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Effect of a Urethane Acrylate-Based Photosensitive Coating on the Reliability of Ag Nanowire Transparent Electrodes

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Abstract: Due to the susceptibility of Ag nanowires to external mechanical and chemical damage, maintaining high optical performance and ambient and mechanical stability during the fabrication process is important for the industrial use of Ag nanowire transparent electrodes (TEs). In this study, urethane acrylate-based photosensitive resin (UAPR) is used as the coating material for Ag nanowire TEs to improve their optical transmittance, ambient stability, and resistance to external wiping damage. In the proposed method, UV-curable UAPR is coated onto Ag nanowire TEs using a simple doctor blade, forming a protective coating that increases the optical transmittance of the electrodes due to the refractive index of the UAPR between the air and the substrate. The UAPR coating successfully protects the Ag nanowires from corrosion in ambient air, with no significant change in their optical or electrical properties observed after 180 h of exposure to ambient air. Mechanical wiping tests also confirm that the UAPR coating is effective in protecting the Ag nanowires from external wiping damage, with no degradation of the optical or electrical properties observed after six wiping cycles.

Keywords: electrode; transparent; coating; Ag nanowire; stability

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1. Introduction

Given the increasing advances in technological innovations for flexible electronics, the demand for chemically and mechanically reliable flexible transparent electrodes (TEs) has increased [1–12]. The current choice for flexible TEs is indium tin oxide (ITO) because it has high optical transparency and electrical conductivity; however, its brittle nature under deformation limits its use in flexible electronics [13,14]. Several candidates to replace ITO have been proposed, including carbon nanotubes [15,16], graphene [17,18], and metal nanowires [19,20]. Of these alternatives, Ag nanowires are considered the most promising due to their higher optical and electrical performance as TEs compared to carbon-based materials [21,22]. In addition, the scalable production of Ag nanowires using polyol reduction processes and their mechanical reliability under bending or stretching also make them suitable for industrial use [23,24].

Despite the advantages of Ag nanowires, they cannot be used alone in practical devices because of their susceptibility to oxidation and sulfidation in ambient air and their low scratch resistance. Due to their large surface-to-volume ratio, Ag nanowires are easily oxidized and sulfidized, which leads to the degradation of the optical and electrical properties of the resulting TEs [21,25]. In addition, flexible electronic devices can be damaged by external scratches during the production process [26]. Bare Ag nanowire electrodes deposited on a flexible polymer substrate are particularly vulnerable to scratch

damage due to the weak adhesion between the two layers [27]. Therefore, a coating layer is required for protection.

To date, several types of coating material have been proposed, including metal oxides [4,25], graphene [6,28], and polymers [29,30]. Metal oxides generally show excellent resistance to chemical and scratch damage, but their brittleness degrades electrical conductivity under bending [4,25]. In addition, metal oxide deposition requires a vacuum-based fabrication process, which increases production costs. Graphene has low permeability to gas and water molecules, which can protect Ag nanowires from chemical damage and has excellent flexibility, making it suitable for flexible electronics [28]. However, its adhesion to polymer substrates is weak; thus, there is no dramatic improvement in scratch resistance when employing graphene as a coating material. The mass production of high-quality graphene is also challenging, which limits its industrial applications [31]. In contrast, polymer-based coating materials can easily be coated onto a substrate using simple printing or coating processes [21,24], reducing production costs compared to other coating candidates, while also offering high chemical resistance and excellent mechanical properties.

Despite the advantages of polymer-based materials, the selection of an appropriate polymer coating material for Ag nanowire TEs is challenging. This is because any mismatch between the physical properties of the coating and the substrate can alter the optical and mechanical performance of Ag nanowire TEs. Due to the difference in the refractive indexes of the coating material and the substrate, light can be reflected at the interface between the two, altering the optical transparency of the TE [32]. In addition, mismatched thermal coefficients and elastic moduli can lead to the delamination of the coating material from the substrate, leading to device failure [33]. Therefore, developing coating materials with the physical properties necessary to overcome these issues is crucial.

In this study, urethane acrylate-based photosensitive resin (UAPR) was investigated as the coating material for Ag nanowire TEs. UAPR solution was coated onto the TEs using a simple blade coating process followed by UV exposure to crosslink the UAPR. By measuring the change in the optical transmittance before and after the application of the coating by using an optical transmittance measurement system, its effect on the optical transmittance of the TEs was assessed. In addition, the thermal stability of the UAPR-coated Ag nanowire TEs was analyzed using thermal cyclic tests. To confirm their chemical stability, TE samples were exposed to ambient air. In addition, scratch resistance was tested by measuring the sheet resistance as a function of the number of surface wiping cycles using an eddy current-based non-contact measuring system.

2. Experimental Details

Figure 1 illustrates the fabrication process for UAPR-coated Ag nanowire TE. A 0.15 wt% Ag nanowire solution in isopropyl alcohol (IPA) was purchased from Nanopyxis and used without post-treatment. The Ag nanowires had an average diameter and length of ~35 nm and ~15 μm, respectively. The Ag nanowire solution was coated on a polyethylene terephthalate (PET, Kolon Inc.) substrate with a thickness of 125 μm using doctor blading, as shown in Figure 1a. The blade height and speed were fixed at 150 μm and 10 mm/s, respectively. To remove the solvent, the as-coated Ag nanowires were dried in ambient air at 80 °C for 10 min using a convection oven. UAPR resin containing ~1.5 wt% 1–hydroxycyclohexyl phenyl ketone as the photoinitiator was provided by BASF, which was then coated onto the Ag nanowire samples using the same doctor blading system (Figure 1b). The blade height and the blading speed for this step were 160 μm and 10 mm/s, respectively. The coated Ag nanowire TEs were then exposed to UV light with a light intensity of 30 mW/cm^2 at 365 nm for 60 s using a UV chamber (KGW–94 N, KAIS) as shown in Figure 1c,d. The resulting cured UAPR coating had a thickness of 3 μm as measured using an Alpha-Step (KLA Tencor, D–500), which was the thinnest layer that could be achieved in this study. Below 3 μm, the thickness of the coating was non-uniform through the sample, while the thickest layer possible using our fabrication system that did

not lose its uniformity was ~10 μm. There was no major difference in the optical properties of the layer up to a thickness of ~10 μm, which showed an optical transmittance of ~91.3% up to ~10 μm. The cured UAPR exhibited a pencil hardness of 2H; in a crosshatch adhesion test using scotch tape, no lifting was observed, confirming that the UAPR had suitable adhesion to the PET substrate.

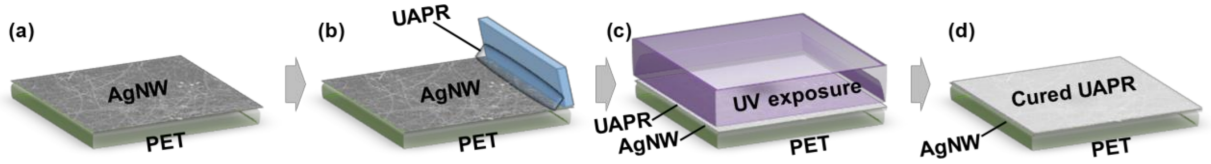


Figure 1. Illustration of the fabrication process for the UAPR-coated Ag nanowire transparent electrode: (**a**) coating of Ag nanowire on PET substrate, (**b**) UAPR coating with a doctor blade, (**c**) UV exposure to cure UAPR, (**d**) UAPR-coated Ag nanowire electrode.

The sheet resistance of the Ag nanowire TEs was measured using a non-contact sheet resistance measurement system (Napson, EC–80) to prevent potential damage caused by the probes of the conventional contact-based measurement system. Before the coating process, the sheet resistance of the Ag nanowire TEs was measured using both the non-contact system and a typical four-point probe method (FPP–2400). For the same samples, the sheet resistance values were similar when measured using the non-contact and four-point probe methods. After the coating had been applied, the resistance measured using the non-contact system did not change significantly when compared to the resistance of the bare electrode. These results confirmed that there was no significant difference in the sheet resistance measured by the contact and non-contact systems. The sheet resistance before and after UAPR coating was ~50.6 ohm/sq and ~51.3 ohm/sq, respectively. Optical transmittance was characterized using an optical transmittance measurement system (BYK, haze-gard i). Bright-field optical images of the UAPR-coated Ag nanowire TEs were taken using an optical microscope (Nikon, Eclipse LV150N). The detailed microstructure of the samples was analyzed using a scanning electron microscope (FE–SEM, XL30 ESEM–FEG, Phillips). Bending tests were performed by using a manual bending tester (Woorim Tech.) under the condition of a 3 mm bending radius and 100 bending cycles. For bending tests, the samples were cut into 5 cm × 5 cm sizes, and both ends of the samples were clamped to the clip of the upper and lower plates of the bending tester. By folding and unfolding the upper and lower plates, the bending strain was applied to the samples. During every 20 cycles of bending, the sheet resistance and optical transmittance were measured in the bent region of the samples.

3. Results and Discussion

To confirm the surface morphology of the Ag nanowire TEs before and after UAPR coating, SEM images were analyzed (Figure 2). The uncoated electrode exhibited a network structure (Figure 2a), while a smooth surface was observed for the UAPR-coated electrode (Figure 2b). Because the Ag nanowire TEs were uniformly covered by the UAPR, the network morphology was difficult to observe in the SEM image. To confirm the effect of the UAPR coating on the optical performance of the Ag nanowire TEs, their optical transmittance with and without UAPR was measured. The optical transmittance of an electrode is determined by its light reflection behavior, which needs to be closely controlled. In the UAPR-coated Ag nanowire electrodes, there were two major light reflection sources: the surface of the nanowires and the interface between the coating and the substrate. The use of long Ag nanowires with a small diameter increases the optical transmittance by reducing the fraction of the light reflected at the surface of the nanowires. However, there is a trade-off between the diameter and the length of the Ag nanowires; thus, controlling

the dimensions of Ag nanowire TEs is challenging. Therefore, an alternative method to enhance the optical transmittance of Ag nanowire electrodes is required.

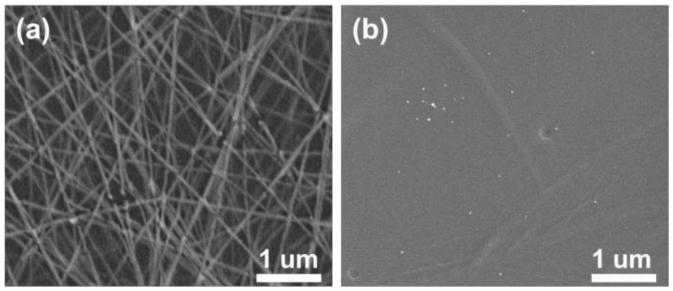


Figure 2. SEM images of (**a**) the bare Ag nanowire electrode and (**b**) the Ag nanowire electrode with UAPR coating.

Employing an appropriate coating material is a promising way to enhance the optical transmittance without controlling the Ag nanowire dimension. According to Fresnel's theory, the optical transmittance of Ag nanowire electrodes on a transparent substrate can be tuned using a coating layer. A coating material with a refractive index between that of the substrate and air reduces the amount of light reflected at the interface between the substrate and the coating, which can enhance the optical transmittance. UAPR, the coating material used in the present study, has a refractive index of ~1.59, which is between that of polymer substrates that are widely used for Ag nanowire TEs, such as PET or polyethylene naphthalate (PEN) and air. Therefore, employing UAPR is expected to enhance the optical transmittance of Ag nanowire TEs on a PET or PEN substrate.

Table 1 presents the optical transmittance of Ag nanowire TEs fabricated on various substrates with and without the UAPR coating. Overall, the optical transmittance of the Ag nanowire electrodes was increased with the UAPR coating. The bare Ag nanowire electrodes on a PET substrate had an optical transmittance of ~90.6%, which rose to ~91.3% after coating. For the Ag nanowire electrodes on a PEN substrate, the optical transmittance before and after UAPR coating was ~88.9% and ~90.1%, respectively. The Ag nanowire electrodes on an ITO-covered PET substrate also exhibited greater optical transmittance. The increase in the optical transmittance with the use of UAPR coating is due to the refractive index of UAPR (~1.59), which is between that of the air (1.0) and the substrates (Table 1), as indicated by Fresnel's theory [29]. Therefore, it was confirmed that UAPR is an appropriate coating in terms of the enhancement of the optical transmittance.

Table 1. Optical transmittance of Ag nanowire TEs with and without UAPR on various substrates.

Substrate (Thickness, Refractive Index)	Transmittance	
	Bare AgNW	AgNW with UAPR Overcoating (3 μm)
PET (125 μm, 1.64)	90.6 ± 0.3	91.3 ± 0.4
PEN (125 μm, 1.77)	88.9 ± 0.2	90.1 ± 0.3
ITO (100 nm, 1.92)/PET (125 μm, 1.64)	84.2 ± 0.2	85.7 ± 0.3

Protecting Ag nanowire TEs from the oxidation or sulfidation caused by the ambient air is also an important role of coating materials. To confirm that the UAPR coating could improve the ambient stability of Ag nanowire TEs, electrodes with and without the UAPR coating were exposed to ambient air for 180 h, and the change in optical transmittance and sheet resistance over time was measured. Figure 3a shows the variation in the optical transmittance of the Ag nanowire TEs with and without the UAPR coating as a function of exposure time to ambient air at 70 °C, which represents standard conditions for the

reliability testing of TEs in industry. The bare Ag nanowire electrodes had an optical transmittance of ~90.6% before exposure, which fell to ~89.0% during exposure to air. On the other hand, there was no significant change in the optical transmittance of the Ag nanowire TEs with the UAPR coating (~91.3% both before and after 180 h of exposure).

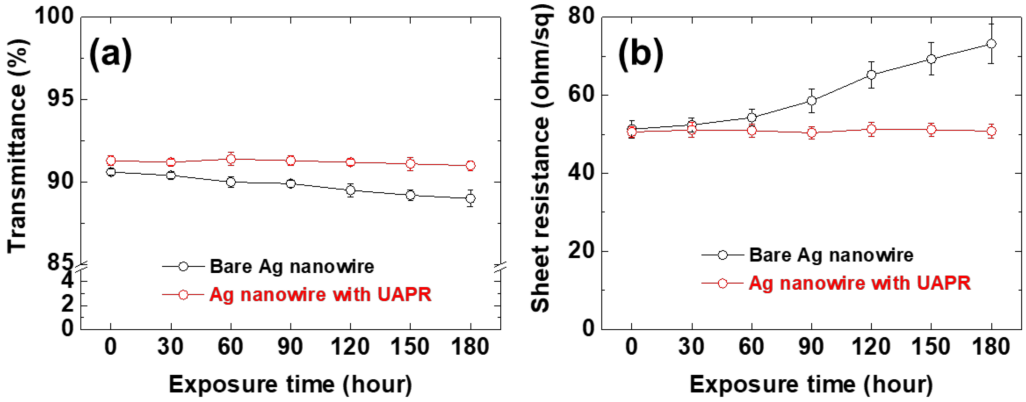


Figure 3. (**a**) Change in the optical transmittance and (**b**) sheet resistance of bare Ag nanowire transparent electrodes and UAPR-coated Ag nanowire transparent electrodes as a function of the exposure time to ambient air at 70 °C.

The trend in the sheet resistance was similar to that for the optical transmittance. While bare Ag nanowire electrodes exhibited an increase in the sheet resistance from ~51.32 ohm/sq to 73.22 ohm/sq after 180 h of exposure, the UAPR-coated Ag nanowire TEs maintained their initial sheet resistance over the entire period (Figure 3b). In ambient air, the surface of Ag nanowires corrodes, leading to the formation of oxide or sulfide derivatives on the surface. The reduction in the cross-sectional area of the nanowires due to this corrosion restricts the current flow, thus lowering the electrical conductivity of Ag nanowire TEs [21,25]. In addition, the oxide or sulfide particles that form on the Ag nanowire surface due to the corrosion increase the probability of light reflecting at the surface of the electrode [25], in turn reducing its optical transmittance. After coating the nanowires with UAPR, however, contact between water vapor or gas molecules in the air with the surface of the nanowires is effectively prevented. Furthermore, in stability testing with nitric acid, Ag nanowire TEs with UAPR coating showed no significant change after 2 h of immersion in 4 vol% nitric acid, while bare Ag nanowire electrodes lost their electrical conductivity after only 30 min of exposure. In summary, in the present study, the optical transmittance and sheet resistance of the proposed UAPR-coated Ag nanowire TE were very stable under prolonged exposure, which proves the effectiveness of the UAPR coating in enhancing the ambient stability of Ag nanowire TEs.

During the fabrication process of electronic devices that employ Ag nanowire TEs, the possibility of mechanical damage to the electrodes due to external wiping forces is high. Because the adhesion of Ag nanowires to the polymeric substrate is low, external wiping forces can remove Ag nanowires from the substrate, decreasing the electrical conductivity of Ag nanowire TEs. Therefore, protecting Ag nanowires from external wiping damage is crucial for the industrial use of Ag nanowire TEs. To investigate the effect of the UAPR coating on the resistance of Ag nanowire TEs to wiping damage, an artificial wiping force was applied to Ag nanowire electrodes with and without the UAPR coating using a cotton textile soaked in IPA. Figure 4a displays the variation in the optical transmittance of the Ag nanowire electrodes as a function of the number of times the wiping force was applied. The bare Ag nanowire electrode had an optical transmittance of ~90.6% before starting the test, which increased to ~91.0% after the first wiping cycle and then to ~91.9% after six wiping cycles. The optical transmittance of the bare Ag nanowire TEs thus approached that of bare PET film (~92.1%) because most of the Ag nanowires were removed from the PET substrate due to the wiping damage. However, the Ag nanowire electrodes with the

UAPR coating exhibited a constant optical transmittance of ~91.2% over the six wiping cycles because there was no significant loss of the Ag nanowires.

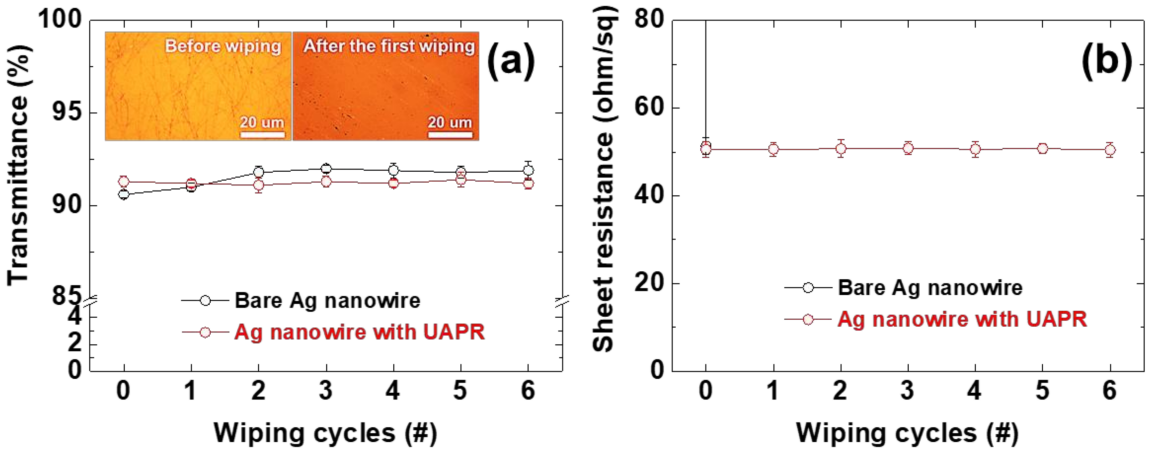


Figure 4. (**a**) Change in optical transmittance and (**b**) sheet resistance of the bare Ag nanowire transparent electrodes and UAPR-coated Ag nanowire transparent electrodes as a function of the number of wiping cycles.

Figure 4b presents the sheet resistance of Ag nanowire TEs with and without the UAPR coating in response to the application of a wiping force. After one wiping cycle, the bare Ag nanowire TEs lost their conductivity due to a significant loss of Ag nanowires. However, the UAPR-coated Ag nanowire TEs demonstrated a similar sheet resistance of ~50.49 ohm/sq after six wiping cycles, providing further evidence that there was no loss of Ag nanowires during the wiping test. These results thus point out the protective action conferred by UAPR coating on Ag nanowire electrodes against external wiping damage.

Bending tests were performed on the Ag nanowires with and without the UAPR coating. Figure 5a,b show the respective sheet resistance and optical transmittance of the samples as a function of bending cycles. There was no significant degradation of the sheet resistance and optical transmittance of both Ag nanowire TEs with and without UAPR coating even after 100 cycles of bending at 3 mm radius. The intrinsic flexibility and the thickness (~3 µm, i.e., relatively thin) of UAPR were able to keep the high flexibility and excellent optical transparency of the Ag nanowire TEs. In-depth research on the bending properties of the sandwich system for different types of flexible material tests under extreme conditions including much higher bending cycles and a smaller bending radius than those of this study might be required for some flexible devices used in harsh conditions, which will be interesting future work.

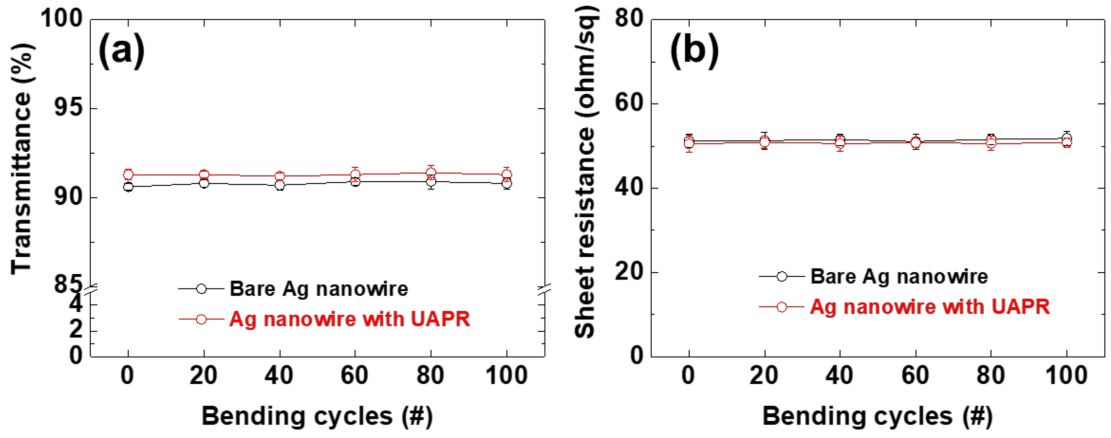


Figure 5. (**a**) Change in the optical transmittance and (**b**) sheet resistance of the bare Ag nanowire transparent electrodes and UAPR-coated Ag nanowire transparent electrodes as a function of the bending cycles.

4. Conclusions

In this study, the effect of UAPR coating on the performance of Ag nanowire TEs was investigated. UV-curable UAPR was coated onto the Ag nanowire TEs, and the resulting effect on the optical performance, ambient stability, and resistance to external wiping damage of the TEs was investigated. The UAPR coating was found to improve the optical transmittance of the Ag nanowire TEs because its refractive index was between that of air and the PET or PEN substrate. Given the importance of the optical transmittance of Ag nanowire TEs, a system including a UV-curable UAPR coating is expected to exhibit significant potential for use in industrial applications utilizing Ag nanowires. In addition, the UAPR coating successfully protected the Ag nanowires from corrosion when placed in contact with ambient air for 180 h at 70 °C, with no significant change in the optical transmittance or sheet resistance, while a significant degradation of optical transmittance and sheet resistance was observed for bare Ag nanowire TEs. Furthermore, mechanical wiping tests revealed that UAPR-coated Ag nanowire TEs experienced no significant change in their optical or electrical performance with mechanical wiping, while bare Ag nanowire electrodes exhibited significantly lower performance. The wiping tests thus confirmed that the UAPR coating was also effective in protecting the Ag nanowires from external wiping damage. In conclusion, the proposed UAPR coating represents a promising strategy for the electronics industry because it confers Ag nanowires a higher optical transmittance, ambient stability, and resistance to external wiping damage, all of which are highly desirable features for Ag nanowire-based TEs.

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Conflicts of Interest: The authors declare no conflict of interest.

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