AC phase sensing of graphene FETs for chemical vapors with fast recovery and minimal baseline drift

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ABSTRACT

This work utilizes an AC phase sensing approach of chemical vapors to achieve minimal baseline drift and fast recovery on graphene-based field effect transistors (FETs). Phase lag signals between channel resistance and gate voltage are detected with ultrafast recovery speed ($\sim$10 s) on defect-rich FETs made of chemical vapor deposition (CVD) graphene as the channel materials without surface functionalization at room temperature. The responses of the phase change upon exposure to water, methanol and ethanol vapors show at least ten times faster recovery speed than those of the conventional DC resistance measurements with minimal baseline drift, large dynamic range, and good stability. The effects of relative humidity on methanol and ethanol gas response properties are also studied. As such, the AC phase sensing scheme could open up a new class of research in gas sensors for improved sensing speed and baseline stability.

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1. Introduction

Over the last decade, 2D materials have been widely studied for fundamental knowledge toward practical applications [1–3]. Materials such as graphene offer good characteristics as the base sensing structures, such as large surface to volume ratio, low electrical noise, low power consumption and process compatibility with integrated circuits [4,5]. Previously, researchers have reported the ability to realize molecule-level detections using a graphene FET for high sensitivity [6,7] as the emerging platform for chemical sensors [8–10]. The most common sensing mechanism is the DC resistance measurement which correlates with charge variations on the graphene surface due to external gas vapors [11,12]. The intrinsic slow process of the charge transfer and the adverse effect of defects on the graphene surface are two great challenges. For example, FET gas sensors made of CVD graphene with a film transfer process suffer from intrinsic slow dynamics in its interface trap states and defect-compensated charge transfer process [13–15]. As such, the recovery speed of CVD graphene sensors is slow, especially at room temperature. For example, the recovery

of DC electrical conductance could take more than 1000 s [16] as vapors and charges are released from interface trap states/defects [17,18]. The resulting baseline drift has been a bottleneck for practical applications [19,20]. To address this issue, UV lights or heaters were previously used to help boost the recovery process [19,21,22], which inevitably increase the power consumption and system complexity.

The baseline drift could be reduced by using different electrical sensing schemes such as at high frequencies (100 kHz). Researchers have previously shown that the channel resistance of a graphene FET is sensitive to the change in the dielectric constant of the gas, rather than the adsorption reaction associated with charge transfer process [23]. In this present study, we report on an AC phase sensing approach in contrast to the conventional DC resistance sensing for minimal baseline drift and fast responses, as illustrated in Fig. 1a. To alleviate the problems stemming from the trap states and defects on graphene, this new scheme takes the advantages of the reversible and stable phase change signals instead of DC resistances. As shown in Fig. 1b, the phase lag between the channel resistance (point A and B between the source and drain of the FET) and the gate voltage is detected when an AC gate voltage at a moderate frequency is applied. Experimental results show that the phase lags of different vapors under various concentrations have fast recovery speeds in the ranges of 10 s, which are at least 10 times faster than those of DC resistance results with similar setups. Furthermore, the

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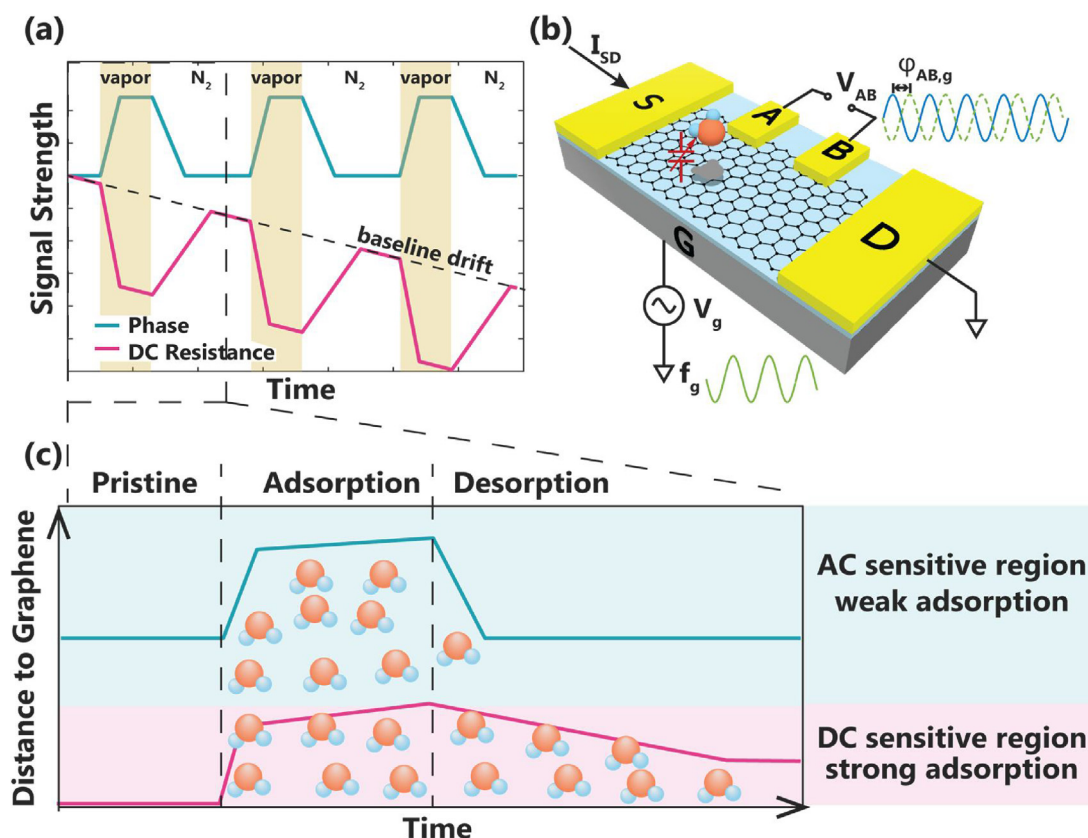
Fig. 1. (a) Schematic diagram illustrating the gas sensing performances between the phase lag detection scheme with minimal baseline drift and fast recovery speed as compared with the conventional DC resistance scheme on graphene-based gas sensors. (b) The setup of phase lag $\varphi_{AB,g}$ between V_{AB} and V_g on a CVD graphene FET sensor after the exposure to a specific chemical vapor. (c) The vapor adsorption and desorption on the graphene surface – the AC sensing scheme is targeting weakly adsorbed gases with a short distance from the graphene surface while the DC sensing scheme is targeting molecules very close to the graphene surface. This results in fast recovery for the AC sensing scheme.

dynamic response of the phase lag is reversible with large dynamic range while the DC resistance tests suffer from baseline drift problems. Fig. 1c illustrates the key differences between the AC and DC domain measurements, where the AC phase lag results are sensitive to the weak adsorption of vapor molecules above a distance to the graphene surface for fast gas adsorption and desorption processes, while the DC resistance results are sensitive to the strong adsorption and desorption process close to the graphene surface. These observations and analyses are explained by an analytical model with good match to the measurements.

2. Device and experiments

2.1. Preparation of graphene FET

The sensing material of our device is the monolayer graphene grown via the CVD method, which is available from Graphenea Inc. on top of a silicon die (10 mm × 10 mm) with 300 nm-thick, thermally grown SiO$_2$ on top. The following fabrication processes were carried out in the Marvell Nanofabrication Laboratory at UC Berkeley and also reported in details in our previous work [24]. First, four electrodes and contact pads (30 nm Pd/25 nm Au) were patterned on graphene by the lithography and lift-off process. Second, the graphene channel was patterned by the lithography and oxygen plasma (50 W, 7 s) etching process. Finally, the electrodes of graphene FET were connected to a custom designed test board by wire bonding. The optical microscopic picture and the SEM photo of the fabricated device are shown in Fig. 2a & b, respectively.

The fabricated device was purged with nitrogen overnight in the test chamber. Then, a milli-ampere level current was applied through the graphene channel for one minute as the thermal annealing process to remove surface contaminants [25]. The charge neutral point of the fabricated graphene FET was around 25 V (p-type doped) due to the unintentional doping of polymer residues during the device fabrication process. Therefore, results in this work were tested in the hole branch of graphene as the DC offset on the gate electrode was kept at 0 V.

2.2. Experimental setup

Fig. 3a illustrates the electrical configuration of the test setup, where the four-point probe method was used to avoid the influence of contact resistance in the measurement. An AC voltage was applied on the gate electrode with a frequency, f_g, ranging from 50 to 1000 Hz. A lock-in amplifier (SRS 860, Stanford Research Systems) was used to reduce the noise level at the source electrode with a reference of 20 kHz AC current (2 μA) through the graphene channel and a 1 MΩ resistor. The voltage between electrode A and B was measured by the lock-in amplifier. A data acquisition device (PicoScope 5242B) was used to collect V_{AB} and V_g simultaneously. With this setup, the phase lag between V_{AB} and V_g can be extracted by fast Fourier transform (FFT).

Fig. 3b illustrates the gas connections as all gas sensing measurements were carried out in a sealed nylon chamber (2 cm × 2 cm × 2 cm) with a waste gas treatment system. The concentration of a specific vapor was applied via the split-stream system with dry nitrogen (or dry air) and gas-saturated nitrogen