

Reducing electrowetting-on-dielectric actuation voltage using a novel electrode shape and a multi-layer dielectric coating

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Abstract This paper presents design and fabrication of an electrowetting-on-dielectric (EWOD) device using a novel electrode shape and a multi-layer dielectric coating that reduce the actuation voltage of the device to less than 12.6 V. The fabrication of the EWOD electrodes is carried out in several steps including laser exposure, wet developing, etching, and stripping. A high-dielectric-constant multi-layer dielectric coating containing a 770 nm thick Polyvinylidene difluoride (PVDF) layer and a 1 µm thick Cyanoethyl pullulan (CEP) layer, is deposited on the EWOD electrodes for insulation. This multi-layer dielectric structure exhibits a high capacitance per unit area, and the novel electrode shape changes the actuation force at the droplet contact line reducing the voltage required to operate the device. In addition, an overlaying Teflon layer of 50 nm is placed on top of the dielectric structure to provide a hydrophobic surface for droplet manipulation. It is observed from the experiments that the electrode shape and the dielectric structure have contributed to the reduction of the actuation voltage of the EWOD device.

1 Introduction

Manipulation of fluids in micro-engineered systems is becoming increasingly desirable for a range of applications including chemical and biological analysis. Despite the reduction from millilitre to microlitre volumes offered by microfluidics compared to traditional laboratory processing

(Mohammed and Desmulliez 2014), it is becoming increasingly desirable to reduce the volumes further and directly deal with individual droplets. Directly sampling droplets is advantageous because of the use of less reagents for a biological/chemical assay, the reduction of the produced waste, and the advantage of surface tension effects for fluid manipulation.

Several approaches including light (Pei et al. 2015; Park et al. 2010; Pei et al. 2010), magnetic force (Zhang and Wang 2012; Okochi et al. 2010; Long et al. 2009), and surface acoustic wave (Ding et al. 2013; Wang and Zhe 2011; Bourquin et al. 2010) have been used for droplet manipulation. Although these approaches provide satisfactory accuracy and good reliability, various limitations such as slow response time, complex fabrication process, and fluid leakage have been observed during their operation. Electrowetting-on-dielectric (EWOD) is a technique that enables manipulation of microlitre volumes of fluid without any physical damage to constituents with the liquid (proteins, cells, etc.). The additional advantages of EWOD devices are: ease of fabrication, small size, no requirement for heat or light (Shah et al. 2010; Sohail et al. 2011), less chance of early electrochemical breakdown, less chance of cross-mixing of fluids across the interface (Ahamedi et al. 2013), negligible leakage, flexibility, reliability, reversibility, high accuracy, fast switching, and low cost.

An EWOD is the voltage-based manipulation of the wettability of liquid on a solid surface (Quinn et al. 2005). An EWOD device includes a base substrate, an array of conducting electrodes, a dielectric layer, and a hydrophobic surface. In such a device, a conducting droplet is placed and manipulated on the hydrophobic surface by sequentially applying an actuation voltage to the conductive electrodes. The applied voltage generates electric charges on the insulating surface resulting in a net force upon the

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droplet. This force changes its contact angle, and beyond a critical value, leads to movement of the droplet. The correlation between the contact angle and the applied voltage is given by Lippmann-Young equation, which depends on the interfacial surface tension of droplet-filler medium and dielectric properties, and is expressed as (Berge 1993):

$$\cos \theta_v = \cos \theta_0 + \frac{\varepsilon_0 \varepsilon_{rd} \varepsilon_{rh} V^2}{2\gamma_{df}(t_d \varepsilon_{rh} + t_h \varepsilon_{rd})} \quad (1)$$

where θ_0 is the initial contact angle, θ_v is the generated contact angle after applied voltage, ε_0 is the permittivity of the free space, ε_{rd} is the relative dielectric constant of the dielectric layer, ε_{rh} is the relative dielectric constant of the hydrophobic layer, V is the applied voltage, γ_{df} is the droplet-filler medium interfacial surface tension, t_d is the thickness of the dielectric layer, and t_h is the thickness of the hydrophobic layer.

EWOD has played an important role in various system including drug delivery (Samad and Kouzani 2014), cell culture (George and Moon 2015; Witters et al. 2011), and lab-on-a-chip (Wang et al. 2014). However, a major limitation of the EWOD technology is the required actuation voltage for existing devices, which has been reported to be high in the range of 30–300 V (Ahamed et al. 2013; Chandee et al. 2012; Sohail et al. 2014; Bansal and Sen 2014; Caputo et al. 2013). An EWOD device operating at high actuation voltage produces extra heat in the device, which evaporates the droplet instead of moving it (Accardo et al. 2013; Shabani and Cho 2013). Moreover, the high actuation voltage brings in the risks associated with high voltage operation. In this work, we aim to overcome this technological shortcoming and present an EWOD device capable of low voltage operation, through the optimised fabrication of novel electrode patterns and a multi-layered dielectric coatings.

Among the wide range of reported EWOD works, several groups have focused on creating low actuation voltage devices. Bormashenko et al. (2014) designed a low actuation voltage EWOD device based on lubricated honeycomb polycarbonate surfaces. The surface was made with the breath-figure and self-assembly technique, where silicon (Si) and castor oils were used. Using Si oil and castor oil, the EWOD device operated at the minimum actuation voltages of 35 and 80 V, respectively. Moreover, a low voltage coplanar EWOD device was reported by Li et al. (2009). The device was fabricated using an anodic tantalum pentoxide (Ta_2O_5) dielectric layer and a thin aFP (amorphous Fluoropolymer) Cytop hydrophobic layer. It was mentioned that a successful droplet movement was achieved with an applied voltage of 15 V where the electrode dimension and the gap distance between the electrodes was $1 \times 1 \text{ mm}^2$ and $3 \text{ }\mu\text{m}$, respectively. In another scheme, Vasudev and Zhe

(2009) proposed an EWOD microgripper to manipulate the micro-size of objects. The microgripper used interdigitated coplanar gold electrodes, and the gap distance between the electrodes was $20 \text{ }\mu\text{m}$. To achieve a low driving voltage, a $0.5 \text{ }\mu\text{m}$ thin barium strontium titanate (BST) dielectric layer was embedded on the interdigitated electrodes. The result showed that the lifting force of $0.1 \text{ }\mu\text{l}$ droplet could be as high as $213 \text{ }\mu\text{N}$ at a driving voltage of 28 V. On the other hand, a parallel-plate configuration based low actuation voltage EWOD device for picolitre droplet manipulation was presented by Lin et al. (2010). The bottom plate of the device contained $100 \text{ }\mu\text{m}$ of electrode pitch, a multi-layer dielectric consisting of 135 nm Ta_2O_5 and 180 nm parylene C, and 70 nm of Cytop hydrophobic layer. The top plate was made of indium-tin-oxide (ITO) ground electrode, covered with a 70 nm thick Cytop layer only. To define a gap for the droplet, a $5 \text{ }\mu\text{m}$ spacer was used. The work reported that the EWOD device with multi-layer dielectric coating reduced the actuation voltage to 7.2 V using a 300 pl of Deionized (DI) water droplet. The droplet actuation was carried out in Si oil. Whilst the achieved actuation voltage is very low, it should be noted that decreasing the electrode size will reduce the actuation voltage. The obtained 7.2 V is for a very small electrode size ($100 \text{ }\mu\text{m}$) suitable for picoliter droplet manipulation only. The focus of this work is microliter droplet manipulation. Also, Silicone oil medium decreases the actuation voltage compared with air. Another low actuation voltage parallel-plate EWOD device was fabricated based on a 700 \AA of BST with 200 \AA Teflon coating (Moon et al. 2002). The device actuated 460 nl of water droplet by an applied voltage of 15 V where the electrode size was $1.4 \times 1.4 \text{ mm}^2$ and the gap between top and bottom plate was 0.15 mm .

Overall in order to reduce the actuation voltage, most of the existing works have focused on varying the dielectric material. Only a few works (Lin et al. 2010) explored the use of multiple layers of dielectric materials. Moreover, the existing works have not considered varying the electrode shape to decrease the actuation voltage. On the other hand for microliter droplet manipulation, in this study we focus on: (1) the development of a multi-layer dielectric coating with two materials that have not been used together previously, and (2) the effect of electrode shape change on reducing the actuation voltage of the coplanar EWOD device.

2 Materials and methods

2.1 Electrode design and fabrication

Electrodes of different shapes including square (Fair 2007), interdigitated (Chen et al. 2012; Jang et al. 2009), crescent

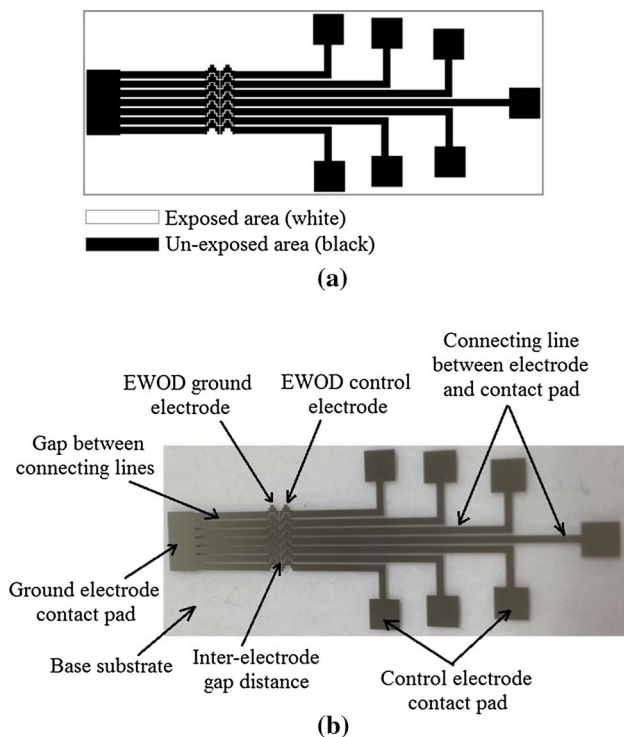


Fig. 1 **a** Mask design for the electrode shape, and **b** Cr patterned single plate EWOD design after photolithography

(Rajabi and Dolatabadi 2010), interlocking (Lin et al. 2010), and wedge (Li et al. 2009) have been used to operate EWOD devices. In this work, a novel electrode shape was designed and fabricated to realise an EWOD device operating with a lower actuation voltage. The fabrication process is initiated by designing a mask using a layout editing software (LayoutEditor, Juspertor UG, Germany). In the design process, several parameters including electrode shape, size, inter-electrode gap distance, connecting line, and contact pad have been considered to obtain a desired design of the EWOD device. The final, optimised EWOD device consists of seven control electrodes and seven ground electrodes. The dimension of the electrodes and the inter-electrode gap distances are fixed to $1.4 \times 1.4 \text{ mm}^2$ and $50 \mu\text{m}$, respectively. Figure 1a shows the optimised electrode mask for the EWOD device.

The proposed EWOD electrode design was fabricated by direct laser lithography exposure. Although the laser lithography process requires more exposure time than the conventional optical lithography process for fabricating an optical mask, the laser lithography process has higher fabrication resolution compared with the conventional optical lithography process. Consequently, smaller and finer electrodes, and also electrodes with specific shapes (e.g., the electrode shape shown in Fig. 1) could be fabricated. To fabricate the proposed device, a soda-lime glass substrate of 1.1 mm thickness was chosen as a base substrate. Upon the glass, an

8 nm layer of Chromium-oxide (CrO) and a 250 nm layer of Chromium (Cr) are deposited by electron-beam vapour technique (Clean Surface Technology, Japan). Here, the CrO layer acts as a barrier layer to protect Cr erosion by sodium elution from soda-lime glass and Cr layer works as the conducting layer. Moreover, an additional 30 nm thick layer of CrO is coated on top of the Cr layer which works as an anti-reflecting layer to protect the laser lithography machine from laser reflections on Cr surface during exposure. Finally, a photoresist coating is required as a forth layer to transfer the pattern of the mask on the conducting Cr layer. A positive photoresist of AZ 1500 (AZ Electronic Materials, Germany) was selected and a 500 nm thick layer was deposited on the CrO layer by spin-coating (WS-400BZ-6NPP/LITE, Laurrell Technologies Corporation, USA).

The designed mask and AZ 1500-Cr-CrO coated glass substrate then undergo a photolithography phase which is carried out by using a laser lithography machine (Heidelberg Instruments μPG101 , Japan). Initially, the designed mask is exported into the μPG101 PC software and the laser lithography machine exposes the photoresist coated substrate according to the mask pattern. The duration of the laser exposure was approximately 4 h. After the laser exposure, the substrate is required to go through three wet etching stages including development, etching, and photoresist stripping. In the development stage, 1:4 diluted AZ 351B developer solution (AZ Electronic Materials, Germany) is used to remove the photoresist from the exposed area of the substrate. In the second stage of the wet etching processes, the substrate is immersed into a Chromium etchant (651826, Sigma-Aldrich, Australia) for removing the unwanted Cr-CrO from the exposed area. In the third stage, the remaining photoresist from the un-exposed area is removed by immersing the substrate into AZ 100 remover (AZ Electronic Materials, Germany). Moreover, for removing the anti-reflecting (CrO) layer from the un-exposed region of the substrate, the Chromium etchant is used again for only 10–15 s. Here, the most important factor for removing CrO while keeping Cr intact is the immersing time in the etchant solution. If the immersing time is more than 15 s, the underneath Cr electrode pattern can be ruined. After the wet etching stages, a clear glass appears in the exposed region while the un-exposed region remains Cr according to the designed conducting pattern of the EWOD device as shown in Fig. 1b. This is a single-plate EWOD design where control electrodes and ground electrodes are placed to maintain a $50 \mu\text{m}$ inter-electrode gap distance. As can be seen in Fig. 1b, the EWOD control electrodes are joined to several control contact pads which allow for the individual spatial addressing of a supply voltage to each electrode. Conversely, all ground electrodes are joined together and connected with a single ground port of the supply voltage.

2.2 Deposition processes of multi-layers dielectric and hydrophobic coatings

The fabrication process of the device was completed by embedding dielectric and hydrophobic layers on top of the Cr electrode surface. The dielectric layer is required to isolate the conducting Cr electrodes from the droplet and the hydrophobic layer to minimise wetting of a droplet upon the EWOD device. Before electrode insulation, the control and ground contact pads of the Cr patterned EWOD design are covered by polyimide tape (RS Components Pty Ltd, Australia) which prevents the dielectric coating from insulating the active voltage contact points. An optimum dielectric coating was developed using a multi-layer approach by stacking layers of Polyvinylidene difluoride (PVDF) and Cyanoethyl pullulan (CEP). The main advantage of the dielectric layers is that both materials have high dielectric permittivity ($\epsilon \sim 7.6$ – 11.6 for PVDF, and $\epsilon \sim 20$ for CEP). As the dielectric strength of the PVDF material is lower than that of CEP, we deposited PVDF underneath CEP. In order to deposit PVDF, a 5 % (wt/wt) solution is made by dissolving PVDF powder (Mr $\sim 530,000$, Sigma Aldrich, Australia) in both Tetrahydrofuran (THF) and *N,N*-Dimethylformamide (DMF) solvents (Sigma Aldrich, Australia). The PVDF solution is then spin coated at a speed of 500 rpm for 5 s, followed by 1500 rpm for 60 s and 2000 rpm for again 60 s. Finally, the sample is annealed at 60 °C for

30 min on a hot-plate. The thickness of the deposited PVDF layer on Cr electrodes was measured using the 3D stack function of an optical microscope (OLYMPUS BX 50, USA) by focusing it at the dielectric surface and the glass substrate. The measured thickness of the PVDF layer was 770 nm. The deposited PVDF layer observed through the optical microscope shows the non-uniformity of its surface, as indicated in Fig. 2a. Due to the non-uniformity and pinholes on the PVDF surface, CEP dielectric material is selected as a second dielectric coating to remove these defects in PVDF layer to yield a more high quality surface uniformity. A 15 % (wt/wt) CEP solution is synthesised from granular CEP (Biddle Sawyer Corporation, USA) dissolved in DMF. 1 μm thick CEP layer is deposited by spin-coating the solution at a speed of 500 rpm for 5 s, followed by 2000 rpm for 90 s (Fig. 2b). The substrate is then dried in the vacuum environment for about 3 h to remove any trapped air-bubbles from the CEP surface and finally baked at 60 °C for 30 min for complete cure. The final multi-layer dielectric coating of 770 nm PVDF and 1 μm CEP, placed upon the Cr electrodes, is shown in Fig. 2c.

The dielectric layer is found to be relatively hydrophilic (contact angle of $\theta_c < 60^\circ$), leading to wetting of the EWOD surface and spreading of a deposited droplet. Therefore, a Teflon-AF layer is applied on top of the CEP layer to improve the surface hydrophobicity and to allow for droplets to form with $\theta_c > 120^\circ$. Teflon-AF (DuPont,

Fig. 2 Electrode insulation using a multi-layer dielectric and hydrophobic coatings

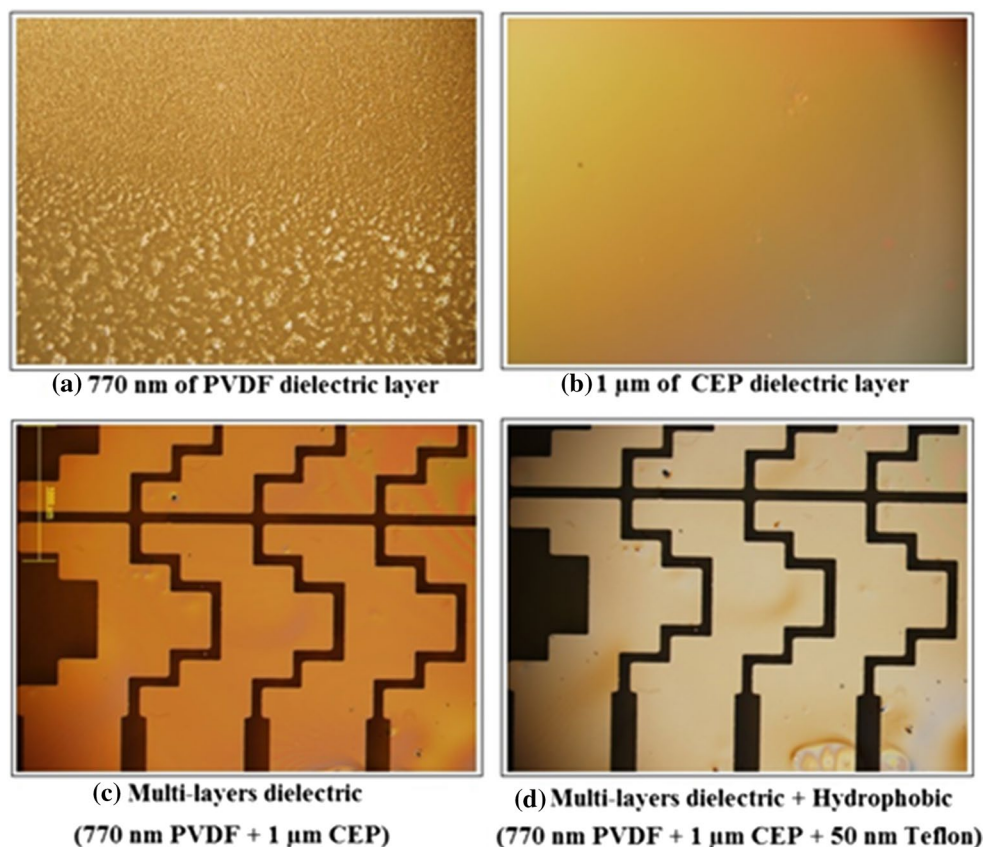
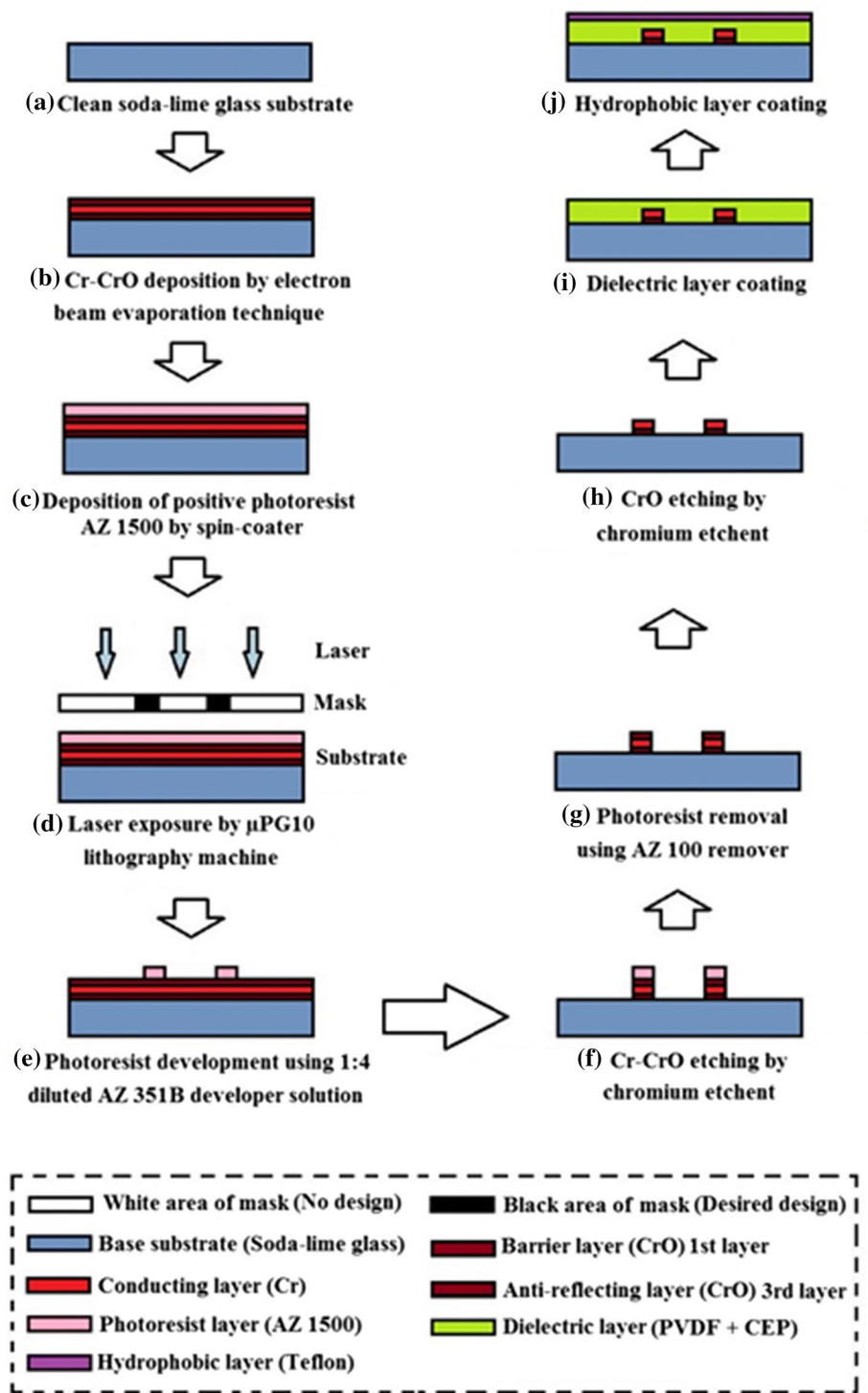


Fig. 3 Outline of the fabrication procedure of the EWOD device



USA) of 1 % wt/wt in Fluorinert FC-40 is spin coated at a speed of 2000 rpm for 60 s and baked at 60 °C for 25 min, resulting in a 50 nm layer thickness. The complete EWOD structure, comprising electrodes with dielectric and hydrophobic layers can be seen in Fig. 2d. The entire fabrication process of the proposed EWOD device is chronologically presented in Fig. 3.

3 Results and discussions

In order to observe the droplet motion on the fabricated EWOD surface, a conducting droplet from 1 M KCL solution is generated using a variable micropipette of 2–20 μ l (PV-20, Labtak, Australia). The volume of the droplet was chosen in such a way that the footprint of the generated

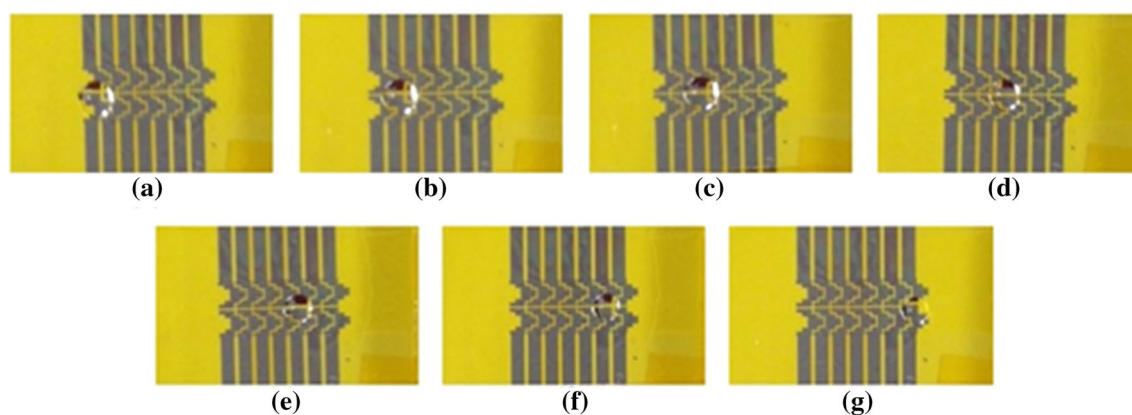


Fig. 4 EWOD droplet movement at the voltage of 12.5 V: **a** initial, and **b–g** position changes after applied voltage












droplet would become slightly larger than the electrode size. A 2.5 μl volume of droplet is placed between the upper most control electrode and the ground electrode. The EWOD device is then connected to a DC power supply and a potential difference is applied between the control and ground electrodes in contact with the droplet. Initially, the contact angle of the 2.5 μl droplet was $\theta_0 = 123^\circ$. An optical contact angle meter (KSV Cam 101, USA) was used to measure the contact angles of the droplet. When a voltage is applied to the neighbouring EWOD electrode, the droplet moves to the actuation electrode due to the resulting change in its contact angle. For the proposed device, the minimum actuation voltage required to initiate the droplet movement was 12.5 V. It was observed that the droplet moved successfully from the first electrode to the last electrode when 12.5 V was applied to the respective electrodes. Figure 4 shows a series of images of the droplet movement along the electrodes at a minimum actuation voltage of 12.5 V.

To verify the performance of the proposed EWOD device, various EWOD devices with different electrode shapes and different dielectric layers including single-layer and multi-layer coating were constructed and tested. Table 1 presents the minimum achieved droplet actuation voltages for different types of EWOD devices. In this experiments, Cr electrodes of $1.4 \times 1.4 \text{ mm}^2$ dimension with 50 μm inter-electrode gap distance and 50 nm Teflon-AF hydrophobic layer were kept constant. When the square-shaped Cr electrodes with different single-layer dielectric coated EWOD devices were examined, the experimental results proved that the required actuation voltage decreases as the thickness of the dielectric layer declines. Moreover, Table 1 states that a dielectric layer having a high dielectric-constant can operate an EWOD device at a low actuation voltage. Thus, the high dielectric-constant of 1 μm thin CEP layer allows the EWOD device to be operated with a minimum actuation voltage of 15.83 V. However, there is a fundamental limit on how thin a single dielectric layer can

be in the EWOD device because of dielectric breakdown. If the deposited dielectric layer is very thin, it may produce a porous surface. Therefore, the droplet would be in direct contact with the underlying electrode. As a result, the current would flow in the droplet and cause electrolysis as well as burn the underneath electrode. To solve this problem, a multi-layer dielectric coating was applied. The second dielectric layer of the multi-layer dielectric filled the porous gaps of the first dielectric layer, so that the desired properties of the surface (such as low roughness, low level of porosity, and suitable thickness) were obtained. Moreover, the droplet cannot be in direct contact with the underlying electrode, and hence the possibility of electrolysis during the experiment is close to zero. Thus, the EWOD device with the multi-layer dielectric coating can be reused many times providing the same result. Although the multi-layer dielectric coating increased the thickness of dielectric layer, the actuation voltage of the EWOD device was relatively reduced compared to single-layer dielectric, due to the combined effect of their dielectric-constants. Thus, the EWOD device with 770 nm of PVDF and 1 μm of CEP based multi-layer dielectric coating required 15.62 V of minimum actuation voltage, where as a single-layer dielectric of 770 nm of PVDF coated EWOD device occurred dielectric breakdown during operation and 1 μm of CEP coated EWOD device operated at a minimum actuation voltage of 15.83 V.

The effect of the electrode shapes on the actuation voltage was also investigated. Considering an electrode dimension of $1.4 \times 1.4 \text{ mm}^2$, an inter-electrode gap distance of 50 μm , a multi-layer dielectric of 770 nm of PVDF and 1 μm of CEP, a hydrophobic layer of 50 nm of Teflon-AF, and a droplet volume of 2.5 μl of 1 M KCL solution, the square-shaped and the proposed-shaped electrodes-based EWOD devices were analyzed. It is observed from Table I that the proposed EWOD device, based on a novel electrode shape and a multi-layer dielectric coating of PVDF

Table 1 Achieved minimum actuation voltage for different EWOD configurations

| Shape of EWOD Electrode | Dielectric Layer | Types of Dielectric Layer | Achieved Minimum Actuation Voltage |
|---|--|---------------------------|------------------------------------|
|  | 770 nm of PVDF layer | Single-layer dielectric | Dielectric breakdown |
|  | 845 nm of PVDF layer | Single-layer dielectric | 18.9 V |
|  | 2.4 μm of PVDF layer | Single-layer dielectric | 38.14 V |
|  | 5 μm of PVDF layer | Single-layer dielectric | 59.62 V |
|  | 1 μm of CEP layer | Single-layer dielectric | 15.83 V |
|  | 1.05 μm of CEP layer | Single-layer dielectric | 16.5 V |
|  | 1.1 μm of CEP layer | Single-layer dielectric | 18.27 V |
|  | 5 μm of CEP layer | Single-layer dielectric | 37.85 V |
|  | 770 nm of PVDF layer + 1 μm of CEP layer | Multi-layer dielectric | 15.62 V |
|  | 1.05 μm of CEP layer | Single-layer dielectric | 13 V |
|  | 770 nm of PVDF layer + 1 μm of CEP layer | Multi-layer dielectric | 12.5 V |

and CEP, obtained the lowest actuation voltage amongst all other EWOD devices examined. The reason for this reduction of the actuation voltage was that the sides of the two adjacent electrodes were placed together in such a way that minimized the inter-electrode gap distance between the electrodes in the array, and provided maximize overlap between the droplet and the adjacent electrode. Therefore, the droplet easily moved by overlapping with the adjacent

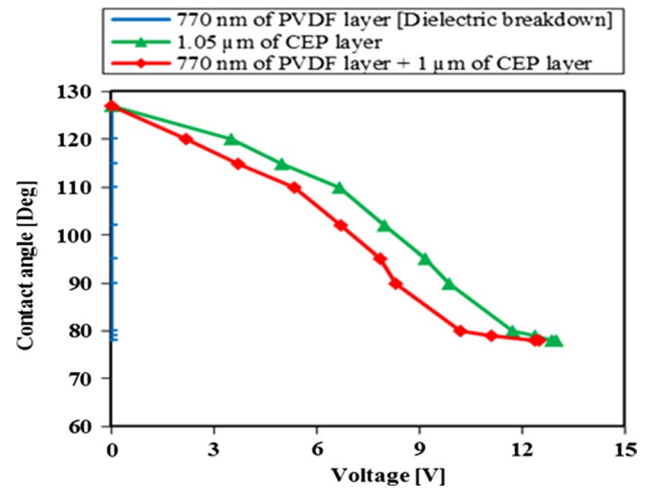


Fig. 5 Changes in the droplet contact angle versus the applied voltage

electrodes at a low actuation voltage compared to the square-shaped electrode.

Changes in the droplet contact angle for variations in the applied voltage on the EWOD devices were also characterised. It was found that a significant change of the droplet contact angle, ranging from 123° to 78°, was observed when the applied voltage was varied. Figure 5 shows a graph of the changes in the droplet contact angle with respect to the applied voltage for different insulating surfaces. It is observed that the graph follows the Lippmann-Young method of EWOD.

From the above-mentioned discussion, it is clear that not only the developed multi-layer dielectric coating but also the shape of the electrodes, along with their dimension and inter-electrode gap distance can reduce the required actuation voltage of an EWOD device. The lowest actuation voltage for the droplet movement was obtained from the proposed EWOD device, which was actuated at an applied voltage of only 12.5 V for the specified design (1.4 × 1.4 mm² of electrodes dimension and 50 μm of inter-electrode gap distances). Since this work operates a microlitre size of droplet on the specified electrode using a low actuation voltage, further reduction of the actuation voltage of the EWOD device can be achieved if the electrode dimension can be reduced further for picolitre droplet manipulation. Moreover, the work should expand the model to other microdroplet operations including mixing, merging, and splitting.

4 Conclusion

This paper presented the design and fabrication of a novel EWOD device which drives a microlitre size droplet using

a lower actuation voltage than other EWOD systems. The single-plate EWOD device was designed and fabricated successfully with a novel shape of seven control and seven ground electrodes on a glass substrate. Moreover, a PVDF layer of 770 nm and a CEP layer of 1 μm were used to form a multi-layered dielectric to reduce the actuation voltage of the EWOD device. The developed EWOD device with the designed electrode shape and the dielectric coating resulted in droplet movement at a minimum voltage of 12.5 V. This actuation voltage was obtained for certain electrode size and inter-electrode gap distance. Although the proposed device was used for droplet movement, it can be utilized for droplet mixing and splitting applications in future. Additionally, the droplet manipulation process can readily be applied to the manipulation of liquids containing biomolecular components (e.g. proteins, DNA, and cells) for medical applications, where samples can be handled in a safe, secure, and efficient manner.

Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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