to avoid the issue of uncertain moving direction. All the cylindrical pillars of a ratchet have their diameter D of 20 μm and height H of 30 μm. An arrowed surface ratchet was composed of three types of pillar design: A, B, and C, as depicted in Fig. 7(b). Pillar design A was composed of three cylindrical pillars with gap d_1 of 10 μm. Pillar design B was composed of two cylindrical pillars with gap d_2 of 20 μm. Pillar design C was composed of one cylindrical pillar with gap d_3 of 30 μm.

This pattern was designed for limiting the droplet to gather in the middle area on the arrowed micropillar array surface. Letting the number of micropillar arrays in the A area be larger than those in the B and C areas, as shown in Fig. 7, and it became the most hydrophobic (the contact angle was the largest) in the A area. The spacing of the arrowed micropillar was lightly smaller in the B area, and it was less hydrophobic. The spacing of the arrowed micropillar was the smallest in the C area, and it was least hydrophobic (the contact angle was the smallest).

The arc micropillar surface was used in the literature,[28] and the authors redesigned the arrowed micropillar surface. Redesigning the arrowed pattern makes the micropillar spacing more nonuniform inside. This arrowed pattern couples the larger difference between the advancing and receding contact angles for enhancing the droplet motion. If there was no such a pattern of pillar spacing, the droplets would not be restricted in a specific direction, and the droplet may lose control. In other words, the redesign of the arrowed micropillar array is used to control the advancing direction.

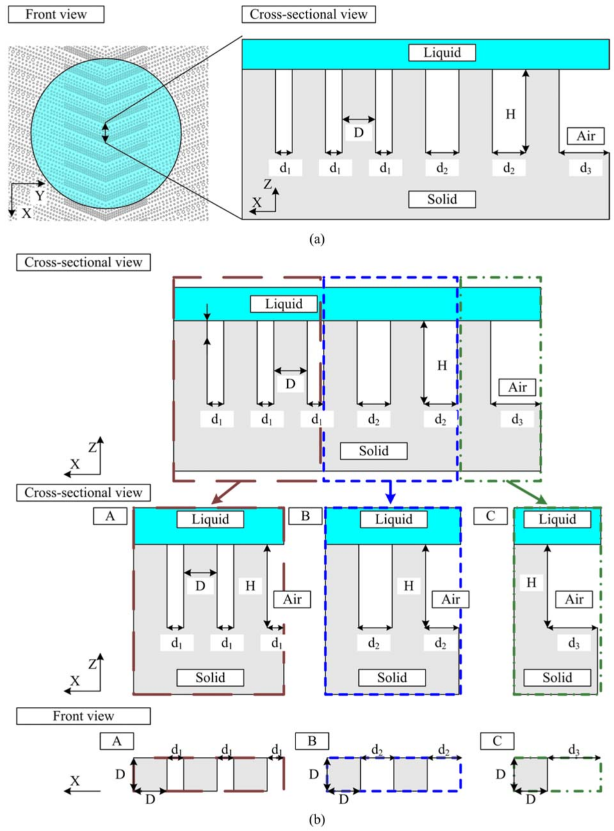

Fig. 7 (a) Design of the arrowed surface ratchets; (b) pillars A, B, and C of a pitch of the arrowed surface ratchet.

2.2.2 *Fabrication of the arrowed surface ratchets*

Two types of treatment on the arrowed surface ratchets were chosen: HMDS was used as the hydrophobic material in type I, and the hydrophobic parylene treated with CF_4 plasma (150 W, 10 sccm, 10 s) was used in type II. The fabrication process of the arrowed surface, as shown in Fig. 8(a), is illustrated as follows:

1. Grow the wet oxide on a 4-in (100) silicon wafer.
2. Define the arrow patterns of photoresist by lithography.
3. Pattern the oxide by buffered oxide etch (BOE) as the opening windows.
4. Etch the silicon wafer to have ratchet pillars of 30 μm deep by inductive couple plasma (ICP).
5. Remove the photoresist by acetone and the oxide by BOE.
6. Deposit HMDS primer or hydrophobic parylene (coating parylene at first and then surface treatment by CF_4 plasma) as hydrophobic material.

After completing the process, one can fabricate a microdevice that includes the pillar structure with arrowed surface, as shown in Fig. 8(b).

2.3 *Experimental Setup*

The experimental setup for transporting the water droplets is shown in Fig. 9. First, a silicon-based pattern was defined by lithography, and the chosen material was deposited onto

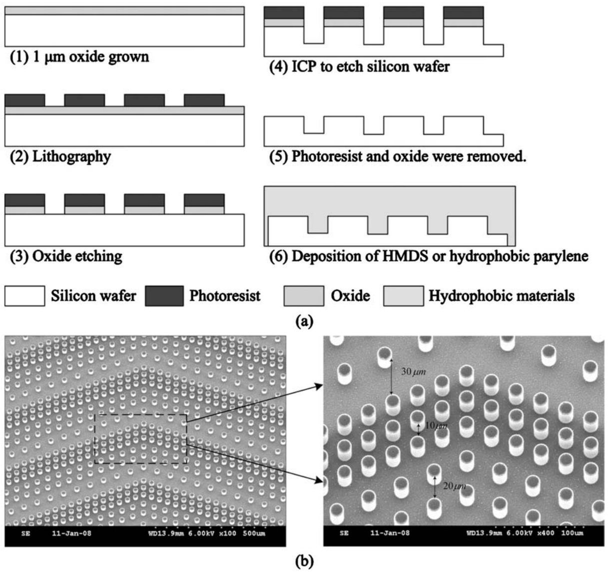

Fig. 8 (a) Fabrication processes of the arrowed surface ratchets; (b) structure of the arrowed surface ratchets device.

the silicon base. If the material was parylene, it was treated by the plasma, and then the droplet contact angle could be measured after the surface modification. The device was put on the speaker membrane at horizontal level, and the added water droplet was dispensed onto the device by the syringe pump. Last, the water droplets were transported by the speaker-induced vibration and function generator, and the image was recorded by the digital video.

3 Results and Discussion

3.1 *Comparison of Droplet Contact Angle between Different Surface Modifications*

The comparison of droplet contact angles between different surface modifications is listed in Table 1. The contact angle of DI water on silicon-based surface ratchets was 111 deg, and it showed the lotus effect. The contact angle of surface ratchets with HMDS was 127 deg, and it enhanced the hydrophobic property of silicon-based surface ratchets. The droplet contact angle of surface ratchets with parylene was 96 deg, and it lowered the hydrophobic property to some extent. The contact angle of surface ratchets with hydrophobic parylene of CF_4 plasma treatment was 131 deg, and it demonstrated that the CF_4 plasma treatment could enhance the hydrophobic property of the surface ratchets. It was found that the increase of the RF power would result in

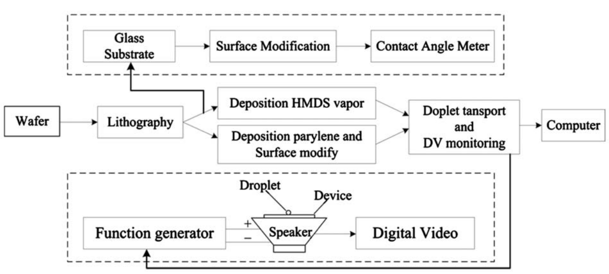

Fig. 9 Outline of the experimental setup.

Table 1 Comparison of contact angle between different surface modifications.

Materials	Contact angle
Parylene	74 deg
Parylene with O_2 plasma treatment	0–5 deg
Parylene with CF_4 plasma treatment in 100 W	110 deg
Parylene with CF_4 plasma treatment in 150 W	123 deg
Parylene with SF_6 plasma treatment in 100 W	108 deg
Parylene with SF_6 plasma treatment in 150 W	110 deg
Silicon-base arrowed surface ratchets	111 deg
Arrowed surface ratchets with HMDS	127 deg
Arrowed surface ratchets with parylene	96 deg
Arrowed surface ratchets with hydrophobic parylene	131 deg

the increase of the droplet contact angle. It was investigated that the contact angle of parylene surface treated with O_2 plasma (50 W, 10 sccm, 10 s) was around 4 deg.

From the results of the literature,[36] it was found that when the parylene-C was treated with O_2 plasma, the contact angle drastically decreased in the initial stage and slowly decreased with increasing power and time. When the power was increased to 100 W, the contact angle decreased to a value of 27.3 deg. Also, when the time was increased to 120 s, the contact angle decreased to a value of 34 deg. The literature[36] has proved that when the parylene surface was treated with O_2 plasma, the parylene surface corresponding to CO_3^- and $C{=}O$ bonds resulting from O_2 plasma–induced oxidation. Also it was able to transform the parylene surface into hyperhydrophilic. In another experiment,[37] the low-density polyethylene (LDPE) was treated with O_2 plasma, and the O_2 functional groups (alcohol, carbonyl, ester, etc.) were the results of complex reactions of free radicals caused by O_2 plasma treatment. This was why the parylene surface could be transformed into hyperhydrophilic by O_2 plasma. These results were similar to those of the devices herein.

Two main factors induce hydrophobicity: The first one is that there is fluorine in the parylene surface after the CF_4 or SF_6 plasma modification and it causes the increase of contact angle. The second one is the effect of roughness, and the contact angle is increased as roughness increases. The SF_6 plasma modification decreased the roughness of the parylene surface, so the droplet contact angle of the parylene surface with CF_4 plasma modification was larger than that with the SF_6 plasma modification. The roughness of the parylene surface treated with O_2 plasma was hardly

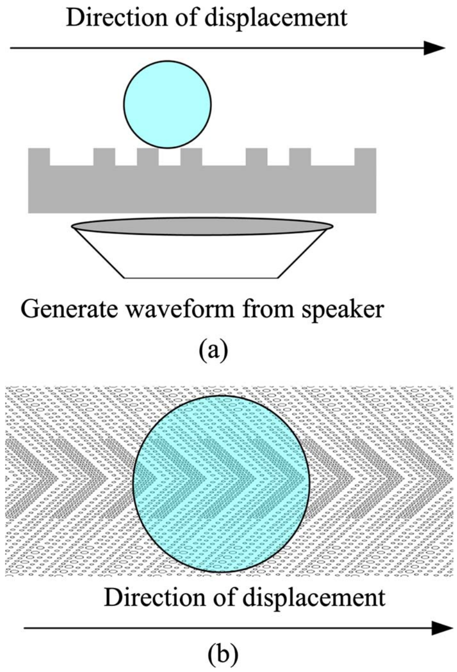

Fig. 10 The relative position of the speaker, device, and droplet and the direction of displacement: (a) side view; (b) top view.

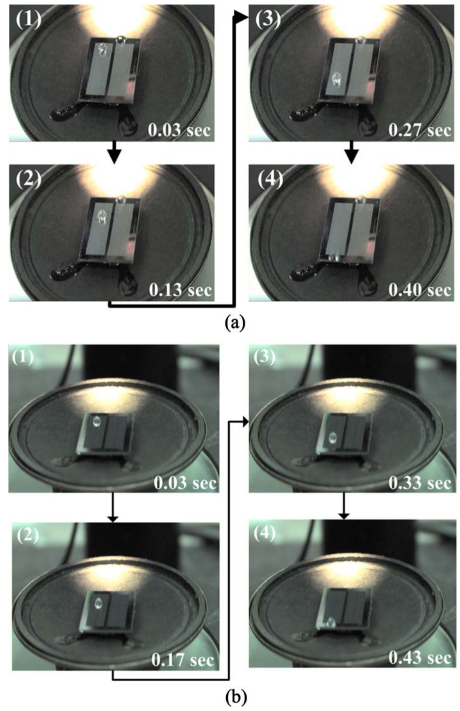

Fig. 11 Time shots of the droplet moving on the arrowed surface ratchets with (a) HMDS, and (b) hydrophobic parylene.

changed, and there were hydrophilic functional groups in the parylene surface, so it would cause the contact angle of the parylene surface to become smaller.

3.2 *Droplet Movement on the Arrowed Surface Ratchets by Speaker Shaking*

The droplet transporting device with arrowed surface ratchets was put onto the membrane of a speaker. Then, square waves of ± 5 V at different frequencies were supplied to induce the vibration of the device and then transport the water droplets. The side and top views of relative positions between the speaker, device, and droplet and the direction of displacement are shown in Fig. 10(a) and 10(b), respectively.

Different frequencies (50 to 55 Hz) were supplied to drive the displacement of the water droplet on the arrowed surface ratchets with HMDS treatment or modified by hydrophobic parylene, and the results were similar. When a square wave of 50 Hz was supplied to the speaker and induced the vibration of the arrowed surface ratchets with HMDS treatment, the water droplet could be transported, as shown in Fig. 11(a). When a square wave of 50 Hz was supplied to the arrowed surface ratchets with hydrophobic parylene, the droplet could also be transported, as shown in Fig. 11(b). When the time was 0.03 s, the displacement of the water droplet was 1.6 mm on the HMDS treatment surface. When the time was 0.13 s, the water droplet arrived at the location of 4.9 mm. When the time was 0.27 s, the displacement of the water droplet was 9.6 mm. When the time was 0.40 s, the water droplet arrived at 11.8 mm. In the case of the device with modified hydrophobic parylene, the displacement of the water droplet was 0.2 mm on the parylene surface when the time was 0.03 s. When the time was 0.17 s, the water droplet arrived at the location of 5.7 mm. When the time was 0.33 s, the displacement of the water droplet was 10.3 mm. When the time was 0.43 s, the water droplet arrived at 11.0 mm. The changing displacement of the water droplet and time is shown in Fig. 12. The average velocities of the water droplets' displacement were 28 and 29 mm/s for the HMDS experiments and hydrophobic parylene cases, respectively. The results demonstrated that the water droplet could be transported by the arrowed silicon surface ratchet device with modified HMDS or hydrophobic parylene coating. Compared with the prior technique of using FOTS, the average velocity of the droplet was only 12 mm/s (Ref. 28). Therefore, the transporting efficiency of the droplet in this research was upgraded around three times.

The authors made the droplet move uphill along the gradient surface in this paper.[38] In the current design, however, this made an apparent difference between the advancing contact angle and the receding contact angle of the droplet,

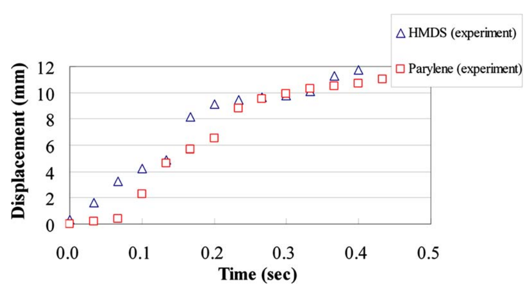

Fig. 12 The relation of displacement and time in different cases.

and therefore influenced the extent that the droplet induced to move by the energy from the speaker shaking was strengthened accordingly.

As a supplementary statement, the authors would like to emphasize that the thickness and uniformity of the modified HMDS and the hydrophobic parylene coating are quite different. The former was composed of several layers of HMDS molecules (the layer number was uncertain over the whole device area); the latter was apt to be grown beyond one micron thick uniformly and would be the main candidate for future applications.

4 Conclusion

This research demonstrated that the property of the silicon-based arrowed surface ratchets could be modified by using different materials and gas plasmas. One could enhance parylene's hydrophobic property or conversely change it to be hyperhydrophilic. More importantly, the droplet was successfully transported by the arrowed surface ratchets coated with hydrophobic parylene or HMDS, and the maximum velocity of transporting the water droplet was measured as 29 mm/s. This research greatly enhanced the transporting velocity of the droplet, and the direction of the water droplet movement was controllable. The devices might have emerging application to microfluidics.

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