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# Influence of fluoropolymer surface wettability on electrowetting display performance<sup>☆</sup>

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## ABSTRACT

Amorphous fluoropolymer (FP), as a material for both insulating and hydrophobic coating, plays an essential role in electrowetting displays (EWD). In this work, three FPs based on Teflon AF1600, Hyflon AD60 and Cytop 809A were studied according to their influence on the EWD performance. Both water/air and oil/water contact angles were utilized to compare the surface wettability of these three FPs. Reversible and fast optical switch could be achieved in the EWD devices fabricated using all three FPs; however, the less hydrophobicity of the Cytop 809A surface would lead to a slower Off-switching speed or even incomplete close of the micro-pixels. The “reflow” temperature for restoring the hydrophobicity of fluoropolymer surface should be high enough to achieve a sufficient surface recovery, and at the same time avoid inducing failures like film dislocation and breakdown. The optimal “reflow” temperature has been investigated and evaluated based on the EWD performance. This work would help us deeply understand the surface wettability effect on fluidic behavior in micro-pixels driven by electrowetting, and related optical phenomena in EWDs as well.

## 1. Introduction

Electrowetting has been widely used to manipulate liquid motion at small scales. It was first exploited by Lippmann in 1875 [1]. Recently, it has attracted a lot of attentions because of its broad applications in electrowetting displays (EWD) [2–4], digital microfluidics [5], lenses [6], and energy harvesting [7].

The working principle of electrowetting on dielectric (EWOD) can be described by Young-Lippmann’s equation [8–11],

$$\cos\theta - \cos\theta_0 = \frac{\epsilon_0 \epsilon_r V^2}{2d\gamma} \quad (1)$$

where  $\theta_0$ , the initial contact angle;  $\gamma$ , the interfacial tension between two fluids;  $\epsilon_0$ , the vacuum permittivity;  $\epsilon_r$ , the relative permittivity of the insulator; and  $d$ , the thickness of the insulator. As can be seen from the equation, the insulator and the surface wettability (contact angle) were key parameters for electrowetting performance. It has also been

proven that the insulating layer and the hydrophobic coating is very important for electrowetting performance, and therefore the related parameters, including driving voltage, degradation of electrowetting effect and leakage current [8–12]. The combination of inorganic thin film as insulating layer and fluoropolymer (FP) as hydrophobic top coating has been widely used to investigate the electrowetting phenomenon. The inorganic insulator materials (such as SiO<sub>2</sub>, TiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub>, and so on) with high dielectric constant were normally used to decrease the electrowetting actuating voltage based on the Young-Lippmann’s equation [9–11]. Teflon AF1600 and Cytop 809A have been commonly used as hydrophobic top layer because of their low surface energy [9–13]. It was reported that Cytop showed superior long-term electrowetting on dielectric (EWOD) performance compared with Teflon AF [12].

Except for EWOD, electrowetting on liquid-infused films (EWOLF) has recently received increasing interest. Dielectric liquid lubricants are spread on the surface and being locked in a membrane to form a smooth

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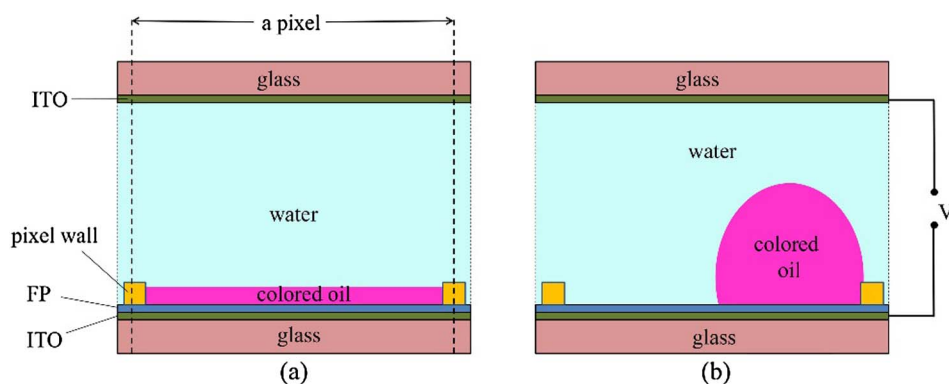


Fig. 1. Schematic drawing of an electrowetting display (EWD) pixel. (a) Off state: in a EWD pixel, without applied voltage, a homogeneous oil film spreads over the pixel area showing the color of the dyed oil. (b) On state: in a EWD pixel, with an applied voltage of  $V$ , the oil film was pushed by water to one corner of the pixel, showing the color of the bottom substrate. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

liquid-infused dielectric layer which could minimize the contact angle line pinning and lead to fast response without sacrificing the desired electrowetting reversibility. EWOLF has been applied for complete reversibility and controlled droplet oscillation suppression in droplet electrowetting devices [14,15].

EWD is a device utilizing a dual fluidic system of colored oil and transparent water to realize display function in a microscale pixel based on electrowetting mechanism. The concept of EWD was first proposed by Beni et al. [16]. In 2003, the functional EWD device was reported by Hayes et al. [2]. Afterwards, a lot of research has been done to optimize the EWD performance from the views of materials, fabrication process or electrical control [17–20]. Fig. 1 shows the schematic drawing of a EWD pixel at “Off” (Fig. 1a) and “On” (Fig. 1b) states. In the absence of a voltage, the oil forms a continuous film in a pixel between the hydrophobic insulator-covered electrode and water, showing the color of the oil film. When a voltage is applied across the top and bottom electrodes, the transparent water is driven to move towards the insulator, pushing the oil film aside or break, showing the color of the bottom substrate. In this way, the optical properties of the stack, when viewed from the top, are tuned between a colored Off-state (dyed oil) and a white On-state (color of bottom substrate).

Recently, FPs have been widely applied in electrowetting devices as both insulating and hydrophobic layer [3,8,17,18]. The breakdown voltage as high as  $100 \text{ V}/\mu\text{m}$  has been achieved for Teflon AF1600 when its thickness was lower than  $1.0 \mu\text{m}$  [8]. Large scale EWD devices have also been processed with both Teflon AF1600 and Cytop 809A materials [3,19]. However, the comparison among different FP’s influence on EWD performance has not been reported yet.

In this work, three FPs based on Teflon AF1600, Hyflon AD60 and Cytop 809A were investigated according to their effect on the EWD performance. The surface wettability of FPs was evaluated by the contact angles of the water droplet in air. To mimic the real situation in an electrowetting display device, the contact angles of the oil droplet in water surrounding were introduced. Two specific processes, named “activation” and “reflow” in EWD fabrication, could be achieved by reactive ion etching (RIE) and thermal annealing. The contact angle changes before and after such processes have been investigated to understand the surface wettability change, and thus their effect on EWD performance. Therefore, an optimum “reflow” temperature for each FP has been obtained and used for EWD fabrication. Moreover, the failure modes caused by annealing at high temperature were also studied to understand the reasons of device damage.

## 2. Materials and methods

### 2.1. Materials

Commercial indium tin oxide (ITO) glass with thickness of  $0.7 \text{ mm}$  and resistance of  $100 \Omega/\square$  was purchased from Guangdong Jimmy Glass Technology Ltd. (Foshan, China). Amorphous fluoropolymer

based on Teflon AF1600 (Dupont, Shanghai, China), Hyflon AD60 (Solvay, Shanghai, China) were dissolved into FC-43 (Minnesota Mining & Manufacturing Company, Saint Paul, USA) with concentration of 3.7 wt% and 6.5 wt%, respectively. Cytop 809A solution was purchased from Asahi Glass Co., Ltd (Kanagawa, Japan) with concentration of 9.0 wt%. Negative photoresist for fabricating pixel walls was co-developed with a local material supplier. The conductive liquid was 1.0 mM NaCl solution with conductivity of  $\sim 110 \mu\text{S}/\text{cm}$ . The color dye was designed and synthesized in our lab. Decane (Micklin, Shanghai, China) was used as dye solvent. The interfacial tension of colored oil (0.21 M dye decane solution)/conductive liquid (1.0 mM NaCl aqueous solution) was  $19 \text{ mN}/\text{m}$ .

### 2.2. Device fabrication

ITO glass was used as the starting substrate which was initially cleaned in a cleaning line (KJD-7072ST, KEJINGDA Ultrasonic Equipment Co., Ltd., Shenzhen, China), and then coated with amorphous fluoropolymers using a spin coater (KW-5, Institute of Microelectronics Chinese Academy of Sciences, Beijing, China) at the speed of  $1000\text{--}2000 \text{ rpm}$  for 60 s. FP coating was then dried on a hot-plate at  $85 \text{ }^\circ\text{C}$  for 5 min and then in an oven at  $185 \text{ }^\circ\text{C}$  for 2 h, obtaining  $\sim 800 \text{ nm}$  thick fluoropolymer film on the ITO-glass. In order to coat the photoresist on it, the FP surface was treated to hydrophilic by using a reactive ion etching (RIE) machine (ME-6A, Institute of Microelectronics Chinese Academy of Sciences, Beijing, China) with slight oxygen plasma (5 W for 10 s plasma treatment). Photoresist was coated on the FP surface, and lithography process was applied using an aligner (URE-2000/35, Institute of Optics and Electronics, Chinese Academy of Sciences, Chengdu, China) to make the pixel walls. A thermal reflow process was applied by putting the substrate in an oven (5FG-01B, Huangshi, China) under a certain degree for 2 h. Afterwards, the ITO-glass with FP layer and pixel walls was filled with colored oil, assembled and sealed with a bare ITO-glass under water. The detailed process has been described in the Refs [19,20].

### 2.3. Measurements

Contact angle was measured using a Contact Angle Meter (POWE-REACH, Shanghai Zhongchen Digital Technology Apparatus Co., Ltd. Shanghai, China). Thickness and surface morphology of the FP and pixels were measured using a stylus profiler (Dektak XT, BRUKER Corporation, Shanghai office, China). A waveform generator (Agilent 33500B Series, Santa Clara, CA, USA) and an amplifier (Agilent 35502A) were used to provide square wave signals with specific voltage amplitude to drive the EWD devices. An optical colorimeter (Arges 45, Admesy, Ittervoort, the Netherlands) was used to measure the optical response of the devices. The incident light was shined at an angle of  $45^\circ$ , and a detector at  $45^\circ$  angle with surface area of  $\sim 1 \text{ cm}^2$  was positioned on the device area. Optical microscope (CTX41, Olympus, Tokyo,

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