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Dielectrophoretic assembly of Pt nanoparticle-reduced graphene oxide nanohybrid for highly-sensitive multiple gas sensor

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ABSTRACT

Reduced graphene oxide (rGO) gas sensors functionalized with platinum (Pt) nanoparticles were fabricated. An alternating current dielectrophoresis technique was used for the precise alignment of the Pt-GO nanohybrid between microgap electrodes, proceeded by the mid-temperature thermal annealing. The gas sensing response was determined for the assembled rGO nanostructure-based devices with and without Pt decoration at various ambient temperature and gas concentrations. The tested device exhibited sensitivities of 14% (7%), 8% (5%), and 10% (8%), for 1000 ppm hydrogen, ammonia, and nitric oxide gases, respectively with (without) Pt nanoparticles, at room temperature. The Pt-decorated samples show an improvement of 100%, 60% and 25% to hydrogen, ammonia, and nitric oxide gases, respectively, over without Pt decorated sensors. Besides, improving the sensitivity, the dielectrophoresis assembled rGO-Pt nanohybrid sensors have been demonstrated as a viable material for multiple gas sensors.

1. Introduction

The gas sensing is critical to various aspects of industrial applications such as chemical process control, environment analysis, and medical diagnostics. Various gases like Hydrogen (H_2), ammonia (NH_3), and nitric oxide (NO, NO_2) are typical target gases in these applications. Hydrogen is being explored as an alternative to fossil fuel for its potential in fuel cells, methanol industries, and in power generation. Ammonia is an indispensable constituent in the production of fertilizers and in various chemical processes like to etch aluminum and to saponify fats and oils. Nitric oxide is widely use to promote vasodilation and muscle relaxant and in semiconductor industry. Besides their wide applications, these colorless gases can be lethal beyond their safety limits, hydrogen is highly flammable and easily explode over 4% concentration, similarly ammonia and nitric oxide poses severe health risk if exposed in excess quantity. Therefore, accurate and reliable gas sensing technique has become indispensable in these areas.

Owing to their small size, low cost, and ease of operation, semiconductor metal-oxide-based gas sensors are commonly used for monitoring the aforementioned gases. Tin oxide and zinc oxide are two typical metal-oxide materials, which are used for gas sensing applications. Metal oxide-based gas sensors must be operated at high temperatures (200–400 °C) and this leads to high power consumption; this consumption is an impediment to downsizing of the hand-held type monitoring system or sensing module of ubiquitous sensor networks (USN) [1–4].

Carbon-based nanomaterials, such as carbon nanotubes (CNTs) and graphene, have emerged as new gas sensing materials that can be operated at room temperature. Various gases have been tested using CNT-based gas sensors. However, these sensors exhibited low sensitivity [5], long response and recovery time [6], or poor reproducibility [7] depending on the purity and assembly processes. In the last few years, graphene has attracted significant attention for gas sensing applications owing to its exceptionally low electrical noise level [8]. Although their sensing properties were relatively independent of the assembly process, the corresponding sensitivity and response time rendered the graphene-based sensors inadequate for practical use. As such, nano-sized metal decoration has been suggested as a means of enhancing the sensing characteristics of both CNT- and graphene-based gas sensors [6,9–12].

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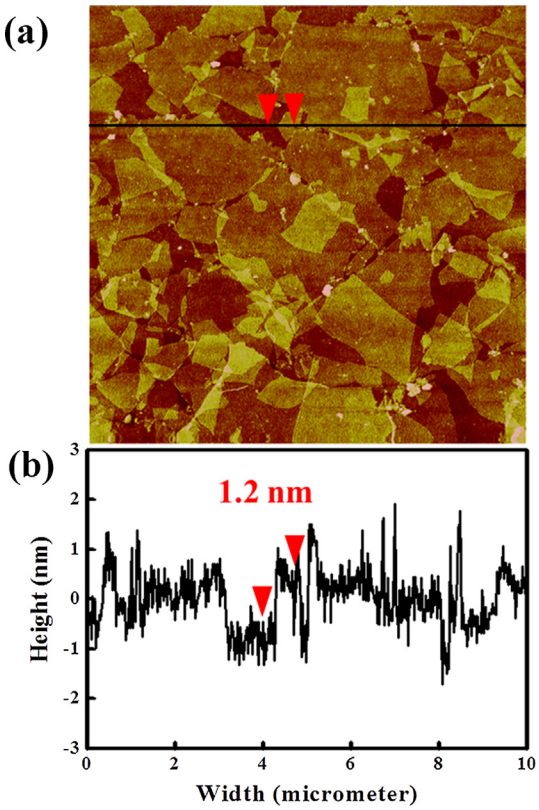

Fig. 1. (a) AFM image of the GO nanostructures and (b) height profile of the GO nanostructures along the line shown in (a); the red arrows correspond to those shown in (a). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

A few-layered reduced graphene oxide (rGO) has recently been proposed as a promising gas sensing element owing to its controllable surface defect density. However, pristine rGO-based gas sensors exhibited relatively low sensitivity [13] or long recovery time [14,15], or required a complicated fabrication process [16].

Improved gas sensing behavior was recently observed in rGO-based gas sensors decorated with rare metals. Although the sensitivity of the sensors was significantly enhanced through nano-sized metal or metal-oxide decoration, the shortened recovery time was, by commercial standards, still inadequate [17–19]. The alternating current (AC) dielectrophoresis (DEP) technique, which precisely controls the location, uniformity, and thickness of the nanostructures [20–29], may reduce the recovery time of rare-metal-decorated r-GO gas sensors.

Therefore, in this work, we compare the sensing behaviors of bare (non-decorated) and Pt nanoparticle-decorated rGO gas sensors fabricated under the same conditions via the AC DEP technique. The performance of each device was evaluated through exposure to H_2, NO, and NH_3 gases.

2. Experimental

GO nanostructures were synthesized using a modified Hummer's method. Using this method, 4 g of graphite flakes were added to a 250 ml round-bottom flask containing 120 ml of H_2SO_4 and stirred for 1 h. A $KMnO_4$ aqueous solution was added to the mixture every 20 min while stirring. The mixture was then slowly heated to, and maintained at 40 °C for 5 h in order to oxidize the graphite. Subsequently, 150 ml of deionized (DI) water was added to the mixture followed by 17 ml of H_2O_2 solution while stirring for 30 min. This mixture was maintained at 40 °C for 24 h and then centrifuged. The resultant mixture was enclosed in a dialysis tube and washed repeatedly with ultrapure DI water in order to obtain a pH level of 5. Finally, the GO was dried in a freeze dryer at −60 °C for 48 h. Prior to the DEP process, the GO solution was ultra-sonicated for 24 h to reduce the size of GO flakes and then filtered. Approximately 1-nm-thick, few microns-wide GO nanostructures were obtained by using the method; Fig. 1 shows the atomic force microscopy (AFM) image of the GO nanostructures.

In this work, we fabricated gas sensors based on Pt nanoparticle-decorated rGO nanostructures via DEP and thermal annealing. Using the DEP technique, the Pt-rGO nanohybrid structures were assembled into the electrodes by controlling parameters such as the applied voltage, applied frequency, and processing time. One ml Pt nanoparticle solution, (size ≈ 15 nm, purchased from Sigma Aldrich) was mixed into 5 ml GO nanostructure solution (1 mg/ml) in this experiment. The mixed solution was ultra-sonicated for ~1 h and 0.1 μl of the resulting well-mixed solution was dropped onto

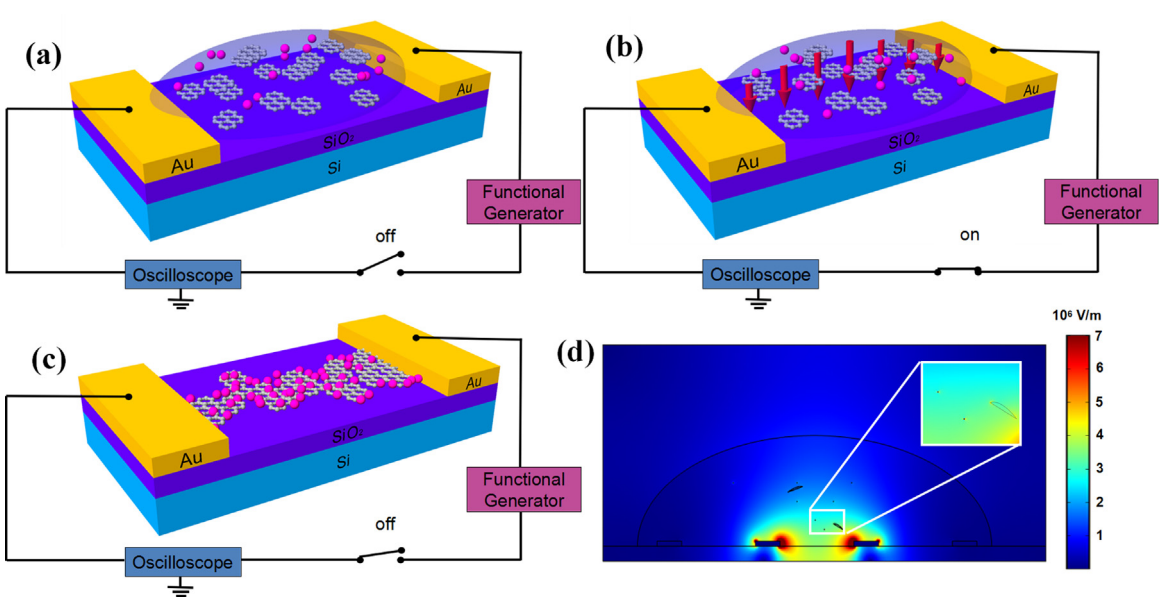

Fig. 2. Schematic of the GO nanostructure assembly process (a) before, (b) during, and (c) after DEP; the pink balls represent the Pt nanoparticles. (d) FEM simulation result of the electric field between the Au electrodes. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

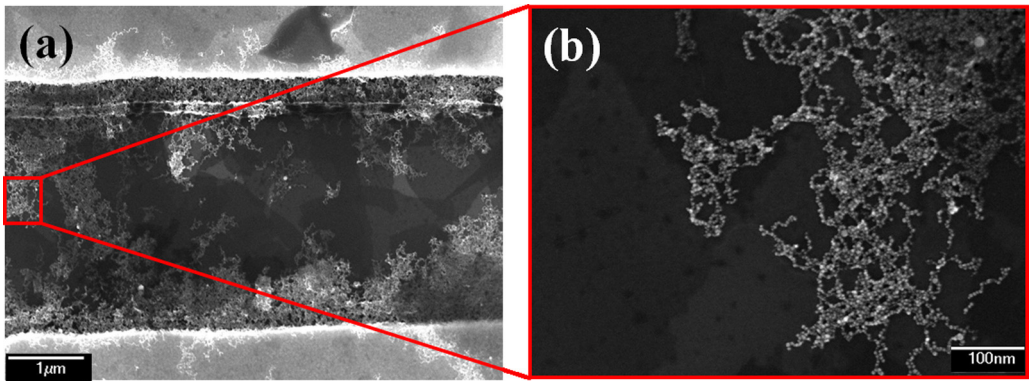

Fig. 3. (a) SEM image of the assembled nanostructures and (b) an enlarged view the region enclosed in the red rectangle in (a). The top and bottom region in (a) show electrodes while the shiny structures as zoomed in (b) are Pt nanoparticles. As seen in (a) that almost all region between the electrodes is covered with assembled rGO with only two small brighter windows showing the beneath SiO_2/Si substrate. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

4-μm-spaced microgap electrodes. An AC voltage was applied from a functional generator connected to an oscilloscope. We used 5 V, 500 kHz, and 30 s as the controlling parameters in our fabrication. The nanohybrid structures were pulled from the solution to the SiO_2 surface and bound between the electrodes by means of the electric field gradient. Fig. 2a–c shows the schematic of the sample setup before, during, and after the DEP process, respectively. The red arrows in Fig. 2b show the downward exertion of the DEP force resulting from changes in the electric field. To explain these changes, the finite element method (FEM) was used to simulate the electric field between the Au electrodes. Fig. 2d shows the result of this FEM simulation.

After the DEP process, the sample was thermally treated at 400 °C for 30 min. The Pt nanoparticles should be closely integrated with the rGO nanostructures after the thermal treatment [17]. The nanostructure assemblies were observed by scanning electron microscopy (SEM). Fig. 3 shows a typical SEM image of the assembled nanostructures. The GO nanostructures were also analyzed via X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). In addition, the sample was placed in a gas chamber in order to evaluate the gas sensing behavior. The sensing response was measured with more than 30 devices and almost all devices exhibits similar response which indicates good reproducibility of the fabrication process.

3. Results and discussion

XPS can be used to analyze the chemical bonding between functional groups and carbon atoms. Fig. 4a and b shows the results of XPS measurements performed on the non-treated and 400 °C-treated GO nanostructures. Peaks 1, 2, 3, and 4, which occur at binding energies of 284.6, 286.7, 288.3–288.4, and 289.9–290.3 eV are attributed to sp^2-hybridized carbon, and the hydroxyl, epoxide, and carboxyl functional groups, respectively. Similar to previous findings [30–33], the high intensity of peak 2 (Fig. 4a) indicates that the non-treated GO nanostructure consists of large amounts of hydroxyl functional groups. However, Fig. 4b shows that the hydroxyl functional groups occur with low intensity in the thermally treated nanostructure. This difference indicates that the hydroxyl functional groups underwent significant reduction during the thermal treatment.

Fig. 4c shows the XRD data of the non-treated GO nanostructures. The insert figure shows the XRD data of the 400 °C-treated GO nanostructures. Comparing the patterns reveals that the (0 0 1) peak of the GO nanostructures shifted from 11.2° to 23.9° during the thermal treatment; the corresponding interlayer distance changed from ~0.7 nm to ~0.37 nm. The interlayer distance of the

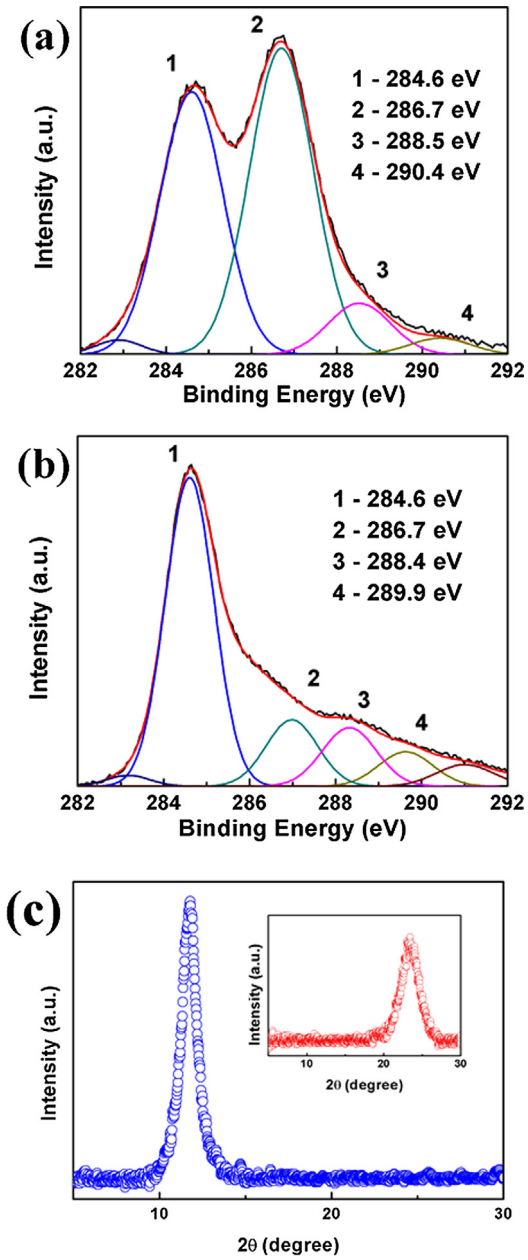

Fig. 4. XPS measurements of the (a) non-treated and (b) 400 °C-treated GO nanostructures. (c) XRD data of the GO nanostructures. The inset shows the XRD data of 400 °C-treated GO nanostructures.

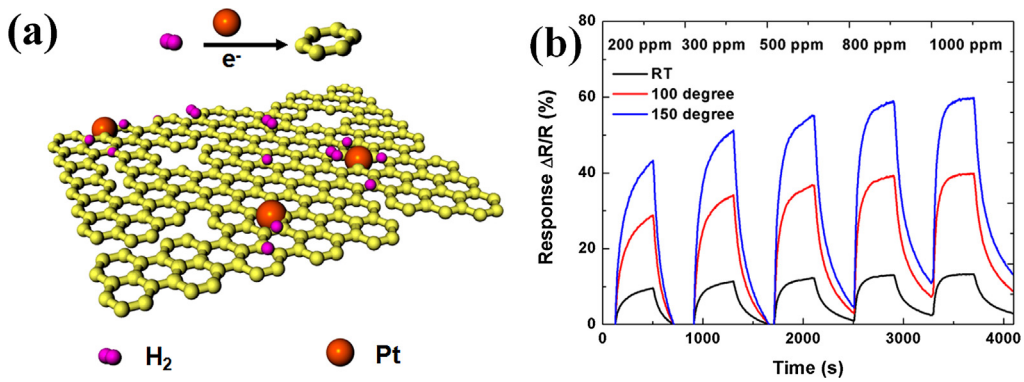

Fig. 5. (a) Illustration of the sensing mechanism of hydrogen gas adsorbed onto Pt-decorated r-GO nanohybrid. (b) Sensing response of DEP-assembled Pt/r-GO nanohybrid to hydrogen gas at different temperatures.

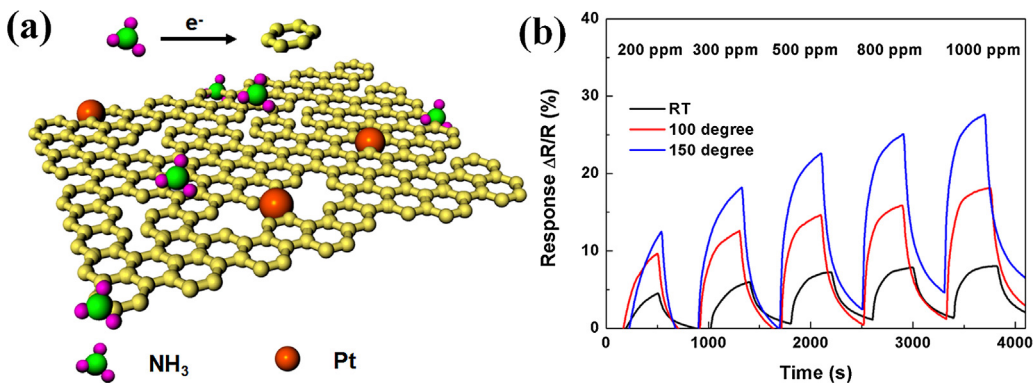

Fig. 6. (a) Illustration of the sensing mechanism of ammonia gas adsorbed onto Pt-decorated r-GO nanohybrid. (b) Sensing response of DEP-assembled Pt/r-GO nanohybrid to ammonia gas at different temperatures.

400 °C-treated GO nanostructures is very similar to that of pristine graphite, owing to a reduction of the hydroxyl functional groups during the treatment; this reduction was previously inferred from the XPS results.

The sensing response characteristics of the device to various gases were evaluated in a gas chamber. The gas sensing test was started after 100 s. This waiting time was considered so as to avoid current variations due to Joule heating induced improvement in the channel and contact resistances. Gas was introduced into, and removed from, the chamber periodically; one period is 800 s, and the gas was moved in and out of the chamber in 400 s. The gas concentration was varied with each period and gas concentrations of 200, 300, 500, 800, and 1000 ppm, were used from the first to the fifth period, respectively. The measurements were performed at room temperature, 100 °C, and 150 °C.

Fig. 5a illustrates the mechanism of the device sensing to hydrogen gas. Previous studies showed that rGO is a p-type material [14]. In the case of hydrogen gas, the Pt nanoparticles acts as a catalytic for hydrogen gas by surface adsorption and spill-over of extra hydrogen over the rGO nanostructures [34]. Electrons introduced into the material from the interaction of hydrogen gas resulted in a decrease in the density of holes, which in turn led to an increase in the resistance of the material. This gas doping is higher with Pt nanoparticles due to spill over of hydrogen from Pt to rGo layers. Fig. 5b shows the sensing response signal of the device to hydrogen gas at different temperatures. As the figure shows, the

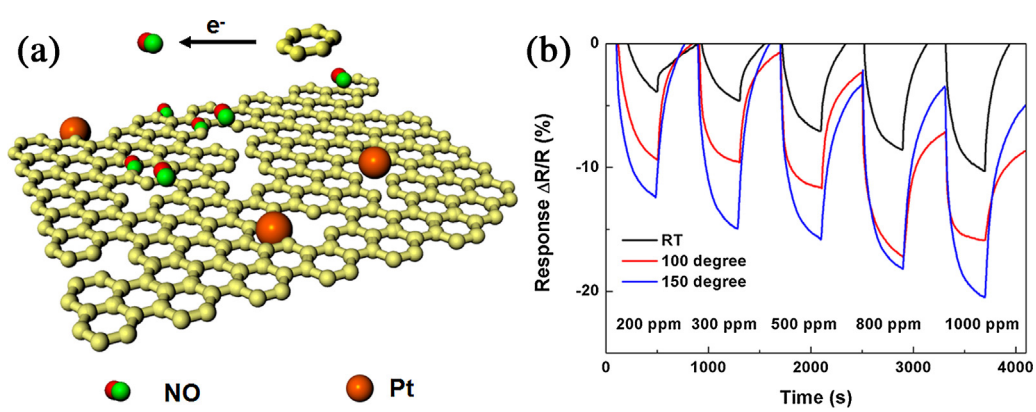

Fig. 7. (a) Illustration of the sensing mechanism of nitric oxide gas adsorbed onto Pt-decorated rGO nanohybrid. (b) Sensing response of DEP-assembled Pt/r-GO nanohybrid to nitric oxide gas at different temperatures.

sample had sensitivity of ∼14% and >60% at room temperature and 150 °C, respectively. These results show that the sample has excellent response to hydrogen gas as compared to similar sensors without Pt [28,29].

Fig. 6 shows the sensing response signal of the sample to ammonia gas. Ammonia by the virtue of a lone pair of electrons is a known electron donor, which when interacts with p-type rGO results in a decrease of the hole density, which increases the device resistance. As there was no evidence that platinum nanoparticles act as a catalyst in the reaction processing between the ammonia gas and the rGO nanostructures, so Pt nanoparticles has almost no effect on the sensing properties. The sample exhibited ∼25% and ∼8% sensitivity to 1000 ppm ammonia gas at 150 °C and room temperature, respectively.

Nitric oxide gas exhibits an opposite sensing response from that of ammonia gas, as shown in Fig. 7; i.e., since the oxygen atom accepts electrons, the resistance of the material was reduced owing to the increasing hole density of GO. As reported by other groups [14] and as the figure shows, the sample exhibits a negative sensing response to nitric oxide gas. The sample exhibited sensitivities of ∼20% and ∼10%, to 1000 ppm nitric oxide gas, at 150 °C and room temperature, respectively. It may be noted that the concentration of gases in the sensing measurement are on the higher side as the measurement with the lower concentration in the range of 1–100 ppm were limited by the hardware limitations of the sensing equipment.

The gas sensing response to various gases were tested in order to determine the effect of the Pt decoration on the rGO-based gas sensor. Fig. 8a–c shows the sensing response to hydrogen, ammonia, and nitric oxide gas, respectively of the devices with and without Pt decoration. As the figure shows, the Pt-decorated device exhibits varying levels of response to each gas. As Pt nanoparticles have no catalytic response to NH_3 and NO gases, therefore the variation in the sensing response could be due to variation in the local doping effect of Pt nanoparticles on rGO when the device is exposed to these gases. The interaction between Pt nanoparticle and reduced graphene oxide (rGO) can be visualized based on the work function difference, Pt (≈5.6 eV) and rGO (≈4.6–4.8 eV), which results in the local hole-doping of the rGO at the interface of nanoparticles. It is because of this favorable work function difference that high work function metals like Pt and Pd are preferred for ohmic contacts to graphene and rGO based devices.

We calculated the sensitivity, one of the most important characteristics of gas sensors, in order to determine the effect of Pt decoration on the gas sensing behavior of the rGO-based device. The sensitivity (S) was calculated from $S = (R - R_0)/R_0 \times 100\%$, where R is the resistance of the sample exposed to the gas and R_0 is the baseline resistance. Fig. 9a–c shows the sensitivities of the devices with and without Pt decoration, to hydrogen, ammonia, and nitric oxide gas, respectively at room temperature. As the figure shows, the sensitivity to 200 ppm hydrogen improved significantly, from ∼3% to ∼10%, with Pt decoration. This improvement resulted from the catalytic action of Pt nanoparticles on rGO nanostructures [15]. However, Pt decoration resulted in modest improvements in the sensitivities to ammonia and nitric oxide gas compared to the sensitivity to hydrogen; in fact, there seemed to be no improvement in the sensitivity to 200 ppm nitric oxide gas after Pt decoration. As explained above, these limited improvements in the case of ammonia and nitric oxide gas could stem due to the variation in the local doping effect of Pt nanoparticles on rGO when the device is exposed to these gases. As the variation in the rGO carrier concentration can predominately change the potential profile in the vicinity of Pt nanoparticles, which can have a direct influence on the device resistance and sensing properties. As the figures show, the Pt-decorated samples had sensing responses of ∼14%, ∼8%, and ∼10%, to 1000 ppm hydrogen, nitric oxide, and ammonia, respectively. Furthermore, the sensitivity of the non-decorated samples to hydrogen, ammonia, and nitric oxide gas were ∼7%, ∼5%, and ∼8%, respectively. Therefore, Pt decoration resulted in modest improvements in the sensitivities to ammonia and nitric oxide gas, but significant improvement (from ∼3% to ∼10% for 200 ppm) in the sensitivity to hydrogen gas. Therefore, the sensing response of the Pt-decorated sample varies significantly with the exposure gas. In this case, the gases could be easily distinguished by the difference in sensitivities. The sensing response suggests that the DEP-assembled GO nanostructure-based gas sensor can be used for multiple gas sensing applications like electronic nose.

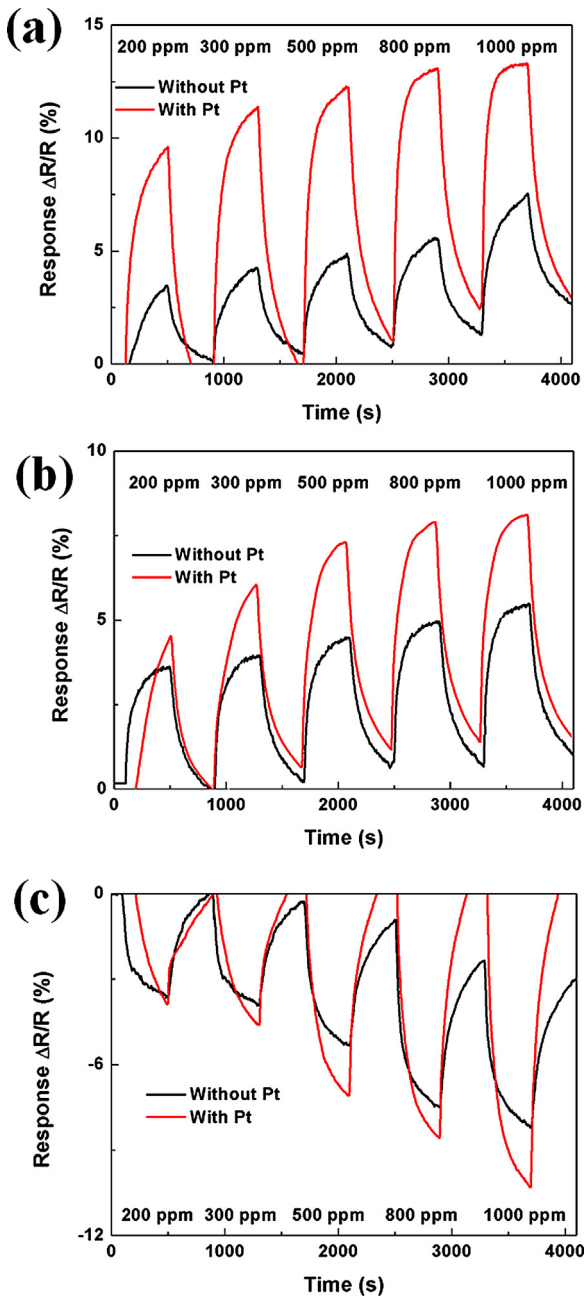

Fig. 8. Sensitivity of the DEP-assembled GO nanostructure-based gas sensors with and without Pt decoration to (a) hydrogen, (b) ammonia, and (c) nitric oxide gas at room temperature.

Response and recovery time are also important characteristics for determining the quality of the gas sensors. The former can be defined as the time required for the response to increase from its base value to 90% of its highest value. The latter can be defined as the time required for the response to decrease from 90% of its

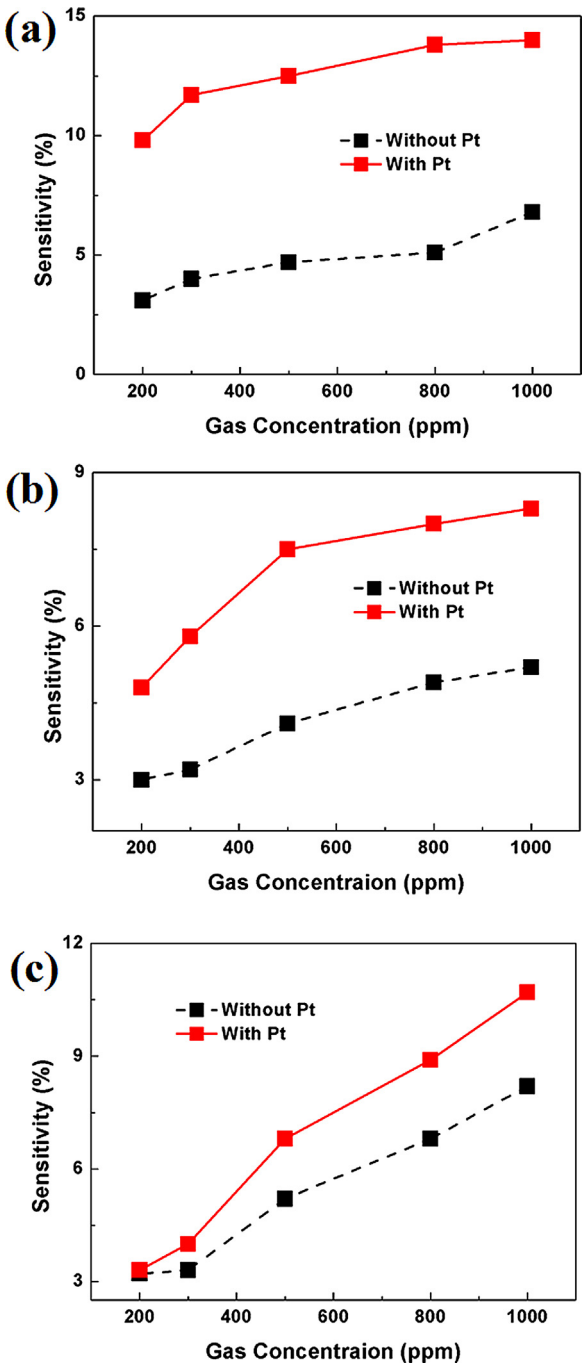

Fig. 9. Sensitivity of the device with and without Pt decoration to (a) hydrogen, (b) ammonia, and (c) nitric oxide gas at room temperature.

highest value to its initial value. However, as seen from Fig. 8, the sensing response sometimes results in a partial recovery of the device to its original condition, this in turn results in the non-flat baseline. Such results can provide non-accurate estimation of the response and recovery time of the sensor. Although, such partial recovery has been reported in other studies based on rGO but further improvement in the device fabrication and sensing conditions can reduce the non-flat baseline. Overall, an approximate estimate of response/recovery time to hydrogen, ammonia, and nitric oxide gas was found to vary from 2 to 6 min. It was also found that the recovery/response time for hydrogen gas decreases with Pt decoration while for ammonia and nitric oxide gas, it shows the opposite behavior. This decrease in hydrogen case could be from the catalytic action of Pt nanoparticles placed over rGO nanostructures. In contrast, the response and recovery time to the ammonia and nitric oxide gas increase after Pt decoration; i.e., although their sensitivities improved modestly, the reaction time did not increase, as in the case of hydrogen.

4. Conclusion

In this study, we used the DEP assembling technique to fabricate a multi-functional device based on a mixture of GO nanostructures and Pt nanoparticles. The DEP-assembled Pt/r-GO nanohybrid exhibited excellent sensing behavior to hydrogen, nitric oxide, and ammonia gas. The sensitivity of the device to these gases was ~14%, ~8, and ~10%, respectively, at room temperature, at which sufficient differences allow sensitivity-based distinction of the gases. This distinction will enable the device to perform effectively for multiple sensing devices like electronic nose.

Acknowledgments

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