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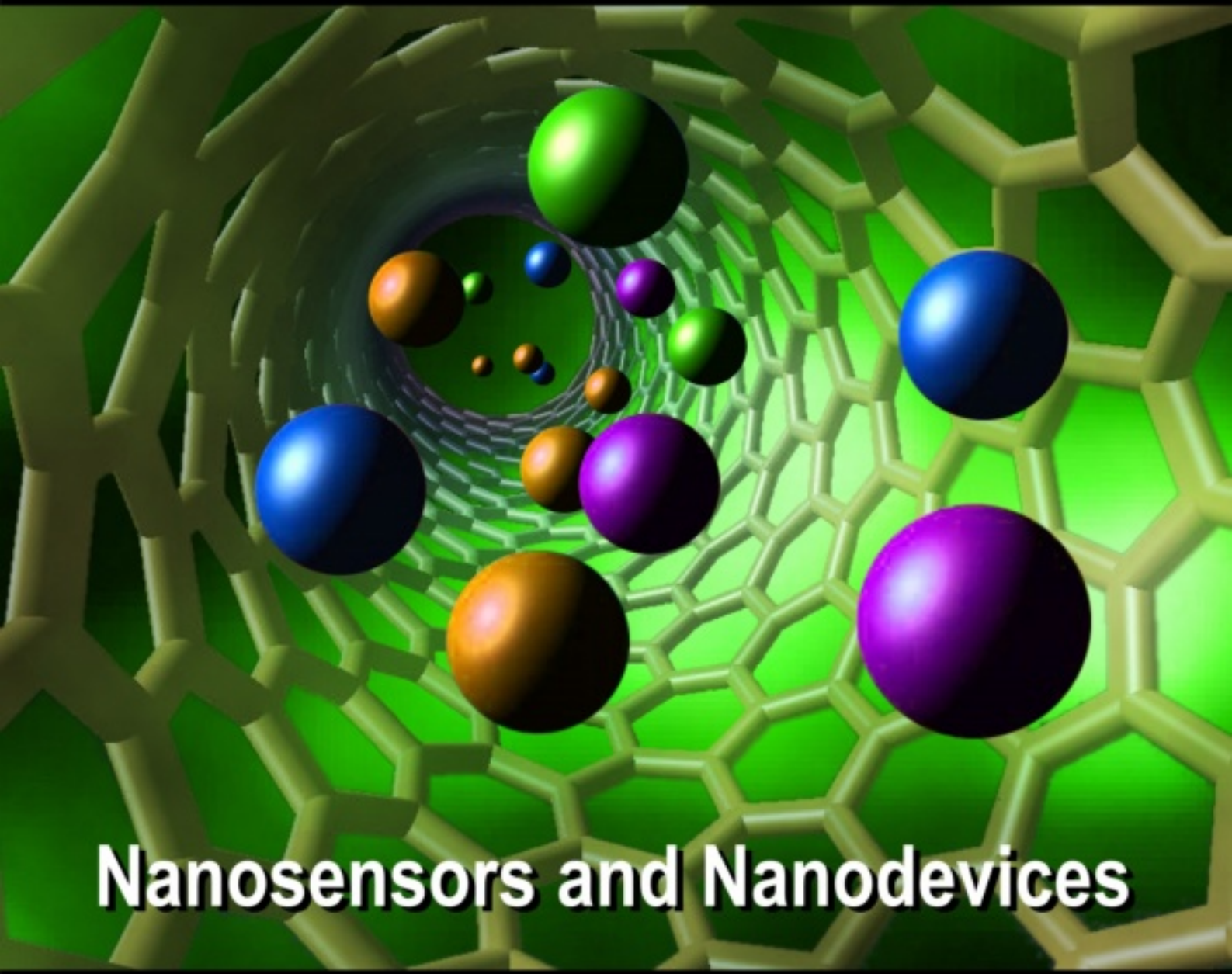

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Gas Detectors Based on Single Wall Carbon Nanotubes by Exploiting the Dielectrophoresis Method

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Abstract: Single wall carbon nanotube is developed for detecting various gases and vapors attributed to its peculiar mechanic structure and electronic property. We implemented a dielectrophoresis method to deposit desired amounts of carbon nanotubes on adjacent electrodes and investigate their conductivity variations at low gas pressures. The sensor exposed to extremely rare traces (in part per million) of CH_4, H_2, CO_2 and acetone is capable of yielding response and showing saturation at a higher concentration. We propose a mechanism to portray that limited reaction surfaces are subjected to be saturated at high gas concentration. It is expected that the resistivity of carbon nanotubes are highly affected by the exposing to polar solvent vapor such as acetone. Much sensing of carbon nanotubes based sensors is highly appraised to detect chemical gases and organic vapors. *Copyright © 2007 IFSA.*

Keywords: Gas sensors, Single-walled carbon nanotubes, Dielectrophoresis method

1. Introduction

Carbon nanotubes with unique structure have many fascinating properties, including excellent mechanical stiffness, quantum confinement effect at low temperatures and excellent gas adsorption. Recently, adsorption of chemical vapors in single wall carbon nanotubes (SWCNTs) has been extensively studied [1-5]. SWCNTs have being considered as chemical sensors on their superior in hollow geometry, large surface area and high aspect ratio, particularly of the sharp change of resistivity upon chemical vapor absorption [6-9]. Theoretical studies suggest the generation of donors

or acceptors of CNTs on absorption of gas molecule [10] in advantages of yielding the sensitivity of an order of one part per billion (ppb) in molecular absorption at room temperature. A higher sensitivity is expected at even lower temperatures.

In this work, we fabricated the chemical sensor by dielectrophoresis (DEP) process and measured the variation of the resistance under different kind of gases. To expel the amorphous carbons in the CNTsample, we utilized the DEP method to deposit pure SWCNTs from the mixed solution. The DEP method immunizes the involved electron beam lithography to directly assembling the CNTs on the desired location. This CNT based sensor is appealed in response to many kinds of gases operating at room temperatures.

2. Experimental Details

In this experiment, commercial SWCNTs (purchased from Shenzhen Nanotech Port Co.) limited with specifications of diameters < 2 nm; tube lengths of 5 ~ 15 μm; purity of SWCNTsof > 80 %; ash contents of ≦ 2 wt%; special surface area of 600 m2/g, and amorphous carbons of < 5 %. The CNT powder was firstly mixed with Triton X-100, a nonionic surfactant, to form aqueous solution, which then was well-dispersed ultrasonically by agitation for 30 minutes and centrifuged for two hours. The SWCNTs are suspended in insulating dielectric liquids which are subjected to move in response of an external nonuniform AC electric field. The SWCNTs were attracted to a region of stronger electric field when their permittivity ε_2 exceeds that of the suspended medium ε_1. According to the theoretical estimation, the DEP force on the SWCNT is $F_{dep} = \frac{\pi r^2 l}{2}\varepsilon_1 \mathrm{Re}[K]\nabla E^2$ where $\mathrm{Re}[K] = \frac{\sigma_1(\sigma_2 - \sigma_1) + \omega^2\varepsilon_1(\varepsilon_2 - \varepsilon_1)}{\sigma_1^2 + \omega^2\varepsilon_1^2}$ [11]. The DEP force on semiconducting carbon nanotube (S-SWCNT) is far smaller than that of metallic CNTs in the operational frequency range of 10 MHz, as shown in Fig.1.

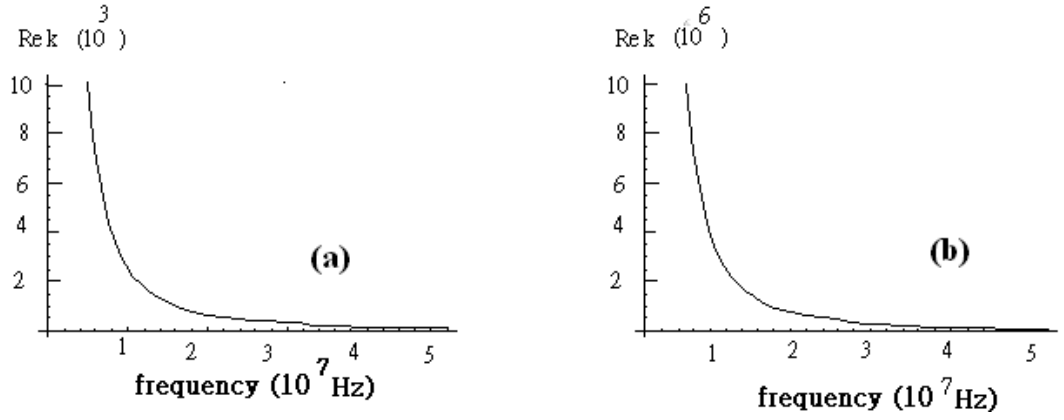


Fig. 1. The DEP method illustrated to be effectively forming a bridge across the microelectrodes expresses a force at various frequencies on (a) a single S-SWCNT. (b) a single M-SWCNT where the SWCNTs of metallic behavior are attracted by the highest electric field intensity.

A CNT based sensor was fabricated by conventional photolithography with the junction formed by the DEP. Accordingly, theavily doped silicon substrate was wet oxidized at 1950 °C to grow SiO_2 film of

500 nm in thickness. The Source and drain were patterned by the conventional photography with electrodes firstly deposited with 150 nm gold and then 10 nm titanium by E-gun deposition and followed by lift-off process in acetone, as shown in Fig. 2 (a). The pre-patterned structure was then mounted on a probe station with the surfactant solute dropped onto the electrodes. The CNTs bridge was absorbed by the DEP process with applying a radio frequency of 10 MHz for 1 minute. After then the sensor was immersed in isopropyl alcohol for 30 minutes to remove the surfactant from CNT surface. Figure 2 (d) shows that multi-CNTs were attached to the metal electrodes. The titanium layer deposited on CNTs is crucial to form ohmic contact instead of Schottky contact to effectively reduce the contact resistance. To improve the contact adhesion, the sensor further treated by rapid thermal annealing at 600 °C for 5min.

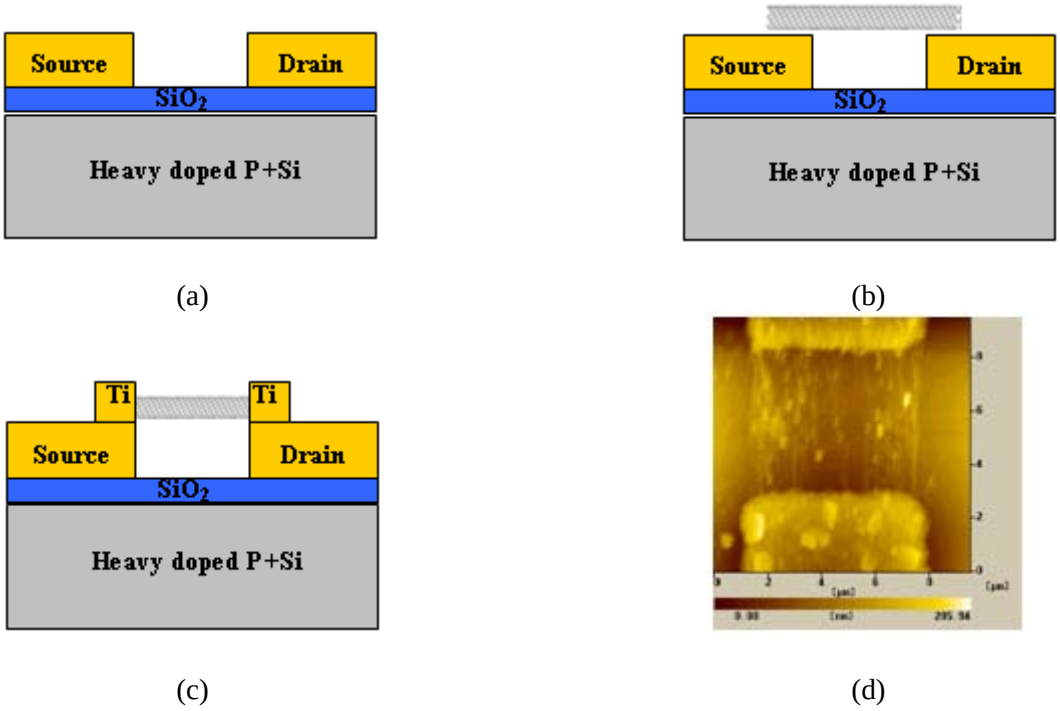


Fig. 2. Processes (a), (b) and (c) are the fabrication procedures for CNTs based sensor, and the AFM image in (d) shows multiple CNTs existed across the adjacent electrodes.

The experimental setup consists of 2.55L steel chamber evacuated by a dry pump which also performs the feeding of organic solvent used in gas sensing, as shown in Fig. 3. This system also provides the exposure of H_2, CO_2, CH_4 and acetone at extremely low level in parts per million (ppm) with implementing of a mass flow controller. The electronic characters were measured by Keithley 2410 Sourcemeter controlled by the LABVIEW software.

3. Results and Discussion

Fig. 4 shows that the resistance changes of SWCNTs at a saturated NH_3 gas. Under ambient condition (point 1), the resistance remains a steady value at vacuum condition. At point 2, NH_3 gas was introduced into the chamber to fill for 5 minutes. The system was pumped after 50 minutes illustrating the recovering of the resistance as indicated at point 3. The SWCNT sensor shows a prominent response upon the exposure to NH_3 gas, which recovers to the initial value after pumping down to vacuum.

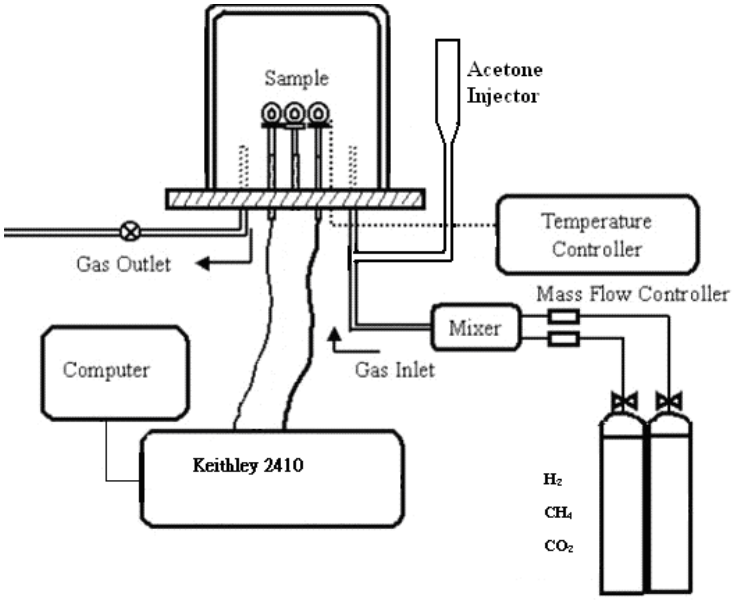


Fig. 3. The schematic diagram of the test chamber.

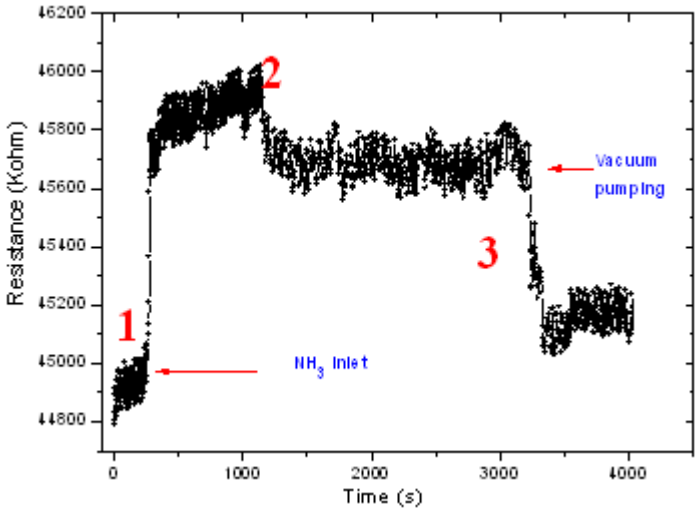
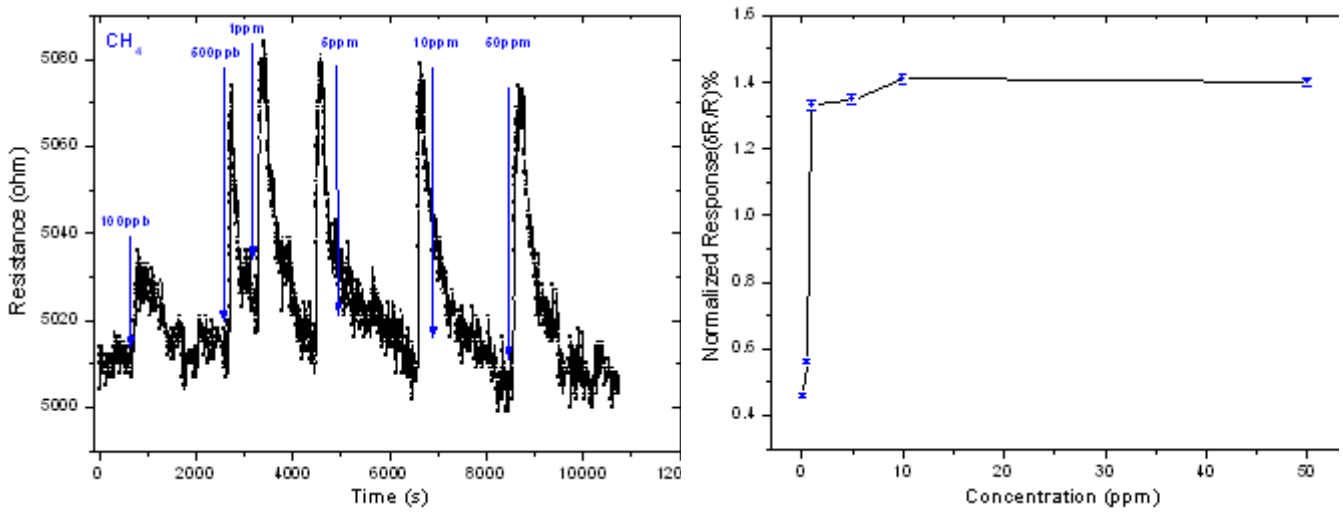


Fig. 4. Electrical resistance of SWCNTs sensor by cycling measurements in NH_3 and in vacuum.

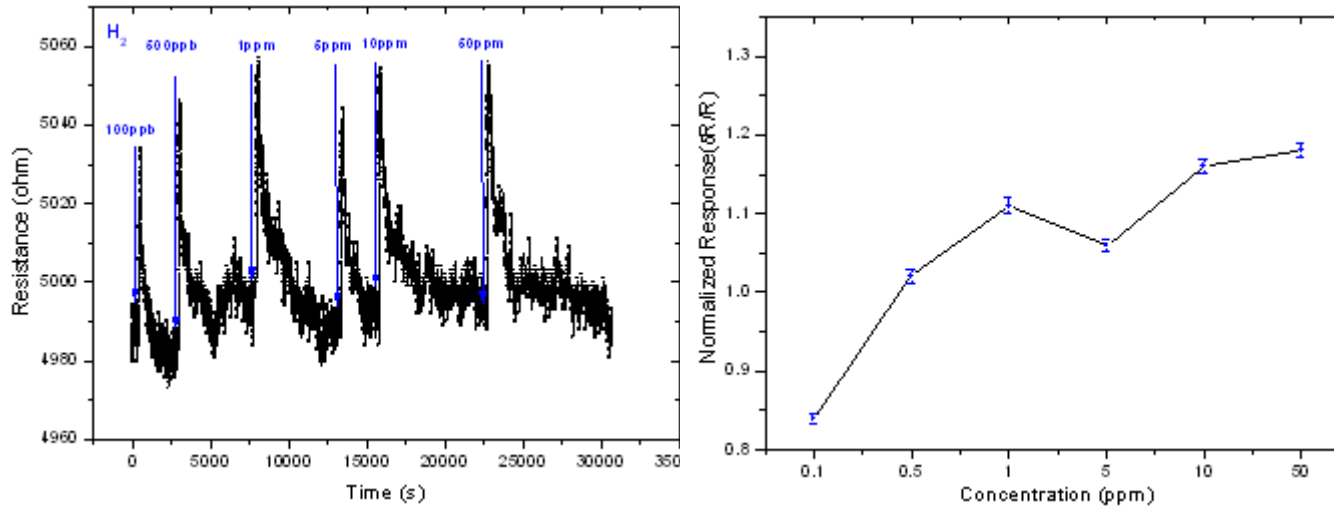
Fig. 5 (a) plots that The time dependence of the resistance for exposures to the concentration ranging from 100 ppb-50 ppm CH_4 that were inserted into chamber for 11000 s. The normalized response can be defined as $\frac{R_{after}-R_{initial}}{R_{initial}}\times 100\%$ where R_{after} is the steady state of the resistance after CH_4 exposure and R_{before} is the initial resistance under the vacuum. Fig. 5 (b) shows the response of distinct concentrations. The response of the sensor displays the abruptly change below 1ppm, on the contrary, the curve above the 1ppm exhibits a nonlinear response. Saturation phenomenon can be observed when the methane concentration exceeds 10 ppm.



(a) (b)

Fig. 5. Resistance response of the sensor when it is exposed to CH_4 at (a) various concentrations of CH_4 gas, and (b) the normalized resistance RAfter-RInitial/RInitial.

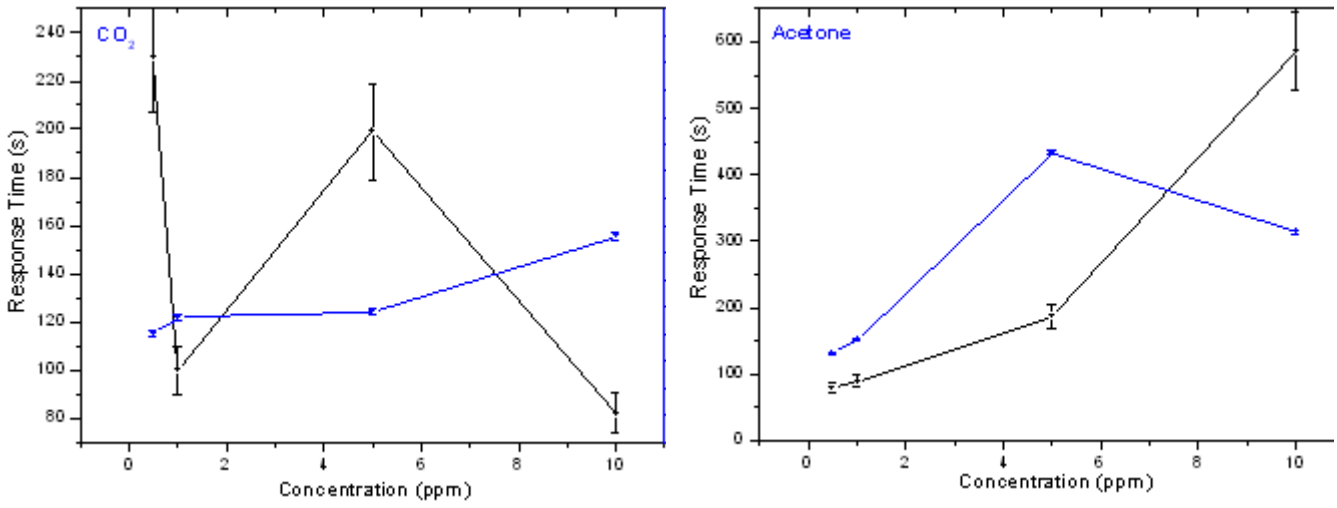
We also investigated the resistance response to hydrogen gas under various concentrations ranging from 100 ppb-50 ppm, as shown in Fig. 6 (a). At lowest H_2 concentration, the steady-state response of CNT sensor is founded to be 0.84 % of that at 100 ppb of H_2. Increasing the H_2 concentration, the steady-state response of CNT sensor is up to 1.2 % and saturated to that above 10 ppm, as shown in Fig. 6 (b). The saturation of the resistance response at higher concentration also reveals in CH_4 gas detection and the saturated concentration in methane gas is lower than that for hydrogen gas.



(a) (b)

Fig. 6. Resistance response of SWCNTs upon exposing to H_2 at (a) Various concentrations of H_2 gas, and (b) the comparison of normalized resistance (RAfter-RInitial/RInitial) and response time under hydrogen gas exposing.

For vapor sensing under saturated vapor condition, the acetone solvent was injected via syringed into the test chamber and allowed to evaporate to reach saturation condition for several minutes. Fig. 7 shows the resistance response sensing to CO_2 and acetone gas at 0.5, 1, 5 and 10 ppm. The resistance of the sensor slowly increases upon CO_2 exposure and nearly remained at the same value above 1 ppm, as shown in Fig. 7 (a). However, the resistance response of the sensor continuously increased even at a lower concentration and was saturated at 5 ppm acetone, as shown in Fig. 7 (b). The higher normalized response may have been attributed to the solvent polarity because acetone is a typical polar vapor [12, 5]. The response times with the concentration of detecting acetone gas displays the opposite result to that of other gases on account of delay times including solvent vaporizing and diffusing to the test chamber.



(a) (b)

Fig. 7. Resistance response of SWCNTs upon exposure to CO_2 and acetone vapor for (a) resistance and reaction times response to the trace CO_2 gas, and (b) resistance and reaction times response to the trace of acetone vapor.

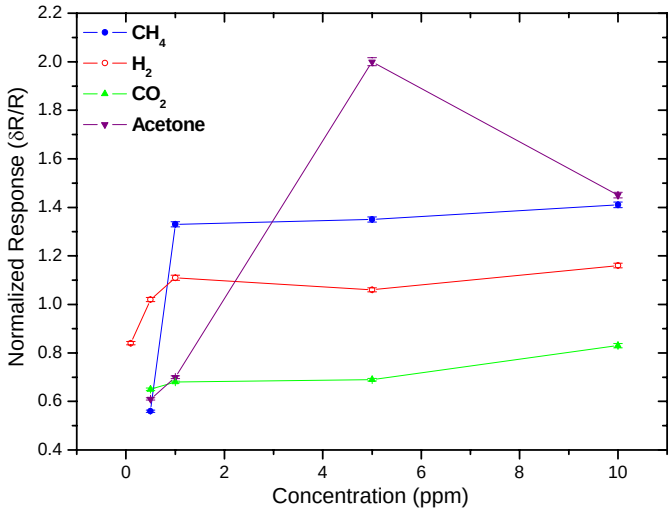


Fig. 8. Normalized response of various gases under different concentrations.

Fig. 8 shows the change of the resistance as a function of concentrations when the sensor is exposed to a various gases. The resistance changes with environments of CH_4, H_2 and CO_2 gases saturated at

1 ppm. These phenomena may be explained that the reactive surface of sensor is limited by the numbers of carbon nanotubes absorbed by DEP, which is much easier to be saturated than by that of SWCNTs film. In addition, detection of acetone vapor is much sensitive to that of non-polar gases at low concentration, such as H_2 and CO_2.

4. Conclusions

We have successfully fabricated a gas sensor based on single wall nanotubes by utilizing of the dielectrophoresis method, which assembling the CNTs on the preferred locations. Methane of ppm level can be detected at room temperature whilst the saturation phenomenon can be observed through a small amount of SWCNTs at a high gas concentration resulting from the decrease in surface reaction area of SWCNTs. Higher electrical response can be induced for the polar solvent such as acetone. The efficiency could plausibly increase by the lowering down of ambient temperatures and the increase of CNT surface.

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Aims and Scope

Sensors & Transducers Journal (ISSN 1726- 5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually.

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Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

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- Virtual instruments;
- Sensors interfaces, buses and networks;
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