EVALUATION OF ANODIC TA$_2$O$_5$ AS THE DIELECTRIC LAYER FOR EWOD DEVICES

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ABSTRACT

We report that anodic tantalum pentoxide (Ta$_2$O$_5$) exhibits severe polarity and frequency dependencies that, when used as the dielectric material for electrowetting on dielectric (EWOD) devices, in many cases result in long-term performance that is worse than when conventional dielectrics (e.g., SiO$_2$) are used. This rather disappointing news is nevertheless relevant to the community and calls for critical assessment. Here, we find that under direct current (DC) actuation Ta$_2$O$_5$ is attractive for EWOD only if the droplet is negatively biased, and that under alternating current (AC) it is acceptable only for low frequencies.

INTRODUCTION

A liquid wets or spreads on a dielectric surface when an electric field is applied between the liquid and the electrode underneath the dielectric layer. Known as electrowetting-on-dielectric (EWOD), this actuation mechanism has been the basis for a number of micro devices in various application areas: lab-on-a-chip [1-2], optics/display [3-4], electric switch [5] and rheometry [6]. For EWOD to be widely used in commercial products, high actuation voltages need to be avoided. The operation voltage can be lowered by using a dielectric with higher dielectric constant and/or reducing its thickness, as evident in the Lippmann-Young Equation below and experimentally confirmed [7].

$$\cos\theta_V - \cos\theta_0 = \frac{\varepsilon_0\varepsilon_r}{2\gamma t_r}V^2 \qquad (1)$$

where θ_V and θ_0 are the contact angles with and without voltage, respectively, ε_0 is the permittivity of vacuum (8.85×10^{-12} F/m), ε_r and t_r are the dielectric constant and thickness of the dielectric layer, respectively, γ is the surface tension at the liquid-fluid interface (the fluid is air in this paper), and V is the voltage across the dielectric layer. High dielectric constant material is a better choice than reduced dielectric thickness because the latter brings with it the drawback of reduced breakdown voltage.

Tantalum pentoxide, as a relatively high dielectric constant (*k*) material (i.e., 18.5-27.5 [8, 9]), can reduce overall electrowetting voltage compared to common dielectrics (SiO$_2$ and Parylene around 2-3). Anodic Ta$_2$O$_5$, widely used in electrolytic capacitors with reproducible thickness and good film quality [10], has recently been reported as an alternative dielectric for its room-temperature fabrication, high-*k*, and high film quality (i.e., pinhole-free) [11]. However, the above study considered only DC actuation with a negatively biased droplet. In this report, we investigate the reliability of anodic Ta$_2$O$_5$ as an EWOD dielectric by testing additional actuation conditions. As a reference dielectric, we also tested thermal SiO$_2$, chosen for its stability across varied fabrication conditions [7].

DEVICES AND EXPERIMENT

In order to evaluate Ta-Ta$_2$O$_5$ EWOD devices, Si-SiO$_2$ EWOD devices were used as control samples for comparison. Figure 1 shows the two types of EWOD devices used in the long-term testing experiments: (a) Ta-Ta$_2$O$_5$ and (b) Si-SiO$_2$ EWOD devices. In order to ensure dielectric film quality, anodic Ta$_2$O$_5$ and thermal SiO$_2$ were chosen.

Long-term performance of an EWOD system was determined by monitoring contact angle change ($\Delta\theta$) of a droplet between before and after voltage application throughout repeated electrowetting actuation. Slower reduction of $\Delta\theta$ implies better reliability of the EWOD device.

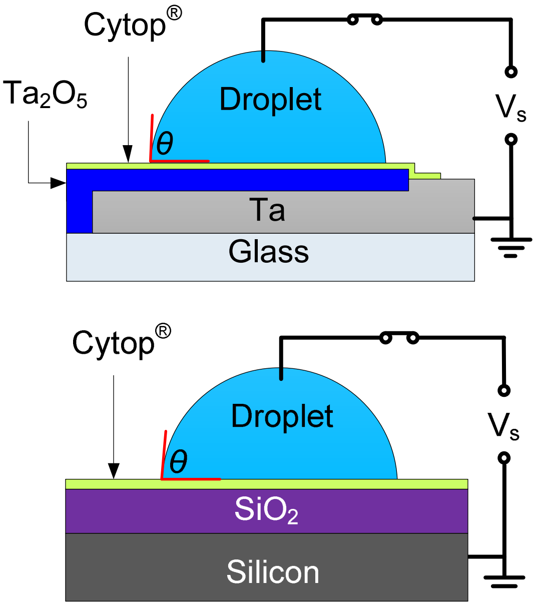

Figure 1: The Ta-Ta$_2$O$_5$ (top) and Si-SiO$_2$ (bottom) EWOD devices tested for repeated EWOD actuations

Fabrication

Process flows for fabricating Ta-Ta$_2$O$_5$ and Si-SiO$_2$ EWOD devices are shown in Figure 2 and Figure 3, respectively. Tantalum of 375 nm thickness was sputtered on clean 2.5 cm × 2.5 cm square glass microscope slides. A 200 nm-thick Ta$_2$O$_5$ film was grown on the tantalum by anodizing in 0.01 wt% citric acid bath using platinum as the counter electrode at room temperature [10]. During anodization, constant current density of 0.8 mA/cm^2 was applied until the voltage reached 100 V, after which 100 V was held for 30 minutes. Constant-current anodization was used to ensure a fixed value of anodization constant (20 Å/V), and the constant voltage phase was used to ensure final desired film thickness [12]. The film thickness was measured by NanoSpec (AFT 010-0180)

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using refractive index of 2.22 reported in [8].

Separately, 560 nm-thick SiO_2 film was thermally grown on a p^{++} Si wafer (with resistivity < 0.0015 Ω-cm) by wet oxidation at 1050°C for 56 minutes. Front side oxide was protected by photoresist AZ5214 during removal of the backside oxide in buffered oxide etching (BOE) solution. The photoresist was then removed in ALEG-380 stripper.

A 1.3% Cytop® solution was made by diluting 9% Cytop® solution with Cytop® solvent (1:6). The resulting 1.3% Cytop® solution was spin-coated on both dielectric layers at 500 rpm for 5 s followed by 1500 rpm for 30 s, giving a 50 nm-thick Cytop® film. After spin-coating, the film was baked at 150°C for 10 minutes to remove the solvent and 195°C for 1 hour to anneal.

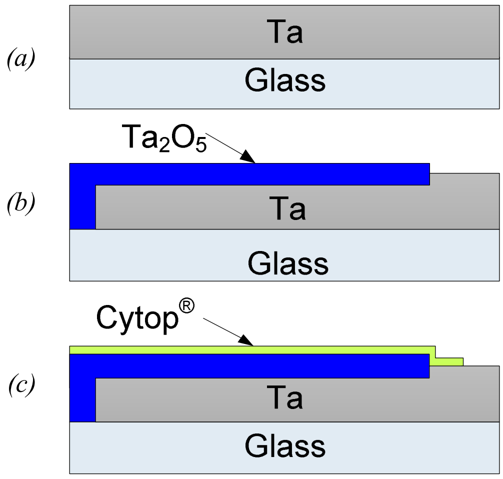


Figure 2: Process flow for Ta-Ta_2O_5 EWOD device. (a) 375 nm of Ta film was sputtered on a glass slide. (b) 200 nm of Ta_2O_5 was anodically grown in 0.01 wt% citric acid bath at 100 V. (c) A 50 nm Cytop® film was spin-coated.

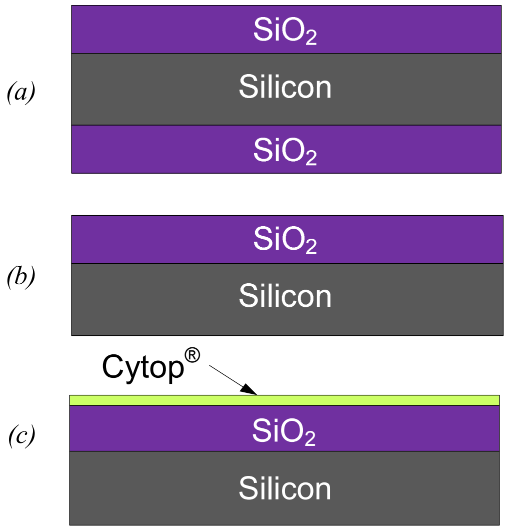


Figure 3: Process flow for Si-SiO_2 EWOD device. (a) 560 nm of SiO_2 film was thermally grown on a p^{++} Si wafer. (b) Backside oxide was removed in BOE. (c) A 50 nm of Cytop® film was spin-coated.

EWOD Experiments

Contact angle measurement of a droplet during EWOD actuation was observed through a CCD camera (PixeLink, model PL B742U). Actuation voltage signals were generated by a LabVIEW program, outputting to a data acquisition (DAQ) system (National Instruments) and amplified to desired voltage values. Voltage was on for 3 seconds and off for 3 seconds, repeating for up to 2000 cycles. Figure 4 shows two images of droplet contact angle captured for each cycle: (a) without and (b) with voltage.

All experiments were conducted using a 10 µL droplet with electrical conductivity of 0.013 S/m. The droplet consisted of 50%v/v glycerin and 50%v/v KCl standard solution (Fluka, σ = 0.1413 S/m). The droplet was conductive enough to ensure the circuit's voltage drop occurred predominantly across the hydrophobic and dielectric layers, not across the droplet.

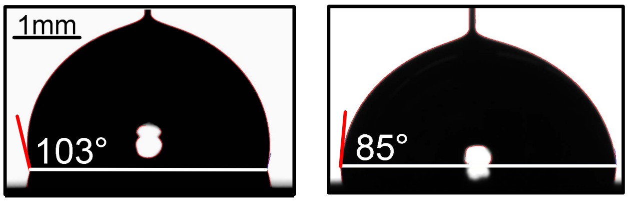


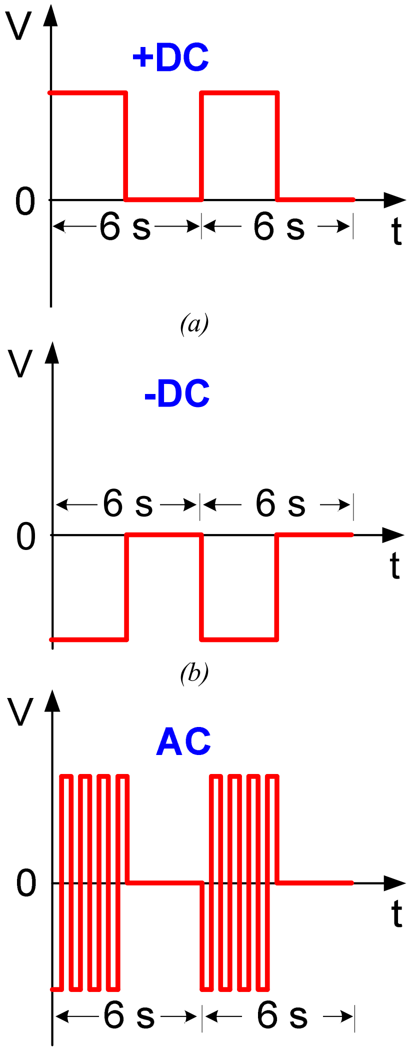
Figure 4: Images of contact angle measurement captured by CCD camera and analyzed by a contact angle measurement program for cases without voltage (left) and with voltage (right).

Three different types of actuation were used for long-term electrowetting testing: +DC, –DC, and an AC square wave, as shown in Figure 5(a-c). The frequency range used for AC actuation was from 50 Hz to 1kHz. In this paper, +DC means positive potential was applied to the droplet through a platinum wire while grounding the buried electrode (e.g., tantalum or silicon), as shown in Figure 5(a). Conversely, applying negative potential to the droplet is denoted as the –DC case, illustrated in Figure 5(b). A square wave was selected over other AC waveforms since its peak value is equal to its root-mean-square (RMS) value, which was set equal to the absolute DC voltage value used in the DC voltage experiments (i.e., $V_{pk} = V_{RMS} = |V_{DC}|$). Hence, maximum force acting on the triple contact line of solid-liquid-air is the same for both DC and AC cases.

In order to start with the same contact angle reduction ($\Delta\theta$), 11.6 V was applied to the Ta_2O_5 and 30 V to the SiO_2 device, corresponding to the same electrowetting number (i.e. $Ew = 0.31$). Electrowetting number represents the degree of EWOD actuation, as shown in the modified Lippmann-Young Equation below.

$$Ew = \cos\theta_V - \cos\theta_0 = \frac{\varepsilon_0 \varepsilon_r \varepsilon_{Cytop}}{2\gamma\left(\varepsilon_r t_{Cytop} + \varepsilon_{Cytop} t_r\right)} V^2 \qquad (2)$$

Here ε_{Cytop} and t_{Cytop} are the dielectric constant and thickness of the Cytop® layer, respectively, and V is the voltage across the dielectric and Cyop® layers together.



(a)

(b)

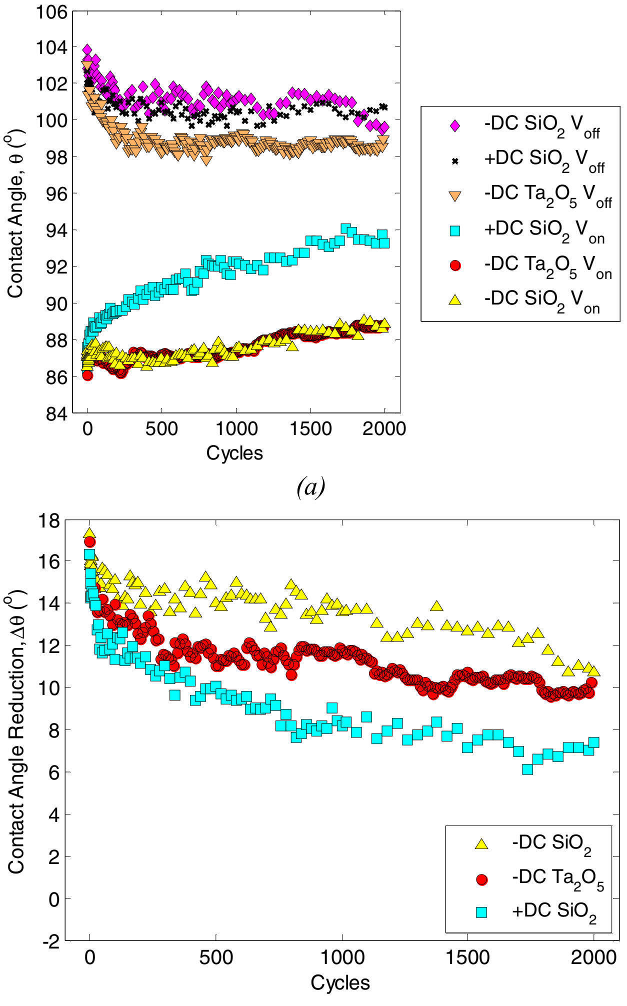
(c)

Figure 5: Actuation signals for long-term electrowetting testing with (a) +DC, (b) –DC, and (c) AC square wave.

RESULTS AND DISCUSSION

The principal piece of information to report about the Ta_2O_5 EWOD device is its utterly poor performance when the droplet is positively biased (i.e., +DC case), an important ramification not described in [10]. Electrolysis occurred immediately once +DC was applied, ruining the device. This dramatic polarity effect on Ta_2O_5 EWOD devices may be understood from the well-known diode-like behavior of Ta-Ta_2O_5 capacitors. Directly underneath the Ta_2O_5 outer surface, there exist negative charges induced by the anodization process [13], providing greater resistance to additional incoming electrons in the –DC case than the +DC case. In other words, current can easily flow from the droplet through the Cytop® and Ta_2O_5 layer to the tantalum electrode when the droplet is under positive potential. The sudden increase of current in the dielectric causes dielectric breakdown [12], visually observed as electrolysis.

Figure 6(a) shows the contact angle with and without DC voltage for three cases (–DC with Ta_2O_5, +/– DC with SiO_2) up to 2000 cycles or 200 minutes of EWOD actuation. In the case of –DC, the on-state angles of Ta_2O_5 (86-88°) are almost the same as those of SiO_2 (86-88°), while the off-state angles of Ta_2O_5 (98-102°) are mostly lower than those of SiO_2 (100-104°). Figure 6(b) presents the same results expressed as the contact angle reduction ($\Delta\theta$) from the off to the on state, i.e., the effectiveness of electrowetting. The contact angle change on the Ta-Ta_2O_5 device decreases faster than that on the Si-SiO_2 device, implying that Ta_2O_5 degraded faster than SiO_2 under repeated EWOD actuations. Although the electrowetting effect diminishes faster with +DC than with –DC on most EWOD devices including the SiO_2 device [14], the polarity dependence is extreme for the Ta_2O_5 devices. While reasonable performance is seen on the Ta_2O_5 device with –DC throughout 2000 cycles, no results were attainable under +DC due to electrolysis.

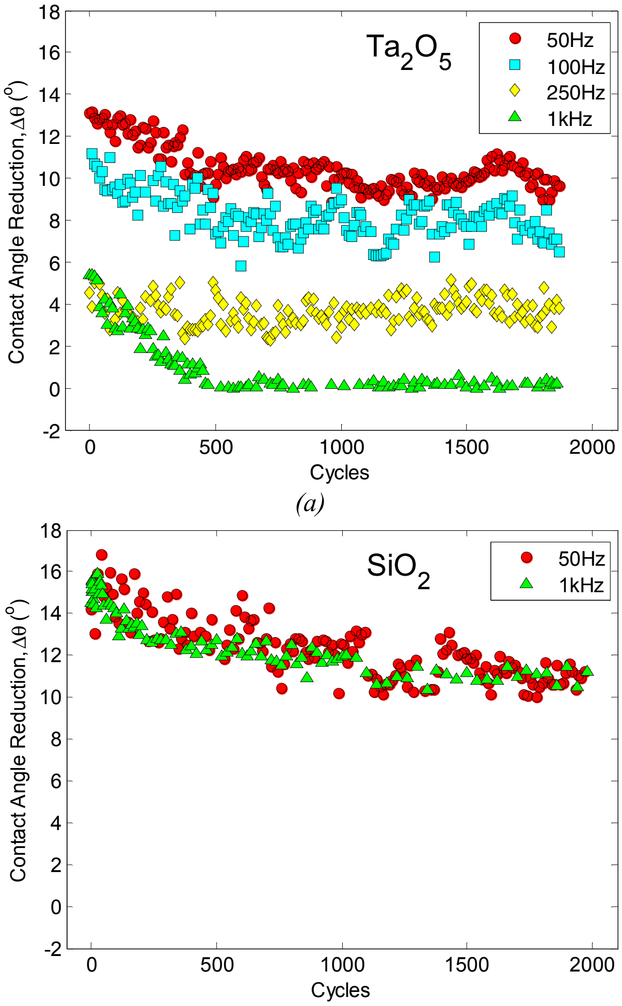


(a)

(b)

Figure 6: Measured data of (a) contact angle and (b) electrowetting-induced contact angle reduction on Ta_2O_5 and SiO_2 EWOD devices with DC actuation cycles of different polarities: +DC and -DC. No data could be collected for Ta_2O_5 with +DC because electrolysis occurred instantly once voltage was turned on.

Figure 7 shows the contact angle reduction with AC signals of different frequencies applied to Ta_2O_5 EWOD devices. The on- and off-state duration in each cycle is the same as for the DC cases above. Figure 7(a) summarizes the results on the Ta_2O_5 device with AC frequencies of 50 Hz, 100 Hz, 250 Hz, and 1 kHz. Among the frequencies used in the experiments, the best performance occurs at 50 Hz (which resembles the –DC performance) and diminishes as frequency increases. In contrast, no obvious difference between 50 Hz and 1 kHz was observed on SiO_2 devices as shown in Figure 7(b).



(a)

(b)

Figure 7: Measured data of electrowetting-induced contact angle reduction on (a) Ta_2O_5 EWOD device and (b) SiO_2 EWOD device with AC signal. Frequencies tested were 50 Hz, 100 Hz, 250 Hz and 1 kHz for Ta_2O_5, and 50 Hz and 1 kHz for SiO_2. The plots show that long-term performance of Ta_2O_5 device worsens with higher AC frequencies, while that of SiO_2 device is not affected much by the AC frequency.

CONCLUSION

EWOD devices made with a Ta_2O_5 dielectric layer exhibit severe polarity and frequency dependencies not observed with common dielectric materials such as SiO_2. Specifically, long-term performance of Ta_2O_5 EWOD devices is acceptable only when the droplet is negatively biased and the actuation frequency is low (< 50 Hz for the current system).

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