Synthesis, functionalization, and environmental stabilization of ZnO nanobridge transducers for gas and liquid-phase sensing

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ABSTRACT

Three methods of functionalizing ZnO NW surfaces with biotin were demonstrated. Biotinylated ZnO NWs were found to dissolve during exposure to deionized (DI) water, so a chemical vapor deposition (CVD) process was developed for parylene-A, a common moisture barrier with an amine group which allows further functionalization. Parylene-A coated ZnO NWs were found to be resistant to dissolution. Electrical measurements on parylene-A coated nanobridge devices showed normal operation with higher dark current and an attenuated response to UV and O_2, indicating the ability to modulate environmental sensitivity. This work demonstrates the novel use of parylene-A coatings as an encapsulation layer as well as a potential starting platform for general functionalization of ZnO NW devices for selective sensing.

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1. Introduction

Metal-oxide nanowire (NW) based sensors have become a highly active area of research due in part to inherently high surface-to-volume ratios that promise enhanced sensitivity to a target species [1–8]. In particular, devices based on ZnO NWs have demonstrated high sensitivity to UV [9–11] and various gases [11–15,8]. Despite significant progress in non-specific sensing using individual devices, commercial applications of NWs as sensors face many challenges. Two of the major challenges are (i) a method for large area electrical integration of these quasi-one-dimensional materials into a lithographically defined process and (ii) a reliable method for achieving highly specific/selective sensing.

There are several predominant approaches to NW integration. The most widely employed approach is known as "pick and place" in which NWs are grown on one substrate, harvested into solution, and then dispersed randomly onto another substrate for device fabrication [16,17]. One of the major drawbacks of this method is the lack of alignment to lithographically defined structures, although the issue of alignment has been addressed recently with the assistance of ac electric fields, i.e. dielectrophoresis [18–21]. Another approach is known as directed integration where NWs are selectively grown and used in place; for example, NWs are formed on one electrode and grow across a gap to terminate on a sec-

ond electrode. Directed integration of nanobridges is appealing due to the simultaneous growth, alignment to lithographic features, and electrical connection of NWs, potentially over a large area. This style of nanobridge formation was first demonstrated by Islam et al. [22] who used off-axis deposited Au films to catalyze the growth of Si NWs between the sidewalls of adjacent Si islands on a silicon-on-insulator (SOI) substrate. Although they were able to create electrically accessible devices [23], drawbacks of their particular method included metal catalyst contamination (Au), the need for reactive-ion-etching (RIE) to form the trenches, and the use of an SOI substrate (costly). Conley et al. [10,24] were able to avoid the use of a metal catalyst by using a thin conformal film of ZnO, deposited via atomic layer deposition (ALD), to seed the growth of ZnO NWs. Despite the advantages of avoiding metal contamination and RIE, the use of an SOI as well as the need for deposition, patterning, and etching of the seed layer complicated the fabrication process [10]. Selective growth of ZnO NWs has recently been achieved without a metal catalyst when carbonized photoresist (C-PR) was used to nucleate NW growth [25]. Huang et al. very recently used photolithographically patterned C-PR to direct the growth and simultaneously electrically integrate ZnO nanobridges into three-terminal sensor device structures. The C-PR method not only avoided the use of a metal catalyst, SOI wafers, and RIE, but because photoresist can be directly patterned, etching of a seed layer was not required. Although it was shown that these nanobridge devices were sensitive to O_2 gas and H_2O vapor, sensor selectivity or sensitivity to particular target species was not demonstrated [11]. In fact, a major challenge of NW sensors is to accomplish *specific* sensing of a single analyte in a complex sample while reducing the impact of ambient/NW surface interactions on

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the electrical properties of the NW device. The goal of the present work is to address this challenge by examining approaches to functionalizing the surface of ZnO NWs so as to bind specifically to an analyte of interest.

One of the most common ways to demonstrate sensor selectivity is by exploiting the strong bonding between biotin and streptavidin. We have been able to demonstrate attachment of biotin to the ZnO NW surface using passive sorptive modification, covalent bonding, and sorptive addition. However, in all three cases, the biotin coated ZnO NWs were found to dissolve completely within 24 h when placed in the deionized (DI) water solution used as a carrier medium for the biomolecules. Demonstration of selective sensing in the liquid-phase will require a way to prevent the ZnO NWs from dissolving in DI water.

Poly(p-xylylene) polymers, generally called parylene, are commonly used as moisture barriers and electrical insulators in the electronics industry. They have several unique properties including high chemical and thermal resistance. Parylene coatings are manufactured as dimers [2,2] paracyclophane, and can be deposited via a chemical vapor deposition (CVD) process that does not require solvents or catalysts. Parylene-N (has no functional group on dimer) and parylene-C (chlorine group on dimer) have been the most widely used coating materials for electronic devices. Parylene-A is made of one of the paracyclophane derivatives, amino-[2,2]paracyclophane [26,27]. Parylene-A coatings are of particular interest for this work because they facilitate further functionalization via the primary amine group.

In this paper, we examine ways to protect ZnO nanobridge devices from dissolving in an aqueous solution during liquid-phase sensing while simultaneously enabling functionalization for high selectivity. We develop a CVD process for parylene-A, demonstrate successful coating of ZnO NWs, and find that the parylene coating effectively protects ZnO NWs from dissolution in DI water. We also assess the impact of the parylene coating on the electrical performance, UV sensitivity, and gas sensitivity of ZnO nanobridge sensors. We find that the parylene coating can serve as an encapsulating layer that protects the NWs without adversely impacting device performance. Because they can be further functionalized via the primary amine group, parylene-A coatings appear to be a potentially viable general route to selective sensing for ZnO NW based devices.

2. Experimental details

2.1. Device fabrication

ZnO nanobridge sensor devices were made on Si substrates with a 300 nm thick steam-grown oxide as an insulating layer. After direct photolithographic patterning, photoresist (Shipley 1818) was carbonized at 900 °C for 60 min and 5 Torr in a reducing atmosphere (95% Ar, 5% H$_2$) [25]. Next, a 150 nm thick layer of molybdenum was sputtered from a 3″ target at 100 W and a pressure of 3.5 mTorr Ar. Vapor–solid growth of ZnO NWs was performed using ZnO (99.99%, Sigma–Aldrich) and graphite powders (99.9995%, Alpha Aesar) mixed in a 1:1 ratio [28]. The mixture was placed in a tube furnace at 920 °C with a carrier gas flow of 150 sccm N$_2$ to carry it downstream to the substrates at 770 °C. 1–2 sccm of O$_2$ was introduced 2.5 cm upstream from the substrates to aid in nanobridge growth.

2.2. Biotin functionalization

Using biotin as a representative bioactive protein [29], ZnO thin film surfaces were modified using three different functionalization procedures: (i) passive-sorptive modification using a protein, bovine serum albumin (BSA), (ii) covalent bonding using ethoxysilane chemistry, and (iii) sorptive addition of biotin via alde-

hyde coupling. Schematics of these functionalization processes are shown in Fig. 1.

(i) Passive sorptive modification of ZnO surfaces with bioactive proteins

Biotin-modified ZnO is prepared by exposing the ZnO surface to phosphate buffered saline (PBS) solution of biotinamidocaproyl-labeled BSA, followed by rinsing with buffer solution. First, biotinamidocaproyl labeled BSA (Sigma–Aldrich) in 1 mM pH 5.6 PBS is applied to ZnO films and ZnO NWs for 1 h at room temperature. After rinsing the samples with 1 mM PBS and drying with N$_2$ gas, NeutrAvidin-FITC (Pierce Biotechnology) in 1 mM pH 9 PBS solution is applied to the ZnO films and ZnO NWs and incubated for 1 h at room temperature. Next, the samples are washed with 1 mM pH 9 PBS solution and dried with N$_2$ gas in the same manner as in the previous step. The concentration ratio of biotinylated-BSA to NeutrAvidin-FITC is approximately 20:1, where the concentration of each solution is in the order of μM.

(ii) Covalent bonding of biotin to ZnO surfaces via ethoxysilane chemistry

This process is performed by exposing the ZnO surface to 10% ethanol solution of aminotriethoxysilane (APTES) for 30 min, followed by rinsing with absolute ethanol and heating in an N$_2$ atmosphere to remove unbound silane molecules.

(iii) Sorptive addition of biotin on the ZnO surface via aldehyde coupling

ZnO surfaces are exposed with biotin in a 2% ethanol solution of 3-trimethoxysilylpropyl aldehyde (TMSPA, with 4% water and 0.1% acetic acid), rinsed with absolute ethanol, and then heated in an N$_2$ atmosphere.

Biotin coating of the ZnO surface was evaluated by binding fluorescently labeled streptavidin-FITC (fluorescein isothiocyanate) to the biotin and measuring fluorescence intensity using a fluorescence microscope (Carl Zeiss Inc., Axio Imager M1).

2.3. Parylene coating

The procedure developed for CVD coating of parylene is illustrated in Fig. 2. In a single vacuum chamber, amino-[2,2]paracyclophane (dix-A, Kisco) was vaporized at 150 °C, pyrolized at 690 °C, and then deposited on ZnO nanobridge devices at room temperature. The thickness of parylene film was adjusted by varying the amount of parylene-A dimer in the vaporization zone (unless otherwise indicated, 0.1 g was used). Coatings were evaluated using water contact angle measurements. Water contact angle measurements were taken before and after surface modification treatment using an FTÅ 135 Contact Angle and Video Analysis System (First Ten Angstroms).

2.4. Device testing

The I–V characteristics and UV response of parylene coated and uncoated ZnO nanobridge devices were measured using a semiconductor parameter analyzer (Agilent 4155C) and a UV excitation source (Mineralight 254 nm, 18.4 W lamp). The sensitivity of parylene coated devices to O$_2$ and H$_2$O was performed using a semiconductor device analyzer (Agilent B1500) and a high temperature measurement cell (NorECs ProboStat). 200 sccm mass flow controllers (MFCs) were used to control gas flow rates.

3. Results and discussion

3.1. Biotin functionalization

It was found that all three procedures were successful in selectively grafting biotin onto ZnO thin film and NW surfaces. Shown
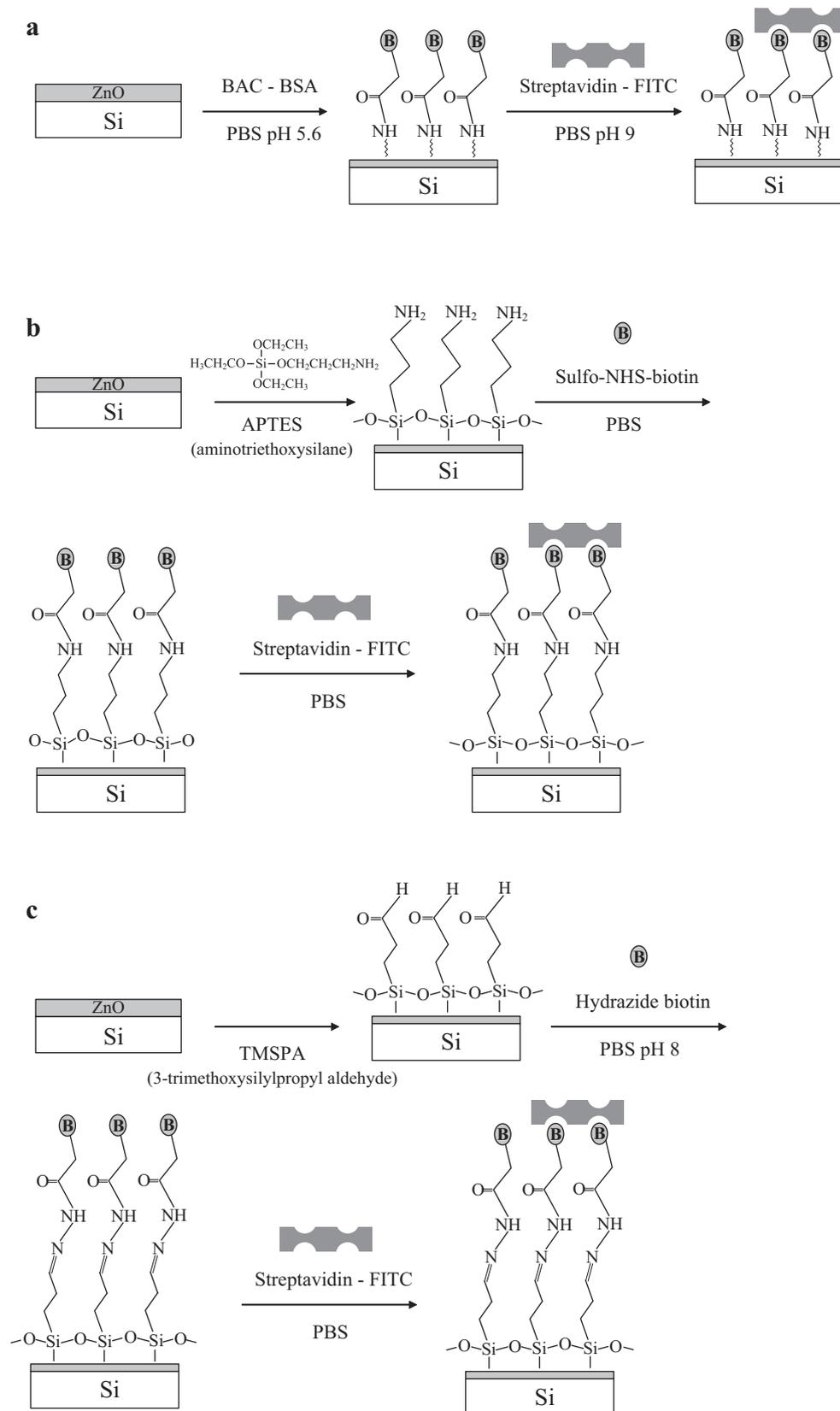
Fig. 1. Specific binding between biotin (B) and streptavidin-FITC on biotin-modified ZnO surfaces by (a) passive sorption, (b) covalent bonding via ethoxysilane chemistry, and (c) soptive addition via aldehyde coupling.
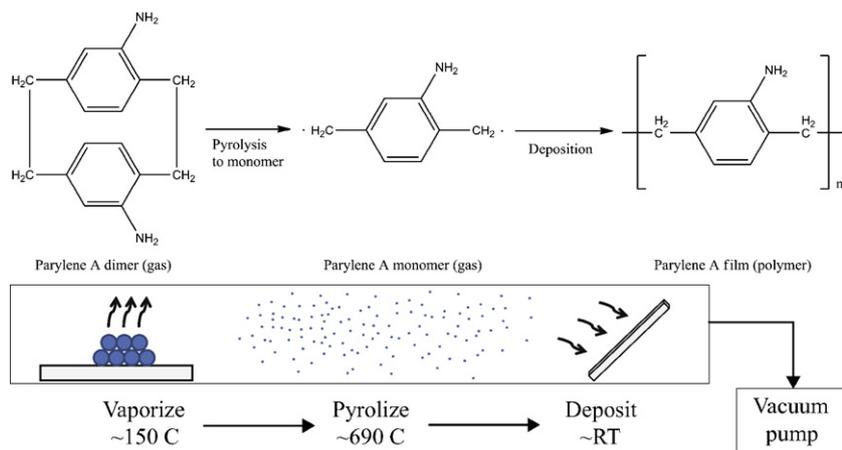
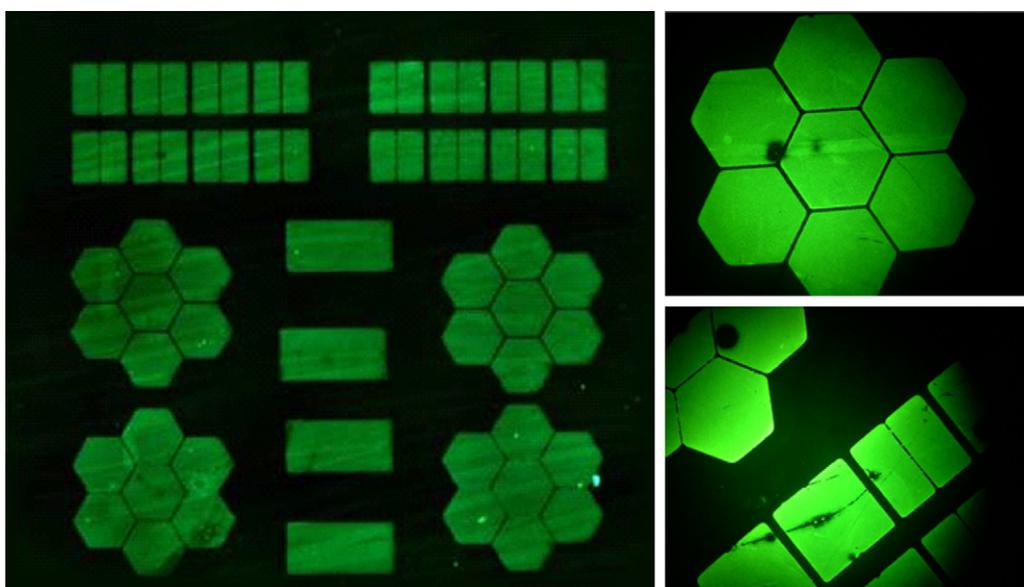**Fig. 2.** CVD procedure for deposition of parylene-A.

Fig. 3. Fluorescence microscopy images of a Si/SiO$_2$ substrate with patterned ZnO NW growth following biotin-modification and streptavidin-FITC conjugation by passive sorption. Bright areas indicate fluorescence and correspond to the regions of selectively grown NWs; dark areas correspond to the SiO$_2$ field.

in Fig. 3 are fluorescence microscopy images of a Si/SiO$_2$ substrate with patterned ZnO NW growth modified with biotin by passive sorption and subsequently labeled with FITC conjugated streptavidin. The ZnO NWs were grown selectively on a pre-patterned ZnO seed layer. The images reveal that streptavidin selectively binds to the patterned ZnO NW regions (bright areas), resulting from the specific binding between biotin and streptavidin on biotin modified ZnO. As a control, the functionalization procedures were also carried out on a blank silicon wafer. Insignificant fluorescence was observed on the control using all functionalization treatments.

It is known that ZnO dissolves in water [30] and as shown in Fig. 4, biotin functionalized NWs dissolve in as well. As a result, the impact of biotin functionalization on ZnO nanobridge device operation could not be determined.

3.2. Parylene coating

In an attempt to protect the ZnO NWs from dissolving in DI, ZnO NWs were coated with parylene-A via a CVD process. The presence of the parylene coating was assessed via water contact angle measurements. Shown in Fig. 5, the average contact angle for the unmodified ZnO surface was $22 \pm 5°$, whereas the average
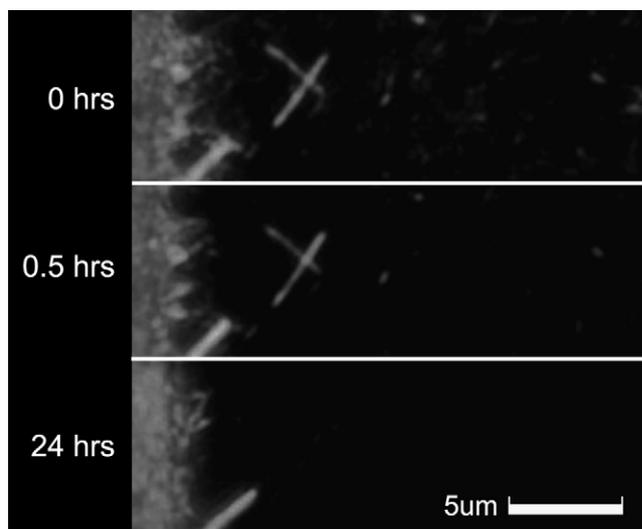

Fig. 4. Dark field microscope images taken as a function of time showing dissolution of biotin-coated NWs in DI water.
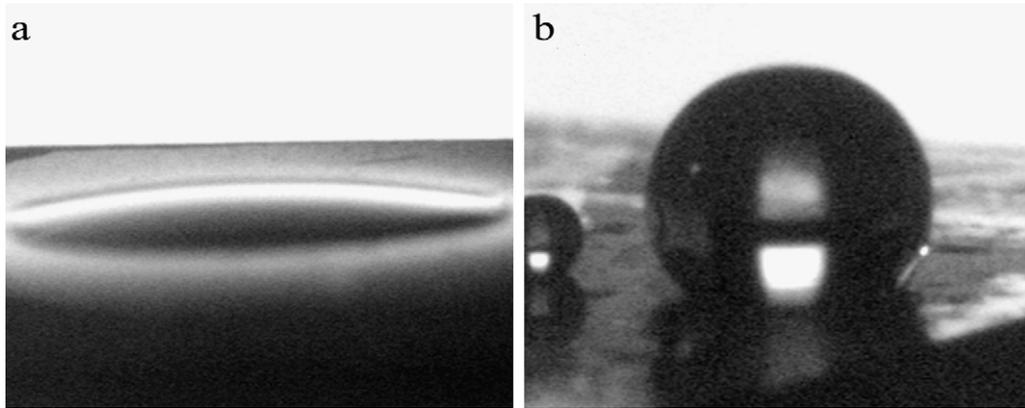
Fig. 5. Water contact angle measurements on (a) unmodified ($22 \pm 5°$) and (b) parylene coated ZnO NWs ($140 \pm 5°$).
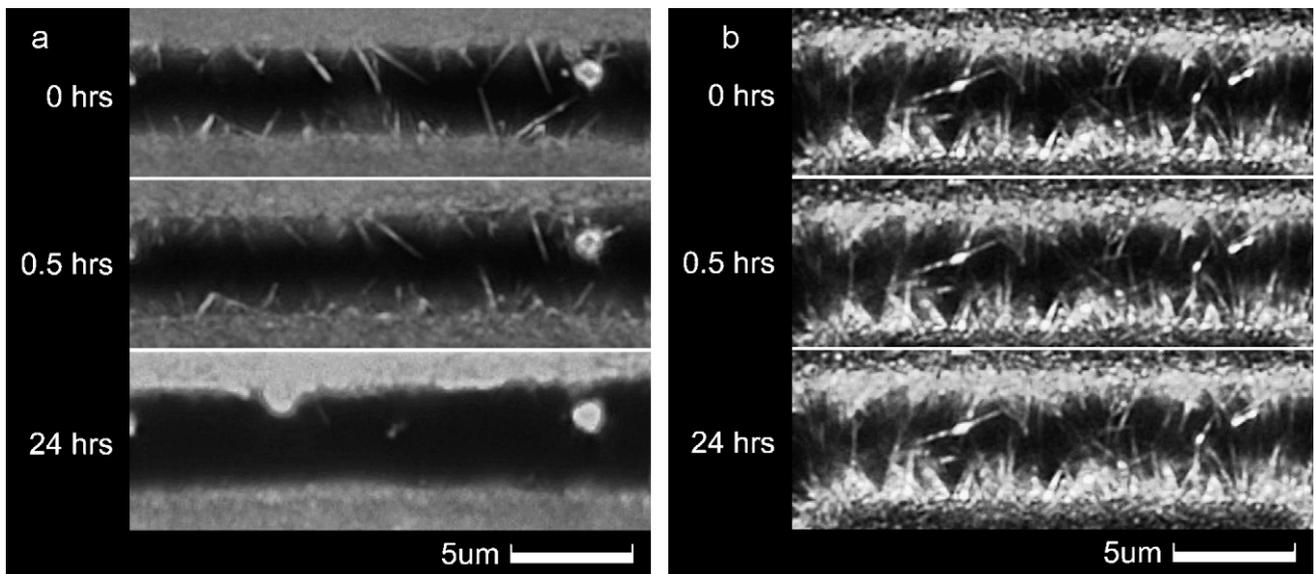

Fig. 6. Dark field microscope images as a function of time for (a) uncoated ZnO NWs and (b) parylene-A coated ZnO NWs left continuously submerged in DI water.

contact angle for parylene-A coated ZnO NWs was $140 \pm 5°$, indicating an increase in hydrophobicity of the surface, consistent with successful parylene coating. Uncoated and parylene-A coated ZnO nanobridge devices were then soaked in DI water for 24 h, as shown in Fig. 6. For an uncoated sample (Fig. 6(a)), the NWs have begun dissolving after only 0.5 h and after 24 h they have disappeared completely. The parylene-A coated sample (Fig. 6(b)), however, shows no dissolution, demonstrating protection of the ZnO NWs.

3.3. Electrical measurements

Although electrical properties for various ZnO NW based devices have been widely investigated, there are no reports about the impact of non-covalently bound polymer passivation layers on ZnO NW device I–V behavior, UV sensitivity, and gas sensitivity. $I_d - V_{ds}$ characteristics are presented in Fig. 7 for a 5 μm gap floating gate nanobridge device with and without UV exposure and before and after parylene coating. The curvature of the $I_d - V_{ds}$ traces suggests that the parylene coated device exhibits a decrease in Schottky behavior [31]. The formation of NW bridges between the opposing C-PR pads can take several forms: (i) direct connection by a single NW, (ii) fusing of two NWs from two pads, and (iii) physical contact between two NWs [11]. In a previous report [11], it was concluded that types (ii) and (iii) dominate. When two NWs are in physical contact, absorbed O_2^- results in upward band bend-
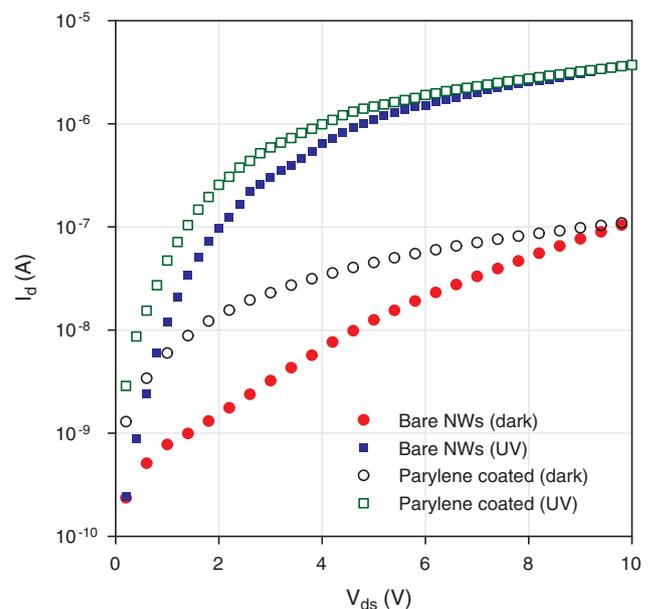

Fig. 7. $I_d - V_{ds}$ plot for a 5 μm gap nanobridge device before and after 0.1 g parylene coating.
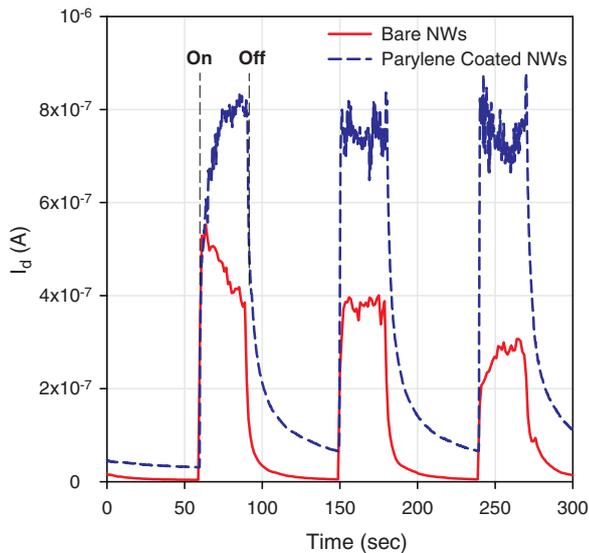
Fig. 8. Plot of I_d vs. time showing the UV photoresponse for the 5 μm nanobridge device from Fig. 7, pre (red solid line) and post (blue dashed line) parylene coating. Devices are exposed to 30 s pulses of UV light with V_{ds} = 3 V and V_g left floating. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

ing and the formation of a double Schottky barrier between two NWs [32]. Since the physically touching NWs far outnumber fused or directly connected bridges, they dominate current flow across the two electrodes, and the double Schottky barrier between the touching NWs will significantly affect the electrical properties of nanobridge devices. The lower Schottky barrier for parylene coated devices can be explained by a reduction of surface sites available for O_2 adsorption.

Fig. 8 is a plot of I_d vs. time for the same 5 μm gap nanobridge device illustrating the UV response pre- and post-parylene coating. Devices were exposed to 30 s pulses of UV light at room temperature in air with the drain to source voltage, V_{ds} = 3 V, and the gate, V_g, left floating. The uncoated device shows an increase in I_d of up to 100× upon UV exposure. Although the parylene coated device exhibits a higher photocurrent, its dark current is also higher and thus UV exposure only produces a 45× increase of I_d. The average rise time (the time it takes for I_d to reach 90% of its maximum value) is roughly 3.6 s for the coated sample vs. 2.8 s for the uncoated one. The decay time (the time it takes for I_d to decrease to 10% of its maximum value) is roughly 30 s for the coated sample vs. 5.6 s for the uncoated one.

Besides the direct photogeneration of carriers, the UV response of ZnO NWs can also be attributed to surface interactions with the ambient, in particular, oxygen adsorption $[O_2\,(g) + e^- \rightarrow O_2^-(ad)]$ and desorption $[O_2^-(ad) + h^+ \rightarrow O_2(g)]$ [33]. In the dark (absence of UV exposure), O_2 molecules are chemisorbed on the NW surface and form a surface depletion region which decreases the conductivity of the NW. The increased dark current in the parylene coated nanobridge devices could be attributed to displacement of surface oxygen or available O_2 adsorption sites by the parylene. Upon UV exposure, electron-hole pairs are immediately photogenerated inside the ZnO NWs. The newly created holes drift towards the surface where they discharge chemisorbed O_2^-, reducing the surface depletion and thus increasing the conductivity. The slower rise/decay time of the UV response observed in the parylene coated nanobridge devices could thus be related to the parylene coating acting as a diffusion barrier and decreasing the desorption and adsorption rates of O_2 on the ZnO surface. Finally, the increased magnitude of photocurrent for the parylene coated device in Fig. 8 can be explained by a decrease in available sites for both O_2 and
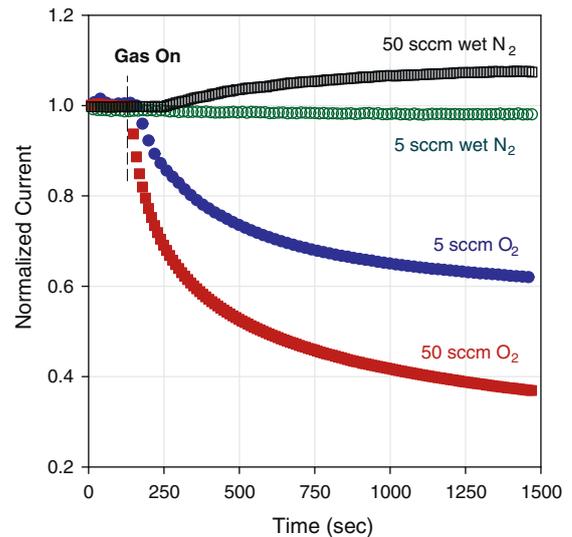

Fig. 9. Plot of normalized current ($I_{d\text{-}O_2}(t)/I_{d\text{-}N_2}$ and $I_{d\text{-wet}}(t)/I_{d\text{-}N_2}$) vs. time showing O_2 and H_2O sensitivity for a 5 μm gap parylene coated nanobridge device. The device was exposed to various O_2 and wet N_2 flow rates and measued at V_{ds} = 3 V in the dark.

H_2O molecule adsorption. During UV illumination the presence of parylene encapsulation reduces the adsorption of H_2O molecules from the ambient. According to Li et al. [33], the existence of H_2O molecules on the surface results in a decrease of photocurrent since the H_2O molecules will attract electrons and holes and decrease the carrier concentration. The O_2 diffusion rate is much higher than H_2O [34]; therefore the UV response will be dominated primarily by O_2.

The plot in Fig. 9 shows O_2 and H_2O sensitivity as a normalized current, I_d ($I_{d\text{-}O_2}(t)/I_{d\text{-}N_2}$ and $I_{d\text{-wet}}(t)/I_{d\text{-}N_2}$), plotted vs. time for a 5 μm gap parylene coated nanobridge device measured at room temperature in the dark with V_g left floating. Prior to each measurement, the sample was purged under 200 sccm of pure N_2 gas for 2 2 h to achieve saturation of I_d at the measurement bias of V_{ds} = 3 V. The O_2 and H_2O concentrations were controlled by changing the ratio of flow rates for wet N_2, dry O_2, and dry N_2 at a fixed total flow rate of 200 sccm. The wet N_2 was generated by passing the dry N_2 through a bubbler filled with DI water at room temperature. Normalized I_d is seen to decrease as a function of time during O_2 exposures and increase during wet N_2 exposures. As discussed, adsorption of O_2 will result in the formation of a surface depletion layer and thus lead to decreased current within the NWs. The formation rate of this depletion layer is faster and saturates at a higher value (lower normalized I_d) when O_2 concentration is higher. Under high concentration of H_2O, obtained by a high flow rate of wet N_2, normalized I_d was observed to increase. The increased conductivity can be explained by the interaction of H_2O molecules with the ZnO NW surface by either direct donation of an electron or by displacement of an adsorbed O_2^- [33].

Compared to a previous report on uncoated nanobridge devices [11], the response to O_2 exposure is attenuated. After 300 s O_2 flow at 50 sccm, the normalized I_d ratio ($I_{d\text{-}O_2}(t)/I_{d\text{-}N_2}$)) was about 1/7 for uncoated NWs compared to only about 1/2 for the parylene coated NWs. This result indicates not only a decrease in O_2 adsorption rate for parylene coated NWs but also a reduction in total amount adsorbed. Interestingly, even though parylene coatings are widely used as an O_2 and H_2O barrier for electronic devices, this device still showed some level of O_2 sensing for the thin parylene coating applied here. This result agrees with the UV sensing results shown in Fig. 7 in which O_2 adsorption and desorption still appear to dominate the sensing properties of parylene coated nanobridge devices as it does in uncoated devices.

4. Summary and conclusion

We have demonstrated three methods for direct functionalization of ZnO NWs with biotin which could be used for selective sensing in a gaseous environment. For vapor or aqueous-phase sensing, protection of ZnO NWs from dissolution is required and parylene-A was investigated as a moisture barrier. We have developed a CVD process for parylene-A and demonstrated that parylene-A coated ZnO NWs do not show any signs of dissolution after 24 h in an aqueous solution. We have also investigated the impact of the parylene-A coating on the electrical performance and sensitivity of ZnO nanobridge devices. Nanobridge devices were fabricated using a novel method in which patterned carbonized photoresist is used for both NW nucleation and electrical contact. A comparison of UV and O_2 sensitivity for uncoated and coated devices revealed that a thin coating of parylene-A attenuates both the UV and O_2 response, suggesting the ability to modulate environmental sensitivity using this non-covalently bound polymer passivation layer.

It has been shown that the available amine groups on parylene-A can bind to the carboxylate groups on BSA by utilizing a crosslinker, such as glutaraldehyde to form a covalent bond [27,35]. Thus, in addition to providing protection of the ZnO NWs for liquid and vapor-phase sensing, parylene-A can also serve as an activation layer for further functionalization. In conclusion, this work demonstrates the novel use of parylene-A coating as an encapsulation layer and as a potential starting platform for general functionalization of ZnO NW devices for selective sensing of arbitrary target species in the liquid or vapor phase.

Acknowledgements

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John F. Conley, Jr. earned a B.S. in electrical engineering in 1991 a Ph.D. in engineering science and mechanics in 1995 from The Pennsylvania State University, where he won a 1996 Xerox Prize for his Ph.D. dissertation. He was with Dynamics Research Corporation from 1995 to 2000 and with the Jet Propulsion Laboratory from 2000 to 2001, where he received an achievement award. From 2001 to 2007 he was with Sharp Laboratories of America. In 2002–2003, he served as an Adjunct Professor at the Vancouver campus of Washington State University. Since 2007, he has been a Professor and an ONAMI Signature Faculty Fellow at Oregon State University, both in the School of Electrical Engineering and Computer Science and the Intercollege Materials Science Program. He has authored or co-authored over 100 technical papers (including several invited) and over 100 conference presentations. He holds 18 U.S. patents. He has presented tutorial short courses on high-k dielectrics at two international conferences and served a guest editor of IEEE TDMR. He has also served on the technical and management committees of several conferences, including General Chair of the 2006 IEEE IRW. Dr. Conley's research interests include atomic layer deposition, high-κ dielectrics, thin film transistors, metal/insulator/metal tunnel diodes, directed integration of nanomaterials and nanodevices, point defects, reliability, and radiation effects in novel electronic materials.