

Chemical Gated Field Effect Transistor by Hybrid Integration of One-Dimensional Silicon Nanowire and Two-Dimensional Tin Oxide Thin Film for Low Power Gas Sensor

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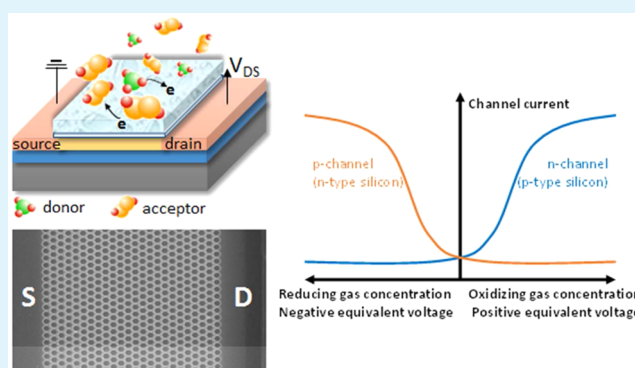
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Supporting Information

ABSTRACT: Gas sensors based on metal-oxide-semiconductor transistor with the polysilicon gate replaced by a gas sensitive thin film have been around for over 50 years. These are not suitable for the emerging mobile and wearable sensor platforms due to operating voltages and powers far exceeding the supply capability of batteries. Here we present a novel approach to decouple the chemically sensitive region from the conducting channel for reducing the drive voltage and increasing reliability. This chemically gated field effect transistor uses silicon nanowire for the current conduction channel with a tin oxide film on top of the nanowire serving as the gas sensitive medium. The potential change induced by the molecular adsorption and desorption allows the electrically floating tin oxide film to gate the silicon channel. As the device is designed to be normally off, the power is consumed only during the gas sensing event. This feature is attractive for the battery operated sensor and wearable electronics. In addition, the decoupling of the chemical reaction and the current conduction regions allows the gas sensitive material to be free from electrical stress, thus increasing reliability. The device shows excellent gas sensitivity to the tested analytes relative to conventional metal oxide transistors and resistive sensors.

KEYWORDS: gas sensor, field effect transistor, nanowire, thin film, tin oxide



INTRODUCTION

Several metal oxides such as tin oxide, indium oxide, and zinc oxide are chemically reactive with a wide range of gas molecules and volatile organic compounds.^{1,2} The resistance of the metal oxide changes upon adsorption of gas molecules, and two terminal chemiresistors and three-terminal chemical field effect transistors (CHEM-FETs) have been widely used to sense gases and vapors.¹ Metal oxide based sensing technology has been around since the 1960s, and the first electronic nose for selective discrimination of vapors was introduced in 1982.³ More recently, there have also been structural variations in sensor construction using one-dimensional (1D) nanowires.^{4–7} In all forms of devices, the resistance value of the metal oxide film usually ranges from a few tens of kilo- to a few mega-ohms. Since the baseline resistance is relatively high, a few volts are needed to obtain reasonable signal response, and the resultant current continuously flows even during the standby period. This requires control circuit overhead such as voltage generator and charge pump. Such demands are undesirable for battery-operated mobile/wearable applications, which are increasingly popular. Typically, nanowire-based CHEMFETs^{6–12} require gate or drain biases in the range of 5–60 V which is excessive, though top-gated devices are biased lower than bottom-gated

devices. Even silicon nanowire transistors on plastic substrates were operated at about 5 V.¹³ In conventional devices, the oxide thin film or nanowire plays the role of both chemically reactive layer and current conducting channel. Separation of these two functions here allows the bias voltage to be reduced to less than 1 V. Furthermore, the sensor can stay at normally off state and consume power only at the sensing event, which is suitable for low-power mobile/wearable electronics applications.

The two-dimensional (2D) thin and thick films offer an advantage of simple fabrication in the case of both chemiresistors and CHEMFETs, but their sensitivity and response time are relatively low. In contrast, the one-dimensional (1D) nanostructures result in high sensitivity due to their high surface-to-volume ratio. For example, Fan and Lu⁸ presented an analysis showing an inverse dependence of gas sensitivity on nanowire radius and supported it with experimental results. However, the bottom-up 1D-based device development faces fabrication challenges at present such as

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formation of the nanostructures, alignment, and making contacts. In addition, the 1D nanostructure formation itself consists of a series of steps involving synthesis, sonication, dispersion, and transfer of the nanowires to the eventual substrate. Therefore, the common 2D and 1D structure-based sensor technologies tend to be limited in applications and amenability to low-cost mass production, respectively. Here we present a scheme to attain the best aspects of both structures by hybrid integration of a 1D nanowire channel and a 2D gas sensitive film. The device presented here, named a chemically gated field effect transistor (CGFET), uses 1D silicon nanowire as the current conducting channel and 2D SnO₂ thin film on top of the silicon nanowire as the gas sensitive material. In conventional bulk silicon-based FET sensors, the subthreshold current flows through the bulk region of silicon whereas the chemical reaction occurs only at the surface. Therefore, these sensors have a fundamental limitation on detection limit and sensitivity. However, as the diameter of 1D silicon nanowire becomes less than the Debye length, the current conduction region is confined within the diameter of the nanowire. The entire volume of the nanowire becomes sensitive to the surface chemical reaction, and therefore, the high surface-to-volume ratio can increase sensitivity and detection limit. The device here is fully processed by conventional top-down silicon technology.

Another problem with the conventional sensors is the limited lifetime due to material degradation caused by not only the chemical fatigue after extended use but also the electrical stress.^{14–16} The performance degradation after a long period, arising from chemical changes or accumulation of unwanted species on the surface, can be reversed in most cases by nominal heating or other means such as exposure to UV light.^{5,9} In contrast, the electrical stress is more permanent and serious in nature. The electrical stress is mainly attributed to the fact that the gas sensitive material is in contact with a metal electrode. The electrode–oxide interface properties degrade due to oxide crack, metal–oxide interdiffusion, and electrode exfoliation. The voltage is constantly applied in chemiresistors and CHEMFETs and the resultant current flows continuously along the sensing layer so that electromigration and Joule heating would degrade the integrity of the sensing layer and the contacts.^{15,16} This problem is avoided here since the CGFET decouples the chemical receptor region from the electrical conduction region. Thus, no metal contact is made on the gas sensitive film, which protects the sensor from the types of degradation mentioned above.

The concept of this chemically gated device is illustrated in Figure 1. In conventional two-terminal chemiresistors, the conducting oxide film is directly connected at two ends to the electrodes as in Figure 1a. In conventional FET-type sensors, the gas sensing film is prescribed as the conduction channel (Figure 1b) and thus, it also suffers from the electrical stress and the contact degradation just as the chemiresistors, as mentioned above. When occasionally the conducting material in the transistor is made electrically floating (Figure 1c), the floating gate material and its target gas have been limited to platinum and hydrogen, respectively.¹⁷ A voltage is applied to the platinum gate, and the work function shift of the platinum is used as the sensing mechanism. Since the device itself is made with bulk silicon substrate, the response of this Pt-based hydrogen sensor is relatively small because the contribution of current through the bulk silicon region is always present regardless of sensing. In the CGFET here (Figure 1d), the

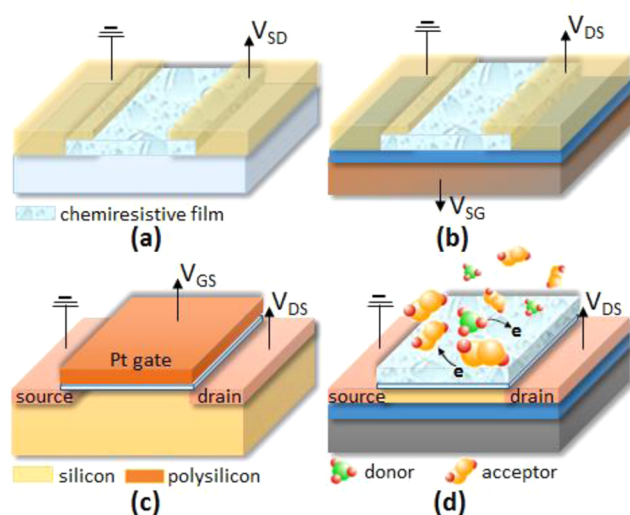


Figure 1. Schematic illustration of various gas sensor structures: (a) two terminal chemiresistor, (b) back-gated FET, (c) platinum gate FET typically used as hydrogen sensors, and (d) metal-oxide floating gate CGFET.

sensor structure is similar to metal-oxide-semiconductor FET (MOSFET) except that the gate electrode is replaced with an electrically floating gas sensitive material. In this work, SnO₂ is selected for the electrically floating gate to demonstrate the reliability and power consumption of the presented device. The material choice can very well be any other oxide film such as indium oxide, zinc oxide, or even graphene and carbon nanotubes. The use of silicon nanowires as the conduction channel eliminates the bulk current leakage and maximizes the sensitivity due to the one-dimensional nature.^{5,8} No external voltage is directly applied to the gate, instead the chemically induced potential modulates the silicon nanowire channel. Despite the two terminal configuration, as the resistance between the source and the drain is controlled by the pseudo potential from the third floating terminal, the present structure can be referred to as chemically gated field effect transistor.

EXPERIMENTAL WORK

The fabrication process of the CGFET follows the conventional MOSFET process except that the polysilicon gate is replaced by a gas sensitive SnO₂ film. Details are provided in Supporting Information along with a schematic of the process flow in Figure S1. The top silicon on a silicon-on-insulator wafer is patterned to form the active region with optical lithography, and then e-beam lithography and plasma etching are used to produce the nanowires in a honeycomb structure (see Figure 2) with a width of 30 nm and a height of 100 nm. Conventional straight nanowires can be used as well to construct the sensors. The honeycomb nanowire structure is electrically improved and mechanically robust compared to the conventional straight nanowire, especially when the aspect ratio is very high as in the case of biosensors and similar nonlogic or memory devices with a feature of the size of few microns.^{18,19} Figure 2 shows the top view scanning electron microscope (SEM) images before and after the SnO₂ process. The thickened nanowire indicates the post-SnO₂ fattening of the nanowire. Figure S2 shows the 150 mm wafer after nanowire formation and the fabricated sensor chip.

RESULTS AND DISCUSSION

The gas sensing capabilities of the CGFET are compared first with a bare nanowire device using diluted 100 ppm nitric oxide (NO) in air in order to clarify the role of the SnO₂ film and Pt

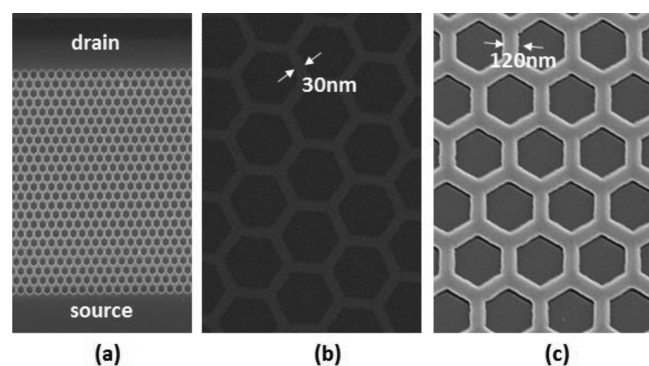


Figure 2. SEM images of the (a) fabricated SnO₂ CGFET; close-up view of the honeycomb channel region (b) before and (c) after the SnO₂ deposition process.

nanoparticle catalyst. The current change upon exposure to NO in the case of SiO₂-coated silicon nanowire is negligible (see Figure S3), whereas the response becomes prominent for the devices coated with the SnO₂ film. The maximum response is found for the SnO₂ film decorated with Pt nanoparticles. The target molecules physically adsorbed on SiO₂ may contribute to modulate the channel current in the bare device. In contrast, the SnO₂ involves both physical and chemical reactions with the target molecule, and the catalyst further enhances these reaction processes.⁷ Accordingly, the SnO₂ with the catalyst shows the largest response among the candidates, which confirms that the gas response depends on the choice of materials.

To demonstrate the advantage of the hybrid integration scheme, a conventional SnO₂ chemiresistor is fabricated and compared with the CGFET. The responses of the CGFET and conventional chemiresistor for oxygen (O₂) are shown in Figure 3a for identical gas injection conditions. The gas sensing experiments were carried out at ambient conditions with dry air as carrier gas and diluted O₂ as the target. The sensor response is defined as the ratio of the conductance difference (before and after gas injection) over the initial conductance. The conductance of the chemiresistor decreases upon oxygen adsorption on the surface^{4–7,11} because the electrons in SnO₂ are depleted by oxidation reaction with O₂. In contrast, the conductance of the CGFET increases because the positive pseudo potential in the gate starts to form an inversion channel in the p-type silicon. The response of the CGFET is more than 2 orders of magnitude greater than that of the chemiresistor as seen in Figure 3a; in addition, the decoupling of the reaction and conducting zones here allows higher response than those reported^{6,7,10–12,20,21} for conventional oxide nanowire transistors. For example, the response of the GCFET in Figure 3a is ~200–2000% for oxygen concentrations in the range from 25 to 100 ppm. ZnO nanowire CHEMFET, which is claimed to be better than the corresponding thin film transistors,⁸ shows a response of ~60% for 50 ppm of oxygen.¹¹

The charge exchange occurs on the current conduction channel itself in the chemiresistor, whereas the chemical reaction region and the current conduction channel are separated in the CGFET as mentioned earlier. The conductivity (σ) in the chemiresistor and CHEMFET is related to the total number of negative and positive free charges (n and p) and their mobility (μ_n and μ_p), as in $\sigma = q(\mu_n n + \mu_p p)$ where q is the unit charge density.²² Therefore, the conductance shifts linearly following the charge exchange reactions. In other words, the

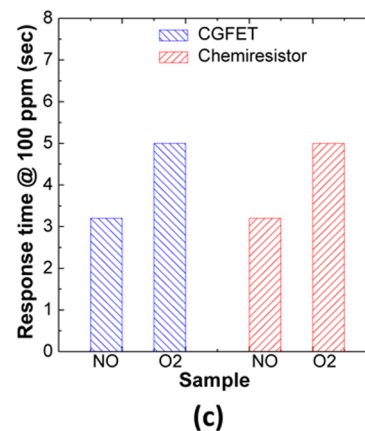
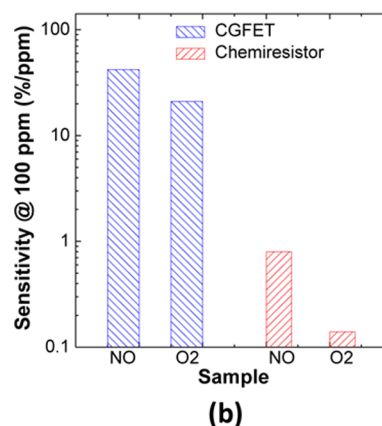
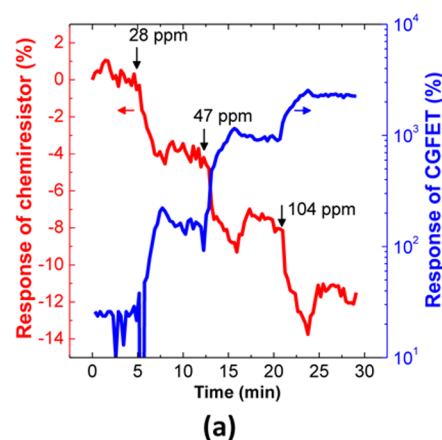


Figure 3. (a) Response of the CGFET and the control chemiresistor for different O₂ concentrations. (b) Sensitivity and (c) response time of the CGFET and the control chemiresistor for NO and O₂.

performance of the conventional type gas sensors is limited by the linear response. The channel current in the CGFET, however, is reflected from the gate potential and it is governed by the drift-diffusion mechanism in the MOS configuration. If the baseline condition is at the subthreshold regime, the subthreshold current (I_{sub}) is governed by the diffusion current that is approximated as $I_{\text{sub}} \sim I_0 \exp[q(n+p)/C_{\text{ox}}/kT]$, where C_{ox} is the gate oxide capacitance, k is the Boltzmann constant, and T is temperature.²³ This indicates that the subthreshold current in CGFET varies exponentially with the change in charge concentration. While only the free mobile charges directly contribute to the resistance change in the chemiresistor, both the free and fixed charges indirectly result in the

subthreshold current shift in the CGFET as the potential induced by the chemical reaction modulates the transconductance of the silicon channel. This inference is conditionally valid when the CGFET is biased under subthreshold regime (i.e., normally off state or enhancement mode). If the sensor is operated as normally on a device such as depletion mode MOSFET then the sensor response follows a linear fashion because the channel current is driven by the drift mechanism and there is not much advantage compared to the chemiresistors. Thus, the normally off CGFET structure can, in principle, exhibit higher sensitivity than the chemiresistor. In the CMOS process, the normally off device is usually attained simply by the complementary ion implantation between the channel and the source/drain. When the bias voltage is fixed at 1 V here, the baseline current is $0.5 \mu\text{A}$ for the chemiresistor while it is only 0.1 nA for the CGFET which proves the value of the normally off characteristics of the CGFET for the low-power operation. Such low bias operation is not possible with current oxide CHEMFETs with thin film or nanowire channels. The standby power consumption here is only 100 pW . Also, previous CHEMFETs were operated in the 5 to 60 V range^{6–12} in contrast to the 1 V bias here.

The sensitivity and response time of chemiresistor and CGFET are compared for 100 ppm of O_2 and NO in Figure 3 (panels b and c, respectively). The sensitivity is defined as the change in response signal per analyte concentration. The response time is the time required for the sensor to respond to a step concentration change from zero to 95% of the target concentration. The sensitivity of the CGFETs is more than 1 order of magnitude higher than that of the chemiresistors, which is attributed to the 1D nature of the conduction channel.^{5,8} However, the response times are independent of the structures as shown in Figure 3c. In other words, the hybrid integration does not accelerate the sensing speed. The sensitivity can be amplified by the 1D properties of the conduction channel, but the response time is limited by the properties of the gas sensitive material such as the surface states, grain size, catalytic treatment, and other extrinsic factors such as the operating temperature. Nevertheless, the response time of a few seconds here is excellent for room temperature operation, which may be due to the relatively thin film. Oxide-based sensors are typically operated at elevated temperatures of $200\text{--}300 \text{ }^\circ\text{C}$.^{1,6,7} The minimum detection limit of the CGFET is estimated by extrapolation from the calibration curves as $\sim 1 \text{ ppm}$ for both O_2 and NO , comparable to or better than previously reported values.^{10–12,20,24–28} Previous polymer-gated CGFETs used a bulk silicon substrate,^{29,30} and their detection limits for toluene and ethanol were on the order of a thousand ppm. The poor detection limit is attributed to the bulk current conduction, while the chemical reaction occurs at the surface as mentioned before. In contrast, the present sensor shows low detection limits of the order of a few ppm due to the 1D nanowire nature.

The sensor degradation can shift the sensor calibration curves over time, which gives rise to concerns of reliability and stability. The degradation can lead to more frequent calibrations and also reduce the sensor lifetime. Once the material is fixed, the chemical stress by cyclical adsorption and desorption due to continuous use may apply to the same degree for different structures. The wearing and aging of the gas sensitive materials associated with the oxidation and reduction process are unavoidable as long as chemical reactions occur during the lifetime of the sensor. Since conventional sensors

use the gas sensitive material also as the current conduction channel, the electrical stress becomes another source of degradation. Since the present CGFET decouples the gas sensitive material and the current conduction channel, the electrical stress to the gas sensitive region can be negligible. The sole contribution of the electrical stress can be assessed by a voltage acceleration test. A voltage five times higher than the nominal operation bias was applied in order to accelerate the voltage stress. Figure 4 shows the current drift for a stress of

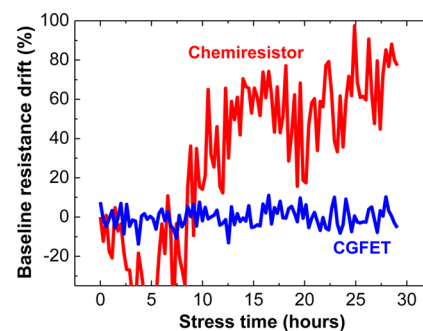


Figure 4. Stress induced device degradation for the chemiresistor and the CGFET for $V_{\text{DS}} = 5 \text{ V}$ at ambient conditions.

$V_{\text{DS}} = 5 \text{ V}$ at ambient conditions. While a sensor drift is seen for the chemiresistor, no current shift occurs for the CGFET, and in addition, the CGFET shows less noise than the chemiresistor.

There is a unique design consideration in the case of CGFET that can be exploited for selective sensing. The target analytes in gas sensing are generally classified into two groups: reducing gases or electron donors (H_2 , H_2S , NH_3 , and CO , for example) and oxidizing gases or electron acceptors (O_2 , NO , and NO_2). In the conventional chemiresistor, the conductance does shift for both oxidizing and reducing gases as shown in Figure 5a. Their current–voltage characteristics would follow a monotonic function (including linear, superlinear, or sublinear). Therefore, the curve can shift either upward or downward depending on the type of the sensing reaction. For n-type semiconductors with electrons as majority carriers such as ZnO , SnO_2 , and In_2O_3 , the conductance decreases for the oxidizing gases while it increases for the reducing gases. Likewise, the conductance of p-type semiconductors such as CuO changes in a reverse manner. This implies that the discrimination of the two groups requires further efforts,^{1,3,5} such as principal component analysis, pattern recognition, etc., but a single sensing material can be used for both oxidizing and reducing gases. If the CGFET is configured to be normally on, the current can either increase or decrease depending on the type of gas as in the case of chemiresistors and conventional CHEMFETs. But as discussed earlier, the normally on mode is not a low-power solution. The present normally off CGFET distinguishes between the oxidizing and reducing gases due to the nonlinearity of the channel. The selection of the conductivity type of the silicon channel results in complementary response to the oxidizing and reducing gases. However, the conductivity type of the gas sensitive film does not have anything to do with this distinguishing ability because the direction of induced potential is not determined by the conductivity type of the gas sensitive film but by the type of the target gas. Figure 5b illustrates the anticipated responses for n- and p-type devices for corresponding gas types. The oxidizing

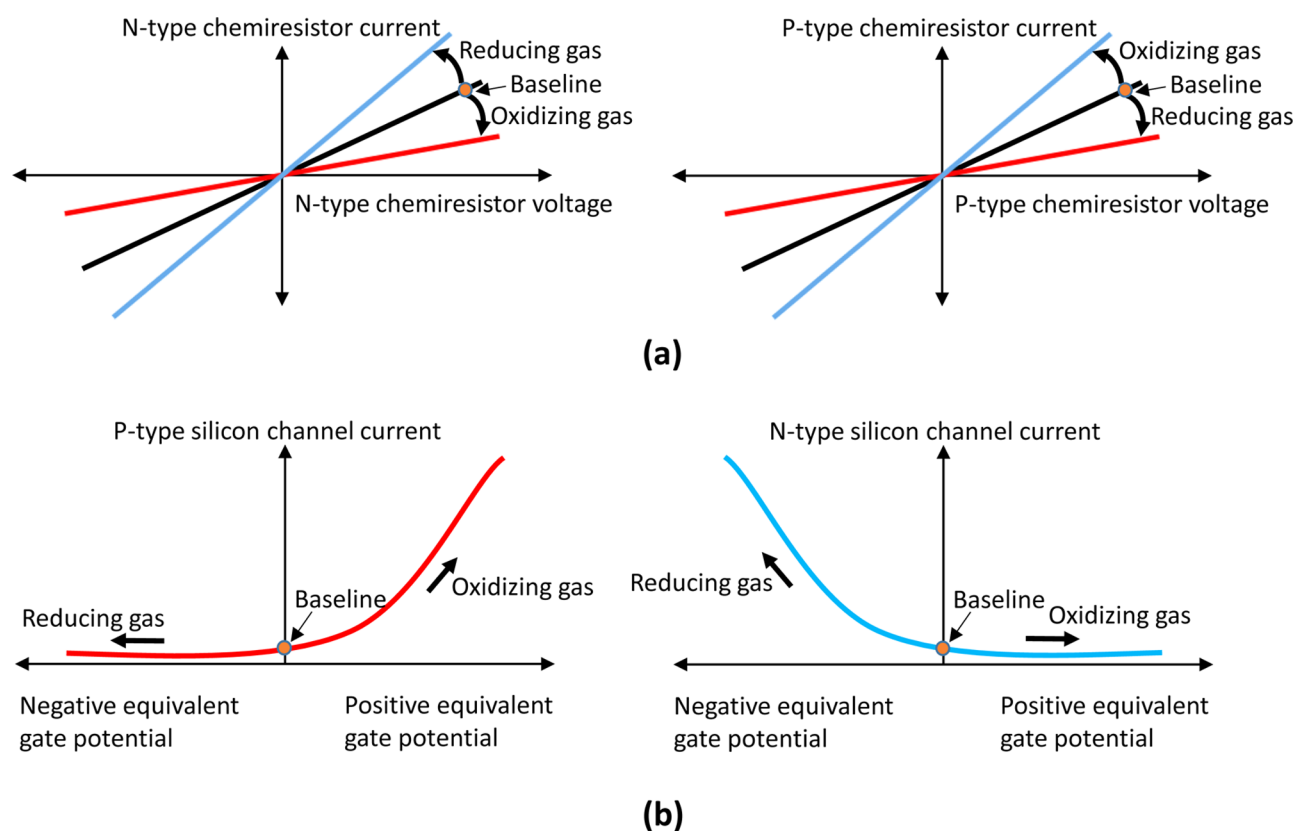


Figure 5. Conceptual illustration of the response of (a) chemiresistor and (b) CGFET for oxidizing and reducing gases. The monotonic function of the chemiresistor results in a response to both oxidizing and reducing gases. The nonlinearity of the normally off CGFET selectively responds to the corresponding type of gas.

gas accepts electrons from the sensing material and thus a positive potential is induced as the electrons in the sensing material are depleted regardless of the conductivity type of the sensing material. Likewise, the reducing gas donates electrons to the sensing material, and thus, a negative potential is induced. In order for the sensor to only respond to oxidizing gases, the n-channel device (p-type silicon) is preferred. For higher concentrations of reducing gases, a higher negative gate bias is induced, and thus the p-channel keeps staying at the off-state and no more sensing is possible.

To verify such a single type gas response, the sensor was exposed to a reducing gas, NH_3 , as shown in Figure 6. While the response to NH_3 is obvious in the chemiresistor, no

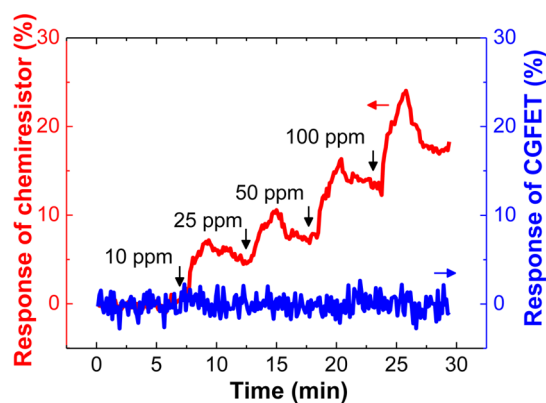


Figure 6. Response of the CGFET and the control chemiresistor to ammonia.

response is found in the n-channel CGFET here; instead, a p-channel CGFET would provide a sensing response. In this regard, the type of channel a priori can provide limited selectivity over the type of gas, but beyond that, an array of sensors (complementary CGFETs) in the form of an electronic nose^{3,5} would be required as with other approaches to distinguish among the same class of gases. This is due to the fact that no sensor material is uniquely selective for a given analyte. Therefore, selectivity is typically achieved by constructing an array of sensors with some variations among sensor elements. Typically chemical variations are preferable over physical variations such as changes in dimensions and geometry. For example, we have previously shown³¹ selective sensing of certain organic vapors and toxic gases using an array of 32 sensors with single-walled carbon nanotubes as the primary sensing material. Variations in materials across the sensor array included dopings, functionalization, and polymer coatings imposed on pristine carbon nanotubes. The sensor responses from the array were processed using a principal component analysis, which is a type of pattern recognition algorithm in order to obtain selective discrimination. Similarly, an array of GCFETs can be constructed here with variations of the tin oxide layer among the sensor elements in the array since the oxide layer constitutes the sensing medium; for this reason, there is no need to introduce any design changes in the silicon nanowire conducting channel across the sensor chip. Common and well-known variations for oxide-based sensors include doping with metals.¹ Metals enhance the catalytic activity of the oxide surface and common metals have included Pt, Pd, Al, and Au. The primary goal of the present work is to show the

possibility of operating a sensor under 1 V and very low power suitable for mobile applications by decoupling of the two regions (chemically sensitive and current carrying). On the basis of the present successful demonstration, future work would involve construction of sensor arrays as discussed above for selective sensing.

CONCLUSION

A chemically gated field effect transistor is presented with an electrically floating metal oxide film working as the gas sensitive gate. The device is fabricated by fully CMOS compatible technology, wherein a 1D silicon nanowire network is used as the current conduction channel with a 2D SnO₂ film on top of the nanowire network serving as the gas sensitive layer. The decoupling of the chemically sensitive layer and current conducting channel allowed a workhorse oxide CHEMFET sensor to become suitable for 20 first century applications of mobile and wearable platforms by reducing the drive voltage to 1 V or below. As the device is configured to operate at a normally off state, the sensing indicator is the subthreshold current which is an exponential function of the gate potential. The pseudo gate potential induced by the adsorption and desorption of the gas molecules on the oxide modulates the silicon channel conductance. The sensor response and sensitivity are enhanced compared to conventional devices. Decoupling the current conduction channel and the chemical reaction region enhances the long-term reliability and enables favorable normally off characteristics, making the device useful for low-power sensing applications. Finally, the presented approach should work with alternate choice of materials such as CNTs, graphene, and other emerging 2D materials.

ASSOCIATED CONTENT

Supporting Information

The material is available free of charge via Internet at The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsami.5b05479.

Detailed procedures for the device fabrication and image of the fabricated chip. The sensor responses exposure to NO between the CGFET and the control device are compared (PDF)

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Author Contributions

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Notes

The authors declare no competing financial interest.

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