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Optimum thickness of hydrophobic layer for operating voltage reduction in EWOD systems

Jeong Byung Chae, Jun O Kwon, Ji Sun Yang, Dohyun Kim, Kyehan Rhee**, Sang Kug Chung*

Department of Mechanical Engineering, Myongji University, Yongin 449-728, South Korea

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ABSTRACT

This paper presents the optimum thickness of a hydrophobic layer for operating voltage reduction in electrowetting-on-dielectric (EWOD) systems by investigating the effects of thickness on the surface wettability and hysteresis using atomic force microscopy (AFM). To investigate the surface wettability, the thicknesses of Cytop and Teflon layers coated with different weight percentages of Cytop and Teflon solutions are precisely measured by AFM. The contact angles of deionized water droplets on the Cytop and Teflon layers of different thicknesses are measured using an optical microscope from the side. The results of the contact angle measurements of deionized water droplets on Cytop layers indicate that layers thicker than 3 nm maintain an angle of approximately 110°; the contact angles of the drops on Cytop layers thinner than 3 nm abruptly drop and decrease as the layer becomes thinner. The Teflon layers show a similar trend to the Cytop layers, except that the critical thickness is larger (7 nm). The contact angle hysteresis for different thicknesses of Cytop and Teflon layers is investigated by the tilting plate method. The results reveal that for thin hydrophobic layers, the contact angle hysteresis is over 10° but decreases as the layer thickness increases. When the layers are thicker than 12 nm, the contact angle hysteresis is reduced by approximately 4° for Cytop and 7° for Teflon and becomes saturated. To investigate the effect of aging on the surface wettability and hysteresis, the film stability of Cytop and Teflon layers is separately tested. The initial contact angles are reduced about 1.2% for Cytop and 1.6% for Teflon within 1 day and then maintained up to 10 days, while the initial contact angle hysteresis is increased about 117% for Cytop and 39% for Teflon within a day and then maintained up to 10 days, regardless of the thickness of the Cytop and Teflon layers. Based on the results of the surface wettability and hysteresis, an optimum hydrophobic layer thickness of 12 nm is established. Finally, the thickness effects of the hydrophobic layer on the operating voltage in EWOD actuation are tested. As expected, the operating voltage for thin hydrophobic layers is lower than that for thick layers. For the Cytop layer, the saturation voltages for 12 nm, 500 nm, and 1500 nm are 80 V, 100 V, and 120 V, respectively. Similarly, for the Teflon layer, the saturation voltages for 12 nm, 600 nm, and 1600 nm are 90 V, 110 V, and 130 V, respectively. The relative differences in the saturation voltages for both materials with respect to thickness are approximately the same.

1. Introduction

In recent years, research interests have been increasing with regard to the applications of lab-on-a-chip and biochip systems for blood sample preparation, real time polymerase chain reaction (PCR), and safety and efficiency testing for new drugs [1–3]. The most important technology in the development of the systems is microfluidics, which handles extremely small fluid volumes, down to less than picoliters. With respect to fluid mechanics, the Reynolds number ($Re = \rho VL/\mu$, where ρ, V, L, and μ are the density, mean velocity, characteristic length, and dynamic viscosity of a fluid, respectively) is a reference parameter to characterize flows and identify the dominant governing forces [4]. As the size of a fluidic system decreases, the Reynolds number linearly decreases, while the viscous forces become more dominant. As a consequence, the fluid pumping techniques commonly used in macrochannels may become inefficient for driving fluid plugs in microchannels because of the extremely high resistance of the viscous forces [5,6].

As alternatives, various microfluidic manipulation technologies have been developed that are mainly based on capillary forces and electrokinetic methods, such as electrophoresis, electroosmosis and dielectrophoresis [7,8]. Electrowetting-on-dielectric (EWOD) is another method of microfluidic manipulation in the form

* Corresponding author. Tel.: +82 31 330 6346.
** Corresponding author.
E-mail address: skchung@mju.ac.kr (S.K. Chung).

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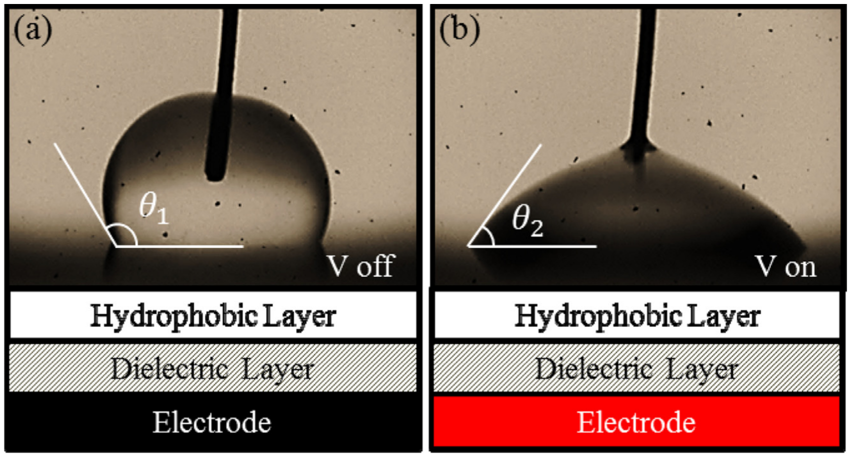

Fig. 1. Electrowetting-on-dielectric (EWOD) principle: (a) initial state and (b) after voltage is applied between conducting droplet and electrode. The electrode is covered with a hydrophobic dielectric layer, causing the droplet to spread over the surface because of the change in surface wettability.

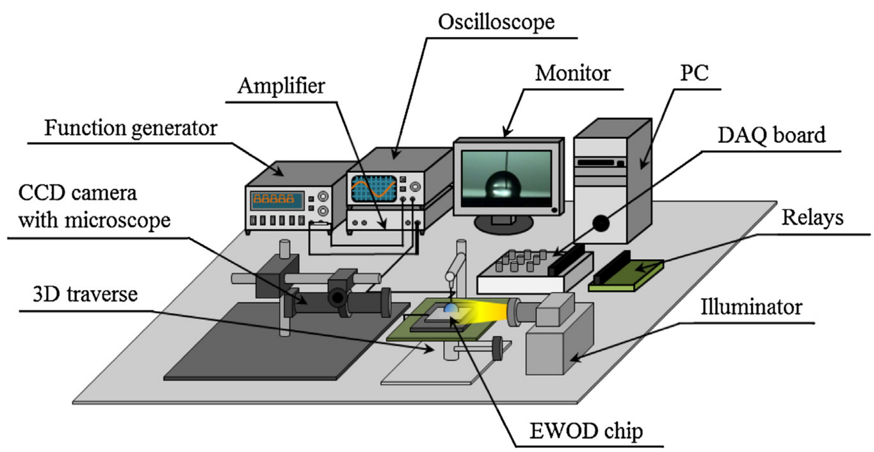

Fig. 2. Schematic diagram of experimental setup, which mainly consist of electrical and optical systems.

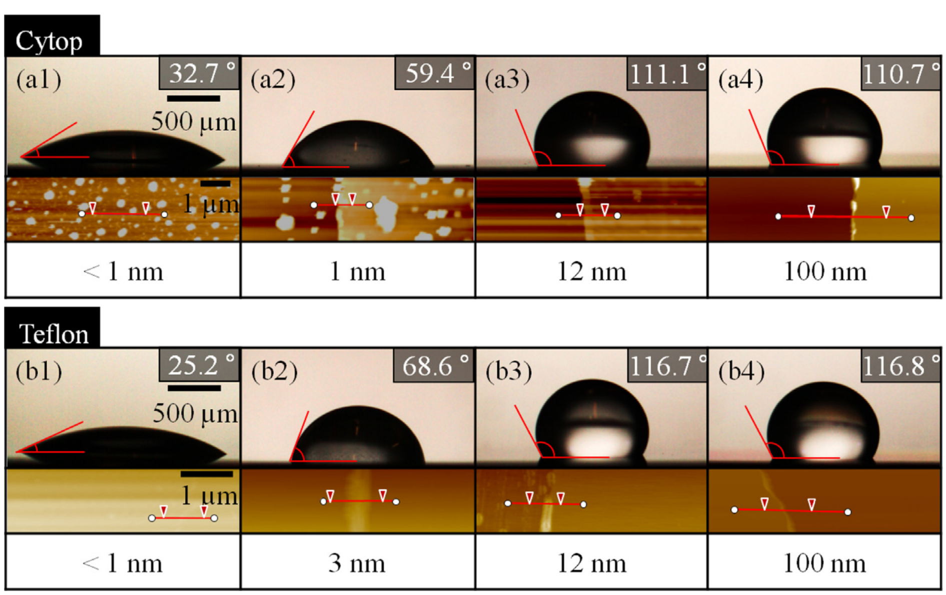

Fig. 3. Contact angles of water droplets on Cytop and Teflon layers with different thicknesses measured by atomic force microscopy (AFM). Note that the images beneath the droplets were captured by AFM during the layer thickness measurements.

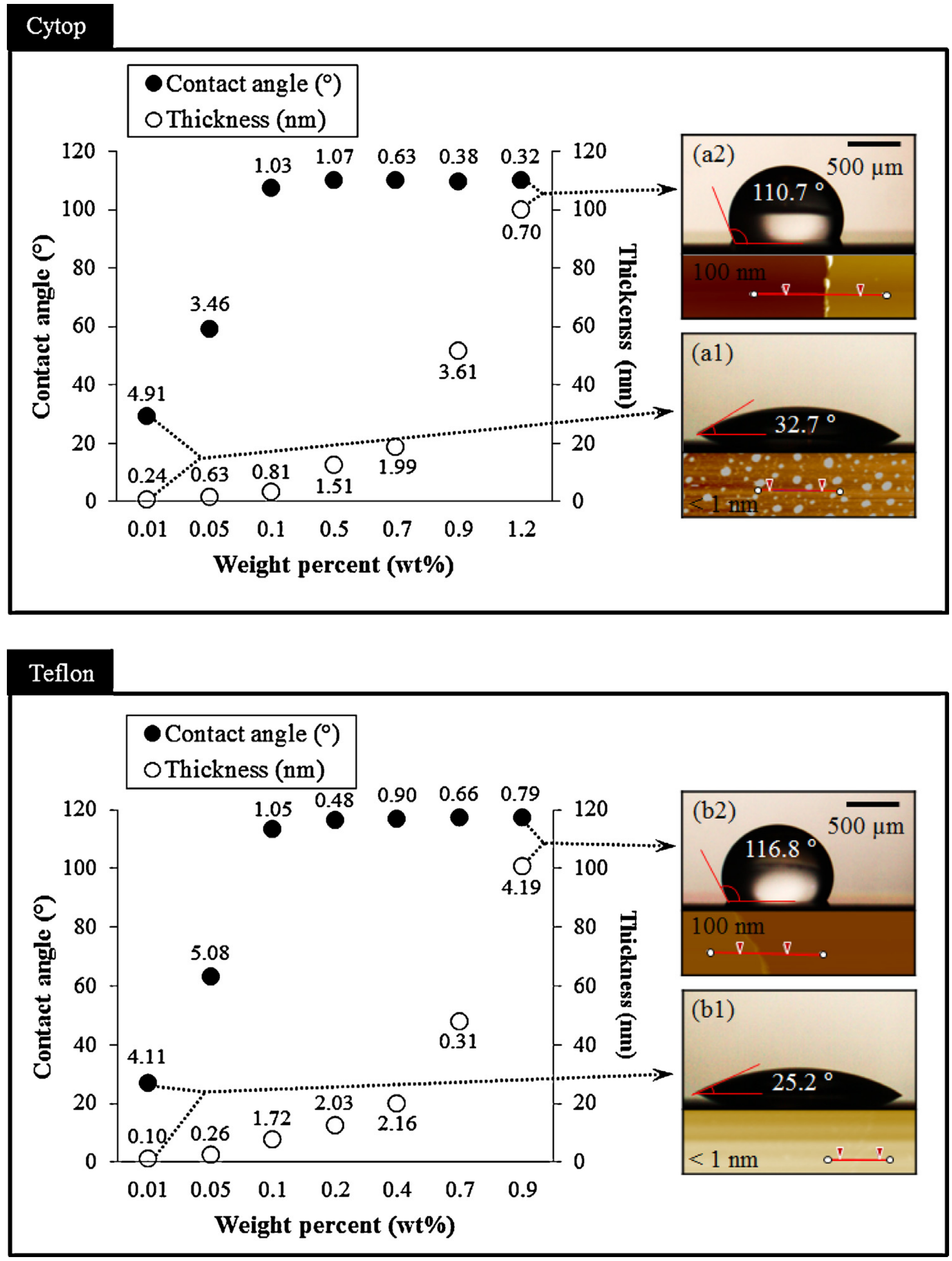

Fig. 4. Contact angles of water droplets and thicknesses of thin layers with respect to weight percentages (wt%) of Cytop and Teflon. Note that the number near each data indicates the standard deviation.

of discrete fluids (most commonly, droplets) without channel networks [9,10]. This is fundamentally different from other existing methods that make use of complicated microchannels. EWOD is known to be one of the most efficient and feasible microfluidic technologies owing to its advantages, such as its use of extremely small fluid volumes, low power consumption, and fast response time [11]. Many research groups have investigated the electrowetting principle and developed useful applications for liquid lens, electrowetting displays, and light guides [12–15].

However, a bottleneck for the commercialization of applications based on the EWOD technology is the use of high voltages. In many previous studies, a thick dielectric layer (>10 μm) was used, so obtaining a significant contact angle change required a high voltage (>200 V) [16,17]. This is much higher than the voltage used for most mobile electronic devices, such as smart phones and PDAs, because they currently operate at less than 15 V. To reduce operating voltages in EWOD, some research groups have applied a thin dielectric material with high dielectric constants, and others have applied multiple dielectric layers with a two-level-metal electrode [18–20]. However, none of them have focused the thickness effects of the hydrophobic layer for the purpose of voltage reduction.

Most research groups have used two separated materials for the dielectric and hydrophobic layers, because EWOD actuation requires an initial high hydrophobic surface (110–120) [21]. Various organic and inorganic materials, such as Parylene and silicon dioxide, have been used for dielectric layers, whereas two materials, Cytop and Teflon, have been widely used for hydrophobic layers in EWOD platforms [22]. Although the hydrophobic

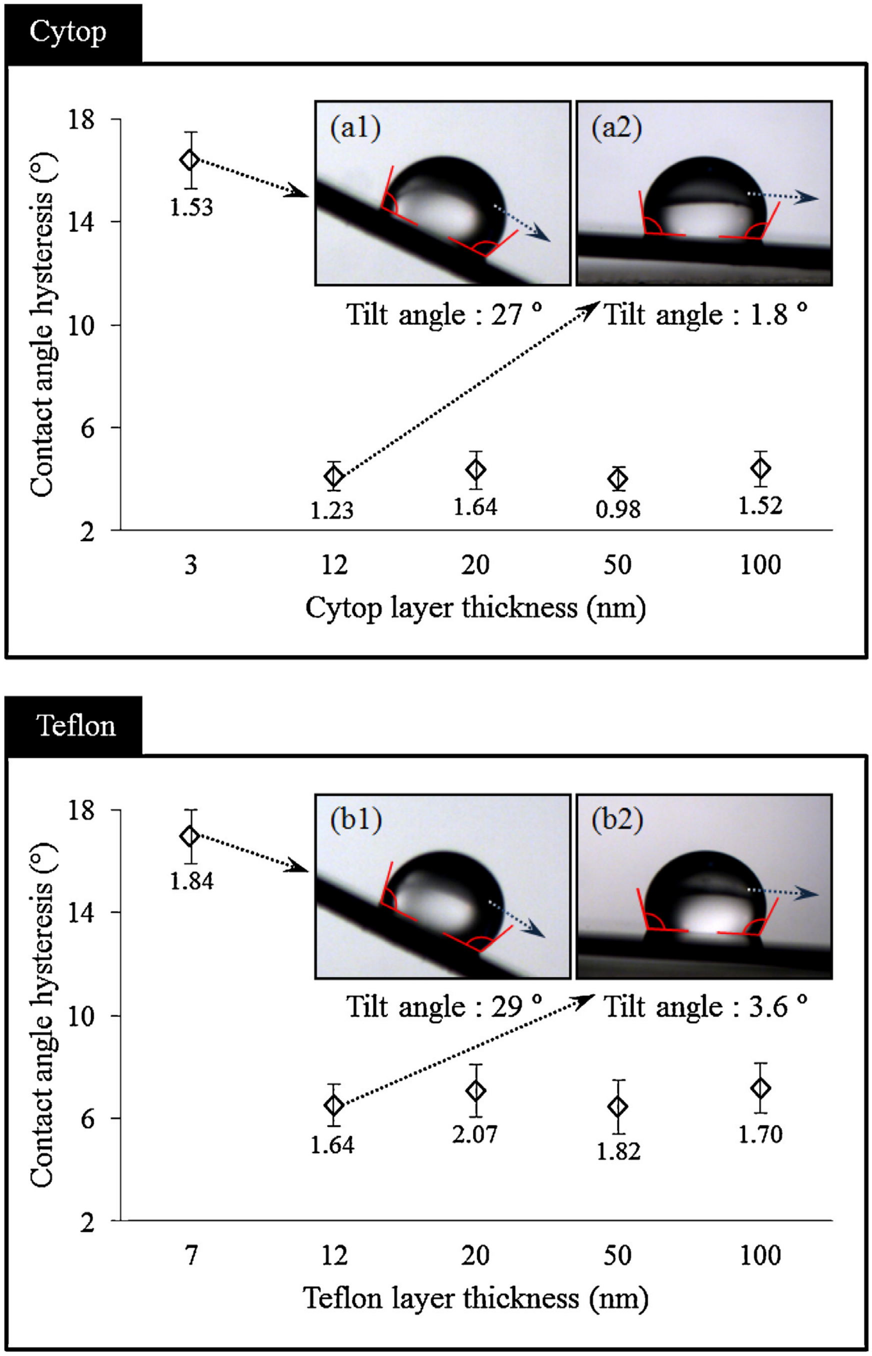

Fig. 5. Contact angle hysteresis with respect to the thickness of Cytop and Teflon layers based on the tilting plate method. Note that the number and bar near each data indicate the standard deviation and error bar respectively.

and dielectric layers were sequentially deposited on the electrode and acted as parallel capacitors during EWOD actuation, the thickness effects of the hydrophobic layer have been often neglected, because the thickness and the dielectric constant of the hydrophobic layer were both smaller than the corresponding parameters of the dielectric layer. However, research groups are applying thin dielectric polymer-based materials, increasing the importance of the thickness effects of the hydrophobic layer. In this paper, we present the investigation of the surface wettability and the hysteresis of hydrophobic layers, Cytop and Teflon, for different thicknesses using atomic force microscopy (AFM) for the purpose of operating voltage reduction in EWOD systems. Note that a preliminary report on this work was presented at the International Conference on Micro Electro Mechanical Systems held in Taipei, Taiwan [23].

2. Theoretical background

About a hundred years ago, Gabriel Lippmann discovered the electrocapillary phenomenon in which the interfacial tension of mercury in an electrolyte solution could be modified by applying electric potentials between the mercury and the solution [24]. However, the electrolysis caused by the flow of electric current through electrolyte solutions obstructed the development of this

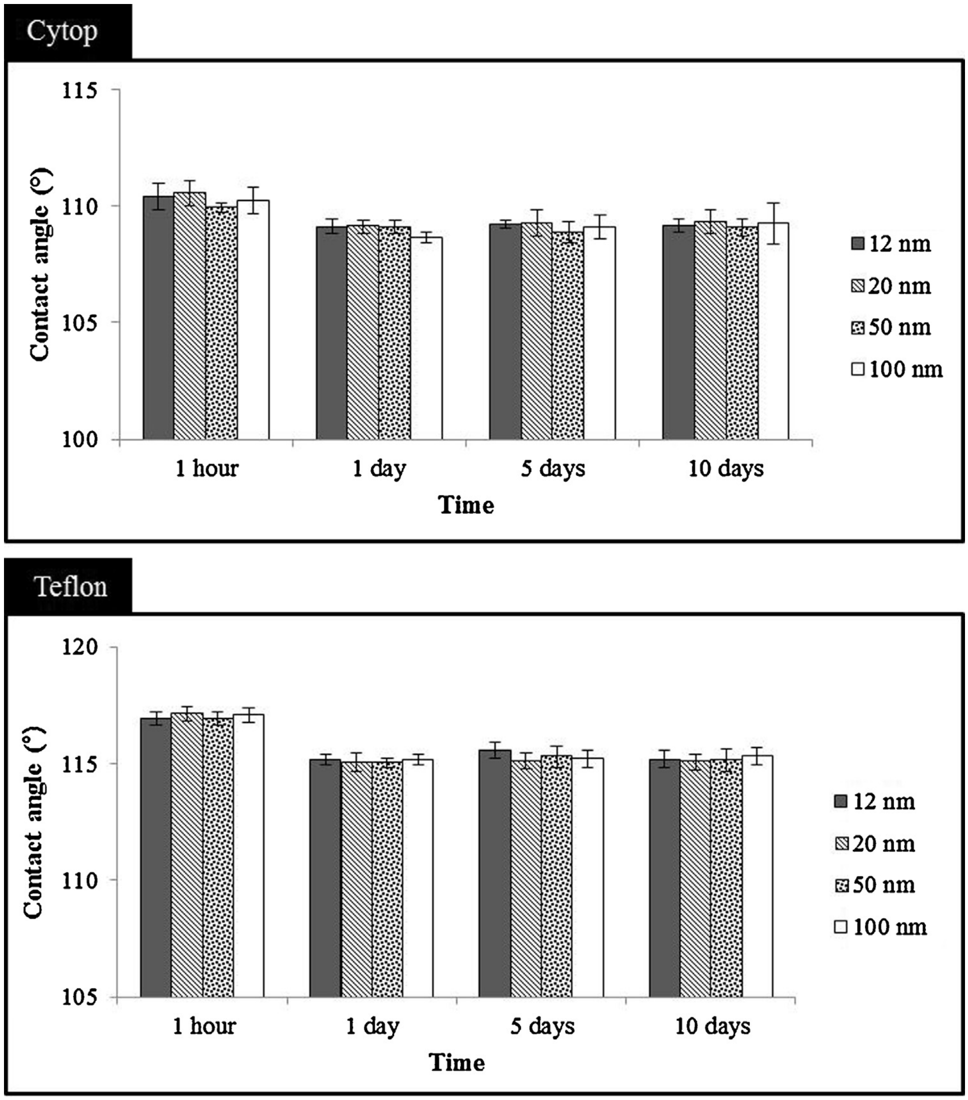

Fig. 6. Contact angles of water droplets on Cytop and Teflon layers for different thicknesses with respect to time.

technology into practical applications. A century later, Berge et al. [25,26] introduced an electrode covered with a thin hydrophobic dielectric layer in a sessile water droplet test. They achieved not only a large modification in the contact angle, but also a reversible droplet operation with minimal electrolysis. This process is called electrowetting, or to be precise, electrowetting-on-dielectric (EWOD) [27,28]. EWOD modifies the surface wettability of an electrode coated with a thin hydrophobic dielectric layer via an applied electric voltage. Hence, EWOD actuation encourages a conductive liquid droplet to expand on the insulated electrode surface by converting the applied electrical energy into mechanical energy, as shown in Fig. 1.

When a voltage is applied between a conductive droplet and a dielectric layer-coated electrode, the system acts as a capacitor, so that electric charges are built up near the solid and liquid interfaces in the dielectric layer. From a thermodynamic perspective, the surface energy decreases because of the accumulated charges around the three-phase contact line (TCL) under the applied electric field [29,30]. Berge et al. [25,26] also derived the relationship between the applied voltage and the change in contact angle by substituting Lippmann's equation with a voltage-dependent effective interfacial tension into the classical Young's equation for the droplet contact angle as follows:

$$\cos\ \theta = \cos\theta_0 + \frac{\varepsilon_0\varepsilon_1\varepsilon_2}{2(\varepsilon_1 d_2 + \varepsilon_2 d_1)\gamma}V^2 \quad (1)$$

where θ is the contact angle under the externally applied electric potential V, θ_0 is the equilibrium contact angle at $V = 0\,\mathrm{V}$, ε_0 is the vacuum permittivity, ε_1 is the dielectric constant of the dielectric layer, ε_2 is the dielectric constant of the hydrophobic layer, γ is the interfacial tension between the droplet and the surrounding fluid, d_1 is the thickness of the dielectric layer, and d_2 is the thickness of the hydrophobic layer. This equation shows that the contact angle can be modulated by the applied electric voltage and is affected by not only the thickness and dielectric constant of a dielectric material but also the thickness and dielectric constant of a hydrophobic material.

3. Fabrication of testing devices and experimental setups

In order to conduct EWOD-driven sessile droplet experiments, testing chips were prepared by standard microfabrication

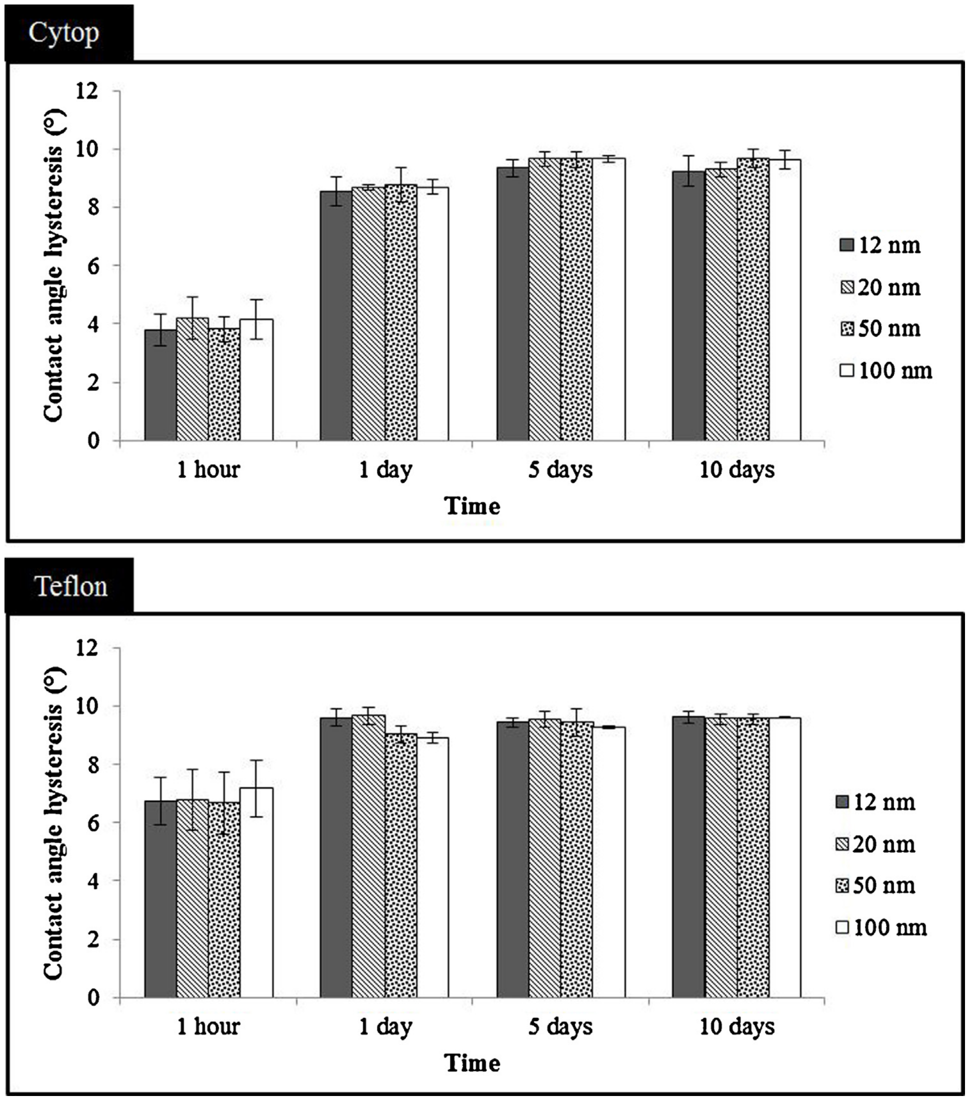

Fig. 7. Contact angle hysteresis of Cytop and Teflon layers for different thicknesses with respect to time.

processes. First, rectangular silicon pieces (3 cm × 2 cm) were cleaned for 10 min in a heated piranha solution, blow-dried by nitrogen gas, and dehydrated for 30 min on a hot plate. Second, a 1.6-μm-thick polyimide (Durimide 7505, Fujifilm Co.) layer was spin-coated (6000 rpm for 30 s) onto the silicon pieces as the dielectric layer. Third, Cytop (CTL-809M + CT-Solv.180, Asahi Glass Co., Ltd.) and Teflon (Teflon AF 1600®, DuPont + FC-40, 3M) layers were also separately spin-coated on the top of the polyimide layer. The thicknesses of the Cytop and Teflon layers were controlled by changing the Cytop and Teflon concentration in the spin-coating solution. The spin-coating parameters were 800 rpm for 20 s for the Cytop layers and 1000 rpm for 30 s for the Teflon layers. Finally, a DI water droplet (5 μl) was placed on the top of each chip using a syringe (600 Series MICROLITER™ Syringes model 62, Hamilton Company) and a sharp tip for a ground electrode was placed inside of the droplet using a three-dimensional traverse system for EWOD driven sessile droplet tests.

For an electrical EWOD signal, a sinusoidal signal with 1 kHz was produced by a function generator (33210A, Agilent Co.) and the signal was increased by a voltage amplifier (PZD700, Trek Co.), as shown in Fig. 2. The signal was transmitted to the EWOD chip through photo-coupled relays (PhotoMos®, AQW614EH, Aromat Co.) controlled by a digital I/O board (DAQ pad-6507, NI co.) and PC-based LabVIEW code. Experimental results were captured by a charge coupled device (CCD) camera (EO-1312C, Edmund Optics) integrated with a zoom lens (VZMTM 450i eo, Edmund Optics) and saved in a personal computer.

4. Experiment results and discussion

Hydrophobic measurements of Cytop and Teflon layers were carried out for different layer thicknesses by depositing different weight percentages of Cytop and Teflon solutions on rectangular silicon pieces (3 cm × 2 cm) with a spinner. The thicknesses of each sample spin-coated with Cytop or Teflon layer were precisely measured by AFM (XE-100, Park Systems Corp.), as shown in the images beneath the droplets in Fig. 3. Note that for the measurement reliability all tests in this work were repeated at least 5 times. The contact angles of the DI water droplets (5 μl) on the different samples were separately measured by a microscope from the side, as described in Fig. 2. The results of the contact angles for different thicknesses of Cytop and Teflon layers were plotted in Fig. 4. The contact angles of DI water droplets on Cytop layers thicker than 3 nm maintain an angle of approximately 110°; the contact angles of the droplets on Cytop layers thinner than 3 nm abruptly dropped and decreased as the layer thickness decreased. The Teflon layers

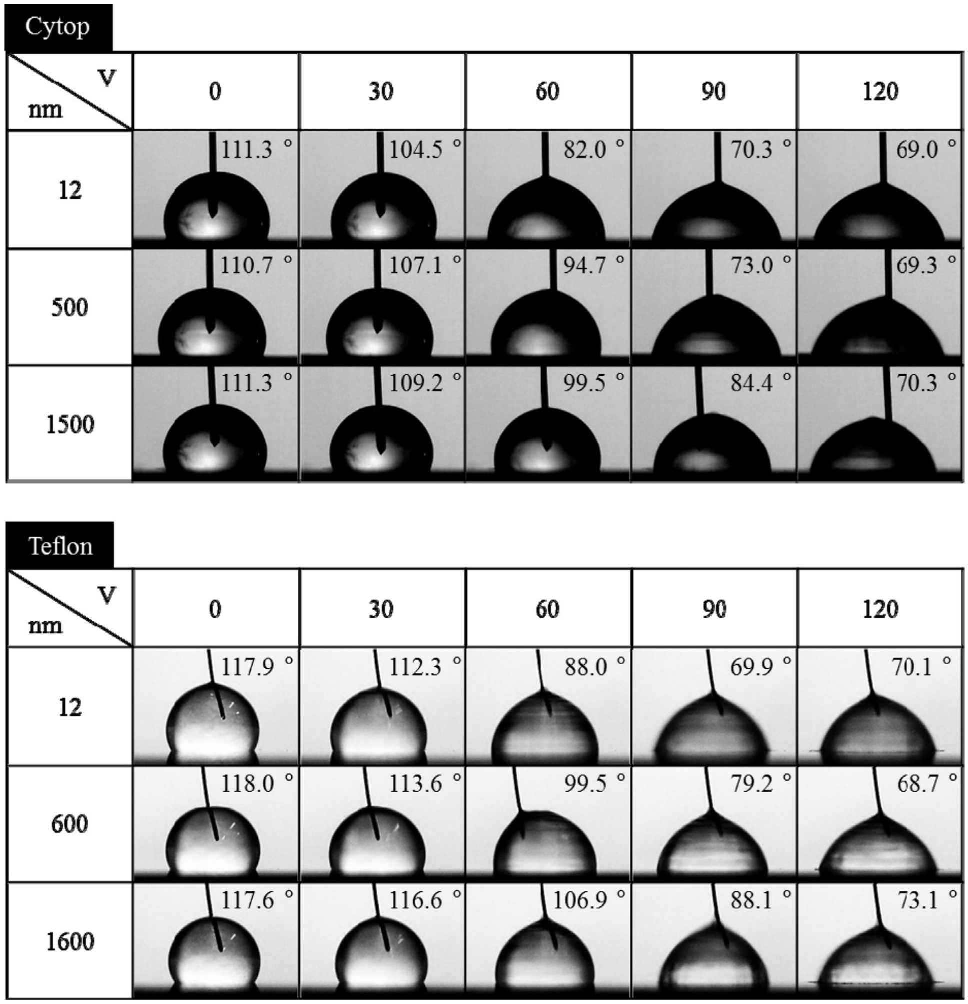

Fig. 8. Snap shots of EWOD-driven water droplets on different thicknesses of Cytop and Teflon layers.

showed a similar trend to the Cytop layers, except that the critical thickness was larger (7 nm). The contact angles of the DI water droplets on the Teflon layers thicker than approximately 7 nm maintain an angle of approximately 117°, whereas the contact angles of the droplets on the Teflon layers thinner than 7 nm abruptly dropped and decreased as the layer became thinner.

The investigation of the contact angle hysteresis of Cytop and Teflon layers for different thicknesses was conducted via the tilting plate method to obtain the optimum thickness of the hydrophobic layer, as shown in Fig. 5 (insets). DI water droplets (5 μl) were dispensed on silicon pieces covered with different thicknesses of Cytop and Teflon layers, and then the pieces were tilted by using a tilt stage installed beneath the pieces until the droplets released and slipped down. As the droplets sitting on the silicon pieces were tilted, the advancing contact angle on the downhill side increased while the receding contact angle on the uphill side decreased. Directly prior to the droplets releasing, the difference between the advancing and receding contact angles for each sample, i.e., the contact angle hysteresis, was measured by a microscope and plotted in Fig. 5. The contact angle hysteresis of the water droplets on the Cytop layers thinner than 3 nm was over 10°; it decreased as the layer thickness increased. When the Cytop layers were thicker than 12 nm, the contact angle hysteresis saturated at an angle approximately 4° smaller. The Teflon layers also showed similar results. When the thickness of the Teflon layer was thinner than 7 nm, the contact angle hysteresis was over 10°; when the thickness was larger than 12 nm, its value saturated at an angle approximately 7° smaller.

The film stability test was separately conducted to investigate the effect of aging on the surface wettability and hysteresis of Cytop and Teflon layers for different thicknesses. The results of the contact angles of DI water droplets (5 μl) on Cytop and Teflon layers of different thicknesses for different times – 1 h, 1 day, 5 days, and 10 days – were plotted in Fig. 6. The contact angles of the DI water droplets were initially 110° for the Cytop layer and 117° for the Teflon layer, reduced about 1.2% and 1.6% respectively within 1 day, and maintained up to 10 days, regardless of the thickness of the Cytop and Teflon layers. The results of the contact angle hysteresis of Cytop and Teflon layers of different thicknesses for different times were also plotted in Fig. 7. The contact angle hysteresis was initially approximately 4° for the Cytop layer and 7° for the Teflon layer, increased about 117% and 39% respectively within 1 day, and maintained up to 10 days. Note that the thickness effect of the Cytop and Teflon layers thicker than 12 nm on the contact angle hysteresis over time was also negligible within the range of measurement errors.

Based on the results of the surface wettability and hysteresis, the thicker hydrophobic layer emerges as a desirable option. However, a thicker layer requires higher voltages for EWOD actuation and is therefore unsatisfactory for low voltage operation. Lastly, the effect of the hydrophobic layer thickness on the operating voltage in EWOD actuation was tested. Six different samples sequentially deposited with a 1.6-μm-thick polyimide layer as the dielectric layer and Cytop or Teflon layers of three different thicknesses as the hydrophobic layers were prepared. Fig. 8 shows the snap shots of

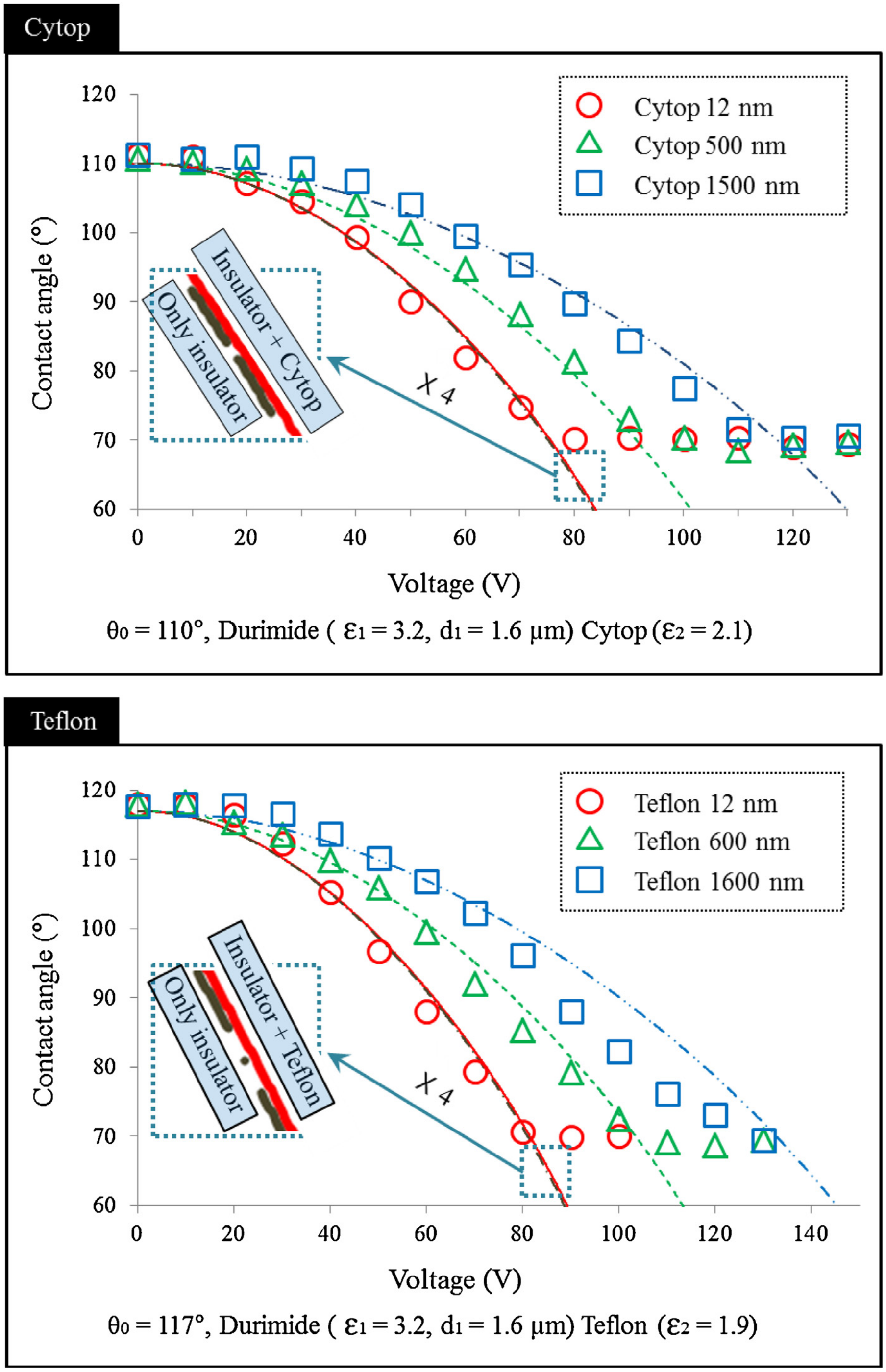

Fig. 9. Contact angles with respect to applied voltages to the Cytop and Teflon layers for different thicknesses. Note that the experimental and theoretical results are plotted as symbols and lines, respectively.

EWOD-driven sessile droplet experiments of the six different samples. When a voltage was applied between the silicon pieces and the ground electrodes placed inside of water droplets, the contact angles of the droplets were modified. The experimental and theoretical (Lippmann–Young equation) results were plotted in Fig. 9. As expected, the operating voltage for thin hydrophobic layers is lower than that for thick layers. For the Cytop layer, the saturation voltages for 12 nm, 500 nm, and 1500 nm are 80 V, 100 V, and 120 V, respectively. Similarly, for the Teflon layer, the saturation voltages for 12 nm, 600 nm, and 1600 nm are 90 V, 110 V, and 130 V, respectively. The relative differences in the saturation voltages for both materials with respect to thickness are approximately the same. Based on the results of the surface wettability, hysteresis, and EWOD tests, an optimum hydrophobic layer thickness is determined to be 12 nm.

5. Conclusions

Electrowetting-on-dielectric (EWOD) is currently contributing greatly in many areas, from microfluidics to optics. However, the high-voltage requirement of EWOD is one of the critical issues that must be resolved for the commercialization of potential applications that are demanded in various industrial fields. This study evaluates the optimum thickness of a hydrophobic layer for operating voltage reduction in EWOD systems. First, the surface wettability of Cytop and Teflon layers with different thicknesses is investigated by measuring the contact angles of DI water droplets on hydrophobic layers. The results indicate that for both Cytop and Teflon layers, the contact angles abruptly drop and continuously decrease with thickness at the critical thickness of each layer; the critical thicknesses are 3 nm and 7 nm for Cytop and Teflon, respectively. Second, the contact angle hysteresis of Cytop and Teflon layers with different thicknesses is investigated via the tilting plate method. This experiment shows that the contact angle hysteresis is over 10° for thin hydrophobic layers, but it decreases as the thickness of the hydrophobic layer increases. Third, the effect of aging on the surface wettability and hysteresis of Cytop and Teflon layers is separately investigated. The results show that the initial contact angles are reduced about 1.2% for Cytop and 1.6% for Teflon within 1 day and then maintained up to 10 days, while the initial hysteresis is increased about 117% for Cytop and 39% for Teflon within a day and then maintained up to 10 days, regardless of the thickness of the Cytop and Teflon layers. Based on the test results, an optimum thickness of 12 nm for the hydrophobic layer is suggested. Finally, the effect of the optimum thickness of the hydrophobic layer on the operating voltage in EWOD actuation is tested and confirmed. This study contributes to the voltage reduction for future EWOD applications without system design modifications.

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Biographies

Jeong Byung Chae received the Bachelor's degree of mechanical engineering from Myongji University in 2012. He currently is a graduate student in Myongji University and his research interests lie on the optimization of EWOD (electrowetting-on-dielectric) systems.

Jun O Kwon received the Bachelor's degree of mechanical engineering from Myongji University in 2012. He currently is a graduate student in Myongji University and his research interests lie on the development of an electromagnetically driven microrobot swimming in blood vessels.

Ji Sun Yang received the Bachelor's degree of mechanical engineering from Myongji University in 2012. He currently is a graduate student in Myongji University and his research interests lie on biomimetic robots and EWOD (electrowetting-on-dielectric) applications.

Dohyun Kim is an assistant professor of Department of Mechanical Engineering at Myongji University, Republic of Korea. After receiving B.S and M.S. degrees (mechanical engineering) at Sogang University, Korea, he earned Ph.D. degree at University of California, Los Angeles (electrical engineering), United States. He also worked as a postdoctoral scholar at University of California, Berkeley (bioengineering). His main research interests are microfabrication, microfluidics, BioMEMS, chemical/biochemical sensors, and protein chemistry.

Kyehan Rhee is a professor of the department of mechanical engineering at the Myongji University in Korea. He received his Ph.D. degree from the University of Minnesota, Minneapolis, U.S.A and worked as a Post-Doctoral Fellow in the Pennsylvania State University, State College, U.S.A. His research interests include hemodynamics, microfluidics, polymer smart material actuators and their application in biomedical devices. He serves as an editor of the International Journal of Precision Engineering and Manufacturing and the Journal of Biomechanical Science and Engineering.

Sang Kug Chung is an associate professor of the department of mechanical engineering at the Myongji University in Korea. He received the Ph.D. degree in Mechanical Engineering and Materials Science from the University of Pittsburgh in 2009 along with the Graduate Research Excellence Award. He received the M.S. degree from Pohang University of Science and Technology (POSTECH) and B.S. from Myongji University. He had worked for the development of the world first Liquid Lens at Samsung Electro-Mechanics from 2003 to 2009 and researched oil spills at Advanced Fluids Engineering Research Center from 2000 and 2001. Upon joining the faculty at Myongji University in 2009, he has directed the Microsystems Laboratory. His research is in microfluidics and MEMS, including design and fabrication of micro/nano actuators and systems.

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