

Study of cyanoethyl pullulan as insulator for electrowetting



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ARTICLE INFO

Article history:

Received 31 January 2014

Received in revised form 21 March 2014

Accepted 29 March 2014

Available online 12 April 2014

Keywords:

Cyanoethyl pullulan (CEP)

Insulator

Dielectric

Electrowetting

EWOD

ABSTRACT

In this paper, the dielectric and electrowetting properties of cyanoethyl pullulan (CEP) are studied as the insulator in electrowetting on dielectric (EWOD) device. The CEP film is formed by spin-coating and low-temperature annealing in atmosphere. The characterization results show that CEP can serve as the dielectric of EWOD with a high dielectric constant of 18 and a great electrowetting performance when annealed at 100 °C in atmosphere. The reversibility, stability, polarity-dependence, and frequency-dependence of electrowetting on CEP are fully investigated. The spin-coated CEP exhibits little polarity-related electrowetting while having better driving performance under negative-potential. Frequency-related electrowetting is more sensitive at low-frequency alternating current (AC) potentials than at high-frequency ones. It also shows reversible and stable electrowetting with a small irreversibility of 2° after a 700-cycle test. Based on the above results, an EWOD device with 1 μm thick CEP dielectric layer is fabricated. The EWOD device successfully manipulates water droplet at a low driving voltage of 20 V and a large velocity under low voltage of DC or AC signals with different frequencies. The easy fabrication and excellent performance of CEP qualifies it as a superior dielectric material in the future EWOD devices and lab-on-a-chip system.

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1. Introduction

In recent years, the manipulation of liquid droplets, referred to as digital microfluidics (DMF), has been widely investigated as a platform for control of liquids on lab on a chip (LOC) in many applications [1–4]. As one of the advanced and promising DMF technologies, electrowetting-on-dielectric (EWOD) technology is feasible and efficient in controlling discrete droplets through electrowetting. During the electrowetting, the wetting property of a hydrophobic surface can be modified by an external electric field [5,6]. The EWOD device generally manipulates liquid by way of droplet creating, transporting, splitting, and merging [7]. A typical EWOD microfluidic device must contain four components: a conductive material serving as the drive electrodes to provide control signal and route, a dielectric material serving as the insulator to provide main capacitance for electrowetting, a hydrophobic material lowering the surface energy for successful liquid movement, and a droplet liquid working as a medium for typical applications [8]. The principle of EWOD device can be described by the Lippmann–Young (L–Y) equation [9]:

$$\cos \theta - \cos \theta_0 = \frac{1}{2\gamma} CV^2 = \frac{\epsilon \epsilon_0}{2\gamma t} V^2 \quad (1)$$

where θ_0 and θ are the contact angles (CAs) before and after the driving voltage, respectively. V is the applied voltage, C the capacitance of the dielectric in the device, and γ the surface tension, respectively. ϵ_0 (8.85×10^{-12} F/m), ϵ , and t denote the permittivity of vacuum, the dielectric constant, and the thickness of the dielectric layer, respectively. As can be seen in Eq. (1), when the voltage is applied on the droplet, the CA of the droplet will be changed, leading to the irregular deformation of the droplet and further electrowetting forces to move the droplet. Furthermore, at a given voltage, the electrowetting ability of a droplet is highly dependent on the electrical properties of the dielectric. Therefore, an appropriate dielectric material layer with a high dielectric constant and a small thickness should be used to lower the actuation voltage for commercial point of care Microsystems [10]. Actually, there have been many researches focusing on fabrication and evaluation of high performance materials in EWOD devices to reduce the driving voltage, such as barium strontium titanate (BST) through metal organic chemical vapor deposition (MOCVD) process [11], Si_3N_4 through plasma-enhanced chemical vapor deposition (PECVD) process [12], parylene through spin-coating [13] or vapor pyrolysis process [14], tetraethoxysilane (TEOS) deposited by PECVD [15], Al_2O_3 deposited by atomic layer deposition (ALD) [16], and Ta_2O_5

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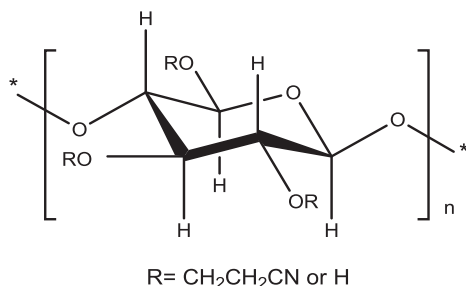


Fig. 1. Molecular structure of CEP.

formed by anodized or sputtered process [17,18]. Although the above materials can reduce the driving voltage greatly, the rigorous and complex fabrication process may limit mass production due to high cost, a great obstacle in the commercialization of EWOD-based LOC. Therefore, an ideal material for electrowetting should have a high dielectric constant and a great dielectric strength with easy fabrication process for LOC applications.

In this paper, we report a novel material, cyanoethyl pullulan, as the insulator for EWOD devices. CEP is a cross-linked polymeric material with a molecular weight of 489,000. The molecular structure of CEP is shown in Fig. 1. It has many nitrile groups with polarization in the molecular side chains, accounting for a high dielectric constant of about 19 (1 kHz) [19,20]. With such attractive properties as easy solution-processability, low industrial cost, and nice flexibility, the CEP material has already been used as the high k gate insulator in organic field-effect transistors (OFET) [21–24]. However, due to the small fraction of the cross-linkable –OH groups and the rigid ring structure of CEP, it is difficult to produce a very condensed film. As a result, it still suffers from the electrical leakage with a very thin layer as required in OFETs [20]. In EWOD field, no thickness limitation of the dielectric exists as in OFET and a thick CEP can also lower the driving voltage efficiently with its high dielectric constant. Moreover, the CEP layer can be easily formed through spin-coating and low temperature post-baking, which is very attractive to commercial applications of LOC. To our knowledge, the CEP material has rarely been used in EWOD devices [25,26] and its performances related to electrowetting have not been well studied. Herein, we study the properties of CEP material in detail and evaluate its performances for application in EWOD devices. The optimized fabrication process of CEP layer is obtained by electrical characterization and general CA measurements for the first time. Then asymmetry, reversibility, stability, and frequency-dependence of electrowetting on CEP are fully studied in the polarity-related, frequency-related, and long-term CA test. Finally, the CEP is used as the insulator in EWOD device, where the droplet manipulation is carried out.

2. Experimental

2.1. Materials and apparatus

The glass substrate coated with 130 nm Indium tin oxide (ITO) layer with the sheet resistance of about $15 \Omega/\square$ was bought from Wesley technology Co., Ltd. The CEP powder was the commercial product from Shin-Etsu Chemical Co., Ltd. N,N-dimethylformamide (DMF) provided by Sigma–Aldrich was used as the solvent of CEP. The hydrophobic material Teflon[®] AF2400 solution (Grade 400S2-100-1, 1 wt%) was from DuPont. The positive photoresist (RZJ-304 from Suzhou Ruihong Electronic Chemical Co., Ltd.) was used in lithography. The glycerin and KCl standard solution (Sigma–Aldrich) were used to prepare the conductive liquid for CA measurements.

The electrodes were patterned on the lithography machine (G-33, Vacuum Machinery Plant in Chengdu, China). A reactive ion

etcher (RIE-10NR, Samco International, Japan) pretreated the substrate for subsequent coating. To deposit the CEP and hydrophobic layer, the spin-coater (WS-400BZ-6NPP, Laurell, US) and the hot plate (HP-303DU, SmartLab, US) were utilized for deposition and post-baking, respectively. Physical vapor deposition (PVD) machine (ASC-4000-C4 Type L, ULVAC, Japan) was applied to deposit Al metal layer.

Film thicknesses were measured by the Profiler (Dektak XT, BRUKER, Germany). The impedance analyzer (4249A, Agilent, US) was used for capacitor–voltage (C–V) test. The CA measurements were carried out by the drop shape analyzer (DSA30, KRUSS, Germany). The driving signals for electrowetting were provided by the signal generator (FG503, MOTTECH, Taiwan) and the amplifier (HA-405, MOTTECH, Taiwan) with the aid of LabVIEW software and other circuit components (USB 6251, National Instruments (NI), US, etc.). The motion of droplets was recorded by the digital camera system (TK-C9201EC, JVC, Japan).

2.2. Preparation and measurements

In our experiment, the glass substrate was first cleaned with acetone, ethanol, and deionized (DI) water in sequence, and then dried with pure N₂. If the bottom ITO electrodes need to be patterned, the lithography and etch process were carried out as follows. Firstly, the substrate was spin-coated with photoresist at 3000 rpm for 30 s and post-baked at 100 °C for 10 min. The substrate was then exposed (5 s, 10 mW/cm²) through a mask, and developed in the developer for 40 s, followed by DI water rinsing and hard baking (120 °C, 5 min). Finally, the wet etch process was carried out, followed by removing of the photoresist, washing, and drying. When the substrate with ITO electrodes was ready, the RIE treatment (30 sccm O₂, 100 W, 30 s) was employed to pretreat the surface for better adhesion between CEP and ITO. The CEP powder was dissolved in DMF to produce a 15% (wt/wt) solution, and then the solution was spin-coated onto the substrate at 3000 rpm for 30 s to form a thin film of about 1 μm. After that, the CEP samples were dried at room temperature (RT) or annealed at low temperatures (from 60 to 200 °C) in atmosphere for an hour in case of different experiments.

For CA measurements, a 60 nm thick Teflon layer was spin-coated on CEP as the hydrophobic surface. 5 μL DI water, or conductive liquid, consisting of 50% (v/v) glycerin and 50% (v/v) KCl solution with an electrical conductivity of about 0.013 S/m, was dripped on the surface of Teflon. A 0.1 mm platinum (Pt) wire was inserted into the droplet as the top electrode. During measurements, voltages were applied to the droplet through Pt electrode while grounding the bottom ITO electrode (see Fig. 2(a)). The DC or AC voltage was applied on and off the droplet with the shift time of 2 s. If a positive (negative) voltage is applied on Pt electrodes, it is denoted as +DC (–DC). The shape of the droplet, changing with the on and off of the applied voltage, was captured and analyzed to obtain the CAs.

For electrical characterization, 150 nm Al layer was deposited and patterned on top of the CEP surface to form the 500 μm circle electrodes through a hard mask by PVD process, as shown in Fig. 2(b). The ITO and the Al electrodes were used as the bottom and top electrodes, respectively, for CV tests to find out the dielectric constant of CEP.

A parallel-plate EWOD device was fabricated with the CEP as the dielectric layer and the ITO drive electrodes of 1.5 mm × 1.5 mm (Fig. 2(c)). The bottom plate consists of ITO drive electrodes, 1 μm thickness CEP dielectric layer, and 60 nm Teflon hydrophobic layer. The top plate was an unpatterned ITO glass as the transparent common electrode and a layer of 60 nm Teflon as the hydrophobic layer. The gap between the plates was 150 μm. To actuate the droplets, the common electrode on the top plate was connected to the ground, while the driving electrodes on the bottom plate

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