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Low voltage electrowetting-on-dielectric platform using multi-layer insulators

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ABSTRACT

A low voltage, two-level-metal, and multi-layer insulator electrowetting-on-dielectric (EWD) platform is presented. Dispensing 300 pl droplets from 140 nl closed on-chip reservoirs was accomplished with as little as 11.4 V solely through EWD forces, and the actuation threshold voltage was 7.2 V with a 1 Hz voltage switching rate between electrodes. EWD devices were fabricated with a multilayer insulator consisting of 135 nm sputtered tantalum pentoxide (Ta_2O_5) and 180 nm parylene C coated with 70 nm of CYTOP. Furthermore, the minimum actuation threshold voltage followed a previously published scaling model for the threshold voltage, V_T, which is proportional to $(t/\varepsilon_r)^{1/2}$, where t and ε_r are the insulator thickness and dielectric constant respectively. Device threshold voltages are compared for several insulator thicknesses (200 nm, 500 nm, and 1 μm), different dielectric materials (parylene C and tantalum pentoxide), and homogeneous versus heterogeneous compositions. Additionally, we used a two-level-metal fabrication process, which enables the fabrication of smaller and denser electrodes with high interconnect routing flexibility. We also have achieved low dispensing and actuation voltages for scaled devices with 30 pl droplets.

1. Introduction

In recent years, microfluidic manipulation of liquid droplets has been widely investigated as a platform for the transport of chemical or biomedical liquids on biochips. Electrowetting-on-dielectric (EWD) is an actuator based on controlling charge at the interface of liquids and insulators over buried electrodes [1,2]. EWD is believed to be the most feasible and efficient microfluidic actuator for lab-on-a-chip applications, as the droplets can be promptly driven to a precise position, which is not achievable by continuous flow microfluidics [3]. EWD actuators can split, mix, and dispense droplets from on-chip reservoirs, which gives more flexibility and better choices for multiple applications performed on a common platform [4–7].

EWD is a principle extended from electrocapillarity, where an electric field changes the effective surface energy between a solid insulator and a liquid interface to induce a driving force [8,9]. EWD creates an electric field by applying a voltage at the interface between the dielectric material and a polarizable or conductive liquid droplet [7]. The electric field induces interfacial electrical charge in the liquid, which then exerts a force relative to the surface of the dielectric. Since a liquid interface is deformable, this force distorts the interface [10]. The interfacial force creates a variation in the local interfacial tension, which changes the effective contact angle [4–8,11]. The effective contact angle changes from θ_0 to θ_V for the applied voltage, V, and follows the Lippmann–Young equation [12]:

$$\cos\ \theta_V = \cos\ \theta_0 + \frac{1}{\gamma_{LG}} \cdot \frac{\varepsilon_r \varepsilon_0}{2t} V^2 \quad (1)$$

where ε_r is the dielectric constant of the insulator layer, ε_0 is the permittivity of vacuum, γ_{LG} is the surface tension between liquid and the filler medium surrounding to the droplet, and t is the thickness of the insulator. When actuating a droplet, opposing sets of forces act upon the droplet: an electrowetting force induced by the electric field and resistant forces that include drag forces caused by interactions with the filler medium and contact line friction [13]. At the moment the droplet is actuated, the electrowetting force is balanced with the sum of all drag forces. The minimum voltage applied to achieve this effect is defined as the threshold voltage, V_T [1]:

$$V_T = \sqrt{\frac{2t\gamma_{LG}}{\varepsilon_r \varepsilon_0}[\tan\ \alpha(\sin\ \theta_{V_T} + \sin\ \theta_0)]} \quad (2)$$

where α is the contact hysteresis angle. Since the surface tension and the contact hysteresis angle are influenced by the filler medium and the hydrophobic material, the threshold voltage is variably determined by the dielectric-thickness-to-dielectric-constant ratio $(t/\varepsilon_r)^{1/2}$. Thus, to reduce the actuation threshold voltage, it is required to reduce t/ε_r.

In order to reduce the actuation threshold voltage of EWD devices, sub-micrometer thin films and high dielectric constant materials were used in the fabrication process. In the past EWD

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devices were fabricated with thick, low dielectric constant fluoropolymer insulators, such as 800 nm of parylene C [8], for which actuation voltages were higher than 60 V. For reduced threshold voltage operation, high dielectric constant materials, such as barium strontium titanate ((Ba,Sr)TiO_3, BST) [14], tantalum pentoxide (Ta_2O_5) [15], and aluminum oxide (Al_2O_3) [16] have been tested as insulators on EWD devices. Al_2O_3 was claimed to actuate droplets at 3 V but with a velocity below 1 μm/s. However, 9 V was acquired to move droplets to a neighboring electrode within 1 s (1 Hz switching rate). Both previously reported BST and Ta_2O_5 EWD devices demonstrated a 1 Hz switching actuation threshold of 15 V. However, the BST and Al_2O_3 devices only demonstrated droplet transport, and the Ta_2O_5 device was only able to dispense with a syringe providing pressure. The most difficult operation for EWD devices is dispensing droplets from a closed, on-chip reservoir without external pressure assistance, which requires more force and hence higher voltage than transporting droplets. It is also required that dielectric breakdown and insulator charging at these higher voltages not occur to avoid major reliability problems.

In this work, the lowest dispensing voltage achieved was 11.4 V to split a 300 pl droplet from a 140 nl reservoir with 100 μm electrodes and a 20 μm gasket layer. The actuation threshold voltage of EWD devices made with a multilayer insulator was 7.2 V at 1 Hz switching rate. A threshold voltage scaling model [1] is verified with single layers of different thicknesses of parylene C and Ta_2O_5. In addition, multilayer insulator devices were fabricated with stacked parylene C and Ta_2O_5 films for improved reliability. The multilayer insulators were also found to follow the scaling model. Scaling of the physical dimensions is also demonstrated with a two-level-metal structure in a device capable of dispensing 30 pl droplets onto 35 μm electrodes with a 10 μm thick gasket.

2. Materials and methods

2.1. High dielectric constant material

Parylene C ($\varepsilon_r \cong 3$) and silicon dioxide (SiO_2, $\varepsilon_r \cong 3.7$) have been the most frequently used materials for EWD actuators [4,5]. The actuation threshold voltages have been reported to be 60–80 V for 800 nm parylene C and a 60 nm Teflon hydrophobic layer [7,8] and 25 V for 100 nm silicon dioxide and 20 nm Teflon [5]. The higher voltages needed for operations such as dispensing and splitting have previously prohibited the use of ultra-thin insulators, as they are more susceptible to electrical breakdown and poor reliability. Thus, the use of high dielectric constant materials becomes another solution to reduce EWD applied voltage. The gate oxide in MOSFETs presents similar issues with regard to insulator breakdown and reliability, so those materials used in CMOS devices are potential solutions for EWD devices. Silicon nitride, Si_3N_4 ($\varepsilon_r \cong 8$), barium strontium titanate, (Ba,Sr)TiO_3 (BST, $\varepsilon_r \cong 180$), aluminum oxide, Al_2O_3 ($\varepsilon_r \cong 10$), and tantalum pentoxide, Ta_2O_5 ($\varepsilon_r \cong 20$–25), have been tested as insulator layers on EWD devices. Since the dielectric constant of Si_3N_4 and Al_2O_3 is two to three times that of SiO_2, the threshold voltage reduction is by a factor $1/\sqrt{2}$ to $1/\sqrt{3}$, which was not considered to be a significant enough improvement. On the other hand, devices with 1 mm × 1 mm electrodes coated with 70 nm BST films deposited by metal-oxide chemical vapor deposition (MOCVD) [14] and 95 nm anodic Ta_2O_5 films [15] both reached the relatively low threshold voltage of 15 V. Both materials were coated with a thin fluoropolymer film.

For example, since BST has a very high dielectric constant with a thin 100 nm film (t/ε_r = 100 nm/180), the ratio of effective insulator thickness to dielectric constant is much smaller than a 70 nm hydrophobic layer (t/ε_r = 70 nm/2), and can be neglected. This is also the reason why the actuation voltage of Moon et al. [14] is the same as Li et al. [15], but using the thinner and higher dielectric constant material, i.e. even in the same structure, the higher dielectric constant material may not result in lower actuation voltage, and it depends on the film thickness and the hydrophobic layer's properties.

The onset of charge trapping has been proposed to occur when the effective dielectric strength of the fluoropolymer layer, D_{FP}, is exceeded [17]. Thus the threshold voltage for the onset of charge trapping in a two-layer film is given by the expression:

$$V_{T_{\text{limit}}} = \frac{\varepsilon_{FP} D_{FP} t}{\varepsilon_r} = D_{FP}\left[t_{FP} + \varepsilon_{FP}\left(\frac{t_1}{\varepsilon_{r1}} + \frac{t_2}{\varepsilon_{r2}} + \cdots\right)\right] \quad (3)$$

where ε and t are the dielectric constant and thickness of each material, and *FP*, 1, and 2 represent the fluoropolymer layer and insulators beneath. When the applied voltage is higher than the voltage limit of Eq. (3), trapping of charges at the surface occurs and higher voltages are then required to achieve similar droplet actuation due to a shift in the threshold voltage. As a result, when the high dielectric constant insulator is relatively thin, the hydrophobic layer, for which the dielectric constant is relatively low, dominates the factor t/ε_r. Most of the voltage drop is across the hydrophobic layer, and higher voltage applied with this thin film would cause it to breakdown more easily. Even though the previously reported threshold voltage values were as low as 15 V, the reliability of these EWD devices was limited by factors such as leakage current and time-dependent dielectric breakdown [14].

Among the materials tested, the dielectric constants of Al_2O_3 and Si_3N_4 are too low for practical use. For BST, the dielectric constant is so high for thin films that this layer's effective t/ε_r ratio is negligible compared to the hydrophobic layer, which becomes the major contributor to the insulator stack. This results in most of the voltage drop occurring across the hydrophobic layer, and causes devices to break down more easily. Thus, one would expect that BST devices could not easily dispense droplets, due to the higher voltage required for dispensing operations. Therefore, the final choice was Ta_2O_5, which has been widely evaluated as the gate oxide in the semiconductor industry because of its high dielectric constant and dielectric strength (~8 MV/cm) [18]. Multiple techniques are available to deposit this material, such as evaporation, sputtering, anodic oxidation of tantalum, pulsed laser deposition, and chemical vapor deposition (CVD) [19]. In this study, Ta_2O_5 was deposited by a RF dielectric sputtering system with a 99.99% Ta_2O_5 target. The deposition power was set at 200 W and the deposition pressure was set at 13 mTorr with 10% oxygen in the chamber, producing a deposition rate of 0.9 nm/min. Testing was performed with a sandwich electrode–insulator–electrode structure, from which the dielectric constant for the 200 nm Ta_2O_5 film was determined to be approximately 23. Numerous reports have shown that post-deposition treatment with an annealing process improves the electrical properties of tantalum pentoxide films prepared by the sputtering method [18–21]. These reports describe various annealing procedures which were performed after deposition, resulting in variations in surface structure, dielectric constant, dielectric strength, and leakage current. Here, EWD chips were annealed by a rapid thermal anneal system (RTA, Jipelec JetFirst 100), a photonic heating system, at 400 °C in a N_2 atmosphere (200 sccm) for 10 min. We found that annealing only slightly influenced the dielectric constant and dielectric strength, but notably EWD devices appeared to function with the same reliability with or without the annealing procedure.

2.2. Device fabrication

A two-level-metal device design was developed to provide the capability of fabricating smaller and denser electrodes with interconnect routing flexibility. The electrodes were designed with a