



Zinc Oxide (ZnO) Nanobridge-based Sensor Platform and Functionalization for Explosive Sensing

by Ashley D. Mason and Brett H. Piekarski

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14. ABSTRACT Nanowire (NW)-based sensors present an alternative to thin films for improved gas sensing because of their inherently high surface-to-volume ratio. Zinc oxide (ZnO) NWs have a well-developed vapor-phase growth method and can be seeded on carbonized photoresist (C-PR). The C-PR also acts as an electrical contact; however, a molybdenum (Mo) layer decreases contact resistance and masks C-PR areas where growth is not desired. The selectivity of the device is refined using chemical functionalization, starting with a parylene-A activation layer applied via chemical vapor deposition (CVD). We present images of the C-PR, Mo, and NW interfaces taken with a microscope and a scanning electron microscope (SEM), as well as atomic force microscopy (AFM) roughness data for the parylene-A coatings. Using the functionalization described here, an array of devices could be created using the same process with only the final layer of functionalization (end-node species) changing. The results gleaned from these experiments can aid in the search for a chemical-based sensor array.					
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1. Introduction/Background

The enhanced surface-to-volume ratio of nanowires (NWs) allows higher sensitivity than thin-film-based alternatives (1). The role of surface states is amplified, which facilitates gas sensing (2–5). Specifically, metal-oxide NWs have been used to detect a number of gases (5–8) and can be fabricated using a relatively easy vapor-solid growth method (9, 10). Although NW-based devices are a highly sensitive platform, the development of wafer-scale integrated selective sensors still remains a challenge.

NW nanobridge (NB) devices can be grown from one electrode and terminate on an opposing electrode, so no additional processing steps are needed to make electrical contact. Figure 1 shows images of the earliest reported NB device. Silicon (Si) NWs were grown on a silicon-on-insulator (SOI) substrate using a patterned gold (Au) catalyst (11, 12). The difficulty of patterning Au, metal contamination, and the need for an SOI substrate are some of the drawbacks of this method. Alternatively, as shown in figure 2, zinc oxide (ZnO) NB devices have been created using a patterned ZnO seed layer (13, 14). Although the use of a metal catalyst is avoided, this method still requires a SOI substrate, and reactive ion etching is required for trench formation, deposition, and patterning of the seed.

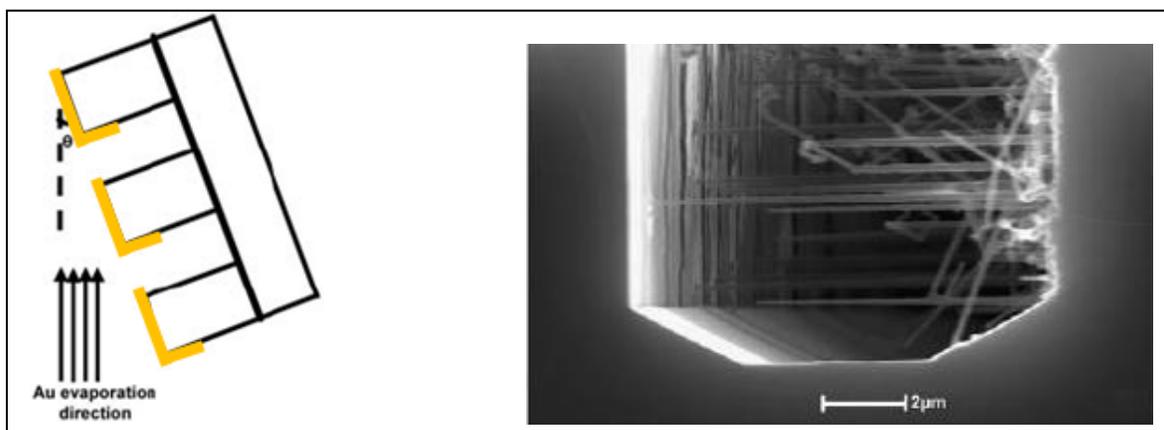


Figure 1. Formation of Si NBs using an Au seed layer (11, 12).

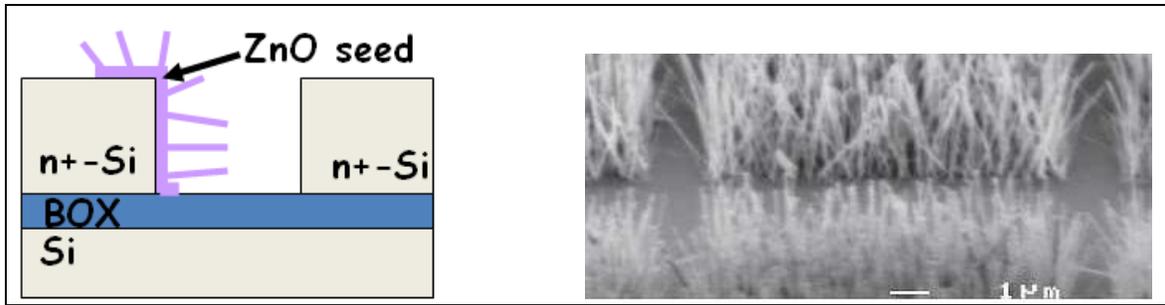


Figure 2. ZnO NWs seeded on a ZnO thin film (13, 14).

Recently, carbonized photoresist (C-PR) has been demonstrated as a nucleation layer for selective and oriented growth of ZnO NWs from evaporated ZnO powder, as shown in figure 3 (15). The advantages of the C-PR nanobridge approach are (1) the NWs can be seeded without the introduction of metal contamination and (2) there are more options for a base substrate, an SOI substrate is not required. Traditional lithography is used to pattern the growth area, which greatly reduces to complication of fabrication.

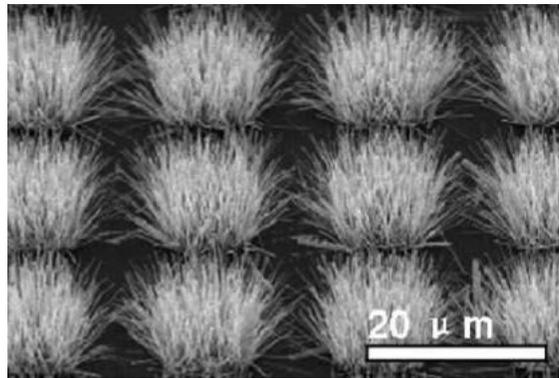


Figure 3. Selective growth of ZnO NWs on C-PR (15).

Preliminary reports of electrical measurements on C-PR based NBs have been reported (16, 17). As a proof of concept, our initial selective sensing demonstrations were based on the exploitation of bonding between biotin and streptavidin. Since ZnO is known to dissolve in deionized (DI) water and all the processes used to attach biotin to the surface require DI water (18), the focus of this work was to investigate potential ways to protect the ZnO NWs.

Parylene or [2,2] paracyclophane was originally manufactured as a dimer and is commonly used as a moisture barrier and an activation layer. There are many different variations of parylene; the monomers of a few examples are shown in figure 4. Parylene-N (no functional group) and parylene-C (chlorine group) have been the most widely used previously. We are developing a chemical vapor deposition (CVD) coating process for amino-[2,2] paracyclophane (19, 20) or parylene-A. The amine group is more useful than the other functional groups for anchoring aptamers, which can be used to selectively sense a target species (21).

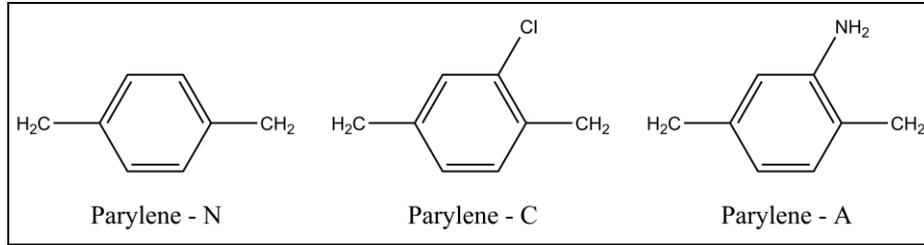


Figure 4. Various types of parylene (N, C, and A).

2. Experiment

2.1 Base Device Structure

The base device structure process flow is shown in figure 5. Wet oxidation was used to grow a 200–300 nm oxide on a Si wafer. Lithography was used to define the device area, then photoresist was carbonized for 60 min at 900 °C and 5 Torr in a reducing atmosphere (95% argon [Ar], 5% hydrogen [H₂]), as in reference 22. Molybdenum (Mo) was sputtered and patterned using a lift-off process. This film was added to reduce contact resistance and reduce the impact of bulk effects by limiting the growth of ZnO NWs to only the bridging area. A burn-in time of 3 min was included prior to each Mo deposition. A ramp time of 1 min was used to reach the full power of 1 kW. A 300-nm film was sputtered using a pressure of 1.1×10^{-6} Torr Ar and a deposition time of 30 min. This initial recipe was successful on silicon dioxide (SiO₂) but not on C-PR (see section 2.1.1); the process was adjusted to fix the initial incompatibility with C-PR.

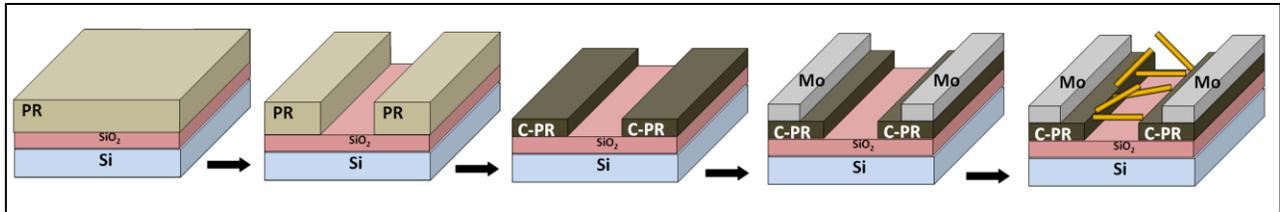


Figure 5. Process flow for fabrication of base device.

NWs were grown on the C-PR and Mo structure via carbothermal reduction of ZnO using pure ZnO (99.99%, Sigma-Aldrich) and graphite powders (99.9995%, Alpha Aesar) mixed in a 1:1 ratio (23). The precursors were placed in a tube furnace at 920 °C. Nitrogen (N₂) was used (150 sccm) to carry the mixture down to the growth substrate (held at 720 °C). We introduced 1–2 sccm of oxygen (O₂) 2.5 cm from the samples to aid in NW growth (shown in figure 6 along with resulting growth).

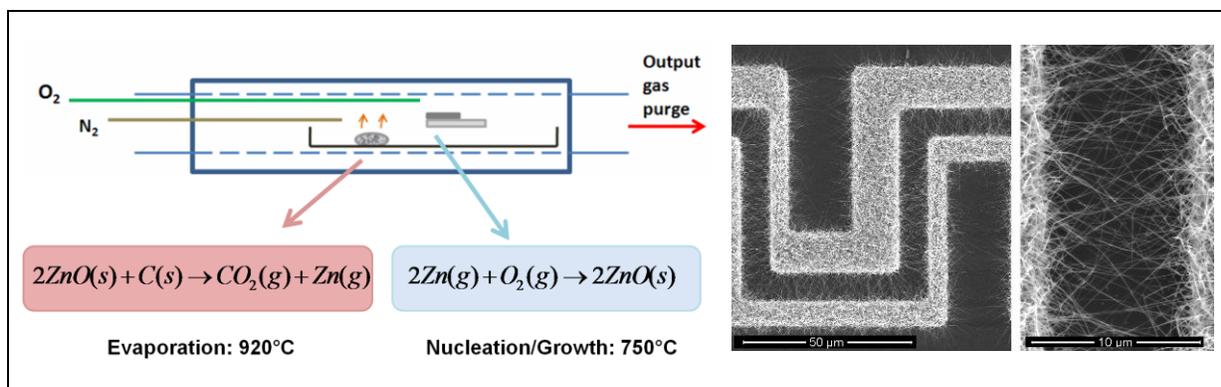


Figure 6. Experimental setup and resulting vapor-solid NW growth.

2.1.1 Molybdenum Sputtering Recipe Development

The initial Mo recipe is listed in section 2.1. This recipe was successful for Mo deposition on Si with a native oxide; however, when the Mo deposition was attempted on the C-PR surface, the films were no longer uniform or smooth, but instead were cracked across the sample. As shown in figure 7, this prevented the complete fabrication of devices. The C-PR films were imaged and appeared smooth so an O_2 plasma clean was included before the next sputter attempt (using the same initial recipe). The films were still cracking so the power was reduced to 300 W to decrease the deposition rate and the resulting film stresses that led to cracking. The successful deposition of Mo on C-PR is shown on the right in figure 7. NWs devices were made using these structures and electrical measurements are presented in section 3.2.

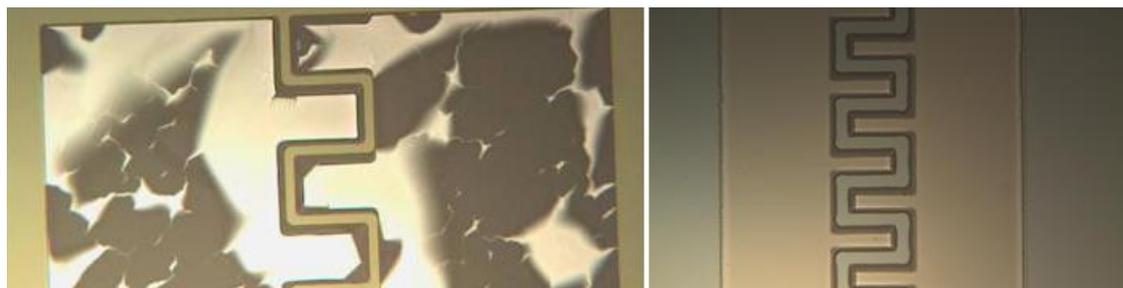


Figure 7. Film cracking when initial recipe was used on C-PR layer (left) and successful Mo deposition on C-PR (right).

2.2 Parylene-A Coating

The parylene-A dimer (dix-A, Kisco) was vaporized at $150^\circ C$, pyrolyzed at $690^\circ C$, and deposited on a ZnO thin film at room temperature, as shown in figure 8. Samples were placed at different locations in the tube to optimize the position for depositing a conformal film over the entire sample area. For characterization purposes, a poly(dimethylsiloxane) (PDMS) mask was applied to part of the sample to compare the uncoated and coated area.

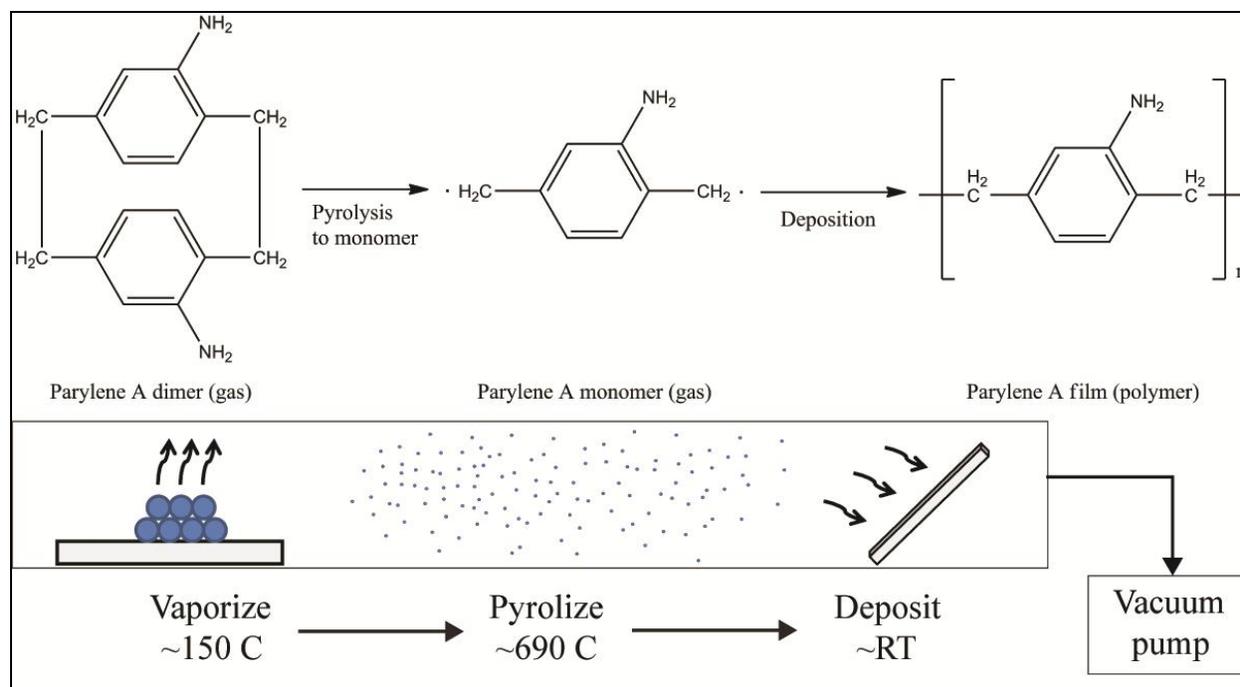


Figure 8. Parylene-A coating procedure.

Uniform films were deposited with 1–10 nm roughness between samples with most of the samples falling between 1–5 nm. The roughness of the deposited film was measured using a Veeco NanoScope V atomic force microscope (AFM) in tapping mode. A 1- μm scan size at 512 samples/line was used. These samples show a significant improvement over the original samples, which were only depositing parylene-A islands rather than a conformal film. By decreasing the pressure (originally 600 mTorr, now below 100 mTorr), nucleation in the gas phase was decreased and prevented island-like deposition (24). Representative AFM data for the conformal films are shown in figure 9. The thickness of the coating was too large to measure a step height. The recipe was altered to obtain a thinner layer prior to electrical measurements. The final substrate position for our system was 29 in away from the parylene-A precursor.

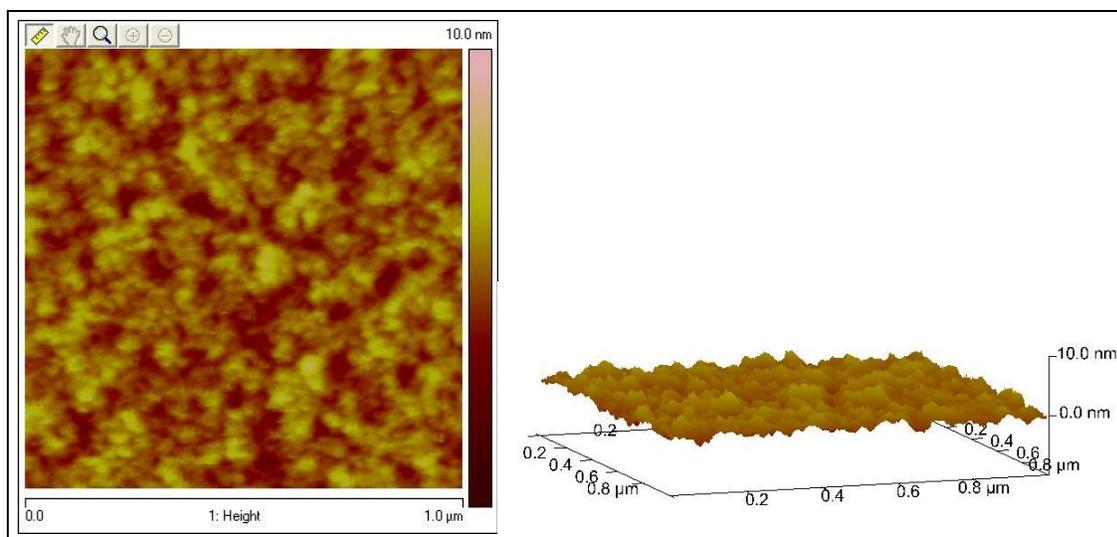


Figure 9. Parylene-A film root-mean-square (rms) roughness and three-dimensional (3-D) plot.

3. Results and Discussion

3.1 Protection of ZnO NWs

Samples with a 20-nm blanket ZnO film were coated with parylene-A. From visual inspection, one can see a difference in color between the coated and uncoated region (figure 10).

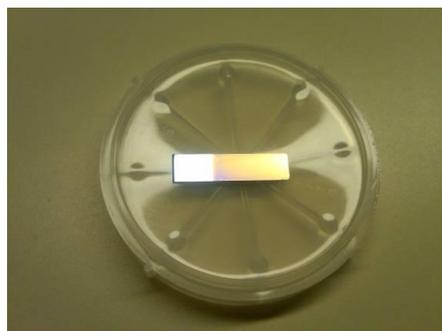


Figure 10. Visual differentiation between uncoated and coated sample regions: the left-hand side is uncoated (silver), the right-hand side is coated.

After developing the recipe for uniform parylene-A films, the next step was to prove that the coating preserved ZnO NWs when they were exposed to DI water. ZnO NWs with and without the parylene-A coating were soaked in DI water for 24 h (figure 11). Uncoated NWs dissolved completely within 24 h and some dissolution was visible after only 20 min. After coating ZnO NWs with parylene-A, they remain unharmed for at least 24 h. The parylene-A surface can then be biotinylated to demonstrate selective sensing utilizing biotin/streptavidin bonding.

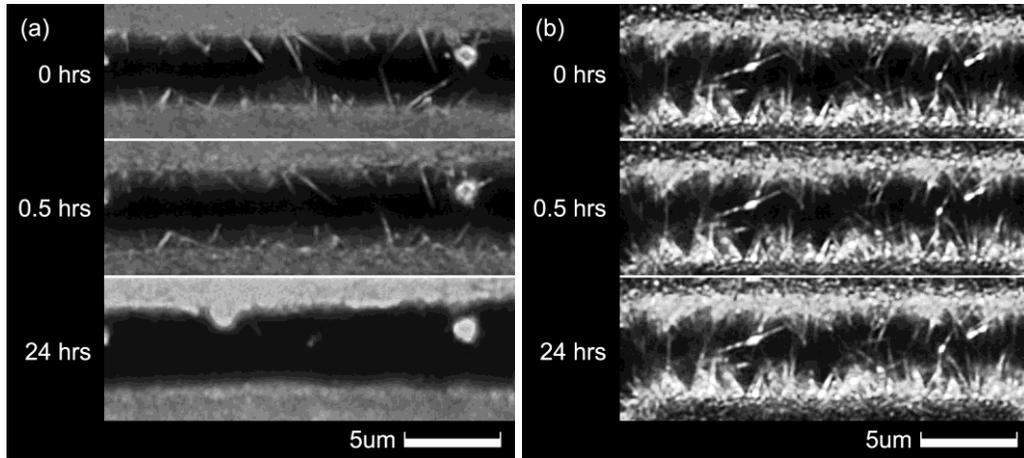


Figure 11. (a) Uncoated ZnO NWs (b) NWs protected with parylene-A coating.

3.2 Electrical Measurements

The current-voltage (I - V) characteristics and ultraviolet (UV) response of bare and parylene-A coated devices were measured using an Agilent 4155C. A Mineralight 254-nm, 18.4-W lamp was used for UV excitation. I_d - V_{ds} characteristics are presented in figure 12 for the same device before and after parylene-A coating. Measurements were taken with and without UV excitation. Another mechanism for the NB UV response, other than just photocarrier generation, is oxygen adsorption and desorption on the surface. When the device is not exposed to UV, the chemisorbed O_2 molecules on the NW surface form a depletion region, decreasing the NW conductivity. The higher dark current for the parylene coated devices (compared to the dark current for bare NWs) could be attributed to the displacement of surface oxygen or the reduction in available O_2 adsorption sites because of the coating. UV exposure generates electron-hole pairs within the NWs, the newly created holes discharge O_2^- chemisorbed on the surface. This reduces the surface depletion region and the conductivity within the wire is increased (25, 26).

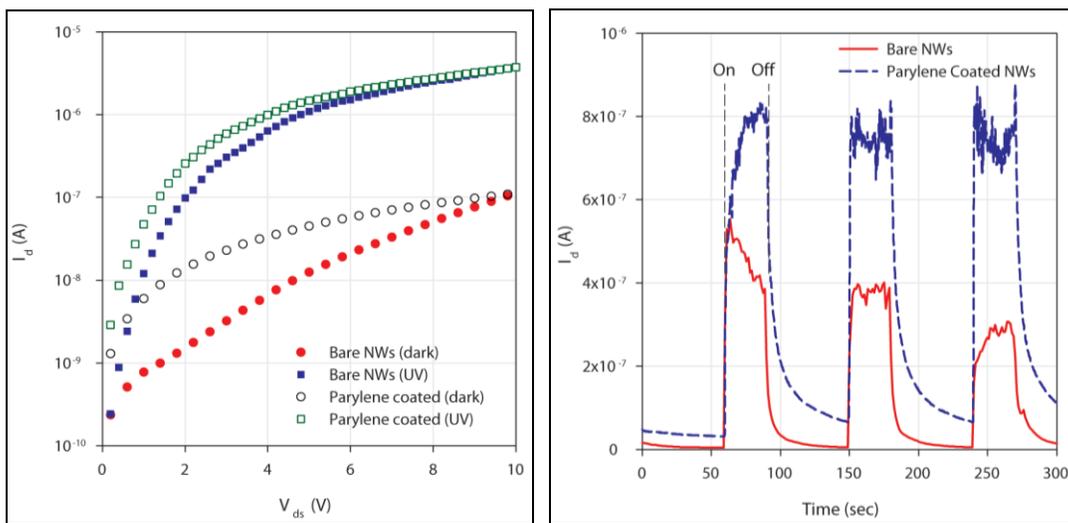


Figure 12. I - V and UV measurements for a bare and parylene-A coated NBs (26).

For the same device (before and after parylene-A coating), I_d versus time is also shown in figure 12. The measurements were made with $V_{ds} = 3$ V and a floating gate. Devices were exposed to 30-s pulses of UV light at room temperature in air. I_d of the bare NBs increases up to 100 times when exposed to UV. Although the parylene-A coated NBs exhibit a higher on current, the dark current is also higher, so the increase in I_d is only 45 times. Both the average rise time (the time it takes I_d to reach 90% of its maximum) and, to a larger extent, the average decay time (the time it takes for I_d to decrease to 10% of its maximum) are increased when the NB is coated. This shows that a parylene-A coating can be used to attenuate the UV response.

4. Conclusions

In this work, ZnO NWs were seeded on C-PR and an electrical connection was made simultaneously during the growth process. This method for fabricating NBs takes advantage of traditional lithography rather than more complicated etching techniques. Previous methods required the use of an SOI substrate whereas the C-PR method can be used on any insulating substrate. To protect ZnO NWs from dissolving in DI water, a parylene-A activation layer was applied via CVD. During the development process, parylene-A was deposited on 20-nm ZnO thick seed layers on Si substrates. Originally, the parylene-A was only deposited in islands on the ZnO film. By changing the sample position and decreasing the pressure (<100 mTorr), blanket films with rms roughness 1–10 nm were obtained. Electrical measurements were performed on a NB device before and after coating. I-V and UV measurements were used to show the impact of the parylene-A on device performance. The parylene-A coating decreased the UV sensitivity of the devices and could potentially be used to modulate environmental sensitivity when used in a vapor-phase selective sensing platform. The methods for further functionalizing and characterizing the parylene-A films will be continued at OSU.

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List of Symbols, Abbreviations, and Acronyms

3-D	three-dimensional
AFM	atomic force microscopy
Ar	argon
ARL	U.S. Army Research Laboratory
Au	gold
C-PR	carbonized photoresist
CVD	chemical vapor deposition
DI	deionized
ECE	Electrical & Computer Engineering
H ₂	hydrogen
I-V	current-voltage
Mo	molybdenum
N ₂	nitrogen
NB	nanobridge
NW	nanowire
O ₂	oxygen
ONAMI	Oregon Nanoscience and Microtechnologies Institute
OSU	Oregon State University
PDMS	poly(dimethylsiloxane)
rms	root-mean-square
SEM	scanning electron microscope
Si	silicon
SiO ₂	silicon dioxide
SOI	silicon-on-insulator
UV	ultraviolet
ZnO	zinc oxide

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