Facile preparation of flexible and highly stable graphene oxide-silver nanowire hybrid transparent conductive electrode

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Abstract
The flexible and highly stable graphene oxide (GO)/silver nanowires (AgNWs) hybrid transparent conductive electrode (TCE) was fabricated by coating AgNWs and GO inks on the surface of polyethylene terephthalate (PET) using a Meyer rod. The as-prepared GO/AgNWs hybrid TCE with a GO concentration of 0.75 mg·ml⁻¹ exhibits excellent optoelectronic performances with a sheet resistance of 25 Ω·sq⁻¹, a transmittance of 87.6% at 550 nm, and a lower surface roughness with a root mean square (RMS) roughness value of 4.86 nm. The existence of protective GO layer endows excellent thermal oxidation resistance and outperforming mechanical stabilities for GO/AgNWs hybrid TCE even at the conditions of temperature 80 °C, relative humidity (RH) 75% for 16 days, at room temperature in ambient air for 3 months, and mechanical bending of 2200 times, respectively. The GO/AgNWs hybrid TCE is a promising candidate for ITO used in optical devices such as organic light-emitting diodes (OLEDs), solar cells and flat panel displays.

1. Introduction

With the development of science and technology, smart devices such as mobile phones, tablet computers and smart watches are constantly evolving toward convenience and flexibility. Therefore, in recent years, flexible transparent electrodes have been rapidly developed [1, 2]. The organic light-emitting diodes have been attracting extensive attention owing to the outperforming advantages for flat-panel displays such as self-luminance, wide viewing angle, prominent mechanical flexibility, low power consumption and high reaction speed [3, 4]. Indium tin oxide (ITO), an n-type semiconductor formed by incorporating Sn into In₂O₃, is typically used for transparent electrode in OLED devices, which presents high visible light transmittance and high electrical conductivity [5]. However, indium is rare element in the earth’s crust and ITO is brittle and prone to cracking [6, 7], which greatly limits the applications for flexible electrodes in the development of flexible electronic devices. Due to such drawbacks of ITO, recently, the second generation of flexible transparent conductive electrode materials, such as carbon nanotubes [8–10], graphene [11–13], metal nanowires [14–16], metal conductive oxides [17, 18] and conductive polymers [19, 20] were investigated.

Among these TCEs, silver nanowires (AgNWs) is a promising candidate as a TCE to replace expensive ITO TCE [21, 22]. AgNWs are one-dimensional silver nanostructures with a diameter of about 10–200 nm and a length of about 5–100 μm, and are widely used for transparent films with excellent electrical conductivity, high light transmittance, good bending resistance, etc [23–25]. However, there are also limitations with the application of AgNWs for transparent electrodes. Firstly, the conductivity of AgNWs-based electrodes is generally poor due to the high contact resistance between AgNWs. Secondly, the roughness of the AgNWs-based transparent conductive film is relatively high, which easily leads to short circuit of the organic electroluminescent devices. In addition, nanoscale AgNWs is easily oxidized in the ambient air, these irreparable oxidative accumulations inevitably lead to the failure of devices [26, 27]. Recently, graphene-based materials are
paid much attention as a candidate for transparent conductive films with high electrical conductivity, high carrier mobility and relatively high optical transmittance in the visible range of the spectrum [21, 22].

Some researchers have reported the combination of AgNWs and graphene-based materials such as graphene, reduced graphene oxide (rGO) and graphene oxide (GO) to lessen the roughness of the surface of the film and to make the graphene-based material act as a protective layer to prevent the oxidation of silver nanowires and the failure caused by external forces. Lee et al [28] fabricated a AgNWs-graphene hybrid TCE by dry-transferring a CVD-grown monolayer graphene on a pristine AgNWs TCE, which exhibits excellent optoelectronic properties and superior long-term stability due to the gas-barrier property of the graphene layer. Ahn et al [29] developed a solution-processable AgNWs–rGO film on glass substrate, which shows high optical transmittance, low sheet resistance and highly enhanced thermal oxidation and chemical stabilities. Meenakshi et al [30] developed a AgNWs/rGO TCE with remarkable optoelectrical properties and enhanced stabilities by dip coating method, and rGO plays a dual role of minimizing the quasi-3D stacking and forming the NW–NW contact tighter and serves as a protective top coat to prevent the AgNWs from oxidation. Although graphene and rGO highly enhance the stabilities of AgNWs TCE, the expensive CVD process and the reduction difficulty of GO largely restrict the application of large-size inexpensive TCEs for industrialized production. Graphene oxide is a new type of carbon material with a high specific surface area and rich surface functional groups. Due to the ionization of carboxyl groups, GO can form a colloidal suspension with an electrostatically stable state in water, ethanol, and specific organic solvents [31, 32], which is suitable for solution-based film preparation. Liang et al [33] reported an all-solution processing method to fabricate a highly conductive AgNWs/GO hybrid network by choosing GO as a soldering material for AgNWs junctions. This method of immersing the AgNWs coating in GO solutions strengthens the connection between the silver nanowires and improves the mechanical strength of the film, but it cannot effectively reduce the surface roughness of the film. Wu et al [27] demonstrated a rapid self-assembled route for the preparation of ultrathin GO film, which was easily transferred onto the surface of AgNWs/PET to form a GO/AgNWs/PET electrode. The GO film fabricated by self-assembly method are ultrathin (the thickness ~1.2 nm), however, this method includes transferring undoubtedly complicates the process, which is difficult to control. Therefore, it is necessary to develop a simple, economical, large-scale solution-processable method for the fabrication of AgNWs/GO TCE, which can simultaneously reduce the surface roughness of the film and improves its stability.

In this work, AgNWs with a diameter of about 30 nm were synthesized by the polyol method, and a certain amount of hydroxypropyl methyl cellulose (HPMC) as adhesive and fluorocarbon surfactant (Zonyl FSO-100) as surface modifier were added to the uniformly dispersed silver nanowire solution to form AgNWs conductive ink. Then AgNWs conductive ink was coated on a PET substrate to prepare a large-sized transparent conductive film by a Meyer rod with a coating rate of 30 mm · s⁻¹, and GO ink was coated on the surface of the silver nanowire film as a protective layer to form a GO/AgNWs hybrid TCE. It has been found that the GO/AgNWs hybrid film shows excellent optoelectronic performances, lower surface roughness, outstanding long-term stability in the air, and prominent mechanical stability. Such a solution-processed, large-sized, flexible transparent conductive film with excellent optoelectronic properties and long-term stabilities is expected to be used as a transparent conductive electrode in various optical devices.

2. Experimental

2.1. Synthesis of AgNWs

Silver nanowires were prepared by polyol method according to the previously reported works [34–36]. AgNWs were synthesized by reducing AgNO₃ (99.8%, Sinopharm Chemical Reagent Co., Ltd) with ethylene glycol (EG, 98.0%, Aladdin Industrial Corporation) and FeCl₃ (98.0%, Aladdin Industrial Corporation). Firstly, 450 ml EG was preheated in an oil bath at 160 °C for 1 h to remove moisture, and 3.0 g PVP was subsequently added to the preheated EG with magnetic stirring speed of 300 rpm for 1 h. After the reagent solutions were prepared, 50 ml of FeCl₃ (600 μM, EG) and 60 ml of AgNO₃ solution (3.6 g, EG) were added rapidly into PVP solution within 5 min. After the formation of AgNWs, it was cooled and centrifuged with acetone (AR, Sinopharm Chemical Reagent Co., Ltd) for at least 12 h until the suspension settled to the bottom and remained clear. Finally, it was washed several times with ethanol (AR, Sinopharm Chemical Reagent Co., Ltd) to remove excess solvent, PVP and some impurities.

2.2. Preparation of AgNWs and GO composite inks

The water-based conductive AgNWs inks were prepared by using AgNWs solution (5 mg · ml⁻¹), HPMC (2.56 mg · ml⁻¹, Sinopharm Chemical Reagent Co., Ltd), Zonyl FSO-100 (1 mg · ml⁻¹, DuPont) and deionized water. HPMC was used as an adhesive to improve the homogeneously distributing of AgNWs, and Zonyl FSO-
100 was used as a surface modifier to enhance wetting ability. The GO inks with three different concentrations (0.5, 0.75 and 1.0 mg · ml⁻¹) were fabricated by using GO solution which diluting graphene oxide gel (GO gel, 1 wt%, Aladdin Industrial Corporation) to 1 mg · ml⁻¹ with water as solvent., HPMC (2.56 mg · ml⁻¹), Zonyl FSO-100 (1 mg · ml⁻¹) and deionized water, and dispersing the mixed solution evenly by ultrasound treatment for 5 min.

2.3. Fabrication of GO/AgNWs hybrid films
The PET substrates with size of 10 × 15 cm² were first ultrasonicated in ethanol for 15 min, rinsed in deionized water, and then pretreated via UV/ozone (UVO) with a UV irradiation power of 300 W for 10 min to make its hydrophilicity exceedingly improved. Firstly, the prepared AgNWs ink was coated on the surface of the pretreated PET using a Meyer rod to form solution-processable silver nanowire transparent conductive films. The coating rate and temperature were set to 10 mm · s⁻¹ and 50 °C, respectively. Then, after the surface solvent naturally evaporated, the GO film was coated on the as-prepared AgNWs film at a coating rate of 30 mm · s⁻¹ and a temperature of 50 °C to form a protective layer. After the solvent in the GO film was evaporated, the GO/AgNWs hybrid film was heated at 110 °C for 20 min to remove residual water and organic solvents.

2.4. Characterization
The morphology and microstructure of the samples were observed by scanning electron microscope (SEM, SU8010, Hitachi Ltd, Japan) and transmission electron microscope (TEM, JEM-1230, JEOL Ltd, Japan). Powder x-ray diffraction (XRD) patterns were performed by a Shimadzu XRD-6000 diffractometer (Cu Ka radiation; 40 kV, 60 m A). Optical transmittance spectra were carried out by a UV-visible spectrophotometer (DU8000, Beckman). Raman spectra were obtained by Raman microscope (LabRAM HR Evolution, Horiba Jobin Yvon, France) under a backscattering geometry (λ = 532 nm). The surface roughness of the film was measured by scanning probe microscope (SPM, MultiMode, VEECO, America). The sheet resistance and haze of the hybrid film were investigated by four-probe conductivity meter (RTS-9, Guangzhou four-point probe technology, China) and transmittance/haze meter (WGT-S, Bogoo, China) respectively. Static contact angles of different hybrid films were characterized by video contact angle meter (Dataphysics OCA20, Germany). X-ray photoelectron spectrum (XPS, Thermo Scientific K-Alpha+, USA) was recorded to characterize the surface composition.

3. Results and discussion

3.1. Characteristics of AgNWs and GO
Figures 1(a) and (b) show the TEM image and XRD pattern of the as-prepared AgNWs. It is seen that the average diameter of AgNWs is about 30 nm, and there are four peaks at 38.2°, 44.4°, 64.6° and 77.5°, corresponding to (111), (200), (220) and (311) of pure face-centered cubic (fcc) silver crystal (ICPDS Card No. 04–0783), respectively [37]. Figures 1(c) and (d) show the TEM image and ultraviolet-visible absorption spectrum of the GO. It is noted that the as-purchased GO is a large-sized, single-layer two-dimensional GO material, and has a relatively high visible light transmittance. There is a distinct absorption peak at a wavelength of about 230 nm, which corresponds to the π-π* transition of C=O bond, and a weak absorption peak at around 300 nm is attributed to the n-π* transition of C=O bond, which is consistent with the spectrogram of GO reported in the previous work [32].

3.2. Optoelectronic performances of GO/AgNWs hybrid TCEs
The fabrication process of the transparent GO/AgNWs hybrid film is illustrated in figure 2(a). The UV-treated PET was coated with silver nanowire ink to form a conductive network. Then, in order to protect the silver nanowire from oxidation, a layer of GO ink was coated on the surface of pristine AgNWs film as a protective layer. After all the solvents have evaporated, the hybrid films were annealed at 120 °C in the air for 20 min to remove excess solvents from the films. Figure 2(b) displays SEM image of GO/AgNWs film, indicating that silver nanowires with 30 nm in diameters are randomly distributed on the substrate surface. These interlaced AgNWs allow electrons to be transported freely throughout the film, resulting in the entire film good conductivity, and the nanometer diameter of AgNWs produces high transmittance of the entire film, which indicates that such a transparent conductive AgNWs film can be obtained.

In order to verify that GO adhered well to the surface of silver nanowires, Raman spectroscopy was used to characterize the surface of the GO/AgNWs film, which is a non-destructive technique which is widely used to obtain structural information about carbon-based materials [32, 38]. Figure 3(a) shows the 532 nm Raman spectrum of GO/AgNWs hybrid film. The measurement was performed at room temperature with a LabRAM HR Evol spectrometer at 532 nm and a 50 × objective. It is seen from figure 3(a) that there are a D-band around
1341 cm$^{-1}$ and a G-band at 1590 cm$^{-1}$, and the intensity ratio of the D- and G-band (ID/IG) is 0.473, which is consistent with the main features of GO reported in the previous works [39, 40]. It indicates that GO is well compounded on the surface of the silver nanowire film. To investigate the surface wetting state of different films, contact angle tests of PET substrate, GO film, AgNWs film and GO/AgNWs hybrid film were performed respectively, as shown in figure 3(b). Each contact angle is the average of three sample measurements, and the corresponding error bars have been added to the graph. It is observed that the contact angle of PET is the biggest ($\theta = 65^\circ$) and the one of GO the smallest ($\theta = 33^\circ$). Compared with the original AgNWs film, the GO/AgNWs hybrid film has a smaller contact angle. With the hybridization of GO film, the contact angle of GO/AgNWs hybrid film decreases from 54$^\circ$ of pristine AgNWs film to 43$^\circ$, which illustrates that GO film coated on the pristine AgNWs film can improve the hydrophilicity of entire film.

Figure 1. (a) TEM image of AgNWs. (b) XRD patterns of AgNWs. (c) TEM image of GO. (d) UV-Vis absorption spectra of GO suspension.

Figure 2. (a) Schematic diagram of preparation of GO/AgNWs hybrid film. (b) SEM image of GO/AgNWs hybrid film.
Figure 4 shows the sheet resistance, haze and optical transmittance spectra of GO/AgNWs hybrid film with different GO concentrations. The inset image in figure 4(a) indicates that the GO/AgNWs hybrid film coated on the PET substrate is a transparent flexible conductive film. The resistance and haze of film are the average values of 9 sample measurements, and the corresponding error bars have been added in the figure. As shown in the figure 4(a), when the GO concentration is less than 0.75 mg · ml⁻¹, the sheet resistance and haze of the AgNWs film rise slowly with the increased GO concentration. However, when the GO concentration exceeds 0.75 mg · ml⁻¹, the sheet resistance and haze of the film rise remarkably. To further investigate the effect of GO layers on the optical properties of AgNWs film, the transmittance spectra were measured by UV-visible spectrophotometer, as shown in figure 4(b). The visible light transmittance of the AgNWs film gradually decreases with enhanced GO concentration. When the GO concentration reaches 0.75 mg · ml⁻¹, the transmittance at 550 nm of GO/AgNWs films only declines from 88.4% to 87.6%, compared with AgNWs film. The results reveal that GO layer has less influence on the optical properties of the AgNWs film when the concentration of GO solution does not exceed 0.75 mg · ml⁻¹.

To further investigate the surface properties of films, the morphologies and roughness of the AgNWs and GO/AgNWs (C_Go = 0.5 mg · ml⁻¹) films were determined by atomic force microscope (AFM) with a tapping mode, as shown in figures 5(a) and (c). It is observed that the root-mean-squared (RMS) roughness value of AgNWs and GO/AgNWs films are 6.13 ± 1.13 and 4.86 ± 1.01 nm, respectively. Obviously, GO film coated on the pristine AgNWs film reduces the roughness of AgNWs film by 20.7%, indicating that the surface of the film becomes much smoother [41]. This may be because AgNWs film is an overlapped NWs-NWs network, which easily generates a large roughness, and a large-sized few-layer GO can improve the unevenness of the film. 3D AFM images (figures 5(b) and (d)) show that the surface of AgNWs film has sharp undulations, while the GO/AgNWs hybrid film becomes obviously smooth, which further verifies that the GO film decreases the surface roughness of AgNWs film. In addition, it is obviously observed from figure 5 that the height shown in the 3D AFM diagram of the GO/AgNWs film is significantly lower than that of the AgNWs film. In the coating, the part of the GO sheet will be filled into the network formed by the overlapping of the silver nanowires, which will reduce the height difference of the film and make the height in the image significantly lower [42].
3.3. Thermal oxidation and mechanical stabilities of GO/AgNWs hybrid TCEs

In order to explore the effect of the GO layer on the thermal oxidation stabilization of AgNWs film \([27, 29, 42]\), the AgNWs and GO/AgNWs films \((C_{\text{GO}} = 0.5 \text{ mg} \cdot \text{ml}^{-1})\) were exposed to air at 80 °C and 75% RH for 16 days, respectively, as shown in figure 6(a). It is observed that the sheet resistance of the AgNWs and GO/AgNWs films enhances gradually with the increase of exposition time. The sheet resistance of AgNWs film increases from 24.58 to 61 Ω sq\(^{-1}\) after 16 days, while the GO/AgNWs hybrid film only increases from 24.89 to 34 Ω sq\(^{-1}\), indicating that the GO layer can effectively prevent the silver nanowires from being oxidized in the air. As shown in inset two images of figure 6, after being put in a high-temperature (80 °C) and high-humidity (75% RH) environment for 16 days, many nanoscale particles are distributed along the silver nanowires on the AgNWs film, which are caused by the easy oxidation of the silver nanowires in the air. The oxidized particles increase the contact resistance between the AgNWs, resulting in an increase in the sheet resistance of the AgNWs film. By comparison, after being put in the same environment for 16 days, the variation of the sheet resistance of the hybrid GO/AgNWs film is small, and the morphology is almost the same as that of the previous hybrid film, which indicates that GO can effectively isolate silver nanoparticles from ambient air. Figure 6(b) shows the changes in sheet resistance of the AgNWs and GO/AgNWs films exposed to ambient air at room temperature for 3 months. Obviously, the sheet resistance of GO/AgNWs film has no significant change, however, the \(R/R_0\) (ratio of measured value to original value) of AgNWs film increases more than 15 times when it was exposed to the ambient air at room temperature for 3 months. The difference in change of sheet resistance confirms the excellent long-term stability performance of GO/AgNWs films in air, which is attributed to the gas barrier layer of GO prevents AgNWs from oxidizing.

![Figure 5. AFM and 3D AFM images of (a, b) AgNWs film and (c, d) GO/AgNWs hybrid film.](image)

![Figure 6. (a) Sheet resistance change of AgNWs and GO/AgNWs films at 80 °C and 75% RH for 16 days. Inset pictures are SEM images of AgNWs and GO/AgNWs films exposed to air at 80 °C and 75% RH for 16 days respectively. (b) Change in sheet resistance of the AgNWs and GO/AgNWs films exposed to ambient air at room temperature for 3 months.](image)
X-ray photoelectron spectroscopy (XPS) was used to characterize the chemical composition of the films. Figures 7(a) and (b) show the wide scan survey spectra of AgNWs and GO/AgNWs films exposed to air at 80 °C and 75% RH for 16 days. It is noted that there exist C, O, F and Ag elements. In figures 7(a) and (b) of C1s spectrum, four different peaks were observed, which indicates the existence of C–C/C=C, C–O bond, carbonyl group (C=O) and carboxyl group (O=C=O), corresponding to the previous work reported [43–45]. The Ag 3d spectrum (figure 7(e)) of AgNWs film exhibits two strong peaks, 3d5/2 and 3d3/2, located at respectively 373.81 and 367.81 eV, corresponding well to Ag(I) [46], which is attributed to Ag2O. After exposing at 80 °C and 75% RH in the air for 16 days, however, the Ag 3d spectrum of GO/AgNWs film (figure 7(f)) shows the two peaks in 374.24 eV (Ag 3d5/2) and 368.24 eV (Ag 3d3/2) corresponding well to Ag [47], which provides direct evidence that silver nanowires have not been oxidized under the protection of GO layer.

Figure 8 shows the mechanical stability of AgNWs and GO/AgNWs (C100 = 0.5 mg · ml⁻¹) films. It can be seen from the figure that the sheet resistance of the AgNWs film rises sharply after 2200 times of the bending test, and finally becomes 89 times of the original sheet resistance. In contrast, the sheet resistance of the GO/AgNWs hybrid film is relatively stable, after 2200 times under the bending test, the sheet resistance only increases by 4.5 times. The morphologies of AgNWs and GO/AgNWs films after 2200 times of the bending test are shown in the inset images in figure 8. The AgNWs film without GO protective layer is severely damaged, and a plurality of fractures appear on one silver nanowire, matching to a sharp rise of sheet resistance. On the contrary, the silver nanowire is relatively intact in the hybrid film with GO as a protective layer except that the junction between the NWs and NWs is broken, as shown in the red circle. Obviously, the combination of AgNWs and GO can greatly improve the mechanical stability of the AgNWs film, which maintains the original electrical properties under severe destructive experiments.
4. Conclusions

In summary, flexible graphene oxide/silver nanowires (GO/AgNWs) hybrid transparent TCEs were fabricated by coating AgNWs and GO inks respectively on the surface of flexible PET substrate. The GO film can be used as a protective layer for AgNWs TCE, and improves the surface properties, optoelectronic performances and long-term stabilities of AgNWs TCE. The GO/AgNWs hybrid film with a GO concentration of 0.75 mg \cdot ml^{-1} possesses a sheet resistance of 25 \Omega sq^{-1}, a transmittance of 87.6% at 550 nm and a root-mean-squared (RMS) roughness of 4.86 nm, and displays excellent thermal oxidation and mechanical stabilities under the conditions of temperature 80 °C and relative humidity 75% for 16 days, at room temperature in ambient air for 3 months, and mechanical bending of 2200 times, respectively. It is expected that such a large-sized, highly stable GO/AgNWs TCE can replace ITO used in optical devices such as OLEDs, solar cells, and flat panel displays.

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Conflicts of interest

There are no conflicts to declare.

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