

Highly Stable Operation of Metal Oxide Nanowire Transistors in Ambient Humidity, Water, Blood, and Oxygen

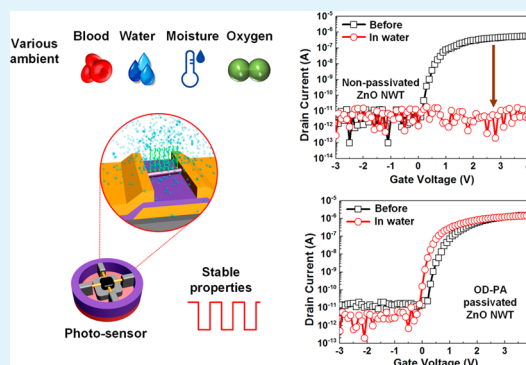
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ABSTRACT: The capability for robust operation of nanoscale transistors under harsh environments is equally important as their operating parameters such as high on-currents, high mobility, and high sensing selectivity. For electronic/biomedical applications, in particular, transistor operation must be stable under diverse conditions including ambient humidity, water, blood, and oxygen. Here we demonstrate the use of a self-assembled monolayer of octadecylphosphonic acid (OD-PA) to passivate a functionalized nanowire transistor, allowing the device to operate consistently in such environments. In contrast, without passivation, the characteristics (especially the threshold voltage) of identical nanowire transistors were dramatically altered under these conditions. Furthermore, the OD-PA-passivated transistor shows no signs of long-term stability deterioration and maintains equally high sensing selectivity to light under the harsh environments because of OD-PA's optical transparency. These results demonstrate the suitability of OD-PA passivation methods for fabricating commercial nanoelectronics.

KEYWORDS: sensing selectivity, nanowire transistor, self-assembled monolayer, environmental stability, hydrophobicity



INTRODUCTION

Over the years, nanoscale devices have attracted tremendous attention because of their high integration, high speed, high flexibility, and transparency. These devices often make use of various carbon-nanotube, nanowire, and graphene materials as highly conductive electrodes or semiconductors. For transistor and sensor applications, semiconducting metal oxide nanowires are particularly attractive because of their simple synthesis, metal–semiconductor transition, high transconductance, and large variation in conductivity as a function of environmental parameters.^{1–3} Concerning such nanoscale transistors, the investigation is aimed at achieving high-field-effect mobility (μ_{eff}), high on-current (I_{on}), high on–off current ratio ($I_{\text{on}}/I_{\text{off}}$), and steep subthreshold slope (SS),^{4–7} and extensive studies have been performed to improve transistor characteristics by annealing,⁸ doping,⁹ and passivation¹⁰ of the active area of the oxide nanowire devices. Metal oxide semiconductors used in these devices are notoriously sensitive to surface effects; their transistor and emission characteristics change with the working conditions, e.g., temperature, light, gas, humidity, water, and blood. Thus, the use of nanoscale metal oxide semiconductor materials in commercial applications such as displays, transistors, gas sensors, and biosensors remains difficult, and intensive research into the reliable and selective passivation of the surface of the nanoscale devices is necessary.

Various recent studies have demonstrated the use of a passivation layer to improve device stability, using inorganic and/or organic passivation layers on the semiconductor channel or light-emitting area of devices and showing improved

stability with exposure to different ambient environments such as bias stressing and light illumination. More specifically, enhanced transconductance of field-effect transistors was obtained using SiO_x passivation;¹¹ surface passivation with a self-assembled monolayer (SAM) of 1-octadecanethiol was used to realize low-noise electronics based on nanowires,¹⁰ and surface passivation by L-lysine was introduced to provide reproducible ultraviolet (UV) photoresponse in humid air and reduce the influence of H_2O molecules, which cause gradual degradation of the UV photocurrent of ZnO nanowires.¹²

At this stage of research, an important goal is the fabrication of robust metal oxide nanowire transistors (NWTs) showing consistent operation in ambient humidity, water, blood, and oxygen. Commercial devices integrated with metal oxide based NWTs currently being used in these operating environments as gas sensors or biosensors may not fully meet the requirements yet, and thus more reliable sensors are still being sought after. In such sensors, it is essential that the link between what is being sensed and the sensor's response be unambiguous. Because the conductivity of a metal oxide nanowire is altered by ambient conditions in a complex manner, it can be difficult to connect a change in the conductivity to a change in the sensor's target.

In this study, we have investigated the environmental stability of ZnO NWTs using an OD-PA SAM as a nanowire-surface-

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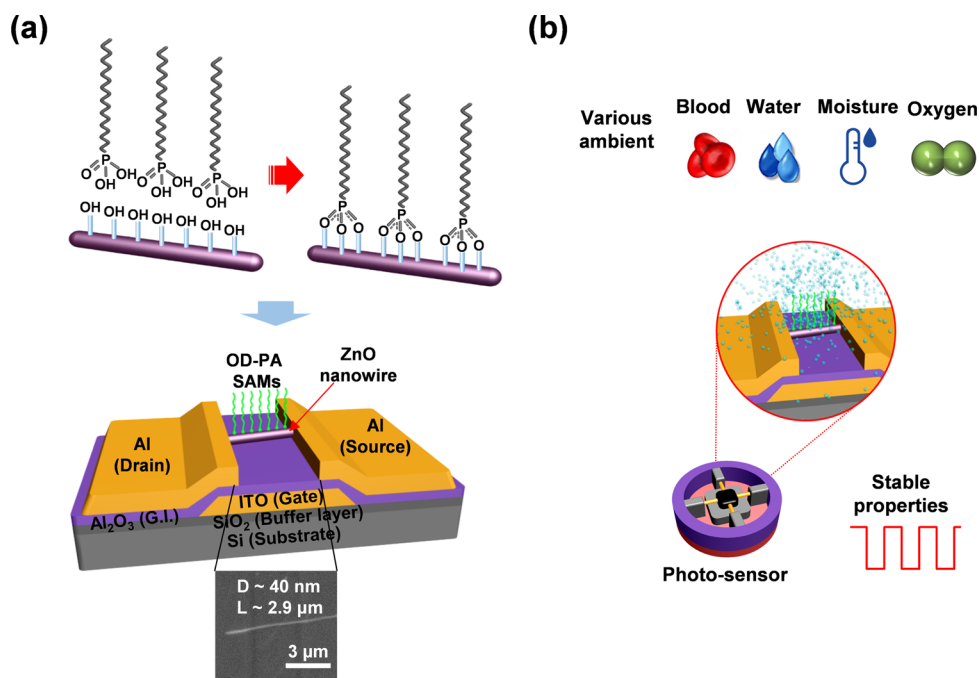


Figure 1. (a) Schematic of the OD-PA SAMs on ZnO nanowires and the bottom-gate-structured ZnO NWT. The inset shows a representative FE-SEM image of the OD-PA-passivated nanowire-channel region between the source and drain electrodes. (b) Diagram of moisture, water, blood, and oxygen exposures on the channel region of ZnO NWTs.

passivation layer. The transistor characteristics before and after application of the SAM passivation layer were compared under different humidity, water, blood, and oxygen conditions. Selective light-sensing properties of ZnO NWTs with a SAM passivation layer were characterized. The conductivity of the metal oxide nanowire was changed only by light and not the relative humidity (RH), water, or oxygen environment. This work demonstrates the use of a SAM to enable selective sensing of an NWT under varied environments.

EXPERIMENTAL SECTION

ZnO nanowires were grown on gold-coated silicon substrates using a conventional chemical vapor deposition (CVD) method in a two-temperature-zone furnace from ZnO powder. Graphite (Alfa Aesar) was added to ZnO powder (99.999%, Sigma-Aldrich) at a ratio of 1:1 (weight percent), and 20 nm gold nanoparticles acting as catalysts for nanowire growth were coated on the surface of the silicon substrate. The temperatures in the ZnO powder and gold-coated silicon substrate zones were increased to 1060 and 650 °C, respectively. During nanowire growth, a mixed gas of argon (48 sccm) and dioxygen (2 sccm) was supplied to the CVD tube for 25 min. The as-grown ZnO nanowires had an average diameter and length of ~40 nm and ~10 μm, respectively.

To remove inorganic and organic contaminants on the as-grown ZnO nanowires to improve the self-assembled condition, the ZnO nanowires were cleaned in semiconductor-grade isopropyl alcohol (SG-IPA) for 5 min and exposed indirectly to UV/ozone treatment with a shielding mask. The cleaned ZnO nanowires were immersed in octadecylphosphonic acid (OD-PA; 2 mM, Alfa Aesar) in SG-IPA for 1 h. OD-PA, which contains hydrocarbon chains, self-assembled on the channel region of the ZnO nanowire. OD-PA passivated the ZnO nanowires, which were then rinsed with fresh SG-IPA and deionized water twice.

The bottom-gate-structured ZnO NWTs were fabricated with a 100-nm-thick indium–tin oxide (ITO) thin film as a gate electrode, a 30-nm-thick Al₂O₃ layer (with an effective dielectric constant $k_{\text{eff}} \approx 9$) as a gate insulator, a single ZnO nanowire (with a band gap of 3.37 eV) as a semiconductor channel, and a 70-nm-thick aluminum thin

film as a source/drain electrode. A randomly distributed ZnO nanowire channel was formed by dispersing the IPA solution with nanowires on a gate-dielectric/bottom-gate-electrode-formed substrate. Radio-frequency and direct-current sputtering methods were used to deposit the ITO and aluminum, respectively. A thermal atomic layer deposition method was used to form the Al₂O₃ thin film. Note that in order to improve the contact resistance, before deposition of the source/drain metal, the UV/ozone was exposed to remove OD-PA on the contact region of the source/drain electrodes. In order to prevent the electrical conductivity of liquids, the aluminum source/drain electrodes were passivated by a 100-nm-thick SiO₂ thin film.

Transistor characteristics of nonpassivated and OD-PA-passivated ZnO NWTs were measured using a semiconductor device analyzer (Agilent B1500A) in ambient air. Prior to measurements, the NWTs were exposed to various conditions: two RH (73% and 95%), water (immersion), blood (immersion), and oxygen (exposure) for 1, 1, 1, and 10 (30) min, respectively. The photosensitivity and gas sensitivity of the devices at three levels of illumination intensity [0 (dark), 1200, and 2100 lx] and at two oxygen gas flow rates (100 sccm for 10 and 30 min in vacuum), respectively, were also examined. The transistor characteristics of the devices were measured during light exposure. Note that μ_{eff} was derived from the calculated gate-to-channel capacitance $C_i = 2\pi\epsilon_0 K_{\text{eff}} L / \cosh^{-1}(1 + t_{\text{ox}}/r)$ using $\mu_{\text{eff}} = (dI_{\text{ds}}/dV_{\text{gs}})(L^2/C_i)(1/V_{\text{ds}})$, K_{eff} is the effective dielectric constant of the Al₂O₃ thin film (~9), t_{ox} is the thickness of the Al₂O₃ thin film (~30 nm), r is the diameter of the ZnO nanowire (40 nm), L is the channel length of the ZnO nanowire (~2.9 μm), and V_{ds} is the drain/source voltage (0.5 V). The oxygen and water-vapor permeabilities of the SAM of OD-PA passivation on ZnO (device size of $5 \times 5 \times 0.1 \text{ cm}^3$) were characterized by permeability analysis (Mocon Inc. OX-TRAN model 2/21 and PERMATRAN-W model 3/33, respectively). The oxygen and water-vapor permeabilities were measured at 23 and 37.8 °C with 100% RH, respectively.

RESULTS AND DISCUSSION

The surface of the ZnO nanowires was functionalized with a SAM of OD-PA. Strong chemical bonds are expected to form between the metal oxide surface and phosphonates via heterocondensation and metal–ligand coordination (M–O–

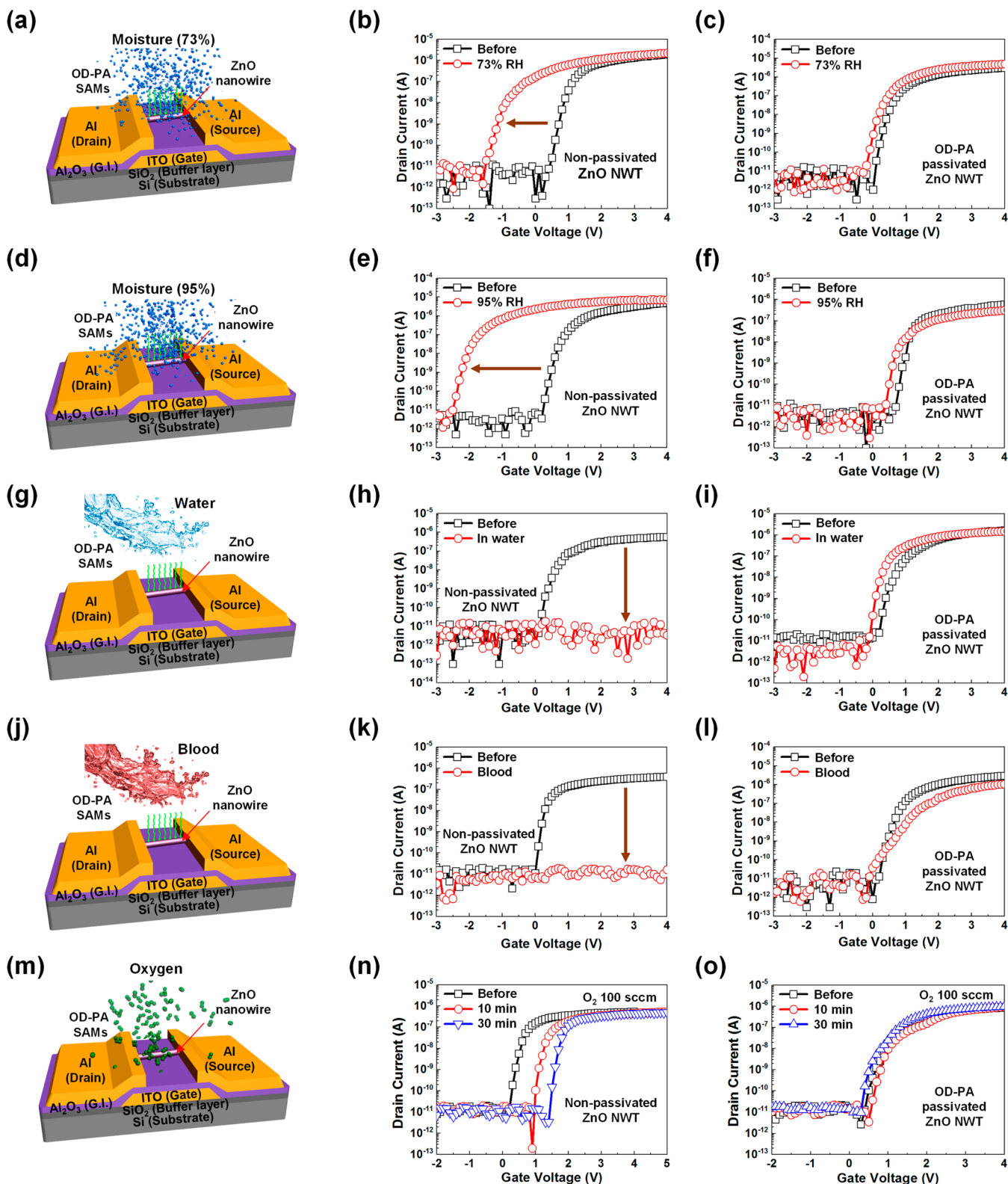


Figure 2. Schematic of 73% RH (a), 95% RH (d), water (g), blood (j), and oxygen (m) exposures on nonpassivated and OD-PA-passivated ZnO NWTs. Log-scale I_{ds} - V_{gs} characteristics of representative nonpassivated and OD-PA-passivated ZnO NWTs after exposure to ambient environments of 73% RH (parts b and c, respectively), 95% RH (parts e and f, respectively), water (parts h and i, respectively), blood (parts k and l, respectively), and oxygen (parts n and o, respectively).

P), as shown in Figure 1a. A single passivated nanowire was used in a bottom-gated ZnO NWT as a semiconductor channel, with ITO as the gate electrode, Al_2O_3 as the gate dielectric, and aluminum as source/drain electrodes, also shown in Figure 1a.

The inset shows a representative field-emission scanning electron microscopy (FE-SEM) image of the nanowire-channel region between the source and drain electrodes; the nanowire-channel diameter and the length of a representative ZnO NWT

were 40 nm and $\sim 2.9 \mu\text{m}$, respectively. ZnO NWTs without OD-PA passivation were also fabricated to observe the difference in transistor characteristics relative to the OD-PA-passivated devices. Figure 1b is a schematic of the various conditions applied during characterization of the NWT, including exposure to moisture, water, blood, and oxygen, as well as light. The Mocon Inc. test was performed to evaluate the reliability of the nonpassivated and OD-PA-passivated ZnO. The low oxygen and water-vapor permeabilities ($1.57 \text{ cm}^3 \cdot \text{mil} \cdot \text{m}^{-2} \cdot \text{day}^{-1}$ and $0.88 \text{ g} \cdot \text{mil} \cdot \text{m}^{-2} \cdot \text{day}^{-1}$, respectively) of the SAM of OD-PA-coated ZnO were obtained to compare with those of the nonpassivated ZnO ($3.48 \text{ cm}^3 \cdot \text{mil} \cdot \text{m}^{-2} \cdot \text{day}^{-1}$ and $1.75 \text{ g} \cdot \text{mil} \cdot \text{m}^{-2} \cdot \text{day}^{-1}$, respectively).

The switching behavior of the NWT devices were characterized using drain/source current versus gate-source voltage ($I_{\text{ds}}-V_{\text{gs}}$) measurements (with $V_{\text{ds}} = 0.5 \text{ V}$) before and after the device was exposed to various environments: two RHs (73% and 95%), water, blood, and oxygen. Schematics of the environment are shown in the first column of Figure 2a,d,g,j,m, representative log-scale $I_{\text{ds}}-V_{\text{gs}}$ characteristics of the nonpassivated devices are shown in the second column (Figure 2b,e,h,k,n), and representative characteristics of the passivated devices are shown in the third column (Figure 2c,f,i,l,o). In all cases, the nonpassivated NWT showed dramatic changes in behavior, while the behavior of the passivated NWT was stable in response to exposure to the varied environments. A more detailed discussion of these measurements is conducted below.

It is well-known that surface adsorbents can shift the conductivity and other electrical properties of metal oxide nanowires. Adsorbed H_2O molecules act as electron donors and thus are expected to shift the threshold voltage (V_{th}) in the negative direction.¹³ Indeed, we observe that, with humidity, V_{th} of the nonpassivated device shifts toward negative gate voltage, by 1.63 V for 73% RH and 2.49 V for 95% RH, shown in the first two rows of Figure 2. The negative shifts of V_{th} on nonpassivated ZnO NWTs in ambient environments of 73% and 95% RH are attributed to adsorbed H_2O molecules on the surface of the ZnO nanowires, which act as electron donors. Although the number of electron donors on the nanowires varied, the SS was unchanged because the interface state between the gate dielectric and nanowire was most likely unaffected.^{14,15} Other transistor properties such as $I_{\text{on}}/I_{\text{off}}$, SS, and μ_{eff} remain almost the same. In contrast, the OD-PA-passivated ZnO NWT showed a constant $I_{\text{ds}}-V_{\text{gs}}$ curve with only a small change in V_{th} .

The water content was increased further by dipping of the ZnO NWTs in water, as shown in the third row of Figure 2g,h,i. The nonpassivated ZnO NWTs lost their transistor characteristics entirely, whereas those of the OD-PA-passivated ZnO NWTs were stable. OD-PA was highly effective in protecting the transistive behavior of the ZnO NWTs under ambient humidity and water because the functional group ($-\text{CH}_3$) of OD-PA SAMs is hydrophobic, isolating the nanowire's surface from H_2O molecules. The passivation decreased the adsorption-desorption effect on the channel surface of the metal oxide nanowires.¹⁶ These results indicate that the OD-PA-passivated ZnO NWTs can provide stable transistor characteristics under harsh operating conditions, including ambient humidity and water.

The effects of humidity and water on the switching properties of the passivated and nonpassivated ZnO NWTs are summarized in Figure 3. Figure 3 shows the average and standard deviation of four major transistor parameters (ΔV_{th} ,

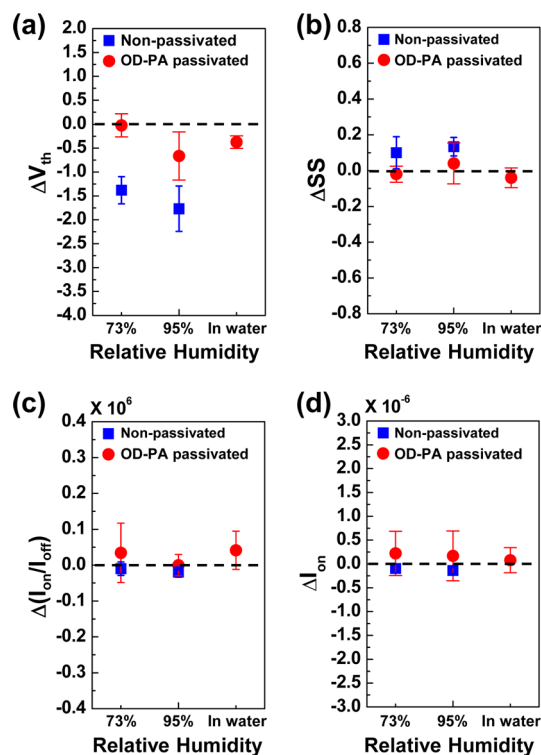


Figure 3. Changes and standard deviations of V_{th} (a), SS (b), $I_{\text{on}}/I_{\text{off}}$ (c), and I_{on} (d) for the representative nonpassivated and OD-PA-passivated ZnO NWTs after exposure to ambient 73% and 95% RH and water.

ΔSS , $\Delta I_{\text{on}}/I_{\text{off}}$ and ΔI_{on}), which were calculated based on five ZnO NWTs before and after OD-PA passivation. Note that the deviation (Δ) of these parameters is the difference between the measured values of the devices before and after exposure to the three different conditions (73% and 95% RH and water). Without OD-PA passivation, ΔV_{th} exhibited negative shifts of $-1.38 \pm 0.28 \text{ V}$ at 73% RH and $-1.77 \pm 0.48 \text{ V}$ at 95% RH. After dipping in water, the ΔV_{th} characteristics could not be measured. On the other hand, in the NWTs with OD-PA passivation, ΔV_{th} showed negligible negative shifts of $-0.02 \pm 0.24 \text{ V}$ at 73% RH, $-0.66 \pm 0.50 \text{ V}$ at 95% RH, and $-0.38 \pm 0.13 \text{ V}$ in water. The ΔSS values of the nonpassivated ZnO NWTs were slightly degraded by 0.10 ± 0.09 in 73% RH and 0.13 ± 0.05 in 95% RH. Meanwhile, the ZnO NWTs after OD-PA self-assembly exhibited unchanged average values of ΔSS ($-0.02 \pm 0.04 \text{ V/dec}$ in 73% RH and $0.04 \pm 0.11 \text{ V/dec}$ in 95% RH). For the ΔI_{on} and $\Delta I_{\text{on}}/I_{\text{off}}$ values, the nonpassivated and OD-PA-passivated devices were not changed with 73% and 95% RH. The ΔSS , $\Delta I_{\text{on}}/I_{\text{off}}$ and ΔI_{on} values could not be extracted from the nonpassivated ZnO NWT in water because the transfer curve of the device showed only low currents over the entire measurement range of V_{g} . In contrast to the unpassivated devices, all parameters of the OD-PA-passivated ZnO NWTs exhibited unchanged values before and after exposure in water.

The effect of blood exposure on OD-PA-passivated and nonpassivated ZnO NWTs was also investigated, as shown in the fourth row of Figure 2j,k,l. The information about an arterial blood gas is crucial for physicians, nurses, respiratory therapists, and other health care personnel. Similar to their behavior with water, after dipping into blood, the nonpassivated ZnO NWTs exhibited a low off-current level over the entire

range of applied gate voltage (Figure 2k), while the OD-PA-passivated ZnO NWTs exhibited stable transistor characteristics (Figure 2l). These results suggest that such passivated transistors have great potential as biomedical sensors for gas detection in blood owing to the stability exhibited by the present device in liquid environments.

The last row of Figure 2m,n,o shows the effect of oxygen ambient on the nonpassivated and OD-PA-passivated devices. V_{th} of the nonpassivated ZnO NWT device was shifted by 0.71 and 1.26 V in 10 and 30 min in the positive direction, respectively. Like H_2O , O_2 can adsorb on the surface of the metal oxide channel. Oxygen can also diffuse into the nanowire, filling oxygen vacancies (V_O , V_O^+ , and V_O^{++}), which act as electron-trapping sites. In contrast to water, oxygen acts as an electron acceptor rather than an electron donor, so both adsorbed oxygen and a decrease in the oxygen vacancies lead to positive V_{th} shifts in n-type metal oxide NWTs, as observed.¹⁷ OD-PA prevents oxygen from reaching and penetrating the surface of the ZnO NWTs, ensuring a consistent performance.

To observe the stability of the OD-PA passivation of the devices, $I_{ds}-V_{gs}$ measurements of nonpassivated and OD-PA-passivated ZnO NWTs were performed over time, both devices were dipped in water. As shown in Figure 4a, the I_{on} value was

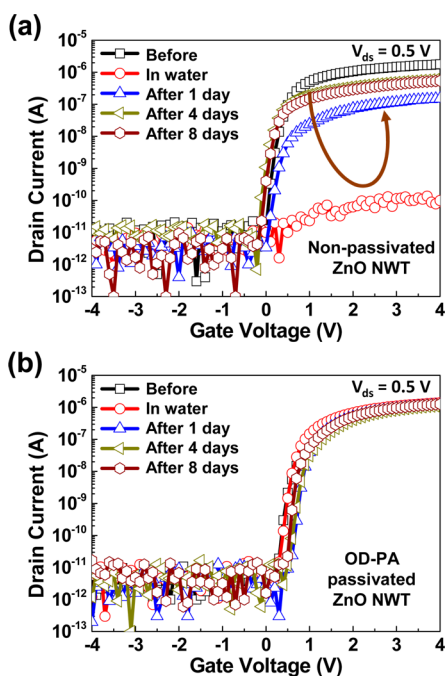


Figure 4. Time variation of the log-scale $I_{ds}-V_{gs}$ characteristics of the (a) nonpassivated and (b) OD-PA-passivated ZnO NWTs after exposure to water for 1 min. Measurements were performed over 8 days.

significantly reduced for the nonpassivated device. Over time, the I_{on} value slowly recovered, almost reaching the original I_{on} value, because water was dried on the surface of the ZnO nanowires. This indicated that electron donors due to H_2O molecules were not active any more on the surface of the ZnO nanowires. In the case of the OD-PA-passivated ZnO NWT, the I_{on} value remained the same in water, and the NWTs exhibited stable device performance without any change in the other transistor properties for 8 days. These results indicate that the OD-PA ZnO NWTs can maintain a stable performance over a long lifetime despite a harsh external environment.

To study their light-sensing properties, OD-PA-passivated and nonpassivated ZnO NWTs were exposed to three light illumination conditions [0 (dark), 1200, and 2100 lx], as shown in Figure 5a. The light illumination is expected to induce significant negative shifts in V_{th} in both types of devices, because of photogenerated electron–hole pairs induced in the metal oxide nanowires. Parts b and c of Figure 5 show the measured log-scale $I_{ds}-V_{gs}$ characteristics ($V_g = 0.5$ V) of representative nonpassivated and OD-PA-passivated ZnO NWTs under the three different illumination conditions. Indeed, a negative shift in V_{th} is observed in both passivated and nonpassivated NWTs: in nonpassivated ZnO NWTs (Figure 5b), V_{th} values were shifted by -0.54 V at 1200 lx and -1.37 V at 2100 lx, while in OD-PA-passivated ZnO NWTs (Figure 5c), V_{th} values were shifted by -0.81 and -1.37 V at 1200 and 2100 lx, respectively. In addition to the effect on V_{th} , the SS values changed under illumination, increasing from 0.40 V/dec at 0 lx to 0.50 V/dec at 1200 lx and 0.60 V/dec at 2100 lx for both passivated and nonpassivate devices. These shifts are due to the photogeneration of electron–hole pairs in the nanowires. The holes generated in metal oxide nanowires are captured by the interface between the nanowire and insulator, increasing the interface trap density.^{18,19} These interface traps may be either shallow-level or deep-level states in the forbidden band gap of the nanowires.¹³ The generated traps cause the negative shift in V_{th} in the ZnO nanowires. Figure 5d, a plot of I_{ds} over time as the NWT is illuminated (with 0, 1200, and 2100 lx), shows a step function with distinct current levels, regardless of passivation. The behavior is unchanged because the SAM of OD-PA does not reduce the transmittance of light; the transmittance of an OD-PA SAM is 98–99% at wavelengths of 300–1200 nm. As a result, the OD-PA-passivated ZnO NWTs can be used as highly selective light sensors that are robust to humid conditions; the OD-PA SAM protects the NWTs from their environment without changing their optical sensitivity.

CONCLUSIONS

In summary, the effect of OD-PA passivation on metal oxide nanowires was examined by observing the changes in transistor characteristics with and without OD-PA passivation under various environments including humidity, water, blood, and oxygen. While the nonpassivated NWTs showed significant changes with exposure to these environments, the OD-PA-passivated NWTs showed robust transistor properties. The representative ZnO NWTs have an I_{on} of 2.04×10^{-6} A, an SS of 0.30 V/dec, an I_{on}/I_{off} of 3.78×10^5 , and a μ_{eff} of 195 cm²/V·s. After exposure of the devices to 73% and 95% RH and water, V_{th} values of the nonpassivated device were shifted in the negative direction (-1.38 V at 73% RH, -1.77 V at 95% RH, and deteriorated transistor characteristics in water) but were highly stable for the OD-PA-passivated device, regardless of the condition. In addition, the transistor characteristics of the passivated device exhibited stable operation over time. After the stability of the passivated NWTs was demonstrated, their light sensitivity was investigated. They showed no significant change in optical response versus unpassivated devices. Because it provides greatly improved stability while retaining the NWT optical sensitivity, OD-PA passivation is attractive for application in sensing devices. Metal oxide NWT-based circuits are used in various applications for their flexibility, transparency, and high mobility; however, the lack of highly stable conductivity under various humidity conditions inhibits the

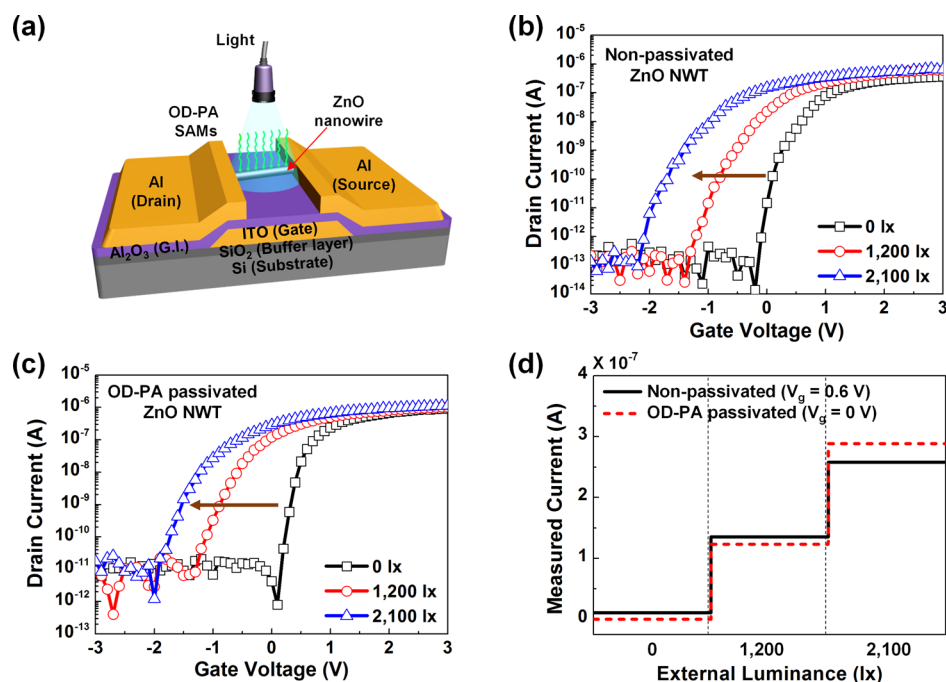


Figure 5. (a) Schematic of light illumination on ZnO NWTs. log-scale I_{ds} - V_{gs} characteristics of representative (b) nonpassivated and (c) OD-PA-passivated ZnO NWTs under light illumination [0 (dark), 1200, and 2100 lx]. (d) Two-step measured currents via external light intensities (1200 and 2100 lx).

incorporation into and good performance of real-world device applications. OD-PA passivation on the surface of metal oxide nanowires is expected to help resolve this issue by providing a stable transistor performance in different ambient environments and over long periods of time.

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Notes

The authors declare no competing financial interest.

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