

Research Article

Transparent Conductive Films of Copper Nanofiber Network Fabricated by Electrospinning

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Cu nanofiber networks can be a good alternative of the Ag nanowire of high electrical conductivity while having the advantage of low price. An electrospinning method was developed to fabricate copper nanofiber network for use as a transparent conductive film on glass substrate. The effects of liquid diluents for electrospinning processability were examined in relation to the subsequent Cu nanofiber formation processes. Electrospinning solutions of copper acetate/polyvinyl alcohol (PVA) and copper nitrate trihydrate/polyvinyl butyral (PVB) were investigated. The polymer mixing solutions influenced the subsequent annealing temperatures for removal of the polymers and reduction of the formed CuO nanofibers to Cu metal nanofibers. The morphology and structures of the formed nanofiber networks were examined by scanning electron microscopy, energy dispersive spectroscopy, X-ray diffraction, and so forth. The mixture with PVB provided lower annealing temperatures suitable for application to flexible substrates.

1. Introduction

Transparent conductive films (TCFs) are the key element of display panels adopted in many human interface devices. Indium-tin oxide (ITO) has been an excellent material used as the TCF for long time [1]. However, the portable electronics require flexibility as a general requirement in the electronics and the hard ITO showed degradation in electronic conductivity with bending [2, 3]. Furthermore, the high price and limited supply of indium demanded a substitution material. A variety of techniques and materials to replace ITO have recently been studied including carbon nanotubes (CNTs), graphene, conducting polymers, metal nanowires, and their composites [4–17]. The web of nanowires of high-conductivity materials are expected to show conservation of the electrical conductivity on bending substrates. In other words, the nanowire-to-nanowire contacts in motion were expected not to degrade the overall film conductivity. Actually, the web structural films revealed excellent reliability under bending of the substrates [13, 14]. It was found that

the performance (which was measured as the conductivity at a given transparency for a film) of silver films approached that of ITO while the CNTs showed lower conductivities [13, 14].

The vulnerability of silver, however, may be the relatively high price as a novel metal. As a consequence, they began to study copper as a candidate to replace ITO mainly because Cu is a relatively inexpensive material while showing similar conductivity. Several chemical methods were developed and commercialized for synthesis of Cu nanowires [18, 19]. However, the length of nanowires was often limited to several micrometers and the conductivity of the films at a transparency was very poor because the uniform percolation of the nanowires was hardly established due to agglomeration [18]. Therefore, elaborate research is required to chemically fabricate long and thin nanowires. Furthermore, copper easily oxidized and the wire-to-wire contacts may reveal high contact resistances resulting in a low conductivity. As such, we studied electrospinning method to overcome the limitation of the method of spreading the Cu nanowires on substrates. Electrospinning (ES) is a process to obtain a fine

thickness of long fibers of a variety of materials in large quantities at a low cost. Considering the complex chemical synthesis and purification process steps in the nanowire synthesis as well as the resulting insufficient length of the nanowires, the electrospinning can be a promising method to synthesize continuous Cu nanowires [20–23]. The figure-of-merit similar to TCF of silver nanowires was demonstrated by electrospinning of Cu [23]. Furthermore, electrospinning is a method that can be easily modified to a large-area synthesis technique.

However, the source materials used for the electrospinning process need to be prepared with appropriate physical properties including the viscosity, which determines the diameter of the nanowires as well as the processability itself. For this purpose, the source chemicals are frequently mixed with viscous polymers that are to be removed later by heating. A typical example is polyvinyl alcohol (PVA) and it can be removed by heating to a rather low temperature below 200°C [24]. We investigated the electrospinning technique to fabricate films of copper nanofiber web having high conductivity for flexible substrate application. We also report the limitation of the technique for the purpose application.

2. Experiments

2.1. Electrospinning of Cu Acetate in PVA Solution. The PVA solution was made by mixing 5 g of polyvinyl alcohol (PVA, Mw; 89,000–98,000) [25–27] in 50 mL of distilled water followed by stirring for 6 hours at 90°C. Copper acetate [CuCH_3CO_2] of 0.4 mol was added in a liter of the PVA solution with stirring for 6 hours at 60°C. The prepared source was injected into a syringe to electrospin on the glass substrates of 3 cm × 3 cm size to form a nanowire web of copper mixed in PVA. The distance of the syringe tip from the substrate was fixed at 10 cm. The bias for electrospinning was fixed at 23 kV, and the electrospinning rate was 0.5 mL/h. The film thickness of the nanowire web was controlled by the amount of solution to be emitted from the syringe (from 0.01 mL to 0.04 mL). Removing of PVA from the nanowire was done by heating up the substrates to 200–500°C for 2 hours in the atmosphere. It was found that only the heat treatment at 500°C completely removed PVA from the nanowires. This heat treatment in the air also oxidized the copper in the nanowires into copper oxide, and thus the copper oxide nanowires were then heat-treated at 200–500°C for 2 hours in a hydrogen atmosphere in order to reduce the copper oxide into copper. The complete reduction occurred only at 500°C. The rather high heat treatment temperatures caused disconnection of Cu nanowires on the substrate. Furthermore, the temperature is too high to apply to the popular flexible substrates such as PEN (polyethylene naphthalate) and PET (polyethylene terephthalate) polymer films. The change of the nanowire chemistry was schematically depicted in Figure 1.

2.2. Electrospinning of Cu Nitrate Trihydrate in PVB Solution. The polyvinyl butyral (PVB) solution was made by mixing 2.2 g of PVB [28, 29] in 20 mL of isopropanol/DI water (8 : 2) solution followed by stirring for 1 hour. Thereafter, 4.41 g of

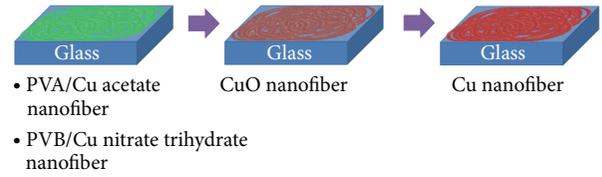


FIGURE 1: Schematic of the synthesis process for Cu nanofiber networks.

copper nitrate trihydrate [$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$] was mixed in the solution for 6 hours. Thus prepared source was injected into a syringe to electrospin on the glass substrates of 3 cm × 3 cm size to form a nanowire web of copper mixed in PVB. The distance of the syringe tip from the substrate was again fixed at 10 cm, but the bias for electrospinning was changed to an optimized value of 15 kV at the electrospinning rate of 0.5 mL/h. The film thickness of the nanowire web was again controlled by the amount of solution to be emitted from the syringe (from 0.01 mL to 0.04 mL). Removing of PVB from the nanowire was done by heating up the substrates to 200–500°C for 2 hours in the atmosphere. It was found that the heat treatment at a lower temperature of 450°C could remove PVB from the nanowires. Furthermore, the reduction could be done at 300°C in this case, and this is the main advantage of using PVB as the mixing material for Cu source.

2.3. Characterization of the Films. The microstructures of the films were examined by the field-emission scanning electron microscope (FE-SEM, JSM-7000F, JEOL, Japan) equipped with energy dispersive X-ray spectroscopy (EDS) and the chemical composition and crystal structures by X-ray diffraction (XRD, Rigaku, D/MAX-RC, Japan). Electrical resistance of the films was measured using a multimeter after formation of conductive pad of silver paste at both edges of the substrate. Light transmission was measured in the wavelength region of 400–700 nm using a UV-Visible spectrophotometer.

3. Results and Discussion

3.1. Cu Nanofiber Web by Electrospinning of Cu Acetate in PVA Solution. Figure 2 shows the surface morphologies of the electrospun nanofiber webs (a) for as-deposited nanofibers of copper acetate + PVA, (b) after oxidation of the nanofibers of (a) at 500°C, and (c) after reduction treatment of (b) at 500°C. The total amount of emission was 0.025 mL in this study unless otherwise specified. The electrospinning of the Cu acetate + PVA solution on the substrate formed smooth continuous fibers. The diameters of the electrospun nanofibers were distributed in the range of 50–150 nm (Figure 2(a)), and the distributed fibers are in amorphous form as indicated by the XRD measurement (Figure 3(c)).

The annealing of the electrospun fibers in air environment changed the physical chemistry of the fibers. In Figure 3(a), we compared the amount of carbon and oxygen atoms in the fibers as measured by the EDS with varying the heat treatment temperatures in the atmosphere. One observes

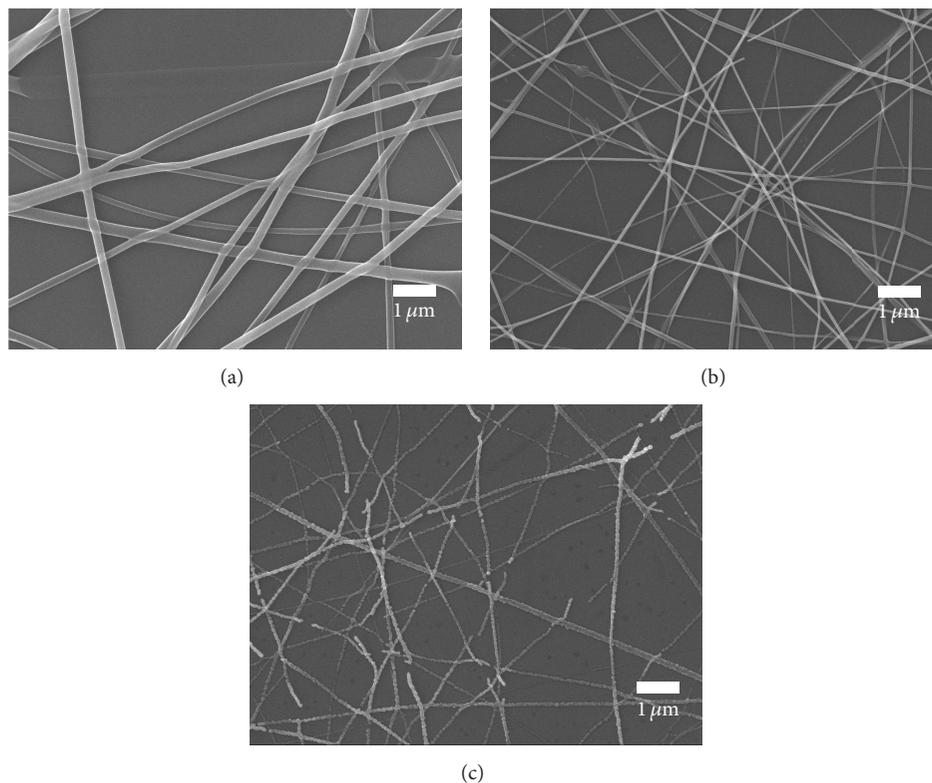


FIGURE 2: SEM images (a) of the Cu acetate/PVA nanofibers electrospun on glass substrate, (b) after annealing at 500°C in air for 2 h and (c) after subsequent annealing at 500°C in hydrogen atmosphere for 2 h.

decreasing of C content and increasing of O content with increasing of the temperatures. The trend indicates that PVA degrades and evaporates with annealing in oxygen environment while Cu converts to CuO. Figure 3(a) showed that the C content approaches zero at 500°C, which indicates that the evaporation of PVA cannot be completed by an annealing below 500°C. At the same time, the content of O also ever increased with the annealing temperature. This observation, or the removal of PVA going together with oxidation of Cu, suggests that Cu is chemically connected with carbon of PVA. It was actually shown that the thermal decomposition temperature of PVA shifts to lower temperature when it is mixed with foreign entities, and the shift was explained by some defects induced in the polymer by the mixing [30, 31]. However, the temperature for completion of the decomposition was not different at 450–500°C. Actually, such a high temperature (500–600°C) was required for calcination of electrospun copper acetate when mixed with polyvinyl alcohol or polyvinyl acetate solution [22, 23].

The results of EDS measurement in Figure 3(a) thus well coincide with the previous reports. The evaporation of PVA and decomposition of Cu acetate through the oxidation processing at 500°C led to a dramatic decrease in diameters as shown in Figure 2(b). The formed Cu oxide showed a stoichiometric CuO structure as clearly shown by the XRD pattern in Figure 3(c) [22]. The monoclinic CuO phase reveals (111) at 35.4° and (200) at 38.5° from JCPDS-5-0661.

The CuO nanofiber films formed through oxidation at 500°C were reduced at different temperatures in hydrogen ambient, and the EDS results are summarized in Figure 3(b). The reduction process could be completed via heat treatment at 500°C [as indicated by zero oxygen content (Figure 3(b))]. The diameter change after reduction was not remarkable, but agglomeration of Cu into particles on the glass substrate is remarkable. The observation indicates poor wetting between Cu and the glass substrate. The effect of reduction temperature on the Cu morphology was examined in more detail in Figure 4, which shows the morphologies after the reduction process at different temperatures, 300–600°C, for 2 hours. The fibers maintained the continuity at 300°C, where the reduction did not occur. The rather complete reduction occurred at 500°C as shown in Figure 3(b), but the liquefaction of Cu with agglomeration already began at 400°C and led to disconnection of the fibers as shown in Figure 4(b).

In conclusion, when PVA solution was electrospun, the requirement of high temperature (500°C) for polymer decomposition also required high reduction temperature for CuO. The latter resulted in melting and shrinking of Cu wires leading to breakage of percolation (or electrical connection) of the fibers. The net result is relatively high resistance of the Cu film as will be compared later in Table 1.

3.2. Cu Nanofiber Web by Electrospinning of Cu Nitrate Trihydrate in PVB Solution. Cu nitrate trihydrate may oxidize

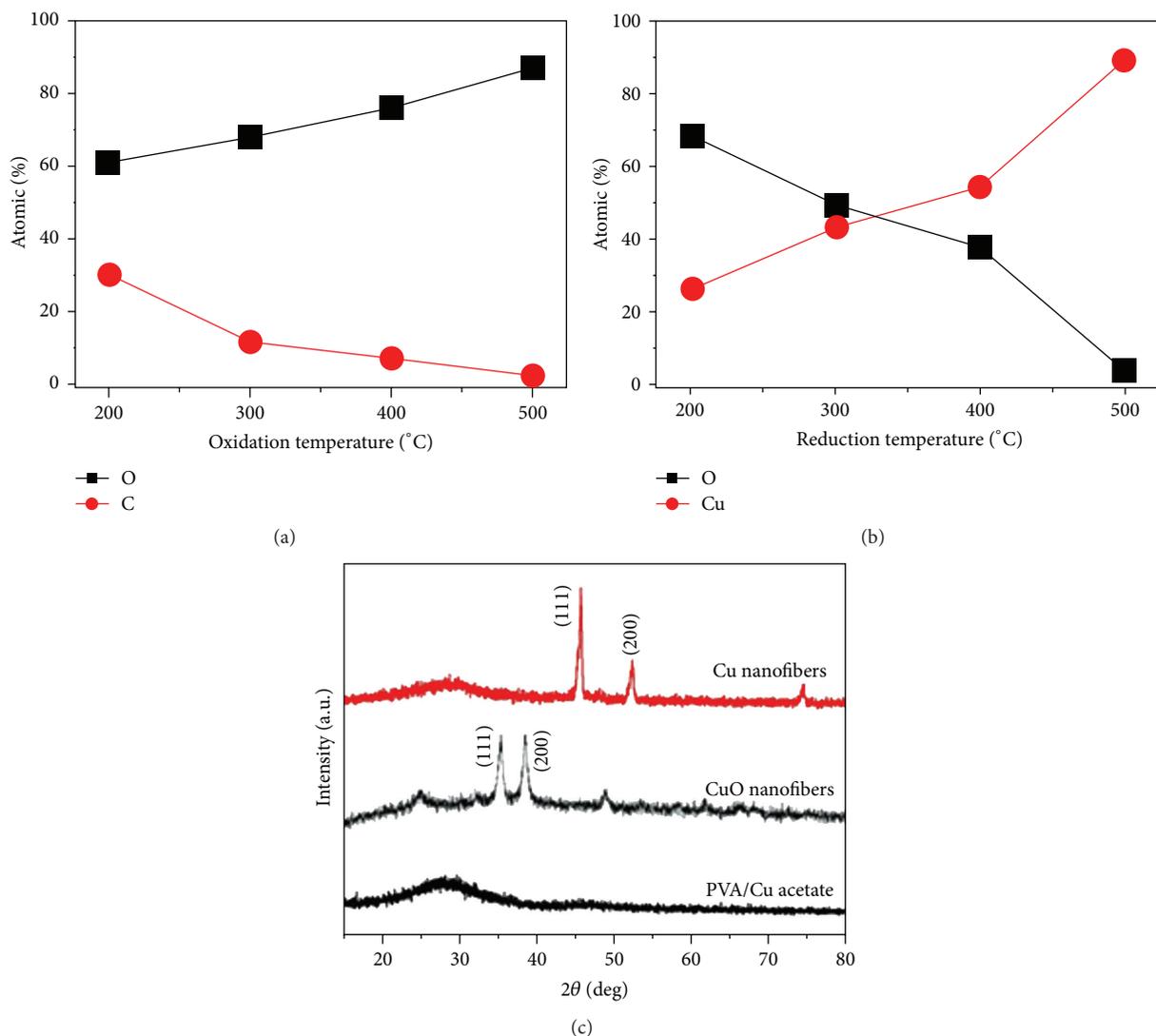


FIGURE 3: (a) Atomic percent of C and O measured by EDS after annealing at different temperatures in the air; (b) atomic percent of Cu and O measured by EDS after annealing at different temperatures in H_2 environment. (c) XRD patterns for the as-electrospun nanofiber sample, after 500 °C oxidation in (a) and after 500 °C reduction in (b).

to CuO at temperatures below $\sim 200^\circ\text{C}$ [32]. The preliminary experiment showed that mixing in PVB revealed much thicker fibers of >300 nm. Figure 5(a) shows the morphology of the as-electrospun Cu nitrate trihydrate + PVB fibers, which clearly shows thicker diameters than the PVA mixture above. Again the total amount of the solution emitted from the nozzle was 0.025 mL. Figure 5(b) shows the morphology after oxidation treatment of Figure 5(a) at 450°C for 2 hours and revealed that the diameters are in the range of 250–350 nm. Figure 5(c) shows the morphology after subsequent reduction treatment at 300°C for 2 hours for the CuO nanofibers of Figure 5(b).

Actually the selected oxidation and reduction annealing temperatures in Figure 5 were obtained from the experimental results of Figure 6, which summarize the EDS measurements after the oxidation and reduction processes carried out at different temperatures. The results of Figure 6(a) clearly

showed that carbon almost disappeared at 400°C as an indication of the complete removal of PVB from the fibers. It was shown that the pure PVB begins to decompose at $\sim 200^\circ\text{C}$ and ends at $\sim 400^\circ\text{C}$ [33–35]. While our observation well coincides with the literature observation, mixing with other materials could result in reduction of the decomposition temperature as observed with PVA [34]. Note also in Figure 6(a) that the O content monotonically increased with increasing temperatures. The steady trend may reflect a linear composition change of the copper oxide such as from Cu_2O to CuO. Therefore, the lower oxidation temperature of 450°C could lead to stoichiometry of Cu_xO ($1 < x < 2$) or defective copper oxide with poorer crystallinity. The significantly lower reduction temperature of 300°C shown in Figure 6(b) can be explained by the defective copper oxide formed at a lower temperature. The mechanism for such a low reduction temperature needs to be explored in more

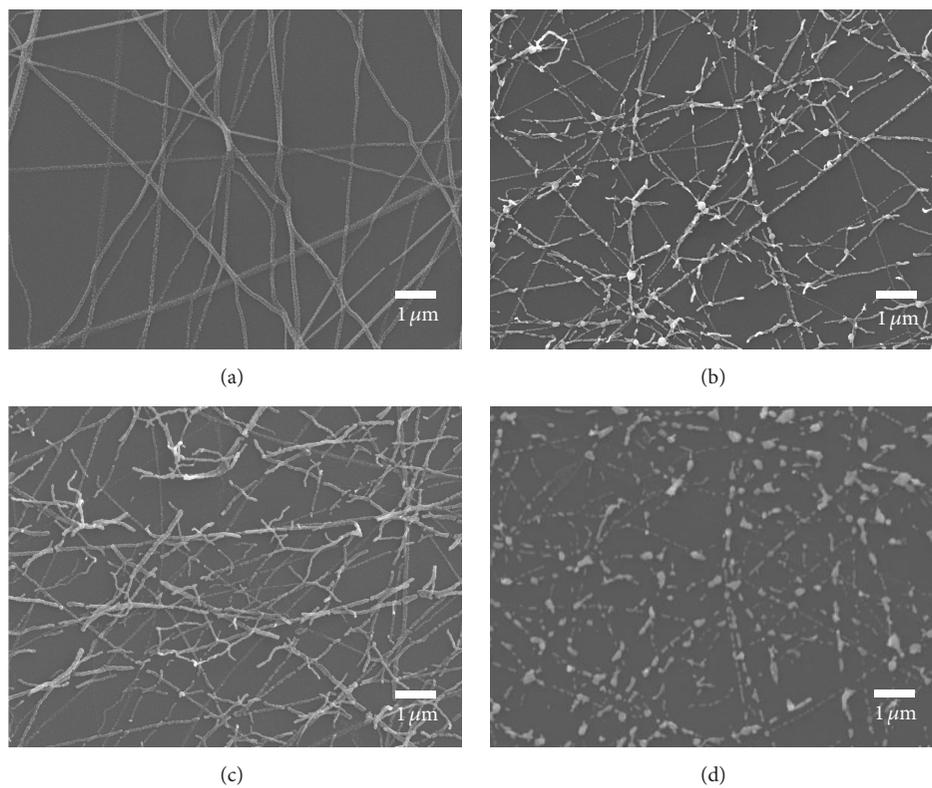


FIGURE 4: SEM image of Cu nanofibers formed via reduction of CuO nanofibers in hydrogen environment at different annealing temperatures of (a) 300°C, (b) 400°C, (c) 500°C, and (d) 600°C.

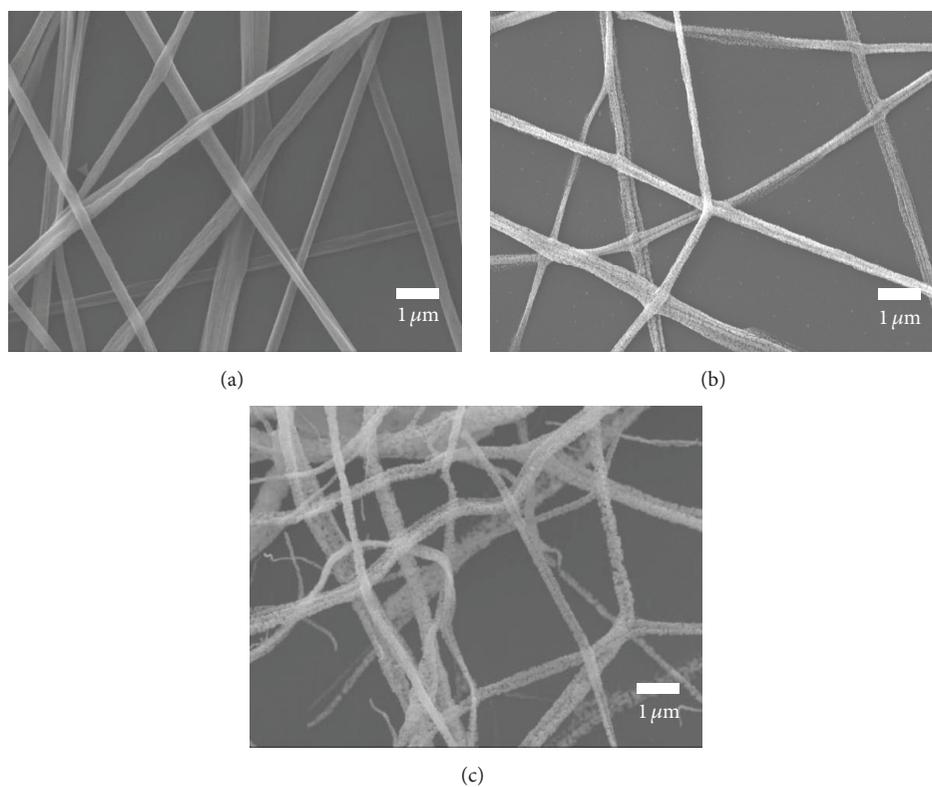


FIGURE 5: SEM images (a) of the electrospun Cu nitrate trihydrate/PVB composite fibers, (b) after oxidation annealing at 450°C in air ambient for 2 h and (c) after subsequent reduction annealing at 300°C in hydrogen atmosphere for 2 h.

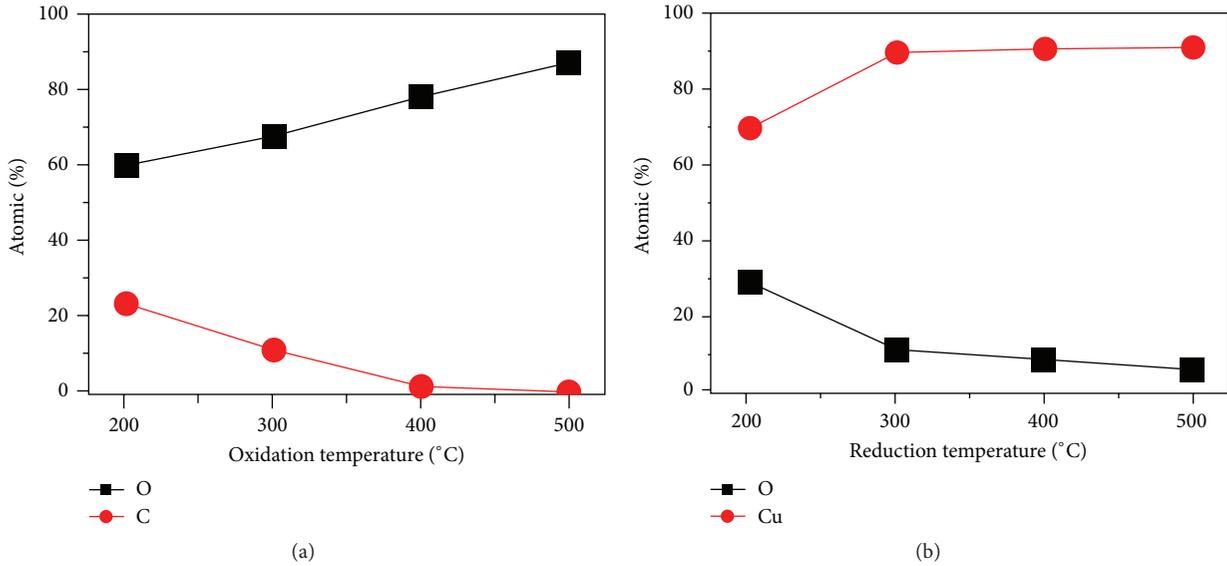


FIGURE 6: Atomic percent of (a) C and O after oxidation annealing at different temperatures and (b) Cu and O after reduction annealing at different temperatures for oxidation treated at 450°C for Cu nitrate trihydrate/PVB nanofibers.

detail, but it was clearly motivated by the decomposition temperature of PVB lower than PVA. Such a low processing temperature is a very useful requirement if variable substrates are to be introduced. Figure 5(c) clearly showed that the Cu nanofibers are now continuous on the substrate because of the lower reduction temperature employed.

3.3. Electrical and Light Transmitting Properties. The total amount of electrospinning for Cu nitrate trihydrate + PVB was varied from 0.010 to 0.040 mL to change the conduction and transparency of the TCFs. The morphologies of the Cu nanofibers fabricated according to the developed conditions (450°C oxidation and 300°C reduction) above are shown in Figure 7. Since Cu nanofibers running on the substrate are not transparent, the open spaces between the fibers determine the transmittance.

The light transparency and electrical conductivity for electrospinning with PVA and PVB mixtures are also compared in Table 1 for different total amount of electrospinning. The light transmission was not significantly different between PVA and PVB mixtures at each amount of electrospinning because total amounts of Cu covering the substrate are similar. However, the electrical resistance of the TCF made from the PVB mixture revealed a 10-fold lower value than from the PVA mixture. The latter result is simply because of the discontinuity of the nanofibers on the substrate caused by the high reduction temperature used in the PVA mixture case.

The light transmittance of the as-electrospun solution, CuO nanofibers, and Cu nanofibers measured at various wavelengths was summarized in Figure 8 for 0.04 mL electrospinning. The light transmittance at the wavelength of 550 nm was 57%, 75%, and 92% for the Cu nitrate trihydrate + PVB, CuO nanofibers, and Cu nanofibers, respectively. The enhancement of the transmittance was thus caused by the

TABLE 1: Optical and electrical properties of the Cu nanofibers fabricated with different amounts of Cu source.

| Amount of Cu source | Cu nanofiber from PVA + Cu acetate | Cu nanofiber from PVB + Cu nitrate trihydrate |
|-------------------------|------------------------------------|---|
| 0.010 mL | | |
| Transmittance (%) | 91.4 | 91.7 |
| Resistance (Ω) | 4502.2 | 284.6 |
| 0.025 mL | | |
| Transmittance (%) | 80.2 | 76.1 |
| Resistance (Ω) | 840.7 | 94.3 |
| 0.040 mL | | |
| Transmittance (%) | 58.7 | 59.2 |
| Resistance (Ω) | 580.4 | 83.5 |

removal of polymer and oxygen from the fibers. The electrospun Cu nanofiber network as a transparent conductive film device was not yet optimized to show a comparable performance with other Cu nanowire films [18].

4. Conclusions

Electrospinning method to fabricate copper nanofibers spread on a substrate as transparent conductive films was studied. The processing steps are electrospinning of Cu solutions (Cu source materials + liquefaction polymer solutions) to form nanofibers, annealing in air ambient to remove polymer material and oxidize Cu, and annealing in hydrogen ambient to change copper oxide nanofibers to copper nanofibers. Two kinds of diluent and liquefaction solutions of PVA and PVB were tested. PVA showed a

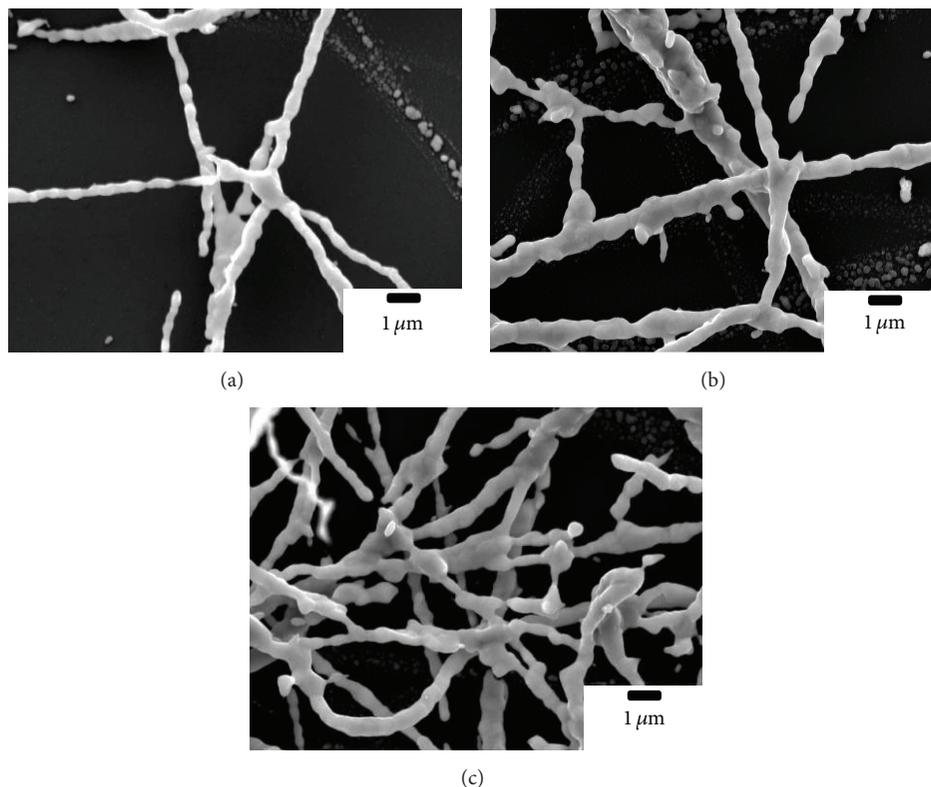


FIGURE 7: SEM images of Cu nanofibers formed by electrospinning of different amounts of the solution: (a) 0.010 mL, (b) 0.025 mL, and (c) 0.040 mL.

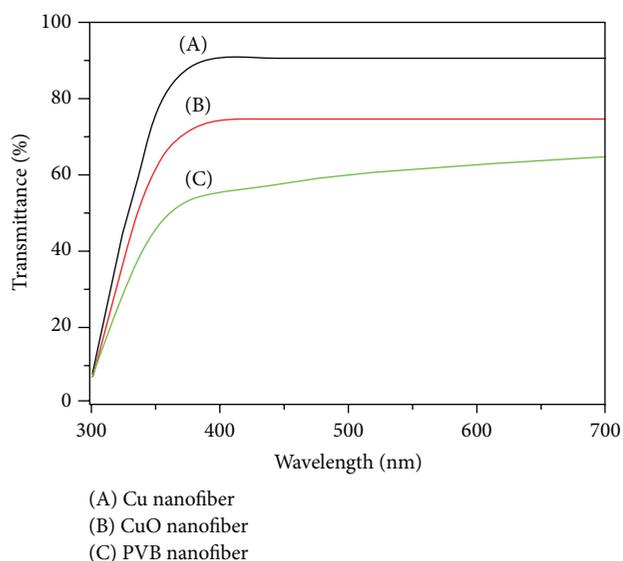


FIGURE 8: Transmittance of nanofiber networks of Cu nitrate trihydrate/PVB, CuO, and Cu (the glass transmittance subtracted).

reaction with Cu acetate with mixing. This led to a high annealing temperature of 500°C for removal of PVA from the mixture. The high temperature oxidation possibly led to a high crystallinity CuO, which in turn increased the reduction temperature required. It was 500°C in hydrogen ambient

for Cu nanofiber network formation. The high reduction temperature was the main reason for the Cu nanofibers melting and consequent disconnection. In the meantime, Cu nitrate trihydrate mixing with PVB required lower oxidation and reduction temperatures of 450°C and 300°C, respectively. The fabricated Cu nanofiber network led to a percolation condition, and a relatively simple economic procedure was developed. However, the heat treatment temperatures need to be further lowered for application to flexible polymer substrates.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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