Size Control of Gold Nanoparticles Grown on Polyaniline Nanofibers for Bistable Memory Devices

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ABSTRACT Controlling reaction temperature for a set time enables the size of gold nanoparticles autoreduced on the surface of polyaniline nanofibers to be controlled. The size of the gold nanoparticles can be used to tune the electrical bistable memory effect in gold/polyaniline nanofiber composite devices. Turn-on voltages and on/off ratios improve with decreasing nanoparticle size, making this a promising method to enhance performance and create smaller devices. Long-term stability of the composites can be improved by the addition of stabilizers following autoreduction of the gold nanoparticles.

KEYWORDS: polyaniline nanofibers · gold nanoparticles · organic−inorganic nanocomposite · nanoparticle size/growth control · bistable memory devices

Metal nanoparticles combined with conducting polymer nanofibers have demonstrated functionality in a variety of applications which include catalysts,[1] sensors,[2] and memory devices.[3,4] One of the key parameters in controlling and optimizing the properties of these devices is the size of the metal nanoparticles. Stable and uniform size control of gold nanoparticles can be difficult, and currently, the best particle solutions utilize stabilizers added during the synthesis.[5,6] For example, amine or thiol groups can function as stabilizers for the synthesis of small (~1−20 nm), stable, monodisperse solutions of gold nanoparticles, with thiols exhibiting the best long-term stability.[7] Previously, we demonstrated that relatively uniform gold nanoparticles can be synthesized directly on the surface of polyaniline nanofibers without the need for stabilizers.[4] This differs greatly from the result obtained with conventional polyaniline films for metal reduction, which generally produce irregularly sized and shaped metal particles.[8] The uniform metal nanoparticles are achieved through direct reduction of chloroauric acid ($HAuCl_4$) by the nanofibrillar conducting polymer. Here the nitrogens along the polyaniline chain provide physical stabilization for the autoreduced gold nanoparticles.[4] The simple water-processable synthesis of polyaniline nanofibers[9,10] matches up well with the ease of the one-step nanoparticle fabrication. This facilitates the tuning of reaction conditions to allow control of the size of the metal nanoparticles formed from the polyaniline nanofiber autoreduction as we report here.

Optimized particle size and stability are especially crucial to the performance of bistable two-terminal memory devices, often called cross-bar memory. The cross-bar device structure is promising due to its potential for scalable electrode fabrication, which could lead to high-density nonvolatile flash-type memory devices. Of the materials used in cross-bar memory devices, bistable molecules[11] and metal organic nanocomposites[3,4,12] are two of the most promising candidates. However, organic metal nanocomposites have distinct advantages, for example, memory devices manufactured with polyaniline allow very fast (<25 ns) write/erase nonvolatile memory responses, facilitated by a rapid charge transfer mechanism.[4,5]

As these memory devices have not been optimized, there is a clear need for studying the synthetic control of the gold polyaniline nanocomposite materials. This investigation examines the solution syntheses of polyaniline nanofibers with different sizes of autoreduced gold nanoparticles and evaluates the relationship between gold nanoparticle size and bistable memory response. Insights gained from changing the

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particle size may also be applicable to other nanoparticle bistable memory devices.

Various sizes of gold nanoparticles (AuNPs) can be grown on polyaniline nanofibers by modifying the reaction conditions (Figure 1). For example, by cooling the reactants to different temperatures prior to reaction, during the reaction and during dialysis purification, average AuNP sizes of <1, 2, and 6 nm can be obtained, as shown in the TEM images Figure 1a–c, respectively. For the synthesis of 6 nm particles, a slightly higher concentration of gold is needed, approximately 1:1.4 of aniline (monomer unit in polymer)/Au^{3+} (ion molar ratio). Reliable generation of particles of 6 nm and larger size is considerably more difficult as larger nanoparticles (>20 nm) were occasionally observed in the 6 nm AuNP particle synthesis (<1% of particles). This size variation is much more prominent with room temperature and reflux reactions, as shown in the TEM images Figure 1d,e. The 20–100 nm sized AuNPs, generated with the room temperature reaction (Figure 1d), are formed *via* a reaction ratio of 1:1.2 aniline/Au^{3+}. The largest particles generated in solution were created by carrying out the polyaniline/chloroauric acid reduction under reflux for 1 h and then purifying at room temperature (Figure 1e). For this synthesis, chloroauric acid is added to a boiling polyaniline nanofiber solution, and the product settles out, producing a red supernatant and dark sediment. The red color is attributed to the surface plasmon resonance of the ~50 nm gold particles in the dispersion.[13] The supernatant and sediment contain essentially the same particles; both possess micrometer scale single crystal gold sheets (crystallinity confirmed *via* TEM diffraction as shown in the inset to Figure 1e) and a small amount of 20–100 nm gold particles found on the polyaniline nanofibers.

The nanoparticle generation in the autoreduced gold polyaniline nanocomposite system is dominated by the temperature-dependent nucleation and growth characteristics, which has been demonstrated in other gold nanoparticle systems.[6,13–15] When the reaction is held at low temperature (0–2 °C) with a low concentration of gold cations, the reaction rate is slow, allowing nucleation of gold nanoparticles in many different locations on the polyaniline nanofibers. Because the polyaniline nanofibers act as the reducing agent, all of the gold nanoparticles are nucleated on the nanofibers and none are found in solution separate from a polyaniline nanofiber. As the temperature and/or concentration of the gold ions are increased, the stability of the nanoparticle generation is compromised, leading to fewer nucleations and aggregation and Ostwald ripening of the least stable, smallest gold nanoparticles.[16] This phenomenon reaches the limit when the temperature of the reaction is increased to reflux temperature and large single crystals of ~15 μm across are generated (Figure 1e). All of the reaction conditions for the nanoparticle syntheses are listed in Table 1.

RESULTS

After generation of the autoreduced gold nanoparticle–polyaniline nanofiber composite system, the memory device performance was tested to determine which samples have the best performance. Current–voltage (I–V) scans were made from 0 to +5 V, and then back to 0 V. In general, after the turn-on voltage is reached, a stable, one-way conductance switching occurs from a low to a high conductivity state. This manifests itself in the device as a high current state (thus demonstrating high conductivity) and can be reliably used in data storage as the "on" state or "1", while the erased device is the low current "off" state or "0". To test the cycling ability of the devices, the turn-on scans were followed by a scan from 0 to −6 V to turn the device back to the low conductivity, or off-state.

The device performance scans for devices made from four different solutions, with the nanoparticles at four distinct size ranges (<1, 2, 6, and >20 nm) are presented in Figure 2. From these characteristic scans, the turn-on voltages of the devices made from the <1, 2, and 6 nm gold nanoparticle solutions were found to be 1.5, 3, and 4.5 V, respectively, while the on/off ratio, that is, the difference between the low conductivity state and high conductivity state measured at 1 V, was 4, 3, and 1.5 orders of magnitude, respectively. The data present typical performance seen after several I–V scans, as the first scan in some cases can be anomalous because of the

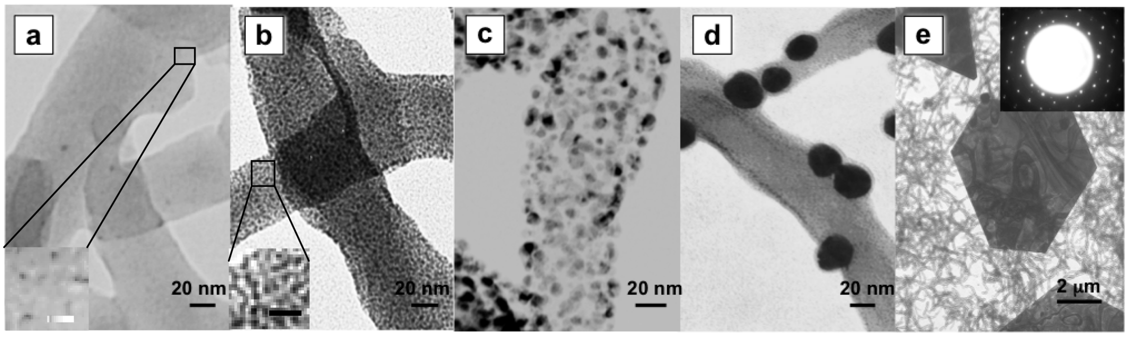

Figure 1. TEM images of autoreduced gold nanoparticles of (a) <1, (b) 2, (c) 6, and (d) >20 nm grown on polyaniline nanofibers. Inset scale bars = 10 nm. (e) Gold crystal [scale bar = 2 μm] with its TEM diffraction pattern (inset).

TABLE 1. Reaction Conditions for the Growth of Gold Nanoparticles on Polyaniline Nanofibers

reactant temp (°C)	synthetic temp (°C)	dialysis temp (°C)	Au NP size (nm)	turn-on bias (V)	on/off ratio	cycles
2	2	2	<1	1.5	10^4	~10
25	2	2	2	3.0	10^3	$>10^2$
25	2	25	6	4.6	10	~10
25	25	25	>20	5	1	1
100	100	25	>10000[a]			

[a] Largest dimension.

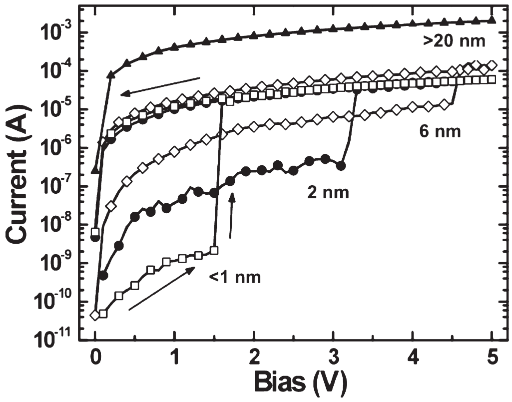

Figure 2. Characteristic *I*−*V* scans for autoreduced gold nanoparticle−polyaniline nanofiber composite memory devices.

residual charge built up in the device during fabrication. The initial scan may exhibit jumps in the performance curve; however, after several cycles, the performance curve becomes stable and repeatable. The >20 nm gold nanoparticle device did turn on once during the first scan before remaining in the high conductivity state, with a turn-on voltage of 6 V. Following the first scan, this device could not be erased and remained in its high conductivity state. This suggests shorting of the device from filament formation and/or agglomeration of the relatively large (>20 nm) gold particles. The major trends from an analysis of all these experiments are that, as the particles get larger, the turn-on voltage necessary to switch into the high conductivity on-state increases and the on/off current ratio decreases. For an ideal device, a larger on/off ratio provides more stability and a lower turn-on voltage requires less power consumption. The larger gold nanoparticles are clearly not ideal for bistable memory devices due to their poor cycling and high turn-on voltages. However, the larger the gold particle, the greater the capacity is for charge storage on the particle.[17] This leads to greater residual charge in the device, thus increasing the off-state current, which results in a smaller on/off ratio. The trend of lower turn-on voltages with smaller nanoparticles is likely due to a decreased barrier from the close proximity of the gold nanoparticles. Hence, it appears that the smaller the nanoparticles, the better the devices perform, with lower voltages needed for device turn-on and larger on/off ratios. Among the devices tested here, the best performance was found with the 2 nm nanoparticle composite due to the low turn-on voltage, large on/off ratio, and the best demonstrated cycling, with the number of write−read−erase cycles being in the hundreds.

In order to further explore the electrical characteristics of the gold nanoparticle−polyaniline nanofiber composite system, tunneling AFM (TUNA) was used to perform *I*−*V* characterization at the nanoscale. In these studies, both autoreduced gold nanoparticle solutions and more traditional stabilized gold nanoparticle solutions were examined. Four different composites were analyzed: a 2 nm autoreduced nanoparticle−polyaniline nanofiber composite solution (Figure 3a), a 5 nm citrate-stabilized colloidal gold solution mixed with polyaniline nanofibers (Figure 3b), a 10 nm citrate-stabilized colloidal gold solution mixed with polyaniline nanofibers (Figure 3c), and a 2 nm dodecanethiol-stabilized colloidal gold solution mixed with polyaniline nanofibers (Figure 3d). For the stabilized nanoparticle devices, the gold nanoparticle solutions were synthesized prior to mixing with the polyaniline nanofiber solutions. One of the obvious differences, visible in the TEM images shown in Figure 3, is that the composite solutions with the stabilized nanoparticles have nanoparticles present where there are no nanofibers, in contrast to the autoreduced nanoparticle solution that only has nanoparticles on the nanofibers. However, for the TUNA measurements, only areas with nanoparticles in contact with a nanofiber were used to observe the electrical performance. Because of the imaging limitations of scanning AFM mode used to locate the material for TUNA experiments, it is likely that multiple nanoparticles are interacting during the measurements. The TUNA device characterization was carried out by applying a linear potential scan from 0 to −9 V from the AFM tip to the gold nanoparticle(s) to trigger the jump in conductivity to the "on-state", followed by a reverse scan from −9 to 0 V. Although linear scans were used to demonstrate the bistable conductivity of the gold nanoparticle−polyaniline nanofiber composite system, complete write−read−erase−read cycling data (demonstrating stable and rewritable memory) using TUNA were not possible due to the thermionic drifting effect experienced by the AFM probe between consecutive measurements. Another drawback to the TUNA system is that the on/off ratios cannot be readily obtained due to a current saturation threshold of ±125 pA for the TUNA module; however, the *I*−*V* curves (Figure 4a) do

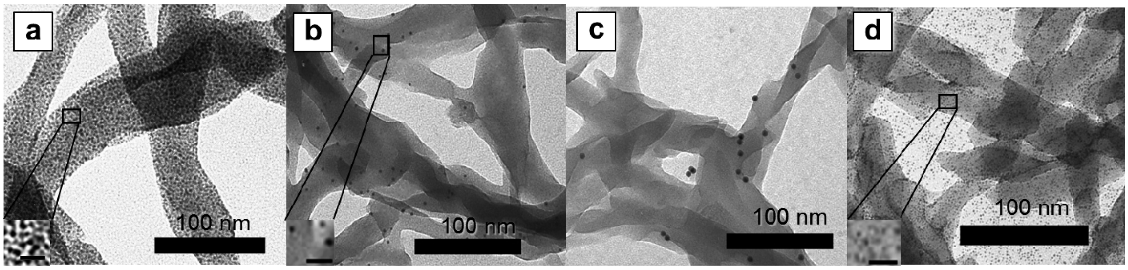

Figure 3. TEM images of (a) autoreduced gold nanoparticles grown on polyaniline nanofibers and (b–d) colloidal nanoparticles and polyaniline nanofiber composites. Inset scale bars = 10 nm.

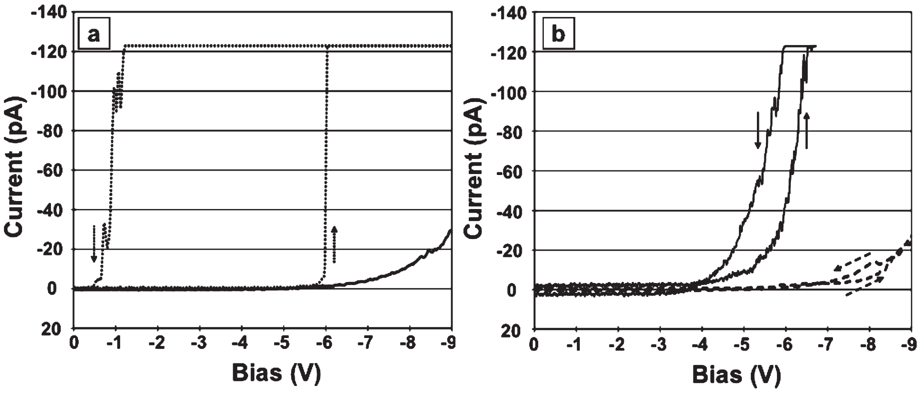

Figure 4. Tunneling AFM *I*–*V* scans of (a) autoreduced gold nanoparticle–polyaniline nanofiber (dotted line) and 2 nm dodecanethiol-stabilized colloidal gold solution mixed with polyaniline nanofibers (black line), and (b) 5 nm citrate-stabilized colloidal gold solution mixed with polyaniline nanofibers (solid line) and 10 nm citrate-stabilized colloidal gold solution mixed with polyaniline nanofibers (dashed line).

show that the conductivity of the gold nanoparticle–polyaniline nanofiber system increases by at least 2–3 orders of magnitude when the system switches from the low conductivity off-state to the high conductivity on-state.

The 5 nm citrate-stabilized colloidal gold nanoparticle–polyaniline composite demonstrates induced hysteresis between the on and off scans, although the difference between the turn-on voltage and turn-off voltage is considerably smaller than that of the 2 nm autoreduced gold nanoparticle–polyaniline nanocomposites (Figure 4). The 10 nm citrate-stabilized colloidal gold nanoparticle–polyaniline nanofiber composite demonstrates a similar hysteretic response, but less pronounced (Figure 4b). Analogous to the results obtained for the bulk cross-bar devices, a general trend of increasing turn-on voltage with increasing gold particle size was observed during tunneling AFM (TUNA) characterization, demonstrated by the responses of the 5 and 10 nm citrate-stabilized colloidal gold nanoparticle–polyaniline composite devices.

A secondary interaction that is demonstrated in the TUNA measurements is the impact of the type of gold nanoparticle stabilizer on the electrical switching characteristics. Dodecanethiol, when used as a stabilizer for gold nanoparticle synthesis prior to combination with polyaniline nanofibers, clearly affects the interactions between the nanoparticles and the nanofibers (Figure 4a). This is in contrast to the autoreduced system where there is a more intimate contact between the nanoparticles and the nanofibers. The electrical characteristics of the autoreduced system have already been described in some detail.[5] The weak stabilization of the citrate ion used with the 5 and 10 nm particles allows relatively normal interactions with charge transfer between the gold colloid and the polyaniline chains; however, the 2 nm dodecanethiol-stabilized gold nanoparticle–polyaniline composite demonstrates a response more like that of much larger particles. The dodecanethiol group appears to inhibit charge transfer between the polyaniline chains and the gold nanoparticles. This is likely due to the addition of the nonpolar stabilizer onto the metal nanoparticles prior to composite synthesis, which creates a physical barrier between the metal nanoparticles and the polyaniline nanofibers. This type of electrical switching has been demonstrated previously with other thiol-stabilized nanoparticle memory systems.[18,19]

The sensitivity of the interface between metal and conducting polymer in gold polyaniline nanocomposites indicates the importance of the method of metal nanoparticle growth when developing composites for nonvolatile memory devices. A weak stabilizer, such as citrate, or direct reduction of metals by the charge transfer agent appears ideal to make composites for bistable memory devices. Still the method of autoreduction of the metal nanoparticles incorporating a

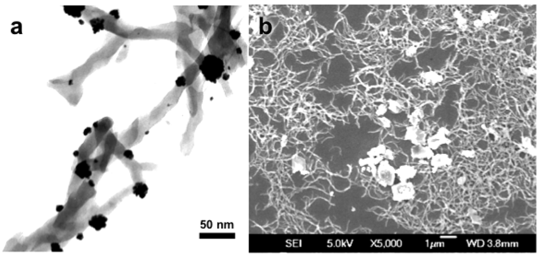

Figure 5. (a) TEM and (b) SEM images of an aggregated nanoparticle–polyaniline nanofiber composite.

separate charge transfer agent can prevent any potential problems related to decreased device performance while maintaining stabilization for long-term solvent storage by postsynthesis nanoparticle functionalization. An area to explore in this regard involves other nonvolatile memory devices created with metal nanoparticles such as virus/platinum nanoparticle composites.[20]

The smaller the metal nanoparticle, the more difficult it is to synthesize in solution without the aid of a stabilization agent.[21,22] This is found to be the case for autoreduced gold nanoparticle–polyaniline nanofiber composites, as well. In the bistable memory devices, the composite is dried and thermoset in a cross-linked matrix of poly(vinyl alcohol), which stabilizes the metal nanoparticles on the polyaniline and produces devices that can be turned on and off months later. However, after several weeks in solution, the metal nanoparticles in the autoreduced gold nanoparticle–polyaniline nanofiber composite solutions form sizable aggregates and crystallites (Figure 5a,b). Solutions with large aggregates lead to devices that exhibit poor memory performance characteristics because of electrical shorting.[23] Prestabilization of the gold nanoparticles can also pose potential device performance problems, so a different method is needed.

A simple method to prevent aggregation after autoreduction of gold onto polyaniline is by the addition of a postsynthesis stabilizer. The polyaniline nanofibers provide some stabilization for particle growth, but the interactions between the polymer chains and gold nanoparticles are not as strong as the covalent gold–thiol bond[22] or as stabilizing as a monolayer of alkanethiol. The addition of hydrogen sulfide (H_2S), which will readily interact with the metal surface of the nanoparticles,[24] will prevent aggregation, as well. Solutions can be treated with dodecanethiol and successfully used to make devices several weeks after the initial synthesis and still demonstrate over a 4 order of magnitude change in current at a read voltage of 1 V with a turn-on voltage of 3 V (Figure 6).

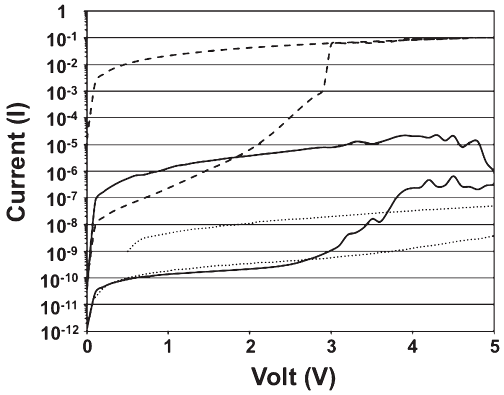

Figure 6. I–V scans of an aggregated nanoparticle–polyaniline nanofiber composite (dotted line) and aggregated solutions treated with hydrogen sulfide [H_2S] (solid line) and dodecanethiol (dashed line).

CONCLUSIONS

We report a unique size control of gold particles from <1 nm gold nanoparticles up to single-crystalline gold microsheets by the aqueous autoreduction of chloroauric acid with polyaniline nanofibers. This technique allows improved turn-on voltages and on/off current ratios, with decreasing nanoparticle size and increasing particle density. Memory devices established with 2 nm average sized gold nanoparticles provide the best overall performance with good cycling ability. The gold nanoparticles must be in direct contact with the polyaniline for device turn on. This is prevented when gold nanoparticles are stabilized with dodecanethiol prior to deposition on the polyaniline nanofibers. Our advantageous synthesis allows reduction of metal cations directly on the conducting polymer nanofibers. The metal cations can be stabilized following the autoreduction reaction so that aggregation and Ostwald ripening are arrested, while still allowing excellent device performance. Both the improved device performance with smaller gold nanoparticles and the size control of autoreduced gold polyaniline nanocomposites offer opportunities for expansion into other areas. For example, smaller metal nanoparticles directly interacting with charge transfer agents may be developed for further advancement of bistable memory devices. Also, the size tunability may prove to be advantageous in applications such as catalysis and sensors, especially with expansion to other noble metals.[1,25]

METHODS

Polyaniline Gold Composite Synthesis. Camphorsulfonic acid (CSA) and ammonium peroxydisulfate were obtained from Fisher Scientific and used as received. Aniline was purchased from Fisher Scientific and purified *via* distillation at reduced pressure prior to use. Gold colloids (5 and 10 nm) were purchased from Sigma-Aldrich and used as received. The dodecanethiol-stabilized 2 nm gold colloid was synthesized by a two-phase arrested growth method.[6] Chloroauric acid, $HAuCl_4$, was purchased from Electron Microscopy Sciences in

0.1 g vials. A fresh 10 mM solution from an unopened vial of $HAuCl_4$ solid was made each time new autoreduced gold polyaniline nanocomposites were needed. Polyaniline nanofibers were synthesized by rapidly combining aniline and ammonium peroxydisulfate in 1 M camphorsulfonic acid, as described previously,[26,27] and purified *via* centrifugation (twice redispersing the pellet with 0.5 M NaOH, followed by redispersing three times with deionized water). The gold nanoparticles were deposited onto the polyaniline nanofibers *via* the autoreduction of chloroauric acid.[4,5] Typically, a ratio of 1 aniline monomer unit to 1.2 gold cations (or a 0.55 mol fraction of gold cation) in solution was used for the reaction mixtures with a reaction time of 2 days. Temperature of the reactant solutions prior to mixing, during reaction, and for dialysis purification was modified to alter the size of the gold nanoparticles deposited onto the metal polyaniline nanocomposites as listed in Table 1.

Microscopy. Transmission electron microscope (TEM) images were taken with a JEOL JEM-100CX, with samples prepared by drop-casting the gold nanoparticle/polyaniline nanofiber composite solution onto carbon-coated gold grids.

Device Fabrication. A two-terminal device was fabricated through thin-film solution-processing methods. Initially, a polymer composite solution that contained about 1−2 mg mL^{-1} of polyaniline/gold nanoparticle fibers and 10−15 mg mL^{-1} of polyvinyl alcohol was spin-cast at 1000 rpm over aluminum electrodes on glass substrates. The polyvinyl alcohol provides adhesion to the substrate, stabilization of the composite, and acts as the matrix layer. The thickness of the composite layer varied from 50 to 70 nm, verified using a profilometer (Dektak), and was of sufficient smoothness to allow deposition of the aluminum electrodes *via* thermal evaporation. The composite layer was thermoset, and then the top aluminum electrodes were deposited perpendicular to the bottom electrodes. The overlap area between the top and bottom electrodes is the area of one device, $200 \times 200\ \mu m^2$.

Device Characterization. Current−voltage ($I-V$) characteristics were measured by a semiconductor parametric analyzer (HP 4155B). AFM characterization and tunneling AFM (TUNA) $I-V$ measurements were taken on a Multimode AFM (MultiMode V, Veeco Instruments).

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