Ethylcellulose/Ag Nanowire Composites as Multifunctional Patchable Transparent Electrodes

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ABSTRACT

Cellulose and its derivatives are attractive for the development of electronic devices because they are naturally abundant and biodegradable. Ethyl cellulose (EtC) is particularly promising as a substrate material for flexible electronics because it is waterproof with low air permeability and high transparency. In this study, adaptable EtC-Ag nanowire (AgNW) composite-based transparent electrodes (TEs) are developed for various electronic devices. Fabrication of EtC-AgNW composites is clean and safe without using toxic chemicals, resulting in highly transparent and conductive electrodes. With the assistance of functional tapes, EtC-AgNW composites are successfully used as attachable flexible transparent interconnects, and sensing electrodes for transparent electrophysiological sensors. In addition, a semitransparent perovskite solar cell is demonstrated by using the EtC-AgNW composites as a top electrode, which shows a high power conversion efficiency of 7.03%.

KEYWORDS: Ethyl cellulose, Nanocomposites, Ag nanowire, patchable, novel coating

1. Introduction

With technological advances in flexible/wearable electronics, there are increasing demands for flexible and transparent electrodes that are easy to prepare for applications related to various electronic devices, such as wearable sensors [1-3], flexible optoelectronic devices [4, 5], and transparent touch panels [6, 7]. The current strategy for the development of flexible transparent electrodes involves metal thin film electrodes formed on flexible substrates, whereby thin films are deposited using a vacuum-based deposition system (e.g., sputtering or evaporation), followed by patterning via lithography to form a mesh-like structure [8-10]. Metal thin film-based electrodes are highly conductive and flexible, making them suitable for wearable electronic devices; however, the complexity of their fabrication process using vacuum-based deposition and lithography increases the fabrication cost, thus limiting their application as electrodes. Printing methods can also be used for metal electrode fabrication, but unfortunately the pattern size is much larger than those formed by lithography, and the formulation of ink for printing is complex and expensive as well.

To overcome the limitations of the current technology, composite electrodes consisting of metal nanowire (NW) fillers and flexible/stretchable polymers have been developed [4, 11, 12]. Metal nanowire fillers were embedded in the polymer matrix of the composites, whereby current flows through the network of metal NW fillers, while excellent flexibility is imparted to the composite by both the stretchable network structure of NW fillers as well as the intrinsic flexibility of the polymer matrix. Because the metal NWs are synthesized in a large volume by solution processes and are coated via a simple coating process, as are the polymeric resins, the fabrication cost of the composites is much cheaper than those fabricated by vacuum-based deposition or printing. Although various composites (e.g., silver nanowires (AgNW)-polyvinyl alcohol (PVA) [13], AgNW-polyacrylate [14], AgNW-colorless polyimide (cPI) [15], and AgNW-epoxy [16]) were reported for different electronic devices,

studies on patchable composite electrodes are lacking. However, these patchable composite electrodes are a key component for wearable electronics or internet on thing systems, where the electronic devices are required to be attachable to the human body or desired surfaces. Therefore, the development of a facile and efficient method to fabricate reliable patchable electrodes with excellent optoelectronic performance is urgently needed.

In our previous study, we developed AgNW/poly(methyl methacrylate) (PMMA) based composite electrodes, which were attachable to quantum dot light-emitting diodes (QLEDs) as a top electrode [17]. The Ag NWs were deposited on a self-assembled monolayer (SAM)-treated substrate with a highly hydrophobic surface on which the PMMA layer was coated. With the assistance of thermal release tape, the AgNW/PMMA composites could be released from the SAM-treated substrate. They could then be attached to the top side of QLEDs via a simple hot-pressing process. The process is facile and quick, but there remains the inconvenience of conducting the SAM treatment using toxic perfluorodecyltrichlorosilane, as well as using polymers, which are not eco-friendly due to extremely long decomposition times.

Cellulose is a naturally abundant and biodegradable biomaterial [18, 19]. There are various derivatives of cellulose, including nitrocellulose [20], ethyl cellulose (EtC) [21], methyl cellulose [22], and bacterial cellulose [23]; among these, EtC is an excellent substrate candidate for flexible electronics due to its unique characteristics of being waterproof, possessing low air permeability, and high transparency [21, 24]. EtC powder is soluble in organic solvents such as acetone and ethanol, and ultrathin films less than submicron scale thickness can be formed via simple spin-casting and drying of the EtC solution [25]. Fabricated ultrathin EtC films can easily be released from the substrate in water without the need of a surface treatment using toxic chemicals or a polymer-based sacrificial layer.

Therefore, utilization of EtC films for electronics has various advantages, such as a safe and facile fabrication process, as well as relying on an eco-friendly biomaterial.

In this study, we utilize an EtC film as a medium for the dry transfer method to fabricate patchable electrodes, thereby developing multifunctional patchable EtC-AgNW composite transparent electrodes (TEs). Ag NWs were coated on a glass substrate, followed by coating with EtC. After applying a functional tape, EtC-AgNW composites were released from the substrate via simply dipping them in water. It is worthy mention that it was not possible to successfully transfer AgNWs to the functional tape without EtC. The functional tape acted both as a supporter for the ultrathin EtC-AgNW composites and as a means to impart patchable characteristics to the composites. By simply changing the types of functional tapes, EtC-AgNW composites were attached to various surfaces, such as the human body, plastic and glass substrates, and the organic layer of solar cells. With the multifunctional patchable EtC-AgNW composite TEs, we successfully demonstrate their versatility as a patchable transparent interconnector, sensing electrode for electrophysiological sensing (electromyogram (EMG) and electrocardiogram (ECG)), and top electrode for a high-performance semitransparent perovskite solar cell (PSC).

2. Material and methods

2.1 Materials

AgNW solution (average diameter and length: ~25 nm × ~25 μm, solid content: 0.5 wt% dispersed in isopropyl alcohol (IPA), Flexio), EtC powder (Sigma Aldrich), transparent double-sided tape (thickness: 30 μm, Teratec), transparent tape (thickness: 40 μm, Teratec), and thermal release tape (Tapeworld) were used. An EtC solution (5 wt%) was prepared by dissolving ethyl cellulose powder in ethanol.

2.2 Fabrication of Patchable Flexible Transparent Interconnects

A schematic for the fabrication of multifunctional EtC-AgNW patchable TEs is shown in Figure 1 and Figure S5 of the Supporting information. The AgNW solution was spin-cast on the substrates at 1000 rpm for 30 s, followed by spin-casting the EtC solution at 500 rpm for 20 s and drying at 150 °C. Because we controlled the volume of AgNW suspension used for a cycle of coating to fabricate the AgNW electrodes a similar value of optical and electrical properties, there were no significant differences in the conducting properties of the fabricated sample depending on the sample size. To fabricate the flexible/patchable interconnects, transparent double-sided tape was attached manually on the EtC-covered AgNWs using a roller in a moderate press. By immersing the samples in a water bath, the EtC-AgNW/transparent double-sided tape was removed from the substrate and dried with an air blower. Fabrication of a patchable electrode in any design is possible by simply cutting the prepared sample with scissors (or an automatic cutter for precise size control). Because of the large surface to volume ratio, AgNWs tended to agglomerate as a ball shape at much lower temperature of ~180 °C compared to the melting temperature of Ag, ~962 °C as can be seen in Figure S6. Since the other materials used for the composites have a higher thermal resistance than AgNW, the highest temperature for the use of the composite electrodes is ~180 °C.

2.3 Electrophysiological Sensor

For application as an electrophysiological sensor, transparent one-sided tape was used as the functional tape. The EtC-AgNW/transparent one-sided tape patchable electrodes, connected to wireless electrophysiological sensing equipment (Biopac Systems), were attached to human skin with a few drops of water, and EMG and ECG signals were detected. The EMG signals were measured from AgNW patchable sensing electrodes attached to the

forearm of the lead author (Figure 4a), and the potential changes between the contraction and relaxation of the muscle were recorded. For the ECG signal, two patchable electrodes were attached on the chest according to the guidelines for Lead II electrode placement. Commercially available gel-type electrodes (Ludlow Technical Products Canada) were also used for the same electrophysiological sensing tests for comparison. For a normal subject, the ECG signals show periodic P, QRS complex, and T waves with constant intervals.

2.4 Perovskite Solar Cells

Thermal release tape was used as the functional tape to apply the patchable transparent top electrode to perovskite solar cells. The patterned ITO glass substrate was cleaned by sequential ultrasonication in acetone, IPA, and deionized water for 180 s at room temperature, and was then treated with UV-ozone for 15 min to tune the surface hydrophilicity for the water-based poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) coating. PEDOT:PSS (AI4083, Heraeus) was filtered through a membrane polytetrafluoroethylene (PTFE), 0.45 μm pore size), spin-coated (3500 rpm, 30 s) on the indium tin oxide (ITO) substrate, and baked at 130 °C for 20 min. The PEDOT:PSS-deposited ITO substrates were directly stored in an Ar-filled glovebox. A solution of PbI_2 (99.999%, 1.2 M in *N,N*-dimethylformamide/dimethyl sulfoxide (9:1 v/v), Alfa Aesar) and CH_3NH_3I (99.5%, 50 mg·mL^{-1} in IPA, Xian Polymer Light Technology) were spin-coated sequentially (3000 rpm, 30 s for both solutions) on the samples. The brownish perovskite-coated substrates were annealed at 100 °C for 2 h to crystallize the perovskites. PCBM ([6,6]-phenyl C61 butyric acid methyl ester) was dissolved in chlorobenzene (25 mg·mL^{-1}) and spin-coated (2000 rpm, 40 s). The PCBM-coated substrate was kept in a dry box overnight. Then, polyethyleneimine (PEI; 1 mg·mL^{-1} in IPA, Sigma Aldrich) was spin-coated (5000 rpm, 60 s). Finally, EtC-AgNW/thermal release tape was attached to the upper side of the samples using a hot-pressing method at 100 °C and 2.5 MPa for 30 s, and then further

annealed at 100 °C for 5 min. Photovoltaic *J-V* curves of the PSCs were measured with a 300 W xenon lamp-based solar simulator (Newport 91160A) and a Keithley 237 source measurement unit under AM 1.5G 1-sun illumination (100 mW·cm^{-2}). The active area of the PSC devices was 9 mm^2.

2.5 Characterization

A 4-point probe system (CMT-SR1000N) and UV-vis spectroscopy (Cary 5000) were used to measure sheet resistance and transmittance, respectively. The microstructure of AgNWs was investigated with scanning electron microscopy (SEM, NOVA 200 Nanolab) images and atomic force microscopy (AFM, XE-100) topography.

3. Results and Discussion

3.1 Fabrication of Multifunctional EtC-AgNW Patchable TEs

Figure 1 shows the EtC-AgNW fabrication process and potential applications as patchable TEs depending on the type of applied functional tapes. AgNWs were coated on the substrate (e.g., glass or silicon wafer), followed by EtC solution coating. During the drying of EtC, the AgNWs were embedded in the EtC films that formed the EtC-AgNW composites. Previously reported AgNW-polymer composites used a sacrificial layer or a substrate treatment requiring toxic chemicals, but such inconvenient and toxic steps for the fabrication of EtC-AgNW composites are unnecessary in our approach because EtC can be released from the substrate simply by dipping it in water (Figure S1). However, the EtC film floating on the surface of the water is ultrathin (of submicron scale thickness), making handling of the EtC film without a supporting layer difficult. Applying functional tapes to the EtC films solved this handling issue while also incorporating an attachable feature to EtC films. In addition, by changing the types of functional tapes, the EtC-AgNW composites can be used for various types of applications. For example, we used typical transparent tape, transparent double-sided

attachable tape, and thermal release tape as functional tapes for the composites, which were applied to the patchable electrodes for electrophysiological sensing, flexible transparent patchable interconnects, and top electrodes of optoelectronic devices, respectively (Figure 1).

To determine the effect of EtC on the optoelectronic performance of AgNW-based electrodes, the optical transmittance and sheet resistance change of AgNW only and EtC-AgNW composites on glass substrates as a function of the number of cycles of AgNW coatings were measured (Figure 2a). The optical transmittance of bare glass (0 cycle of AgNW coating) was ~100%, and the transmittance decreased from 93.8 to 68.3% as the number of AgNW coating cycles increased. The sheet resistance also decreased from 28.3 to 4.2 ohm/sq as the number of AgNW coating cycles increased. Increasing the number of coating cycles formed denser AgNWs, which resulted in lower transmittance due to more light scattering as well as lower sheet resistance due to formation of more conduction paths. However, there were no significant differences in optical and electrical properties of the bare AgNW electrodes on glass substrates and the EtC-AgNW composites. This confirmed that the thin and transparent EtC had no significant effect on the optical transmittance of the composite electrodes. Moreover, the similar sheet resistance values of the bare AgNW electrodes and EtC-AgNW composites showed that there was no significant loss of AgNWs during the fabrication process of the composite electrodes. Furthermore, the value of the figure of merit (FOM), defined as the quantitative performance of the TE film, is also calculated to figure out the optimized coating conditions of AgNWs for the best optoelectronic device performance (Figure 2b). For the network structure TEs, the FOM is defined as $T^{10} \cdot R_s^{-1}$, where T is the transmittance and R_s is the sheet resistance. For the EtC-AgNW composites, the samples fabricated with two cycles of AgNW coating showed the highest FOM value of ~2.3×10^{-2}; thus, all the devices demonstrated in this work used samples prepared with two cycles of AgNW coating. The FOM value was comparable to

previously reported TEs using AgNWs dispersed in cellulose matrix, for example, 3.99×10^{-2} for AgNWs in cellulose nanofibrils [26], or 1.22×10^{-2} for AgNWs in bacterial cellulose [23].

3.2 Application to Patchable Flexible Transparent Interconnects

The principal advantage of the fabricated patchable EtC-AgNW TEs is their multifunctionality through changing the functional tapes to suit a different purpose. For example, EtC-AgNW transferred to the transparent double-sided tape could be used for patchable transparent and flexible interconnects. Depending on the size of the substrate on which the AgNWs were coated, large-scale patchable EtC-AgNW TEs could be fabricated; in this case, 4″ Si wafer samples were fabricated (Figure S2). Once large patchable TEs were fabricated, we could fashion a patchable electrode in any shape by cutting with scissors (or a cutting machine for better precision), as shown in Figure 3a. The EtC-AgNW TEs, trimmed in the desired design, could be attached to any type of substrate, including polyethylene terephthalate (PET) film (25 μm), copier paper (100 μm), a paper cup, or human skin, and functioned as interconnects even under harsh deformable conditions (Figure 3b–e). In addition, the patchable EtC-AgNW TEs were mechanically reliable and showed no significant change in resistance over a variable bending radius of 1.3−12.5 mm (Figure 3f), or ~0.8% and ~2.3% increase after 300,000 cycles of bending with a radius of 5 mm (Figure 3g) on the PET and paper substrates, respectively. The PET substrate was thinner than paper substrate, which resulted in the better mechanical reliability. The decrease in resistance in the initial stage of bending was due to the newly formed junctions by mechanical welding [27].

3.3 Application as Sensing Electrodes in an Electrophysiological Sensor

The patchable EtC-AgNW TEs could also be used as the sensing electrodes of an electrophysiological sensor. In this case, the conventional one-sided transparent tape was used as the functional tape (Figure S3). The patchable transparent tape-supported EtC-AgNW TEs were connected to an electrophysiological sensing system, as well as to the forearm and chest of the human subject, to measure electrophysiological EMG and ECG signals, respectively (Figure 4a and Figure 5e). While gel-type electrodes are disposable because they lose their adhesiveness after a few tests, patchable EtC-AgNW TEs can be used repetitively by simply applying additional transparent tape. Figure 4b shows the EMG signals measured by the EtC-AgNW TE attached to a forearm. The electrical potential change generated by the activation of muscle movement was clearly characterized, showing a potential change during contraction of the muscle and steady-state potential while relaxed.

For the ECG signals, EtC-AgNW TEs were attached on the chest according to the Lead II electrode placement guidelines (Figure 5e). The ECG signals, which consist of a P wave, QRS complex wave, and T wave, were in the normal range with constant intervals, exhibiting periodic results during the duration of the test (Figure 5a and c). In addition, the EMG and ECG signals delivered results similar to those measured with commercially available gel-type electrodes, as shown in Figure 4c and Figure 5b, thus confirming the potential of patchable EtC-AgNW TEs as electrophysiological sensors. Transparent electrophysiological electrodes could help optically examine skin conditions during measurements for emerging digital healthcare applications.

3.4 Applications to top electrodes of PSCs

Multifunctional patchable EtC-AgNW TEs also enabled the fabrication of semitransparent PSCs. The EtC-AgNW TEs developed for these purposes employed thermal

release tape as the functional tape (Figure S4). During hot pressing, the thermal release tape lost its adhesiveness and then the EtC-AgNW TE was successfully transferred to the PSC layer to function as the top electrode. PSCs with the structure illustrated in Figure 6a were fabricated. The PSCs comprised stacks of ITO/PEDOT:PSS/$CH_3NH_3PbI_3$/PCBM/PEI/EtC-AgNW, with all the layers formed by solution processes except for the ITO layer. Figure 6b displays the transmittance values of the PSCs before and after the formation of the top electrodes using the EtC-AgNW TEs. The PSC layers with and without the AgNW top electrodes were semitransparent, showing relatively low average transmittance values of ~16.4% and ~16.1%, respectively. Light passing through the EtC-AgNW TE was absorbed in the PSC layers; thus, the transmittance change by the transparent AgNW top electrode was negligible.

The electrical characterization of semitransparent PSC performance is shown in Figure 6c. The current density and voltage curves (*J-V* curve) of the bottom and top electrodes display similar trends because the bottom and top electrodes have similar transmittance (the transmittance of the ITO bottom TE and EtC-AgNW top TE are 89.4% and 87.2% at 550 nm, respectively.) The short circuit current densities (J_{SC}), open circuit voltages (V_{OC}), and fill factors (FF) are shown in Table I, and the power conversion efficiency (PCE) is 6.74% and 7.03% for bottom and top sides, respectively. Compared to single-sided PSCs with evaporated top Ag film (100 nm), the PCE of this semitransparent PSC is lower than that of the single-sided PSC (9.24%). However, the total PCE is higher (Table I), which indicates that the patched EtC-AgNW is bound strongly with the underlayer (PCBM/PEI) and is able to function as a transparent top electrode for optoelectronics.

4. Conclusions

In this study, EtC-AgNW composite-based multifunctional patchable and flexible TEs were developed and applied to various electronic devices, including patchable and flexible interconnects, electrophysiological sensors, and PSCs. AgNWs and EtC were sequentially coated on a glass substrate, followed by the application of functional tape. The EtC-AgNW composites were then released from the glass substrate by dipping them in water. The use of EtC showed no significant change in optical and electrical properties of the AgNW electrodes. In addition, the EtC layer was separated from the glass substrate without the need for a toxic surface treatment. Thus, the use of EtC is highly beneficial for the safe and convenient production of composite electrodes. Furthermore, EtC-AgNW composites can be modified by simply using a different functional tape for different applications. For example, EtC-AgNW composites using transparent double-sided tape as the functional tape were applied as flexible and patchable interconnects. These were fabricated on a large scale (4″ wafer), attached to various substrates such as PET film (25 μm), copier paper (100 μm), a paper cup, or human skin, and functioned as interconnects even under harsh deformation conditions. EtC-AgNW composites with conventional one-sided transparent tape were used for the electrophysiological sensing of EMG and ECG signals, exhibiting stable detection performance that was similar to that obtained from commercially available gel-type electrodes. We also demonstrated semitransparent PSCs with EtC-AgNW composite top electrodes by using thermal release tape as the functional tape. With the highly transparent and conductive EtC-AgNW composite top electrodes, PSCs showed excellent total power conversion efficiency of 13.77%.

Notes

The authors declare no competing financial interest.

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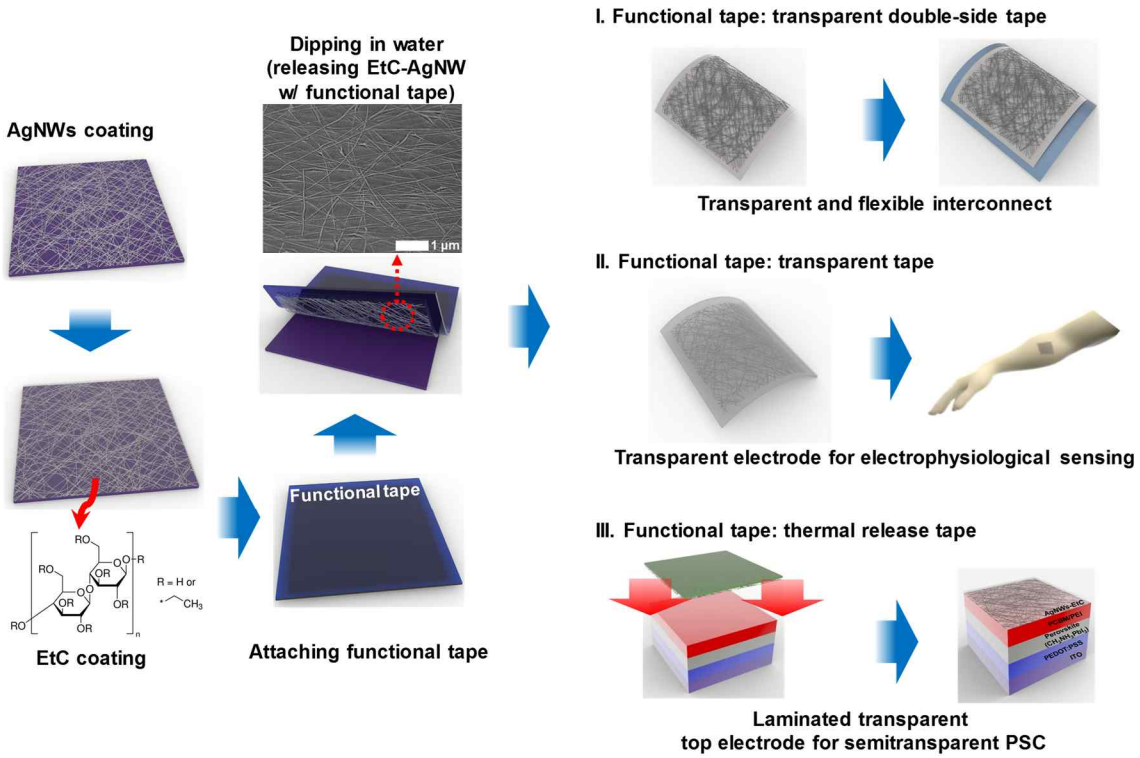

Figure 1. Schematic illustrations of the fabrication process of EtC-AgNW composite TEs and the various applications depending on the type of functional tape used. SEM images show the surface of AgNWs covered with EtC film.

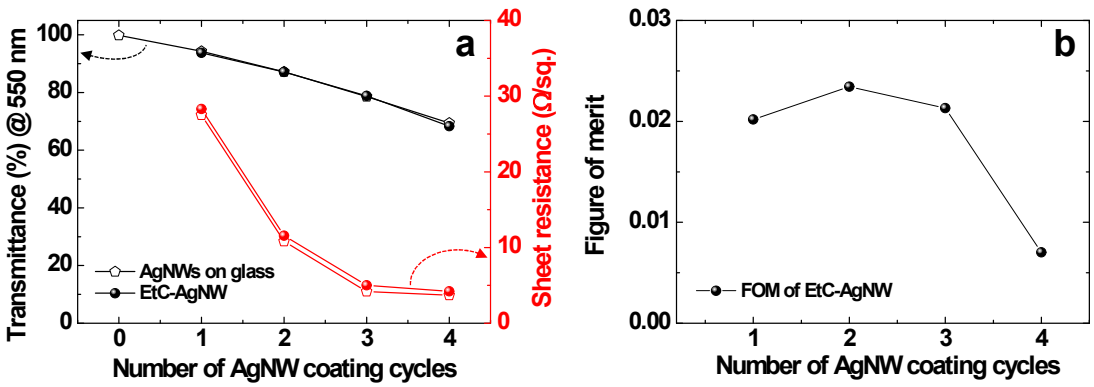

Figure 2. a) Transmittance at 550 nm and sheet resistance of AgNWs on glass substrates and EtC-AgNW composites and b) Figure of merit of EtC-AgNW composites as a function of the number of AgNW coating cycles (1–4).

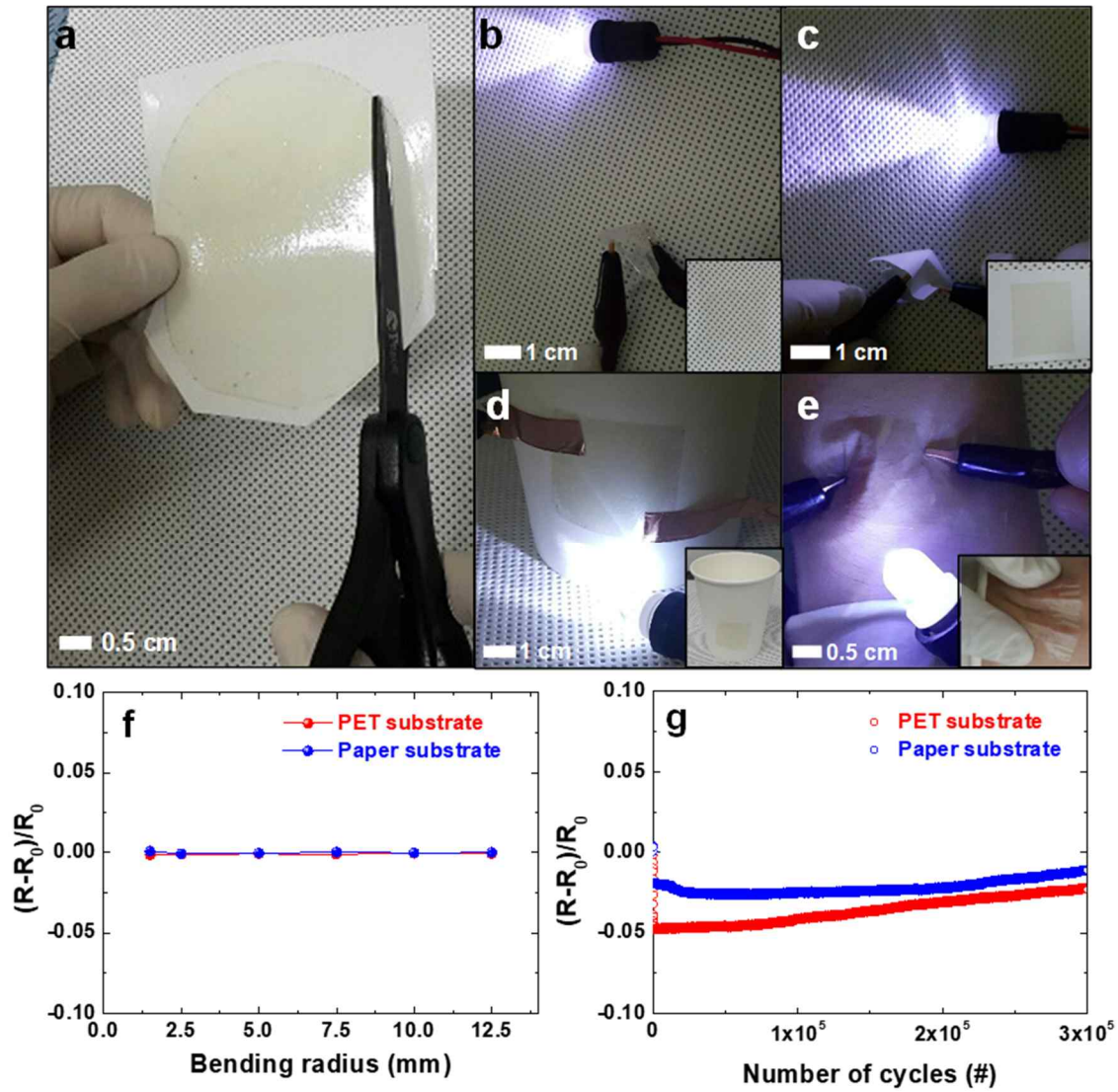

Figure 3. Application of the EtC-AgNW TEs to transparent and flexible interconnects. a) Patchable interconnects with the desired designs were made by cutting large-scale EtC-AgNW TEs with scissors. Operation of LEDs on different substrates: b) PET film, c) copier paper, d) paper cup, and e) human skin. The inset photos show the attached TEs on the different substrates without LEDs. Changes in the normalized resistance of the EtC-AgNW TEs on the PET and copier paper substrates as a function of f) the bending radius and g) number of bending cycles at a radius of 5 mm.

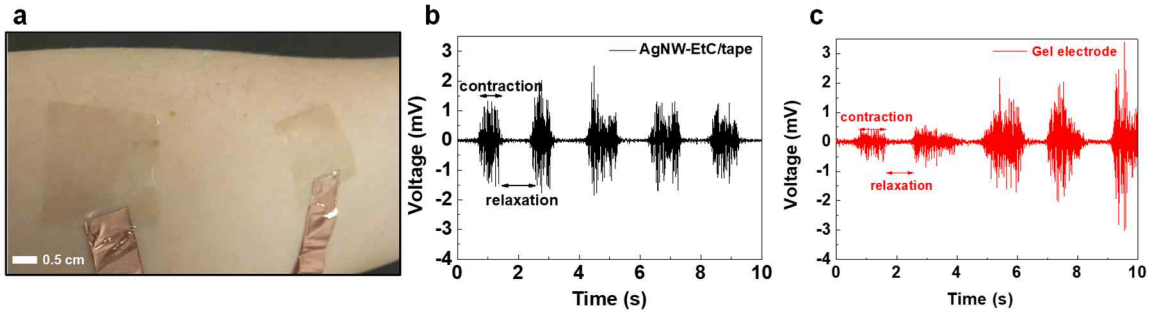

Figure 4. Application of the EtC-AgNW TEs to electrophysiological sensing. a) Photos of the EtC-AgNW/transparent one-sided tape attached on the forearm of the lead author for EMG signal sensing. EMG signals detected using b) patchable EtC-AgNW TE sensing electrodes and c) gel electrodes.

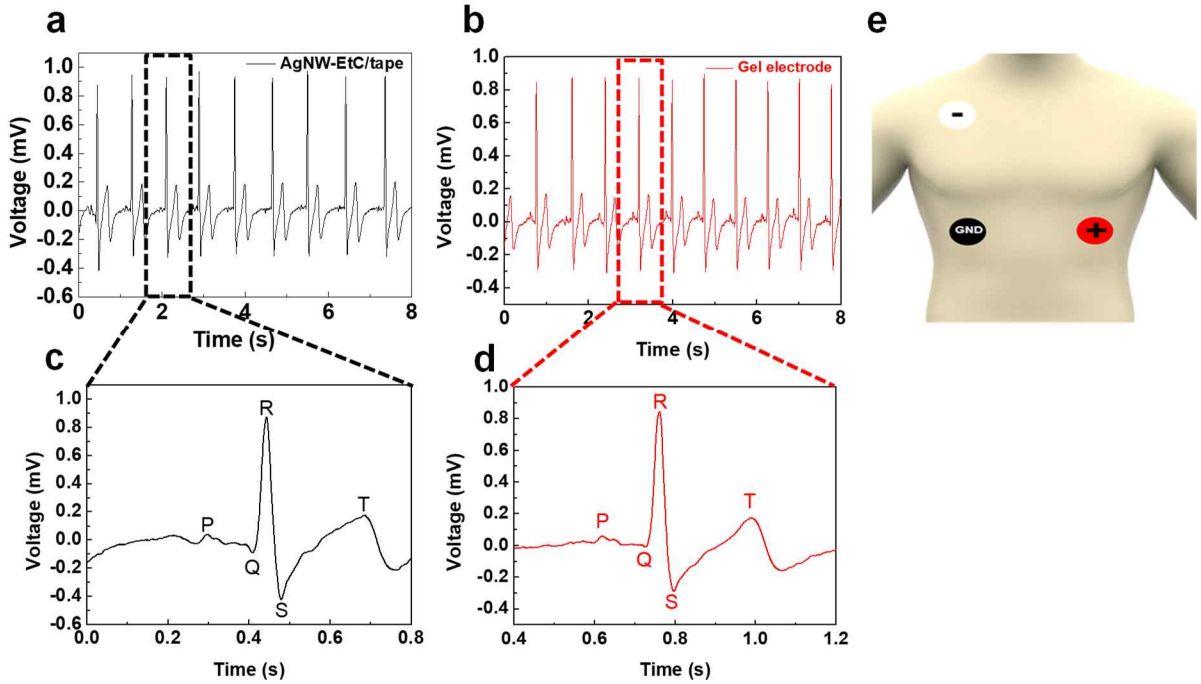

Figure 5. ECG signals detected using a) EtC-AgNW TEs sensing electrodes and b) gel electrodes; c) and d) single-interval ECG signal enlarged from the dotted squares in a) and b), respectively; e) schematic of the positions of the three electrodes according to Lead II electrode placement guidelines for ECG signal detection.

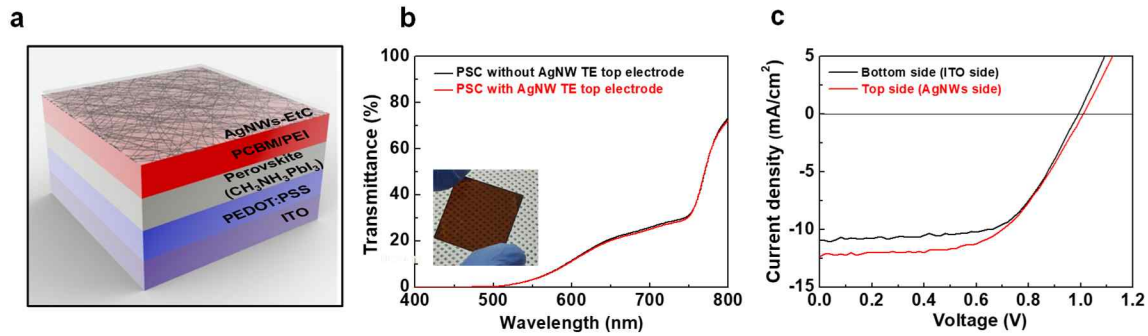

Figure 6. a) Schematic structure of PSC using the EtC-AgNW TE as the top electrode. b) UV-vis transmittance spectra of PSCs with and without the EtC-AgNW TE top electrode. The inset is a photo of the PSC with the EtC-AgNW TE. (c) *J-V* curves of the PSC illuminated through the bottom and top electrodes.

Table 1. PSC performance of the semitransparent device (EtC-AgNW composite top electrode) and single-sided device (opaque Ag film top electrode).

Illumination side	J_{SC} (mA cm^{-2})	V_{OC} (V)	FF (%)	PCE (%)
Bottom side	10.8	0.99	62.58	6.74
Top side	12.3	1.00	56.55	7.03
Bottom side (evaporated Ag film)	16.0	0.90	63.8	9.24