Title

The influences of temperature, humidity, and O_2 on electrical properties of graphene FETs

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Abstract

The influences of temperature, humidity, and O_2 to the gas sensing characteristics of graphene field effect transistors (FETs) have been studied as these environmental factors are often encountered in practical gas sensing applications. Both empirical results and theoretical analyses are characterized for heated graphene FET gas sensors from room temperature to 100°C under a wide range of applied gate voltages. It is found that at a constant applied gate voltage of -20 V with respect to the gate voltage at the neutrality point, the sensitivity of the device to humidity decreases; while the sensitivity to O_2 decreases first, and increases afterwards as the operation temperature increases. These phenomena are explained by using the physisorption and chemisorption models between gases and the graphene surface. Furthermore, devices operate in the hole regime (the majority carrier is hole in the prototype devices) result in lower sensitivity to humidity and O_2 as compared to those results of gas sensors operating in the electron regime due to the p-type doping effects of moisture and O_2. As such, this work provides good foundations for graphene-based FET gas sensors in practical application environments under the influences of ambient air, temperature, and humidity.

Keywords

Graphene field effect transistor, gas sensor, temperature, humidity, oxygen, ambient air

1. Introduction

Recently, a demand for miniaturized gas sensors with high sensitivity, low power consumption, and low price has been rapidly growing for emerging new applications: wireless sensor networks for gas and oil industry, indoor air quality monitoring, and personal health care monitoring by using mobile electronic devices. While several gas sensor technologies have been proposed to replace the bulky and expensive traditional technologies in the past, none of the existing technologies can satisfy the above requirements at the same time [1,2]. On the other hand, new gas sensor technologies with nanomaterials and two-dimensional (2D) materials have emerged in the last decade [3,4]. Among those new technologies, graphene-based field effect transistors (FETs) have been extensively studied not only for gas sensors, but also for various other applications [5–8,4] due to the unique properties of graphene, including high surface area to volume ratio, zero-band gap electron band structure, high carrier mobility at room temperature, low Johnson noise, tunable electrical properties, ambipolar carrier transport, and chemical sensing capabilities [5,8–14]. Particularly, graphene is inherently suitable for chemical sensing applications with high sensitivities [5,8]. For graphene-based FET gas sensors, the low power consumption (~ a few tens of µW) and high sensitivity at room temperature are attractive features [5]. This is very different from gas sensors based on metal oxide, which typically require high temperature >200°C operations [15] with power consumption is in the mW range. Intensive studies on room-temperature graphene gas sensors [4,16–19] have revealed some key obstacles. The first challenge is the influence of temperature due to the temperature-dependent properties of graphene and other peripheral materials such as metals and semiconductors [20–23]. The second key issue is the influences of humidity and O_2 as several studies have reported that graphene FETs are sensitive to humidity [24–26]. Previously, the influences of temperature, humidity, and O_2 on gas sensors based on metal oxide have been extensively studied [27–29] but very few studies have been reported for gas sensors based on graphene FETs [24,30–32]. Furthermore, there has been no prior study on the influences of these parameters to the gas sensing results of graphene FETs under different applied gate voltages. In this work, we study these factors toward the performances of graphene FETs for practical applications. By sweeping the applied gate voltage, we analyze the doping behavior of graphene by tracking the position of the neutrality point (NP) at which the source-drain resistance reaches the maximum value. We extract the carrier mobility, μ ($\mathrm{cm^2/(V \cdot s)}$) from the obtained results to analyze the graphene resistance changes.

2. Material and Methods

2.1. Fabrication and electrical configuration of graphene FET

Graphene FETs are fabricated with a standard photolithography process with two photo masks. The detailed fabrication process has been reported previously [32,33] with a short summary here. We use commercial pre-transferred graphene substrates (Monolayer graphene on SiO_2/Si, 10 mm x 10 mm, Graphenea) synthesized by chemical vapor deposition (CVD). The metal contacts, Au/Pd (30 nm/25 nm) are patterned on the graphene substrate by a lift-off process. Afterwards, the graphene channels are defined by an oxygen plasma etching process (50 W for 7-10 s). A scanning electron microscope (SEM) image of the fabricated graphene FET is shown in **Fig. 1a**. The defect states of the fabricated graphene FET is evaluated based on Raman spectrum (**Fig. 1b**) as it is important factor for both the gas sensitivity and the recovery time [34,35]. The high $I(2D)/I(G)$ ratio (~3) agrees with typical Raman spectrum of a monolayer graphene, where $I(2D)$ and $I(G)$ are intensities of 2D and G peaks, respectively. The relatively higher $I(D)/I(G)$ ratio (~0.2) indicates that some defect states are induced during the fabrication process. The grain size, L_a, can be roughly estimated to be ~20 nm by using a relationship, $I(D)/I(G) = C(\lambda)/L_a$, where $C(\lambda)$ is ~4.4 nm, and this size is about half compared with the one in the data sheet provided by the vendor [36]. It is noted that the transferred CVD graphene may have impurities such as poly(methyl methacrylate) and H_2O at the graphene/SiO_2 interface and also on the surface, and thus they may degrade the performance of graphene FETs, e.g., the carrier mobility [37]. We have confirmed that the degradation due to our fabrication process is acceptable level in our previous study [18]. The fabricated graphene FETs are fixed onto ceramic packages by using the conductive silver paste. A typical electrical configuration is shown in the **Fig. 1c**. A constant source-drain current (or voltage) is supplied between the source (S) and drain (D), and the voltage (or current) across the channel is measured via contacts A and B. The gate voltage is applied to the Si substrate.

2.2. Experimental setup

The experimental setup is designed to monitor and control the device temperature and the relative humidity (R.H.) level. **Figure 1d** illustrates the gas system and major components in the chamber. A test chip is placed in the chamber and electrically connected to power supplies (Keithley 6220 and Agilent 6613C), a semiconductor parameter analyzer (HP 4145B, Hewlett Packard), and a digital multi meter (Agilent 34401A) outside the chamber via feed-through wires. A ceramic heater (18 mm x 12 mm x 1.2 mm) is fixed to the backside of the chip by a mechanical clamp, and two thermocouples are attached to the topside (TC1) and to the backside (TC2), respectively. The heater is controlled by a temperature controller (Digi-sense, Oakton). A commercial humidity sensor (HIH-4000, Honeywell) is placed near the test chip to monitor the R.H. level inside the chamber. LabVIEW (National instruments) is used to control the equipment and for the data acquisition. The R.H. level is controlled by the ratio of the flow rates of the two mass flow controllers, MFC1 and MFC2. The carrier gas (N_2) is branched to the path connected to MFC2 for water vapor to the chamber. Additional target gas is injected via the third mass flow controller, MFC3. No pump is used in the exhaust line so that the pressure in the chamber is always close to the atmospheric pressure. The volume of the chamber is about 400 cm^3 and the total flow rate of the gas is maintained at 200 sccm throughout the experiments.

3. Results and Discussion

3.1. The influence of temperature in N_2

This section describes the electrical properties of graphene FETs with respect to temperature in the N_2 atmosphere. **Figure 2a** shows the device operation temperature increases in a stepwise manner and the R.H. level maintains relatively at a constant level, 4±1%. **Figure 2b** shows the recorded results of V_G-V_{NP0} and R_{SD} (source-drain resistance) under different applied gate voltages with respect to time, where V_G is the applied gate voltage and V_G-V_{NP0} is the offset gate voltage in the first cycle - which is used as the carrier concentration in a graphene FET is determined by this value, rather than the absolute value of the gate voltage. The experimental results indicate that R_{SD} changes as the device operation temperature changes and the relationship is further characterized in **Figure 2c,** where the results of R_{SD} versus V_G-V_{NP} under different operating temperatures are plotted. It is observed that R_{SD} increases as the temperature increases and the relationship is nonlinear. The analytical investigation starts with the analyses of the carrier mobility, μ ($cm^2/(V\cdot s)$), versus V_G-V_{NP} as drawn in **Figure 2d** at different operation temperatures. The abrupt change of μ at the neutrality point is due to the singularity for the expression of μ [10]:

$$\begin{aligned} \mu_e &= \frac{L}{W} \cdot \frac{I_{SD} - I_{SD,NP}}{V_{SD}} \cdot \frac{1}{C_G\left(V_G - V_{NP}\right)}, \quad V_G - V_{NP} > 0 \\ \mu_h &= \frac{L}{W} \cdot \frac{I_{SD} - I_{SD,NP}}{V_{SD}} \cdot \frac{1}{C_G\left(V_{NP} - V_G\right)}, \quad V_G - V_{NP} < 0 \end{aligned} \tag{1}$$

where μ_e, μ_h, L, W, I_{SD}, $I_{SD,NP}$, V_{SD}, and C_G are the electron mobility, the hole mobility, the length and width of the graphene channel, the source-drain current, the source-drain current at the neutrality point, the source-drain voltage, and the gate capacitance per unit area of the graphene FET ($\sim 1.15\times 10^{-8}$ ($C/(V\cdot cm^2)$)). It is assumed that the carrier concentration does not change drastically in this temperature range (under the same gate voltage) such that the increase in R_{SD} can be interpreted as a result of the decrease in the carrier mobility (**Fig. 2d**) due to the electron-phonon scattering, or remote interfacial phonon scattering effects [20]. Furthermore, temperature-related calibration curves (**Figure S1**) are fitted by an empirically derived simple monomial equation,

$$\frac{\Delta R}{R_0} \cdot 100 = aT^k, \qquad \Delta R \square 0 \tag{2}$$

where a and k correspond to a factor and an exponent, and the k is expressed as,

$$k = \frac{\log(\Delta R_2 / R_0) - \log(\Delta R_1 / R_0)}{\log(T_2) - \log(T_1)} = \frac{\log(\Delta R_2 / \Delta R_1)}{\log(T_2 / T_1)} \tag{3}$$

where R_0, R_1, R_2, T_1(°C), and T_2(°C) are the initial resistance, the resistances at two different temperatures, and the corresponding temperatures, respectively. It is observed that the experimental results are fitted well with ranges of value of k=2.16~2.85 and a=3.3×10^{-5}~8.7×10^{-4}, respectively, with the coefficient of determinations, R^2, ranging from 98.6% to 99.8% (**Table S1**). The gate voltage dependent sensitivity curves shift downwards in the electron regime as the applied gate voltage increases, i.e., the carrier concentration increases, while those in the hole regime shift upwards as the applied gate voltage increases. These phenomena suggest that the significant reduction in the hole mobility contributes to the upward trend of R_{SD} in the hole regime as shown in **Fig. 2d** (V_G-$V_{NP} < 0$ V) [20]. As such, equation (2) can be used as the foundations for the source-drain resistance sensing calibrations under different operation temperatures.

3.2. The influences of humidity in N_2 under various temperatures

Figure 3a is the control profile of R.H. with respect to time under a fixed temperature by increasing the R.H. level linearly from 4±1% to ~70% in the first 20 minutes and then linearly decreasing the R.H. level to 4±1% in the next 20 minutes, and keeping it at 4±1% in the last 20 minutes. **Figures 3b-c** show the 3D plots of V_G-V_{NP0} and R_{SD} (source-drain resistance) under different applied gate voltages with respect to time at room temperature (22°C) and 100°C, respectively. Other experimental results at 40°C, 60°C, and 80°C can be found in **Figs. S2b-d**. **Figures 3d-e** show the 3D plots of mobility under different applied gate voltages with respect to time at room temperature (22°C) and 100°C, respectively (**Figs. S2g-i** for other cases of 40°C, 60°C, and 80°C). In general, the adsorption and desorption process for water moisture on graphene is reversible for a physisorption-like process (non-covalent binding), while the changes in R_{SD} are larger in the low temperature regime as compared with those at the high temperature regime. In addition, it is also observed that the changes in the carrier mobility are reduced as the temperature increases. The sensitivity of R_{SD} (resistance changes divided by the initial resistance) with respect to time is calculated under representative applied gate voltages (V_G-$V_{NP} = 10$ V and -10 V, respectively) as shown in **Figs. 3f-g** as well the changes in the hole concentration under room temperature (22°C) and 100°C, respectively (results at other temperatures of 40°C, 60°C, and 80°C are shown in **Figs. S2l-n**). These results illustrate that the responses of graphene FETs to moisture depends not only on the operation temperature, but also on the applied gate voltage. As the device temperature increases from room temperature to 100°C, the moisture sensing sensitivity in the electron regime (V_G-$V_{NP} = 10$ V) reduces from ~70% to less than 20%, while the moisture sensing sensitivity in the hole regime (V_G-$V_{NP} = -10$ V) remains within 15% either at room temperature or 100°C. Results from other environments at different testing temperatures also suggest large variations in the moisture sensing sensitivity in the electron regime when compared with those in the hole regime at various applied gate voltages (**Figs. S2p-t**). Furthermore, results show the trend of resistance sensitivity changes in the electron regime will follow the changes in the hole concentration, Δn_h (cm^{-2})=(C_G/e)ΔV_{NP}, where e is the elementary charge, and ΔV_{NP} is the shift of V_{NP}. Importantly, the moisture sensing sensitivity decreases as the temperature increases in the electron regime. Further analyses are made in the later section (section **3.4**) regarding the mechanisms.

3.3. The influences of O_2 in N_2 under various temperatures

Figure 4a is the profile of the concentration of O_2 with respect to time under a fixed temperature. The concentration of O_2 is estimated from the ratio of the flow rates of the mass flow controllers by linearly increasing it from 0% to ~30% in the first 20 minutes and linearly decreasing it to 0% in the next 20 minutes, and keeping it at 0% in the last 20 minutes. **Figures 4b-c** show the results of V_G-V_{NP0} and R_{SD} and the carrier mobility (**Figs. 4d-e**) changes with respect to time over various gate voltages at room temperature (22°C) and 100°C, respectively. The results for 40°C, 60°C, and 80°C can be found in **Figs. S3b-d, g-i**. As mentioned previously, the abrupt change of μ at the neutrality point is due to the singularity in the equation for μ. It is observed that in the low temperature regime, the R_{SD} responses do not recover to their original positions as the R_{SD} profiles shift to the p-type doped regime (V_G-$V_{NP} > 0$). This result indicates that the desorption process of O_2 is very slow at low temperature. On the other hand, in the high temperature regime, the fast recovery process is observed. These features suggest that the interactions between O_2 and graphene is chemisorption-like (covalent binding). The O_2 sensing sensitivity under representative applied gate voltages of V_G-V_{NP} = 20 V and -40 V, and the hole concentration changes with respect to time are shown at room temperature (22°C) and 100°C in **Figs. 4f-g**, respectively (results at other temperatures of 40°C, 60°C, and 80°C are shown in **Figs. S3l-n**). In general, large sensitivity variations are found with respect to both temperature and the applied gate voltage. Furthermore, high temperature condition seems to help the recovery of R_{SD}. For example, at V_G-V_{NP} = 20 V (chosen due to high sensitivity), the R_{SD} sensitivity decreases from ~160% (at room temperature) to ~60% (60°C) and increases to ~130% at 100°C. This transition can be attributed to a competing effect of the increasing desorption rate and increasing chemisorption rate [39]. On the other hand, at V_G-V_{NP}=-40 V, the sensitivity remains within ~30%. Similar to the testing results for the case of varying humidity, the electron regime tends to be more sensitive to O_2 as compared with those in the hole regime under different applied gate voltages (**Figs. S3p-t**). Furthermore, the variation trends of the R_{SD} sensitivity align well with those of the variation trends of the hole concentration. These results are reasonable as more electrons can be transferred from graphene to O_2 molecules in the electron regime.

3.4. The analyses on the temperature dependent sensitivities for humidity and O_2

In order to understand the fundamental mechanisms of the operation temperature to the humidity and O_2 sensing sensitivities of graphene FETs, further investigations are performed under a constant applied gate voltage instead of sweeping the gate voltage. **Figure 5a** shows the R_{SD} changes with respect to humidity (R.H. is 40%) versus time from room temperature to 80°C, with a constant applied gate voltage of -3 V (corresponding V_G-V_{NP} ~ -20 V). In this case, the specific applied gate voltage is chosen to have a relatively stable response but less sensitivity based on the aforementioned characterizations. **Figure 5b** shows the extracted R_{SD} sensitivity versus temperature, where markers are experimental data and the line is a fitting curve. The resistance change, ΔR_{AV}, is defined as the change between the 75-minute and 90-minute markers during the exposure to water vapor and is normalized by the initial resistance, R_0, to define the sensitivity, $(-\Delta R_{AV}/R_0)\times 100$ (%). As mentioned in the previous section, the reversible sensor response and the decrease in the sensitivity as operation temperature increases all imply the nature of physisorption-like process for moisture on graphene. An energy profile between physisorption-like gas molecules and a surface of a solid can be represented by a single Lennard-Jones potential, where the equilibrium distance between gas molecules and a surface of solid increases as the temperature increases due to higher kinetic energy of the gas molecules. Therefore, a higher device operation temperature results in a higher desorption rate for the gas molecules due to higher kinetic energies. The rate constant of desorption can be described by the following equation with the Boltzmann factor [40]:

$$k_d = A\exp\left(-\frac{E_d}{k_B T}\right) \tag{4}$$

where k_d, A, and E_d (>0) are the rate constant of desorption, a factor, and the energy for desorption, respectively. Assuming the flux of the incident gas molecules is independent of the device operation temperature, the sensitivity can be considered to be proportional to the inverse of the rate constant of desorption, k_d^{-1}, thereby the sensitivity, S_{H2O}(T), can be expressed as,

$$S_{H2O}(T) \sim k_d^{-1} = B\exp\left(\frac{E_d}{k_B T}\right) \tag{5}$$

where the factor B is the inverse of A. Based on this consideration, the experimental data in the **Fig. 5b** are fitted by equation (5), labeled as "Fitting 1." The fitting curve reasonably agrees with the experimental data with a relatively high correlation, 91.8% of R^2. This agreement suggests that the interaction between moisture and the surface of graphene FETs can be modeled by a physisorption model. As such, the sensitivity to humidity is considered to decay exponentially as the device operation temperature increases based on Eq. (5) and Figure 5b.

Figure 5c shows the R_{SD} changes under the influences of 20% of O_2 versus time at various device operation temperatures, from 70°C to 100°C, with a constant applied gate voltage of -3 V (corresponding V_G-V_{NP} ~ -20 V). Again, this test is selected for relatively stable response based on the aforementioned characterizations in the previous sessions. **Figure 5d** shows the extracted resistance sensitivity (in logarithmic scale) from the results in **Fig. 5c** versus the inverse of temperature. The markers are the experimental data and the line is a fitting curve. The definitions of the notations are the same as **Figs. 5a-b**. In contrast to the results for humidity, the sensitivity increases as the temperature increases in the given temperature range. This phenomenon can be explained in the following way. For chemisorption-like gas molecules, the potential energy at a surface of solid has two local minima [40]. At the first local minimum with a longer distance from the surface, the state of gas molecules is in a physisorption state, while at the second local minimum with a shorter distance from the surface, the state of gas molecules is in a chemisorption state. For a transition from a physisorption state to a chemisorption state, the gas molecules have to overcome a potential energy, which is called the activation energy, E_{act}. Therefore, additionally supplied thermal energy can contribute either to increase the rate of desorption, and/or to overcome the activation energy. As a result, the interactions of gas molecules with graphene surface will increase as the supplied thermal energy reaches the activation energy, due to the increase in the chemisorption states. This trend will prevail up to a certain temperature at which the supplied thermal energy exceeds the energy to desorb the chemisorption gas molecules. This model leads to the following Arrhenius equation [44],

$$S_{O2}(T) \sim A\exp\left(-\frac{E_{act}}{k_B T}\right) \tag{6}$$

where A and E_{act} (>0) are a factor and the activation energy, respectively. This relationship is often described in the Arrhenius plot by taking the natural logarithm of the both sides,

$$\ln\left[S_{O2}(T)\right] \sim \ln A - \frac{E_{act}}{k_B T} \tag{7}$$

The obtained experimental data are well fitted by equation (6) with a high correlation, 97.5% of R^2 as shown in **Fig. 5d** (labeled as "Fitting 2"). This agreement suggests that the interaction between O_2 and the surface of graphene FETs can be modeled by a chemisorption model in the given temperature range. As such, the sensitivity to O_2 increases exponentially as the device operation temperature increases. The chemisorption-like behavior in the relatively low temperature regime can be attributed to an existence of dangling bond defects on the grain boundaries of the graphene channel. Previous studies have suggested that the dangling bond defects on the edges of graphene can serve as adsorption sites for O_2 with larger than 1eV of adsorption energy, which corresponds to strong chemisorption [41].

A key conclusion here is that the sensitivity to humidity decreases exponentially as the device operation temperature increases, while the sensitivity to O_2 increases exponentially. These different temperature dependencies can be attributed to the different natures of physisorption and chemisorption as described above. These analyses suggest that the sensitivity to targeted chemisorption-like gas can be enhanced by increasing the device operation temperature depending on the activation energy, while the influence of humidity is mitigated. In other words, selectivity to humidity for chemisorption-like target gas can be improved by controlling the device operation temperature.

4. Conclusion

In conclusion, this work studies the gas sensing properties of graphene FETs under various conditions of temperature, humidity, O_2, and applied gate voltage. It is found that the source-drain resistance can be influenced by temperature throughout the range of the applied gate voltages and empirical equations can be used as the calibration curves for temperature between room temperature to 100°C. The influences of humidity and O_2 to the gas sensing performances are also analyzed at various temperatures and applied gate voltages. It is found that the gas sensing results can be drastically changed due to the existence of moisture and O_2 as well as the combination of the operation temperature and the applied gate voltages. Specifically, in the electron regime (V_G-V_{NP} > 0 V), graphene FETs are very sensitive to both humidity and O_2 at low device operation temperatures. As the device operation temperature increases, the sensitivity to humidity decreases, while that of O_2 decreases first, and then increases. These different temperature dependencies are well explained by the physisorption and chemisorption models. On the other hand, in the hole regime (V_G-V_{NP} < 0 V), the sensitivities against both humidity and O_2 are much smaller than that of the electron regime. As such, at higher device operation temperatures, the sensitivity to humidity can be reduced regardless of the applied gate voltage, while the sensitivity to O_2 can be increased. The analyses further suggest that selectivity to humidity for targeted chemisorption-like gas can be improved by controlling the device operation temperature. The results in this study can be applied to manipulate or compensate the influences of temperature, humidity, and O_2 for the gas sensing applications of graphene FETs.

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