

Fused Silver Nanowires with Metal Oxide Nanoparticles and Organic Polymers for Highly Transparent Conductors

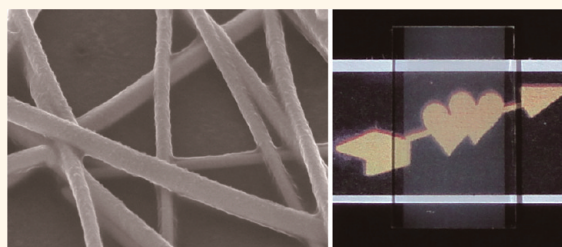
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Currently, the most popular transparent conductive film is indium tin oxide (ITO). High optical transparency and low sheet resistance have ensured its wide application in many areas. Although it has been used for several decades, the limited supply of indium has raised concerns as to the long-term future of ITO, and there is an urgent need for novel transparent conductive electrodes. There are several candidates, for example, carbon nanotubes,¹ graphene,^{2–4} metal grid,⁵ or thin metal films.⁶ Compared with commercial ITO substrates, these candidates make compromises between optical transparency and electric conductivity.⁷ Recently, pioneering methods^{8–16} have been developed using silver nanowire (AgNW) networks to achieve transparent conductors, which show comparable performance to commercial ITO.

For the large-scale fabrication and wide application of AgNW films, there are several issues that need to be addressed. First, effective connections between AgNWs are the key parameter to achieve high conductivity and transparency. However, due to the possible surfactant coating of polyvinylpyrrolidone (PVP) on the surface of AgNWs⁷ and the loose contact between AgNWs, extra treatments are often required to fuse the crossed AgNWs together. These treatments include high-temperature (>150 °C) or long-time thermal annealing,^{8,10} applying extra pressure,^{12,13,15,16} vacuum filtration,^{9,16} and HCl vapor treatment,¹⁷ etc. Second, strong adhesion between AgNWs and the substrate is critical to obtain stable and robust AgNW films for wide application. Substrate surface modification has been

ABSTRACT



Silver nanowire (AgNW) networks are promising candidates to replace indium–tin–oxide (ITO) as transparent conductors. However, complicated treatments are often required to fuse crossed AgNWs to achieve low resistance and good substrate adhesion. In this work, we demonstrate a simple and effective solution method to achieve highly conductive AgNW composite films with excellent optical transparency and mechanical properties. These properties are achieved *via* sequentially applying TiO₂ sol–gel and PEDOT:PSS solution to treat the AgNW film. TiO₂ solution volume shrinkage and the capillary force induced by solvent evaporation result in tighter contact between crossed AgNWs and improved film conductivity. The PEDOT:PSS coating acts as a protecting layer to achieve strong adhesion. Organic photovoltaic devices based on the AgNW–TiO₂–PEDOT:PSS transparent conductor have shown comparable performance to those based on commercial ITO substrates.

KEYWORDS: silver nanowire · transparent electrode · solar cells · TiO₂ · solution process · self-assembly · capillary force

used to improve the adhesion of nanowires on substrates.^{16,18} Applying strong conformal pressure can also enhance the adhesion.^{12,13,15,16} Moreover, nail polish liquid,¹⁷ *in situ* polymerization,^{11,14} and surface encapsulation¹⁰ have also been reported to improve the adhesion of AgNWs on substrates.

From a practical application point of view, the formation of AgNW films should be simple and cost-effective, while the performance (such as conductivity and transparency)

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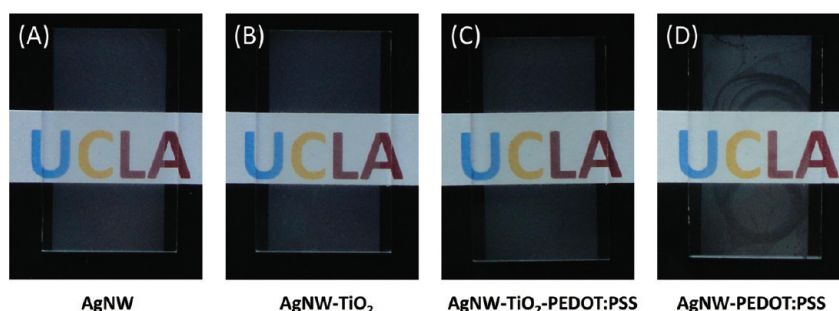


Figure 1. Pictures of AgNW films with different treatments. (A) Pristine AgNW film; (B) treated with TiO_2 sol–gel solution; (C) treated with TiO_2 sol–gel and PEDOT:PSS solutions sequentially; (D) treated only with PEDOT:PSS solution. Sample size: $15\text{ mm} \times 25\text{ mm}$.

should be retained, if not improved. In this work, a simple and efficacious solution-based method is demonstrated to achieve highly conductive AgNW films with good optical transparency and excellent adhesion on the substrate. Titanium dioxide (TiO_2) sol–gel solution and poly(3,4-ethylenedioxythiophene)/poly(styrenesulfonate) (PEDOT:PSS) solution are used to treat AgNW films sequentially. We found that the TiO_2 nanoparticles and PEDOT:PSS polymer hybrid composite can bind the AgNW network together. As a result, the connections between AgNWs and their adhesion on substrates are greatly enhanced. The AgNW– TiO_2 –PEDOT:PSS composites show excellent electric, optical, and mechanical properties, with excellent film uniformity.

RESULTS AND DISCUSSION

AgNWs used in this work are commercially available or prepared according to previous reports.¹⁰ AgNWs are dispersed in isopropyl alcohol (IPA) and then coated onto glass substrates by spray-coating^{19,20} or Mayer rod coating^{10,17} methods. In the experiments here, it was found that the spray-coating method is better for small-scale samples. By adjusting the spray-coating parameters such as the nozzle size, air flow rate, dispersion concentration, and the distance between nozzle and substrate, uniform AgNW films can be obtained with little agglomeration, as shown in Figure 1A. The pristine AgNW films are first treated with TiO_2 sol–gel solution (0.1 wt % in ethanol),²¹ which will be converted into TiO_2 nanoparticle clusters after low-temperature hydration in air ($\sim 80^\circ\text{C}$, 10 s), resulting in uniform AgNW– TiO_2 hybrid films (Figure 1B). Commercial PEDOT:PSS solution is then coated onto the AgNW– TiO_2 film and dried at 80°C for another 15 s, providing the AgNW– TiO_2 –PEDOT:PSS hybrid film with excellent uniformity and transmittance (Figure 1C).

It is notable that, if the pristine AgNW mesh is only treated with PEDOT:PSS water solution, clear rings can be observed on the AgNW–PEDOT:PSS film, as shown in Figure 1D. The rings obviously come from the edges of the PEDOT:PSS solution drops when they were dripped onto AgNWs in the spin-coating process.

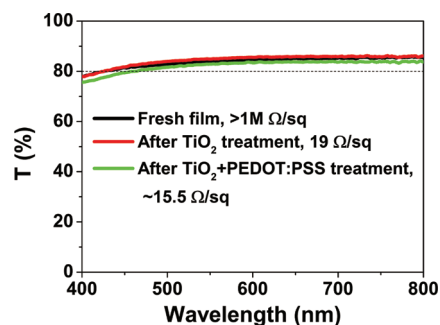


Figure 2. Transmission spectra of AgNW films after different treatments.

AgNWs at the edges of PEDOT:PSS drops will come off from the substrate due to the high surface tension between water solution and AgNW film. This indicates poor adhesion between AgNWs and the substrate. In contrast, spin-coating TiO_2 sol–gel from its ethanolic solution onto AgNWs can retain film uniformity (Figure 1B); this is ascribed to better wetting and lower surface tension of the ethanol solution on AgNW films. By comparing panels B and D of Figure 1, we conclude that treating AgNW films with TiO_2 sol–gel solution will provide an indispensable binding force between the AgNWs and the substrate. The binding forces between AgNWs and their adhesion on substrates are strong enough so that the AgNW film can survive the subsequent PEDOT:PSS solution coating.

Figure 2 shows the transmission spectra of the AgNW film before and after different treatments. For the pristine AgNW film (without any treatment), the film transmittance is 84% at 550 nm. Due to the PVP surfactant on the surface of AgNWs and the loose contact between AgNWs in the pristine film, its sheet resistance is rather high ($>1\text{ M}\Omega/\text{sq}$).⁸ After a TiO_2 sol–gel solution treatment, the film transparency is almost the same as that before treatment, but the sheet resistance is greatly reduced from $\text{M}\Omega/\text{sq}$ to $\sim 19\text{ }\Omega/\text{sq}$. After further treatment by PEDOT:PSS solution, the film transmittance is slightly decreased to 83% at 550 nm, while the sheet resistance is further decreased to $15\text{ }\Omega/\text{sq}$. The decreased transparency is primarily because of the weak absorption of PEDOT:PSS

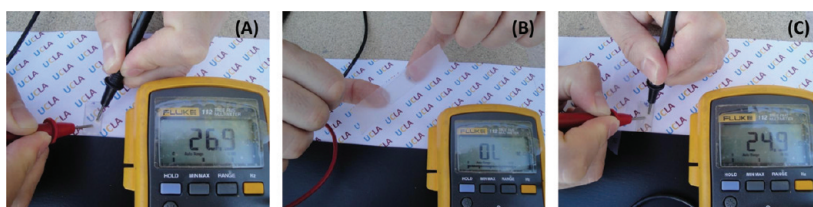


Figure 3. Mechanical adhesion test of the conductive AgNW–TiO₂–PEDOT:PSS film. (A) Resistance measurement before tape peeling; (B) use adhesive tape to peel the film; (C) resistance measurement after tape peeling.

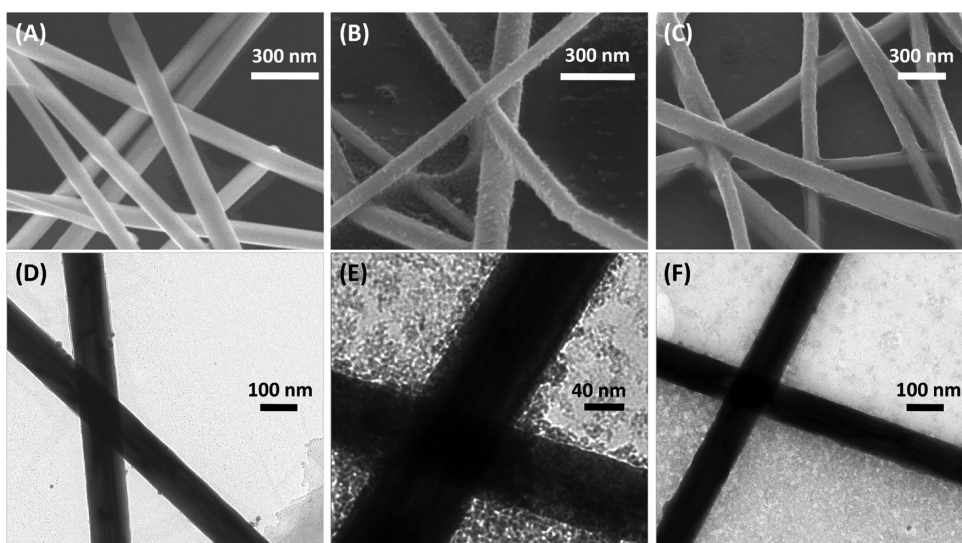


Figure 4. (A–C) Scanning electron microscopy and (D–F) transmission electron microscopy images of the AgNWs before and after different treatments: (A,D) pristine AgNWs without any treatment; (B,E) AgNWs after TiO₂ sol–gel treatment; (C,F) AgNW–TiO₂–PEDOT:PSS hybrid film.

in the visible range. This solution treatment method has been repeated on more than 30 samples, showing excellent reproducibility. The results of both transparency and resistance are comparable to reported results for transparent AgNW films and commercial ITO substrates.^{8–17} Beyond excellent optical and electrical performance, the AgNW–TiO₂–PEDOT:PSS composite film also shows strong mechanical adhesion on substrates. The adhesion between AgNW–TiO₂–PEDOT:PSS films and substrates is evaluated by mechanical tape testing,¹⁰ as shown in Figure 3. For the as-prepared AgNW–TiO₂–PEDOT:PSS films, the resistance measured with the two-point probe method shows a value of $\sim 26.9 \Omega$. After peeling the film with 3M Scotch tape three times, the resistance remains $\sim 25 \Omega$. This simple experiment indicates the strong mechanical adhesion of the conductive AgNW–TiO₂–PEDOT:PSS composite film on glass substrates. The current low-temperature solution treatments are much simpler and more suitable for low-cost large-scale fabrication of high-performance AgNW films. Moreover, TiO₂ is also a stable material under either acidic or basic conditions. This will ensure its wide application and great stability on different substrates or supports.

To understand the roles of both TiO₂ sol–gel solution and PEDOT:PSS solution treatments, the films are

characterized using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Figure 4 compares the SEM and TEM images. For pristine AgNW films, the connection between crossed AgNWs is mainly driven by gravity, van der Waals forces between AgNWs, and capillary forces from solvent evaporation. From the SEM image of the pristine AgNW film (Figure 4A), charging can be observed at the crossed positions between AgNWs. This indicates low conductivity due to loose connections between AgNWs. In Figure 4B, the AgNW film is treated with TiO₂ sol–gel solution. Solvent evaporation provides the capillary force that drives TiO₂ nanoparticles to assemble and to aggregate around AgNWs randomly and discontinuously (Figure 4B,E).^{22,23} It is notable that there are meniscus TiO₂ nanostructures formed around crossed AgNW positions that bind the crossed AgNWs together. These TiO₂ meniscus structures are ascribed to the nanoparticle assembly under capillary forces induced by solvent evaporation.²² Moreover, the meniscus TiO₂ nanoparticle structures can also be found between the substrate and AgNWs, and this helps AgNWs to stick onto the substrate with improved adhesion. However, TiO₂ nanoparticle aggregations are usually weak and brittle and cannot provide strong adhesion and tension. The AgNW–TiO₂

film can still be peeled off from the substrate with adhesive tape. To enhance mechanical connections and adhesion further, the AgNW–TiO₂ film is treated with PEDOT:PSS solution. Figure 4C shows the SEM image of the AgNW–TiO₂–PEDOT:PSS hybrid film. After PEDOT:PSS treatment, the PEDOT:PSS polymer composite forms a thin and continuous coating on the TiO₂-coated AgNWs. From the SEM image, it is clear that all of the TiO₂ meniscus structures at the AgNW intersection points or the positions between AgNWs and substrate are further covered by PEDOT:PSS polymer film. The PEDOT:PSS coating acts as the protective layer on the AgNW–TiO₂ film and provides much stronger binding forces through the entire AgNW networks. With these results, we can understand the strong adhesion of the AgNW–TiO₂–PEDOT:PSS film together with good conductivity, as demonstrated in Figure 3.

On the basis of the above discussion, we know that the TiO₂ and PEDOT:PSS treatment can help to improve the adhesion and conductivity of AgNW films. The improved adhesion is ascribed to the strong binding force induced by TiO₂ and PEDOT:PSS hybrid composites, which can be seen in Figure 4. However, the reason for the great enhancement in conductivity after TiO₂ treatment is not clear. It has been reported that growing Au nanoparticles (or Au–Ag alloyed nanostructures) on AgNWs helps to improve the film conductivity because Au nanoparticle conductors bridge AgNWs at their intersection points and provide improved conduction between crossed AgNWs.¹⁰ In the current case, TiO₂ is a semiconducting material and much less conductive than Au nanoparticles, but only TiO₂ sol–gel solution treatments can improve the film conductivity from M Ω /sq to several tens of Ω /sq. It is unlikely that the TiO₂ nanoparticles alone act as conducting connectors between AgNWs because TiO₂ nanoparticles do not have sufficient free carriers to yield the high conductivity of Au–Ag alloyed nanostructures.¹⁰ The most probable explanation is that TiO₂ solution treatment helps AgNWs form closer and better contacts at their intersections. During solvent evaporation of the TiO₂ sol–gel solution, capillary forces will drive TiO₂ sol–gel clusters to assemble around the intersection of two crossed AgNWs. When the solution is dried and hydrated, the volume of TiO₂ sol–gel clusters will shrink and become TiO₂ nanoparticle aggregates with much closer packing. The driving force from the volume shrinkage will drive the crossed AgNWs closer and result in tighter contact between AgNWs at the intersections. The meniscus nanostructures formed around the intersections provide evidence for the capillary forces induced by solvent evaporation. To understand the contact between AgNWs at the intersection, focused ion beam (FIB) milling was used to obtain cross-sectional images of the crossed AgNWs. To protect the underlying layers

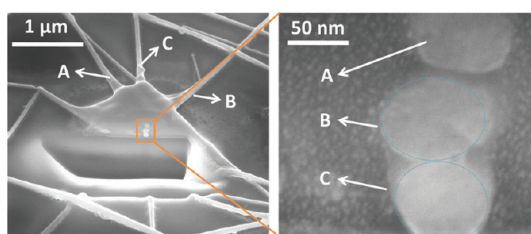


Figure 5. Cross-sectional SEM image of the AgNWs intersection point. The sequence of nanowires A, B, and C are top, middle, and bottom, respectively. The FIB cut only the intersection point between B and C but did not reach the intersection point between A and B.

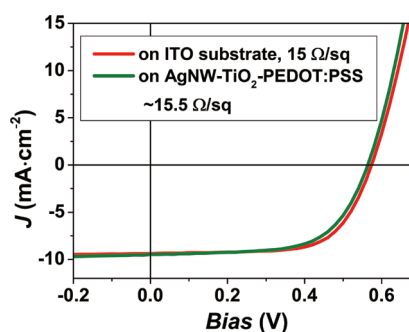


Figure 6. *J*–*V* characterization of P3HT:PC₆₀BM photovoltaic devices using ITO or AgNW–TiO₂–PEDOT:PSS substrates.

from ion beam damage, platinum pad was deposited to cover the intersection of the crossed AgNWs. The intersection point of crossed AgNWs was then cut out by FIB. Figure 5 shows the cross-sectional SEM image of the AgNW intersection point. From the enlarged SEM figure, we can observe that nanowires B and C stack closely, indicating good contact between AgNWs.

With the transparent AgNW–TiO₂–PEDOT:PSS conductive substrate, we made an organic photovoltaic (OPV) device to evaluate its performance. Poly(3-hexylthiophene) (P3HT) and phenyl-C₆₁-butyric acid methyl ester (PC₆₀BM) were used as the donor and acceptor, respectively. An ITO substrate for an otherwise identical device was used for comparison. Figure 6 shows the current density *versus* voltage characterization. For the device using ITO as the anode electrode, a power conversion efficiency (Eff) of 3.5% was obtained with a short-circuit current density (*J*_{sc}) of 9.3 mA·cm^{−2}, an open-circuit voltage (*V*_{oc}) of 0.57 V, and fill factor (FF) of 66.5%. When the AgNW–TiO₂–PEDOT:PSS substrate was used, similar performance was achieved, with *V*_{oc} = 0.56 V, *J*_{sc} = 9.5 mA·cm^{−2}, FF = 63.4%, and Eff = 3.4%. This indicates that the AgNW–TiO₂–PEDOT:PSS substrate works well as a transparent conductive substrate and is a promising replacement for commercial ITO substrates in OPV devices. It should also be noted that the rough surface of AgNW films is always a challenge for device

fabrication on AgNW films.^{10,14,15} In these experiments, modified PEDOT:PSS²⁴ was coated onto the AgNW—TiO₂—PEDOT:PSS substrate to bury the AgNWs into a thicker transparent and conductive PEDOT:PSS film (~200 nm). The details of the modified PEDOT:PSS have been reported in our previous work.²⁴ With this thicker but conductive and transparent modified PEDOT:PSS coating, the possible electrical short between the AgNW substrate and back electrode^{10,15} in the devices can be avoided.

CONCLUSIONS AND PROSPECTS

In conclusion, we have developed a simple and effective method to achieve highly conductive AgNW composite meshes with excellent optical transparency and mechanical properties. This is achieved *via* applying TiO₂ and PEDOT:PSS solutions sequentially to treat AgNW films, resulting in AgNW—TiO₂—PEDOT:PSS composites. Thin films coated from this composite possess excellent mechanical, electric, and optical properties.

METHODS

Materials. AgNWs were obtained from Blue Nano Inc. or synthesized according to previous reports.¹⁰ Poly(3-hexylthiophene) (P3HT) was purchased from Rieke Metals, Inc. [6,6]-Phenyl-C₆₁-butyric acid methyl ester (PC₆₀BM) was purchased from Nano-C. Poly(3,4-ethylenedioxythiophene)/poly(styrenesulfonate) (PEDOT:PSS, CLEVIOS P VP Al 4083) was purchased from H. C. Starck. All materials were used as received. TiO₂ sol—gel solution was prepared according to previous reports.²¹

AgNW Films Preparation and Treatments. Small-area AgNW films were prepared by spray-coating with a Tamiya airbrush.^{19,20} Large-scale AgNW films were prepared by Mayer rod coating following previous reports.^{10,17} To achieve good uniformity and avoid agglomeration in the AgNW films, diluted AgNW dispersions (0.05 mg/mL in IPA) were used. The dispersion was sonicated for 10 s and well shaken before spray-coating. In the TiO₂ sol—gel solution treatment, the TiO₂ sol—gel was dispersed in ethanol with a concentration of 0.1 wt %. The solution was dripped onto the AgNW film and spin-coated at 3000 rpm for 30 s. The film was then dried at 80 °C for 10 s under ambient conditions. After that, the PEDOT:PSS water solution (0.8 wt %) was dripped onto a AgNW—TiO₂ film and spin-coated at 3000 rpm for 30 s. The film was also dried at 80 °C for 10–30 s under ambient condition to complete the AgNW—TiO₂—PEDOT:PSS transparent conducting film.

P3HT/PC₆₀BM Photovoltaic Device Fabrication. The devices were fabricated on ITO-coated glass substrates or AgNW—TiO₂—PEDOT:PSS substrates with a sheet resistance of ~15 Ω/sq. Modified PEDOT:PSS was spin-coated on the substrates providing the anode modification layer with a thickness of ~200 nm.²⁴ The P3HT:PC₆₀BM (1:1, 2 wt % in dichlorobenzene) solution was then spin-coated onto the modified PEDOT:PSS layer following the slow growth method in our previous report.²⁵ After annealing at 110 °C for 10 min, device fabrication was completed by thermal evaporation of Ca (20 nm) and Al (100 nm) as a cathode under vacuum (2×10^{-6} Torr).

Optical, Electrical, and Microscopic Characterization. The transmittance spectra were taken using a Hitachi ultraviolet–visible spectrophotometer (U-4100). *J*–*V* characteristics of photovoltaic cells were taken using a Keithley 2400 source unit under a simulated AM1.5G spectrum with an Oriel 91191 solar simulator. The light intensity was ~100 mW·cm⁻², as calibrated using a Si photodiode. The surface resistance (<100 Ω/sq) was measured

The SEM images indicate that the TiO₂ is used to prebind the AgNWs and to improve the connection between crossed AgNWs, while PEDOT:PSS enhances the film conductivity further and acts as a protecting layer to achieve much stronger adhesion between AgNWs and the substrate. The quality of the AgNW—TiO₂—PEDOT:PSS transparent conductor has been compared with commercial ITO substrates in P3HT/PC₆₀BM-based OPV devices. Results indicate that the AgNW—TiO₂—PEDOT:PSS can achieve device performance comparable to ITO glass substrates. It is also concluded that the TiO₂ solution volume shrinkage and the capillary force induced by solvent evaporation might be the primary reasons for the tighter contact between crossed AgNWs, resulting in the greatly enhanced conductivity. Using insulating nanomaterials to enhance the AgNW connection and conductivity is surprisingly effective and promising. Further work is in progress to understand the details of the conductivity enhancement by semiconducting and insulating nanoparticle solution treatments.

using the four-point probe method with a surface resistivity meter (Guardian Manufacturing, model SRM-232–100, range = 0–100 Ω/sq). The two-point probe method (regular multimeter) was used to estimate the surface resistance of the films with surface resistance >100 Ω/sq. Scanning electron microscopy images were taken on a JEOL JSM-6700F electron microscope. Transmission electron microscopy was conducted with a JEOL 2010F electron microscope. Cross-sectional SEM images were taken using a FEI Nova 600 Nanolab Dualbeam SEM/FIB.

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Supporting Information Available: Comparison between high-temperature annealing treatment and TiO₂ sol—gel solution treatment. Optical transmittance spectra of AgNW—TiO₂ films with different sheet resistances. Plot of sheet resistance *versus* film transmittance at 550 nm for AgNW—TiO₂ films. Cross-sectional SEM images of the AgNW intersection point in the untreated AgNW film. This material is available free of charge *via* the Internet at <http://pubs.acs.org>.

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