

Control and Detection of Organosilane Polarization on Nanowire Field-Effect Transistors

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ABSTRACT

We demonstrated control and detection of UV-induced 3-aminopropyltriethoxysilane (APTES) polarization using silicon nanowire field-effect transistors made by top-down lithograph technology. The electric dipole moment in APTES films induced by UV-illumination was shown to produce negative effective charges. When individual dipoles were aligned with an externally applied electric field, the collective polarization can prevail over the UV-induced charges in the wires and give rise to an abnormal resistance enhancement in n-type wires. Real-time detection of hybridization of 15-mer poly-T/poly-A DNA molecules was performed, and the amount of hybridization-induced charges in the silicon wire was estimated. Based on these results, detection sensitivity of the wire sensors was discussed.

Hetero-interfaces between organic and semiconductor oxides have attracted extensive attentions^{1–4} due to the critical role of molecule assembly in the sensing electronics involving hybrid structures. APTES⁵ (3-aminopropyltriethoxysilane) and other compounds such as PTS⁶ (*n*-propyltrichlorosilane), OTS⁷ (*n*-octadecyltrichlorosilane), TCTS⁸ (*n*-triacontyltrichlorosilane), OTMS⁹ (*n*-octadecyltrimethoxysilane), and AHT-MS⁹ (*n*-aminoheptadecyltrimethoxysilane) with head-and-tail functional groups are widely used interfacing molecules,

and assembly of these molecules is essential in surface-modification technologies. Silanization of oxidized semiconductor surfaces is a commonly employed scheme for functionalization of sensors. The functional groups would then provide binding sites for attachment of probe molecules, such as single-strand DNA (ssDNA), on the semiconductor sensing devices. The nanowire-based sensors have been demonstrated^{10–14} as an ultra sensitive detector for probing molecular charges at the wire surface. However, surface modification of the functional groups on the nanowire surface is not a trivial task. Extensive studies in the surface-modification were reported in the past years,^{5–9} but issues concerning monolayer molecule ordering in terms of the electric dipole moment remain unexplored. Taking APTES as an example, in this study, we proposed a simple method to align the molecule dipoles, and the degree of alignment was examined by underneath Si-nanowire (SiNW) field effect transistors. This method provides a sensitive way for structure investigation of few molecules at the nanometer scale, which is otherwise unfeasible by the present-day examination tools.

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The idea of using SiNWs fabricated by a standard “top-down” semiconductor process has unrivaled advantages including well-controlled wire properties, no positioning issue, easy integration with micro-fluidic channels, and compatibility with the present-day semiconductor process. Sensors with SiNWs fabricated with “top-down” processes are thus readily available for a smooth technology transfer into the industry.

In the nanowire sensors, variation in molecular charge is reflected in a change in the wire conductance. Just as in conventional metal-oxide semiconductor transistors, the polarized molecules play the role of the metal gate, and the polarization of the molecules is directly manifested by wire conductance. These sensors hold great potential for various applications due to their intrinsic advantages such as having high sensitivity with a minimal requirement for the amount of target molecules, capability for multiplex parallel processing,¹⁵ and no tedious sample labeling process required. In this work, SiNW sensors were modified with APTES on the surface. With this device, the effect of UV illumination on the APTES charge configurations in vacuum was investigated and was found to produce negative effective charges. Furthermore, we proposed and demonstrated that the individual molecule dipoles could be aligned by an applied electric field to yield a collective dipole moment which can induce conduction carriers in the nanowires by gating effect. The induced carriers can, in turn, compensate and even prevail over the UV-induced charge carriers in the wires and give rise to an abnormal photoinduced resistance enhancement in n-type nanowires. This study demonstrates a simple method not only for alignment of surface molecules but also for evaluation of the quality of assembly at the molecular level. The method provides a sensitive way for structure investigation on a small number of molecules at the nanometer scale, which cannot be achieved by the present-day examination tools.

As an application of the APTES modified SiNWs, detection of hybridization between 15-base single-strand DNA (ssDNA) molecules was demonstrated. The SiNWs were made using silicon-on-insulator (SOI) wafers, which offer a layer of high quality single-crystal silicon (referred to as “device layer”) separated from the bulk substrates by a layer of buried oxide (abbreviated as “BOX layer”). The SOI wafers are presently the basis of state-of-the-art metal-oxide-semiconductor field-effect-transistors (MOSFETs) in the mainstream semiconductor industry. SOI-based SiNW sensors were reported previously¹⁴ and demonstrated for detection of hybridization of 12-mer oligonucleotides with a sensitivity of 25pM. In a recent paper, Winkelmann et al.¹⁶ reported detection of photoelectric effect of porphyrin derivative. Four-inch p-type SOI wafers with 50 nm-thick silicon top layer and 400 nm-thick buried oxide layer were used in this work and the resistivity of the device layer was 100 $\Omega\cdot\text{cm}$. Figure 1 shows a scanning electron microscope (SEM) image of a typical SiNW FET. The wires were defined by standard electron beam lithography and photolithography processes and were thermally oxidized to form a SiO₂ insulating layer which inhibited charge transferring

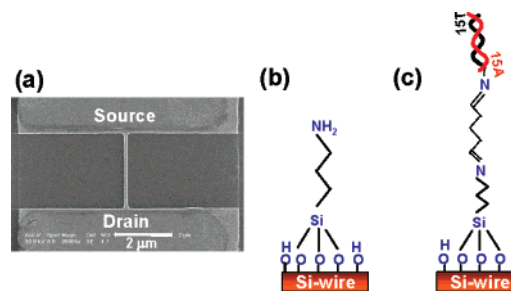


Figure 1. (a) SEM image of the SOI-based Si-wire FET device. The width, length and thickness of the wire are 100 nm, 3 μm , and 30 nm, respectively, and the wire is covered by a 10-nm thick thermally oxidized SiO₂ layer. The schematic drawings illustrate (b) APTES surface modification and (c) detection of DNA-hybridization.

between attached molecules and wires. Detailed device fabrication procedures can be found in the Supporting Information. Separately, we also made PDMS (polydimethylsiloxane) slabs with fluid inlet/outlet on the top and micro fluidic channels on the bottom.^{17,18} These PDMS slabs were to be aligned and bonded to Si-chips, so that buffer solution containing target DNA could be introduced and guided to the location of the Si-nanowire sensors. The fluidic channels were 100 μm wide and 100 μm high. DNA solution was injected into the device with a syringe via the PDMS fluidic channel across the nanowire sensors.

Prior to the immobilization of the capture DNA molecules, the silicon nanowires were first subjected to an APTES modification process^{19–22} to form a hydrophilic surface with hydroxyl groups on the silicon oxide surface. APTES contains triethoxysilane at one end for binding to the hydroxyl group on the SiO₂ surface and the NH₂ functional group at the other end for bindings with glutaraldehyde which in turn covalently binds to amine-modified ssDNA molecules via the nitrogen atom. Repeated atomic force microscope (AFM) inspections of the modified/unmodified border show a thickness of about 0.7 nm for the APTES layers, verifying formation of a uniform layer. A detailed description of this process is given in the Supporting Information.

The first part of this work is devoted to the effect of ultraviolet light (UV) illumination on APTES molecules. After APTES modification, the SiNW source-drain current exhibited dramatic enhancement upon UV illumination. Figure 2 shows a comparison of the responses for a p-type SiNW before and after modification. The response for n-type SiNWs can be found in the Supporting Information. The UV was generated by a light emit diode (LED) with a center wavelength of 405.8 nm and a half-width at maximum-height (FWMH) of 15 nm. UV-LED is a compact and convenient light source whose light intensity can be easily varied by bias current. To prevent influences from moisture or air contaminations, the measurement was carried out in a vacuum pressure of about 10^{−4} mbar. Before modification, the SiNW exhibited weak dependence on UV intensity, and the source-drain current increased with the light intensity. After modification, the current also increased with light intensity but with a much stronger dependence. It is worth noting that the net current contributed from APTES (i.e.,

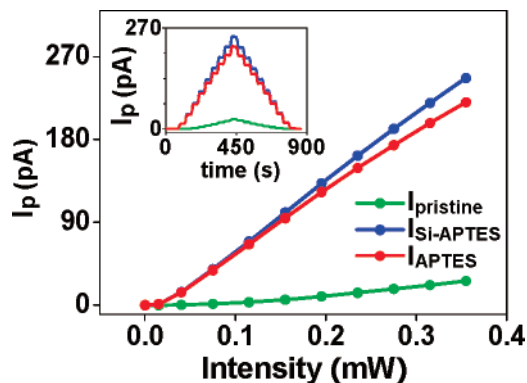


Figure 2. Response in source-drain current of a p-type SiNW-FET upon UV-LED illumination with varying light intensity controlled by the LED current. The green and blue curves are wire current before and after APTES-modification, respectively, and the red curve indicates the portion of current induced by APTES polarization alone. The SiNW was biased at 0.5 V with a back-gate voltage of 0.5 V, and the current was measured with a lock-in amplifier operated at 0.777 Hz. Inset shows the same measurement data but as a function of time, in which the UV intensity was increased and then decreased to reveal reversible behavior.

subtracting current of pristine wires from that of modified-wires, shown in red in Figure 2) increased with light intensity, indicating that the surface molecules are responding to the UV light. The most noteworthy feature is that the effect of UV on APTES was reversible as shown in the inset of Figure 2: the current returned exactly to the original value as the UV-light intensity decreased, suggesting prompt recovery of the charge configuration without permanent molecular conformation change. Surprisingly, for an APTES-modified n-type SiNW, we found that the resistance increased upon UV-light illumination, as shown in Figure 3a. This contradicts our previous understanding that UV illumination always decreases resistance of semiconductor wires.

Upon UV illumination, two effects take place simultaneously as illustrated in Figure 4: (A) UV illumination excites additional carriers in the wires, and thus decreases wire resistance. This is true for both p-type and n-type wires, whether pristine or modified wires. (B) UV may induce APTES polarization and generate a layer of surface charges, which may accumulate or deplete carriers in the wire depending on the type of the wires. For p-type wires, as illustrated in Figure 4a, the induced negative charges accumulate hole-carriers and increase the current; this is the case shown in Figure 2. For n-type wires (see Figure 4b), the negative charges deplete conduction electrons in the wires, producing an effect opposite to that of UV on the wire itself. Depending on the strengths of these two competing effects, the resultant resistance may increase or decrease. The increased resistance shown in Figure 3a is a manifestation for the case in which effect B is stronger than effect A. The same type of experiment using p-type SiNWs is described in the Supporting Information.

In the attachment of APTES, the molecules are not likely to be well assembled into a highly ordered monolayer.²³ Depending on reaction conditions, chemistry of the organosilane, and intra-/intermolecular hydrogen bonds interactions between ligands, a number of surface-binding struc-

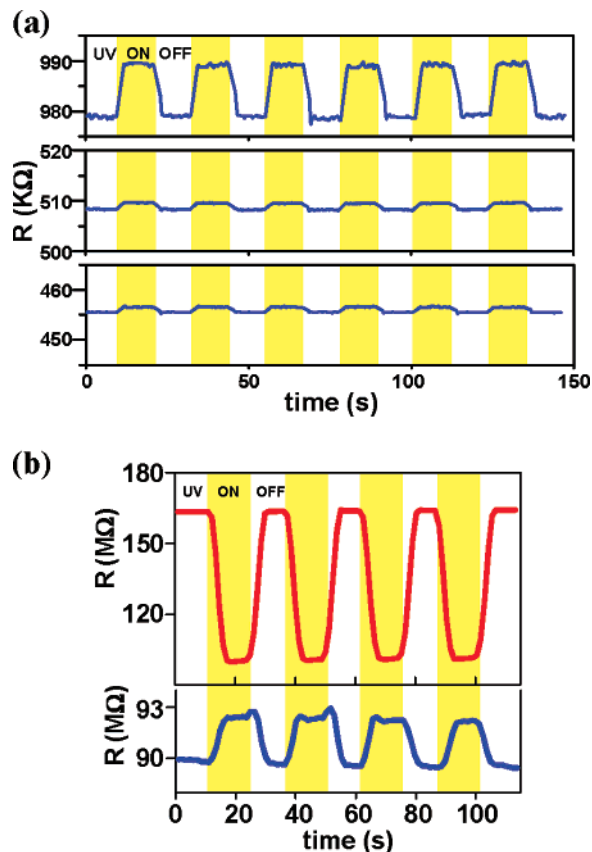


Figure 3. Resistance responses upon UV-illumination (marked by yellow shades) of APTES-modified n-type Si-nanowires. (a) responses at $V_g = -9.7$ V (top), 0 V (center), and +10 V (bottom panel). Note that the resistance increases upon UV-illumination. (b) UV responses of another wire before (top panel) and after (bottom panel) subjecting to the “dipole alignment” process. In the former, the resistance decreases under UV-illumination, indicating that effect A dominates. In the latter, effect B surmounts effect A, and the device shows a reversed response. Both curves were taken at $V_g = 0$. The resistance after dipole alignment process is decreased. This is probably due to presence of residual positive charges in the bulk handling wafer, as explained in the Supporting Information. The intensity of the illumination is the maximum intensity used in Figure 2, and is about 0.36 mW.

tures²⁴ and molecular configurations²⁵ may form. In the as-modified molecule layer, net polarization of the layer is quite insignificant because of the disordered configuration (as illustrated in Figure 4c), and the effect of UV illumination on the molecule is small; that is, effect A is stronger than effect B, as shown in the top panel of Figure 3b. This is illustrated as the red dashed line in Figure 4b. Since assembly of APTES molecules is a prerequisite for the UV-induced net polarization of the layer, we introduced a “dipole alignment” process which can significantly enhance the UV response. In this process, as illustrated in Figure 4d, the back-gate voltage is ramped to a large positive voltage and then ramped back to zero. The result is depicted in the bottom panel of Figure 3b and also illustrated as the blue solid line in Figure 4b, which shows alignment of the individual molecules to the electrical field. In this case, UV-induced APTES dipoles may eventually cause a reversed response in the resistance of n-type SiNWs. This phenomenon is attributed to the collective polarizability of the APTES layer.

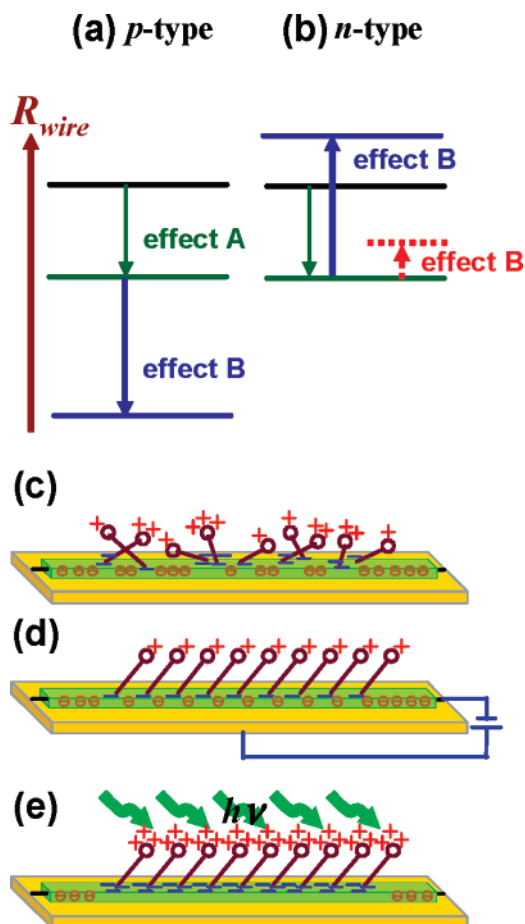


Figure 4. (a and b) Resistance changes upon UV illumination. Two effects as explained in the text are illustrated and their sum gives the resultant resistance changes. The black lines indicate the resistance of the modified wires in the absence of UV. For p-type wires (a), the resistance always decreases, whereas for n-type wires (b), the resistance may increase (solid blue line) or decrease (dashed red line) depending on the strength of induced APTES polarization. (c–e) Schematic illustration of APTES polarization on a n-type SiNW: (c) random orientation of molecules with internal dipoles; (d) because of the internal field, a high E-field helps to align APTES; (e) UV illumination strengthens the internal dipoles.

We found that this ordered state may persist for a few days and then relax to a disorder state. In the Supporting Information, results of dipole alignment using different positive back-gate voltages are presented, and a control experiment with negative back-gate voltages is described. The above results establish a foundation for the study of organic molecule polarization on a nanometer scale without involving complicated charge-transfer mechanism. It provides a method for quantitative analysis of degree of ordering in the assembly and thus a useful link between biological functionality and electronic signal.

In subsequent experiments, detection of hybridization between 15-mer poly-T and poly-A single-strand DNA molecules (abbreviated as 15T and 15A, respectively) with SiNWs was carried out.^{26–30} After APTES modification, the devices were further modified with poly-T ssDNA (1 μ M in Tris-HCl, pH 8). This process is described in the Supporting Information. Probe DNA molecules (poly-A ssDNA) were then injected into the micro-fluidic channel, and the wire

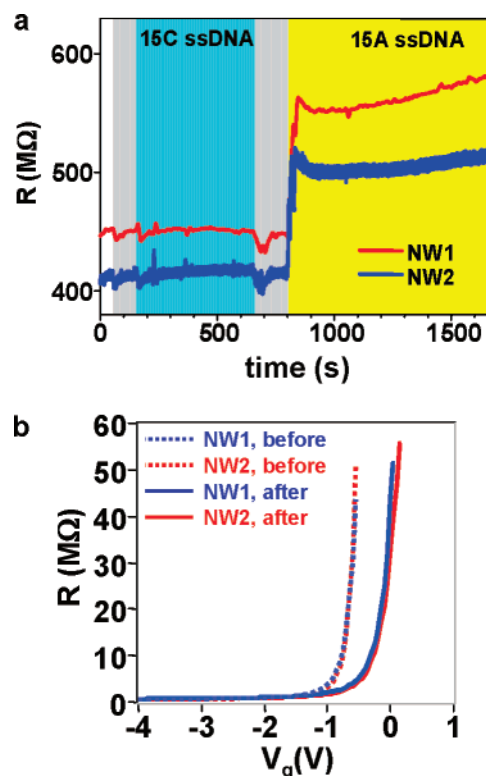


Figure 5. Measured nanowire resistance in response to the injection of ssDNA molecules. Prior to the measurement the wires were modified with 15T ssDNA. (a) Subsequently, 15C ssDNA (cyan shade) and buffer (gray shade) solution were added at $t = 150$ and 650 s, respectively, and the wire showed no change in resistance. At $t = 800$ s, 15A ssDNA (yellow shade) was added and an abrupt increase in the resistance was observed. The blue and red curves are for two different SiNWs measured simultaneously, and they showed the same behavior. (b) The resistance of two p-type wires on the same chip measured simultaneously as a function of gate voltage before addition of 15A ssDNA (dotted curve) and after hybridization (solid curve).

resistance was measured and recorded in situ. In our SOI-based SiNW sensing devices, several wires were made in the neighborhood on the same chip with similar electrical properties that could be measured simultaneously. This design provides a check for local environment variation. Figure 5a shows the measured resistance of two n-type SiNWs modified with 15T ssDNA. As a control experiment, 15-mer poly-C ssDNA (20 pM in Tris-HCl, pH 8) was first added into the system, and no change in the wire resistance was found. Subsequently, 15A ssDNA (20 pM in Tris-HCl, pH 8) was introduced to hybridize with the immobilized 15T ssDNA, and a clear increase in the resistance of both wires was observed. The increased resistance in the n-type SiNW implies an increased negative charge on the wire surface. This is consistent with the fact that DNA molecules with an isoelectric point^{31,32} of about 5.0 should possess negative charges in a pH 8 electrolyte. Figure 5b shows resistance of two p-type wires measured simultaneously as a function of gate voltage before and after introduction of 15A ssDNA. The effect of hybridization on the wire resistance can be represented by a shift in gate voltage ΔV_g of 0.535 V. A gate capacitance C_g of 110 aF³³ would correspond to an addition of conduction carriers of $\Delta V_g C_g \approx 370e$.

The Debye length, defined as the distance over which mobile charge carriers screen out electric fields, in the solution is given by $\lambda_D = [\epsilon k_B T / e^2 N_i]^{1/2}$. Here ϵ is the dielectric permittivity, k_B is the Boltzmann constant, T is the absolute temperature, e is the electron charge, and $N_i = 2N_a I$ is the ionic concentration of the solution, N_a is the Avogadro constant, and I is the ionic strength. This length is estimated to be about 1 nm, which is considerably shorter than the DNA-to-SiNW distance taking into account the lengths of APTES and glutaraldehyde molecules. The fact that DNA hybridization is still detectable suggests that hybridization may induce charge redistribution in the DNA–glutaraldehyde–APTES molecule chains. This should otherwise be undetectable without the chemical bonding process. On the other hand, the Debye length inside the SiNWs is an important factor regarding the charge sensitivity of the SiNWs. The equation has the same form as that for molecules in solutions, but the ionic concentration N_i is replaced by the doping concentration instead. For our SiNWs, a very low doping concentration of $\sim 10^{14}/\text{cm}^3$ was used so as to yield a long Debye length of ~ 400 nm, much longer than the thickness and the width of our SiNWs. Therefore, we are working in the full-depleted region which ensures a high charge sensitive. This is in line with the fact that our experiments on wires with widths ranging between 70 and 400 nm did not show apparent differences in detection sensitivity.

The point of zero charge of silicon oxide is 2.2. In our experiment with an electrolyte pH value of 8.0, cation concentration is high near the negatively charged oxide surface and decays rapidly with distance whereas the anion concentration increase gradually, and both of them reach bulk ion concentration at approximately 5 nm. Since DNA molecules are negatively charged, they have negligible possibility in entering the vicinity of the wire surface within the range of Debye length. This can explain the insensitive of the SiNW to the introduction of 15C DNA shown in Figure 5, and the sensing signal is mostly contributed by the hybridization.

In summary, SOI-based nanowire FET has been utilized as a sensor to detect UV-induced APTES polarizations and hybridization-induced charges in poly-T/poly-A 15-mer DNA molecules. The effect of UV illumination on the APTES charge configurations in vacuum was investigated and shown to produce an electric polarization yielding negative effective charges. Furthermore, when APTES dipoles were aligned, this effect could prevail over the UV-induced charge carriers in the wire and gave rise to an abnormal photoinduced resistance enhancement in n-type SiNWs. This dipole moment change can be induced by photoexcitation, molecular configuration/orientation change, bond formation, or local environment change. To demonstrate application of the modified SiNW FETs, hybridization of 15-mer poly-T/poly-A DNA molecules was detected in real time and was confirmed by a control experiment. The hybridization induced charges can be simulated by a shift in the gate voltage, from which the effective amount of induced charge

was estimated. This result provides a foundation for quantitative analysis for single-molecule sensing.

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Supporting Information Available: Response for n-type SiNWs, results of dipole alignment using different positive back-gate voltages, and control experiment with negative back-gate voltages. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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