High Aspect Ratio and Post-Processing Free Silver Nanowires as Top Electrodes for Inverted-Structured Photodiodes

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ABSTRACT: Silver nanowires (Ag NWs) as transparent conducting electrodes are widely used in many applications such as organic light-emitting diodes (OLEDs), polymer light-emitting diodes, touch screens, solar cells, and transparent heaters. In this work, using a large-scale synthesis, the synthesized Ag NWs had a high aspect ratio of 2820. The Ag NWs could be applied as a top transparent electrode in a device by simple drop-casting without any post-processing steps. The fabricated device comprised 4,4′-bis(carbazol-9-yl)biphenyl/MoO3 organic/inorganic layers which are parts of the inverted structure OLEDs or solar cells. The photodiode characteristics at the UV range were observed in the device. The ability of Ag NWs to replace opaque metals as top electrodes in a device has been demonstrated.

1. INTRODUCTION

Nanoparticles and nanowires (NWs) are highly attractive materials in the field of electronic devices as well as other fields.1−15 In a typical optoelectronic device structure, the bottom electrode is often a transparent conducting electrode, while the top electrode is normally a metal thin film.16−24 The device performance is hence limited as light is not transmitted through the top electrode which is opaque. The device performances can be enhanced by using a transparent metal conducting material as the top electrode. Among transparent metal conducting materials available, silver NWs (Ag NWs) are widely used as transparent conducting bottom electrodes because of their highest electrical conductivity and ease in synthesizing.12,23−34 Ag NWs have been reported to be used in many applications such as organic light-emitting diodes (OLEDs), polymer LEDs, touch screens, solar cells, and transparent heaters.18−26,34 This is because Ag NW electrodes are exceptional in terms of transparency, conductivity, and flexibility and suitable to be applied in optoelectronic devices.23,25,26,28,29 Besides that, the processing of Ag NW electrodes is cost-effective because Ag NWs can be processed in solution.22,27,32,33,35

Several studies have reported the application of Ag NWs as top electrodes but their Ag NWs mostly consist of a polyvinylpyrrolidone (PVP) layer.35−40 Typically, post-processing procedures such as high temperature annealing, chemical annealing, light annealing, and tedious purification processes are required to remove the PVP layer in order to improve the conductivity and connectivity of the Ag NW network.41,42 The Ag NWs without PVP as capping agent are thus attractive as the complicated post-processing can be eliminated. Generally, when Ag NWs are applied as top electrodes, the choice of the post-processing process is often limited to annealing by heat treatment. Hence, the application of Ag NWs without the need of post-processing is desirable especially for the devices that contain heat-sensitive layers, where the post-processing temperature is critical.

The present work aims to explore the applicability of high aspect ratio and post-processing free Ag NWs as top electrodes for device application. We first synthesized Ag NWs with a high aspect ratio of 2820. The synthesized Ag NWs are then

Supporting Information
applied as a top electrode in an inverted structure photodiode by simple drop-casting at room temperature without the need for annealing after deposition. In this work, the organic/inorganic photodiode with 4,4′-bis(carbazol-9-yl)biphenyl (CBP)/MoO₃ layers is fabricated. This is because CBP/MoO₃ is widely used as hole-injection and hole-transport layers in inverted structure LEDs or solar cells. After the fabrication of the CBP/MoO₃ photodiode with Ag NWs as the top electrode, the device performances are analyzed to investigate the potential of Ag NWs as top electrodes for inverted structure optoelectronic devices. The capability of Ag NWs as a transparent conducting top electrode is important for making double-sided, transparent flexible devices.

2. RESULTS AND DISCUSSION

2.1. Crystal Structure and Morphology of Synthesized Ag NWs. Ag NWs were synthesized at various Fe³⁺ concentrations (Figures S1 and S2). The aspect ratios of Ag NWs synthesized with 40, 80, and 120 mM Fe³⁺ are 490, 1156, and 236, respectively, as summarized in Table 1. The optimal concentration of Fe³⁺ for obtaining the highest aspect ratio is found to be 80 mM. To investigate the feasibility of scaling up the synthesis, the synthesis was scaled-up by 10 times (namely, large scale) with using 80 mM Fe³⁺ as the catalyst. The synthesized Ag NWs in large-scaled synthesis has an aspect ratio of 2820. We found that in large-scale synthesis, the AgCl seeds were bigger than those in small-scale synthesis (Figure S3 and Table S1). Hence, the number of seeds becomes smaller in large-scale synthesis, causing Ag NWs to grow longer than in small-scale synthesis.

High aspect-ratio NWs are desirable as transparent conducting electrodes since percolation of the NW network can be achieved with low density of NWs. Since the Ag NWs produced in the large scale synthesis is found to have the highest aspect ratio, and large amount of NWs can be obtained in a single synthesis, these Ag NWs were used to fabricate the top electrode for the photodiode. Figure 1a,b shows the scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images of the synthesized Ag NWs after purification, respectively. The average length of Ag NWs was 110 µm and the average diameter was 40 nm. The dispersion of Ag NWs in isopropanol (IPA) with a concentration of 9.76 mg/mL is shown in Figure 1c. X-ray diffraction (XRD) data in Figure 1d show that the synthesized NWs had peaks in 2θ equal to 38.12° and 44.28°, identical with that in the reference pattern of Ag. The UV-vis spectrum (Figure 1e) of NWs dispersed in IPA shows the peaks at 384 and 353 nm, where the former corresponds to the localized surface plasmon resonance of Ag NWs in transverse mode.

2.2. Conductivity and Transparency of the Ag NW Electrode. The Ag NW electrode was prepared by depositing Ag NWs onto a cleaned glass substrate. The Ag NW electrode has an average sheet resistance of 58 Ω/sq which is suitable to be used as a transparent conducting electrode. Figure 2 shows the transmission spectra of the fabricated Ag NW electrode and a commercial indium tin oxide (ITO) electrode. The transparency of the Ag NW electrode is around 76% at the visible light range, which is lower than that of ITO (around 83% transparency). Even though the transparency of Ag NWs is lower than that of ITO in the visible light region, the application of Ag NWs as the top electrode in our device is feasible. In UV wavelengths (320–380 nm), the Ag NW electrode has approximately 68.2% transparency, which is comparable to ITO (67.8% transparency) that is used as the bottom electrode. The transparency of the Ag NW electrode in the UV region is important because the active layers of our photodiode consist of CBP and MoO₃ layers, which are responsive to UV wavelengths.
we hence applied the Ag NWs into a photodiode as the top electrode. The organic/inorganic photodiode in our study consists of ITO as the bottom electrode, CBP/MoO$_3$ as the active layers and Ag NWs as the top electrode, as shown in Figure 3a. The energy band diagram of the device using Ag NWs as the top electrode is shown in Figure 3b wherein the work functions for ITO, CBP, MoO$_3$, and Ag NWs in the device are obtained based on references.\textsuperscript{17,43}

NWs as the top electrode is shown in Figure 3b wherein the energy value of the MoO$_3$ layer is the measured value for MoO$_3$ in our previous study.\textsuperscript{17} Figure 3c shows the actual device under measurement with Ag NWs as the top electrode under UV irradiation. The Ag NW top electrode has well-contacted and connected NW networks that comprise of several layers of NWs, as shown in Figure 3d.

The measurements to obtain photodiode characteristics were carried out by connecting the crocodile clips of the electrometer Keithley 2425, to Al wires which were connected to the electrodes of the photodiode via a silver paste. A xenon light source with band-pass filters of 254 ± 5, 270 ± 5, 310 ± 5, 340 ± 5, 365 ± 5, 380 ± 5, and 410 ± 5 nm was used. Since Ag NWs provide transparency for the light source to pass through as discussed in the previous section, the measurements were obtained by illuminating the device from the Ag NW side. Figure 4a shows the responsivity of the CBP/MoO$_3$ photodiode using Ag NWs as the top electrode with the flow directions of photogenerated electrons and holes across the device, (c) photo of the real device under UV light illumination with the inset showing the device on a paper under room light, and (d) SEM image of Ag NWs as the top electrode in the device. The work functions for ITO, CBP, MoO$_3$, and Ag NWs in the device are obtained based on references.\textsuperscript{17,43}

The $I$−$V$ characteristics in the UV range using Ag NWs as the top electrode are shown in Figure 4b. The dark current measured is 1 nA at biases of 0.4 and −0.4 V. The dark and the photocurrent $I$−$V$ curves show symmetric and linear behaviors. At a bias of −0.4 V, the photocurrent is −1.5 nA for 1.2 mW incident power, which represents a photocurrent to dark current ratio of 1.5. The symmetry of the $I$−$V$ curves might be due to the very less work-function difference between ITO (∼4.8 eV) and Ag NWs (∼4.6 eV) and the energy levels of the CBP and MoO$_3$ layers. The dark and illuminated $I$−$V$ curves for the region near 0 V demonstrate the presence of a photovoltaic effect under illumination at 340 nm light. The maximum short-circuit current is −1.5 nA for an incident power of 1.2 mW. The open-circuit voltage is 0.4 V. To prove that the effect is not due to hysteresis, voltage was scanned from both directions (forward bias to reverse bias and vice versa) and the corresponding current was measured. The direction of scan has negligible effect on the photovoltaic response. This photovoltaic response, which allows separation of an electron–hole pair at zero applied bias is the indication for a type II heterostructure between the MoO$_3$ and CBP layers, where the built-in field in the device under zero bias is sufficient to promote charge separation.

Figure 4c shows the relationship of photocurrent with the optical power of the light source. The current is negligible under dark conditions. When the optical power increases, the light intensity increases and hence the photocurrent also increases. It is clear that the photocurrent does not increase linearly with incident power. In the low power region, the response is quite linear, suggesting that during recombination, the rates of the absorbed photon and photo-generated electron–hole pairs in reaching the electrodes are constant. Above ∼0.2 mW, the photocurrent increase is slower with increased power (increased intensity). The slower increment of photocurrent in the high intensity region may be due to the saturation of electron traps. To investigate the switching behaviors of Ag NWs as the top electrode with ITO as the bottom electrode of the photodiode, the device was illuminated from both Ag NW and ITO sides. The device shows a good on/off switching behavior as shown in Figure 4d,
upon illumination from both Ag NW and ITO sides. The slight difference in the photocurrents observed for the top (Ag NW side) and bottom (ITO side) may be due to the absorbance of Ag NWs. Based on the UV–vis spectra (Figure S4), the device with ITO/CBP/MoO3/Ag NWs layers shows slightly higher absorbance at 340 nm than the device with ITO/CBP/MoO3 layers, suggesting that some UV light is absorbed by the Ag NW electrode before reaching the CBP/MoO3 layers. However, overall UV light is mainly absorbed by the CBP/MoO3 layers.

3. EXPERIMENTAL SECTION

3.1. Materials. Silver nitrate (AgNO3, Sigma-Aldrich), ethylene glycol (EG, Wako), 1,2-dodecanediol (DD, TC1), sodium chloride (NaCl, Wako), iron(III) nitrate nonahydrate [Fe(NO3)3, Wako], ammonia solution (28% NH4OH, Wako), acetic acid (AcOH, Wako), IPA (Wako), CBP (Lumtec), and molybdenum oxide (MoO3, Sigma-Aldrich) were used as received.

3.2. Synthesis of High-Aspect Ratio Ag NWs. Ag NWs were synthesized based on the modified polyol method, as shown in Figure 5, with the reference to reports by Sim et al.44,45 First, 6.30 mL of EG was added into a flask and heated at 110 °C for 1 h under magnetic stirring. Simultaneously, 80 mM Fe(NO3)3 solution and 30 mM NaCl solution were prepared. Then, AgNO3 solution was prepared in another vial by adding AgNO3 into 1.50 mL of EG. After 1 h of heating, 0.15 mL of Fe(NO3)3 solution and 0.03 mL of NaCl solution were injected into the preheated EG sequentially and heated at 110 °C for another 20 min. The magnetic stirring was then removed from the solution, followed by injection of 1.50 mL of AgNO3 solution. The reaction was maintained at 110 °C for 15 h to allow the growth of NWs. The reacted solution was subsequently quenched down to room temperature and centrifuged at 5000 rpm for 30 min with DD, NH4OH, and AcOH consecutively. The purified Ag NWs were dispersed in IPA for further characterization. The concentration of Fe3+ was varied from 40 to 120 mM to acquire the optimal concentration for obtaining NWs with the highest aspect ratio. For the large scale synthesis of Ag NWs, the concentration of AgNO3, NaCl and Fe(NO3)3 was kept the same as small scale synthesis (X1) but the volumes of the solutions were increased by 10 times. Same procedures were carried out and the detailed parameters are summarized in Table 2.

3.3. Fabrication of the Ag NW Electrode and CBP/ MoO3 Photodiode. Ag NWs were dispersed in IPA to form 1.22 mg/mL dispersion. To fabricate the Ag NW electrode, quartz glass was used as the substrate. Before use, the glass substrate was cleaned with nonionic detergent, distilled water (resistivity = 18.2 MΩ cm−1), acetone, ethanol, and IPA sequentially under sonication. The substrate was then dried and subjected to a UV lamp (Ushio Corp.) under a pressure of 10 Pa air for 30 min. Next, the electrode was prepared by simple drop-casting of Ag NWs onto a glass substrate and leaving it to dry for 15 min naturally. To fabricate the photodiode, the commercial ITO-coated glass substrate with a resistivity of 7−15 Ω sq was used as the bottom electrode. First, the ITO-coated substrate was etched to a narrow strip by using hydrochloric acid and zinc dust. Then, the substrate was cleaned using the same cleaning process as for the glass substrate. A layer of 60 nm thick CBP was then deposited onto the ITO-coated substrate, followed by deposition of an inorganic layer, MoO3 with 125 nm at vacuum level 10−4 Pa by using a thermal evaporation method. Ag NWs as the top electrode were drop-casted onto the MoO3 and dried naturally.

3.4. Characterization. The crystalline and phase structures of Ag NWs were characterized using XRD (Rigaku MiniFlex II X-ray diffractometer, Cu Kα radiation, λ = 1.5418 Å, scanning speed of 2° min−1). The morphologies of the synthesized NWs were examined using SEM (JEOL-JSM-6701F and Hitachi TM3030 Plus, 15 kV) and TEM (JEOL JEM-2000FX, 200 kV). The average length of the NWs was measured based on 50 NWs and the average diameter was measured based on 30 NWs in the SEM and TEM images. The absorbance and transmission of Ag NWs, the electrode, and the photodiode were characterized by using ultraviolet–visible spectroscopy (UV–vis, Shimadzu UV Spectrophotometer UV-1800 with UV Probe software and JASCO V-650). The average sheet resistivity was measured based on 10 NWs and the average diameter was measured based on 30 NWs in the SEM and TEM images. The synthesis of Ag NWs was confirmed using XRD (Rigaku MiniFlex II X-ray diffractometer, Cu Kα radiation, λ = 1.5418 Å, scanning speed of 2° min−1). The morphologies of the synthesized NWs were examined using SEM (JEOL-JSM-6701F and Hitachi TM3030 Plus, 15 kV) and TEM (JEOL JEM-2000FX, 200 kV). The average length of the NWs was measured based on 50 NWs and the average diameter was measured based on 30 NWs in the SEM and TEM images. The absorbance and transmission of Ag NWs, the electrode, and the photodiode were characterized by using ultraviolet–visible spectroscopy (UV–vis, Shimadzu UV Spectrophotometer UV-1800 with UV Probe software and JASCO V-650). The average sheet resistivity was measured based on 10 measurements on the Ag NW electrode by using a four-point probe method (Loresta-GP, MCP-T610, Mitsubishi Chemical Analytech, Japan). A high-power Xenon lamp (300 W, Max-301, Asahi Spectra Co., Ltd.) with band-pass filters of 254 ± 5, 270 ± 5, 310 ± 5, 340 ± 5, 365 ± 5, 380 ± 5, and 410 ± 5 nm was used as the UV light source. The responsivity, photocurrent as function of optical power, current–voltage (I–V) and switching characteristics data of the photodiode were obtained with a Keithley 2425 electrometer connected with a computer.

4. CONCLUSIONS

The synthesis of Ag NWs with a high aspect ratio of 2820 is achievable by using 80 mM Fe3+ as a catalyst and scaling up by 10 times. The sheet resistivity and transparency of the NW network are suitable for using as a transparent conductive top electrode to replace opaque metal electrodes. The Ag NWs can be deposited by simple drop-casting into the photodiode device at room temperature and without any post processing. The ability for the synthesis of Ag NWs to be scaled up, the simplicity of deposition and the elimination of complicated post-processing are beneficial for industrial production where large-scale production and simple procedures are preferable. Our demonstration on using high-aspect ratio Ag NWs as a top

Table 2. Concentration and Amount of Fe(NO3)3, NaCl and AgNO3 Used in Syntheses of Ag NWs

| Concentration (mM) | Volume of Fe(NO3)3 (mL) | Volume of NaCl (mL) | Volume of AgNO3 Used (mg) | Volume of AgNO3 (mL) |
|--------------------|------------------------|---------------------|--------------------------|---------------------|
| 1                  | 40                     | 0.15                | 30                       | 0.03                | 26                  | 1.50                  |
| 1                  | 80                     | 0.15                | 30                       | 0.03                | 26                  | 1.50                  |
| 1                  | 120                    | 0.15                | 30                       | 0.03                | 26                  | 1.50                  |
| 10                 | 80                     | 1.50                | 30                       | 0.30                | 260                 | 15.00                 |
electrode in an inverted ITO/CBP/MoO₃/Ag NW photodiode illustrates the potential of such Ag NWs to be integrated as top electrodes in full inverted structure OLEDs or solar cells in the future.

**ASSOCIATED CONTENT**

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.9b01479.

SEM images of Ag NWs synthesized with different concentrations of Fe³⁺, XRD data of Ag NWs synthesized with different concentrations of Fe³⁺ and scaling-up, XRD data and crystalline size for AgCl seeds in small and big scale syntheses at 80 mM Fe³⁺, UV–vis spectra of the glass substrate, CBP, MoO₃, Ag NWs, ITO/CBP/MoO₃ and ITO/CBP/MoO₃/Ag NWs (PDF)

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Notes

The authors declare no competing financial interest.

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