



Poly(3,4-ethylenedioxythiophene):polystyrene sulfonate-free silver nanowire/single walled carbon nanotube transparent electrodes using graphene oxide

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ABSTRACT

Poly(3,4-ethylenedioxythiophene):polystyrene sulfonate (PEDOT:PSS) is often used in transparent electrodes as a transparent conductive polymer, however encapsulation away from atmospheric water is often required. Here, we fabricate transparent, flexible silver nanowire (AgNW):single walled carbon nanotube (SWCNT) electrodes using graphene oxide (GO), mitigating the use of PEDOT:PSS. These GO/AgNW:SWCNT electrodes were found to be highly conductive with a sheet resistance of $2.2 \Omega/\square$ – $5.9 \Omega/\square$ and a work function of ~ 4.8 eV. The optical transmittance was $75.00 \pm 0.02\%$ with a haze value of $21.8 \pm 0.8\%$. The figure of merit was shown to be comparable to indium tin oxide (ITO), at $297 \Omega^{-1}$. Finally, the electrodes were shown to be flexible with no reduction in conductivity after 1000 bend cycle/cycles. These PEDOT:PSS free electrodes have the potential to replace rigid commercial ITO electrodes in applications that require flexible, highly conductive transparent electrodes.

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1. Introduction

Indium tin oxide (ITO) based electrodes are one of the more common types of electrodes used commercially [1]. However, its production is relatively expensive and the electrode material itself is brittle and therefore cannot be used in any application that relies on a high degree of flexibility [2], such as large scale roll-to-roll production. For this to be achieved other materials need to be developed that can withstand prolonged and repetitious mechanical stress, a result of flexing, while still retaining the desired electronic properties. Stapleton et al. [3–5] have recently developed silver nanowire (AgNW) and single walled carbon nanotube (SWCNT) nanocomposite electrodes. These electrodes have been shown to have high performance with an average sheet resistance as low as $5.5 \Omega/\square$, and an average light transmission of $>86\%$ [5].

The advantage AgNW:SWCNT:PEDOT:PSS/epoxy electrodes is that they can be fabricated in a roll-to-roll process using flexible substrates such as polyethylene naphthalate (PEN). A downside of using flexible substrates such as PEN is that during the fabrication uneven and inconsistent electrode surfaces can result which may decrease electrode performance when coupled with a device. Xu et al. [6] overcame this planarization issue by using graphene oxide (GO), a graphene sheet

containing oxygen functional groups such as epoxy, hydroxyl and carboxyl groups [7], to create a PEDOT:PSS/AgNW/GO composite electrode. By using GO as a coating to cover the AgNWs they created a smooth surface on the electrode and improved the wire-wire contact at the AgNW junctions [6].

However, the real major downside of these electrodes is the need of the conductive polymer poly(3,4-ethylenedioxythiophene):polystyrene sulfonate (PEDOT:PSS) [8]. This highly hygroscopic polymer is an integral part in the fabrication of these electrodes, used to fill in the gaps between the AgNW and the SWCNTs and aids the conductivity of the electrodes. This results in better charge transfer between the AgNWs and SWCNTs as well as during the operation of a device i.e., from the active layer to the external circuit [9]. Its intrinsic hygroscopic nature makes electrodes prone to degradation thus restricting the utility and lifetime of devices. As a result, electrodes containing PEDOT:PSS suffer from decreased long term stability and reduced overall conductivity [10,11]. Han et al. [12] investigated AgNW:SWCNT transparent conducting films in the absence of PEDOT:PSS for haze and conductivity. They showed that haze measurements of 1.6% could be achieved with a high ratio of SWCNTs to AgNWs (2:3). However, the film sheet resistances were relatively high at $120 \Omega/\square$ even with this 2:3 ratio. No mechanical testing was carried out on the electrodes.

There is a clear need to remove PEDOT:PSS polymer from these hybrid electrode materials while still maintaining the electronic and mechanical properties. To this end, here GO/AgNW:SWCNT/epoxy/PEN

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electrodes were fabricated in the absence of PEDOT:PSS and characterized using Raman spectroscopy, atomic force microscopy (AFM) and scanning electron microscopy (SEM). Bulk properties were systematically investigated using a 4-point probe in the linear regime for sheet resistance, UV photoelectron yield spectroscopy (PYS) for the work function, UV–Visible–NIR spectrophotometer with an integrating sphere for haze measurements, and mechanical strength through bending tests.

2. Experimental

AgNWs were purchased from Seashell Technologies (San Diego, USA), which were supplied as a suspension (20.4 mg/mL) in isopropyl alcohol (IPA). This solution was diluted to 0.1 mg/mL with IPA (99.5% AR grade). Carboxylate functionalized (P3 type) SWCNTs with carbonaceous purity of >90% were purchased from Carbon Solutions (California, USA). The carboxylate functionalized SWCNTs (50 mg) were purified by treatment with 3 M HNO₃ at reflux for 12 h, followed by collection via vacuum filtration. It has been shown that mild acid treatment of SWCNTs improves dispersability in water and also the performance of interwoven AgNW and SWCNT films [13]. A suspension of the acid refluxed SWCNTs (12.5 mg) in deionized water (20 mL) was achieved using probe sonication (Sonics Vibracell™) at 40% amplitude for 2 min. The suspension was then diluted to a concentration of 0.25 mg/mL with deionized water.

A modified method of the laminar assisted stamp transfer process, developed by Stapleton et al. [5], was used to fabricate the electrodes. AgNWs (572 μ L, 0.1 mg/mL in 2-propanol) and SWCNTs (58.3 μ L, 0.25 mg/mL in DI water) were mixed in approximately 300 mL of DI water. A network of AgNW:SWCNT was then cast using vacuum filtration through a mixed cellulose ester membrane (MF-Millipore Membrane, USA, hydrophilic, 0.45 μ m). By using a secondary cellulose ester membrane with a smaller pore size (MF-Millipore Membrane, USA, hydrophilic, 0.025 μ m) underneath the first 0.45 μ m membrane allowed for a specific pattern to be cast for the electrode (Fig. 1A). Following the filtration, the filtrated AgNW:SWCNT film was then placed on top of polyethylene phthalate (PEN) (Teonex films, Teijin DuPont Films) (between 4 and 9 cm²) (Fig. 1B). GO in ethanol (5 mg/mL) (ACS Materials, US) was drop cast onto PEN (between 4 and 9 cm²). Once there was significant coverage of the solution on the surface, the sample

was spin coated using a spin coater (WS-400B-6NPP/LITE, Laurell Technologies Corporation, USA) at 2000 rpm for 10 s and 3500 rpm for 30 s followed by heating at 140 °C for 3 h. The mixed cellulose ester filter membrane containing the AgNW:SWCNT film and the GO coated PEN sheet were then passed through a 130 °C laminator (Fig. 1C). After lamination the mixed cellulose ester filter membrane was removed and approximately 20 μ L of epoxy (Epotek 301) was drop cast onto the centre of the GO/AgNW:SWCNTs/PEN electrode, followed by another PEN sheet that had been surface treated for adhesion (Teonex films, Teijin DuPont Films) (Fig. 1D). The electrode was then heated at 60 °C for 1 h to cure, resulting in a PEN/GO/AgNW:SWCNT:epoxy/PEN stack (Fig. 1E). After curing, the GO coated PEN sheet was peeled off leaving the planar GO-coated GO/AgNW:SWCNT/epoxy/PEN electrode (Fig. 1F). Fig. 1, bottom shows a schematic of the proposed GO layers and its exfoliation.

Sheet resistance measurements were performed using the 4-point probe technique in a linear configuration (KeithLink Technology Co., Ltd., New Taipei City, Taiwan). The values reported were an average of 10 measurements on two separate 625 mm² samples. The correction factor used was 4.3882.

The mechanical integrity of a single planarized electrode (625 mm²) was measured using the 4-point probe in order to investigate the change in sheet resistance after a certain number bend cycle/cycles. The electrode was bent over a 5 mm radius metallic tube 1000 times while measuring the sheet resistance intermittently throughout the bend test. In an individual bend experiment, 1 bend cycle/cycles was flexing the electrode over the tube and releasing back to the starting point.

Scanning electron microscopy (SEM) images were acquired using an Inspect F50 (FEL, Oregon, USA) with an accelerating voltage of 10 kV and 2 kV, with a working distance of 10 mm. The sample was used without metal sputter coating and was placed on conductive tape with one corner of the tape flipped onto the edge of the sample surface. This was carried out so that the conductive top layers of the electrodes were in contact with the conductive tape in order to prevent charge build up due to the PEN separating the top layers with the sample stage.

AFM topographical images were obtained using a Bruker Multimode 8 AFM with a Nanoscope V Controller in tapping mode in air. Silicon cantilevers (Mikromasch model HQ:NSC15 Mikromasch) were used with a nominal resonance frequency of 325 kHz, spring constant of

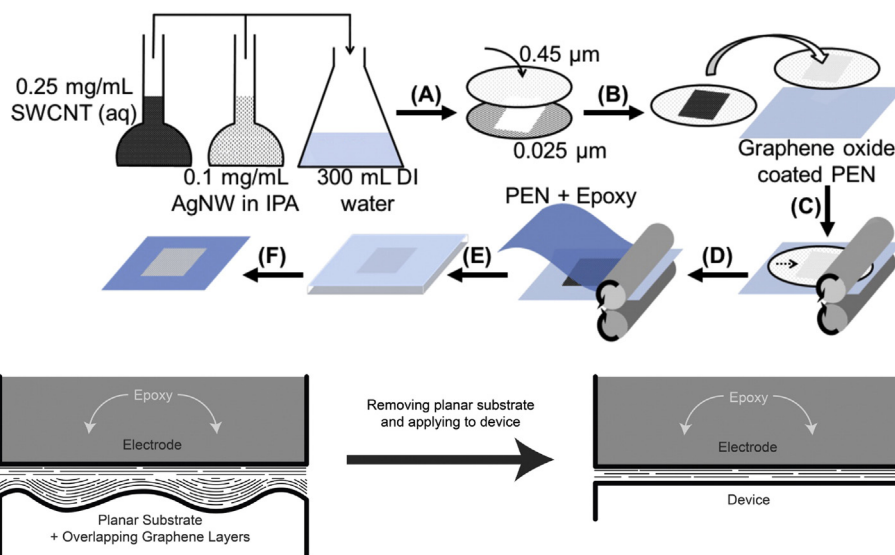


Fig. 1. Top - schematic of GO/AgNW:SWCNT/epoxy/PEN electrode fabrication. (A) Filtration and patterning of the AgNWs and SWCNTs onto a mixed cellulose ester membrane, (B) transfer to a GO-coated PEN planar template, (C) stamp transfer of the electrode to the GO-coated planar template, (D) removal of the mixed cellulose ester membrane, and addition of the PEN and epoxy substrate, (E) curing of the epoxy at 60 °C, and (F) peeling away the GO-coated planar template. Bottom - idealized depiction of removal of GO-coated planar template for device application.

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