HIGH OPTOELECTRONIC PERFORMANCE OF LAYER-BY-LAYER ASSEMBLED CARBON NANOTUBE THIN FILMS

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1 Introduction

Transparent electrodes have been studied because of increasing demand for optoelectronics, such as solar cells and electronic displays. In many cases, this electrode must transmit visible light because it is applied directly onto the surface of a transparent substrate. Therefore, it is desirable for electrodes to exhibit high transparency and electrical conductivity. Various materials have achieved a combination of conductivity and transparency to satisfy the needs of these optoelectronic applications.

Because of increasing demand for flexible and robust optoelectronic devices, alternatives to metal oxides for transparent electrodes are desired. Metal oxides such as indium tin oxide (ITO) have been widely used for more than a half century as the most prominent transparent electrodes due to low electrical resistance and high visible light transmittance [1-4]. However, despite having low sheet resistance and high transparency, these films have brittleness that easily leads cracks and poor adhesion by the thermal mismatch with polymer substrates [3-8]. The difference in thermal expansion coefficient and elastic properties of ITO films and polymer substrates results in large mechanical stresses. Flexing an ITO coated polyethylene terephthalate (PET) film causes stress cracks that reduce its electrical conductivity. Several studies have examined the effect of deflection on the resistivity of ITO on a PET or polycarbonate substrate, focusing on the relationship between cracking and electrical properties [3-5]. These studies show that cracks are initiated at a strain of 1.28%, when films are stretched, and at a curvature of 10 cm, when films are bent. These limitations have led to extensive exploration of alternative transparent electrodes (i.e., ITO replacement).

Another option is intrinsically conductive polymer, such as poly(3,4-ethylenedioxythiophene) (PEDOT), but it exhibits low transparency and unstable performance because of photo-oxidative degradation [9,10].

The promising alternative to brittleness and low transparency is carbon nanotube (CNT) thin films. CNTs have been intensively studied because of their impressive electrical, thermal, mechanical, structural and chemical properties. Recently, functional CNT thin films have been investigated as electronic devices including transparent conductive thin films [11-13].

A technique known as layer-by-layer (LbL) is examined here as a means of tailoring the optical transmittance and electrical conductivity of CNT-based thin films. LbL has been widely used to make thin films with homogeneous and controlled manner. It is widely accepted that the multilayer buildup depends on the electrostatic attraction between oppositely charged molecules and the entropy gain from small counterions entering the water [14]. As shown in Fig. 1 (reproduced from [15]), this method produces thin films by harnessing electrostatic interactions through alternately exposing a substrate to positively and negatively-charged aqueous solutions at room temperature. The surface charge on the new surface is reversed because of overcompensation of the original surface charge after the adsorption of oppositely charged molecules. Repetitive adsorption cycles with pairs of other charged ingredients leads to growth of LbL films. The mechanism of LbL assembly is not limited to electrostatic attractions, but including hydrogen...
bonding, weak electrostatic interactions with the aid of strong π–π interaction, various biochemical interactions, charge transfer interactions, and sol-gel reactions used to generate thin films [16-23].

![Diagram of LbL process](image)

**Fig. 1.** Schematic of the layer-by-layer process that involves alternately substrate to cationic PDDA and DOC-stabilized anionic CNT mixtures, along with rinsing with deionized water and drying with filtered air. These steps generate one bilayer (BL) and are repeated to deposit thicker films (reproduced from [15]).

One major advantage of LbL technique is the ability to incorporate several different materials into precise levels within sub-micron films. In addition to conventional polyelectrolytes, various functional materials have been assembled by electrostatic LbL assembly, including DNA and proteins, metal oxides, clay nanosheets, and nanotubes [24-28]. In addition to the versatile spectrum of LbL assemblies, operating parameters add to the versatility of film characteristics for each assembly. Parameters affecting LbL film growth include surface charge of the substrate, concentration of the solution, pH of the solution, deposition time, rinsing, drying, stacking sequence, humidity, temperature, etc. Additionally, the influences of stacking sequence, humidity, and operating temperature on LbL assemblies have been widely investigated. All of these parameters result in a combined effect, so precise control of parameters should be chosen for the optimal films.

This study focused on improving the transparency and conductivity by alternately depositing CNTs as a thin film with a transparent polycation and a negatively charged stabilizer to well disperse individual CNT in the water solution. The ultimate goal is to develop high conductivity and transparent thin films capable of replacing ITO. In an effort to achieve true ITO transparency and sheet resistance, double-walled carbon nanotubes (DWNTs) stabilized with deoxycholate (DOC) were assembled with poly(diallyldimethyl ammonium chloride) (PDDA). Replacing SWNTs from previous studies with DWNTs provides lower sheet resistance due to greater metallic nature [29,30]. The optoelectronic behavior of these nanotube-based thin films was characterized before and after acid treatment. DOC is known to be a good surfactant for dispersion of individual nanotubes [31,32], with its negatively charged tail causing nanotubes to behave as negatively-charged particles, which facilitates LbL assembly [15,28,33]. Exposing nanotube films to strong acid has been shown to enhance conductivity [15,33].

Although the mechanism of acid treatment for nanotube-based assemblies is still uncertain, the increase in electrical conductivity is believed to be a combination of acid anion doping [34,35] and removal of insulating materials (i.e., stabilizer and polymer) [36]. A 5 BL DWNT assembly exhibits a significant reduction in sheet resistance, from 309 to 112 Ω/sq, after two minutes of exposure to HNO₃ vapor. This film is highly flexible, transparent, and electrochemically stable, making it a potential alternative to ITO. After 100 bending cycles, these films exhibit the same sheet resistance, but ITO-coated PET increases two orders of magnitude. In addition to mechanical stability, cyclic voltammetry was used to demonstrate the electrochemical stability of these transparent electrodes. This combination of optoelectronic performance, mechanical flexibility, and electrochemical stability...
make this a suitable candidate for a variety of applications requiring a transparent electrode. Also, the ability to tailor thin film resistance can be potentially useful for anti-static films and EMI shielding.

2 Experimental

2.1 Materials

Purified electric arc SWNTs (0.5–3 μm length and 1.4 nm diameter, C ≥ 90 wt%), synthesized using Ni/Y catalysts, from Carbon Solutions, were used as a base of comparison for HiPCO DWNTs (+1 μm length and ~3.0 nm diameter, C ≥ 90 wt%), purchased from Continental Carbon Nanotechnologies used in this study. PDDA, with a molecular weight of 100,000–200,000 g/mol, and DOC (C\textsubscript{23}H\textsubscript{32}NaO\textsubscript{4}, ≥98%) were purchased from Sigma-Aldrich. Fuming sulfuric acid (oleum, H\textsubscript{2}SO\textsubscript{4}·(SO\textsubscript{3}), 20% free SO\textsubscript{3} basis), fuming nitric acid (HNO\textsubscript{3}, 99.5%), and fuming hydrochloric acid (HCl, 37%) were also purchased from Sigma-Aldrich and used for acid treatment. All chemicals used as received. PET (0.175 mm thickness) films were used as flexible substrates for CNT assemblies. A 100 Ω/sq ITO-coated PET sheet was purchased from Sigma-Aldrich and cut to size to measure the optical transmittance and the change in sheet resistance during bending cycles.

2.2 Layer-by-Layer Assembly

A cationic 0.25 wt% PDDA solution was prepared with 18.2 MΩ deionized water. 0.05 wt% CNTs were dissolved in deionized water containing 1 wt% DOC, followed by 10 W tip sonication for 20 min. Each cycle of LbL assembly consists of substrate immersion into an aqueous mixture, with rinsing and drying after each deposition, beginning with the cationic solution. Cycles were repeated to deposit the desired number of BLs. All films were stored in a dry box for a minimum of 12 hours prior to testing or acid treatment. A schematic of this LbL deposition process is shown in Fig. 1. For acid treatments, assembled CNT films were held in a saturated acid vapor environment. Three types of concentrated acids (fuming sulfuric, nitric, and hydrochloric) in petri dishes were maintained at 70 °C with a water bath. After a 2 to 30 min exposure to acid vapor, the CNT films were rinsed with deionized water and dried with filtered air.

2.3 Thin Film Characterization

Thickness of assemblies was determined by averaging the values of at least five different positions using a PHE-101 Ellipsometer equipped with a 632.8 nm laser. Absorbance and transmittance were measured between 300 and 850 nm with a USB2000 UV-vis spectrometer. All absorbance values in the text are for one-sided coating (i.e., absorbance from slides coated on both sides was halved). Sheet resistance of nanotube assemblies on PET was measured with a Signatone Pro4 four-point probe system with 0.4 mm probe tip diameter and 1.0 mm tip spacing. An Agilent E3644A DC power supply, a Keithley digital multimeter, and LabVIEW using a SCB-68 shield I/O connector were used for voltage and current data collection. The Fourier-transform infrared (FT-IR) spectra were obtained with an ALPHA FT-IR. Scanning electron microscopy (SEM) was performed with a Quanta 600 FE-SEM at an operating voltage of 10 kV. Thin film cross-sections were imaged with a JEOL 1200 EX TEM with an operating voltage of 100 kV. For TEM specimens, the thin films on PS substrates were embedded in an epoxy resin comprised of Araldite 502 (modified bisphenol A) and Quetol 651 (ethylene glycol diglycidyl ether), along with dodecenyl succinic anhydride (DDSA) hardener and benzyldimethylamine (BDMA) accelerator (2:1:1 mole ratio of Araldite 502;Quetol 651:DDSA and 0.2 ml BDMA per 10 g of total weight), which were purchased from Electron Microscopy Sciences. The specimens were sectioned down to ~90 nm and placed on 300 mesh nickel grids to dry prior to imaging. An Epsilon 851 electrochemical workstation was used for cyclic voltammetry. A CNT- or ITO-coated PET electrode, platinum wire, and an Ag/AgCl (3M KCl) electrode were used as the working, counter, and reference electrodes, respectively. These measurements were performed at ambient temperature (22±2 °C) in 0.1 M Na\textsubscript{2}SO\textsubscript{4} solution that had been bubbled with N\textsubscript{2} gas for more than 20 min prior to measurement. Cyclic voltammetry was performed between two potential limits, −0.2 and 0.8 V, at a scan rate of 100 mV/s.
3 Results and Discussion

3.1 Growth and Microstructure of Assemblies

Fig. 2 shows the linear growth of DWNT LbL assemblies up to 10 BLs, denoted as [(PDDA/(DWNT+DOC))]$_n$, where $n$ is the number of bilayers deposited. The average thickness of one DWNT-containing BL is 4.4 nm, which is much thicker than the individual thickness of DWNT (1.5–3 nm), suggesting that the DOC stabilizer and PDDA fully enveloped the DWNT network deposited. In agreement with film thickness, optical absorbance confirms that these CNT-based LbL assemblies grow linearly up to 10 BL. A [(PDDA/(DWNT+DOC))]$_5$ film on PET is highly transparent, with 84.2% transmittance at 550 nm, which is comparable to 84.6%T of ITO-coated PET, as shown in Fig. 3. These complementary measurements (ellipsometry and UV-vis) further suggest a constant concentration of DWNTs and polymer in every bilayer.

![Figure 2: Thickness of PDDA/(DWNT+DOC) thin films as a function of the number of bilayers deposited. The solid lines are linear curve fits. Transmittance was calculated from absorbance data. White points are values after 20 min of exposure to nitric acid vapor (reproduced from [15]).](image)

![Figure 3: (A) Photograph of a 100 Ω/sq single side ITO-coated PET and a 5 BL DWNT assembly on both sides of PET. (B) Optical image of [(PDDA/(DWNT+DOC))]$_n$ ($n = 1–5$) assemblies on both sides of PET substrates. (C) Transmittance spectra of an ITO-coated PET and [(PDDA/(DWNT+DOC))]$_n$ ($n = 1–5$) on PET. Absorbance of the LbL thin films, coated on both sides of PET was divided by two to produce the data shown here. The dotted lines are transmittance of [(PDDA/(DWNT+DOC))]$_5$ after sulfuric, nitric, or hydrochloric acid treatment (reproduced from [15]).](image)

Sheet resistance of [(PDDA/(DWNT+DOC))]$_n$ and [(PDDA/(SWNT+DOC))]$_n$ assemblies on PET was measured as a function of visible light transmittance, which decreased with increasing the number of BLs deposited, as shown in Fig. 4. Sheet resistance significantly decreases with increasing the number of BLs, along with a gradual decrease in %T. [(PDDA/(DWNT+DOC))]$_{10}$ thin films achieve sheet resistance as low as 127 Ω/sq (with 67.2% transmittance at 550 nm) at 10 bilayers, but this system is 1180 Ω/sq (with 93.5% transparency) at just two BLs. The PDDA/(SWNT+DOC) system
exhibits the same optoelectronic trend, with higher sheet resistance than, and similar transmittance to, the DWNT system. This reduction in sheet resistance is due to increased thickness and connectivity of the nanotube network. A 4 BL PDDA/(DWNT+DOC) film has the best optoelectronic performance of 413 Ω/sq and 87.3% transmittance, which meets the key criteria for touch screens (500 Ω/sq, 85% transmittance).

**Fig. 4.** Optoelectronic performance, sheet resistance as a function of optical transmittance of the [PDDA/(SWNT+DOC)]ₙ, [PDDA/(DWNT+DOC)]ₙ systems (reproduced from [15]).

SEM surface images of [PDDA/(DWNT+DOC)]₆ thin films, before and after nitric acid treatment, are shown in **Fig. 5**. What appears to be a bundled DWNT network is more clearly seen following acid treatment (**Fig. 5(B)**). DOC exfoliation of individual DWNTs in deionized water ultimately results in a uniform distribution on the substrate. During deposition, the nanotubes bundle somewhat due to their high concentration in the deposited film, which creates a strong network and high electrical conductivity. Before treatment (**Fig. 5(A)**), the surface of the film was covered by an insulating layer (polymer and/or surfactant) that made the DWNT network difficult to observe. After nitric acid treatment for 20 min, an extensive DWNT network was exposed due to removal of PDDA and DOC (**Fig. 5(B)**). This removal of insulating material enables more direct contact between these highly conductive nanotubes. It is also believed that HNO₃ enhances conductivity through the formation of a charge-transfer complex between DWNTs and NO₃⁻ layers around the individual nanotubes, which promote bundling into thicker nanotube ropes and improved alignment of these ropes. TEM cross-sectional micrographs (**Fig. 5(C) and 5(D)**) illustrate a highly inter-diffused nanostructure, in which the dark and light grey areas are DWNT-rich and PDDA-rich regions, respectively, and the darkest black dots are catalyst impurities from the HIPCO process. In addition to highlighting thin film structure, the TEM images verify the inner diameter of an individual DWNT (~3 nm) and the negligible change in the [PDDA/(DWNT+DOC)]₁₀ film thickness due to nitric acid treatment.

**Fig. 5.** SEM surface images of [PDDA/(DWNT+DOC)]₆ on PET (A) before and (B) after 20 min exposure to nitric acid vapor. TEM cross-sections of [PDDA/(DWNT+DOC)]₁₀ (C) before and (D) after 20 min treatment with nitric acid (reproduced from [15]).

### 3.2 Influence of Acid Treatment

Even when SWNT is replaced by DWNT, the sheet resistance of as-assembled nanotube films is higher than the requirement for replacing the best ITO. It is believed that excessive polymer and surfactant disrupt connections between CNTs. Strong acidic anions form a charge-transfer complex around individual CNTs. Additionally, strong acid removes insulating material from these PDDA/(CNT+DOC)
films. In the present study, films were exposed to acid vapor by heating the acid solution in a 70 °C water bath. Fig. 6 shows sheet resistance of DWNT assemblies after exposure to nitric, hydrochloric, or sulfuric acid vapor for increasing time periods. Nitric acid (b–e in Fig. 6) dramatically reduced the sheet resistance of as-assembled 5 BL PDDA/ (DWNT+DOC) thin films, from 309 to 107 Ω/sq, after a 10 min exposure. Acid type plays a significant role in reducing sheet resistance. Nitric acid treatment provides the lowest resistance, while hydrochloric and sulfuric acids result in values that are nearly double that of HNO₃.

![Fig. 6. Sheet resistance of [PDDA/(DWNT+DOC)]₅ after 2 to 20 min exposure to nitric acid, hydrochloric acid, and sulfuric acid, respectively. (a) is the sheet resistance of an as-assembled 5 BL DWNT thin film (reproduced from [15]).](image)

To better understand the effect of each acid treatment, doped assemblies were characterized by SEM. The DWNT networks after each acid treatment were also characterized by SEM (see Fig. 7). 20 min nitric acid treatment exposes an extensive DWNT network, while more hazy DWNT networks are observed after 20 min hydrochloric and sulfuric acid treatments due to less removal of insulating molecules. SEM images suggest that the removal of insulating material is the main factor in resistance reduction of DWNT-based assemblies, rather than a true acid doping effect. Nitric acid seems to be the most effective at degrading PDDA and DOC, despite having a lower vapor pressure than HCl. The low solution concentration of hydrochloric acid (37%) may account for this discrepancy.

![Fig. 7. SEM surface images of [PDDA/(DWNT+DOC)]₅ on PET before and after acid treatment (reproduced from [15]).](image)

Fig. 8(A) shows the effect of nitric acid treatment on [PDDA/(SWNT+DOC)]₃, [PDDA/(DWNT+DOC)]₅, and [PDDA/(DWNT+DOC)]₁₀ as a function of exposure time (from 2 to 20 min). In just two minutes, all three films exhibit a remarkable decrease in sheet resistance. Further acid treatment, however, results in only a modest decrease in sheet resistance, relative to the first 2 min, especially beyond five minutes of exposure. 20 min nitric acid treatment did not achieve significantly lower sheet resistance than a 10 min treatment. It is likely that any insulating material present is degraded very quickly due to the very thin nature of these films (<50 nm).

To better understand the compositional changes due to nitric acid treatment, FT-IR analysis of [PDDA/(DWNT+DOC)]₂₅ films was performed, as shown in Fig. 8(B). The 3386 cm⁻¹ peak is attributed to a strong O–H stretching vibration in DOC. As expected, this peak disappeared from the spectrum after nitric acid treatment, along with a decrease in the intensity of the peaks at 2928 and 2864 cm⁻¹, both attributed to CH stretching from PDDA and DOC. Additionally, the C=O in DOC exhibits a sharp band at 1715 cm⁻¹ before acid treatment, but this became a broad band centered at 1695 cm⁻¹.
afterward, which indicates that this vibration band changed due to PDDA and DOC removal. FT-IR spectra after nitric acid treatment, from 2 to 20 min, did not show any significant differences, indicating removal of insulating materials due to nitric acid vapor was largely completed in the first two minutes.

Fig. 8. (A) Sheet resistance of three films, \([\text{PDDA}/(\text{SWNT}+\text{DOC})]_5, [\text{PDDA}/(\text{DWNT}+\text{DOC})]_5,\) and \([\text{PDDA}/(\text{DWNT}+\text{DOC})]_{10}\) as a function of nitric acid treatment time. (B) FT-IR spectra of \([\text{PDDA}/(\text{DWNT}+\text{DOC})]_5\) before and after nitric acid treatment, from 2 to 20 min. These spectra are magnified and overlaid with arbitrary offset for clarity (reproduced from [15]).

Furthermore, the SEM surface images in Fig. 9 support this assertion by showing that most insulating materials were removed after 2 min exposure to nitric acid vapor (and a 5 min treatment exhibited the same DWNT network as a 20 min treatment). Additionally, acid treating beyond 10 min produced no additional improvement for DWNT films and an increased sheet resistance for SWNT films. It is believed that some modest damage of CNTs occurs with excessive acid treatment (e.g., sidewall oxidation), causing the observed increase in sheet resistance.

Fig. 9. SEM surface images of \([\text{PDDA}/(\text{DWNT}+\text{DOC})]_5\) on PET (A) before and (B) after 2 min, (C) 5 min, and (D) 20 min HNO₃ exposure (reproduced from [15]).

\([\text{PDDA}/(\text{SWNT}+\text{DOC})]_5, [\text{PDDA}/(\text{DWNT}+\text{DOC})]_5,\) and \([\text{PDDA}/(\text{DWNT}+\text{DOC})]_{10}\) achieved 227, 107, and 43 Ω/sq sheet resistance, respectively, after 10 min exposure to HNO₃ vapor. These values are low enough to use for many optoelectronic applications. Visible light transmittance of these films is 86.8%, 84.0%, and 67.1%, respectively, as shown in Fig. 10. The influence of acid treatment on the optoelectronic performance of several other CNT-based LbL films is also included in Fig. 10 (reproduced from [15]), which emphasizes the relationship between sheet resistance and transmittance [33,37]. Sheet resistance of SWNT films, due to nitric acid treatment, is consistently reduced by a factor of five in this study, regardless of number of bilayers, while that of DWNT films was reduced by a factor of three. DWNT films with more than five bilayers exhibit sheet resistance below 100 Ω/sq. This value is comparable with the best SWNT-based films produced using any other
technique and is competitive with high transparency (~85% T) ITO coatings [36]. The transmittance of the 5 BL DWNT film (>84%) is unaltered by the acid treatment. This optoelectronic performance demonstrates the ability of LbL assemblies to simultaneously achieve high transparency and low sheet resistance. Taking the inverse of the product of sheet resistance and thickness shows that the electrical conductivity of 5 BL and 10 BL DWNT films reach 4100 and 5300 S/cm, respectively, after nitric acid treatment for 10 min. This conductivity is higher than most CNT films made by any method, except for vacuum filtration whose films have relative low transparency (~70%) that eliminates their ability to be used as ITO replacement [38].

Cyclic voltammograms of an HNO₃-treated 5 BL DWNT film and an ITO film (both on PET) were used to compare electrochemical stability, as shown in Fig. 12(A). The potential was cycled between −0.2 and 0.8 V vs. an Ag/AgCl reference electrode, with a scan rate of 100 mV/s, in 0.1 M Na₂SO₄ aqueous solution. ITO shows oxidation-reduction (redox) peaks at 0.1 and 0.3 V, respectively, indicating that ITO undergoes chemical changes under potentiodynamic stresses. A relatively large peak-to-peak separation of 0.2 V (between 0.1 and 0.3 V) is due to this transformation (irreversible change) that occurs in the ITO film during electrochemical cycling. The DWNT LbL films exhibit no redox peaks, suggesting they are electrochemically stable in this potential range. Cyclic voltammograms of nitric acid-treated DWNT films were also collected as a function of the number of BLs deposited, as shown in Fig. 12(B). These rectangular shaped curves are indicative of the capacitive behavior found in carbon materials [39].
These voltammograms show little change before and after acid treatment, which suggests there is no chemical damage of the nanotubes. Integrated charge density from adsorbed and desorbed ions on the DWNT film was calculated by integrating these CV curves with potential differentials. Fig. 12(C) shows the linear increase in charge density as a function of the film thickness. This thickness-dependent electrochemical behavior demonstrates the tailorability of electrochemical behavior in these LbL assemblies by varying the number of BLs.

![Fig. 12. (A) Cyclic voltammograms of a [PDDA/(DWNT+DOC)]₅ thin film on PET and a commercial 100 Ω/sq ITO-coated PET. (B) Cyclic voltammograms of [PDDA/(DWNT+DOC)]ₙ assemblies deposited on PET. (C) Integrated charge density of each film, as a function of film thickness, was calculated from cyclic voltammograms and electrode area.](image)

4 Conclusion

Highly transparent thin film electrodes were assembled through the alternate exposure of flexible PET substrates to aqueous mixtures of positively-charged poly(diallyldimethyl ammonium chloride) and CNTs, stabilized with negatively-charged deoxycholate. Double-walled carbon nanotubes were substituted for SWNTs in an effort to achieve lower sheet resistance in these flexible transparent electrodes. The layer-by-layer film growth is linear for these nanotube-based thin films. DWNT thin films exhibit a significant increase in electrical conductivity, and simultaneous decrease in sheet resistance, after exposure to nitric acid vapor due to removal of insulating material (polymer and surfactant). Additionally, these DWNT LbL coatings on PET substrates have excellent flexibility without any loss of conductivity after 100 bending cycles, unlike commonly used ITO. The sheet resistance, transparency, mechanical flexibility, and electrochemical stability of these CNT-based LbL assemblies meet the criteria for ITO replacement for most electronics applications (%T >85 and Rₛ <500 Ω/sq). Achieving these exceptional properties with fewer than five bilayers is also important in terms of being commercially viable.

Notes

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