Modification of carbon nanotube transparent conducting films for electrodes in organic light-emitting diodes

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Abstract
Single-walled carbon nanotube (SWCNT) transparent conducting films (TCFs) were fabricated for the electrodes of organic light-emitting diodes (OLEDs); three types of film were studied. The as-prepared SWCNT TCFs displayed a relatively low sheet resistance of 82.6 \( \Omega/\text{sq} \) at 80.7 T% with a relatively large surface roughness of 30 nm. The TCFs were top-coated with poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS) to obtain PEDOT:PSS-coated TCFs. The PEDOT:PSS cover improved the conductivity and decreased the surface roughness to 12 nm at the cost of film transmittance. The SWCNT TCFs mixed with PEDOT:PSS-coated TCFs exhibited a high conductivity (70.6 \( \Omega/\text{sq} \) at 81 T%) and a low surface roughness (3 nm) and were thus selected as the best TCFs for OLEDs. Blue flexible OLEDs with 4,4’-bis(2,2’-diphenylvinyl)-1,1’-biphenyl (Dpvbi) as the emitting layer were fabricated on TCFs with the same structures to evaluate the performances of the different types of SWCNT films for use in OLEDs. Of these three types of OLEDs, the PM-TCF devices exhibited the optimal performance with a maximum luminance of 2587 cd m\(^{-2}\) and a current efficiency of 5.44 cd A\(^{-1}\).

This result was explored using field-emission scanning electron microscopy and atomic force microscopy to further study the mechanisms that are involved in applying SWCNT TCFs to OLEDs.

(Some figures may appear in colour only in the online journal)

1. Introduction

An organic light-emitting diode (OLED), particularly, a flexible OLED (FOLED) is necessary for flexible displays because of its excellent properties, such as its light weight, low power consumption with high light, wide angle of view, fast response, low voltage and excellent mechanical flexibility. The material that is traditionally used for OLED anodes is indium tin oxide (ITO), which has a high transmittance and a low sheet resistance [1]. However, this material cracks easily due to its brittleness and poor adhesion to flexible substrates. Additionally, indium is a rare metal with ever-increasing price and a limited supply [2, 3]. Importantly, ITO, whose work
function is ~4.8 eV, has a barrier height greater than 0.3 eV for most hole transporting materials [4]. For transparent conducting films (TCFs), there is significant interest in replacing ITO films with carbon nanotubes (CNTs) due to the excellent electrical and mechanical properties, exceptional flexibility and proper work function (4.5–5.2 eV) of single-walled CNTs (SWCNTs) [5]. CNT electrodes can be used for flexible electroluminescent devices [6] and CNTs can also be introduced as a hole injection buffer layer on ITO to achieve a high-luminance OLED [7]. Many problems must be overcome before SWCNT TCFs are used in OLED devices. One of the most significant problems is the higher sheet resistance compared to that of ITO films, which will lead to a higher turn-on voltage and to poor device performance [8]. Because of the larger aspect ratio, protruding tubes may cause a tremendous local electric field shorting of the device. Additionally, the roughness of CNT films will affect the carrier mobility and shorten the lifetime of the device [9, 10]. Thus, a lot of research based on poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS) has been conducted to lessen the roughness, increase the conductivity and adjust the work function of CNT films, included inkjetting and spin-coating PEDOT:PSS on top of CNT films [11, 12]. Additionally, CNTs can be dispersed in PEDOT:PSS solutions to obtain highly conductive polymer–CNT hybrid composite films [13]. Due to the hydrophobicity of CNTs, the film conductivity is limited by the CNT concentration and the degree of dispersion in the solution [14].

Although there have been many studies on preparing the mixture of carbon nanotubes and PEDOT:PSS as anode layer in OLEDs, it is necessary to conduct a systematic study of the fabrication of high-quality SWCNT TCFs and of their application in OLED devices. In this work, TCFs were obtained by dispersing SWCNTs in a sodium dodecyl benzene sulfonate (SDBS) aqueous solution and spraying the solution onto polyethylene glycol terephthalate (PET) (pristine-TCFs). Then, the pristine-TCFs were post-treated with nitric acid and thionyl chloride (SOCl₂-TCFs). A diluted PEDOT:PSS solution was spray-coated onto the SOCl₂-TCFs to form PEDOT:PSS-coated TCFs (PC-TCFs). An acid oxidized SWCNT mixture with a fixed proportion of PEDOT:PSS was sprayed onto PET to obtain PM-TCFs. The parameters of mixing nanotubes and PEDOT:PSS are optimized for better result according to our own materials and experiment condition. The concentration and centrifugal condition of SWCNT–dimethyl sulfoxide (DMSO) solution were optimized in the range of 0.05–0.3 mg ml⁻¹. One more centrifugation was conducted after the blending of SWCNT–DMSO and PEDOT:PSS solutions for better blending and dispersion. The SOCl₂-TCFs displayed a relatively low sheet resistance of 82.6 Ω/sq at 80.7 T% and a relatively large surface roughness of 30 nm, while, for the PC-TCFs, the PEDOT:PSS cover improved the conductivity and decreased the surface roughness to 12 nm at the cost of the film transmittance. The PM-TCFs exhibited an optimal performance with a sheet resistance (70.6 Ω/sq at 81 T%) and a surface roughness of 3 nm. The films show excellent film performance with rather low sheet resistance and low surface roughness compared to previous published works [12, 13]. The three types of SWCNT TCFs were used as anodes to obtain blue OLED devices with the same structures: TCF/N,N-diphenyl-N,N-bis(1-naphthyl)-1,1-biphenyl-4,4-diamine (NPB)/4,4′-bis(2,2′-diphenylvinyl)-1,1′-biphenyl (Dpvyb)/1,3,5-tris(N-phenylbenzimidazol-2-yl)benzene (TPBI)/LiF/Al. The performances of the three types of OLED devices were compared; the PM-TCF devices exhibited the best device performance with a maximum luminance of 2587 cd m⁻² and a current efficiency of 5.44 cd A⁻¹. The significance of our work is that we compared different types of carbon nanotube and PEDOT:PSS films in a systematic way to further explore the phenomenon and mechanism involved in applying flexible carbon nanotube films in OLEDs and to identify the key factors of influence of SWCNT films in use in high-performance OLED devices.

2. Experimental details

2.1. Fabrication of SOCl₂-treated and PEDOT:PSS-coated SWCNT films

Highly purified arc discharge SWCNTs (96 wt% purity) were purchased from Iljin Nanotech Co., Ltd and were dispersed in deionized water at a concentration of 0.3 mg ml⁻¹ using SDBS as a dispersant. The solution was sonicated in a sonic bath at 150 W for 10 h followed by centrifugation at 10000 rpm for 10 min. The 80% supernatant was carefully decanted and fed into an air brush pistol (GUNPIECE GP-1). The PET substrate was sonicated with acetone and alcohol for 5 min each, rinsed with deionized water, dried in a dry oven and irradiated with a 360 nm monochromatic UV light source for 2 min at a distance of 5 cm [15]. The supernatant in the air brush was directly sprayed onto a heated PET substrate (110 °C) at a constant spray velocity to form a series of SWCNT TCFs with different thicknesses [16]. The sprayed TCFs were then rinsed several times in deionized water to remove the SDBS that was attached to the SWCNTs and dried at 80 °C for 10 min to obtain pristine-TCFs. The pristine-TCFs were then immersed in 12 M nitric acid for 1 h, rinsed with deionized water, dried in a dry oven and irradiated with a 360 nm monochromatic UV light source for 2 min at a distance of 5 cm [15]. The supernatant in the air brush was directly sprayed onto a heated PET substrate (110 °C) at a constant spray velocity to form a series of SWCNT TCFs with different thicknesses [16]. The sprayed TCFs were then rinsed several times in deionized water to remove the SDBS that was attached to the SWCNTs and dried at 80 °C for 10 min to obtain pristine-TCFs. The pristine-TCFs were then immersed in 12 M nitric acid for 1 h, rinsed with plenty of water to wash off any residual nitric acid and dried again (acid-TCFs) [17, 18]. Finally, the acid-TCFs were soaked in thionyl chloride for 30–40 min and dried (SOCl₂-TCFs) [19].

A PEDOT:PSS solution (Clevios PH1000) was filtered using a 0.45 μm polyvinyl difluoride (PVDF) syringe filter, diluted with ultrapure water at a volume ratio of 2:3 and sonicated for 10 min. Then, the diluted PEDOT:PSS solution was directly sprayed onto the SOCl₂-TCFs on a heated panel and dried in a vacuum oven at 80 °C for 2 h to obtain double-layer PEDOT:PSS-coated SWCNT films, PC-TCFs. The thickness of the PEDOT:PSS layer was fixed for the same spraying velocity, time and pressure.

2.2. Fabrication of PEDOT:PSS-mixed SWCNT films

SWCNTs were treated with a mixture of nitric acid and sulfuric acid (1:3 v/v) and refluxed at 80 °C for 12 h. Then,
the acid oxidized SWCNTs were washed with plenty of water until the pH reached approximately 7, washed with absolute ethanol and dried in an oven at 60 °C for 24 h to obtain COOH–SWCNTs. The COOH–SWCNTs were dispersed in a dimethyl sulfoxide (DMSO) solution at a concentration of 0.1 mg ml\(^{-1}\), sonicated for 2 h at 150 W and centrifuged at 8000 rpm for 10 min. The supernatant was carefully decanted and mixed with a PEDOT:PSS solution (v/v 9:1) [20]. The blended solution was sonicated for 30 min and centrifuged at 6000 rpm for 10 min. The supernatant was carefully decanted, air-sprayed onto a heated PET substrate and dried for 2 h to obtain PM-TCFs, incorporating SWCNT films with a low sheet resistance (70.6 Ω/sq, 81 T%) and a low surface roughness (3 nm) [21].

2.3. Fabrication of OLEDs using SWCNT TCFs as the electrodes

The three types of SWCNT TCFs were patterned for use as OLED anodes using radio-frequency oxygen-plasma through a shadow. The OLED structures in this study were SOCl\(_2\)-TF (A), PC-TCF (B) or PM-TCF (C)/NPB (60 nm)/Dpvbi (60 nm)/TPBI (45 nm)/LiF (0.8 nm)/Al (120 nm). All of the organic materials were thermally evaporated onto the patterned anodes using a thermal evaporation coating system at a pressure of approximately \(2 \times 10^{-4}\) Pa with an evaporation rate of 0.1–0.3 nm s\(^{-1}\); the corresponding evaporation rate of LiF and Al was 0.3 nm s\(^{-1}\).

2.4. Characterization

A Keithley 2400 and a PR 650 spectra scan spectrometer were used for current density–voltage–luminance (\(J\–V\–B\)) measurements in an air environment without encapsulation [22]. The sheet resistance and film transmittance were measured using a four-point probe method (Keithley 2700 multimeter) and a UV–vis spectrophotometer (TU-1901). The surface morphologies of the SOCl\(_2\)-, PC- and PM-TCFs were observed using atomic force microscopy (AFM) (5500AFM/SPM) and field-emission scanning electron microscopy (FE-SEM) (Hitachi S–4800). Fourier transform infrared (FT-IR) spectroscopy (TENSOR37) was performed to detect the functional groups that were grafted onto the SWCNTs.

3. Results and discussion

Different types of SWCNT TCFs were fabricated for OLED electrodes. For SWCNT and PEDOT:PSS hybrid films, the content and dispersion degree of the SWCNTs play key roles in the film conductivity, as evidenced by the excellent conductivity of the CNT film compared with that of PEDOT:PSS [23–25]. Here, DMSO was used as a solvent rather than water to generate a doping effect in PEDOT:PSS, thus further increasing the conductivity [26]. To better disperse the SWCNTs in the DMSO solution and mix them with PEDOT:PSS, the SWCNTs were treated with a mixture of nitric acid and sulfuric acid and refluxed to obtain COOH–SWCNTs. The amount of COOH that was grafted onto the SWCNT surfaces was characterized using x-ray photoelectron spectroscopy (XPS) survey spectra and FT-IR spectroscopy. Figure 1(a) shows the general XPS survey spectra; the main elements are C and O for both the pristine– and COOH–SWCNTs. However, the O 1s content of the COOH–SWCNTs increased to 20 at.%, compared with that of the pristine-SWCNTs of 5 at.%. Additionally, FT-IR spectroscopy gives direct proof of the grafting of COOH groups onto the SWCNTs for the COOH–SWCNT sample, as shown in figure 1(b). The width and degree of the peak near 3432 cm\(^{-1}\), which is mainly caused by the O–H stretching vibration, are larger than those of the pristine-SWCNT sample. Meanwhile, the peak that appears near 1730 cm\(^{-1}\) corresponds to C=O stretching vibrations that are related to carbonyl and/or carboxyl groups [27, 28].

Figure 2(a) summarizes the film performances using the sheet resistance versus transmittance at 550 nm for different film thicknesses of the pristine–, PC–, SOCl\(_2\)-, and PM-TCFs, which shows that the sheet resistance increases with increasing film transmittance. For the pristine-TCFs that were fabricated using the SWCNT solution, the sheet resistance decreased dramatically after treatment with nitric acid and further decreased after immersion into thionyl chloride; the SOCl\(_2\)-TCFs have a typical resistance value of 83 Ω/sq at 81.6 T%. The SOCl\(_2\)-TCFs were then top-coated with dilute PEDOT:PSS to form a double-layer conductive film, whose typical resistance value is 67.75 Ω/sq at 77.1 T%. The sheet resistance, \(R_s\), can be calculated from
Figure 2. (a) Sheet resistance–transmittance curves at 550 nm for the pristine-, PC-, SOCl₂- and PM-TCFs. (b) Transmittance versus wavelength in the visible region for various TCFs with comparable sheet resistances.

The performance of the PM-TCFs is optimal in that the conductivity is much better than that of PEDOT:PSS films due to the addition of highly conductive SWCNTs. The SWCNTs that were treated with mixed acid would introduce some defects, which will have an effect on the SWCNT conductivity; however, the macromolecules of PEDOT:PSS wrap up the SWCNTs, thus decreasing the contact resistance between the tubes. Meanwhile, DMSO produces a secondary-doping effect that improves the conductivity of the PM-TCFs [30].

FE-SEM images (figure 3) were obtained to reveal the surface morphologies of the SWCNT TCFs. The pristine-TCFs that were only rinsed with deionized water show some residual SDBS particles attached to the surfaces of the SWCNT films or between the tubes (white dots in figure 3(a)), which significantly decrease the film conductivity due to their insulating properties. After the SWCNT film was treated with 12 M nitric acid for 60 min and then immersed in SOCl₂ for 30 min, most of the SDBS residue was removed; few SDBS particles are observed, and the bundle size is smaller than that of the pristine films. The removal of insulating SDBS between the SWCNT networks and the p-type-doping effect, which is due to nitric acid and SOCl₂ with electron acceptors, enhanced the electrical conductivity [19]. The side view of the FE-SEM images, in the inset, show that some nanotubes protrude from the surfaces of the pristine-TCFs, while post-treatment with nitric acid and SOCl₂ flatten the protruded tubes to some extent (figure 3(b)). The surfaces of the films should be further treated for OLED applications because the residual protruding tubes may short the circuit and cause severe damage once an OLED is deposited onto them. The diluted PEDOT:PSS solution that was sprayed onto the SOCl₂-TCFs to fill the voids between the tubes further decreased the roughness of the PC-TCFs (figure 3(c)). The FE-SEM images of the PM-TCFs (figure 3(d)) show smoother surfaces because small bundles or even individual nanotubes are well dispersed in the PEDOT:PSS matrix and are wrapped up by the PEDOT:PSS macromolecules, leading to a flat surface morphology without SWCNT aggregation [27].

Several samples for each different type of SWCNT films were selected for AFM test. Figure 4 shows the representative AFM images and roughness profiles of the SOCl₂-, PC- and PM-TCFs. The average surface roughness of the SOCl₂-TCFs, in which clear individual or bundled SWCNTs can be seen, is 30 nm, while that of the PC-TCFs, in which the SWCNT bundles can be seen under the PEDOT:PSS layer, is dramatically reduced to 12 nm on
average. The PM-TCFs display the best performance, with a mere 3 nm roughness on average, and the SWCNT bundles can barely be seen on the film surface. The planarization of the surface will contribute to a longer OLED device lifetime, a better charge injection, a high efficiency and a reduction in the leakage current.

Figure 5(a) depicts a schematic of the OLED devices, including the patterned anodes and the region where organic materials and a cathode deposit form a 3 mm × 3 mm emitting point. The energy level of the OLED devices is shown in figure 5(b). After modification with PEDOT:PSS, the work function of the SOCl$_2$-TCFs improved and was favorable for the hole injection of an adjacent hole transporting layer [23, 35–38]. The decrease in the barrier height between the SWCNT TCFs and the NPB layer enhance the carrier mobility and, hence, the brightness and efficiency of the OLEDs. Figures 5(c) and (d) are photographs of working OLEDs for the PC-TCF and PM-TCF devices, respectively, under the driving bias. The emitted light of the PC-TCF device is
Figure 5. (a) Structure of the OLED devices using patterned SWCNT TCFs. (b) The energy diagram of the SWCNT OLED devices, and photographs of the emitting (c) PC- and (d) PM-TCF OLED devices.

Figure 6. OLED device performances for the (a) current density versus operating voltage of the SOCl$_2$-, PC- and PM-SWCNT TCFs and (b) light output versus operating voltage, (c) current efficiency versus current density and (d) emission spectra of the PC- and PM-TCFs.

non-uniform with a brighter edge due to the edge effect of a larger roughness, compared with that of the PM-TCF device.

Figure 6 shows the current density–voltage–luminance (J–V–B) behaviors of the OLEDs that were fabricated using the SWCNT TCFs. For the SOCl$_2$-TCFs devices, nearly all of the devices failed to emit; thus, only current density–voltage characteristics were measured, as shown in figure 6(a). According to the results, the PM-TCF devices displayed the
highest current density under the same voltage, suggesting better conductivity and favorable carrier recombination and mobility. The luminance–voltage (L–V) characteristics of the OLEDs are shown in figure 6(b); the device performance of the PM-TCFs is optimal with a low turn-on voltage of 7 V and a high luminance of 2587 cd m\(^{-2}\). The PC-TCF devices, which have a higher turn-on voltage of 8 V, break down at the voltage of 17 V with the edge sparking first, at which there may be protruding tubes that cause a tremendous local electric field shorting of the device at a high working voltage. OLED devices with SWCNT-TCF anodes still have higher turn-on voltage of 7–8 V compared to that of ITO anodes devices of 3–4 V [39]. This response of the SWCNT-TCF device is likely due to the porous nature of the SWCNT network. Possible voids may remain even after the PEDOT is mixed or coated. The SWCNT TCFs still have higher sheet resistance and larger roughness than ITO film. High sheet resistance and large roughness led to poor device performance with a high turn-on voltage, low brightness and a short device lifetime. Another evaluation standard of the performance of OLED devices is the current efficiency, which was maintained within 8–17 V for the PC-TCF devices and 7–19 V for the PM-TCF devices, as shown in figure 6(c). The maximum efficiency for the PM-TCF devices was 5.44 cd A\(^{-1}\), compared with 4.52 cd A\(^{-1}\) for the PC-TCF devices, partially due to surface planarization improving the injection of holes at the anode interface. Figure 6(d) shows the electroluminescence spectra of the PC-TCF and PM-TCF devices, which are characteristic of a DpVbi OLED with an emission peak at approximately 460 nm [40], indicating the good compatibility of the SWCNT films with the OLED devices that were fabricated with ITO films.

4. Conclusion

In this work, SWCNT films were modified with PEDOT:PSS and deposited for use in DpVbi blue OLED devices. This work innovatively explored problems and solutions by applying CNT films to OLED devices and by comparing three types of OLED devices that were fabricated with SOCl\(_2\)-TCFs, PEDOT:PSS-coated and PEDOT:PSS-mixed SWCNT TCFs. We conclude that a lower sheet resistance led to a better performance with a lower turn-on voltage and a larger luminance. The roughness of the SWCNT film anode has an effect on the charge injection and on the device lifetime. The excellent performance of the PM-TCFs with a relatively low sheet resistance and roughness make them a proper anode candidate to replace the ITO films that are used in FOLEDs. To obtain FOLED devices with superior performances, SWCNT films should be further treated to increase the film conductivity and to reduce the surface roughness; additionally, a modification of the work function should be considered for a better combination of an SWCNT anode with organic materials.

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