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Enhancement of electrical and thermal properties of silver nanowire transparent conductive electrode by Ag coating

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ABSTRACT

In this study, Ag nanowires (AgNWs) were synthesized by the polyol method at 160 °C under nitrogen atmosphere. AgNW transparent conductive electrodes (TCE) were fabricated on a glass slide, and an Ag layer was coated onto the AgNWs to improve their electrical and thermal properties. The AgNWs had diameters in the range of 25–54 nm, aspect ratio up to 1000 and high yield (>90%) when used with the mixture of halide ions Cl⁻ and Br⁻. After coating an Ag layer on the surface of AgNW of TCEs by electrodeposition, the electrical and thermal properties was improved significantly. The sheet resistance decreased from ~247 Ω/\Box to approximately 10 Ω/\Box and the thermal stability was improved by 20 °C in comparison with bare AgNW TCEs. The best sample was observed at an electrodepositing current of 1 mA for 40 s; the sheet resistance, transmittance, and figure of merit of this sample were ~8.5 Ω/\Box , 85.4%, and 271 Ω^{-1} , respectively.

1. Introduction

Transparent conductive electrodes (TCEs) have attracted considerable attention for use as components of optoelectronic devices with flexible touch screens, organic optoelectronic devices, flexible heaters, electroluminescent displays, thin-film solar cells, and electronic tattoos [1-6]. In recent decades, high-transmittance and conductive indium tin oxide (ITO) has been widely used as a TCE. However, the use of commercially available ITO is limited because of its high cost and mechanical brittleness [7]. This issue has motivated the finding for low-cost TCEs, such as Al-doped ZnO (AZO) [8], carbon nanotubes [9], graphene [10], and metal nanowires [11,12], to replace sputtered ITO. Among such materials, silver nanowire (AgNW) is the most promising candidate due to its outstanding properties, such as the high intrinsic conductivity of Ag, high transmittance of up to 97.9% in the visible spectrum at 550 nm, and low sheet resistance of 36.3 Ω / \Box [13]; moreover, AgNW TCEs can be can be fabricated under a nonvacuum process and are suitable for flexible substrates. Many studies have attempted to synthesize AgNWs with small diameter and high aspect ratio [13-16].

Although Ag has the highest electrical conductivity among metals, the electrical properties of AgNW networks cannot be compared with those of bulk materials because of the high contact resistance at the junctions of wires [17]. High contact resistance at the junctions will cause high sheet resistance of TCEs and low thermal stability as applying a current on them. For this reason, the finding of solutions to reduce such resistance has received considerable attention from the researchers around the world. Electrochemical cleaning is used to remove Polyvinylpyrrolidone (PVP) and enhance the conductivity of the AgNW network [18]. Physical methods, such as mechanical pressing [19], heat treatment [20], and illumination [21], can be used to reduce the contact resistance between AgNWs. Although such methods can minimize the sheet resistance without significant change in transmittance, they are difficult to control due to the low melting temperature of AgNWs. Electrical properties are also significantly improved when metal oxide coating is applied to the wire surface or TCO nanoparticles are coated on the surface of the AgNW TCEs; However, these methods cause a large loss of the transmittance [22,23]. AgNW networks can be coated with a metal layer, such as Cu, Ni, or Ag, via traditional electrodeposition

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Fig. 1. Diagram of TCE fabrication by doctor-blade printing, then coating a silver layer on AgNWs by electrodeposition.



Fig. 2. SEM images of AgNWs synthesized with different KBr concentrations of (a) 0 mM, (b) 1.2 mM, (c) 2.4 mM and (d) 4.8 mM.

method. The AgNW network coated with Ni showed a reduced sheet resistance from $100 \Omega/\Box$ to $10 \Omega/\Box$ [24]. With Ag coating, some reports have also used electroplating to weld the contact points between nanowires [25-27]. However, the diameter of the nanowires after electroplating is very large or the electroplated silver layer exists in the form of particles on the wires, which will increase the surface roughness of the TCEs and reduce the transmittance; while to reduce the surface resistance simply reduce the contact resistance between the nanowires. The high surface roughness of TCEs will affect in some applications such as bottom electrodes for LED devices or solar cells. In Ag coating by electrodeposition, the pH, electrical current and time are key factors; they will affect to the phase structure and morphology of the electrodeposited layer and sheet resistance uniformity of TCEs.

This work focused on the synthesis of AgNWs with high aspect ratio and enhancement of electrical and thermal properties of AgNW TCEs by coating an Ag layer on the surface of AgNWs. By controlling the electrodeposition current and time, and the pH of electrolyte solution, we obtained a smooth Ag coating layer on AgNWs. In addition, the sheet resistance was decreased from ~250 Ω/\Box to ~10 Ω/\Box ; and the thermal stability has also improved by 20°.

2. Experimental

2.1. Materials

The raw materials used in this work included silver nitrate (AgNO₃, purity of \geq 99%, VWR Chemical), ethylene glycol (EG) (purity of 99.8%, Sigma-Aldrich), PVP (average molecular weight: ~1,300,000; Sigma-Aldrich), potassium bromide (KBr, purity of \geq 99%, Sigma-Aldrich), sodium chloride (NaCl, purity of \geq 99%, Sigma-Aldrich), acetone (\geq 99%), sodium thiosulfate (Na₂S₂O₃, purity of \geq 98%, Sigma-Aldrich), and sodium bisulfite (Na₂S₂O₅, purity of \geq 99%, Sigma-Aldrich).



Fig. 3. Performance of AgNWs synthesized with 2.4 mM KBr by polyol method: (a) SEM image before purification, (b) SEM image after purification, (c) diameter distribution and (d) XRD pattern of AgNWs.

2.2. AgNW synthesis

First, 0.6 g PVP was dispersed in 15 ml EG in a three-neck flask under magnetic bar stirring with a rotation speed of 500 rpm at room temperature for 30 min. This step ensured that PVP was completely dissolved in EG and became a transparent solution. Second, 120 and 240 μL solutions of KBr (0, 1.2, 2.4 and 4.8 mM in EG) and NaCl (0.5 M in EG), respectively, were injected to the solution in the three-neck flask. After 15 min, the mixture was heated up to 160 °C, and a solution of 0.25 g AgNO₃ and 10 ml EG was slowly injected into the flask with a typical injection speed of 30 ml/h. The solution temperature was kept at 160 °C for 2 h to ensure a complete reaction. During injection, the solution gradually changed from a white to a reddish-brown color, corresponding to the formation of AgCl_xBr_{1-x} precipitate mixture. The solution color turned gray after 10 min and to clear gray after 30 min. The whole reaction process was completed under nitrogen atmosphere to avoid oxidation. Then, 30 ml deionized water was injected to cool down the solution quickly. Acetone (100 ml) was slowly poured into the reaction solution until the precipitate appeared. After 10 min, most of the precipitates have settled, and the suspended materials in the solution, which were mainly nanoparticles and short AgNWs, were sucked out by a pipette. The final product was washed by ethanol and acetone several times to remove most of the nanoparticles, PVP, EG, and short AgNWs. Finally, AgNWs were dispersed in 5 mg/ml ethanol.

2.3. Electrodeposition

The electrolyte solution included 0.024 M AgNO₃, 0.2 M Na₂S₂O₃, and 0.015 M Na₂S₂O₅, and pH was adjusted in the range of 5.3 – 6. Three solutions were prepared at room temperature to avoid the appearance of Ag₂S precipitate. First, the AgNO₃ solution was slowly poured into Na₂S₂O₅, and a white precipitate was formed. Second, this precipitate was dissolved in the Na₂S₂O₃ solution under magnetic stirring. The final solution was transparent and light yellow in color. AgNW electrodes coated onto glass slides and heated to 140 °C for 2 min had a high sheet resistance of ~285 Ω/\Box and a transmittance of ~96%. Cu tapes were attached to the pristine AgNW film as the cathode for electrodepositing. The areas of AgNW electrodes and Ag plate anode (one side) were fixed at 2 × 2 cm². The applied current was controlled at 1, 2, and 4 mA with the plating time ranging from 10 s to 50 s. The diagram of TCE fabrication is described in Fig. 1.

2.4. Characterization

AgNW length and diameter were viewed by field-emission scanning electron microscopy (SEM) JSM-7600F, JEOL). Thickness of the electrodeposition layer and lattice planes was confirmed by Transmission Electron Microscope (TEM; JEM2100, Jeol). Phase structure and preferred orientation of AgNWs were characterized by X-ray diffraction



Fig. 4. Low TEM (a), HR-TEM (b) images of the AgNWs synthesized with 2.4 mM KBr. The inset in (b) is SAED pattern.

(XRD, Siemens D-5005) with the radiation of Cu-K_{λ} ($\lambda = 154,056$ Å). The sheet resistance ($R_{\rm SH}$)of the AgNW electrodes were measured by a fourprobe system. Transmittance (*T*) was recorded on an Agilent Cary 5000 UV–Vis-NIR. Infrared thermal images of AgNW electrodes were captured by a thermal camera (Ti300+, Fluke).

3. Results and discussion

Fig. 2 shows the SEM image of AgNWs synthesized at different KBr concentrations. KBr concentration showed a strong influence on the size

and content of AgNWs. The diameters of AgNWs synthesized at KBr concentrations of 0, 1.2, 2.4, and 4.8 mM were in the range of 54–103, 48–72, 25–54, and 48–115 nm, respectively. AgNWs synthesized at a concentration of 2.4 mM were the smallest and the most uniform. In the case of 4.8 mM KBr, the yield of AgNWs was low, and numerous large Ag particles were formed during synthesis. For TCE applications, the diameter of AgNWs should be as small as possible because a large diameter will cause a large loss of the transmission and high roughness of the TCE. Therefore, AgNWs synthesized at a concentration of KBr 2.4 mM were the most suitable for TCE applications (Fig. 2(c)).



Fig. 5. SEM image of AgNW TCEs without (a) and with coating Ag at different electrical currents of (b) 1, (c) 2 and (d) 4 mA for 50 s.

Fig. 3(a) and 3(b) are SEM images of AgNWs synthesized at 2.4 mM KBr before and after purification. After cleaning, the Ag nanoparticles completely disappeared. The length of AgNWs was from several micrometers to \sim 30 µm. The aspect ratio was \sim 1000, and the yield was over 90%. The distribution of AgNWs via diameter is shown in Fig. 3(c). AgNWs have diameters in the range of 24-54 nm. The largest number of AgNWs is observed at the diameter of \sim 40 nm. Fig. 3(d) shows the XRD pattern of AgNWs after cleaning. Diffraction peaks were observed at 38.36° and 44.98°, which denote the (1 1 1) and (2 0 0) crystal orientations of Ag, respectively. The XRD pattern showed no impurities, implying that only AgNWs remained in the final product. The AgNWs synthesized at 2.4 mM KBr were used for fabricating TCEs in the next experiments. Further analyzing the diameter size and crystal structure, TEM measurement was performed and the results are depicted in Fig. 4. As shown in Fig. 4(a), the diameter of AgNWs is quite uniform, and in the range of 27-36 nm; this is like the result of SEM measurement. Fig. 4 (b) is HR-TEM image of an AgNW. The lattice planes are seen quite clearly on the entire wire, which indicates the high crystallinity of the synthesized AgNWs. The distances between two consecutive lattice planes are seen to be 2.42 Å and 2.12 Å, which correspond to the lattice planes (111) and (200). This result is consistent with the XRD result as shown in Fig. 3(c). The inserted figure in Fig. 4(b) is the selected area electron diffraction, shows that the AgNWs synthesized are polycrystalline.

Fig. 5 shows the SEM image of AgNW TCEs deposited an Ag layer on the surface of wires by electrochemical deposition method with different currents of 1, 2 and 4 mA for 50 s. The dimeter of AgNWs was increased with the increase of the current; namely, the average diameters of AgNWs after electrodepositing with 0, 1, 2 and 4 mA for 50 min are 36 nm, 56 nm, 71 nm and 94, respectively. Regarding the intersections of AgNWs, the NWs were wielded together; this can be seen clearly at the samples electrodeposited at 2 and 4 mA; the effect of this phenomenon on the electrical property will be discussed below.

Fig. 6 is low and HR-TEM image of AgNW electrodeposited at 1 mA for 40 s. As shown in Fig. 6(a), the diameter of Ag-coated AgNWs is in the range of 48-53 nm; this result quite matches with SEM image as mentioned above. The surface of the wires is smooth, indicating the high uniformity of Ag electrodeposited layer on the AgNWs. To analyze the performance of Ag-coating layer we carried out a HR-TEM measurement and the result is shown in Fig. 6(b). The thickness of Ag-coating layer is around 10 nm. The lattice planes are seen very clearly, indicating the high crystallinity of the Ag-coating layer. The SAED image shows that the Ag-electrodepositing layer is seemingly monocrystalline. We can see that the PVP layer always exists on the surface of AgNWs both before and after electrodepositing; this means that the Ag-electrodepositing layer was grown from the surface of the AgNWs instead of from the surface of the PVP layer. This can be explained as Ag⁺ ions passed through the PVP layer during electrodepositing to grow on the surface of AgNWs.

Fig. 7 shows the sheet resistance of AgNW TCEs electrodeposited at the currents of 1, 2 and 4 mA for different times. Sheet resistance decreased rapidly with different electrodepositing times. During the first 10 s, the sheet resistances of all TCEs at different currents dropped sharply. Specifically, the sheet resistances of samples electrodeposited at 1, 2, and 4 mA decreased from 251 Ω/\Box to 74.46 Ω/\Box , 250 Ω/\Box to 35.42 Ω/\Box , and 253 Ω/\Box to 7.12 Ω/\Box , respectively. The sheet resistance of TCEs was mainly due to the contact resistance between NWs. Ag itself is highly conductive. Thus, the resistance of each AgNW is small compared with the contact resistance. This phenomenon has been proven by previous studies [17]. Therefore, the decrease in sheet resistance in the first 10 s may be due to the improved contact resistance. Over a period of 10–50 s, the sheet resistance of the TCEs decreased



Fig. 6. Low TEM (a) and HR-TEM (b) images of Ag-coated AgNWs with electrodepositing current of 1 mA and coating time of 40 s. The inset in (b) is the selected area electron diffraction (SAED) on the electrodeposited Ag area.

slowly, and this finding was probably due to the increased size of the AgNWs. Thus, the electrical properties of AgNW TCEs were substantially improved after an Ag layer was coated onto the AgNWs.

Fig. 8 shows the transmittance spectra of TCEs and the values at 550 nm wavelength under different electrodepositing currents and times. In the first 10 s, the transmittance dropped rapidly at all three currents and gradually decreased afterward. The rapid decrease in transmittance in the first 10 s was due to the high Ag^+ concentration near the TCE,

resulting in the fast Ag deposition rate on the TCE. The decrease in transmittance was due to the increase of NW diameter and electrodepositing of a part of Ag onto the glass substrate. The transmittance value at the wavelength of 550 nm of TCE is described in Fig. 8(d). In the case of 1 mA the transmission loss slowly; however, when the current increases to 2 and 4 mA, the transmission decreases quite rapidly.

To evaluate the performance of the TCEs, we calculated figure of merit (FOM) value for the TCEs using the following formula:



Fig. 7. Sheet resistance of AgNW TCEs with different Ag electrodeposition times.

$$\frac{\sigma_{DC}}{\sigma_{OP}} = \frac{188.5}{R_{SH}^* (T^{-\frac{1}{2}} - 1)}$$

and the results are shown in Fig. 9(a). Where R_{SH} is the sheet resistance and T is the transmittance at the light wavelength of 550 nm. The FOM value of the TCEs increased sharply after electrodepositing the silver, in which the samples were electrodeposited at 1 mA was the largest increase. Over a period of 0 to 40 s, the FOM value increases strongly with electrodeposition time, namely from ~43 Ω^{-1} up to ~270 Ω^{-1} . Further increasing time to 50 s, the FOM tends to decrease. The highest FOM value of 271 Ω^{-1} is observed at the current of 1 mA for 40 s. The increase in FOM is due to the significant improvement in the sheet resistance of the TCEs as mentioned in Fig. 7 above. In the range of 40-50 s, the decrease of sheet resistance is insignificant while transmittance lose quickly, and therefore causing a decrease in FOM value. In briefly, the performance of the TCEs were greatly improved after coating silver on the NWs. The achieved FOM value of the Ag-coated AgNWs (271 Ω^{-1}) was higher than that of other electrodes, namely, AgNW/Ag-grid [28], photoresist-passivated AgNWs [29], atomic layer deposited AZO [30], AuCl₃-modified grapheme [31], Cu thin films [32], and AgNW/NOA63 [33], previously published by other research groups but was lower than that of AZO/AgNWs [34]. Thus, Ag-coated AgNWs are promising for TCE applications in photoelectronic devices. The relationship between optical transmittance and sheet resistance for four values of FOM is shown in Fig. 9(b). With a similar FOM value, AgNWs TCEs coating Ag at 1 mA are the most appropriate for the TCE applications.



Fig. 8. Transmittance of AgNW TCEs coating Ag at different electrodeposition currents of (a) 1, (b) 2 and (c) 4 mA for various times of 0, 10, 20, 30, 40, and 50 s, and (d) transmittance value at the wavelength of 550 nm with different electrodeposition times.



Fig. 9. The variation of sheet resistance with electrodepositing time (a) and (b) optical transmittance versus sheet resistance of AgNW TCEs.

Fig. 10 shows the thermal performance of TCEs at different the applied voltages. As shown in Fig. 10(a), When the applied voltage is 2 V, corresponding to the sample temperature ~ 100 °C (see Fig. 10(b)), the distribution of temperature on the sample is quite uniform. However, as increasing the applied voltage up to 4, 6, 8 and 10 V the temperature mainly concentrated at the center of the TCEs. Further increasing the voltage to 12 V, a dark red line appeared along the middle of the sample and the temperature at this location increased sharply compared to the rest (see Fig. 10(b)). After increasing the applied voltage to 12 V for 10 s, the current suddenly decreased to zero (see Fig. 10(c)); this indicates that the TCE was destroyed due the breakage of AgNWs at the center of sample. We also compare the thermal stability TCEs with and without Ag coating with the same sheet resistance. As shown in Fig. 10(c) the maximum temperatures for these TCEs 235 and 215 °C. The higher thermal stability of Ag-coated AgNW TCEs can be explained as follows: when an electrical current is flowing through TCEs, the maximum temperature is generated at the intersection points



Fig. 10. Infrared thermal image (a) and temperature on a crossing line (b) of AgNW TCE with Ag coating at 1 mA for 40 s. (c) the thermal stability of AgNW TCE without and with Ag coating at the different applied voltages.

between two wires because the resistance here is large compared with that of each wire. Thus, these points will be destroyed first. Ag-coated TCEs have low contact resistance, making them more difficult to destroy than Ag-uncoated TCE and explaining the higher destructive temperature of Ag-coated TCE than uncoated TCE.

4. Conclusions

AgNWs with a high aspect ratio of ~1000 were synthesized successfully by polyol method. The diameter and length of AgNWs are in the ranges of 25–54 nm and 5–30 µm, respectively. the obtained AgNWs shows highly crystallinity. The sheet resistance of the AgNW TCEs was considerably reduced from hundred to ten Ω/\Box after coating an Ag layer onto the AgNWs. The best Ag-coated AgNW TCE was observed at an electrodepositing current of 1 mA for 40 s and showed a sheet resistance of 8.5 Ω/\Box , transmittance of 85.4% at 550 nm wavelength, and FOM value of 271 Ω^{-1} . The Ag-coated TCEs showed higher thermal stability in comparison with uncoated one; namely, maximum thermal stability was increased from 215 to 235 °C. Ag-coated AgNW TCEs are very promising for application in optoelectronic, photovoltaic as well as thermal heater devices.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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