Selective Sensing of Individual Gases Using Graphene Devices

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***Abstract*— Graphene chemiresistors have enabled gas and vapor detection with high sensitivity. However, changes in graphene resistivity under the equilibrium gas exposure cannot be used to determine both the gas concentration and its type, making the sensing selectivity with resistive detection one of the key barriers to overcome. In this paper, we report on using low frequency noise to define the new characteristic parameters, which, in combination with the resistance changes, form unique gas signatures. The noise measurements can also be used in combination with evaluating "memory step" effect in graphene under gas exposure. The "memory step" is an abrupt change of the current near zero gate bias at elevated temperatures T > 500 K in graphene transistors. The "memory step" in graphene under gas exposure can be also used for high-temperature gas sensors, and is attractive for harsh-environment applications.**

***Index Terms*— Gas sensing, grapheme, noise, noise signature of gas, memory step.**

I. INTRODUCTION

GRAPHENE has extremely high mobility [1]–[5], thermal conductivity [6], [7], and ultimate surface-to-volume ratio. The last property in combination with the low thermal and $1/f$ noise [8], [9], relatively low contact resistance [10], [11], and high electrical conductivity (tunable in a graphene field effect transistor) makes graphene promising for gas sensing applications [8], [12], [13].

The high-gas sensitivity of graphene, (with ability to detect ultra-low concentrations down to < 1 ppb), and the linear dependence of the response to the gas concentration have been already demonstrated for a number of gases (see reviews [12], [13] and references therein). Different DC parameters of graphene including resistivity, shift of the charge neutrality voltage (Dirac voltage), and frequency of the surface acoustic waves have been proposed as sensing parameters [12], [13]. However, the selectivity of gas sensing using the pristine graphene and above-mentioned sensing parameters has been limited.

In this paper, we discuss two new sensing parameters: low frequency noise [14] and "memory step" [15].

Noise is usually considered as one of the main limiting factors for the detector sensitivity. However, the electronic noise itself can be used as a sensing parameter [16]. We show that low frequency noise measurements, in combination with other DC parameters, enhance the graphene gas sensor selectivity (see also [14]).

Another graphene property, which can be used for sensing applications, is so-called "memory step" [15]. At elevated temperatures (> 500 K), current-voltage characteristics of graphene transistors exhibit an abrupt change of the current near zero gate bias (this effect was called the "memory step" in [15]). The slower the gate voltage sweeps make memory steps more pronounced. Despite differences in current voltage characteristics, particularly in charge neutrality voltage, the memory step always appears at zero gate voltage. This new phenomenon in graphene can be used for applications in sensors at high temperatures, in combination with noise measurements.

II. GRAPHENE DEVICE FABRICATION AND MEASUREMENT PROCEDURE

Graphene samples were produced by mechanical exfoliation from highly oriented pyrolytic graphite and placed on Si/SiO_2 substrates using a standard procedure. Graphene flakes were selected using micro-Raman spectroscopy through the 2D/G-band deconvolution. Drain and source contact areas were defined by Leo1550 electron-beam lithography. The degenerately doped Si substrate acted as a back gate (some transistors had top gate as well). Fig. 1 shows the transistor current-voltage characteristics measured in atmosphere environment. The mobility and contact resistance were estimated using transmission line model structures, four probe measurements (see inset in Fig. 1) and by analyzing the current voltage characteristics. We found the mobility of both electron and holes within the range 5000–1300 cm^2/Vs. The value of the contact resistance per unit width varied from

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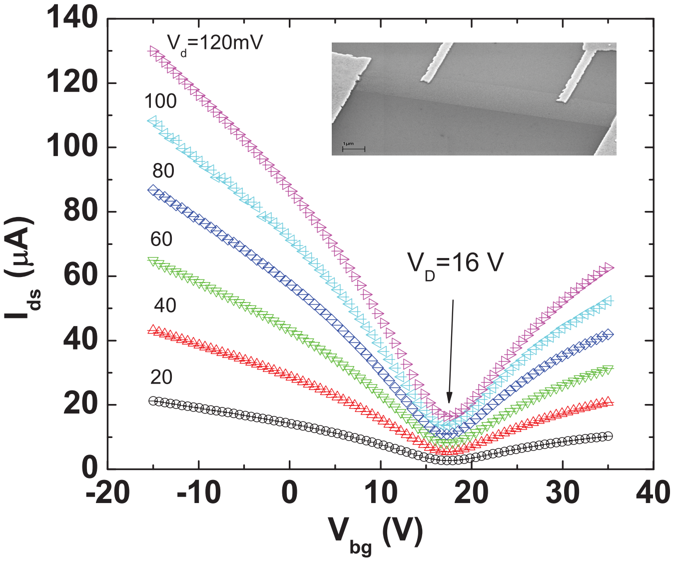

Fig. 1. Current–voltage characteristics of bottom gate transistor. Inset shows SEM image for four probe measurement.

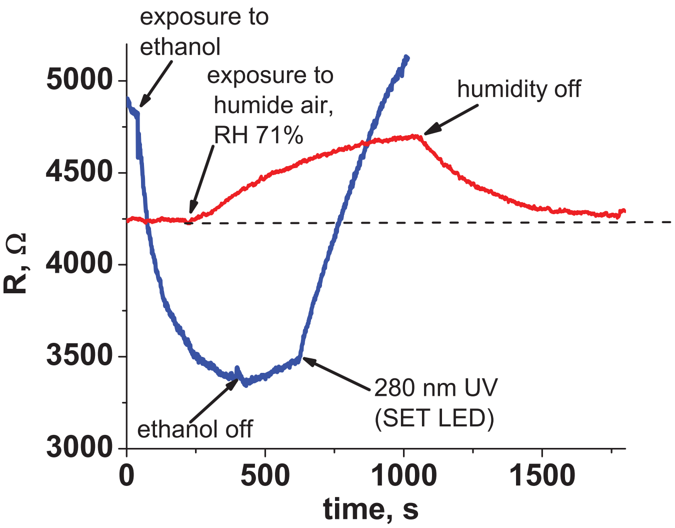

Fig. 3. Dependence of resistance on time under the exposure to ethanol and change of the humidity.

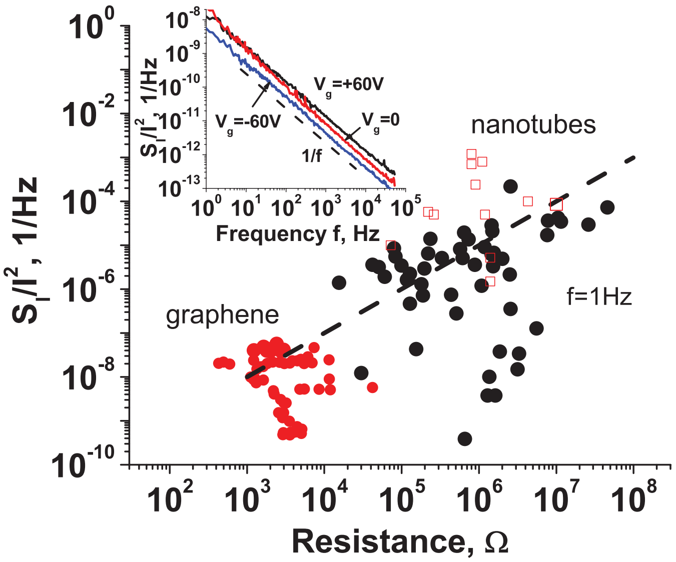

Fig. 2. Comparison of noise in graphene and carbon nanotubes. Data for nanotubes are from [18], [19]. Inset shows noise spectra in graphene at different gate voltages.

0.2 to 2 Ω mm. At high absolute value of the gate voltage this contact resistance makes a significant contribution to drain to source resistance.).

The low-frequency noise was measured in a common source mode in the frequency range from 1 Hz to 50 kHz at 300 K. Inset in Fig. 2 shows examples of the noise spectra at different gate voltages. Comparison of the noise in graphene and nanotubes demonstrated that graphene is less noisy (see Fig. 2).

Current-voltage and noise characteristics were measured in atmosphere environment and under the exposure to the laminar flow of the following vapors: methanol, ethanol, tetrahydrofuran, chloroform, acetonitrile, toluene, and methylene chloride. Different vapors were generated by bubbling dry carrier gas (air) through a respective solvent and further diluting the gas flow with the dry carrier gas. All vapors were generated at concentrations of ~0.5 P/Po, where P is the vapor pressure during the experiment and Po is the saturated vapor pressure.

When the constant voltage was applied to the gate, the drain current relaxed slowly to its steady-state value. This effect is known [17] and attributed to the deep traps in oxide. In order to avoid this drift all measurements were performed at zero gate voltage, i.e. on the "hole" part of the current voltage characteristic (see Fig. 1).

III. RESULTS AND DISCUSSION

The graphene transistor exposure to the vapor changed the resistance and noise spectra. The changes in the resistance and noise were not always correlated and can be used as independent parameters in the analysis of the sensor response.

Fig. 3 shows the resistance changes under the exposure to ethanol and different levels of humidity. As seen, the resistance response is rather slow and takes several hundreds of seconds to reach the steady state condition. Note also the different characteristic times of the response to ethanol and humidity. Surface functionalization [20] and operation at elevated temperatures [21], [22] significantly reduce the response and recovery times of carbon allotrope sensors based on carbon nanotubes and graphene. The process of degassing can be also accelerated by the exposure to ultraviolet (UV) light. We used 280 nm SET Inc. light emitting diodes to study the effect of the UV light on the degassing process (see Fig. 3). However extending exposure to UV damages the graphene and alters the device characteristics.

While some vapors change the electrical resistance of graphene devices without changing their noise spectra, others introduce distinctive Lorentzian bulges with different characteristic frequencies f_c. Fig. 4 shows the noise spectra measured in open air and under the exposure to different vapors. As seen, the noise spectra in the presence of the vapors reveal characteristic bulges over $1/f$ noise background.

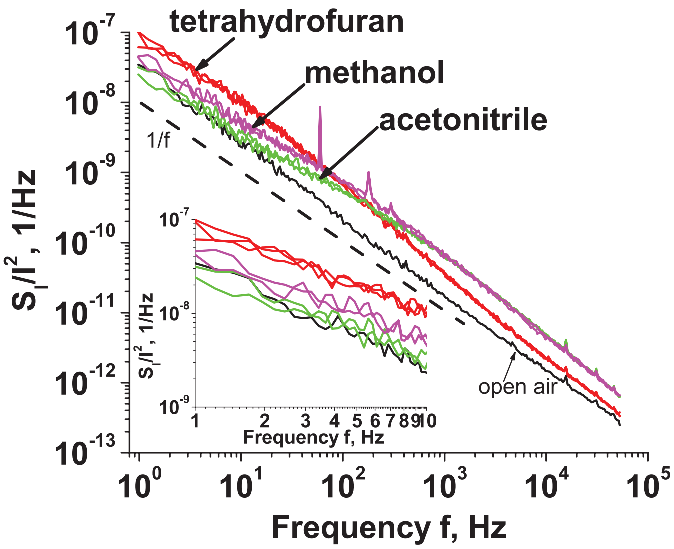

Fig. 4. Noise spectra of grapheme transistor in different gas environments. Inset shows the same noise spectra in the narrower frequency span. Multiple spectra shown for the same condition demonstrate perfect reproducibility of the noise measurements.

The characteristic frequencies of these bulges are different for different gases and can serve as components of the gas signatures. Noise spectra under the vapor exposure were reproduced very well-see multiple overlapping spectra in Fig. 4 and in the inset.

Noise analysis has also been recently performed for graphene and SnO_2/graphene nanocomposites with the goal of improving the signal-to-noise of conventional electrical current measurements [21]. Unlike this paper, our focus is to extract the vapor-dependent information from the analysis of the noise spectra.

The characteristic frequency f_c is easier to extract from the noise spectra multiplied by frequency f, versus f as shown in Fig. 5.

Another unique parameter, which can serve as a part of a gas signature, can be defined as:

$$\gamma = \left| \frac{S_I(f_{c5})}{I^2} f_{c5} - \frac{S_I(f_{cn})}{I^2} f_{cn} \right| \Big/ \frac{S_I(f_{c5})}{I^2} f_{c5}.$$

Parameter γ describes the rate of change of the noise times frequency product $S_I/I^2 \times f$ as a function of frequency. This parameter reflects the properties of noise sources, such as traps. In case of a pronounced generation-recombination noise, the value of γ correlates with the width of the recombination-generation bulge in the noise spectrum. The inset in Fig. 5 shows the value of this parameter γ along with the relative resistance change ΔR/R. Table I shows the whole set of parameters for different gases, which can serve as distinctive signature components for specific vapors enabling highly selective gas sensing with a single graphene device.

These findings demonstrate that a selective) sensing of individual gases is possible with a single) graphene transistor and does not require an array of sensors functionalized for each chemical separately.

The capability for discriminating gases using a single graphene transistor was evaluated using a principal components analysis (PCA) technique [23] done in MATLAB (The Mathworks Inc., Natick, MA). PCA is a commonly used unsupervised and robust pattern recognition approach for analysis of multivariate data. PCA projects the data set onto a subspace of lower dimensionality with removed collinearity. PCA achieves this objective by explaining the variance of the data matrix in terms of the weighted sums of the original variables without significant loss of information. These weighted sums of the original variables are called principal components (PCs). In PCA, the scores plots show the relations between analyzed samples (different gases in our studies). For the development of our PCA model, we analyzed the entire measured noise spectra from 1 Hz to 50 kHz. A more detailed estimation of the precision of our PCA determinations is the focus of our upcoming study.

TABLE I

CHARACTERISTIC FREQUENCY f_c ΔR/R, AND PARAMETER γ FOR DIFFERENT GASES

Gas	f_c, Hz	γ	ΔR/R %
Open air	none	0.055	-
Toluene	none	0.055	15
Tetrahydrofuran	10–20	0.175	18
Chloroform	7–9 and 1300–1600	0.031	−25
Acetonitrile	500–700	0.0535	−35
Methanol	250–400	0.134	−40
Ethanol	400–500	0.048	−50

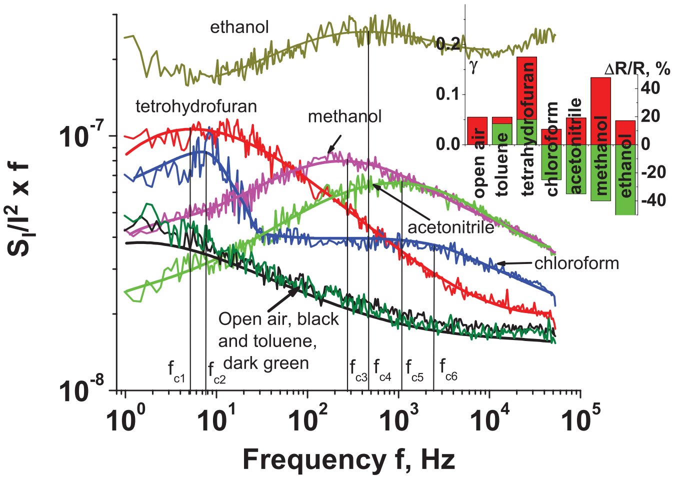

Fig. 5. Noise spectrum frequency products for different gas environments. Inset shows parameters γ and $\Delta R/R$, which serve as a signature of the given vapor.

The scores plot of the first two PCs is shown in Fig. 6, illustrating the diversity of noise features originated from all gases. The larger the distance between the data points in the scores plot, the larger the difference in the response pattern between the respective gases. The first two PCs explained over 99% of the total variation captioned by the PCA model. The difference in the response patterns of tested gases could provide the insights to the details of different mechanisms of interactions of different gases with graphene.

At elevated temperatures (> 500 K) current-voltage characteristics of graphene transistors exhibit an abrupt change of

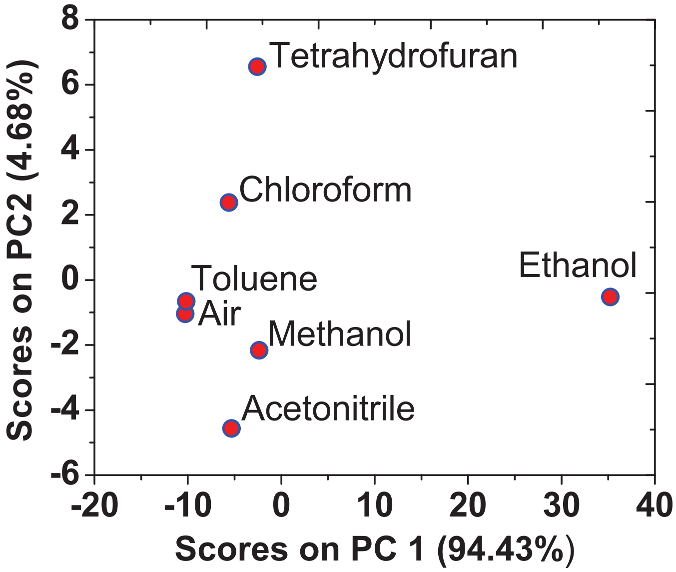

Fig. 6. PCA scores plot of the first two principal components of the response of the graphene transistor to different gases. For PCA classification, noise spectra were autoscaled.

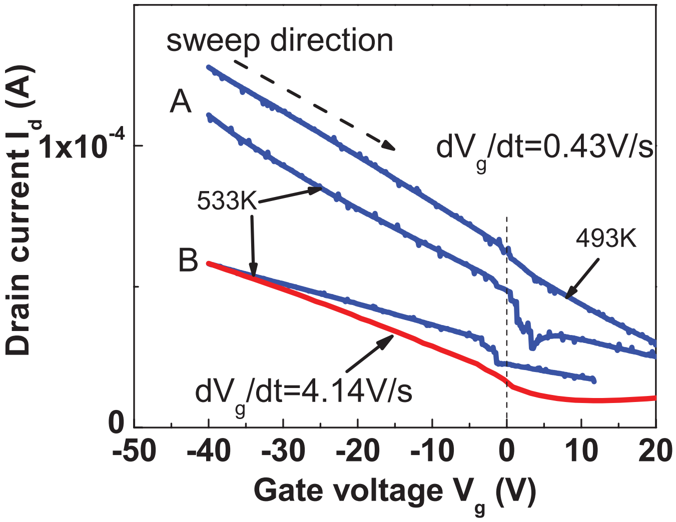

Fig. 7. Current–voltage characteristics exhibiting the memory step for samples A and B for different temperatures and sweep speed.

the current near zero gate bias (called the "memory step"), see Fig. 7. The slower the gate voltage sweep-the more pronounced is the step in the current. Despite differences in current voltage characteristics, particularly in charge neutrality voltage, the memory step always appears at zero gate voltage. Since the memory step is very sensitive to temperature and gate voltage changes, we expect it to be sensitive to the environment changes as well. This new phenomenon in graphene can be used for applications in sensors at high temperatures, in combination with noise measurements. Such studies will be reported elsewhere.

IV. CONCLUSION

We found that while the noise spectra of graphene transistor in open air are close to the $1/f$ noise, vapors of different chemicals produce distinctive changes on the noise spectra. Most vapors introduce bulges with different characteristic frequencies f_c. In this case the frequency dependences of the noise spectra multiplied by frequency f have one or more maxima at different values of f_c with different widths. The frequency f_c of the vapor-induced bulge, the relative resistance change $\Delta R/R$, and newly introduced parameter γ can serve as distinctive signatures for specific vapors. This allows us achieving selective gas sensing with a single graphene device. We also propose that the new phenomenon in graphene (called the "memory step") can be used for sensing applications in sensors at high temperatures, in combination with the noise measurements.

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