Actuation and Control of Droplets by Using Electrowetting-on-Dielectric

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Electrowetting-on-dielectric (EWOD) controls directly the wettability of liquids on the solid surface by applying an electric potential to the microelectrode array under the dielectric layer. A prototype of the EWOD droplet actuator is put forward, consisting of Si used as the substrate of the microelectrode array, Si_3N_4 film as the dielectric layer and fluorocarbon polymer (p-C:F) film deposited by plasma enhanced chemical vapour deposition (PECVD) as the hydrophobic layer. The p-C:F film was uniform and compact, and the contact angle of deionized water on the p-C:F film reached 110°. The actuator successfully actuated deionized droplets surrounded in silicone oil at the voltage of 35 V.

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Micro total analysis systems (μTAS) or laboratory on a chip (LOC) has been widely applied to many fields, such as chemical analysis, biochemical sensing, drug delivery, molecular separation, amplification, sequencing and synthesis of nucleic acids, environmental monitoring, and others. The manipulation and control of microlitre or nanolitre liquids are the basis of μTAS. Early research focused on the manipulation of continuous-flow. In recent years, however, a major research effort has been directed towards the manipulation and control of discrete droplets. Similar to the traditional operations in biochemical analysis laboratories, the system of discrete droplets can realize the biochemical analysis processes in LOC. In contrast to continuous-flow, little or no excessive liquids are required to prime or fill microchannels so that much higher utilization of sample and reagent volumes is possible.

To date, several structures of manipulating droplets have been proposed, including the use of air pressure,[1] asymmetric electrode arrays,[2] thermocapillarity effect,[3,4] dielectrophoresis,[5] electrostatic forces,[6] and electrowetting-on-dielectric (EWOD).[7,8] EWOD directly changes the wettability and local contact angle of droplets on the solid surface by changing the electric potential applied to the microelectrode array under the dielectric layer, and thus results in the asymmetric deformation of droplets to realize the actuation and control of droplets. Compared to other ways, EWOD realizes the integration and automation of creating, transporting, merging, and cutting droplets, and thus builds a solid foundation for droplet-based LOC.

In all reported EWOD droplet actuators, the microelectrode array was fabricated on a glass substrate, and the key hydrophobic film directly contacting liquids was fabricated by spinning Teflon® AF 1600. Owing to the high price and low electric breakdown field of Teflon® AF 1600, other dielectric films, such as Parylene C and SiO_2, had to be fabricated under a glass substrate first. Furthermore, because the glass substrate could not stand high temperature, the films had to be fabricated in low temperature, which gave the films the problems of loose structure, poor insulation and large leakage current.[7,8] The prototype of the EWOD droplet actuator is developed, in which Si is used as the substrate of the microelectrode array, the Si_3N_4 film as the dielectric layer, and the p-C:F film deposited by PECVD as the hydrophobic layer.

The perspective and cross-sectional image of the EWOD droplet actuator is shown in Fig. 1. A deionized droplet is sandwiched between two parallel planar electrodes. The top plate is a single continuous ground electrode, while the bottom plate is an independently addressable microelectrode array. To avoid direct contact between droplets and electrodes and to acquire good electric breakdown performance, a thin insulator film is deposited on the bottom plate as the dielectric layer. The surfaces of the two plates are coated with a thin p-C:F film, which can increase the initial contact angle of droplets on the solid surface. The droplet size and the gap between the two plates are controlled such that the footprint of the droplet can overlap at least three adjacent electrodes while also making contact with the top plate. The medium surrounding deionized droplets is an immiscible liquid (silicone oil) to prevent the evaporation of liquids and to reduce the resistance during the droplets movement.

Initially, all the electrodes are grounded and the contact angles around the droplet are the same, where we ignore the effect of the gravity on droplets. Based

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on the Lippmann equation,[9] the surface tension at solid/droplet interface $\gamma_{\text{sol-drop}}$ is determined by the electric potential applied to the interface,

$$\gamma_{\text{sol-drop}}(V) = \gamma_{\text{sol-drop}}(0) - \frac{C}{2}V^2, \quad (1)$$

$$C = \frac{\epsilon\epsilon_0}{d}, \quad (2)$$

where C, ϵ, d are the capacitance per unit area, the dielectric constant and the thickness of the dielectric layer, respectively; V is the electric potential applied to electrodes. At the solid–oil–droplet three-phase contact line, the relation between contact angles of droplets on the solid surface θ and the surface tensions is given by Young's equation,

$$\cos\theta = \frac{\gamma_{\text{sol-oil}} - \gamma_{\text{sol-drop}}}{\gamma_{\text{oil-drop}}}, \quad (3)$$

where $\gamma_{\text{sol-oil}}$ is the surface tension at solid/oil and $\gamma_{\text{oil-drop}}$ the surface tension at oil/droplets; $\gamma_{\text{sol-oil}}$ and $\gamma_{\text{oil-drop}}$ are constant and independent of the applied potential.[9] Thus the relation between the contact angle θ and the potential V is

$$\cos\theta(V) = \cos\theta(0) + \frac{1}{2}\frac{1}{\gamma_{\text{oil-drop}}}CV^2. \quad (4)$$

From Eq. (4), the contact angle is a function of the applied potential. The actuation force applied to the droplets is related to the difference between $\theta(0)$ and $\theta(V)$. If the applied potentials remain the same, the more the initial contact angle of droplets on the solid surface is, the more the change of the contact angle is, and thus the more the actuation force applied to droplets is. As shown in Fig. 1, the droplet overlaps three adjacent electrodes. The potential is applied to the right electrode, while the middle and left ones are still grounded. The applied potential reduces the surface tension at solid/droplets interface $\gamma_{\text{drop-sol}}$, so does the contact angle of droplets on the solid surface above the right energized electrode. However, the contact angle above the left electrode keeps constant without the potential. The asymmetric deformation of the droplet, which establishes a pressure gradient between two ends of the droplet, gives rise to the droplet movement towards the right energized electrode.

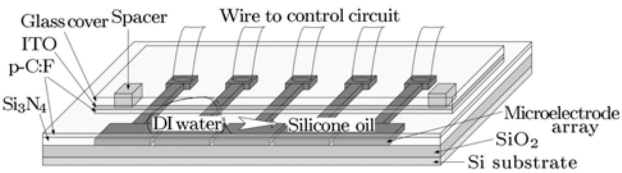

Fig. 1. Perspective and cross-sectional image of the EWOD droplet actuator.

Here Si was used as the substrate of the microelectrode array in the bottom plate. Firstly, 6000Å SiO_2 was thermally grown at 1050°C. Then 200Å Ti and 1800Å Pt were sputtered onto SiO_2 and were patterned to form the microelectrode array by a lift-off process. The size of each electrode was $1.4 \times 1.4\,\text{mm}^2$. The electrodes were coated with a 2800Å Si_3N_4 film deposited by low pressure chemical vapour deposition (LPCVD) as the dielectric layer. The permittivity of Si_3N_4 is about 7, which is about 4 times and twice of that of Parylene C ($\epsilon \approx 2$) and SiO_2 ($\epsilon \approx 4$), respectively. The actuation force applied to droplets is directly proportional to the capacitance of the dielectric layer.[9] Thus, if the thicknesses of all films are the same, Si_3N_4 film has the performance better than Parylene C and SiO_2. Lastly, a 200Å p-C:F film was deposited on the Si_3N_4 film as the hydrophobic layer by PECVD on the conditions with a C_4F_8 flow rate of 5sccm, the deposition pressure of 10 Pa, and rf power of 100 W at room temperature.

Figure 2 presents the AFM image of the surface of the p-C:F film. The surface of the film was uniform and compact. The peak-to-peak roughness of the surface of the film was about 20 nm and the root-mean-square roughness was about 1 nm. The contact angle of deionized water on the p-C:F film measured by the microscope was about 110°, as shown in Fig. 3. The contact angle of deionized water on Teflon® AF 1600, which is the best hydrophobic material acquired from the business, is 103°–120°.[7,8] Thus, the wettability of the p-C:F film deposited by PECVD is similar to that of Teflon® AF 1600. Furthermore, the uniformity and conformality of the p-C:F film is better than that of Teflon® AF 1600, which may be used for the large-scale production in the foundry.

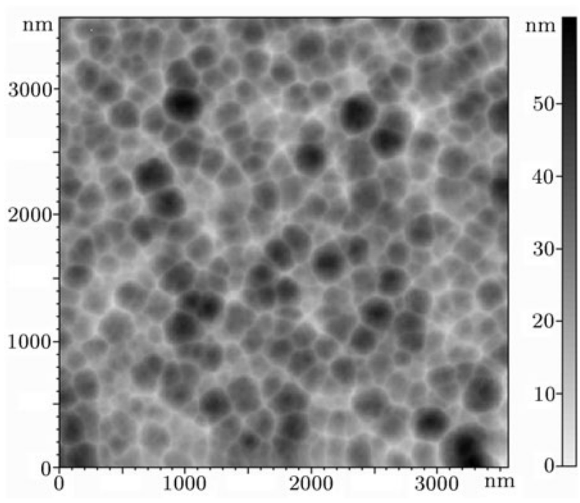

Fig. 2. Image of the surface of the p-C:F film measured by an AFM.

The top ground plate of the actuator was a plate of glass coated with a conducting layer of transparent indium–tin-oxide (ITO). Similarly, a 200Å p-C:F film was also deposited on ITO by PECVD as the hydrophobic layer. Since the materials of the top plate were transparent, it was convenient to observe the droplets movement from the top of the actuator. Both p-C:F films on the microelectrode array and the ground plate served to provide a hydrophobic surface that enabled large changes in contact angle. Furthermore, the p-C:F film above the microelectrode array also acted as the dielectric layer to block the electron transfer and to acquire good electric breakdown performance.

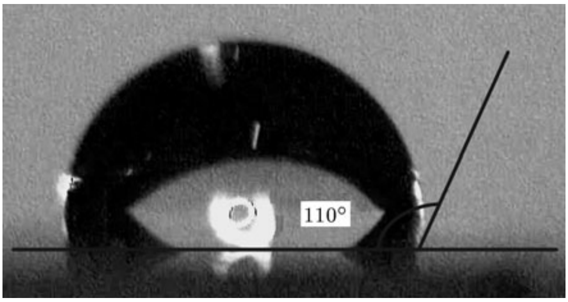

Fig. 3. Contact angle of deionized water on the p-C:F film taken by the microscope.

The top plate was supported by the spacer, which was made by double-stick tapes and its thickness determined the gap spacing between two plates. The results showed that the gap spacing of 150 μm could obtain the good actuation performance. When the actuator was tested, a droplet about 1 μL was dispensed using a pipet onto the surface of the microelectrode array in the bottom plate, which was covered by silicone oil, and then the top plate was placed onto the spacer.

To actuate the droplets without electric breakdown of the p-C:F film and Si_3N_4 film, the pulse voltage, whose frequency and duty-ratio were changeable, was applied to the microelectrode array. The pulse voltage was controlled by the microcomputer system built by ourselves, including the dc power, the photoelectronic relay, the single-chip microcomputer and its peripheral circuits. The appropriate pulse voltage applied to electrodes was generated by amplifying the signal from the single-chip microcomputer, whose frequency and duty-ratio were changeable, by the photoelectronic relay through opening/closing the path between electrodes and the power/ground.

The results showed that when the voltage was 5 V, the obvious phenomenon of electrowetting was observed, and the oscillation frequency of droplets could follow that of the pulse voltage. When the voltage exceeded 30 V, the rapid and repeatable droplets movement was observed. The higher the applied voltage was, the more rapid the droplets movement was. However, it is easier for higher voltage to cause the dielectric layer electric breakdown. Figure 4 presents the three video frames of a moving droplet at the 35-V voltage, 2-Hz frequency, and 1:1 duty-ratio.

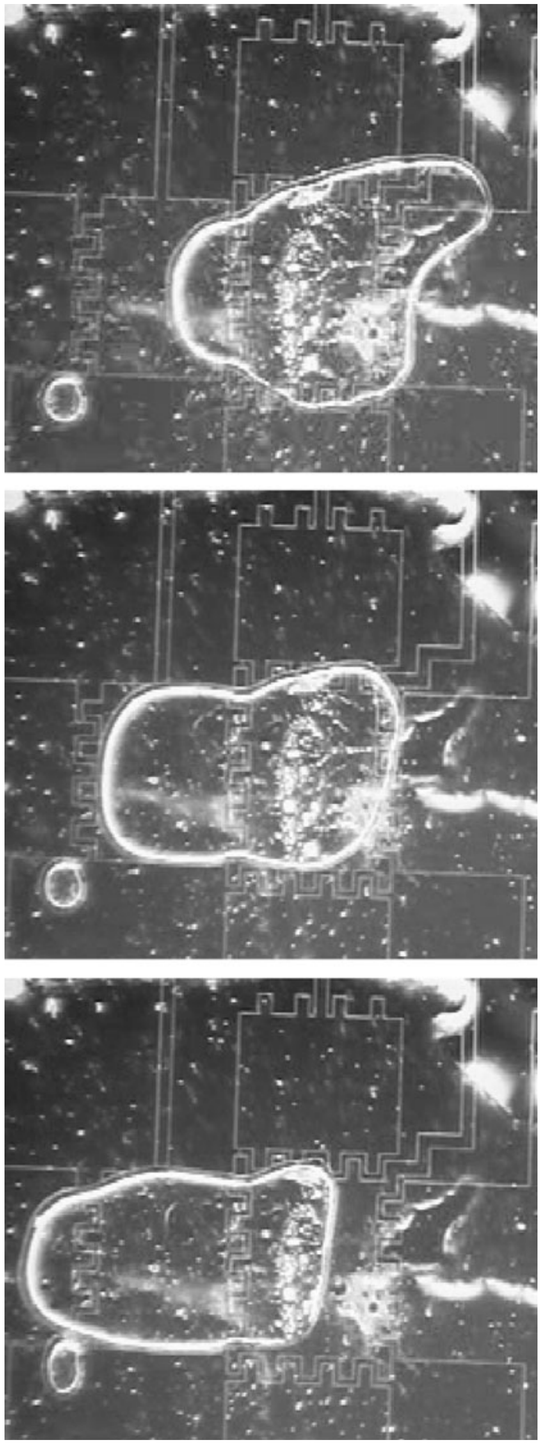

Fig. 4. Three video frames of a moving droplet at 35-V voltage, 2-Hz frequency, and 1:1 duty-ratio.

Droplets were trapped by minute granules on the surface of the bottom plate during the droplets' movement, which were mainly introduced during the processes of sputtering the metal film and depositing Si_3N_4 film. These granules not only impeded the droplets' movement, but also caused the p-C:F film and Si_3N_4 film electric breakdown where the actual electric field was far less than its electric breakdown field, and the actuator was invalid. At present, we are decreasing and eliminating the effect of minute granules on droplet movement by improving the processes, the materials and the structure of the EWOD droplet actuator.

In summary, we have fabricated an EWOD droplet actuator consisting of top and bottom plates. On the bottom plate, Si was used as the substrate of the microelectrode array, the Si_3N_4 film as the dielectric layer, the p-C:F film deposited by PECVD as the hydrophobic layer, while the glass plate with ITO was used as the top ground plate. The p-C:F film deposited by PECVD was uniform and compact, and had good hydrophobicity of deionized water. Deionized droplets surrounded in silicone oil were successfully actuated at the voltage of 35 V. The EWOD droplet actuator has the potential to realize the simultaneous manipulation of several droplets and makes available the process of creating, merging and separating droplets on an $M \times N$ planar microelectrode array by improving the processes and structure of the actuator.

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