Electrowetting devices with transparent single-walled carbon nanotube electrodes

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Microfluidic devices based on the electrowetting principle, more specifically electrowetting on dielectric, were fabricated using transparent single-walled carbon nanotube films as electrodes. The films were spray coated on glass and polyethylene terephalate substrates. The transmittance and sheet resistance remain unchanged after patterning the films using typical photolithography and plasma etching. Operation of water droplets over the patterned nanotube electrodes was demonstrated, and the performance was found to be comparable to that over the usual metal electrodes. The requirement of transparent electrodes is estimated for displays based on electrowetting mechanism, and nanotube films indicate promise for such a type of devices. © 2007 American Institute of Physics. [DOI: 10.1063/1.2561032]

Electrowetting is a promising principle for the generation of microscale fluid movement, currently led by the configuration of electrowetting on dielectric (EWOD).1 Wettability of a solid surface increases when a voltage is applied between the surface and a conducting liquid on it. Under certain circumstances, a motion of droplets can be induced. The technology has been used for many applications including electronic papers,2 adaptive lenses,3 and optical filters.4 The technology has been used for many applications including electronic papers,2 adaptive lenses,3 and optical filters.4

Typically, the sheet resistance of ITO is less than 100 Ω/sq with 80% transmittance in the visible range.9 The liquid resistance and capacitance depend on the type of solution and ion concentration. For typical ionic water solution (e.g., 0.01–1 M KCl), Cw can be ignored and Rw is in the 1–500 Ω range.5 Cd is in the 1 pF–1 nF range in microfluidic devices based on the electrowetting principle, more specifically electrowetting on dielectric, were fabricated using transparent single-walled carbon nanotube films as electrodes. The films were spray coated on glass and polyethylene terephalate substrates. The transmittance and sheet resistance remain unchanged after patterning the films using typical photolithography and plasma etching. Operation of water droplets over the patterned nanotube electrodes was demonstrated, and the performance was found to be comparable to that over the usual metal electrodes. The requirement of transparent electrodes is estimated for displays based on electrowetting mechanism, and nanotube films indicate promise for such a type of devices.

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Fig. 1. (Color online) (a) Cross-sectional schematic of an EWOD device (adapted from Ref. 2). (b) Equivalent circuit for the EWOD configuration (adapted from Ref. 1). (c) dc charging time constant for EWOD activation vs connecting resistance. (d) ac voltage loss during EWOD activation vs connecting resistance.
idics devices when the electrode is small (<10 mm) and the dielectric layer is thin (<1 µm). In our devices, the electrode is 1.5 × 1.5 mm² in dimension, the dielectric is 500 nm thick Si₃N₄, and the hydrophobic layer is 200 nm thick AF1600 Teflon. The calculated C_d per electrode pad is approximately 30 pF. For an applied dc signal [Fig. 1(c)], the capacitor charging time constant R_sC_d is less than 1 ms even for a 1 MΩ resistance, which is fast enough for typical display applications (refresh rate is usually less than 100 Hz). For an applied ac signal, high connecting resistance could play applications.

SWCNT films were spray coated on the glass and PET substrates. Arc-discharged nanotube powders purchased from Carbon Solution Inc. were dispersed in water with 1% weight sodium dodecyl sulphate and sprayed onto the substrates that have been heated to 80 °C. Before the spraying, the substrates need to be soaked in 1% weight 3-aminopropyltriethoxysilane (silane) water solution for 5 min to improve the adhesion of nanotube films to the substrates. Figure 2(a) shows the atomic force microscope (AFM) image of films on glass substrate. The transmittance of sprayed nanotube films can be tuned simply through the spraying steps and the solution concentrations. Typically, five spraying steps with 1 mg/mL nanotube solutions will result in films with 80% transmittance. The sheet resistance and yield an approximate 30% voltage drop.

The patterned film’s size is limited to that of the membrane. However, the sprayed film coverage area can be as large as the substrate. We tested various types of plasma and found that Ar, O₂, CF₆, or CF₄ plasma can effectively etch nanotubes. The fabrication steps are shown in Fig. 3(a). First, the nanotube films are spray coated, and then photosensor (PR) is spin coated on top, exposed, and developed. After photolithography, gas plasma within a reactive ion etcher (RIE) system (i.e., 100 W rf power and 5 min etching with Ar plasma) removes the nanotubes in the unprotected areas. Finally the PR is stripped off by acetone, and the sample is rinsed in de-ionized (DI) water and blown with nitrogen gas to dry. An alternative process to fabricate patterned films is the lift-off process [Fig. 3(b)]. In this method, PR is first patterned, and the substrate is then treated with silane solution. The nanotube film is sprayed over the substrate and the remaining PR is removed by soaking the substrate in acetone. Finally, the substrate is rinsed in DI water and blown to dry.

We applied both methods to deposit patterned nanotube films. Figure 4(a) shows a patterned nanotube film on a 4 in. glass wafer and (b) shows a patterned film on a PET flexible substrate. The square pads visible in the pictures are gold contact pads; the nanotube thin-film electrodes are vaguely visible. Patterning resolution of 4 µm is achieved on both substrates [shown in Fig. 4(c)]. There are no distinguishable changes in sheet resistance and transmittance observed before and after repeating the lithographic patterning process five times. Figure 4(d) displays the transmittance of films before and after the patterning using method (a), where the
The Au pad is 2 conductive on glass is 1200 S/cm. Thus the Au pad resistance of a nanotube film reveals a 5 nm roughness, comparable to the top hydrophobic layer. AFM image of the Teflon coated chemical vapor deposition, and open the contact pads by various plasma etching recipes offers a simple method to fabricate patterned nanotube films with high resolution.

To test nanotube films as the transparent conducting layers for electrowetting applications, we made EWOD devices using the configuration shown in Fig. 1(a). The steps are given as follows. (1) Spray coat nanotube films (80% transmittance) on 4 in. glass or PET substrates; (2) evaporate and lithographically pattern (by wet etching) the 20 nm Cr/200 nm Au as electrical contact pads for applying driving voltage; (3) lithographically pattern (by plasma RIE) the nanotube film to define EWOD pixel electrodes; (4) coat 500 nm Si3N4 as dielectric layer by plasma-enhanced chemical-vapor deposition, and open the contact pads by CF4/O3 RIE pattern; (5) spin coat 200 nm AF1600 Teflon as the top hydrophobic layer. AFM image of the Teflon coated on a nanotube film reveals a 5 nm roughness, comparable with the roughness (2.5 nm) of Teflon spin coated on an ITO/glass substrate; and (6) dice 4 in. glass or PET substrate into pieces for electrowetting tests. Figure 4(a) shows the 4 in. glass wafer with 12 devices before the dicing.

We evaluated the contact resistance between the metal pad and the patterned nanotube lines, because it can also be a source of high resistance between the applied voltage and EWOD electrodes. By probing the metal pad and nanotube pixel area [Fig. 5(a)], the measured resistance was 20 kΩ, which includes $R_{metal}$, $R_{contact}$, and $R_{nanotube}$. The dimension of the Au pad is $2 \times 2$ mm$^2$ with 200 nm thickness, and the nanotube line is 100 μm wide, 7 mm long, and 30 nm thick. Au conductivity is $0.45 \times 10^9$ S/cm, and sprayed nanotube conductivity on glass is 1200 S/cm. Thus the Au pad resistance is only several ohms, and the nanotube line resistance is 19.4 kΩ. The contact resistance of a 100 μm wide nanotube film with Au pad is about 0.6 kΩ, which is more than 20 times less than the resistance of the nanotube line.

To verify the electrowetting principle, we first measured contact angle versus applied voltage for pure water on SWCNT devices. Contact angles were measured by an optical contact angle measurement system (FTA 1000) and the measured data [Fig. 5(b)] follow the same trend found with metal electrodes. We also tested the droplet translation on the nanotube EWOD devices. To provide the electrical ground, 200 nm AF1600 Teflon was spin coated onto a non-patterned nanotube glass plate and used as the top plate shown in Fig. 1(a). A 200 nL droplet was then sandwiched between the two parallel plates with a 100 μm spacer. Then, through the corresponding Cr/Au contact pads, a 50 V dc voltage was applied to the desired EWOD electrode to translate the droplet toward it. Figures 5(c)–5(e) show snapshots of a droplet translated between the electrodes. Droplet translation rate was as fast as that of devices using ITO electrodes in a similar condition (10 mm/s).2

In conclusion, we tested patterned nanotube films as electrodes in EWOD devices, the performance of which was indistinguishable from those obtained from ITO electrode devices. We also evaluated the effect of high sheet resistance on the time delay and the voltage drop and found that high sheet resistance up to 10 kΩ/sq has negligible effect. Therefore, highly conductive ITO is not necessary and, under certain circumstances, not suitable. For example, in flexible device applications based on the electrowetting mechanism, such as electronic paper, highly conducting ITO is normally around 100 nm thick and easy to crack. However, thinner ITO is difficult to form into a continuous conducting network due to its one-dimensional properties. For nanotubes with high length/width ratio, the percolation threshold is extremely small and a conducting network is formed at low coverage. Even such a monolayer, nanotube network will be conductive enough for EWOD-type devices and obviously will possess near-perfect optical transparency.

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