Room-Temperature Nanosoldering of a Very Long Metal Nanowire Network by Conducting-Polymer-Assisted Joining for a Flexible Touch-Panel Application

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As an alternative to the brittle and expensive indium tin oxide (ITO) transparent conductor, a very simple, room-temperature nanosoldering method of Ag nanowire percolation network is developed with conducting polymer to demonstrate highly flexible and even stretchable transparent conductors. The drying conducting polymer on Ag nanowire percolation network is used as a nanosoldering material inducing strong capillary-force-assisted stiction of the nanowires to other nanowires or to the substrate to enhance the electrical conductivity, mechanical stability, and adhesion to the substrate of the nanowire percolation network without the conventional high-temperature annealing step. Highly bendable Ag nanowire/conducting polymer hybrid films with low sheet resistance and high transmittance are demonstrated on a plastic substrate. The fabricated flexible transparent electrode maintains its conductivity over 20 000 cyclic bends and 5 to 10% stretching. Finally, a large area (A4-size) transparent conductor and a flexible touch panel on a non-flat surface are fabricated to demonstrate the possibility of cost-effective mass production as well as the applicability to the unconventional arbitrary soft surfaces. These results suggest that this is an important step toward producing intelligent and multifunctional soft electric devices as friendly human/electronics interface, and it may ultimately contribute to the applications in wearable computers.

1. Introduction

As an example of modern technology, transparent conductors have been regarded as an extremely important component in optoelectronics applications such as solar cells, organic light-emitting diodes (OLEDs) displays, and touch panels.[1–4] The most common material for transparent conductors is indium tin oxide (ITO), which has become a market leader due to its high transparency in thin film. However, its brittle ceramic properties and expensive vacuum deposition process are limitations to its further progress.[5] Moreover, the growing costs and dwindling reserves of ITO are also regarded as unsolvable problems. Therefore, several other candidates, including carbon nanotube (CNT),[6,7] graphene, and metal nanostructures have been studied as alternatives to ITO[8] especially as flexible and solution processible transparent conductors. Although many researchers have focused on unconventional nanostructure materials, each material has evident limitations as a future substitute. For instance, carbon-based materials require an expensive vacuum environment and a toxic chemical process to enhance performance. Conducting polymers have good compatibility for cost-effective solution processible mass production such as roll-to-roll process or spray deposition, but its poor conductivity should be enhanced before it can be applied to commercial products.[5,9] Although 1D metallic nanowires have emerged as a best candidate for ITO substitute with a sheet resistance at a low level of tens of ohms per square,[10] a high-temperature thermal treatment should be conducted to reduce the contact resistance at the nanogap junctions between the nanowires. Moreover, the net resistance in a nanowire network is considerably influenced by the length of the metallic nanowires with a percolation threshold.[11–13]

The shortcomings from relying only on a single component could have been overcome by applying hybrid composite materials that can compensate the disadvantages while maintaining the advantages of individual components. For example, composite of metallic nanowires, graphene,[14] CNT, and conducting

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polymer[15] may produce high performance transparent conductors as ITO substitutes. For example, silver nanowire (AgNW) could be placed on a graphene layer as an electrical bridge between grain boundaries.[16] Although a single graphene layer has resistivity as low as 10^{-6} ohm cm,[17] in many studies, numerous grain boundaries increase the resistance, making it difficult to create a single graphene layer on a large scale.[18] On the other hand, a graphene layer was assembled on a metallic nano structure as a 2D conductive cover.[17] Electrons can be transported through the covered graphene, even in the empty space between metal nanostructures. To tighten the nanowire junction, metallic or metal oxide nanoparticles have been assembled via capillary force.[12]

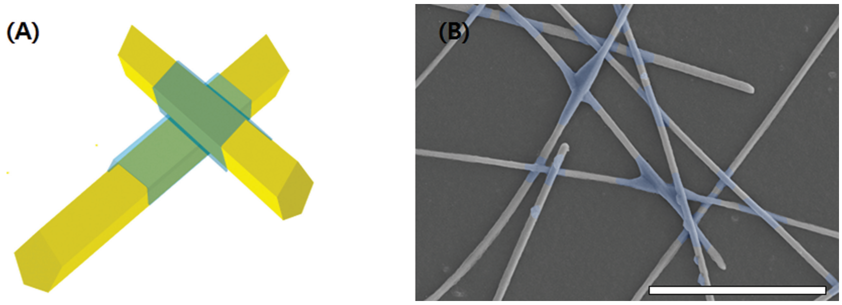

Figure 1. Illustratation and SEM image of room temperature AgNW nanosoldering by PEDOT:PSS assisted joining at the nanowire junctions. A) Schematics of the pentagonal AgNW (yellow colored) juntion with nanosoldering (blue colored) by PEDOT:PSS coating (blue). B) SEM image of AgNWs joined by PEDOT:PSS (red colored) thourgh nanosoldering. The inset scale bar is 4 μm.

Here, we developed a novel hybrid approach using AgNW-conducting polymer composite to demonstrate a high performance, very large area transparent conductor by a simple, room temperature, solution processible nanosoldering by a rod coating. Many studies have shown that AgNWs can serve as a good replacement for ITO because silver is the most conductive material on earth due to its high free-electron density. Silver can also be highly transparent at a very small dimension while maintaining good electrical conductivity.[19–22] However, the need for a high temperature thermal annealing step to reduce the contact resistance and its poor adhesion to the substrate was one of the biggest problems for the AgNW transparent conductors.[23] We demonstrated that using a AgNW/poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate)(PEDOT:PSS) hybrid composite, contact resistance and substrate adhesion problem could be significantly enhanced even without a high temperature annealing step. Through the strong adhesion enhancement and the flexible nature of the AgNW and conducting polymer, a flexible transparent electrode on a flexible polymer substrate could be demonstrated. Furthermore, PEDOT:PSS assisted tight joining at the nanowire junction during the conducting polymer drying makes this process possible at room temperature. Typical AgNW network electrodes should be annealed at high temperature (over 200 °C, for several hours) to create a strong inter-nanowire junction. A thermal annealing is a necessary step to create a low resistance electrode in previous AgNW transparent conductor researches. Therefore, plastic substrate could not be used because the substrate also needs to be heated and may be thermally damaged. To reduce the production costs and create a flexible and mechanically strong transparent conductor electrode, a glass substrate should be replaced with a flexible plastic substrate which will experience thermal degradation problem. The proposed PEDOT:PSS assisted room temperature nanosoldering of AgNW mesh electrode can remove high temperature annealing steps while having similar high conductivity. Moreover, the developed simple fabrication process could be easily expanded to demonstrate a very simple, large-scale roll-to-roll process. In this experiment, glass and PEN (polyethylene naphthalate) substrates up to A4 (210 mm × 279 mm) size were used to create a large transparent electrode. Finally, we demonstrated high performance touch panels of various sizes on various substrates with the fabricated AgNW/PEDOT:PSS hybrid composite electrode. Strong adhesion could maintain good functionality with long term stability in devices under various external force.

2. Result and Discussion

As shown in **Figure 1**A, The neighboring AgNWs were strongly joined by PEDOT:PSS coating at the nanowire junction. Two types of AgNWs (regular and very long AgNWs) in this experiment were synthesized via a modified polyol process as described in the experimental section. Isopropyl alcohol (IPA) was added to the cleaned AgNWs to make nanowire dispersion. Then, firstly, pristine AgNW mesh network was formed on the substrate by Mayer rod coating method. In a similar manner, PEDOT:PSS mixed in IPA was coated onto the previously prepared pristine AgNW mesh network film. The major challenge came from the peeling-off of the pre-deposited AgNWs during the PEDOT:PSS coating. Pre-deposited pristine AgNWs mesh film can be easily destroyed or swept away during the subsequent PEDOT:PSS coating by the strong surface tension of PEDOT:PSS solution because pristine AgNWs are gently sitting on the substrate by weak adhesion force with line or point contacts. Hence, to prevent the peel-off problem, the surface tension of PEDOT:PSS solution and the solvent evaporation rate was adjusted by adding IPA. The IPA concentration in the PEDOT:PSS was optimized to maintain the spreading properties and to good dispersion of the AgNW in PEDOT:PSS solution. After complete drying, PEDOT:PSS solution acted as soldering materials and was accumulated at the nanowire junction to form droplets and filled the gap between nanowires through the strong capillary force during the solvent evaporation. That capillary force assisted nanosoldering by PEDOT:PSS at the nanowire junctions is clearly shown in Figure 1B in a scanning electron microscopy (SEM) image (red colored). PEDOT:PSS nanosoldering could greatly enhance the electrical conductivity and decrease contact resistance of AgNW percolation networks at the nanowire junction by increasing the contact surface area and tightly binding the nanowires via capillary force induced stiction of the conducting polymer solution. As

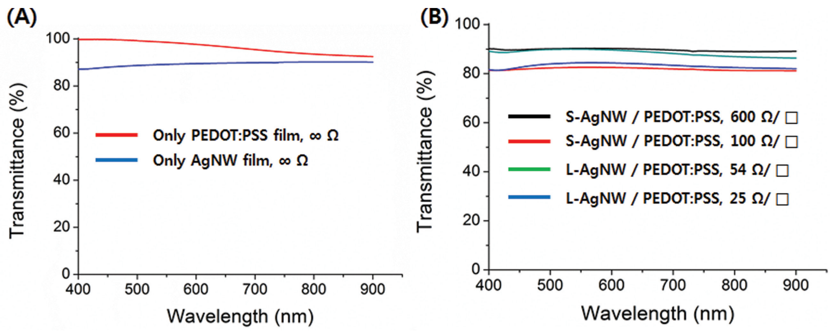

Figure 2. Optical characteristics of transparent conductors. A) Optical transmittance spectra of single component transparent film of only PEDOT:PSS (red) and only AgNW percolation network electrode (blue). B) Optical transmittance spectra of hybrid composite transparent film; short AgNW/PEDOT:PSS hybrid film (black and red) and very long AgNW/PEDOT:PSS hybrid film (green and blue). Note the dramatic sheet resistance drop from ∞ to tens of ohm/sq after nanosoldering of AgNW by PEDOT:PSS coating.

a result, uniform and stable hybrid films were created by the Mayer rod coating method and the resistance and transparency were easily controlled by adjusting the number of layers. Previously, the sheet resistance of pristine AgNW film without thermal annealing was found to be rather high when a capping agent was used on the surface of AgNWs, leading to weak contact.[5] Therefore, a high temperature thermal treatment has been considered as an obligatory process to make such an electrode useful. However, high temperature annealing process may cause the deforming or degradation of the plastic substrates such as polyethylene terephthalate (PET) and PEN films due to their low glass transition temperatures. By applying the PEDOT:PSS as a nanosoldering material for AgNW network electrode, a low-temperature process could be achieved with only a simple coating method and applying plastic substrate was also possible.

The transmission spectra of the single component film (only AgNW network film or only PEDOT:PSS film) is shown **Figure 2**A. For the fabricated only AgNW films (blue line), the transmittance is over 90% (at 550 nm wavelength) but the sheet resistance was very high because thermal annealing process was not performed. Similarly, the only PEDOT:PSS film (red line) fabricated by means of the Mayer rod coating method also shows very high resistance due to its intrinsic poor conductivity and small thickness (10 nm). However, the composite of AgNW and PEDOT:PSS of the same condition with the single component films showed dramatic changes. After the PEDOT:PSS coating on the AgNW network mesh, although the transmittance of the AgNW/PEDOT:PSS hybrid films was slightly reduced, the sheet resistance of the hybrid films after nanosoldering showed a several orders of magnitude decrease as compared to that of the single-component film (only AgNW network film or only PEDOT:PSS film). As shown in Figure 2B, the hybrid composite film of AgNW/PEDOT:PSS showed sheet resistance of few hundreds of ohms/sq for short AgNW (S-AgNW, black and red lines) with 80–90% transmittance. For a comparison, each two electrodes (S-AgNWs, L-AgNWs) with different sheet resistance were fabricated by changing the AgNW deposition thickness. The difference in AgNW thickness caused the change in the sheet resistance and transmittance for the same length AgNWs in Figure 2B. The sheet resistance and transmittance can be further enhanced by using longer AgNWs for the same thickness AgNW transparent conductor. The hybrid composite film of long AgNW (L-AgNW)/PEDOT:PSS showed sheet resistance of few tens of ohms/sq (green and blue lines) with 80–90% transmittance. From the percolation threshold theory, the length of a 1D object determines the random network conductivity.[24,25] For instance, a transparent conductive film created via scalable rod coating with short AgNWs and a hydrogen chloride (HCl) vapor treatment achieved over 100 ohm/sq with 75% transmittance.[26] In this experiment, we changed the length of the AgNWs and explored the nanowire length effect on the electrode resistance. Regular short AgNW (S-AgNW) is mainly 20–30 μm in length, while that of long AgNW (L-AgNW) ranges in 50–100 μm. The sheet resistance of AgNW/PEDOT:PSS hybrid films is decreased considerably from several hundreds of ohm/sq to 25 ohm/sq with 85% transmittance (at 550 nm) by increasing the length of the AgNWs. Although PEDOT:PSS thin films have transparency as high as 95%, the transmittance value of the hybrid (AgNW/PEDOT:PSS) films is a little bit lower because increase surface area by AgNW mesh might accommodate more amount of PEDOT:PSS than bare glass substrate.

SEM images (**Figure 3**) clearly show the difference between a pristine AgNW film (before PEDOT:PSS coating) and a hybrid (AgNW/PEDOT:PSS) film on a glass substrate. In the case of the pristine AgNW film, each AgNWs gently sit on the neighboring nanowires or substrate with very small area point contact or line contact. Therefore, as observed in Figure 3A, many nanowires were suspended without direct contact with the substrate and this caused very poor adhesion of the pristine AgNW mesh to the substrate or to other nanowires when the high temperature thermal annealing process was not applied. Therefore, a pristine AgNW film has very poor mechanical stability under mechanical stress and very high sheet resistance. As shown in Figure 3B, PEDOT:PSS was uniformly coated onto AgNW mesh and substrates. Even though there were many complicated junctions, all of the nanowires showed direct and conformal contact with the substrate due to strong capillary force

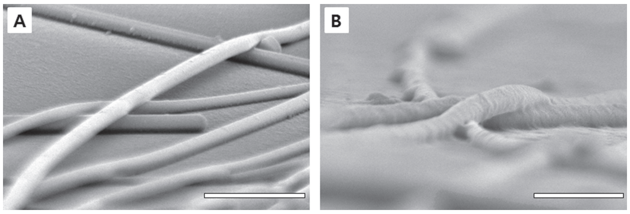

Figure 3. SEM images of A) fabricated pristine AgNW network before nanosoldering and B) AgNW/PEDOT:PSS hybrid film after nanosoldering. The drying PEDOT:PSS induces strong capillary force and stiction of the AgNW with other AgNWs or substrate. After nanosoldering, AgNWs strongly adhere to the substrate and join the junction of NWs. The inset scale bar s are A) 1 μm and B) 500 nm.

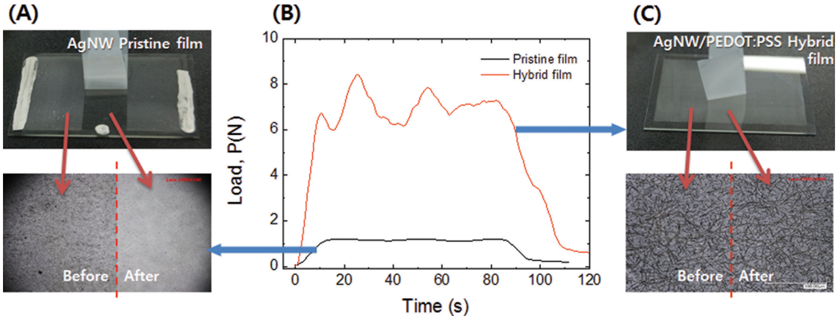

Figure 4. Adhesion force test (3M tape test) of AgNW electrodes of A) pristine AgNW network before nanosoldering and C) AgNW/PEDOT:PSS hybrid film after nanosoldering. B) Transient adhesion force test via 90° peel of test with linear stage. Note that the pristine AgNW film without nanosoldering was easily removed from the substrate as shown in microscope image. However, nanosoldered AgNW/PEDOT:PSS hybrid film demonstrated 7 times stronger adhesion force than pristine AgNW film and maintained initial transparent percolation network.

induced stiction during PEDOT:PSS drying and thus yielded enhanced substrate adhesion. Although PEDOT:PSS has high resistance, as shown in Figure 3, the PEDOT:PSS tightly binds the crossing nanowires and serves as an electrical bridge or nanosoldering material at the nanowire junction network. The post-deposition PEDOT:PSS was uniformly done, with a thickness of about 10 nm (Supporting Information Figure S1). Previously, many approaches reported a post-process without high-temperature annealing to enhance the conductivity of AgNW mesh, such as Au particles grown on a cross-section, mechanical cold pressing, and graphene covering, as noted above. However, a large-scale, all-solution process has not been reported so far, and we suggested a roll-to-roll applicable all-solution process by the conducting polymer assisted AgNW nanosoldering. After this simple process development, besides the electrical conductivity enhancement, it was clearly shown that the adhesion between the substrate and the thin film was significantly increased as shown in **Figure 4**. While pristine AgNW network film could be easily removed from the substrate during the 3M tape testing (Figure 4A), after the PEDOT:PSS treatment, the AgNW/PEDOT:PSS hybrid film showed very strong adhesion to the substrate while maintaining its original conductivity even after tape testing (Figure 4C, Supporting Information Figure S3). This strong and stable substrate adhesion might come from the film uniformity after the filling the gap between nanowires network with conducting polymer.

The peel-off test was conducted to measure the adhesion strength of pristine and hybrid films quantitatively. As shown in Figure 4B, the hybrid films can sustain six times larger force than the pristine film. Moreover, no visible difference in the hybrid film was observed before and after the peel-off test. Particularly, peeling off the film is detrimental in the presence of an external force or a mechanically harsh environment, such as bending, stretching and touching, even if the film has high conductivity as a result of thermal annealing.

While ITO is a very fragile material, AgNWs are very flexible. However, high temperature annealing makes AgNWs not directly applicable to the flexible polymer substrate. Due to the room temperature process nature of the proposed nanosoldered of AgNW/PEDO:PSS hybrid film, flexible transparent conductor could be realized. To ensure the mechanical stability of the AgNW/PEDOT:PSS hybrid film, the substrate was changed from glass substrate to PEN film for a cyclic bending test. As shown in **Figure** 5A, 20 000 cyclic bending test was conducted at a frequency of 1 Hz and a bending ratio of 1 cm to explore the stability of the electrical conductivity. The resistance value was maintained at a constant value after the initial decrease during the cyclic bending test. This initial resistance drop may have been caused by electrical joule heating and annealing during the long time test. Furthermore, the conductivity was sustained even during a stretching test at 5–10% strain (Figure 5B). Unlike fragile glass substrate, a polymer substrate increases a device's lifetime due to high flexibility and strength under external force or an impact. In addition, the low price of a polymer substrate is another advantage for industry.

To validate the practical application of current technology, fabricated AgNW/PEDOT:PSS hybrid transparent electrodes were applied to a touch panel screen demonstration. Compared with the commercial ITO-coated PEN film (**Figure** 6A) which requires an expensive vacuum process, in this experiment, all electrodes was fabricated by solution processes (Figure 6B). A high performance small touch screen (7.5 cm × 5 cm) was first demonstrated in Figure 6C. Both sides of the fabricated panel were AgNW/PEDOT:PSS hybrid film. Moreover, the current technology can be easily scaled up for the larger device demonstration due to the solution processible nature of current process. Finally, an A4-sized (250 mm × 275 mm) hybrid film was fabricated for large-scale applications, as shown in **Figure** 7. Pristine AgNW network film (Figure 7B, right) was coated with the PEDOT:PSS solution (Figure 7B, left). In Figure 7C, the functionality of the A4-size touch panel was demonstrated, writing the characters “ANTS”. However, the touch panel size can be easily scaled up for

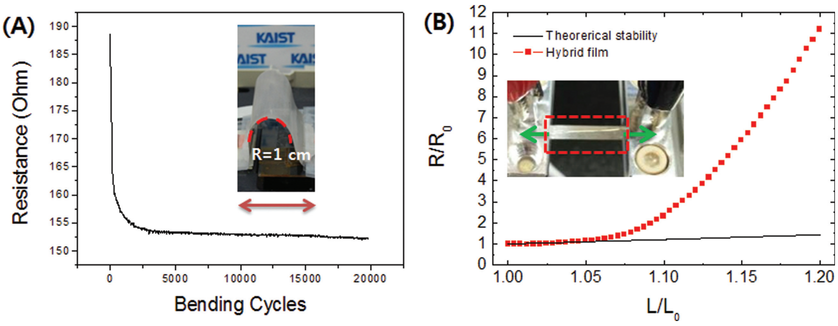

Figure 5. A) Cyclic bending and B) stretching test of nanosoldered hybrid film on a flexible substrate and the electrical property change. A) After 20 000 cycles of bending, nanosoldered hybrid film maintained low resistance. Initial decrease came from the electrical annealing. B) Hybrid film maintained its electrical conductivity over 10% stretching.

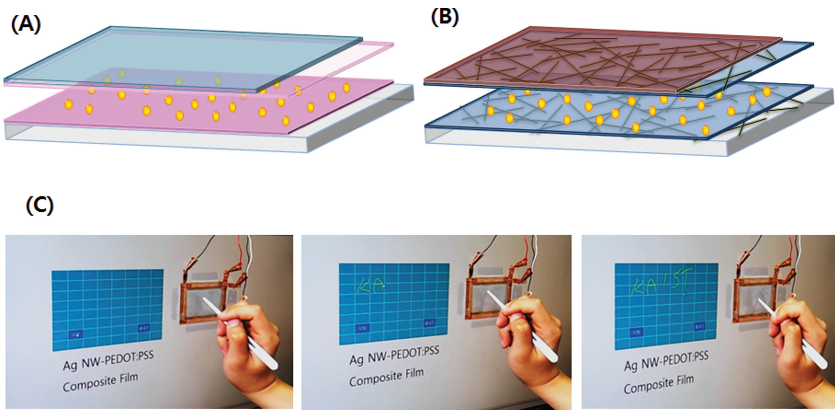

Figure 6. High performance touch panel demonstrated by nanosoldered AgNW/PEDOT:PSS transparent conductor. Schematic illustration of A) commercial touch panel screen with PEN/ITO/spacer/ITO/glass from top to bottom and B) fabricated touch screen with hybrid transparent films. C) Video clips of the touch screen operation fabricated by all solution processed AgNW/PEDOT:PSS hybrid film. A word "KAIST" was written on the touch panel.

much larger size by using larger Mayer rods and even for roll-to-roll process. Video clips of each touch panel demonstration are included in the Supporting Information. Therefore, if we use a more specific and controlled fabrication device, uniform and large-scale electrodes can be formulated by a simple and economical process.

The low temperature process nature could allow building highly flexible transparent conductor which can be mounted on the non-flat curved surfaces. To demonstrate the mechanical flexibility of the fabricated transparent electrodes, a bendable touch screen was made and its performance was demonstrated as shown in **Figure 8**. The bending radius was 3 cm and the substrates were 75 mm × 50 mm in size. The letters "ANTS" were written on the touch screen, which was placed on a cylinder. The flexible touch screen can be mounted even on the soft surface such as human arm (Supporting Information Figure S2). In this manner, our AgNW/PEDOT:PSS hybrid electrode developed in this work enables transparent conductors to be mounted at any locations where to date we have been unable to provide electrical and mechanical functionalities. This may open a new application field such as human friendly electronics in wearable computer.

3. Conclusions

In summary, as an alternative to the brittle and expensive ITO transparent conductor, we developed a very simple, room temperature nanosoldering of Ag nanowire percolation network with conducting polymer to demonstrate highly flexible and even stretchable transparent conductors. The drying conducting polymer on Ag nanowire percolation network is used as a nanosoldering material inducing strong capillary force assisted stiction of the nanowires to other nanowires or to the substrate to enhance the electrical conductivity, mechanical stability and adhesion to the substrate of the nanowire percolation network without conventional high temperature annealing step. Due to the room temperature nature of current process and very long Ag nanowire, highly bendable Ag nanowire/conducting polymer hybrid films with low sheet resistance (25 to 54 ohm/sq) with a high transmittance (90% at 550 nm) were demonstrated on a plastic substrate. Unlike the highly brittle

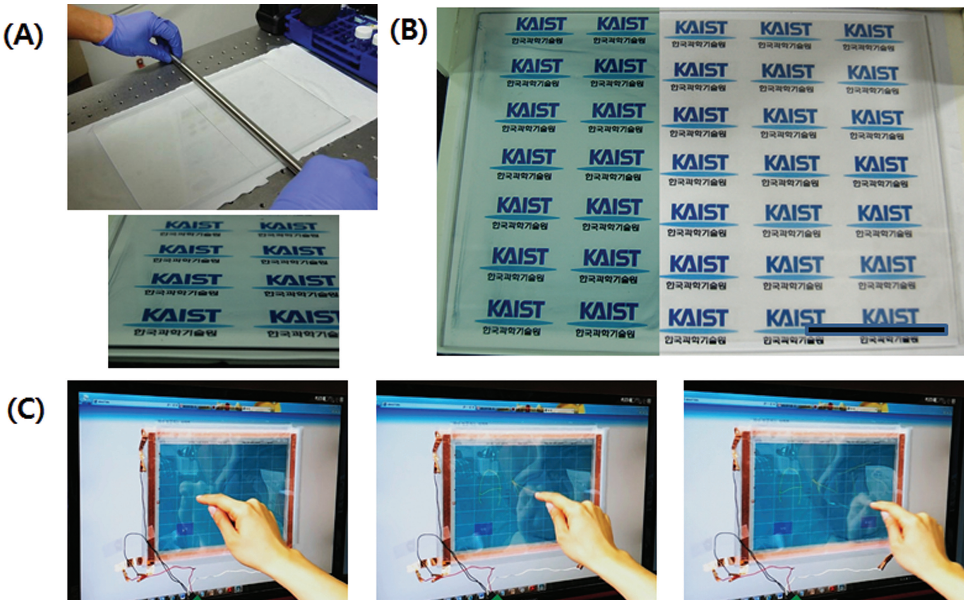

Figure 7. Demonstration of large scale hybrid transparent electrodes and a touch screen. A) Initial A4-sized electrode fabrication with the Mayer rod. B) Digital images of nanosoldered AgNW/PEDOT:PSS hybrid transparent conductor (left) and pristine AgNW network before nanosoldering (right). C) Video clips of the A4-sized touch panel operation fabricated from the nanosoldered hybrid transparent conducting film. Letters of "ANTS" were written.

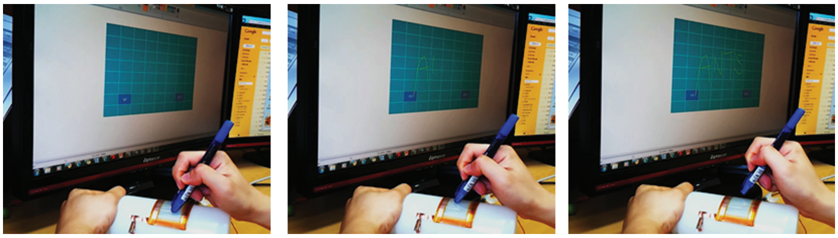

Figure 8. Demonstration of a bendable touch screen on a non-flat surface. Letters "ANTS" were written.

and fragile ITO transparent conductor, the fabricated flexible transparent electrode maintained its conductivity over 20 000 cyclic bending and 5 to 10% stretching. Finally, a large area (A4-size) transparent conductor and a flexible touch panel on a non-flat surface were fabricated to demonstrate the possibility of cost-effective mass production as well as the applicability to the unconventional arbitrary soft surfaces. We believe that our research suggests an important step toward producing intelligent and multifunctional soft electric devices as friendly human/electronics interface, and ultimately contribute to the applications in wearable computers.

4. Experimental Section

Ag NW Synthesis: AgNWs were synthesized by a modified polyol method through a CuCl$_2$(Sigma Aldrich) mediated process.[4,27,28] A Cu-additive solution was added to 5 ml of EG in an oil bath at 151.5 °C. After 5–10 min, 1.5 mL of 147 mM PVP (Sigma Aldrich) solution and 1.5 mL of a 94 mL AgNO$_3$ (Sigma Aldrich) solution were added sequentially. Heating and stirring was maintained for 1 h. The synthesized AgNW was cleaned with acetone and ethanol at a 10:1 v/v ratio three times for characterization.

Very Long Ag NW Synthesis: The first synthesized AgNW was heated at 95 °C for successive multistep growth (SMG) as seed NWs to grow very long AgNWs. 5 mL of EG was heated at 151.5 °C with contitnious magnetic stirring for second step growth. 1 mL of synthesized AgNWs was injected into heated EG as seeds. After 5 min, the PVP and AgNO$_3$ solution was added at the injection rate of 0.1 mL/min using a syringe pump. For 2 h, the reaction was maintained for very long NW synthesis.[29,30]

AgNW/PEDOT:PSS Hybrid Film Fabrication: The cleaned AgNW was dispersed in IPA solution at a concentration of 0.003 mg/mL. This mixed solution was deposited onto a glass or polymer substrate by pipetting and coating by a Mayer rod. The resistance of the film was adjusted by repeating the coating steps for larger thickness. After drying IPA, the PEDOT:PSS (Sigma Aldrich) solution was deposited following the same method. To enhance the evaporation rate, IPA was added to the PEDOT:PSS solution at a 2:1 v/v ratio.

Characterization: The fabricated film was coated with 10 nm of gold before the SEM analysis. In order to investigate the adhesion strength of the transparent electrode, a 90° peeling test was conducted. A 12-mm-wide and 30-mm-long piece of 3M scotch tape was attached to an electrode sample and the peeling strengths of the electrode specimens were directly measured by a mechanical testing machine. The main components of this machine are a PI M-413.3PD linear stage and an Interface SMA-60N load cell. By pulling the tape at a constant displacement rate of 5 mm/s, the applied load was continuously monitored and analyzed as a function of time.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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