

# A Fast and Room-Temperature Operation Ammonia Sensor Based on Compound of Graphene With Polypyrrole

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**Abstract**—We report a new hybrid sensor in which an ultrathin polypyrrole (PPy) layer is deposited on the CVD-grown graphene (G) by electropolymerization. The sensor exhibits an excellent performance in the selective sensing of ammonia (NH<sub>3</sub>) at room temperature, having a high, fast, and reversible response. It also shows rather good stability, reproducibility, and immunity to humidity. These performances are attributed to the synergistic effect between the PPy layer and G. Specifically, the NH<sub>3</sub> molecules are adsorbed on the PPy layer, electrons transfer from NH<sub>3</sub> to the PPy layer, changing the PPy layer resistance. Electrons can also be transferred to the G through the ultrathin PPy layer, in certain way changing the G resistance. These promote the sensor sensitivity. Moreover, the ultrathin PPy layer with porous nature plays an important role in the sensor response, selectivity, and immunity to humidity. Graphene is not only a support material for PPy electropolymerization but also provides an efficient pathway for electron transfer, thereby, accelerating the sensor response and recovery. Our research indicates that the combination of PPy and G is a very promising as a chemical sensor material. We implement a smart prototype with the present sensor in an electronic board for real-time monitoring the NH<sub>3</sub> concentrations. The smart prototype can be connected to a computer by a USB port for demonstrating NH<sub>3</sub> concentrations, transmitting the data and analyzing the sensor response curves. Our results are beneficial forward the commercial design and fabrication of sensors fulfilling the specifications of practical applications.

**Index Terms**—Graphene, polypyrrole, ammonia, hybrid sensor, smart prototype.

## I. INTRODUCTION

AMMONIA (NH<sub>3</sub>) is one of the most harmful pollutant gases. NH<sub>3</sub> injures the human eyes, skin, respiratory tract, liver and kidneys beyond 25 ppm concentration [1], [2].

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Meanwhile, it plays a decisive role in particulate matter (PM) formation. Most of NH<sub>3</sub> in our life environment is emitted directly or indirectly by chemical industries and human activities. Although plenty of NH<sub>3</sub> sensors have been studied and investigated in the literature [3], [4], it is still a great challenge to develop high performance devices. Recently, NH<sub>3</sub> sensors have found a new potential in medical applications. Specifically, NH<sub>3</sub> sensors can be used to diagnose certain diseases through detecting NH<sub>3</sub> concentration from human breath. It was reported that the patients with renal disorders or ulcers exhale NH<sub>3</sub> concentration in the range from 0.8 to 14 ppm, whereas the exhalation of the good health person is in the range of 0.15–1.8 ppm [5]. Metal oxides, catalytic metals and conducting polymers were used as sensing materials in NH<sub>3</sub> sensor fabrication. Some of them may be expensive, need high-temperature operation and high power consumption, have a short lifetime, or low selectivity. Therefore, there is an urgent requirement to develop NH<sub>3</sub> sensors which are sensitive, specific, fast, reliable, operating at room-temperature and suitable for real-life humidity condition.

Graphene (G) has attracted much attention for sensor applications. Single layer graphene with a large specific surface area of 2630 m<sup>2</sup>/g [6] provides the number of binding sites to yield high sensitivity. The graphene resistance is very sensitive to its carrier concentration. The adsorption of target molecules leads to the electron donation or withdrawal from graphene, which makes the graphene resistance significantly change. Particularly, the high carrier mobility of graphene (200 000 cm<sup>2</sup>/s.V) [7] inherently ensures low electrical noise and low power consumption. As a result, extremely small change in the graphene resistance caused by target adsorption is detectable, possessing the potential ability to detect one single molecule [8]. It is well known that pristine graphene is chemically inert and weakly binding with target molecules [9]. However, graphene is easy to be functionalized by organic compounds, such as polymers that can interact with target molecules [10]. Graphene functionalization can be either covalent or non-covalent [11]. The former disrupts graphene electronic and mechanical properties, whereas the later improves the graphene binding ability and maintains the graphene original properties simultaneously. It is important to optimize functionalization methods with non-covalent bonds, which act only as specific links.

TABLE I  
SUMMARY OF RECENT RESEARCH RESULTS FOR NH<sub>3</sub> SENSORS BASED ON PPy FILM, rGO, THEIR COMPOUNDS,  
AND RELATED MATERIALS, OPERATING AT ROOM TEMPERATURE

Sensor Type	Sensing Material	Synthesis Method	Sensitivity	Detection Limit	Response Time	Recovery Time	Reference
IDA	PPy film	Electropolymerization	55%/500ppm	8 ppm	>100 min	>120 min	[28]
IDA	PPy film	Electropolymerization	16%/40ppm	3 ppm			[29]
IDA	PPy+rGO	Chemical Reduction	2,4%/1ppb	1 ppb	1,4 s	76 s+IR	[27]
IDA	PPy nanofibers	Chemicalpolymerization		10 ppm	10 s	25 s	[30]
IDA	PPyNTs+Ag	Chemicalpolymerization	54%/40ppm	10 ppm		500 s	[31]
IDA	G-PEDOT:PSS	Chemicalpolymerization	9,6%/500ppm	5 ppm	3 min	5 min	[32]
IDA	PPy+rGO	Chemical Reduction	22%/100ppm			310 s	[33]
IDA	PANI+rGO	Chemical Reduction	37,1%/50ppm	20 ppm	18 min	2 min	[34]
IDA	PANI+G	Chemicalpolymerization	3.65/20ppm	1 ppm	50 s	27 s	[35]
IDA	PPy+s-CoPc	Electropolymerization	2%/25ppm	25 ppm	<1 min	<1min	[36]
Chemiresistor	G (CVD)		3%/500ppb		360 min	360 min	[37]
Chemiresistor	PPy+rGO	Chemical Oxidation	34,7%/500ppm	3 ppm	400 s		[26]
Chemiresistor	PPy NTs/NWs	Electropolymerization	10%/1,25ppm	1 ppm		> 40 min	[4]
Chemiresistor	PPy+rGO+TiO <sub>2</sub>	Chemicalpolymerization	102%/50ppm	1 ppm	36s	16 s	[38]
Chemiresistor	PPy+MWCN	Chemical Oxidation	3,07%/200ppm		34 s	3 min	[39]
Chemiresistor	PPy NTs/NRs	Chemical Oxidation	44%/100ppm	20 ppm	5 min	5 min	[2]
Chemiresistor	PPy/GO aerogel	Freeze dried	40%/800ppm		10 min		[40]
Chemiresistor	PANI+rGO	Chemicalpolymerization	344.2/100ppm		20 s	27 s	[41]
Chemiresistor	PPy NW	Chemicalpolymerization	0,06%/1ppm	40 ppm	15 min		[42]
FET	PPy Nribbons	Electropolymerization	2%/0.5ppm	0.5 ppm	8 min	3 min	[43]
Chemiresistor	PPy+G(CVD)	Electropolymerization	1.7%/1ppm	1 ppm	2 min	5 min	This work

G: graphene, IDE: interdigitated electrodes, FET: field-effect transistor, PANI: polyaniline, PPy: polypyrrole, rGO: reduced graphene oxide, NWs: nanowires, NRs: nanorods, NTs: nanotubes, MWCN: multi-walled carbon nanotubes, s-CoPc: n-sulfonated cobalt phthalocyanines. The rest of the abbreviations can be found in the related references.

Since the Manchester group demonstrated the detection of a single molecule (NO<sub>2</sub>) in 2007 [8], graphene-based sensors have been exploited for detecting various types of gases, for example NH<sub>3</sub>, O<sub>2</sub>, NO<sub>2</sub>, H<sub>2</sub>, CH<sub>4</sub>, SO<sub>2</sub>, H<sub>2</sub>S, and VOCs at concentrations ranging from percentage levels down to parts per billion (ppb) [12]–[16]. For the NH<sub>3</sub> sensors, the research works mainly focus on reduced graphene oxide (rGO). Although rGO can be prepared at relatively low cost and in large scale [17], the contact resistance between metal electrodes and rGO makes the sensor response complex and not well understood. The literature presents contradictory reports: Fowler *et al.* [18] claimed that the contact resistance does not influence their sensing process. Whereas Lu *et al.* [19] believed that the contact resistance contributes to the overall sensing response since the Schottky barrier variation is induced by the adsorbates.

Polypyrrole (PPy) has shown good performance for gas sensing. Apart from that, PPy is stable and biocompatible [20]. The most interesting feature of the PPy sensor is its ability to properly operate at room temperature. The first NH<sub>3</sub> sensor with PPy as sensing material goes back to 1983 by Nylander *et al.* [21]. They used a filter paper impregnated by PPy to measure NH<sub>3</sub> vapor. The compounds of rGO with PPy have been extensively exploited as electrode materials for

electrochemical supercapacitors [22], [23]. On the other hand, these materials can be used in gas sensors because of their high sensitivity, easy synthesis, and cost effectiveness [24]. Jang *et al.* [25] indicated that the improved sensitivity of the PPy/rGO sensor is mainly due to the effective electron transfer between NH<sub>3</sub> and PPy, as well as the efficient electron pathway in rGO. Tiwari *et al.* [26] further found that incorporation of rGO into PPy improves not only the sensitivity but also the response time. Although the PPy/rGO sensor reported by Hu *et al.* [27] exhibits a good sensitivity and selectivity to NH<sub>3</sub>, the sensor recovery is relatively slow (longer than 5h) in ambient condition.

According to the literature survey, we summarized in Table I the recently published NH<sub>3</sub> sensors based on G, rGO, PPy film, compounds of PPy and rGO, and related materials, operating at room temperature. For the pure PPy nanoribbon sensor, the minimum limit of detection to NH<sub>3</sub> is 0.5 ppm. Its response time and recovery time are about 8 min and 3 min, respectively. The hybrid sensors based on PPy and graphene are still rarely studied. We did not find NH<sub>3</sub> sensors made by the compound of the CVD-grown graphene and PPy. In this work, we designed a new NH<sub>3</sub> sensor, in which an ultra-thin layer of PPy is deposited on the CVD-grown graphene by electropolymerization. Hereafter, our sensor refers to the

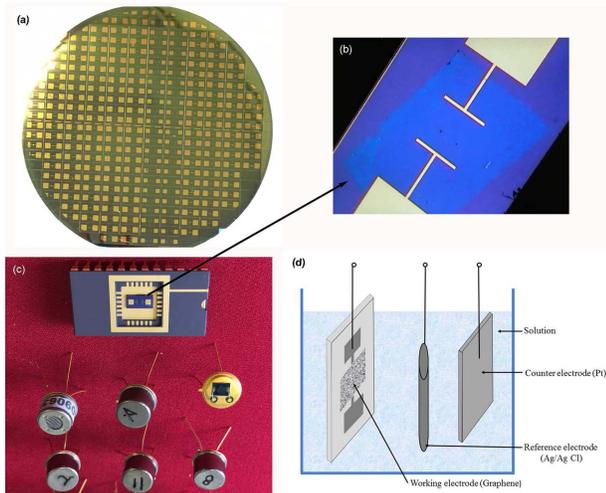


Fig. 1. (a) 3-inch Si wafer with predefined Au/Ti electrodes; (b) the CVD-grown graphene transferred on top of both electrodes; (c) PPy/G sensors bonded to TO-8 package or 24 pin dual-in-line package; and (d) a schematic illustration of electropolymerization setup.

PPy/G sensor. The sensor response, selectivity, reproducibility, and stability are examined. The influence of humidity on the sensor resistance is also tested. The key parameters of our sensors are also listed in table I for comparison purpose. We accomplished a smart prototype, which can monitor the sensor responses and the related  $\text{NH}_3$  concentrations in real-time on its liquid-crystal display. The smart prototype can also be connected to a computer by a USB port to transmit/analyze the data and show the response curves.

## II. EXPERIMENTAL

### A. Hybrid Sensor Fabrication

The fabrication of the PPy/G sensor consists of three steps: (i) the electrode deposition by a lift-off process, (ii) graphene transfer, and (iii) PPy electropolymerization. A 3-inch Si wafer is used as a starting substrate. 90-nm-thick  $\text{SiO}_2$  is thermally grown on the substrate for easily observing graphene with an optical microscope. The wafer is spin coated with positive photoresist to pattern electrodes with different sensing areas. Titanium (as an adhesion layer with the  $\text{SiO}_2$  surface) and gold layers with thicknesses of 5 and 50 nm respectively are deposited onto the photoresist. The wafer is then soaked with acetone for the lift-off process. As a result, many pairs of Au/Ti electrodes are predefined on the wafer (see Fig. 1a). The wafer is then divided into dies with an area of  $3 \times 6 \text{ mm}^2$ . Each die includes a pair of electrodes (see Fig. 1b). The graphene used for the sensor fabrication is grown on a Cu foil by CVD with methane as a precursor. The CVD-grown graphene is transferred on top of both electrodes by using poly(methyl methacrylate) (PMMA) as a mechanical support. The details of the graphene growth and transfer can be found in our previous work [44], [45]. The die with the CVD-grown graphene is glued and wire-bonded in a 24 pin dual-in-line package or TO-8 package (see Fig. 1c). Both kinds of packages can be directly contacted with our smart prototype, which will be demonstrated in the following section. It is important to point out that we use CVD-grown graphene for mass production of sensors. Moreover, the post-step graphene

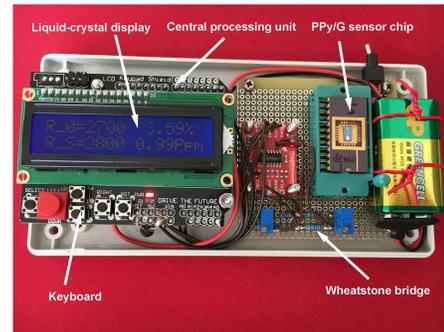


Fig. 2. A smart prototype with a PPy/G sensor for  $\text{NH}_3$  detection.

transfer is compatible with CMOS technology. These choices make it possible to design and fabricate graphene sensors with integrated circuits onto the same Si wafer in future work.

The last step in the sensor fabrication is the PPy synthesis on the CVD-grown graphene, which is carried out by electropolymerization at  $20^\circ\text{C}$ . Figure 1d illustrates the electropolymerization setup, in which the bonded die with the CVD-grown graphene is used as working electrode. The counter and reference electrodes are made of Pt and Ag/AgCl, respectively [46]. The electropolymerization bath consists of a solution of pyrrole (0.35 ml) and  $\text{NaClO}_4$  (0.6 g) in acetonitrile (50 ml). When a voltage pulse of 0.9 V is applied to the working electrode for 2 cycles, an ultrathin PPy layer of 20 nm with a porous nature is synthesized on the CVD-grown graphene. To study the effect of the PPy layer thickness on the sensor performance, a thicker PPy film with a thickness of 500 nm is also synthesized by increasing the number of voltage pulses under the same conditions. After the electropolymerization, the samples were washed with ethanol.

### B. Sensing Measurements

The relative change in resistance between the two electrodes is used to define the sensor response  $\Delta R/R_0 = (R_S - R_0)/R_0(\%)$ , where  $R_0$  and  $R_S$  are the sensor resistance before and after exposure to the target gas, respectively. The response time is defined as the time to reach 90% of the total resistance change, whereas the recovery time refers to the time required for recovering the measured resistance to 90% of its original value. Hereafter, we refer to the sensing measurements carried out at atmospheric pressure with a temperature of  $20^\circ\text{C}$  and a wet air (relative humidity of 50% RH), unless otherwise noted. Wet air and  $\text{NH}_3$  are used as carrier gas and target gas, respectively. The desired  $\text{NH}_3$  concentration is obtained by dilution of 100 ppm  $\text{NH}_3$  with wet air. For example, for 5 ppm  $\text{NH}_3$  concentration, we mix 50 ml/min of 100 ppm  $\text{NH}_3$  in air with 450 ml/min of dry air and 500 ml/min of saturated wet air (100% RH). The total gas flow is always kept at 1000 ml/min throughout a testing chamber of  $3 \text{ cm} \times 3.5 \text{ cm} \times 4 \text{ cm}$  (ca.  $42 \text{ cm}^3$  in internal volume) in all experiments.

### C. Smart Prototype Preparation

Figure 2 demonstrates our smart prototype, which includes a Wheatstone bridge, a central processing unit (CPU), a liquid-crystal display (LCD), a “keyboard”, and a PPy/G sensor. The Wheatstone bridge is biased by a 9V battery. To avoid the polarization effect on the PPy/G sensor, we ensure that

the applied voltage for the sensor is less than 0.5 V. The voltage differences across the galvanometer of the Wheatstone bridge are measured to calculate the sensor resistances  $R_0$  and  $R_S$ . Both resistances are provided to an analog-to-digital converter and then the sensor response is directly read by CPU. Using a relationship curve between  $\Delta R/R_0$  and different concentrations of  $\text{NH}_3$ , the measured gas concentrations (in ppm) can be derived and also displayed on LCD. The initial resistance of the sensor  $R_0$  is recorded in a non-volatile memory (by pressing the red bottom in the keyboard). The measurement interval is set, by default, to 1 second. This smart prototype can also be connected to a computer by a USB port for real-time monitoring, transmitting the data and analyzing the response curves of the sensors. It is worth noting that other gas sensors can be assembled with the present smart prototype.

### III. RESULTS AND DISCUSSION

#### A. Working Principle of the $\text{NH}_3$ Sensor

Schedin *et al.* reported that  $\text{NH}_3$  is an electronic donating molecule [8]. The PPy layer behaves like a  $p$ -type semiconductor [47]. The adsorption of  $\text{NH}_3$  on the PPy surface induces an interaction between the  $\text{NH}_3$  molecules and the PPy. The electron transfer from the  $\text{NH}_3$  molecules to the PPy layer reduces the hole concentration of the PPy layer, leading to an increase of the PPy resistance. Since the PPy electropolymerization cannot be performed directly on insulators, an intermediate layer, such as metal or graphene, must be used as a growth support. However, metallic layers are too conductive to detect small resistance changes upon exposure to  $\text{NH}_3$ . In consequence, we propose to use graphene (instead of metal) because it allows a measurable modification in resistance and tuning the PPy thickness down to nanoscale range. On the other hand, graphene behaves like a  $p$ -type semimetal [48]. When it is functionalized with the PPy, the electrons from the  $\text{NH}_3$  molecules can also be transferred to graphene through the PPy layer, in certain way thereby increasing the graphene resistance. We thus stack the PPy layer and graphene to combine their respective properties in a synergistic way. In summary, the ultrathin PPy layer with a porous nature plays an important role in electron exchange with  $\text{NH}_3$ , whereas graphene not only serves as a support material for PPy synthesis, but it is also an electron reservoir and a suitable pathway (i.e., not too conductive) for electron transfer.

#### B. Physical Characterization

The morphology of the CVD-grown graphene and PPy/G compound is characterized by optical microscopy and scanning electron microscopy (SEM). Figure 3a shows a top-view optical microscopy image of the CVD-grown graphene before the PPy synthesis. It can be seen that the CVD-grown graphene is composed of isolated and contiguous hexagonal flakes. Most part of graphene is single-layer. Bi- and trilayer graphene areas are found in certain regions. Figure 3b exhibits a top-view SEM image of the PPy/G compound. The PPy layer has a thickness of about 20 nm, measured by a micro system analyzer (PolyTec MSA-500). Some cavities can be observed

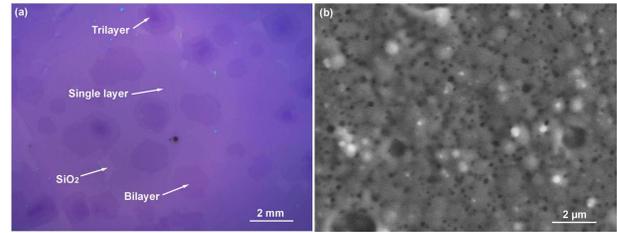


Fig. 3. (a) A top-view optical microscopy image of graphene before the PPy synthesis; and (b) a top-view SEM image of the PPy/G compound, showing a porous nature.

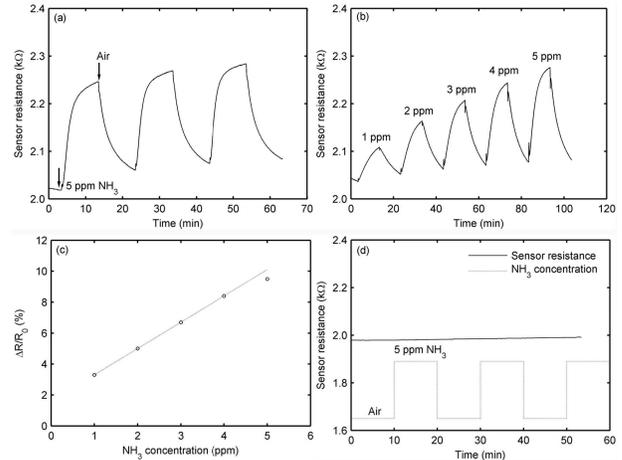


Fig. 4. Sensor behavior of a typical PPy/G sensor at relative humidity of 50% and at 20°C: (a) the sensor resistance exposed to 5 ppm  $\text{NH}_3$ ; (b) the sensor resistance exposed to  $\text{NH}_3$  concentration in a range from 1 to 5 ppm; (c) the sensor response as a function of  $\text{NH}_3$  concentration; and (d) the sensor resistance of a graphene sensor without PPy layer exposed to 5 ppm  $\text{NH}_3$ .

in the ultrathin PPy surface, which is crucial to allow the  $\text{NH}_3$  molecules to quickly come into the PPy/G compound and react with it. A detailed discussion can be found in the following sections.

#### C. Sensor Response

Figure 4a shows the resistance of the PPy/G sensor, exposed to three cycles of 5 ppm  $\text{NH}_3$ . The baseline resistance of the sensor is 2036 Ohm in wet air (50% RH at 20 °C). The  $\text{NH}_3$  exposure time is 10 min, followed by 10 min of purge with wet air. The sensor has a repeatable response of about 10%. When the PPy/G sensor is exposed to  $\text{NH}_3$ , its resistance is increased. This phenomenon is attributed to the electron transfer from the  $\text{NH}_3$  molecule to the PPy/G sensor [49]. Figure 4b and 4c present the sensor resistances and the sensor responses to different concentrations of  $\text{NH}_3$ , respectively. A linear relationship between the sensor response and  $\text{NH}_3$  concentration can be observed in the range from 1 to 4 ppm. Above concentrations of 4 ppm, the sensor response deviates from the linear curve due to the saturation of the PPy/G layer. The sensor sensitivity is evaluated to 1.7%/ppm by the slope of the linear curve. This value is comparable to those of the most sensitive  $\text{NH}_3$  sensors reported in table 1. It is emphasized that the PPy/G sensor also shows a much higher response to  $\text{NH}_3$ , compared with the graphene sensor without PPy layer (see Fig. 4d). This fact confirms that the ultrathin PPy layer plays an important role in the present sensor. Pristine graphene

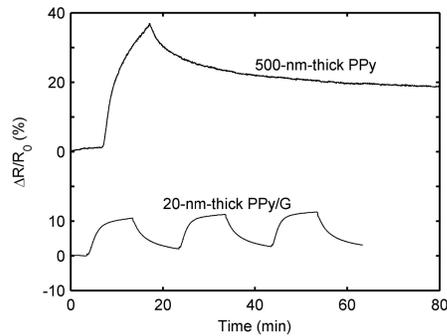


Fig. 5. Resistance responses of the PPY/G sensors with a PPY thickness of 20 nm (bottom) and 500 nm (top), exposed to 5 ppm  $\text{NH}_3$  at a relative humidity of 50% and at 20°C.

interacts very weakly with  $\text{NH}_3$  because it is chemically inert and free of dangling bonds [50]. In conclusion, the high response of our sensor is mainly attributed to the PPY layer.

#### D. Influence of PPY Thickness on Sensor Response and Recovery

We calculate the average response and recovery times of the 20-nm-thick PPY/G sensors, which are 2 and 5 min, respectively. Figure 5 displays the real-time responses to 5 ppm  $\text{NH}_3$  for the 20-nm-thick and 500-nm-thick PPY/G sensors. The response curves are shifted in the figure relatively to each other for easy comparison. Although the response of the thin PPY/G sensor is lower than that of the thick PPY sensor, the former can be fully recovered in a shorter time. The thin PPY layer provides less reactive sites for the adsorption of  $\text{NH}_3$  molecules, leading to a lower response. However, the desorption of  $\text{NH}_3$  molecules from the cavities existing in the thin PPY layer is much faster since the porous structure makes the  $\text{NH}_3$  diffusion easier and the thin PPY layer reduces diffusion pathways. The slower desorption of  $\text{NH}_3$  from the thick PPY sensor is due to the longer diffusion pathways which translate to a longer recovery time. The insufficient recovery of the thick PPY sensor would lead to unreliable sensor outputs and hysteresis effects.

It is worth noting that  $\text{NH}_3$  is mainly adsorbed on the PPY layer rather than on graphene (graphene has little affinity for  $\text{NH}_3$ ), donating electrons to the PPY layer. In turn, the PPY layer takes holes from graphene for electron recombination, depleting holes in graphene. As a result, the resistance of the PPY/G sensor is increased. During desorption,  $\text{NH}_3$  has to withdraw its electrons from the PPY layer and holes return back to graphene. So graphene serves as a reservoir allowing then a rapid electron transfer through the PPY layer. These observations may explain why the PPY/G sensor shows faster recovery time. This is one main advantage of our sensor over the thick PPY sensor. As discussed in Section I, most of the rGO-based  $\text{NH}_3$  sensors have poor reversibility, because the interaction between  $\text{NH}_3$  molecules and defects in rGO is quite strong so that thermal energy is insufficient to overcome the activation energy for the  $\text{NH}_3$  desorption [17].

Consequently, the PPY thickness has a strong influence on the PPY/G sensor response and recovery. More specifically, the thick PPY layer gives rise to high response and slow recovery. A trade-off result is that the PPY layer should be a single

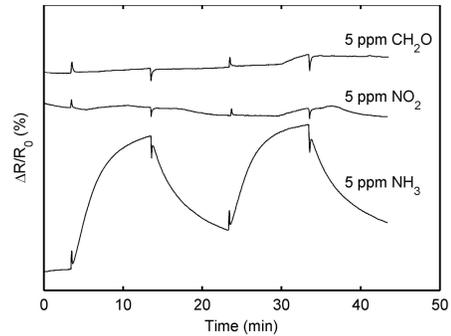


Fig. 6. Resistance responses of a typical PPY/G sensor exposed to 5 ppm  $\text{CH}_2\text{O}$ ,  $\text{NO}_2$  and  $\text{NH}_3$ , at a relative humidity of 50% and at 20°C.

layer with homogeneous and porous nature, which can expose all of its chemical bonds to the environment or the target molecules. Graphene under the thin PPY layer provides the efficient diffusion pathways for accelerating electron transfer, improving the PPY/G sensor response and recovery.

#### E. Sensor Selectivity

Schedin *et al.* indicated that graphene can interact with various molecules, including nitrogen dioxide ( $\text{NO}_2$ ) and formaldehyde ( $\text{CH}_2\text{O}$ ). The adsorption of hole donors ( $\text{NO}_2$ ) should enhance hole density in the existing p-type PPY/G compound and generate a decrease in resistance, whereas the adsorption of electron donors ( $\text{CH}_2\text{O}$ ) should cause hole depletion and hence increase in resistance [51]. We therefore investigate the PPY/G sensor selectivity towards these two gases. Figure 6 presents the PPY/G sensor response exposed to 5 ppm  $\text{CH}_2\text{O}$ ,  $\text{NO}_2$  and  $\text{NH}_3$ . For the cases of  $\text{CH}_2\text{O}$  and  $\text{NO}_2$ , no significant sensor responses are observed except for some abnormal peaks due to pressure peaks during introduction of the gases. Notice that 5 ppm of  $\text{NO}_2$  or  $\text{CH}_2\text{O}$  is much higher than the concentration in normal air conditions (less than 1 ppm). This confirms that the PPY/G sensor has an excellent selectivity to  $\text{NH}_3$  towards  $\text{CH}_2\text{O}$  and  $\text{NO}_2$ . This is associated with the fact that both  $\text{NO}_2$  and  $\text{CH}_2\text{O}$  molecules have no interaction with the PPY layer.

The presence of the ultrathin PPY layer on top of graphene hinders the interaction between interfering gases and graphene. Hernandez *et al.* [42] proved that PPY is insensitive to  $\text{NO}_2$ , since the electrostatic interaction is weak between them. Therefore,  $\text{NO}_2$  molecules can be easily desorbed from PPY. This suggests that the ultrathin PPY serves as a filtering layer, greatly improving the selectivity of the present sensor. Very small change in resistance (in Figure 6) is due to the fact that the PPY filtering layer does not perfectly cover 100% of the graphene surface area, thus small regions of graphene are exposed to the interference gases.

#### F. Humidity Effect

$\text{H}_2\text{O}$  is the most significant interfering vapor for most sensors, especially at room temperature. We tested the influence of the RH on the PPY/G sensor response. Figure 7 is the test result for the resistance variation as a function of time, exposed to 2 ppm  $\text{NH}_3$  in the range from 20 to 80% RH (the real-life RH). Table II summarizes the sensor response

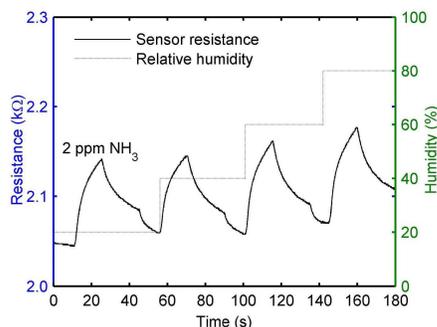


Fig. 7. Resistance behavior of a typical PPy/G sensor exposed to 2 ppm  $\text{NH}_3$  in the range from 20 to 80% RH at 20°C.

TABLE II  
SUMMARY OF THE SENSOR RESPONSE EXPOSED TO 0 AND 2 ppm  $\text{NH}_3$  AT VARIOUS RELATIVE HUMIDITY VALUES

$(R_{RH}-R_D)/R_D$ in %	RH = 20%	RH = 40%	RH = 60%	RH = 80%
$\text{NH}_3 = 0$ ppm	0.2	0.6	1.1	2.2
$\text{NH}_3 = 2$ ppm	4.2	4.5	4.9	5.2

for 0 and 2 ppm  $\text{NH}_3$  at various relative humidity values. In the table,  $R_D$  and  $R_{RH}$  are the sensor resistance under complete dry atmosphere (0% RH) and various relative humidity values, respectively. It can be seen that the response variation is about 1% in the real-life RH. Specifically, a change of 10% in RH brings a response variation of 0.17% in average. This value is 10 times smaller than the sensor sensitivity of 1.7%/ppm in wet air. We can conclude that the present sensor has a good immunity to humidity. In general, inert closed-cell  $\text{H}_2\text{O}$  can adsorb on the CVD-grown graphene through non-covalent bonds. They should influence the graphene resistance by redistributing electrons within the graphene sheet [52]. After exposure to  $\text{NH}_3$ , the adsorbed  $\text{H}_2\text{O}$  molecules interact with  $\text{NH}_3$ , making the sensing process more complicated and the sensing results unpredictable. The addition of the PPy filtering layer avoids the interaction between  $\text{H}_2\text{O}$  and graphene due to the PPy hydrophobic nature [53]. This improves the humidity immunity of the PPy/G sensor. Lin *et al.* [54] showed that the PPy resistance is not sensitive to humidity in the range from 12 to 90% RH. Sizun *et al.* [36] also reported that the PPy conductance is not sensitive to humidity in the range from 0 to 80% RH. Recently, Yin *et al.* [55] indicate that the detection capability of the PPy/rGO sensor is not severely affected by the relative humidity in the range from 0 to 80% RH. In our case, the resistance of the PPy/G hybrid material is still not sensitive to humidity in the range from 20 to 80% RH. Almost all the surface of the PPy/G sensor is uniformly covered by the PPy filtering layer. This explains why the PPy/G sensor shows only a small fluctuation to humidity variations.

#### G. Sensor Stability and Process Reproducibility

The present sensor is stable in ambient air since its resistance response almost keeps 12% for 5 ppm  $\text{NH}_3$  after one year, as shown in Fig. 8a. It can also be seen that after one year the sensor recovery seems to become slower. This may be due to the surface pollution since the sensor was left in

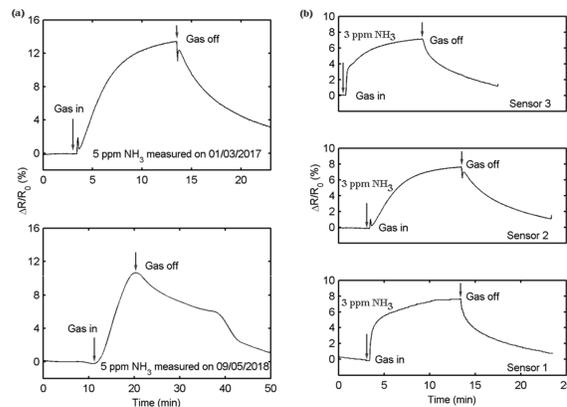


Fig. 8. (a) Resistance response of a typical PPy/G sensor exposed to 5 ppm  $\text{NH}_3$  at 20°C and 50% RH, top measured on 01/03/2017 and bottom on 09/05/2018; (b) Resistance response of three typical PPy/G sensors exposed to 3 ppm  $\text{NH}_3$  at 20°C and 50% RH, three sensors were fabricated in different batches under the same conditions.

ambient air without any protection. Although the sensor was not submitted to the same cycle, its sensitivity is similar.

To transfer the present sensor from laboratory concept to industrial application, it is necessary to investigate the process reproducibility. Therefore, we performed 3 experiments under the same conditions. Figure 8b demonstrates the resistance response of 3 typical PPy/G sensors from different batches, exposed to 3 ppm  $\text{NH}_3$  at 20°C and 50% RH. These results indicate that the present process is quite reproducible.

#### IV. CONCLUSION

In this work, we developed a new hybrid sensor, in which a 20-nm-thick polypyrrole (PPy) layer is synthesized on top of the CVD-grown graphene (G) by electropolymerization. The sensor shows a linear response for ammonia ( $\text{NH}_3$ ) in the range from 1 to 4 ppm at room temperature, with a sensitivity of 1.7%/ppm, response and recovery times of 2 and 5 min, respectively. The sensor exhibits structural stability and durability for at least one year. The sensor has a strong immunity to humidity and a good selectivity for  $\text{NH}_3$  towards interface gases ( $\text{CH}_2\text{O}$  and  $\text{NO}_2$ ). The ultrathin PPy layer with a porous nature plays an important role in the sensor sensitivity, selectivity and immunity to humidity. Graphene not only serves as a support material for PPy electropolymerization, but also accelerates the electron transport, shortening the response/recovery times. Apart from that, the ultrathin PPy layer on graphene allows the  $\text{NH}_3$  molecules to pass through the cavities to react with  $\text{sp}^2$ -bonded carbon atoms of graphene. This synergistic effect may multiply the reaction between  $\text{NH}_3$  and the PPy/G compound, thereby increasing the sensor sensitivity. Moreover, low contact resistance of graphene with Au electrode results in a high signal/noise ratio and low energy consumption. Our results indicate that the simultaneous use of PPy and graphene (the PPy/G compound) is promising as a chemical sensor material. We implement a smart prototype with the present sensor in an electronic board able to display the  $\text{NH}_3$  concentrations *in situ* and transmit the data to a computer for real-time monitoring via a USB port. This research is a step for the commercial design and mass production of room temperature  $\text{NH}_3$  sensors.

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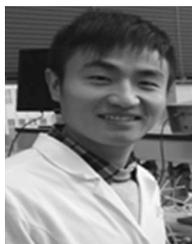
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