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A capillary pumping device utilizing super-hydrophobic silicon grass

Chun-Fei Kung\textsuperscript{1}, Chien-Cheng Chang\textsuperscript{1,2} and Chin-Chou Chu\textsuperscript{2}

\textsuperscript{1} Division of Mechanics, Research Center for Applied Sciences, Academia Sinica, Taipei 115, Taiwan, Republic of China
\textsuperscript{2} Institute of Applied Mechanics and Taida Institute of Mathematical Sciences, National Taiwan University, Taipei 106, Taiwan, Republic of China

E-mail: mechang@iam.ntu.edu.tw and chucc@iam.ntu.edu.tw

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Abstract

In this study, we show that a compact silicon grass surface can be generated by utilizing the induced coupled plasma method with suitably chosen fabrication parameters. This super-hydrophobic structure suspends deionized water on top of the grass and keeps the contact angle at around 153°. The silicon grass is used to improve the driving efficiency of a capillary pumping micro-duct (without sidewalls), which is completely defined by a bottom hydrophilic stripe (adjacent to a Teflon substrate) and a fully top-covered hydrophobic Teflon surface which is coated on a glass substrate. The channel has a height of 3 \( \mu \text{m} \) and a width of 100 \( \mu \text{m} \). In this work, the Teflon substrate is replaced with the silicon grass surface. When the fluid is flowing through the micro-duct on the stripe, the interface between the silicon grass and the hydrophilic stripe forms a stable air cushion barrier to the fluid, thus effectively reducing the frictional force. By changing only the interface with this replacement, we demonstrate that the average measured velocities of the new design show improvements of 21\% and 17\% in the driving efficiency over the original design for transporting deionized water and human blood, respectively. It is also shown that the measured data of the present design are closer to the values predicted by a theoretical analysis which relates the flow velocity to the contact angles, surface tension and fluid viscosity.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

It is known [1, 2] that super-hydrophobic surfaces are formed by the additive action of two basic conditions: a low polarity or low surface energy material composition and a particular surface roughness. Some surfaces in nature, such as lotus leaves and water striders, have self-cleaning properties, the so-called lotus effect. Such surfaces are extremely difficult to wet, with the water contact angles being in excess of 150° and the slide angles as little as 1°. The water droplets on the surface readily roll off even when the contact angle hysteresis is negligible, and subsequently surface dust and debris can be removed easily.

Inspired by the novel repellent properties of lotus leaves, scientists have created artificial super-hydrophobic surfaces using various techniques to develop certain surface topographies, such as electro-deposition and chemical etching [3, 4], plasma etching [5–9], laser treatment [10, 11], chemical vapor deposition [12–14], sol–gel processing [15, 16], lithography [17, 18] and so on. In order to fabricate a surface with a water contact angle larger than 150°, two key factors must be considered: low surface energy and high surface roughness. It must be noted that even a material with the lowest surface energy alone gives a water contact angle of only around 120°. To obtain a surface with higher hydrophobicity, we need to take into consideration an appropriate surface roughness. It is worthwhile to mention a few previous studies by describing the specific methods and hydrophobic surfaces produced. Shieh \textit{et al} [9] created the nanograss-on-nanopillar structure by plasma etching wherein the contact angle can reach close to 180°. The work of Baldacchini \textit{et al} [11] consists of irradiating silicon wafers with femtosecond laser
pulses wherein a contact angle around 160° can be achieved. In the study of Latthe et al [15], they used silica films which were synthesized by the sol–gel process and modified by two kinds of silylating reagents to improve the surface properties. In the case of surface modification by trimethylchlorosilane (TMCS), the surface wettability can be changed from hydrophilic to super-hydrophobic with a contact angle around 162°.

A simple theory would be helpful in understanding the surface property. In the simplest case, the wettability of the solid surface is commonly evaluated by the contact angle given by Young’s equation [19] as follows:

$$\gamma_{SV} = \gamma_{SL} + \gamma_{LV} \cos \theta,$$

where $\gamma_{SV}$, $\gamma_{SL}$ and $\gamma_{LV}$ are the interfacial free energies per unit area of the solid–gas, solid–liquid and liquid–gas interfaces, respectively. This equation is applicable to flat surfaces, but not to rough ones. In order to describe the contact angle on rough surfaces, two distinct models have been proposed to incorporate this effect. First, the Wenzel model [20] considers the increase of the contact area due to the surface roughness:

$$\cos \theta_w = r \cos \theta,$$

where $\theta_w$ is the apparent contact angle on the rough surface, $\theta$ is the contact angle on the flat surface of the same material, and $r$ (0 ≤ $r$ ≤ 1) is a roughness factor, defined as the ratio of the actual area of a rough surface to the geometric projected area. The Wenzel model shows that a hydrophobic surface becomes more hydrophobic when the surface is rougher; its new contact angle becomes greater than the original one. In contrast, a hydrophilic surface becomes more hydrophilic; its new contact angle falls below the original one. In brief, the microstructure of a surface amplifies the natural tendency of the surface.

Next, the Cassie model [21] assumes that some air remains trapped between the drop and the cavities of the rough surface, which means that the liquid is suspended on top of the microstructures:

$$\cos \theta_c = f \cos \theta + f - 1,$$

where $\theta_c$ is the apparent contact angle at a surface composed of solid and air, and $f$ is the area fraction of the solid that touches the liquid.

The Cassie model shows that when the area fraction $f$ is smaller, the contact angle becomes greater. Whether the surface property is hydrophilic or hydrophobic, a rougher surface is more hydrophobic. This effect shows that if we want to fabricate a more hydrophobic surface, the structures should be sharp, like needles, to minimize the contact area between the liquid and the solid.

In this study, we propose a simple method to form a super-hydrophobic surface on the silicon substrate. The idea was originated by Pradeep et al [22], who used the induced coupled plasma (ICP) process to generate a grass structure on a silicon substrate under suitable conditions with specific parameters, such as the SF$_6$ flow rate and ratio of etching and passivation cycle times. Later, these authors used the grass formation structure to enhance the overall performance of heat dissipation of electronics/MEMS components [23]. Stubenrauch et al [24] used deep reactive ion etching to fabricate needle-like structures (black silicon) on the surface, which are interlocked with each other to form a bonding interface at room temperature. The bonding strength of black silicon interfaces is about 3.8 MPa, and may find applications in microfluidics with catalysts and micro-optical or carrier wafer bonding in microelectronics.

The present purpose, however, is to find the optimal ICP process that generates dense or compact silicon grass on the bottom such that the grass is sufficiently hydrophobic to suspend liquid on top of the surface. The structure is further applied to enhance the driving efficiency of our previous capillary pumping design [25]. The capillary pumping design is a micro-duct defined by a bottom hydrophilic stripe (adjacent to the Teflon surface) and a fully top-covered hydrophobic Teflon surface which is coated on a glass substrate. In this study, the Teflon surface is replaced with the silicon grass. Once the fluid is pipetted into the entrance of the duct, the capillary pumping generated by the liquid meniscus at the hydrophilic interface will drag the fluid into the microchannel. With the bottom Teflon surface replaced by the super-hydrophobic silicon grass, the deionized (DI) water can flow 1.21 times faster in the new design than in the original design, while human blood can flow 1.17 times faster. The key effect is reduction of the frictional force due to the super-hydrophobic characteristic. As the fluid flows in the channel on the hydrophilic stripe, it may extend over to slightly wet the Teflon edge. Therefore, the contact interface still produces frictional force when the working fluid flows in. With the Teflon replaced by the super-hydrophobic surface, the new interface can form a stable air cushion barrier to the fluid, thereby diminishing effectively the frictional force on the edge of the channel and improving the driving efficiency. The evidence is also shown by comparing the measured data of the present and previous designs to the values predicted by a theoretical analysis which relates the flow velocity to the contact angles, surface tension and fluid viscosity.

2. Basic method and application

In this section, we describe a detailed procedure used to fabricate the dense silicon grass, and apply this structure to improve the driving efficiency of a capillary pumping device, which was proposed some time ago by the present authors [25].

2.1. Method of producing silicon grass

The Bosch process [26] is one silicon etching technique for making patterns with high-aspect ratios. It uses the ICP technique and alternates flows of SF$_6$ and C$_4$F$_8$ gas for etching and passivation processes to etch patterns.

Unlike other reactive ion etching processes, this method allows patterns with high-aspect ratios to be attained, as shown in figure 1(a). The first cycle is ion-assisted etching of the silicon substrate by the etching gas (SF$_6$), followed by the sidewall passivation step with polymer-producing gas (C$_4$F$_8$). The passivation cycle coats the sidewalls with a protective polymer, i.e. poly-tetra-fluoro-ethylene (PTFE),
Figure 1. Sketch of the Bosch process by repetitions of the alternating etching and passivation cycle. The first cycle is the ion-assisted etching of the silicon substrate by the etching gas (SF$_6$). Then, the sidewall passivation step is performed with polymer-producing gas (C$_4$F$_8$). The passivation cycle coats the sidewalls with a protective polymer, i.e. poly-tetra-fluoro-ethylene (PTFE), which prevents lateral etching. Next, the mixture of SF$_6$ and O$_2$ gas is used in a continuous etching step. The sidewalls remain protected by the Teflon-like polymer. (b) Silicon grass is formed due to micromask formation. which prevents lateral etching and keeps the vertical angle around 90° ± 2°.

The mixture of SF$_6$ and O$_2$ gas is applied in continuous etching steps. Oxygen ions etch the bottom polymer, while fluoride ions etch the silicon at the bottom of the through-hole. The sidewalls remain protected by the Teflon-like polymer. The continuous repetitions of such etching and passivation cycles result in highly anisotropic etching with a high etching rate.

It is noticed that the silicon grass is the by-product during the Bosch process, and most previous studies have focused on eliminating these silicon grasses. The silicon grass is formed due to the residual passivation layer, as shown in figure 1(b). It is caused by the C$_4$F$_8$ deposition, and since the passivation layer is not removed completely by SF$_6$ etching, it makes a micromask. These micromasks may remain with certain gas flows and thus produce silicon grass as an undesirable product.

However, in order to get a super-hydrophobic surface, the main purpose of this study is to retain the grass formation to obtain a rough surface instead of a smooth plane. By controlling the etching/passivation time and coil power carefully, we can find suitable parameters for the formation of a dense silicon grass structure on the silicon surface in the experiment.

2.2. Application to the capillary pumping device

The design of the micro-duct of this study is shown in figure 2. The channel has a height of 3 μm and a width of 100 μm. The fabrication process is shown in figure 3. The starting material is the n-type (1 0 0) orientation standard silicon wafer. The cleaning procedure consists of the following sequence of steps.

1. Remove insoluble organic contaminants with a 5:1:1 H$_2$O:H$_2$O$_2$:NH$_4$OH solution.
2. Dip in diluted 50:1 H$_2$O:HF solution to remove the residues of thin silicon dioxide layer and possible metallic contaminants as a result of step 1.

3. Remove ionic and heavy metal atomic contaminants using a solution of 6:1:1 H$_2$O:H$_2$O$_2$:HCl.
4. Piranha solution [H$_2$SO$_4$ (98%):H$_2$O$_2$ (30%) = 3:1 is used for 10 min for further cleaning.

All steps were separated by rinses with deionized water and, after the final rinse, the wafers were dried with nitrogen.

The fabrication proceeds in the following order. (a) After the cleaning process, the substrate was coated with hexamethyldisilazane (HMDS) to enhance adhesion of the photoresist (S1813). The HMDS was spin-coated on the wafer for 5 s at 500 rpm and followed by 25 s at 3000 rpm, and hard bake for 5 min at 100 °C. After that, the photoresist was spin-coated on the wafer and patterned by photolithography. The ICP etching process was used to form the spacer for channel height definition. The wafer was dipped in an acetone solution to remove the photoresist. (b) Then plasma enhanced chemical vapor deposition (PECVD) was used to deposit 0.5 μm silicon oxide to create the hydrophilic surface property. The photoresist (S1813) was patterned to
define the microchannel. (c) The wet etching process was used to remove the exposed silicon oxide, and the etchant [BOE:H2O2 (30%):3:2] was applied for 1 min. (d) The ICP process was used to generate silicon grass on the bottom to form the super-hydrophobic property. (e) Teflon was coated on each side of the Pyrex 7740 glass as the channel cover. (f) Finally, the top and bottom substrates were bonded with a clamping apparatus.

3. Results and discussion

In order to identify the factors that affect the surface roughness, we varied (i) the etching time and passivation time, and (ii) the coil power, while the platen power was fixed at 100 W, and the gas flow rates of SF6 and C4F8 were kept at 300 and 200 sccm, respectively. It is noted that the coil power supplied to the inductive coupler and platen power was from an additional generator powering the cathode on which the sample was placed.

3.1. Comparisons of different etching and passivation times

First, we consider the etching times of 2–12 s for gas SF6 and the passivation times of 2, 4 and 6 s for gas C4F8, respectively. The coil power was fixed at 800 W, and 300 etching cycles were applied. Figure 4 shows the results of contact angle variation with different etching and passivation times, where the contact angle is measured by the contact angle meter. It can be observed that a contact angle around 153° can be achieved when the etching time and passivation time are 4 s and 2 s, respectively. As a comparison, a smooth silicon surface has a contact angle of approximately 95°. This value is larger than the normal value 70°–75° (of clean water–silicon interface). This discrepancy may be a consequence of the diluted HF solution. The residual fluorine ions on the silicon surface may increase the contact angle. This indicates that after the ICP process, the surface indeed becomes super-hydrophobic due to the formation of a needle-like structure (silicon grass). As the etching time is increased gradually, the surface maintains the hydrophobic property, but the contact angle does not exceed 150°. This result reveals that with a longer etching time in one cycle, it was more difficult to generate the grass structure on the silicon surface because the passivation layer will be etched out completely by the subsequent SF6. In other words, the micromasks will be eliminated with a longer etching time and unable to form a silicon structure, so the surface will become flat.

With a passivation time of 6 s, it can be observed that the contact angle increases gradually with increased etching time. Figure 5 shows a scanning electron microscope (SEM) image of a sample with an etching time of 4 s, passivation time of 2 s and coil power of 800 W. From the figure, it can be observed that under these fabrication parameters, the ICP process generates the compact needle-like silicon grass on the surface of silicon. This structure can hold liquid on top of the grass by forming a stable air cushion between the liquid and the rough surface. Hence, the contact area can be minimized between the solid and liquid, yielding a super-hydrophobic surface with a contact angle around 153°. The height of the silicon grass is about 24 μm in this case.

Recall the Cassie model in equation (2). Assume that the contact angle of DI water on a smooth silicon surface is around 95°. We can obtain that the area fraction \( f \) is 0.11. In order to
Figure 5. SEM images of the silicon grass: (a) magnification of 1470; (b) magnification of 3690. The coil power is fixed at 800 W, platen power is fixed at 100 W, gas flow rates of SF₆ and C₄F₈ are kept at 300 and 200 sccm, respectively. The number of etching cycles is 300. The etching time is 4 s and passivation time is 2 s in this case. From the figure, it can be observed that the height of the silicon grass is around 24 μm.

Figure 6. (a) Microscope image of the top view of the silicon grass. The dark areas are the roots of the silicon grass, and the bright areas are the bottom surface. (b) Contact interface between the DI water and silicon grass is assumed to be located at half the height of the grass. The area fraction $f$ therefore can be estimated as 0.25 of the root area of grass.
verify this factor, an imaging technique was used to measure the distribution of silicon grass, as shown in figure 6(a). The image, taken by optical microscope from the top view, focuses on the root of the silicon grass. So the dark area should be the silicon grass root region; its fraction of dark area is around 0.38 from the image process technique. However, in order to estimate the area fraction $f$, we assume that the contact interface between the DI water and silicon grass is located at half of the height of the grass, as shown in figure 6(b). The grass distribution proportion can be estimated to be around 0.095. This value is quite close to the theoretical prediction of 0.11.

3.2. Comparison of different coil powers

Next, we varied the coil power from 200 to 1200 W at a fixed etching time of 4 s, passivation time of 2 s and 300 etch cycles. The experimental results are shown in figure 7. It is known that the coil power dominates the etching rate in the silicon surface and therefore increased simultaneously. The grass structure was generated on the surface of the silicon when the coil power was 800 W, and a contact angle around 153° could be obtained.

3.3. Comparison of driving efficiency

From the experiments, we determined the optimal parameters to form dense or compact silicon grass on the silicon surface. The flow rate of the etching gas, SF6, is 300 sccm for 4 s, the flow rate of the passivation gas, C4F8, is 200 sccm for 2 s, the platen power is 100 W, the coil power is 800 W and the etching cycle is 300 cycles. The resulting structure can sustain the DI water on top of it with a contact angle of 153°. We expected that the DI water frictional force of its contact area could be reduced by the super-hydrophobic interface. For this reason, we replaced the bottom Teflon surface in our original capillary pumping design with the silicon grass, and compared the driving efficiencies of these two designs.

The channel has a height $h = 3$ μm, width $w = 100$ μm and total length $L = 4$ cm. It is noted that the top cover was double-sided Teflon coated, because the ICP method cannot etch glass substrate operatively to form a super-hydrophobic surface. Therefore, the top design in this experiment is that of the original device. Figure 8 shows the experimental results with different hydrophobic interfaces, with DI water and blood used as the working fluids in the test. The data presented are averages of five experiments. It can be clearly observed that when the hydrophobic interface was Teflon, the DI water could be transported 18.3 mm in 15 s, as shown in figure 8(a). After replacement of the Teflon by the super-hydrophobic surface, it could flow farther, 22.1 mm, within the same period.

In the same plot, we also compare the measured data for our present and previous designs to the theoretical values. In previous studies, we derived simple equations which relate the flow velocity to the contact angles, surface tension and viscosity for DI water and blood. The detailed derivation can be found in [27, 25], and the velocity expression for DI water is

$$x(t) \approx \frac{1}{2}\sqrt{\frac{\gamma h}{24 \mu w t}} [w (\cos \theta_b + \cos \theta_t) - 2h], \quad (3)$$

where $w$ is the channel width, $h$ the channel height, $\mu$ the dynamic viscosity, $\gamma$ the surface tension, $\theta_b$ and $\theta_t$ the contact angles at the bottom and top interfaces, respectively. In this study, we take the channel width $w = 100$ μm, channel height $h = 3$ μm, dynamic viscosity $\mu = 1.002$ mPa s and surface tension $\gamma = 72.8$ dynes cm$^{-1}$ along with the contact angles $\theta_b = 20°$ and $\theta_t = 95°$. The theoretical curve is obtained by integrating equation (3) using the above-mentioned values of parameters.
It is noted that in deriving equation (3), the fluid is assumed to be fully confined over the hydrophilic stripe. Compared to our earlier design, the situation of the present design is closer to this assumption, as the super-hydrophobic silicon surface better prevents wettability of the water over its surface. This is indeed the case as we can see from the comparison in figure 8(a). The driving efficiency of the new design with the super-hydrophobic surface is closer to the theoretical value than the previous design with the Teflon surface. This evidence explains the advantage of using the super-hydrophobic silicon grass. This is because the Teflon surface used in the previous design may still generate frictional force due to the wetting area on the contact region. After replacing Teflon by the super-hydrophobic surface (the condition is closer to the ideal assumption), the frictional force is reduced and the measured data are reasonably close to the theoretical value.

The experimental results with whole blood as the working fluid are shown in figure 8(b). This figure shows that the displacement within 60 s was 17.3 mm for Teflon and 19.9 mm for silicon grass, respectively. The average velocity was 1.17 times that of the original design. Similarly, a simple equation for blood has been derived in [28] with the blood viscosity model [29], which gives a relation between velocity, contact angles, surface tension and viscosity. The conservation of momentum equation can be derived as follows:

\[
\frac{d}{dx} \left[ (M + \rho_{blood} w h x) \frac{dx}{dt} \right] = F_c + F_f,
\]

where \( M \) is the total mass in entrance, \( \rho_{blood} \) is the local density of whole blood, \( w \) is the channel width, \( h \) is the channel height, the distance measured from the entrance is \( x \). There are two force components: \( F_c \) the capillary force and \( F_f \) the viscous frictional force; they are given respectively by

\[
F_c = [w (\cos \phi_b + \cos \phi_t) - 2h] \gamma
\]

\[
F_f = -12 \eta_b \frac{w}{h} \frac{x}{h} \frac{\eta_{plasma}}{\kappa} \left( 1 + \frac{b}{\kappa} \right) \left( 1 - \frac{H}{H} \right) e^{\frac{-x}{2 h}},
\]

where \( \gamma \) is the surface tension, \( \phi_b \) and \( \phi_t \) denote the contact angle of blood at the bottom and top interfaces, respectively, \( \eta_{plasma} \) is the viscosity of plasma, \( \kappa \) is the shear rate, \( b \) and \( n \) are simple constants and \( H \) is the effective hemocrit along the channel. The shear rate \( \kappa \) can be estimated by \( \kappa = 2 u_{max} / h = 4x / h \); where \( u_{max} \) denotes the velocity at the center line of the channel.

In this study, we take the channel width \( w = 100 \, \mu m \), the channel height \( h = 3 \, \mu m \), the local density of whole blood \( \rho_{blood} = 1063 \, kg \, m^{-3} \), the surface tension \( \gamma = 79.6 \, dyne \, cm^{-1} \) along with the contact angles \( \phi_b = 18^\circ \) and \( \phi_t = 107^\circ \), viscosity of plasma \( \eta_{plasma} = 1.2 \, mPa \, s \), \( b = 6s^{-1} \) and \( n = 0.75 \) for human blood, the effective hemocrit \( H = 0.51 \).

As in the case for DI water, the measured data of the velocity for the new design with the super-hydrophobic interface are closer to the theoretical values for whole blood. This discrepancy here may be caused by some other blood property (such as aggregation, change of viscosity), which may increase the drag force when blood is flowing in the channel. Hence the velocity is still smaller than the theoretical value.

As deduced from the results, we know that the new design, using the super-hydrophobic interface to confine the channel sides, improved the driving efficiency substantially. It is perceived that the super-hydrophobic interface can effectively reduce the frictional force on the contact interface. To be thorough, we must also point out that when fluid flows in the hydrophilic oxide channel, the drop may partially contact the edge of the Teflon, as shown in figure 9(a). From the enlarged picture, it can be observed that although the Teflon is a hydrophobic surface, it still has a wetting area (water over a small part of Teflon) on the contact region along the channel and thus generates some frictional force. In contrast, figure 9(b) shows the silicon grass (super-hydrophobic property) surface that allows the formation of a stable air cushion beneath the fluid, and it can greatly reduce the wetting area to nearly the contact point, thus effectively reducing the frictional force. The profile is closer to our ideal expectation, and the driving efficiency is improved. This is the reason why the driving efficiency can be improved by 21% with the new interface.

4. Concluding remarks

In this study, we presented a simple method to fabricate a dense or compact silicon grass structure that is a super-hydrophobic surface on a silicon wafer. The needle-like structure can improve the driving efficiency of a capillary pumping microduct without sidewalls. Some important progress is reported in this paper.

(1) The silicon surface is cleaned only by the ICP method, without the need to employ any other lithography process.

(2) It was found that under suitable parameters (the flow rate of etching gas SF₆, 300 sccm for 4 s; the flow rate of passivation gas C₆F₈, 200 sccm for 2 s; the platen power, 100 W; the coil power, 800 W; and the etching cycles, 300) the ICP process produces a dense or compact silicon grass structure, which yields a super-hydrophobic surface with a contact angle of 153°.

(3) The silicon grass was used to replace the Teflon substrate in our previous capillary pumping design. The driving efficiency was shown to have improved by 21% and 17%
over the original design for transporting DI water and human blood, respectively.

It is noted that the new design eliminates the frictional force on the edge of the channel when the working fluid flows in. Hence, the improvement of the driving efficiency is quite significant. The measured data of the present design are closer to the values predicted by a theoretical analysis which relates the flow velocity to the contact angles, surface tension and fluid viscosity. It is also expected that if other even more super-hydrophobic interfaces are used, the driving efficiency for either the DI water or the whole blood would get closer to the theoretical value.

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