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Large scale, highly conductive and patterned transparent films of silver nanowires on arbitrary substrates and their application in touch screens

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Abstract
The application of silver nanowire films as transparent conductive electrodes has shown promising results recently. In this paper, we demonstrate the application of a simple spray coating technique to obtain large scale, highly uniform and conductive silver nanowire films on arbitrary substrates. We also integrated a polydimethylsiloxane (PDMS)-assisted contact transfer technique with spray coating, which allowed us to obtain large scale high quality patterned films of silver nanowires. The transparency and conductivity of the films was controlled by the volume of the dispersion used in spraying and the substrate area. We note that the optoelectrical property, σ_{DC}/σ_{Op}, for various films fabricated was in the range 75–350, which is extremely high for transparent thin film compared to other candidate alternatives to doped metal oxide film. Using this method, we obtain silver nanowire films on a flexible polyethylene terephthalate (PET) substrate with a transparency of 85% and sheet resistance of 33 Ω/sq, which is comparable to that of tin-doped indium oxide (ITO) on flexible substrates. In-depth analysis of the film shows a high performance using another commonly used figure-of-merit, Φ_{TE}. Also, Ag nanowire film/PET shows good mechanical flexibility and the application of such a conductive silver nanowire film as an electrode in a touch panel has been demonstrated.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

High optical transparency and high electrical conductivity are two critical parameters in the choice of electrodes for many optoelectronic devices, such as liquid crystal displays, light emitting diodes, solar cells and touch panels [1–3]. Currently, tin-doped indium oxide (ITO) and fluorine-doped tin oxide (FTO) are used as the primary choice for transparent conductive electrodes (TCE) for such applications due to their high electrical conductivity and optical transparency [4–7]. However, the high cost of the indium source, lack of flexibility of ITO and the high processing temperature for its production has resulted in the quest for new electrode materials for flexible optoelectronic device applications. Various alternatives have been pursued by different groups towards this objective, among which thin metal films [8, 9] and metal grids [10, 11] have shown a performance comparable to ITO. However, these approaches suffer from the requirement for high vacuum equipment for processing and fabrication and they show limited bending ability. Carbon nanotube (CNT) films [12–16] and, more recently, graphene films [17–20] have been successfully used as TCE in organic light emitting diodes [21–23] and solar cells [24, 25], and have attracted significant interest. However, their performance in terms of sheet resistance and transparency are still inferior to ITO. Thus there is a need to continue the quest for new cheap and reliable

materials that can be used in a highly scalable method for a TCE material with comparable performance in terms of sheet resistance and transparency to that of ITO.

Metal nanowire networks have recently been used to fabricate TCE [26, 27] and the resulting metal nanowire TCE were successfully used in organic solar cells [26]. Both these reports represent promising results in the effort to replace ITO; however, a high temperature (200 °C) annealing step was required in the method used by Lee *et al* [26] to obtain good conductivity, as the silver nanowires used had a surfactant coating of polyvinylpyrrolidone (PVP) molecules, thus limiting these metal nanowires' application on flexible polyethylene terephthalate (PET) as a substrate. The transfer method reported by De *et al* [27] enables transfer of silver nanowire films to flexible substrates, but this method suffers from a long fabrication time and is inherently a chemical-intensive process requiring very careful and exhaustive transfer techniques. Very recently, large scale fabrication of TCE using silver nanowires has been demonstrated by our group [28] and others [29], but this still lacks the ability to obtain conductive electrodes on the arbitrary substrates required for applications in smart fabrics, thus creating a need for a versatile way of fabricating TCE using silver nanowires. Using a spray coating technique we tried to address some of the issues with our earlier work [28], in which fabrication of TCE was sensitive to the substrate and also required surface treatment in the case of glass before the transfer of nanowires. The size of the TCE fabricated was restricted by the size of the AAO membrane, thus limiting the throughput of the TCE. It also required several steps of PDMS mold fabrication, vacuum filtration and an intermediate step of AAO application to fabricate TCE using our earlier technique, as opposed to simple spray coating directly on the substrate of interest reported here to obtain similar high performance. No additional surface treatment on the substrate is required and, unlike earlier reported techniques [26–29], which require a flat smooth substrate, arbitrary substrates (glass, plastic and fabric) with various geometrical features can be coated using the spray coating technique.

In this paper, we demonstrate the use of a simple spray coating technique to fabricate highly conductive flexible TCE using silver nanowires with a sheet resistance (R_{sh}) value of 33 Ω/sq at an optical transmittance (T) of 85%, which is at least an order of magnitude lower than that obtained by CNT and graphene, and comparable to the values previously obtained using silver nanowires. Another advantage includes the ability to produce patterned silver nanowires of various line widths and geometries to obtain pixelated electrodes and also the production of conductive film on arbitrary substrates, including fabrics. We also demonstrated a reliable application of metal nanowire TCE as a touch screen, thus creating the possibility for its application in many optoelectronic devices. The spray deposition technique is a major coating technique used in the industry, is a simple and cheap method to fabricate TCE for large area application, and can be used on almost any substrate [30, 31]. It has been used previously by others to make conductive CNT films [32].

2. Fabrication and experimental details

Ag nanowires were purchased from Seashell Technologies as supplied with a concentration of 12.5 mg ml^{-1} in isopropyl alcohol (IPA). The mean length of the nanowires was 12.5 μm with a mean diameter of 75 nm as provided by the supplier. The dispersion was further diluted to a concentration of 0.208 mg ml^{-1} in isopropyl alcohol and was sprayed onto the substrate using a Paasche airbrush. As the dispersion is highly volatile, we did not need to heat the substrate to accelerate the evaporation of the fine droplets on the substrate. PDMS stamps were fabricated using a SYLGARD 184 silicone elastomer kit (Dow Corning, Inc.). Patterned PDMS was made by standard optical lithography using an SU-8-50 resist (MicroChem Corp.).

Scanning electron microscopy (SEM) micrographs were obtained using a JEOL 7001 field emission scanning electron microscope. Optical transmission spectra were obtained using a Varian Cary 50 Conc spectrophotometer, using bare PET or glass as the reference. The sheet resistance was measured using a four-probe technique by depositing silver paint at the corners in a square shape with sides of ∼3 mm and at least ten locations across the sample; values reported are the average value obtained across the entire film. An Agilent 4156B Semiconductor Parameter Analyzer was used as a source meter for sheet resistance measurements. Bending tests were performed by placing the film between two platforms and bending the films by reducing the distance between the platforms. Sheet resistance measurements were performed at various bending angles by placing four probes on the films with silver paint at the corners (see the inset in figure 4 for details).

3. Results and discussion

Figures 1(a) and (b) shows the optical micrographs of spray coated Ag nanowire film on glass and polyethylene terephthalate (PET) substrates respectively. The films are highly transparent, as the letters in the background can be clearly seen through the film and hence demonstrate the ability to fabricate highly transparent and scalable TCE on both rigid and flexible substrates. The spray conditions, such as the compressed air pressure, the distance between the nozzle and substrate, and the nozzle diameter were optimized to obtain uniform silver nanowire films. As the spray gun used for the demonstration of this technique is not fully automated, we could only increase/decrease the nozzle diameter, but did not know the exact size of the opening. Therefore any characterization of the film uniformity with the nozzle diameter could not be studied. However, we did observe that the uniformity of deposition improved as the nozzle diameter was decreased but that it resulted into a lower deposition rate. Therefore we optimized the size of the nozzle diameter during our deposition to obtain uniform TCE films in a reasonable deposition time. The uniformity of the films was also improved by decreasing the concentration of silver nanowires in isopropyl alcohol (IPA), although in this study we discuss only one typical concentration that we used to form a uniform film. The nozzle–substrate distance

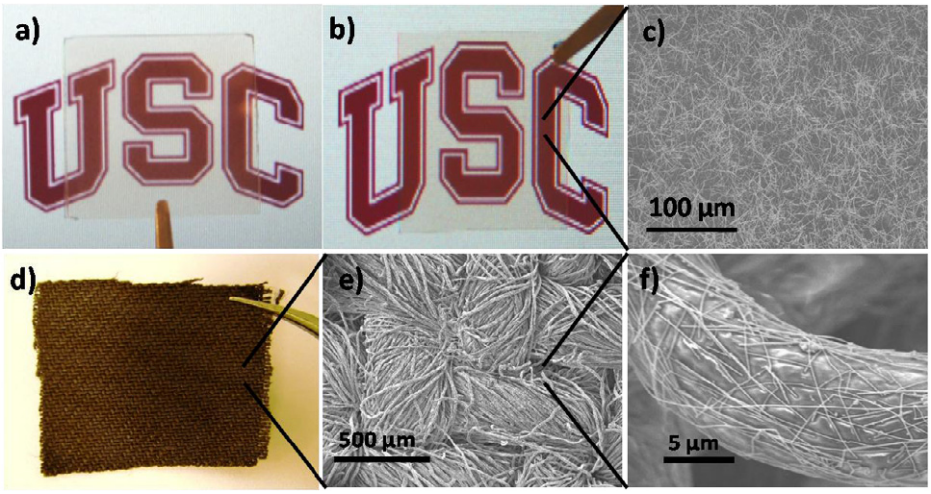

Figure 1. Silver nanowire films on various substrates. (a) Photograph of a silver nanowire film deposited on glass with dimensions, transmittance and sheet resistance 2 cm × 2 cm, 80% and 23 Ω/sq respectively. (b) Photograph of a silver nanowire film deposited on a PET substrate with dimensions, transmittance and sheet resistance of ∼2 cm × 2 cm, 80% and 25 Ω/sq respectively. (c) SEM image of the percolating nanowire distribution of the sample shown in (b), thus showing the uniform silver nanowire distribution. (d) Photograph of a Ag nanowire/fabric composite with dimensions of ∼2.5 cm × 2.5 cm. (e), (f) SEM images of the Ag nanowire network at various magnifications, showing nanowire distribution on individual fiber.

was maintained at ∼3 cm, as we observed that a further reduction in this distance resulted in non-uniform films, as the air flow on the substrate was sufficiently high to disturb the sprayed nanowire solution on the substrate before the solvent could evaporate. The uniformity of the film was analyzed by conducting SEM analysis at random different regions located far from each other on the sample, as shown in figure 1(b). One such SEM image is shown in figure 1(c). No apparent difference in the density of the nanowires was observed, and hence a highly uniform film over the entire substrate can be obtained by this technique. Wearable, lightweight and conductive fabrics can find applications in varied fields, such as static charge dissipation, wearable antennas and interconnects. Previously, a CNT/fabric composite has been fabricated by other groups [33]. Figure 1(d) shows the Ag nanowire/fabric composite that was fabricated by spraying Ag nanowire solution uniformly on a non-conducting fabric. Figures 1(e) and (f) show the SEM images of the fiber of the fabric at different magnifications. It can be seen that the Ag nanowires wrap around the fibers and the nanowire network is above percolation.

The spray coating technique cannot only be used to deposit uniform Ag nanowire films, it can also be used to obtain patterned nanowire films, which is a critical requirement to obtain in-plane pixelation for display devices such as OLED. We integrated the spray coating technique with the PDMS-assisted dry transfer technique [16, 28, 34–38] to obtain a patterned Ag nanowire film, thus giving an unprecedented ability to obtain both large scale patterned and unpatterned uniform nanowire films. PDMS stamp fabrication is described in section 2. To obtain the patterned films, Ag nanowires were first sprayed uniformly onto patterned PDMS, as schematically illustrated in figure 2(a). The receiving substrate was placed on the hot plate at 120 °C, and a PDMS stamp bearing the nanowires was pressed against it for ∼1 min. The PDMS stamp was removed slowly, resulting in the transfer of the silver nanowire film to the substrate, as schematically illustrated in figure 2(b). Although the nanowires were sprayed uniformly on the entire PDMS stamp, only the nanowire deposited on the raised pattern on PDMS was transferred onto the receiving substrate, as shown in the photograph of a patterned Ag nanowire on PET in figure 2(c). The inset illustrates the transparency of the patterned Ag nanowire film. The length of the printed squares is 1 mm, with a spacing of 0.5 mm between them. Figure 2(d) shows the SEM image of the region, with Ag nanowire distributed uniformly in the pixel, suggesting high fidelity of the patterned transfer process. Several kinds of patterns can be fabricated, with the minimum feature size limited by the resist used in optical lithography to pattern the stamp. The PDMS-assisted dry transfer method for silver nanowire has been described in detail elsewhere [28].

Various Ag nanowire films were deposited to study the sheet resistance behavior with transmittance. The density was controlled by the amount of nanowire material sprayed on the substrate. Figure 3(a) shows the plot of sheet resistance, R_{sh}, versus transmittance, T, at 550 nm wavelength, for films with various nanowire densities. It was observed that the sheet resistance value for $T \sim 85\%$ was 33 Ω/sq, which is comparable to ITO, and far superior to other candidates for TCE. As expected, samples with higher transparency typically showed higher sheet resistance. The sheet resistance of film on glass can be further decreased by annealing the film at 200 °C, resulting in fusion of nanowires, as shown in our earlier work [28]. In general, equation (1) relates the transmittance and sheet resistance for thin metallic films, where $\sigma_{Op}(\lambda)$ is the optical conductivity and σ_{DC} is the DC conductivity of the film [39].

$$T(\lambda) = \left(1 + \frac{188.5}{R_{sh}} \frac{\sigma_{Op}(\lambda)}{\sigma_{DC}}\right)^{-2}. \quad (1)$$

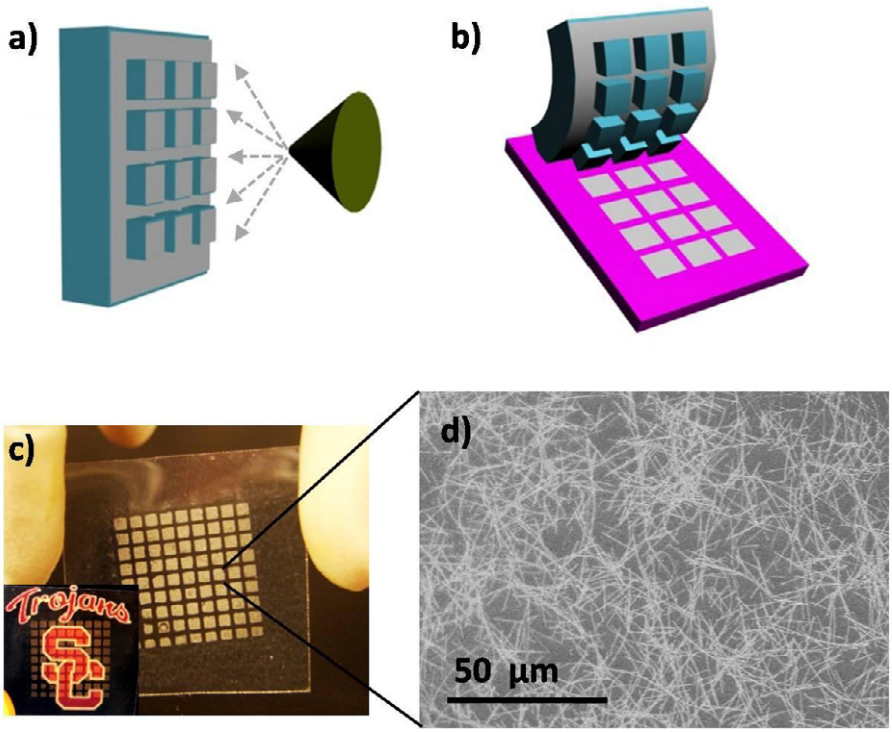

Figure 2. Patterned transfer of silver nanowire film. (a) Ag nanowire solution was sprayed uniformly on the patterned PDMS stamp. (b) Schematic diagram of the contact transfer of Ag nanowire from the patterned PDMS stamp to the substrate. (c) Photograph of a patterned nanowire film transferred on the PET substrate. The size of each pixel is 1 mm × 1 mm with a spacing of 0.5 mm. Inset shows that the patterned nanowire film is highly transparent, as the background image can be clearly seen. (d) SEM image showing the nanowire network from one pixel.

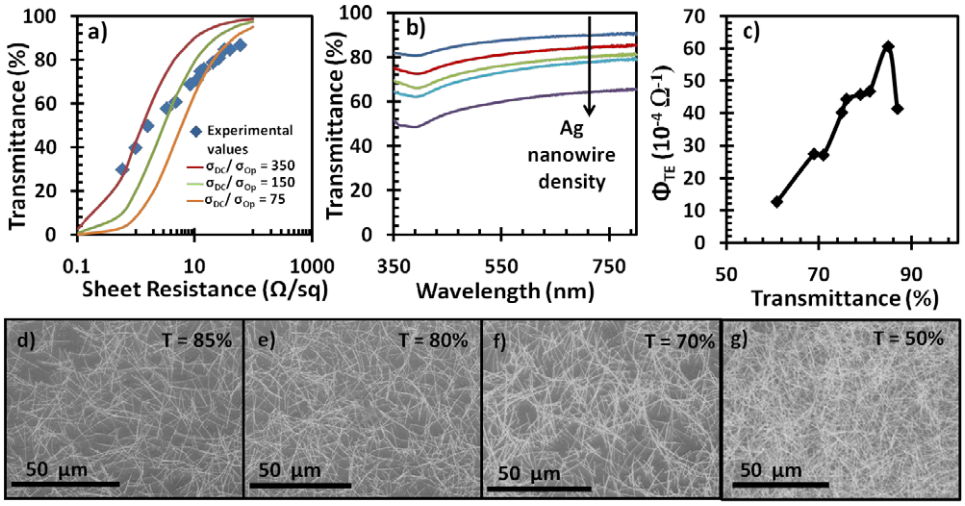

Figure 3. (a) Plot of Ag nanowire film transmittance ($\lambda = 550$ nm) versus sheet resistance data for films deposited on PET. The corresponding fits to equation (1) by using σ_{DC}/σ_{Op} 350, 150 and 75 are plotted as solid lines. (b) Transmittance spectra for various Ag nanowire films. (c) Plot of Φ_{TE} versus λ for Ag TCE with different nanowire densities. SEM images of nanowire films prepared using various amounts of nanowire material with corresponding transmittance values of (d) 85%, (e) 80%, (f) 70% and (g) 50%.

This expression has been fitted to the data in figure 3 (a) using three different values of $\sigma(\mathrm{DC})/\sigma(\mathrm{Op}) = 350$, 150 and 75 (solid lines). This ratio has previously been used as a figure-of-merit for thin conducting films by other workers [14, 27]. The maximum value of σ_{DC}/σ_{Op} for CNT films reported so far is 25, which is far inferior to that of Ag nanowire film [14]. We speculate that this is because of the low junction resistance in the case of Ag nanowire as compared to the extremely high nanotube–nanotube junction resistance of ∼50 kΩ [40]. Recently, a value of $\sigma_{DC}/\sigma_{Op} = 500$ has

been reported, supporting the superiority of Ag nanowire films over other candidates [27]. This ratio is dependent on the uniformity of the Ag nanowire film, which in turn depends on the ability to spray uniformly over the entire substrate. As the spraying technique used here to demonstrate the application of silver nanowires to make transparent conductive electrodes is not fully automated, due to manual handling of the spray gun, the uniformity in deposition increases with density of nanowires deposited. Therefore, the performance of these films improves at lower transmittance. We believe much higher values of $\sigma(\mathrm{DC})/\sigma(\mathrm{Op})$ can be obtained, even at high transmittance, by application of fully automated spraying techniques, as used in industrial applications. The values of the transmittance used for the plot correspond to $\lambda = 550$ nm and the full transmittance spectra are shown figure 3(b) for films with different silver nanowire densities. Again, Ag nanowire films of different densities were fabricated, using different dispersion volumes and Ag nanowire concentration. Figures 3(d)–(g) shows the SEM image of Ag nanowire films on PET with different densities, with transmittance values of 85%, 80%, 70% and 50% respectively. It is evident from the image that the nanowires form a percolated network that becomes denser with increasing dispersion volume, thus demonstrating the application of this method to obtain Ag nanowire films of various transmittance that can be used for different applications.

Another commonly used figure-of-merit for transparent conducting electrodes is Φ_{TE}, defined by Haacke [41],

$$\Phi_{\mathrm{TE}} = \frac{T^{10}}{R_{\mathrm{sh}}} \tag{2}$$

where T is the optical transmittance and R_{sh} is the electrical sheet resistance. To compare our silver nanowire TCE, we plotted Φ_{TE} for silver TCEs with the different nanowire densities shown in figure 3(c). The optimum value of Φ_{TE} from the plot was 0.0059 Ω^{-1} for $T = 85\%$. This value is much higher than recently reported optimum values by others using thin metal films as TCE [42, 43], thus demonstrating the superiority of silver nanowires over other candidates as TCE for optoelectronic devices.

Another important requirement of TCE nowadays is its ability to be flexible [44]; recently, many groups have shown much interest in flexible devices, reporting flexible OLED [45], flexible photovoltaic devices [46], flexible supercapacitors [47] and flexible logic inverters [48], among other applications. Silver nanowire film can fulfil this requirement as a flexible transparent conductive electrode, replacing ITO, for which the brittleness has restricted its application in flexible electronics. We carried out the measurement of its sheet resistance under different bending angles in order to test the durability of nanowire films under stressed conditions. The bending test was performed by placing the film between two platforms, the distance between which was reduced to cause the bending. Sheet resistance measurements were performed at various bending angles by placing four probes on the films with silver paint at the corners. The bending behaviors of two films of higher and lower transmittance values are shown in figure 4. The inset shows the setup for the measurement of change

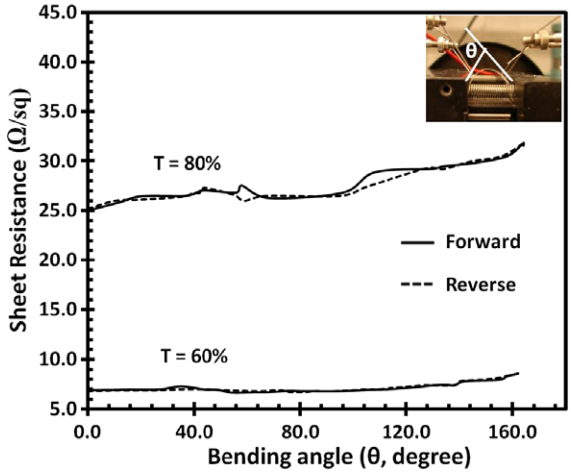

Figure 4. The plot of sheet resistance versus bending angle of two Ag nanowire films deposited on PET with transmittance values of 60% and 80%. The solid lines represent the change in sheet resistance during bending and the dotted lines represent the change in sheet resistance during unbending. The inset shows the photograph of the measurement setup and the definition of bending angle [28]. The Ag nanowire film remains conductive even under severe bending.

in sheet resistance with bending angle, where the bending angle is the angle between the tangents drawn from the bent substrate, shown in the inset [28]. As evident from the plot, the sheet resistance for a film with transmittance of 60% changes only from ~7 Ω/sq to ~8.5 Ω/sq and that for a film with transmittance of 80% only from 25Ω/sq to 33.2Ω/sq when subjected to a bending angle of up to 160°, as compared to a third order magnitude change in the sheet resistance of ITO on PET after bending it to only 60° [49]. The integrity and conductivity of the nanowire film remains intact when the stress is released, as no visible defects were observed. We estimate that the average normal strain value [50] varies from 0.007 to 0.012 for bending angles from 10° to 160° using an equivalent thickness of 100 nm for a film with a transmittance of 80% [27]. No failure was observed up to a bending angle of 160°. SEM inspection revealed that there was no noticeable change in nanowire morphology or the network (images not shown). The potential of silver nanowire film as TCE becomes even stronger due to the fact that this added advantage of flexibility does not come at the cost of its performance in terms of sheet resistance and transparency.

Furthermore, flexible TCE are crucial for touch panels as well as other optoelectronic devices. A simple touch screen includes an electrode separated from another electrode by an insulating spacer. To demonstrate the application of silver nanowire films as an electrode in touch screen devices, we fabricated a prototype touch screen device using a silver nanowire electrode on PET. Figure 5(a) shows the design of the prototype of the touch screen fabricated using silver nanowire TCE. Briefly, silver nanowires were sprayed over the entire 3 cm × 3 cm PET substrate used as the base electrode for the touch panel and the top electrode was made by spraying silver nanowires on a PDMS stamp with two stripes of 3 cm × 0.5 cm and stamping on the PET substrate, as shown in figure 2. A parafilm spacer was placed between the two TCE to avoid any

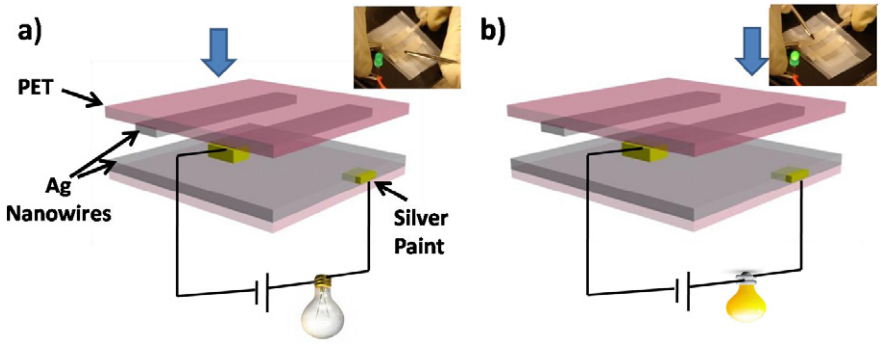

Figure 5. Prototype demonstration of a touch screen using silver nanowire TCE. Schematic of a touch screen fabricated (a) without and (b) with complete connection. Insets show the actual working devices.

contact between the layers. A drop of silver paint was placed at one of the edges of the bottom TCE and at the end of the two stripes of the top TCE to improve the contact. Electrical connections were made between the top stripe and bottom electrode, as shown in figure 5(a), with the actual device shown in the inset. Wire clips, which were connections to the LED and batteries, were connected to the base electrode and the edge of one of the top stripes.

To demonstrate the touch application, the top stripe with no electrical connection to the LED was pressed against the base electrode, resulting in no illumination of the LED, as schematically shown in figure 5(a). The LED was illuminated when the stripe with the wire connection was pressed against the base electrode, thus completing the connection, as shown in figure 5(b). The inset shows the LED lit up on the completion of the circuit. This was repeated 20 times to test the reliability of the setup. No visual change in the morphology of TCE was observed during the test. Thus, silver nanowire TCE has the potential to replace currently used ITO as electrodes for future touch screen panels.

4. Conclusion

In summary, we demonstrated the application of a spray coating technique to obtain high quality and scalable silver nanowire films on arbitrary substrates. The combination of spray coating and a patterned PDMS stamp gives the ability to obtain patterned nanowire films with high fidelity over a large area. Films thus obtained have an extremely low sheet resistance of 33 Ω/sq at a transparency of 85%, which is comparable to ITO films, showing a high value of σ_{DC}/σ_{Op} in the range 75–350 and $\Phi_{TE} = 0.0059\ \Omega^{-1}$. A silver nanowire film retains its good conductivity even under bending and recovers to the original low resistance once the film is released. A demonstration of a touch screen prototype using silver nanowire TCE has been given.

Acknowledgments

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