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PERSPECTIVES OF FLUCTUATION-ENHANCED GAS SENSING BY TWO-DIMENSIONAL MATERIALS

INVITED TALK

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We present the results of gas sensing using the fluctuation-enhanced sensing method in selected two-dimensional materials (2DMs). We claim that gas sensing selectivity can be improved further by considering semiconducting two-dimensional materials doped by noble metal nanoparticles. The 2DMs' structures exhibit some imperfections defined by their structure, occurring repeatedly there. These imperfections are adsorption-desorption centers responsible for gas-sensing properties and generating flicker noise of various intensities. We consider how these imperfections can be modulated and utilized for fluctuations-enhanced gas sensing. We propose the decoration of 2DMs by noble metal nanoparticles that impact flicker noise. Additionally, we consider utilizing localized surface plasmonic resonance induced by irradiation at selected wavelengths.

Keywords: gas sensors; flicker noise; two-dimensional materials; noble metals.

1. Introduction

Due to their unique and repeatable physical properties, two-dimensional materials (2DMs) have been extensively investigated in recent decades. These materials are commonly used for gas-sensing applications and are characterized by high sensitivity [1-3]. The 2DM sensors operate as chemoresistors or field effect transistors (FETs). Their DC resistances or current-voltage characteristics are used to detect various gases but still at limited gas selectivity [4,5]. Thus, the fluctuation-enhanced gas sensing (FES) method, utilizing low-frequency resistance noise measurements, was applied to these attractive materials to improve the selectivity and sensitivity of gas sensors [6,7]. The method was successfully

used in grainy metal oxides (e.g., SnO_2, WO_3) by observing a change in power spectrum intensity of flicker noise or its slope versus frequency [8,9]. The method was also applied to two-dimensional materials (e.g., graphene, MoS_2) to enhance gas detection. Graphene back-gated FETs were used to observe Lorentzians in resistive noise power spectral density and having characteristic frequencies for a few detected organic vapors [10,11].
Our study encloses recent results of applying low-frequency noise measurements for gas sensing in two selected 2DMs (e.g., graphene, MoS_2). We present new ideas and preliminary experimental results on enhancing the FES method utilizing 2DMs as a platform for gas sensing exhibiting the limited number of imperfection types that determine the adsorption-desorption of various gas molecules. We consider two methods of modulating these imperfections: (i) by their selective decoration with noble metal nanoparticles and (ii) by the use of localized surface plasmonic resonance (LSPR) induced by irradiation of noble metal nanoparticles at selected light wavelengths. The LSPR phenomenon generates an additional charge that modifies the electrical characteristics of such sensors. The charge generation efficiency is very sensitive to an ambient atmosphere and, therefore, can be used for gas sensing.

2. Two-dimensional materials and gas-sensing

Two-dimensional materials are desirable for gas sensing because of the extremely high ratio of their active area to volume. A flake of a single atom layer of 2DM can be considered a gas-sensing element of very high sensitivity, reaching a level of a single gas molecule detection [10]. Worse but of practical importance, gas sensing was observed for an ink-printed layer of various 2DM flakes [12]. This technology can be commonly applied to wearable sensing devices because of its low cost and straight sensing methods limited to voltage or current measurements. The FES method was successively applied in 2DMs of various electrical properties, like semi-metallic graphene [10,11], semiconducting materials (e.g., MoS_2 [13]), and graphene decorated by TiO_2 nanoparticles [14].
A few Lorentzians at selected characteristic frequencies were observed in graphene back-gated FET, and different volatile organic solvents (e.g., ethanol, tetrahydrofuran, methanol, chloroform) were identified. The same effect was observed for a graphene gate of various dimensions, from tens of μm^2 to thousands of μm^2 [10,11]. The imperfections of graphene structures are responsible for gas molecules' adsorption-desorption event, generating Lorentzian component $S(f)$ in the noise power spectrum at specific corner frequency f_c and intensity S_0:

$$S(f) = \frac{S_0}{1+(f/f_c)^2} \quad (1)$$

Experimentally observed f_c values were in the frequency range between a few Hz and a single kHz (Fig. 1: $f_c \cong 30$ Hz for chloroform, $f_c \cong 2$ Hz for acetonitrile, $f_c \cong 1$ Hz for tetrahydrofuran). These imperfections are extensively studied by various experimental (e.g., Raman spectroscopy, atomic force microscopy) and theoretical methods (Density Functional Theory—DFT) to determine their binding energy and propose methods of modulation (e.g., by nanoparticle decoration to make gas sensing hybrid materials) [15].

The Lorentzians were also generated within an ink-printed layer of graphene flakes decorated with TiO_2 nanoparticles [15]. The Lorentzian corner frequency shifted from tens of Hz to a few kHz when an ambient atmosphere changed from synthetic air to a few ppm of NO_2 gas.

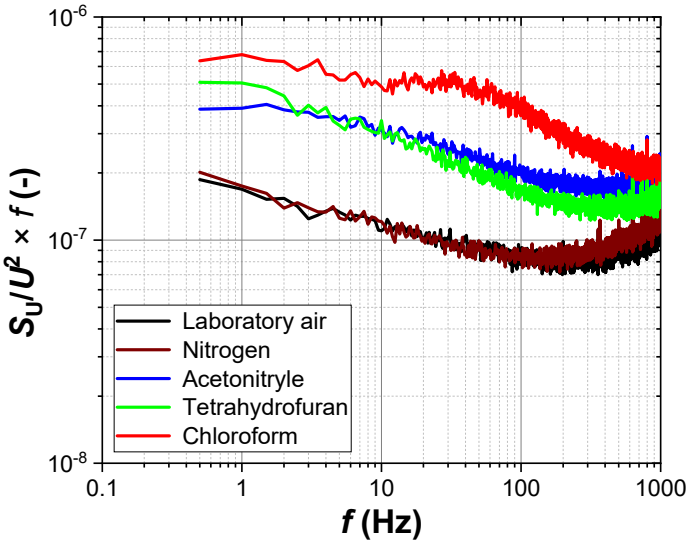

Fig. 1. Power spectral density $S_U(f)$ of voltage noise normalized to the squared voltage U across the channel of the graphene back-gated FET versus frequency f recorded in a few selected ambient atmospheres: laboratory air (black line), nitrogen (brown line), acetonitrile (blue line), tetrahydrofuran (green line) and chloroform (red line).

Semiconducting 2DMs (e.g., MoS_2, WS_2) generate relatively intense flicker noise compared with the graphene layer but are more challenging to measure because of high DC resistance and the impact of external interferences [16,17]. You can compare flicker noise intensities in back-gated FETs comprising a single atom layer of graphene or MoS_2 in the channel by considering current spectral density fluctuations $S_I(f)$ normalized to squared polarizing current I^2 and multiplied by active area A of the applied 2DM [13]. It was reported that the product $S_I(f)/I^2 \cdot A$ is about 2-3 orders higher for MoS_2 than for graphene when the single atom layers of 2DMs were investigated in back-gated FET structures at selected low frequency (e.g., $f = 10$ Hz [13]).

Flicker noise in MoS_2 back-gated FET also depends on the ambient atmosphere and can be utilized for enhanced gas sensing (Fig. 2). The mechanisms of flicker noise generation in graphene and semiconducting 2DMs are different. Mobility fluctuations prevail in graphene [18]. Noise-generating adsorption-desorption events of gas molecules generate Lorentzian, with selected corner frequency. Therefore, the Lorentzian is visible over the low-intensity background flicker noise generated by mobility fluctuations.

Semiconducting 2DMs follow the McWhorter model of flicker noise induced by separate generation-recombination events of distributed time constants, resulting in 1/f-like noise

[13]. It is possible because semiconducting 2DMs exhibit a direct energy gap (e.g., single atom layer of MoS_2: 1.8 eV, WS_2: 2.05 eV) that is much higher when compared with semi-metallic graphene. The band gap can be modulated to give the distribution of its values and result in the observed 1/f-like noise (e.g., within defects in 2DMs structure or doping by various nanoparticles). This means it is less likely to observe the dominant Lorentzian component generated by gas molecules' adsorption-desorption events in noisy semiconducting 2DMs. Still, some exceptions are observed (e.g., at the ambient atmosphere of acetonitrile with $f_c \cong 90$ Hz – Fig. 2).

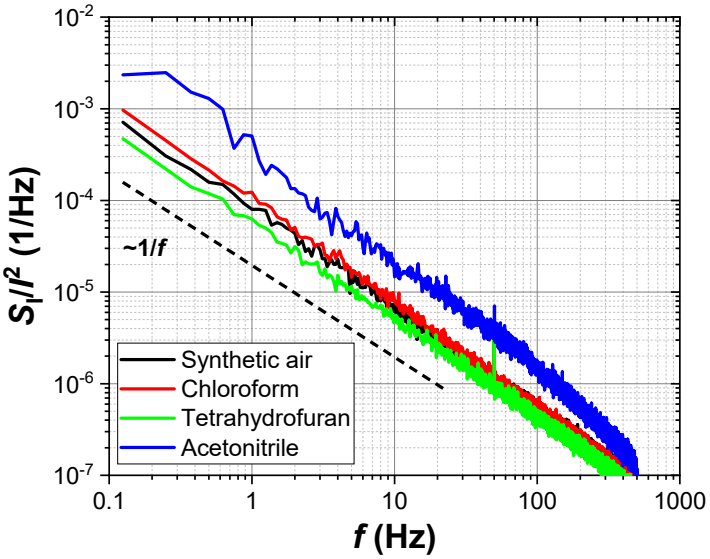

Fig. 2. Power spectral density $S_I(f)$ of current noise normalized to the squared polarization current I in ink-printed MoS_2 layer versus frequency f recorded in a few selected ambient atmospheres: synthetic air (black line), chloroform (red line), tetrahydrofuran (green line) and acetonitrile (blue line).

The semiconducting properties of 2DMs can be easily modified by operating at elevated temperatures and reducing the energy gap or by UV irradiation that generates weakly bounded oxygen ions and activates adsorption-desorption centers. These actions enhance gas sensitivity and response dynamics, reducing humidity's impact [19]. These remarks are especially important for some graphene sensors (e.g., graphene back-gated FETs) that are very sensitive to moisture and require mundane refreshments after a few days of operating in laboratory air. This detrimental effect can be reduced by not applying SiO_2 substrate under the graphene flake (e.g., replacing it with n-doped Si [20]). Unfortunately, none of these methods can improve gas selectivity. This issue is crucial when we must detect gas mixture components using a limited number of gas sensors. These limitations are the driving force behind proposing other modulation methods to enhance gas selectivity in 2DMs.

3. The perspective of gas sensing improvements

The gas sensing by the FES method can be improved by modulating imperfection centers responsible for the adsorption-desorption of the detected gas molecules. Recent experimental results suggest that MoS_2 layer imperfections can be switched on and off by noble metal nanoparticle decoration. It was proved theoretically by DFT calculations and experimentally for Au, Pt, and Pd nanoparticles that selected imperfections are more likely to be decorated by nanoparticles of different noble metals due to their various binding energy [21]. This effect drastically enhanced gas selectivity because only some imperfections of selected binding energy remain active (noble metal nanoparticles occupy others) and attract selected gas molecules. A change in MoS_2 gas sensor response (%) reached a few times for H_2 and NH_3 gas molecules when MoS_2 flakes were decorated with selected noble metals (Au, Pt, Pd). This result was obtained by applying a simple technological method of nanoparticle decoration by the solution process [21].

The results presented for enhancing gas selectivity by noble metal nanoparticles considered only the DC characteristic of the gas sensor. We can expect that flicker noise generated in 2DMs is sensitive to noble metal decoration and can be used for gas detection. There is experimental evidence that metal nanoparticles modify flicker noise very strongly in the presence of plasmonic resonance (e.g., in ZnO-based photodetectors, Au/Cu doping reduced the power spectral density of flicker noise a few orders [22]; a similar effect was observed in Au-decorated TiO_2 nanoparticles used in UV photodetector [23]). It is possible because metal nanoparticles are rather weakly bound to semiconductors and can operate as a charge source between metal nanoparticles and semiconductors. This effect reduces flicker noise when induced by generation-recombination processes in semiconducting materials. Moreover, the nanoparticles operate like spring-attached massive balls [24], absorbing the energy of acoustic phonon scattering in imperfections and reducing flicker noise when generated by mobility fluctuations. Thus, the same effect of flicker noise reduction can be expected for 2DMs of semiconducting or semi-metallic (graphene) properties. We are conscious that flicker noise generation in 2DMs is still an open research area, but the proposed explanations and the reported experimental results support our point of view.

The LSPR phenomenon induces additional charge within the nanoparticles (Fig. 3) when irradiated by light of a selected wavelength longer than the nanoparticle diameter [25]. The charge can be transferred between the primary material and the nanoparticle. The efficiency of this process depends on local conditions between the metal nanoparticle and the 2DM. It was proved experimentally that such structures exhibit gas-sensing properties [26,27]. Other sensing properties, utilizing different physical properties, can also be expected from such structures [24].

We underline that utilizing the LSPR phenomenon to advance the FES method is new and can be very fruitful. It has potentially two main advantages: (i) enhanced sensitivity due to the reported significant changes in low-frequency noise intensity and (ii) improved selectivity by modulating the LSPR induced by different irradiation wavelengths taking place in the nanoparticles of suitable noble metal. Moreover, the technology of decorating

2DMs with noble metal nanoparticles can be a solvent-based method easily implemented in laboratory or low-cost sensor production. The way of inducing the LSPR phenomenon by applying a few LEDs emitting various wavelengths is also attractive from a practical point of view. This method can be used for ink-printed gas sensors made of 2DM flakes.

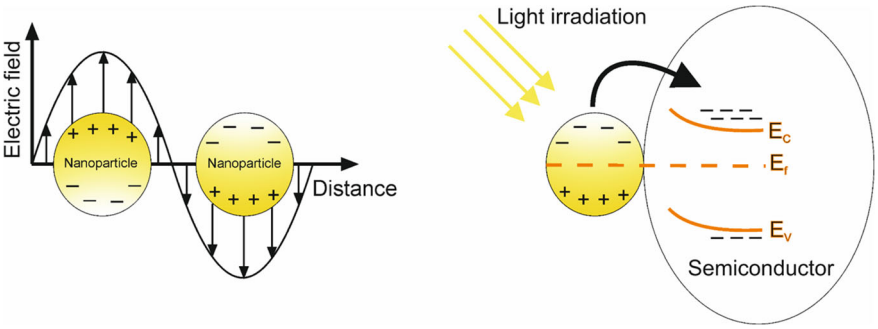

Fig. 3. Principles of a localized surface plasmonic resonance (LSPR) phenomena: a) charge shifts in noble metal nanoparticles when irradiated (left panel), b) electrons flow induced by an LSPR from noble metal nanoparticle into the adjacent semiconductor (right panel). E_f – Fermi level, E_c – the conduction band edge, E_v – the valence band edge.

The idea of decorating a gas sensor made of 2DM by noble metal and its impact on flicker noise was investigated in graphene-silicon Schottky diodes operating as a gas sensor under irradiation of selected wavelengths [20]. Its exponential DC characteristic is sensitive to the ambient atmosphere, and some gases can be detected at low concentrations (e.g., NO_2, with an estimated detection limit of 75 ppb). The sensors utilize a single-atom graphene layer of an active area of about 500 μm^2 or 2 500 μm^2 between the golden contact electrode and the edge of n-doped Si. The Schottky junction was created between graphene and n-doped Si. Au mono-sized (8 nm diameter) nanoparticles decorated such a gas sensor. Au nanoparticles were fabricated and deposited onto the graphene surface by the Advanced Deposition Technique, as presented in detail elsewhere [28]. The density of the Au nanoparticles was set not to exceed the percolation threshold. It secured sufficient number of Au nanoparticles to occupy numerous imperfections at the graphene surface and avoid short circuits of the graphene layer.

Flicker noise was measured in the prepared sensors and compared for a bias of 3.64 V in conductive mode and different ambient conditions. As discussed earlier, we compared the same product, $S_I(f)/I^2 \cdot A$, for the electronic devices made of MoS_2 or graphene flakes. This product was almost two orders lower when the sample was Au-decorated (Tab. 1). It confirmed our presumptions about the impact of Au nanoparticles on low-frequency noise. Next, flicker noise was measured when the Au-decorated Schottky diode was in the dark or irradiated by yellow light (592 nm). We considered power spectral densities of resistance fluctuations normalized to the squared serial resistance of the studied Schottky diode [28]. Our results confirmed that the observed changes in noise intensity with two selected gas

molecules of various chemical properties (tetrahydrofuran, being a Lewis base, and chloroform, being a hard acid) were more significant when the sensor was irradiated by yellow light (about 2.8 times). The changes were minor (<1.4 times) in dark or under blue irradiation. The amplified gas detection effect was attributed to the LSPR phenomena induced by yellow light irradiation. This means the FES method can be applied more efficiently when the LSPR phenomenon occurs.

Table 1. Flicker noise product $S_I(f)/I^2 \cdot A$ observed at a frequency f = 10 Hz in graphene-Si Schottky diodes (Au-nanoparticles decorated and without decoration) biased by voltage 3.64 V in conductive mode at dark and ambient atmosphere of synthetic air.

Schottky diode	$S_I(f)/I^2 \cdot A$ [$V^2/Hz^2 \cdot \mu m^2$]
Au-decorated	$48 \cdot 10^{-7}$
Not decorated	$38 \cdot 10^{-5}$

The gas sensing mechanisms of 2D layered materials include two effects: (i) surface charge transfer induced by the adsorbed molecules and (ii) Schottky barrier and built-in voltage modulation [29]. When the sensor is decorated with noble metal nanoparticles, both mechanisms can be present, and both mechanisms can impact flicker noise generation at various voltage biases in different ways. It suggests that voltage bias shifting can expose various gas sensing mechanisms present in such sensors, enhancing gas detection. In the case of the reported experiment, when the bias voltage was 3.64 V in conducting mode, the mechanism of surface charge transfer prevails.

The impact of the LSPR phenomena on flicker noise generation is yet an open question. Nevertheless, there are reports that the LSPR induced flicker noise at various intensities when the bias voltage was changed (e.g., in ions crossing golden nanopores [30]). Our studies of flicker noise in Au-decorated graphene-Si Schottky diodes included estimating a noise probability distribution. We observed a change in the symmetry of noise probability distribution only when the sensor was irradiated by yellow light, inducing the LSPR phenomenon [31]. Other light sources (e.g., blue light) did not introduce such an effect. This suggests that the area where flicker noise was generated was tiny, and some discrepancies from normal distribution can be identified.

We expect even better gas sensing by the FES method in the presence of the LSPR phenomena in semiconducting 2DMs (e.g., MoS_2, WS_2) with different band gaps. These materials can be decorated with other noble metals (e.g., Pb, Pd, Au) that selectively occupy various imperfections, as reported elsewhere [21]. The LSPR phenomenon amplifies gas selectivity and can be realized when a few different light wavelengths irradiate a single gas sensor.

Any practical application of the FES method for gas sensing requires an algorithmic tool to determine gas concentration. A few algorithms were developed to evaluate flicker noise changes (intensity and slope versus frequency) for the FES method [32,33]. We propose to model the noise power spectrum as a sum of 1/f noise and a few Lorentzians, as proposed in a theoretical model of adsorption-desorption noise generation in gas sensors [34]. The

algorithm can identify the Lorentzians' characteristics for selected gases using deep learning methods and noise power spectrum as an input data vector. This approach enables the automatic detection of gas mixture components occurring in the ambient atmosphere of gas sensors. It can be developed to avoid the necessity of an algorithm adjustment by the operator. Based on 2DMs, the sensing platform and the algorithm create the measurement system ready to detect gas mixtures.

4. Conclusions

We presented perspectives on enhancing the FES method in 2DMs using the LSPR phenomenon. The exemplary experimental results confirm that the sensitivity of gas sensing when using the FES method can be improved by decorating the 2DM with noble metal nanoparticles. The decoration reduces flicker noise, and its intensity is sensitive to the ambient atmosphere – the conditions between nanoparticles and the 2DM applied. When the LSPR phenomenon takes place, the gas sensitivity increases. The LSPR can be induced by light irradiation selectively using different light wavelengths for different noble metal nanoparticles decorating 2DM. The presented idea was confirmed for Au nanoparticles, but more in-depth studies are necessary when other noble metal nanoparticles are applied. Our approach enhances the effects induced by noble metal nanoparticles that were commonly used to advance gas sensing due to their catalytic properties [35].

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