



# Enhanced acetone sensing properties of monolayer graphene at room temperature by electrode spacing effect and UV illumination

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## ABSTRACT

An improvement in the response of a graphene-based acetone sensor is achieved by the combination of optimizing the spacing between the electrodes and UV illumination during the sensing measurement. A monolayer graphene membrane was grown on Cu foil by low pressure chemical vapor deposition, and then, it was transferred to an interdigitated electrode to achieve a resistive-type gas sensor. The response of the sensor to acetone was small without UV light illumination, but improved 7 times for an acetone concentration of 1000 ppb with UV illumination continuously applied during the measurement. In addition, the sensor response for a spacing of 400  $\mu\text{m}$  was approximately 2X higher compared to that for a spacing of 50  $\mu\text{m}$  for acetone concentrations of 100–1000 ppb. Furthermore, short-time UV illumination performed on these graphene-based sensors resulted in low power consumption and achieved similar results compared to continuous UV illumination. The origin of these improvements could be due to the reduction of hole carriers by oxygen and water molecule desorption from the graphene surface due to UV illumination. The surface area ratio of graphene on the glass showed a strong positive correlation to the acetone response and sensitivity, which could be related to the higher affinity in the unit sensing area. The substrate doping effect could be an important factor for the graphene-based gas sensors.

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## 1. Introduction

Graphene-based gas sensors have gained increased attention in recent years, mainly due to their two-dimensional nature and large specific surface area with the ability to achieve single-molecule detection [1–3]. A graphene based gas sensor was first demonstrated by Schedin et al. [1] by taking advantage of the change in carrier concentration, according to electron transfer theory, following gas adsorption. Inspired by this work, various studies have reported on the detection of  $\text{NH}_3$  [4],  $\text{NO}_2$  [5],  $\text{CO}_2$  [6],  $\text{CO}$  [7],  $\text{H}_2\text{S}$  [8] and  $\text{CH}_3\text{COCH}_3$  [9]. Few studies could be found regarding the incorporation of metal oxide nanoparticles in graphene for

high-performance acetone sensors especially at room temperature [9–13]. Liu et al. reported that a sensor based on graphene with  $\text{ZnFe}_2\text{O}_4$  operating at 275°C exhibited good selectivity and reproducibility for 10 ppm acetone vapor [9]. Zhang et al. demonstrated a room temperature acetone sensor with a fast response time and good repeatability using a  $\text{SnO}_2$ -reduced graphene oxide (RGO) hybrid composite as the sensing film [10]. However, the acetone concentration ranged only from 10 to 2000 ppm. Kehayias et al. combined electronic transport and Kelvin probe microscopy measurements to quantify the amount of charge carriers transferred to RGO in acetone and ammonia, respectively [11]. The change in the density of the charge carriers after acetone adsorption was calculated as  $5 \times 10^{11} \text{ cm}^{-2}$ , which was smaller than that of ammonia.

The presence of acetone in breath could be an important biomarker for diabetes patients [12]. [The current practice of diagnosing diabetes involves blood sampling, which is cumbersome, invasive and painful. Therefore, diagnostic sensing devices using

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the exhaled breath have been presented to obtain a better solution for this problem [13]. The detection of acetone is also of great interest in various other applications such as industrial production, environmental monitoring and chemical leakages [14]. Acetone acts as a weak p-type dopant on graphene [15]. Acetone sensing performance could be enhanced by increasing the available specific reaction area. The reaction area has been shown to be affected by the percentage of strain [16,17] as well as the presence of defects and grain boundaries. Domain boundaries consist of a wide variety of defective structures under mechanical strain [18], but the most common are alternating pentagon-heptagon (5–7) rings, which are similar in structure to Stone-Wales defects [19]. Bissett et al. demonstrated that the chemical reactivity of graphene can be tuned by applying external strain via stretching of the supporting flexible substrate [20]. Dutta et al. used van-der-Waals-corrected density functional theory to calculate the adsorption energy of CO<sub>2</sub> on strained graphene, in which the highest adsorption energy occurred at 20% compressive strain [21]. Sun et al. demonstrated UV illumination-induced molecular desorption from graphene, leading to an increase in the reaction area for adsorbing acetone on the graphene surface [22]. Until now, UV illumination and strained graphene for acetone sensing have not been clearly investigated. In this study, the effect of optimization of the spacing between the Au electrodes and UV illumination is presented to improve the performance of graphene-based acetone sensors at room temperature. Four different spacing distances and three different of UV illumination modes (no illumination, short-duration and continuous UV illumination) were used as the experimental parameters. The monolayer graphene could be strained by the different electrode gaps, which was also investigated using Raman mapping of the shift of the G band at different locations.

## 2. Experimental

### 2.1. Graphene growth

The monolayer graphene was grown on commercial 25- $\mu\text{m}$  thick polycrystalline copper foils (Alfa Aesar, US, No.13382, thickness: 25  $\mu\text{m}$ , purity: 99.8%) by low pressure chemical vapor deposition [23,24]. First, the copper foil was loaded into a quartz tube evacuated down to 1 mTorr. The temperature of the furnace (TF55030, Lindberg/Blue/M) was increased to 1000 °C with 80 sccm hydrogen flow and the pressure was controlled at 1 Torr for 60 min. Annealing the Cu foil in hydrogen (H<sub>2</sub>) helps to improve the surface quality by reducing the native oxide layer. Methane (CH<sub>4</sub>) and H<sub>2</sub> were injected into the quartz tube at a flow rate of 100 and 1 sccm, respectively to obtain a fully covered monolayer of graphene on the Cu surface in 20 min. Finally, the quartz tube was cooled down to 200 °C with a cooling rate of 5 °C\*s<sup>-1</sup> before removing the Cu foil.

### 2.2. Sensor fabrication

A resistive gas sensor was fabricated with a monolayer of graphene transferred onto the interdigitated gold electrodes. The width of the single electrode was designed to be 50  $\mu\text{m}$ , and the spacing between the neighboring interdigitated electrodes (IDEs) was designed with different gaps including 50, 100, 200 and 400  $\mu\text{m}$  in the mask for pattern transfer during the photolithography process. Following the lift-off process, a photoresist was patterned on the glass substrate. Then, Cr (10 nm)/Au (150 nm) was deposited on the surface. The Cr and Au layers deposited on the photoresists could be removed using a lift-off process with acetone solution. IDEs made by the Cr and Au layers on glass were obtained after the lift-off process. Next, the top side of the copper foil with graphene was covered with polymethyl methacrylate

(PMMA; M~950,000, MicroChem), and the copper substrate was removed in a FeCl<sub>3</sub> (97%, Alfa Aesar) solution (1 mol/l). The PMMA layer on the graphene structure was then washed with deionized water and transferred to the top of the IDEs. Then, PMMA was dissolved in acetone solution. The graphene was defined by photolithography and another mask for the sensing area, followed by O<sub>2</sub> plasma etching. Then, the photoresist was dissolved in acetone solution. Fig. 1 shows the schematic of the experimental setup with an image of the sensor chip with four different IDE designs.

### 2.3. Gas sensing measurement setup

Fig. 1 shows the acetone sensor system including the gas generator and electrical measurement setup. The volume of the testing chamber was designed to be 3 liters. A UV light-emitting diode (LED) (370 nm, 70 mW, KOODYZ) was fixed at a distance of 5 mm in front of the resistive gas sensor with a monolayer graphene. The UV LED was connected to a power supply with the ability to adjust the bias during illumination. For gas sensing measurements, a small amount of acetone was carried by dry N<sub>2</sub> through a permeation tube into the test chamber. The acetone concentration could be automatically controlled by the 491M type gas standards generator from KIN-TEK Laboratories Inc. (La Marque, TX, USA). Various concentrations of acetone were produced by adjusting the ratio of the flow rate of the test gas to that of the carrier gas. The gas sensing response was monitored by the change in resistance caused by different acetone concentrations. The resistance values were recorded by a multimeter (HP34401A) to study the real-time response of the acetone sensor. A purge process with pure N<sub>2</sub> was used to remove the residual acetone in order to test the recovery performance of the gas sensors. All measurements were performed at room temperature and atmospheric pressure to match the conditions of real biomedical applications. Normally, the gas response in units of percentage of resistance is defined as  $(R_t - R_i)/R_i \cdot 100$ , where  $R_t$  is the resistance of the sensor measured at different time, and  $R_i$  is the initial resistance before acetone injection in first cycle [25].

### 2.4. Material analysis

The spatially resolved maps of the G band for the monolayer graphene on the IDEs with different gaps were obtained using an NT-MDT confocal Raman microscope system at the excitation wavelength of 473 nm. The Raman maps were used to check the G band differences between graphene on the glass and IDEs. The spot size of the laser was 0.5  $\mu\text{m}$ , and the pixel size of the map was 1  $\mu\text{m}$ . The Raman scattering peak of Si at 520 cm<sup>-1</sup> was used as a reference for wavenumber calibration before all measurements.

### 2.5. UV illumination and acetone sensing measurements

Before the gas sensing measurements, all sensors were stabilized by N<sub>2</sub> at a flow rate of 5000 sccm for 25 min for initialization. During the actual gas sensing measurement cycle, the concentration of acetone gas was controlled at 100 ppb, 500 ppb and 1000 ppb for 4 min by using the 491M type gas standards generator. Next, a desorption process using pure nitrogen for 4 min removed the residual acetone. There were three groups with different UV illumination periods designed in this study. In the first group, no UV illumination was used during the whole measurement as the control group, called as the “No UV” group. Next, a short UV illumination was introduced only for the first 5 min of the nitrogen purge, termed as the “Short UV” group. This time limit was chosen because 5 min recovery time is acceptable in sensor characterization. In the third group, UV illumination was turned on continuously during

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