



Charge injection in dielectric films during electrowetting actuation under direct current voltage



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ABSTRACT

Electrowetting-on-dielectric technology suffers from a lack of accuracy and reliability under DC voltage. The root of this phenomenon is related to charge injection within dielectric materials polarized and in contact with electrolytes. Correlation between contact angle measurements and transient current is used to investigate electrowetting stability under DC voltage. In particular, nature of ionic species and polarization effect are scrutinized. Asymmetric mechanisms appear between positive and negative polarizations from both wetting stability and leakage current, attributed to charge injection and allowing electrets-type actuation. A first occurrence of large amplitude reverse electrowetting is also observed.

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1. Introduction

As research continues to push the envelope on micro-electromechanical system (MEMS) devices, research has begun turning to new physical phenomena to drive breakthroughs and innovations. One such physical phenomenon at the forefront of research efforts is electrowetting [1] and more specifically electrowetting-on-dielectric (EWOD) [2,3].

Electrowetting, first discovered in 1875 through Lippmann's experiments on mercury, has seen rapid growth since the use of a dielectric layer between the electrode and electrolyte in the 90s [4], inducing a much more reversible actuation and thus allowing many applications. In its simplest form, electrowetting can be demonstrated by placing a water droplet on a hydrophobic dielectric plate. By introducing a voltage potential across the water drop and dielectric system, charges will build up on both sides of the dielectric layer in the same manner as a capacitor. The charges on the side of the water droplet will induce an electrostatic pressure that tends to spread the conductive liquid.

EWOD has found itself being utilized across many different fields of technology including lab-on-chip devices [5], lenses [6,7], and displays [8]. In practice, EWOD devices typically employ AC voltage sources to avoid the effects of charge injection where charges will penetrate the dielectric layers. AC sources indeed constantly swap the polarity of the applied potential, thus preventing injected charges from sufficiently accumulating. However, relying on AC sources for stability presents its own disadvantages as they add increased circuit complexity and size

as well as additional power requirements. As a DC voltage is applied to an EWOD device, charges will build up within the dielectric layers. As these injected charges accumulate, they can cause unwanted behavior as they screen the original applied charges, thus reducing the effective applied voltage. Therefore, there is a need to study and understand the mechanisms of charge injection for DC EWOD devices. This paper looks to study the mechanics and effects of charge injection to better understand and control this effect, especially on Si₃N₄ known as a high dielectric constant material ($\epsilon_r = 7$) and less commonly used in EWOD technology compared to SiO₂ [9]. This breakthrough could lead to much smaller, more stable, and more energy efficient EWOD devices.

2. Theory

The basic electrowetting effect observed from a conductive liquid standing on a polarized dielectric surface is well described using the equation [4]:

$$\cos(\theta) = \cos(\theta_0) + \frac{C_d}{2\gamma} V^2 \quad (1)$$

where θ_0 is the natural contact angle measured from the conducting fluid on a non-polarized surface, θ is the angle with respect to the applied voltage, C_d is the dielectric surface capacitance, γ is the surface tension from the conductive fluid, and V is the applied voltage to the dielectric material. Surface capacitance is given by $C_d = \frac{\epsilon\epsilon_0}{d}$ where ϵ is the relative dielectric constant from the dielectric layer, d the thickness of the dielectric material and ϵ_0 the vacuum permittivity. For a stack of

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layers the equivalent surface capacitance is obtained from $\frac{1}{C_{eff}} = \sum \frac{1}{C_i}$. When two non-miscible liquids are used, considering an oil spreading with a contact angle θ in a conductive medium, the electrowetting relation becomes [10]:

$$\cos(\theta) = \cos(\theta_0) - \frac{C_d}{2\gamma_{ow}} V^2 = \cos(\theta_0) - \frac{V^2}{V_0^2} = \cos(\theta_0) - \eta \quad (2)$$

where θ is the oil drop natural contact angle, γ_{ow} is the oil/water interface tension and η is the dimensionless electrowetting number [2,3]. In Eq. (2), the voltage pre-factor $\frac{C_d}{2\gamma_{ow}}$ has consequently the dimension of the inverse of a square voltage and $V_0 = \sqrt{\frac{2\gamma_{ow}}{C_d}}$ has the same dimension as a voltage and dimensionless electrowetting number is equal to $\eta = \frac{V^2}{V_0^2}$.

For a given system, V_0 describes both the surface capacitance and the interface tension, and we call it driving voltage as it corresponds to the voltage required to induce a cosine variation of one unit, i.e. an electrowetting number value equal to one, which is usually close to the maximum variation. Physically speaking, it represents the voltage wherein the interface surface energy constant γ_{ow} is equal to the electrostatic surface energy $\frac{1}{2}C_d V^2$ from the capacitance. Hence, and in any electrowetting device, varying both in liquid nature and dielectric materials, V_0 is a more universal constant to describe its actuation capability. V_0 is also incidentally close but not always directly related to saturation voltage, as described in reference [11].

We deliberately used a two-liquid configuration, studying oil contact angle in a conductive medium. This setup benefits from a reduced hysteresis as a result of the lower initial contact angle of the oil in the liquid solution [12].

When the electrowetting effect is attenuated due to charge injection within the dielectric layer, contact angle variation can be described using an effective voltage:

$$\cos(\theta) = \cos(\theta_0) - \frac{V_{eff}^2}{V_0^2} \quad (3)$$

where the effective voltage, V_{eff} represents the effective voltage potential inducing contact angle modification, after screening by injected charges. If no charge is injected, V_{eff} will be equal to the applied voltage, whereas injected charges will reduce effective voltage of the system. Information on the charges in the system can also be extracted.

From Eq. (3), the effective voltage can be calculated by

$$V_{eff} = V_0 \sqrt{\cos(\theta_0) - \cos(\theta)} \quad (4)$$

and surface charge density and injected charge density can be deduced by

$$\sigma_{surface} = \sigma_{eff} = \frac{\epsilon\epsilon_0}{d} V_{eff} \quad (5)$$

and

$$\sigma_{injected} = \sigma_{tot} - \sigma_{eff} = \frac{\epsilon\epsilon_0}{d} (V - V_{eff}). \quad (6)$$

Choi et al. [13] and Raj et al. [14] have shown that the use of larger cations and anions can drastically increase breakdown resistance as their larger size prohibits travel through any dielectric defects. We propose to study if ionic size also influence charge injection by studying a selection of cations under positive bias and anions under negative bias. Through these experiments, we expect to observe both the effects of ions on charge injection as well as reveal any variations between charge injection in forward and reverse bias.

However charge injection also has the possibility of being utilized in a positive light. After charges have been injected into a dielectric layer

during a voltage application, they will remain trapped temporarily in this layer when bias application turns off or when the system is switched to open circuit. These injected charges will induce their own effective voltage across the wetting system. Taking advantage of this effect could lead to the production of bi-stable devices that maintain a set contact angle without the need for an applied voltage [15,16]. Charge injection is also widely used to produce electrets, i.e. materials having a permanent electrical field, being used as transducers for microphones [17], and as micropower generators [18].

It is thus critical to independently study leakage current and charge injection, as both phenomena are linked to the electric field but don't necessarily have the same consequences; leakage is related to the migration through dielectric volume whereas injection is more a liquid-dielectric interface mechanism. Despite several authors carefully studied the effect of leakage and dielectric breakdown in EWOD systems [19,20,21,22], very little is known on the exact mechanism and relevant parameters of reversible charge injection [15].

3. Materials and methods

3.1. Samples

EWOD experiments were performed on silicon nitride (Si_3N_4) dielectric with a Cytop® hydrophobic coating. Silicon nitride with a thickness of 100 nm, deposited on top of 1–20 Ω cm doped silicon wafers using plasma-enhanced chemical vapor deposition, was purchased from CIME Nanotech (Grenoble, France). A top coat of the hydrophobic fluoropolymer Cytop CTX 809 (supplied by AGC Chemicals) was deposited on the dielectric surface using spin coating. 1 wt.% Cytop mixed in CT-Solv180 was spun at 4000 rpm for 90 s with an acceleration of 100 rpms/s. Substrates were annealed at 50 °C for 30 min followed by 180 °C for 60 min to ensure proper adhesion between the fluoropolymer and the dielectric. After sample preparation, ellipsometry measurements (Ellipso-Porosimeter EP-A SOPRA GES 5E) were taken and the thickness of the fluoropolymer was found to be 25 nm corresponding to an overall theoretical surface capacitance of 0.35 mF/m² assuming a dielectric constant for the fluoropolymer and silicon nitride of 2.1 and 7.5 respectively.

3.2. Solutions

Electrowetting experiments were performed using an oil drop submerged in a conducting liquid. Various conducting liquid solutions were prepared to study the effect of ionic size on charge injection. We chose to work with a combination of four cations and anions, calcium, sodium, acetate, and chloride.

Dodecane and polypropylene glycol were purchased from Sigma Aldrich and used without further purification. Salt concentration was kept constant at 0.045 M and 0.5 wt.% of propylene glycol was added to the ionic solution as a surfactant. In all experiments, dodecane was used as an oil. The surface tension γ_{ow} in this case is around 15 mN/m.

3.3. Electrical properties of the samples

To further characterize the interactions between the substrates and solutions chosen, capacitance measurements were done using the cell of Fig. 1(a): The sample is squeezed with springs between a teflon container and a copper plate. The electric contact between the copper and the back silicon side of the sample is made using indium–gallium eutectic. An o-ring is inserted around the hole in the middle of the container to avoid electrolyte leakage and ensure a constant interfacial area of 2.3 cm².

The voltage is applied through the platinum electrode immersed in the electrolyte and the copper plate and current is measured with a picoammeter Keithley 6487. In this setup, the current through the capacitive layer is measured as a function of time. In order to get a

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