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DNA-functionalized single-walled carbon nanotube-based sensor array for gas monitoring

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Abstract. Nine deoxyribonucleic acid (DNA) sequences were used to functionalize single-walled carbon nanotube (SWNT) sensors to detect the trace amount of methanol, acetone, and HCl in vapor. DNA 24 Ma (24 randomly arranged nitrogenous bases with one amine at each end of it) decorated SWNT sensor and DNA 24 A (only adenine (A) base with a length of 24) decorated SWNT sensor have demonstrated the largest sensing responses towards acetone and HCl, respectively. On the other hand, for the DNA GT decorated SWNT sensors with different sequence lengths, the optimum DNA sequence length for acetone and HCl sensing is 32 and 8, separately. The detection of methanol, acetone, and HCl have identified that DNA functionalized SWNT sensors exhibit great selectivity, sensitivity, and repeatability with an accuracy of more than 90%. Further, a sensor array composed of SWNT functionalized with various DNA sequences was utilized to identify acetone and HCl through pattern recognition. The sensor array is a combination of four different DNA functionalized SWNT sensors and two bare SWNT sensors (work as reference). This wireless sensing system has enabled real-time gas monitoring and air quality assurance for safety and security.

Keywords: DNA-SWNT sensor; wireless sensor array; gas monitoring; pattern recognition

1. Introduction

Carbon nanotubes (CNTs), first discovered in (Iijima 1991), have been widely used for decades as one-dimensional nanomaterials utilized in various types of electronics, optoelectronics and sensor systems. By now, abundant applications of CNTs have been reported in various fields including-chemical sensors (Kong *et al*. 2000, Kong *et al*. 2001, Collins *et al*. 2000), gas sensor (Britto *et al*. 1996, Davis *et al*. 1997), biosensor (Britto *et al*. 1999), field emission materials (Tans *et al*. 1998), electronic devices (Saito 1997) and actuators (Park *et al*. 2004). Particularly, the single-walled carbon nanotubes (SWNTs) are more widely utilized, whose molecular structure can be conceptualized by wrapping a one-atom-thick layer of graphite into a seamless cylinder. SWNTs are more favored due to their specific electrical, mechanical, optical, chemical, and thermal properties. Diverse applications of SWNTs have been explored, including

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chemical/biological sensors (Kim *et al*. 2007), electrical interconnects (Close *et al*. 2008), thermal heat sinks (Kordas *et al*. 2007, Iwai and Awano 2007), agents for drug delivery (Liu *et al*. 2007, Prato *et al*. 2008), low voltage, cold-cathode field-emission display (Choi *et al*. 1999) and nanoscale circuits for beyond silicon based complementary metal-oxide-semiconductor (CMOS) electronics (Wong 2000, Thompson *et al*. 2006, Avouris 2007). The electronic properties of SWNTs are one of their most important features, which permit extensive applications in nanoelectronics and sensors (Appenzeller *et al*. 2002, Roberts *et al*. 2009). The electronic properties of carbon nanotubes can undergo dramatic changes in the presence of trace amount of gases, mechanical deformations or variations in certain operating conditions such as temperature, and pressure. The electronic properties of CNTs, correlated with their optical, mechanical, chemical and thermal properties, have enabled carbon nanotubes to be a novel material for diverse sensing applications. Changes caused by external stimuli can easily be electrically evaluated in resistor, transistor, or capacitor (Roberts *et al*. 2009, Abraham *et al*. 2004, Izadi-Najafabadi 2010).

Thus, it is possible to exploit SWNTs as sensing sites for various interested molecules, from toxic chemical vapors to bio-macromolecules (Besteman *et al*. 2003, Pengfei *et al*. 2003, Mahar *et al*. 2007). However, a major disadvantage of SWNT sensors is the lack of specificity. To solve this problem, an effective scheme to functionalize the SWNT sensors is required which can enable the SWNTs to specifically respond to a wide spectrum of analytes. Modification of SWNTs with polymers (Snow *et al*. 2005, Novak *et al*. 2003, Bradley *et al*. 2003) and biomolecular complexes (Wong *et al*. 1998, Staii *et al*. 2005, Zhang *et al*. 2007) has shown great enhancement in the specificity and sensitivity of the SWNT-based sensors. Among these molecules, DNA can nonspecifically bind to the sidewalls of SWNTs through hydrophobic interactions, π-π bonding (Zheng *et al*. 2003), and possibly amino-affinity. DNA (deoxyribonucleic acid) is composed of two long polynucleotide chains which run in the opposite directions and are twisted around each other right-handedly. Each strand of the double helix is a linear chain with a backbone made of sugars and phosphate groups joined by ester bonds. Attached to each sugar is one of the four types of bases, including the purines: adenine (A) and guanine (G), and the pyrimidines: cytosine (C) and thymine (T). The aromatic structures in oligomers' bases bind to the aromatic structures on SWNTs via the π-π stacking interactions.

DNA decorated carbon nanotubes are novel nanoscaled materials that consist of SWNTs coated with a self-assembled monolayer of single-stranded DNA (ss-DNA). This unique system offers an intriguing combination of properties of an essential and ubiquitous biomolecule-DNA and one of the most heralded inorganic nanomaterials-SWNT. It has integrated the selective odorant interactions of ss-DNA (White *et al*. 2008) with the sensitivity of SWNTs to the changes in its surface electronic environment when exposed to analytes (Johnson *et al*. 2008). Moreover, the response of these devices to a particular chemical of interest can always be optimized by changing the base sequence of the ss-DNA. Functionalization of SWNTs with DNA has demonstrated attractive prospects in various fields including the detection of chemical vapors, solubilization in aqueous media, and the nucleic acid sensing (Staii *et al*. 2005, Daniel *et al*. 2007 Meng *et al*. 2007).

DNA decorated SWNTs have illustrated great potential in a variety of fields, ranging from homeland security to disease diagnosis. Aravind et al. has successfully fabricated ss-DNA immobilized, Platinum (Pt) nanoparticles decorated MWNT composites to selectively detect dopamine, which is one of the most important neurotransmitters that affect the function of brain (Aravind *et al*. 2011).Their hybrid biosensors decorated with sequence AC (bases adenine and cytosine) of ss-DNA exhibit linearity of detection up to 0.45 μM and a detection limit about 0.8

μM towards dopamine, while the GT (bases guanine and thymine) sequence of ss-DNA demonstrates linearity of detection to 800 μM and detection limit about 0.45 μM. Moreover, their group's nafion coated DNA-decorated MWNT biosensor has achieved much better results including good stability, short response time (<3s) and selective detection of dopamine even with presence of ascorbic acid and uric acid. Apart from the medical field, such novel hybrid nanostructures have found potential applications in the security maintenance of public places by the detection of explosives. Staii *et al*. has conducted some study on this topic (Staii *et al*. 2005).

The odor responses of the ss-DNA/SWNT-FET sensors measured were towards DNT and DMMP, which are simulants for explosive vapor and nerve gas, respectively, showing prospects in the security safeguard. Their sensors could detect various odors with very short response time and the recovery time on the scale of seconds. In addition, the sensor surface can refresh in an ambient environment, enabling the samples to maintain a constant response through at least 50 gas exposure cycles.

In the field of electronic devices, Chen *et al*. have successfully integrated ss-DNA-decorated SWNT-based chemical sensors onto the complementary metal oxide semiconductor (CMOS) circuitry, which showed great promise towards the development of ultra-small electronic nose (Chen *et al*. 2010). SWNTs were assembled onto the CMOS circuitry by a low AC voltage DEP process, and ss-DNA was non-covalently decorated on SWNTs in a humid environment. The decoration of ss-DNA on SWNTs was found to increase the resistance of SWNTs by approximately 57.02%. A dramatic enhancement of the sensing response of the gas sensor obtained by decorating ss-DNA on SWNT (up to ~300% for methanol vapor and ~250% for isopropanol alcohol vapor) as compared to bare SWNTs has been demonstrated. Liu et al. have developed a single chip nanosensor composed of SWNT integrated on CMOS circuitry with custom designed on-chip amplifiers for chemical agent detection (Liu *et al*. 2011). The SWNTs were integrated on CMOS circuitry using a low temperature and low voltage Dielectrophoretic (DEP) assembly process. Different sequences of DNA were incorporated onto SWNTs and demonstrated improvements of their response to DMMP by 9 times and DNT by 12 times. The responses are reversible and the change in resistance correlated well with the change in concentration of analytes. Not only does this single chip of SWNT sensors provide an attractive platform to realize high sensitivity, portable and compact nanosensing clusters, but it also shows great promise for toxic and explosive gas detection.

Air is composed of 78% N_2, 21% O_2, 0.03% CO_2, 0.94% inert gas and 0.03% other gases, water and impurity. Due to the inference of multiple gases, it is hard to differentiate the target analyte in mixed gases. In order to accomplish the goal of recognizing certain chemicals in the air, just one DNA decorated SWNT sensor is not enough, as it may respond the same way to another chemical. An array of such sensors with different response characteristics and an appropriate pattern recognition algorithm will be eligible to distinguish one chemical from the others in a mixture.

Here we introduce a wireless sensor array with six channels to measure the responses of the six SWNT sensors simultaneously when exposed to different gases, which can certainly reduce the undesirable noises of SWNTs to interference analytes. Ultrathin films of SWNTs were assembled onto the microelectrodes by a low temperature, and also low cost DEP assembly process. Then ss-DNAs of different sequences are noncovalently bonded to the SWNT surfaces, which have dramatically improved their response to the gas vapors. Different DNA decorated SWNT sensors respond differently when exposed to gas vapors. Thus, this real-time wireless sensor array

generates a specific pattern for one particular gas, which can be utilized to recognize certain chemicals in the future.

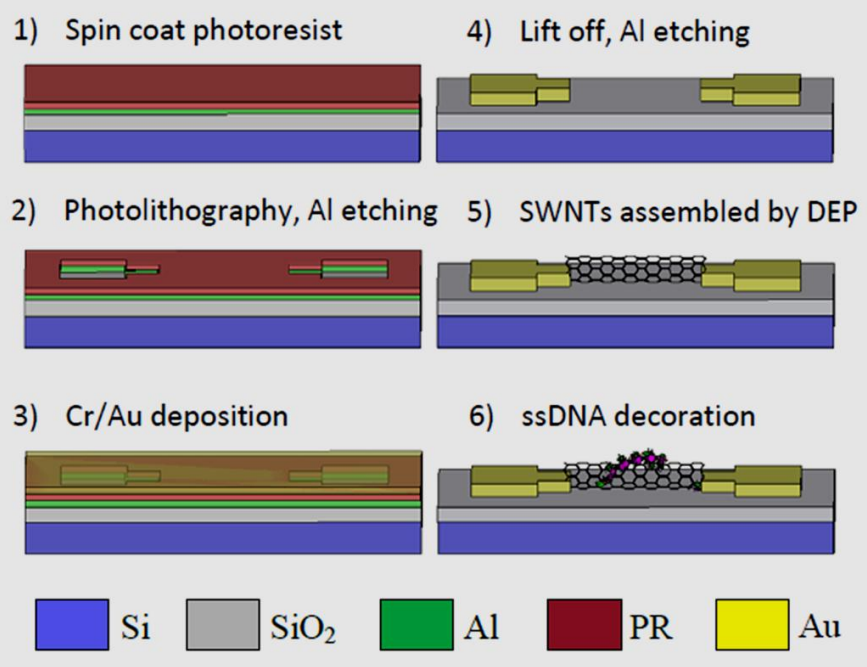

Fig. 1 Fabrication procedure for DNA decorated SWNTs on microelectrodes

2. Experimental procedures

The sensing system includes the DNA functionalized SWNT sensor array and a wireless sensing package. We first tested our sensing system with methanol vapor, and then detected acetone vapor and HCl gas, all of which are hazardous to human health.

2.1 Fabrication of ss-DNA decorated SWNT sensor array

First, microelectrodes with 3 μm gap were fabricated by photolithography followed by sputtering Cr/Au (20 nm/150 nm) layer onto a silicon oxide substrate, while aluminum was deposited on the top of SiO_2 layer as the sacrificial layer before photolithography (Figs. 1 (1)-(4)). Then by the solution-based DEP assembly which is a low temperature, low cost, but very efficient method for placing nanotubes, SWNTs were assembled between the microelectrodes, just as bridges (Fig. 1 (5)). Fig. 2 shows nanotubes assembled at one microelectrode. In this assembly, a large number of individual nanotubes bridged the microelectrodes. Majority of the nanotubes assembled were reasonably aligned along the gap between the two microelectrodes, while there were few mis-aligned nanotubes. The few misalignments may be caused by the sudden remove of the electrical field. Some nanotubes didn't have enough time to be well aligned between the two

electrodes before the electrical field was put off. The SWNTs used here (diameter: 1~2 nm; length: 2~5 μm) were purchased from Brewer Science Company. They were then dispersed in DI water to reach the concentration of 4 g/L. An AC (Alternating Current) signal of 1 V_{pp} and 10 MHz frequency was applied between the electrodes after the placement of 2 μL of the dispersed SWNTs solution onto the top of the electrode gap. After aligning SWNTs between the microelectrodes, DNAs with different sequences were utilized to functionalize SWNTs (Fig. 1 (6)). The chosen DNA sequences in this study are shown below

DNA 24G: GGGGGGGGGGGGGGGGGGGGGGGG
DNA 32G: GGGGGGGGGGGGGGGGGGGGGGGGGGGGGGGG
DNA 24A : AAAAAAAAAAAAAAAAAAAAAAAA
DNA 24Aa: amine-AAAAAAAAAAAAAAAAAAAAAAAA-amine
DNA 24GT: GTGTGTGTGTGTGTGTGTGTGTGT
DNA 24Ma: amine-GTCTTACGCTAGCTGGGCATTACG-amine
DNA 8GT : GTGTGTGT
DNA 16GT: GTGTGTGTGTGTGTGT
DNA 32GT: GTGTGTGTGTGTGTGTGTGTGTGTGTGTGTGT

As mentioned earlier, DNA can be self assembled on SWNTs by noncovalent bonding by the π-π stacking force. This can be easily achieved by dropping 5 μL of 100 μM ss-DNA solution onto the SWNTs and incubating in a chamber with 100% humidity for 1 hour. The extra DNA solution was removed by compressed air. This fabrication procedure is schematically exhibited by Fig. 1.

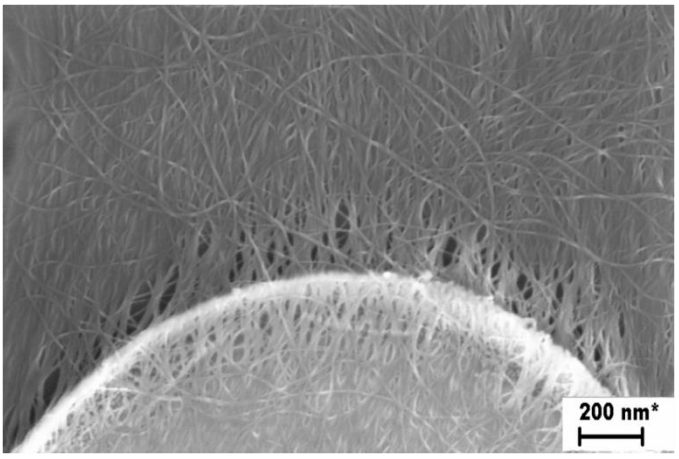

Fig. 2 SEM photograph of SWNTs assembled on the microelectrode

During the DEP process, the SWNTs were aligned between the two electrodes by the applied AC electrical field. Dielectrophoresis is a phenomenon in which a force is exerted on a dielectric particle when it is subjected to a non-uniform electric field. The strength of the force depends strongly on the medium and particles' electrical properties, on the particles' shape and size, as well as on the frequency of the electric field, which can be certified by the formula

$$F_{DEP} = \frac{\pi r^2 l}{3} \varepsilon_m \mathrm{Re}\left\{\frac{\varepsilon_p^* - \varepsilon_m^*}{\varepsilon_m^*}\right\} \nabla \left|\vec{E}\right|^2 \quad (1)$$

Besides voltage, the amount of assembled SWNT can also be determined by the applied frequency, current, shape of the electrodes, duration time, the particles' shape and size and the concentration of carbon nanotubes. The optimum amount of SWNT is determined by the best sensing performance. Too much SWNT been assembled on the electrode will decrease the contact area of naotubes to gas molecules, thus will lower down the resolution; while too little SWNT will increase the signal's instability. The amount of the assembled SWNT is represented by the resistance between the two electrodes, and the best sensing performance is provided by the SWNT with a resistance ranging from 5 to15 kΩ.

Although there are some other approaches to position nanotubes onto electrodes, such as self-assembly (Rueckes *et al*. 2000), random spreading (Martel 1998), direct growth (Franklin 2001), and nanomanipulation (Fukuda *et al*. 2003), each of these techniques has its limitations, e.g., high temperature of 900^{o}C (Franklin *et al*. 2001). The DEP assembly has overcome these limitations and is a versatile and flexible way to assemble carbon nanotubes onto CMOS circuitry. It is a simple and powerful method to manipulate and trap micro-and nanoparticles (Pohl 1978). As a CMOS compatible method, DEP assembly can quickly be utilized to incorporate carbon nanotubes onto platforms as components of microsystems or for in-line characterization or as nanostructure after manufacturing. From Fig. 3, we can clearly see that the SWNTs bundles were successfully aligned between the microelectrodes.

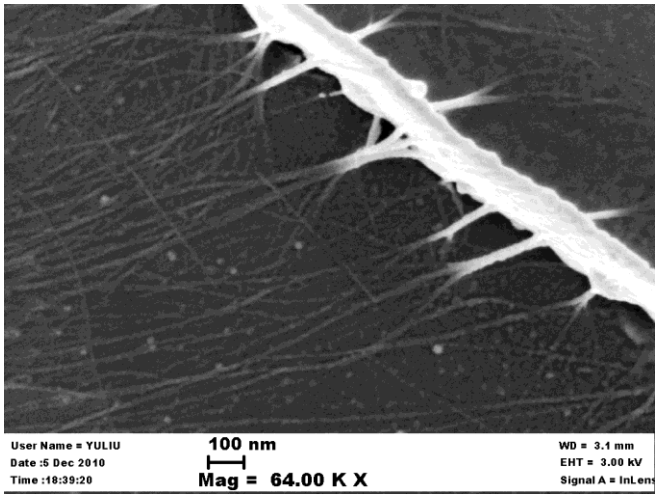

Fig. 3 SEM photograph of ssDNA-decorated SWNTs assembled on the microelectrode

The observed tiny white dots, which were believed to be aggregated ss-DNA molecules (according to one previous report (Jeng *et al*. 2007)), were randomly dispersed in the SWNTs bundles. These decorated DNA molecules have almost spread everywhere in the SWNTs bundles.

ss-DNA was coated onto the SWNT by wrapping around the surface wall of SWNTs, which relied on the π-π stacking of nitrogenous bases of DNA on the aromatic rings of SWNT. The reason why we could not see the increase in the thickness of the SWNT bundles is that the SEM imaging was not sensitive enough to resolve such difference. It has been reported by Staii *et al*. that ss-DNA formed a ~1nm layer on SWNTs (Staii *et al*. 2005). However, the observation of DNA aggregation, displayed as the tiny white dots, is consistent with the AFM results reported by Jeng *et al*., where DNA was adsorbed and folded on the surfaces of the nanotubes in a non-uniform manner (Jeng *et al*. 2007).

2.2 *Wireless sensing package*

The functional block diagram of our wireless nanosensor array is shown in Fig. 4. This design is based on the modular design concept and has been developed further. The major advantage of this module design method is that it provides a flexible and universal platform to approach a wide variety of applications. For different sensors, the signal conditioning module can always be reprogrammed and adjusted, allowing diverse sensors to be integrated in this wireless sensing platform.

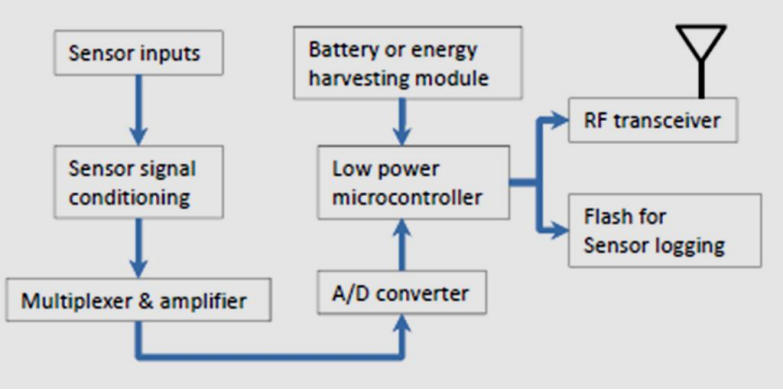

Fig. 4 The wireless sensor node functional block diagram for the wireless sensor node

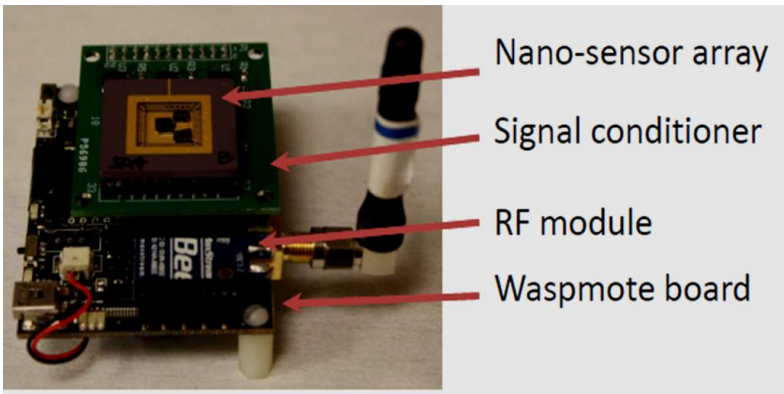

Fig. 5 Photograph of the wireless nanosensor array system

The actual system developed based on this modular concept is shown in Fig. 5. In the figure, the nanosensor array module is on the top layer, the signal conditioner is on the middle one, while the bottom layer contains low power microcontroller board called Waspmote board and a RF module.

The Waspmote board contains an in-built accelerometer and when associated with the GPS module, can be utilized to measure real-time speed, direction and location of the sensor. This board also possesses an outstanding power management which allows it to be used in some remote places or under adverse conditions, and an optional solar panel can permit almost any indefinite operation. In addition, the RF module can be chosen for a different mode with various protocols such as Bluetooth, ZigBee, IEEE802.15.4 and standard RF depending on the wireless range of certain given applications and the need for bidirectional communications which varies from 30 m to 10 km. Furthermore, the usage of flash memory tool enables the remote nodes to acquire data on command from a base station, or by an incident sensed by one or more inputs to the node. The major specifications are listed in Table 1.

Table 1 Specifications of the system

Microcontroller:	ATmega1281
Frequency:	8 MHz
Temperature	[-20℃, +65℃]
Clock:	RTC (32 KHz)
Power (ON):	(3.3 - 4.2)V X 9 mA
Input/Output	7 Analog (I), 8 Digital (I/O), 1 PWM, 2 UART, 1 I2C, 1USB

2.3 Sensing strategies and characteristics

The collected data with above nanosensor array is wirelessly transmitted to a Waspmote gateway which is attached to the PC via a USB port. A graphic user interface (GUI) program developed by LabWindows/CVI was designed to enable customer applications after collecting the data from Waspmote gateway. Fig. 6 shows the GUI interface for this purpose. The six windows show the responses of six different sensors concurrently when exposed to analytes vapor where the x-axis represents the obtained data sequence at sampling rate of 2 Hz and the y-axis is the resistance of the nanosensor. The reason of this sequence dependent sensing response could be due to the amount of SWNTs assembled and the different conformation of DNA decorated on SWNT. In our application, data acquired was real-time monitored, plotted simultaneously and stored for further analysis.

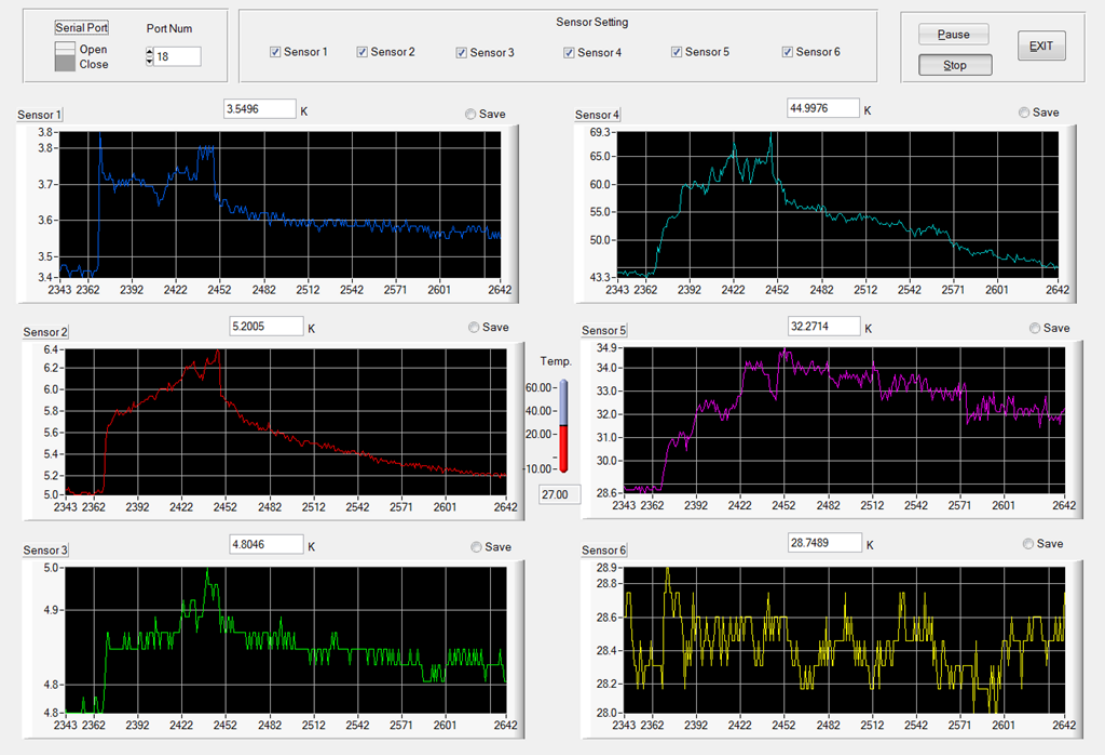

Fig. 6 GUI interface for the wireless nanosensor array

3. Chemical sensing

3.1 Sensor characteristics and methanol detection

Methanol is a clear, colorless liquid that looks like water and has no discernible odor in low concentration. It is used primarily for the manufacture of other chemicals and as a solvent, but acute exposure to methanol is very harmful to human. In one study, symptoms of blurred vision, headaches, dizziness, nausea and skin problems were reported in teacher aides exposed to duplicating fluid containing 99% methanol (Frederick *et al*. 1984). The concentration in the breathing zones near the machines in twelve schools ranged from 485 to 4096 mg/m^3 (365 to 3080 ppm) for a 15 minute sample.

It is reported that the binding affinities between ss-DNA and SWNT follow the trend d(G)$_{21}$-SWNT > d(A)$_{21}$-SWNT > d(C)$_{21}$-SWNT > d(T)$_{21}$-SWNT (Khamis *et al*. 2010), and from earlier work in our group, the DNA length of 24 is the optimum DNA sequence length for methanol sensing (Liu *et al*. 2011). Thus, DNA 24G- and DNA 32G-decorated SWNT sensors were chosen to test the effectiveness of this sensing system. We built the sensor array with two bare SWNT sensors, two DNA 24G-functionalized SWNT sensors, and the other two DNA 32G-functionalized SWNT sensors, and the responses when exposed to methanol vapor were collected and compared.

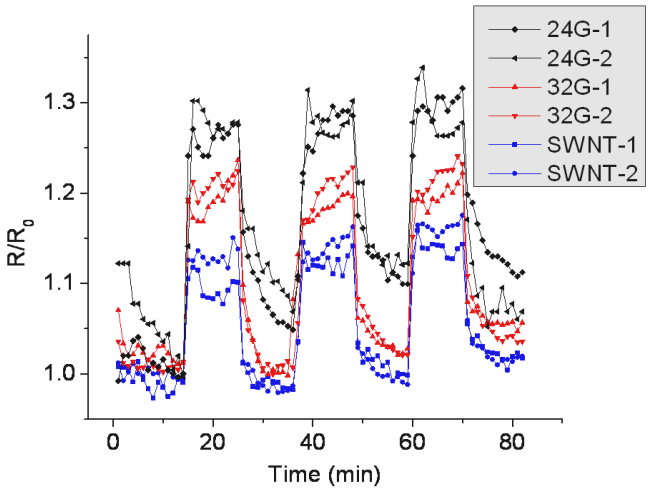

Fig. 7 Normalized resistances of DNA 24G, DNA 32G-functionalized SWNT nanosensors and of bare SWNT when exposed to methanol vapor

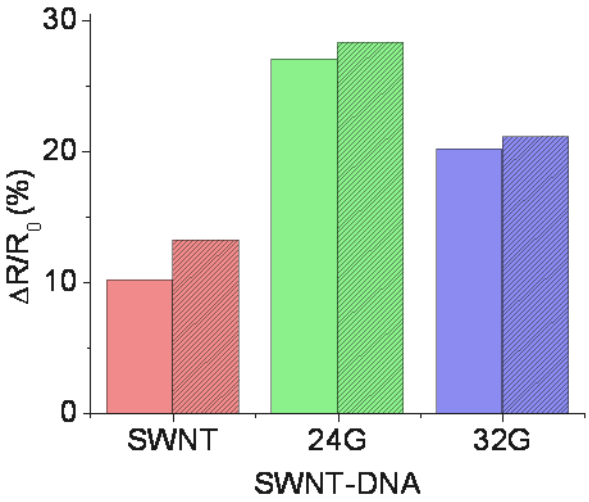

Fig. 8 Resistance changes of DNA 24G, DNA 32G-functionalized SWNT nanosensors and of bare SWNT sensors when exposed to methanol vapor

The sensor array was applied to detect methanol vapor (50 ppm) at room temperature. It was mounted onto a testing board with the signal processing unit and a wireless transmitter (Fig. 5).

This system functions as an autonomous unit for gas monitoring. Then the board and the petri dish containing methanol were confined in a sealed chamber. Changes in resistance, which can

reveal the presence of certain chemicals, were recorded and displayed on the GUI interface (Fig. 6).

The sensing part was exposed to air, methanol vapor, and air consecutively for three times, and the normalized resistances of the six sensors were collected and plotted in Fig. 7. It is reported that the resistance of SWNT sensors remained almost the same when they were exposed to the ambient air until certain specific gases became the subjects (Chen *et al*. 2010, Khamis *et al*. 2010). Our group's former research also confirmed this point (Liu *et al*. 2011). The bare SWNT sensors had an instant response to the methanol vapors and could almost return to their initial resistances in air. But, functionalizing the SWNT sensor with DNA-24G and DNA 32G improved the performance of bare SWNT sensors by 300%, and 200%, respectively. The responses of the six sensors are displayed in Fig. 8. For SWNT sensors functionalized with the same DNA sequence, the changes in resistance for methanol were almost the same. This confirmed the sensing characteristics of DNA decorated SWNTs with two independent sensors. For SWNTs functionalized with different DNA sequences, the results demonstrated that odor responses of DNA-decorated SWNT sensors are sequence dependent.

After illustrating the feasibility of this sensing system as a gas sensor, we emphatically studied and compared the detection of acetone vapor and hydrogen chloride gas using our highly sensitive DNA-SWNT sensor array. The seven DNA sequences utilized in the detection of acetone and HCl are 24 A, 24 Aa, 24 GT, 24 Ma, 8 GT, 16 GT, and 32 GT. The reason that we selected DNA sequences of 24A, 24Aa was to examine the effect of amine group for the sensing performance of such DNA sensors, while DNA 24 Ma (a mixed sequence with two amine groups at the two ends, shown earlier) could clarify whether increasing the diversity of the DNA bases will improve the sensing results. On the other hand, wrapping of CNTs by ss-DNA was found to be sequence-dependent. DNA-GT series can readily wrap around SWNTs based on their electrostatics of the DNA-CNT hybrid. Additionally the $d(GT)_n$-CNT hybrids have a much more uniform periodic structure (Zheng *et al*. 2003), which makes them a great candidate to study the length dependence upon the gas sensing on acetone and HCl.

3.2 Acetone detection by sensor array

Besides methanol, some other volatile organic compounds are also required to be monitored, as they evaporate rapidly, and the vapors are undoubtedly hazardous to human health. For example, acetone, which is commonly used in chemical reagent industry, can cause illnesses to human bodies. According to the Environmental Fact Sheet from New Hampshire Department of Environment Service, irritation of the eyes and respiratory system, mood swings, and nausea are seen in humans exposed to Acetone with the concentration of 500 ppm in air and greater. It was reported that individuals may develop headache, tiredness, dizziness, allergy, and even unconsciousness when exposed to the air which contained high acetone vapor concentration (Godish 1989). Thus, it is necessary to detect and monitor the level of acetone vapor concentration in the environment for our safety. On the other hand, acetone vapor can also be utilized as a marker for some disease diagnostics. It is reported that acetone concentration in the breath of a healthy person is around 5 ppm, while the level of acetone for a patient with diabetes mellitus reaches 300 ppm. Thus, acetone can be a breath marker for diagnosis of diabetes (Cao 2006).

Acetone is a very volatile organic compound, thus it is very easy to obtain acetone vapor from acetone solution. In order to obtain 50 ppm acetone vapor, we used dipropylene glycol (DPG) to adjust the acetone partial pressure in atmosphere. DPG is a mixture of three isomeric chemical

compounds: 4-oxa-2, 6-heptandiol, and 2-(2-Hydroxy-1-methyl-ethoxy)-propan-1-ol. It is a colorless, nearly odorless liquid with a high boiling point and low toxicity. Due to its extremely low vapor pressure (0.06 mm Hg @ 25^{o}C), DPG has been widely employed to modify the partial pressure of analytes. In addition, SWNTs have no response to DPG, thus the responses of SWNT sensor to acetone solution diluted with DPG is purely caused by acetone (Liu *et al*. 2011). The 50 ppm acetone vapor was generated by mixing 6 μL acetone solution with 50 mL DPG. The vapor pressure of acetone at room temperature is about 184 torr (according to Sigma-Aldrich), while that of DPG at 20^{o}C is less than 0.01 torr (from Sigma-Aldrich), which is extraordinary smaller than that of acetone.

$$\text{Vapor pressure of acetone in DPG solution is } P^{i}_{acetone} = \chi_{acetone} \times P_{acetone} \tag{2}$$

$$\chi_{acetone} = \frac{n_{acetone}}{n_{acetone}+n_{DPG}} = \frac{\frac{0.791\times6\times10^{-3}}{58}}{\frac{1.023\times50}{134.17}+\frac{0.791\times6\times10^{-3}}{58}} = 2.15\times10^{-4} \tag{3}$$

Here, $\chi_{acetone}$ is the mole fraction of acetone, and $P_{acetone}$ is the vapor pressure of pure acetone solution. Since the vapor pressure of acetone at room temperature is about 184 torr, the vapor pressure generated by this diluted acetone solution is 0.03956 torr. Divided by 760 torr atmospheric pressure, the concentration of acetone vapor in air is around 50 ppm. The acetone in DPG solution was put into one petri-dish and was exposed to the sensor array in a sealed chamber during the detection of acetone vapor, as shown in Fig. 9. When the chamber was opened, the sensors was exposed to ambient air, the resistance of the six sensors all dropped simultaneously.

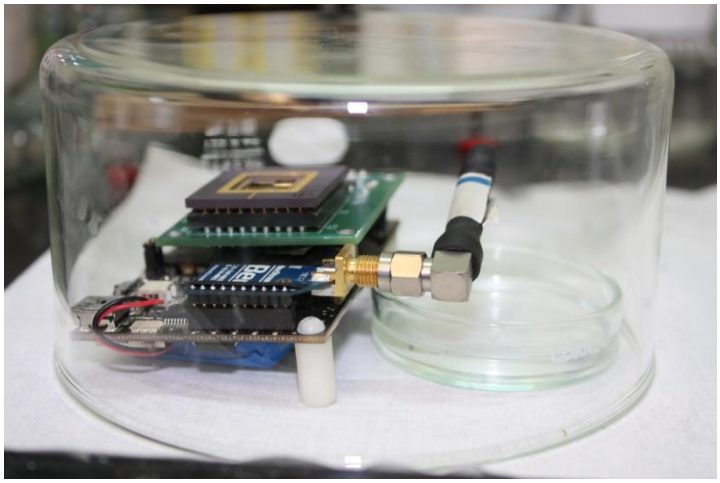

Fig. 9 Testing setup diagram

The sensor array was exposed to air, acetone vapor, and air consecutively for two cycles, and the normalized resistances of the six sensors were collected and plotted in Fig. 10. The resistances of the sensors when exposed to air almost plateaued. Therefore they can be used to normalize the resistances of the sensors when exposed to acetone. Each exposure time was about 20 minutes, and

each time the acetone vapor was replaced by air, the sensor could almost regenerate. The reason for continued increase in the resistance over time, when exposed to acetone for 20 minutes is probably because the acetone molecule is much bigger than methanol one, so the adsorption of acetone onto the sensor is more flexible, taking more time to reach an ultimately stable stage. The sensor made by Salgado *et al*.'s group also displayed instability towards detecting acetone (Salgado *et al*. 2006). Moreover, it's reported that the sensitivities vary at different operating temperatures and the sensor is more sensitive to methanol than acetone until reaching very high temperature (above 325^{o}C) (Wen *et al*. 2010). The time it takes acetone to get saturated in the air is also temperature dependent (Sahay 2005). But as long as the resistance can sustain itself in air, the resistance changes when exposed to subject gases can reliably be used for detection of certain gases. The two cycles of testing confirmed the repeatability and reversibility of the nanosensors to acetone.

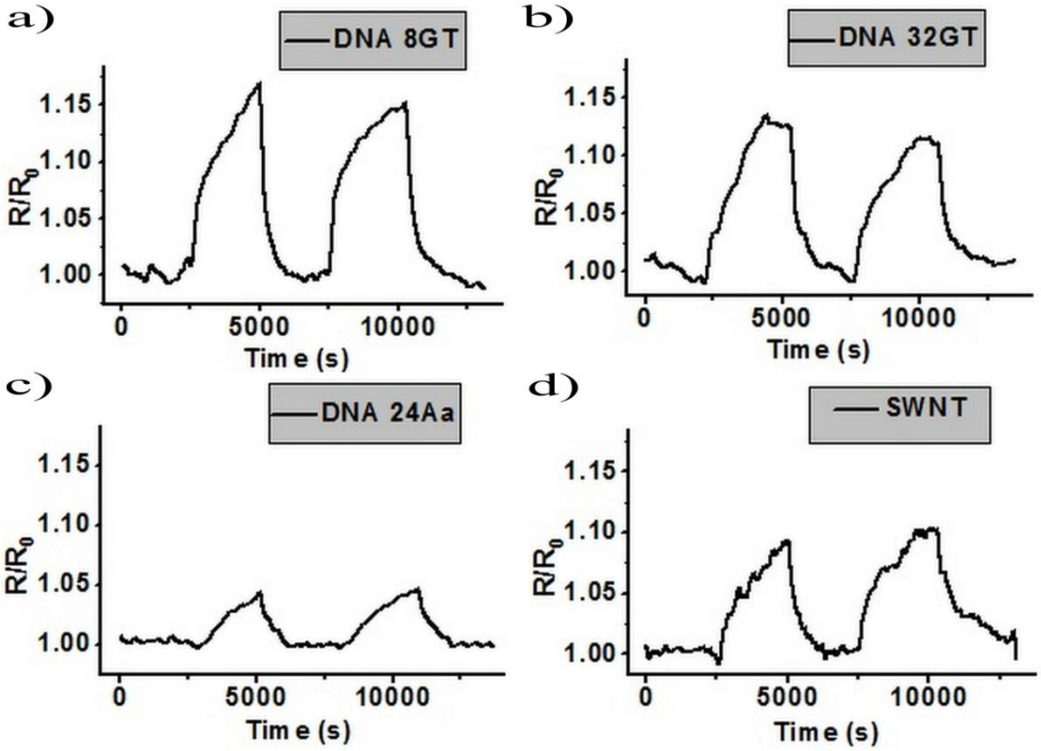

Fig. 10 Normalized resistances of DNA 8GT (a), DNA 32GT (b), DNA 24Aa, (c) - functionalized SWNT nanosensors and of bare SWNT and (d) when exposed to acetone

Compared to the responses of other DNA sequences with the same length-24 bases (Fig. 11(a)), the DNA Ma-functionalized SWNT nanosensor exhibited the highest resistance change (10.15%), and it is much larger than the response of bare SWNT sensor (3.03%). The DNA A, DNA GT, and DNA Aa-functionalized ones showed resistance changes in a decreasing order. The responses of DNA GT with different sequence lengths when exposed to acetone were compared in Fig. 11(b).

The changes in resistance of SWNTs functionalized with DNA of different sequence lengths varies.

As shown in Fig. 11(b), the DNA 32GT functionalized SWNT sensor gave the best performance, followed by DNA 8GT, DNA 16GT and DNA 24GT. All DNA decorated SWNT sensors demonstrated larger changes in resistance towards acetone vapor, which indicated that the DNA modification had greatly improved the sensitivity of bare SWNT sensors. For the reproducibility, revealed by the variation of $\Delta R/R_0$ among different sensors of the same type, bare SWNT sensors have the smallest value (0.138); while the DNA functionalized SWNT sensors show slightly bigger deviations (0.277~1.125) (Table 2). The variation of the sensing performance between different DNA functionalized SWNT sensors is because the decoration conditions cannot be the same, and the affecting parameters can be assembly temperature, moisture level, operations, etc.

Table 2 Responses of DNA decorated SWNT sensors to acetone at room temperature

Acetone	SWNT	DNA 24A	DNA 23Aa	DNA 24Ma	DNA 8GT	DNA 16GT	DNA 24GT	DNA 32GT
$\Delta R / R_0$ (%)	3.03 ± 0.138	8.88 ± 0.466	5.82 ± 0.338	10.15 ± 0.667	9.85 ± 1.04	6.71 ± 0.844	8.23 ± 0.277	11.03 ± 1.125

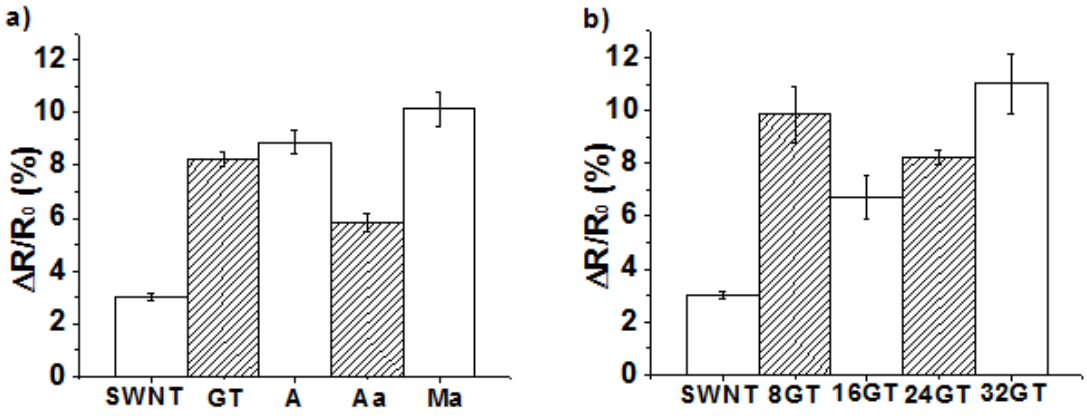

Fig. 11 Resistance changes of DNA 24GT, DNA 24A, DNA 24Aa, DNA 24Ma (a), and DNA 8GT, DNA 16GT, DNA 24GT, DNA 32GT (b)-functionalized SWNT sensors and of bare SWNT nanosensors when exposed to acetone vapor

3.3 Hydrogen chloride detection by sensor array

During the last few decades, the emission of pollutant gases, such as HCl, SO_2, SO_3, CO, NO, NO_2, and CO_2, has become a serious problem. HCl is one of the major contributors to the acid rain as it can easily dissolve in water and become a strong corrosive acid. HCl gas is mainly generated from burning fuels which contain chlorine like coal, and incinerating waste which includes plastic.

The review reports that the coal burning plants produce 14-220 ppm HCl gas while incinerators

show 215-1250 ppm of HCl gas emission (Williams 1990). HCl, is a pulmonary irritant with intermediate water solubility that can cause acute damage in the upper and lower respiratory tract. It is also highly corrosive to metals and skins. Many countries have introduced some strict regulations to control and reduce the emission level of HCl gas, since the quality of environment is directly related to the health of humans and wildlife. Thus, monitoring of the HCl gas is urgently needed.

Unlike acetone which can simply be generated from its evaporation from a solution, HCl vapor generated from 37% HCl solution will contain water vapor which can significantly influence the sensing response of DNA-SWNT sensors. We therefore utilized a chemical reaction between NaCl and concentrated H_2SO_4 solution to generate HCl gas. The concentrated H_2SO_4 solution will not generate vapor, thus it will not interfere with the HCl gas testing, and it can also act as a dehydrate agent. Using the same test setup in Fig. 9, we placed concentrated H_2SO_4 into the petri dish at first, and then added 0.5mg NaCl. Due to the heat release from the chemical reaction, we wait for 1 minute. Then the sensor array was put into the chamber with generated HCl gas. The concentration of HCl gas in the chamber was estimated to be 50 ppm.

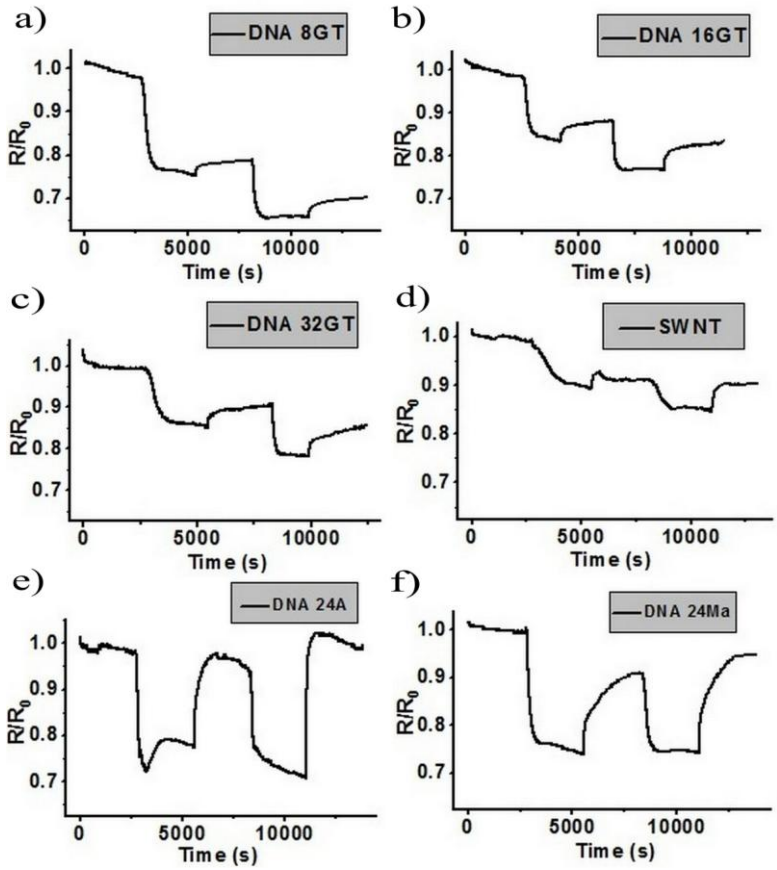

Fig. 12 Normalized resistances of DNA 8GT (a), DNA 16GT (b), DNA 32GT (c), DNA 24A (e), DNA 24Ma (f) - functionalized SWNT nanosensors and of bare SWNT (d) when exposed to HCl gas

Unlike the positive change of resistance when the nanosensors were exposed to acetone vapor, they exhibited a decrease in resistance when exposed to HCl gas. Besides, the reduced resistance of most nanosensors cannot recover to the initial value when the HCl gas is switched back to air.

For SWNTs decorated with DNA 24 A and DNA 24 Ma, the resistances returned to their initial values when HCl gas was replaced by air, while the nanosensors functionalized with other DNA sequences and the bare SWNT sensor could hardly regenerate. The different responses may result from the different affinities between SWNTs and DNAs of different sequences. Such drift of the baseline, represented by the resistance in ambient air, is possibly caused by the intercalation of the SWNT bundles with HCl molecules and the interaction of HCl with adventitious impurities. The interaction of HCl with adventitious impurities may change the intertube contact resistance. Some of the binding sites were irreversible, and once they were bonded to nanotubes, they were very hard to get desorbed, which made the sensor unable to regenerate in an ambient environment. The incomplete recovering of SWNTs after testing with HCl gas was consistent with the results reported by other groups (Bekyarova *et al*. 2010). In order to solve this problem, several methods have been reported to recover the sensing ability of SWNT sensors. For instance, Bekyarova *et al*. functionalized the SWNT with different organic chemicals and facilitated the regeneration of SWNT sensor to HCl gas (Bekyarova *et al*. 2010). They used covalent bonding to decorate different organic chemicals onto the single-walled carbon nanotube to attract HCl. The presence of these functional groups allows the indirect attachment of the HCl molecules on SWNTs as the functional groups will contact the HCl molecules before HCl molecules get the chance to attach on SWNTs. In this case, the HCl will not affect the properties of the SWNT bundles. The best recovery performance was introduced by the group-$COOC_5H_4N$, as its conjugate acid might have the necessary oxidation potential to enable the electron transfer from the semiconducting SWNTs. It resulted in introducing holes into the valence band and decreasing the resistance. A similar mechanism has been reported and verified (Bekyarova *et al*. 2004, Bekyarova *et al*. 2007). There are some other methods for the SWNT sensors to regenerate, including heating the device to high temperature (Kong *et al*. 2000) and UV irradiation (Pengfei *et al*. 2003).

As to our functionalization, the DNA molecules were non-covalently bound to the SWNT, which may not be as strong as covalent bonding. And more importantly, DNA molecules cannot provide holes into the valence band to allow electron transfer from SWNTs, while the organic functional groups used in Bekyarova et al.'s study can. So, maybe we can improve the sensor's regeneration by functionalizing SWNT with some organic functional groups before decorating DNA onto it as long as the organic functional group won't affect the functions of DNA. Even without rectification of the sensor's recovery condition, we can still detect trace amount of HCl gas based on the data collected with the sensor array.

Table 3 Responses of DNA decorated SWNT sensors to HCl at room temperature

HCI	SWNT	DNA 24A	DNA 24Aa	DNA 24Ma	DNA 8GT	DNA 16GT	DNA 24GT	DNA 32GT
$\Delta R / R_0$ (%)	1.170 – 8.56 ± 1.170	– 21.92 ± 1.257	– 17.01 ± 1.299	– 14.75 ± 1.009	– 19.62 ± 0.972	– 12.31 ± 1.006	– 8.53 ± 0.650	= 11.82 ± 0.776

Comparing the different DNA sequences with the same length-24 bases (Fig. 13(a)), DNA

24A-functionalized SWNT nanosensor shows the highest resistance decrease (21.92%), which is much larger than the value of the bare SWNT sensor (8.56%). The DNA decorated SWNT sensors react with HCl gas in the order of DNA 24A> DNA 24Aa> DNA 24Ma> DNA 24GT. DNA 24GT gave even a little less resistance change than bare SWNT (Fig. 13(a)), which means it barely improved the performance of the sensing of HCl gas. For DNA GT composed of different sequence lengths, the responses of DNA-SWNT sensors were also different (Fig. 13(b)). As to HCl gas, the DNA 8GT functionalized SWNT nanosensor gave the best performance, while DNA 16GT and DNA 32GT were almost the same, ranking second.

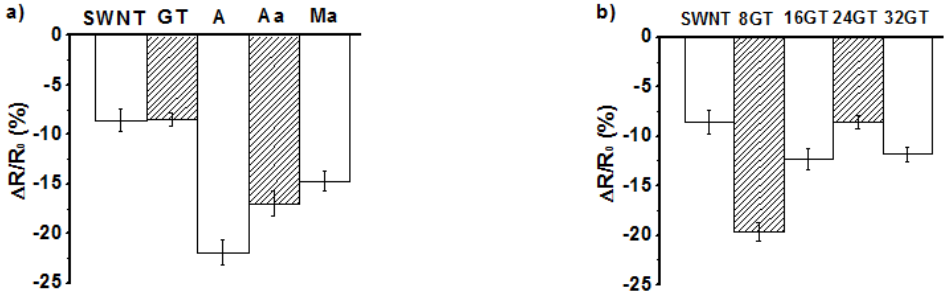

Fig. 13 Resistance changes of DNA 24GT, DNA 24A, DNA 24Aa, DNA 24Ma (a), and DNA 8GT, DNA 16GT, DNA 24GT, DNA 32GT (b)-functionalized SWNT nanosensors and of bare SWNT when exposed to HCl gas

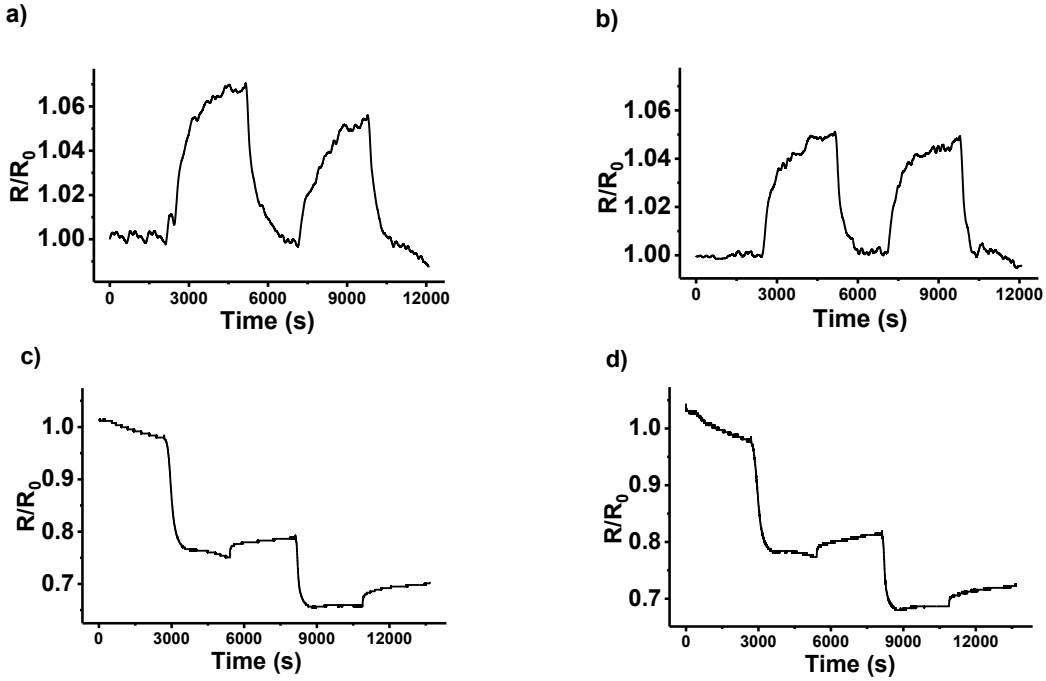

Fig. 14 Repeatability with different sensors on one array. (a), (b) Normalized resistances of different DNA 16GT-functionalized SWNT nanosensors when exposed to acetone; (c), (d) Normalized resistances of DNA 8GT-functionalized SWNT nanosensors when exposed to HCl gas

The detection results of DNA-functionalized carbon nanotube sensors are highly repeatable for different sensors; no matter the analyte is acetone vapor (Figs. 14(a) and 14(b)) or HCl gas (Figs. 14(c) and (d)). The device to device variation is around 5%, so the DNA modified SWNT sensors provide particular high accuracy.

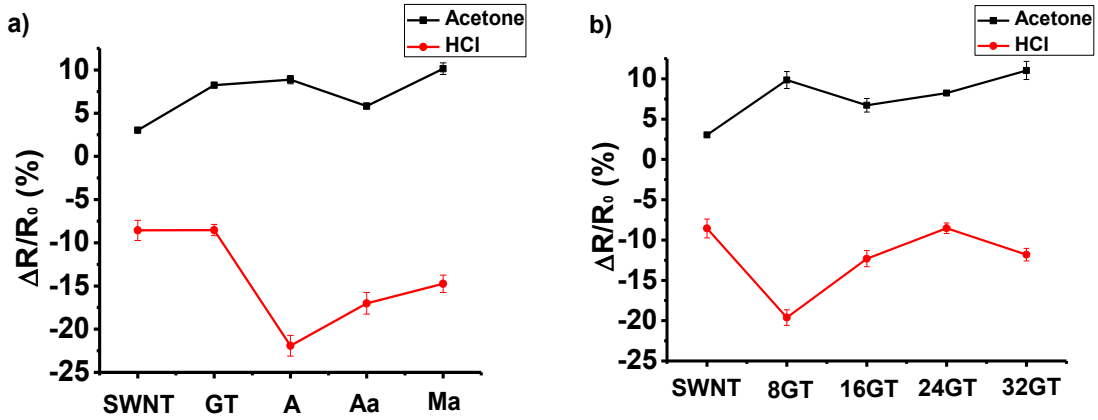

Fig. 15 Comparison of the sensing responses to acetone vapor and to HCl gas

Fig. 15 clearly shows that one specific SWNT nanosensor, regardless of whether it was with functional groups or not, demonstrates different responses to different analytes. The nature of ss-DNA-SWNT interactions plays an indispensable role in the sensing performance of the DNA-SWNT sensors. Stronger binding sites permit greater amount of ss-DNA to be bonded onto the SWNT surfaces, which facilitates the adsorption of polar analytes (Khamis *et al*. 2010), and this may explain the specificity of the DNA sequence used to decorate the device on the performance in gas sensing. Normally, functionalized SWNT nanosensors reveal better sensitivity than bare SWNT ones. From the results of seven different DNAs functionalized SWNT sensors, it is very likely the DNA 24 Ma and DNA 32 GT can be used to sense acetone vapor, while the DNA 24 A and DNA 8 GT can be employed to detect HCl gas. However, with our sensor array, we can simply use the pattern generated by the different sensors in just one array to distinguish different analytes, like those two in Fig.15. Such pattern recognition is applicable to the situation when all gas subjects are present at the same concentration.

4. Future work

One sensor array with DNA 24 Ma, DNA 32 GT, DNA 24 A, DNA 8 GT functionalized SWNT nanosensors and two bare SWNT nanosensors will be built in order to distinguish acetone vapor from HCl gas. This can be accomplished because the DNA 24 Ma and DNA 32 GT decorated SWNT sensors are responsive to acetone vapor while the DNA 24 A and DNA 8GT decorated ones are responsive to HCl gas. Once they are all in an array, the DNA 24 Ma and DNA 32 GT decorated SWNT sensors will show much larger resistance change to acetone than to HCl; while the DNA 24 A and DNA 8 GT decorated ones show much larger resistance change to HCl than to

acetone.

Another subject worth studying is the sensing ability on different concentrations of analytes. A DNA functionalized SWNT sensor array was demonstrated to be able to distinguish different analytes, so we will further study the sensing capabilities of this sensor array on a large gas concentration range and the device resolution. The results will shed light on the utilizing of this kind of sensor array onto the environmental monitoring and also for health diagnosis.

5. Summary

It is obvious that the sensing performance of DNA functionalized SWNT sensors is highly sequence dependent. Different sequences of DNA can selectively respond to certain chemicals.

Samuel and their group studied four kinds of homo-DNA functionalized SWNT field effect transistors (SWNT-FETs) for detecting several gaseous analytes (Khamis 2010). The response to trimethyl amine (TMA) followed the trend $d(G)_{21}$-SWNT > $d(A)_{21}$-SWNT > $d(C)_{21}$-SWNT >$d(T)_{21}$-SWNT. They also suggested a comparable trend of $d(G)_{21}$ > $d(A)_{21}$ > $d(C)_{21}$ >$d(T)_{21}$ exists for DNA-SWNT binding affinities. They concluded that stronger binding between DNA and SWNT will be formed with a greater amount of DNA adsorbed to SWNT surface. This provides a more hydrophilic environment around the hydrophobic SWNT core and thus facilitates the adsorption of polar analytes. The work of A. T. Charlie Johnson and his colleagues also confirmed this conclusion as the nanosensors functionalized with strands comprised of purines (A or G) were responsive to methanol vapor and TMA while the ones with strands of pyrimidines (C or T) were not (Johnson *et al*. 2010). However, it was also displayed that for SWNT sensors with multi-oligomer DNA sequences, the cases are complex and cannot be easily explained. Besides, the sequence length of DNA decorated on the SWNT sensors also affects the sensing performance.

Yu *et al*. found that the decoration of 24 bases of poly-G ss-DNA on SWNTs led to the highest resistance change when the sensors were exposed to methanol and IPA vapors (Liu *et al*. 2011).

But in this paper, we found the optimum length of DNA GT decorated on SWNT sensors for detection of acetone and HCl is 32 and 8, separately. So the sensing performance must also be analyte related other than sequence dependent.

All DNA decorated SWNT sensors demonstrated better sensing performance than bare SWNT nanosensors towards both methanol and acetone. Methanol and acetone are both polar organic compounds. A possible reason for the DNA based sensitivity enhancement of SWNT nanosensors to polar molecules could be that the decoration of DNA has converted the SWNT surface properties from hydrophobic to hydrophilic. Zhao *et al*. suggested that the hydrophobic groups on the nitrogenous bases of oligonucleotides tend to bind onto the surface of SWNTs, while the hydrophilic phosphate groups along the backbone of oligonucleotides do not bind to SWNTs (by the method of Molecular Dynamics Simulation), (Zhao *et al*. 2007). Thus after DNA was coated onto the carbon nanotubes, the outer surface of the DNA-SWNT structure would be covered by the hydrophilic backbones of DNA. This hydrophilic surface environment would then attract polar molecules including methanol and acetone. On the other hand, although HCl is also a polar molecule, the way SWNT nanosensors respond to HCl is different. When SWNT nanosensors were exposed to HCl, there existed the interaction of HCl with adventitious impurities and the intercalation of the SWNT bundles with HCl molecules. The interaction of HCl with adventitious impurities may change the intertube contact resistance. In addition, some of the binding sites were irreversible. Once they were bonded to nanotubes, they were very hard to get desorbed. Moreover,

the variety of DNA sequences may also change the 3D structures of DNA-SWNT. This explains the variation of the sensing results between methanol/acetone and HCl.

Our objective is to find which DNA sequence and length is able to identify a specific chemical, therefore a fixed concentration of 50 ppm was applied in all tests here. The DNA sequences used in this paper were chosen based on prior work in our group. If we want to try many possible DNA sequences to find the specific one for our analyte, it would be very costly and time consuming.

Thus, a well-organized and convenient method to screen out the proper DNA sequence for specific use is required. We are exploring computational system, e.g., information theory (IT) and molecular dynamics (MD) for simulating and analyzing these sensing processes to clarify the mechanisms behind this sequence-dependent sensing performance.

6. Conclusions

Here, we have designed and fabricated a wireless sensor array based on ss-DNA-decorated SWNT on micro devices. Nine DNA sequences were utilized to decorate SWNTs and their responses were recorded and simultaneously displayed on a GUI interface. The sensor array was tested with three different gases: methanol, acetone and HCl. The responses of the DNA 24 Ma decorated SWNT sensor and the DNA 24 A decorated SWNT sensor were strongest to acetone and HCl respectively. As to the DNA GT composed of different sequence lengths, the length of 32 bases showed the biggest resistance change to acetone and length 8 to HCl. When chemicals like HCl are detected, we can further improve the sensor regeneration by changing the binding methods (Bekyarova 2010) or heating the sensor (Kong *et al*. 2000). Also, the concentration dependence of the analytes will be explored and specified. An array-based sensing approach is enormously efficient in real-time, highly sensitive and capable of fast air quality monitoring because of its high selectivity, good sensitivity, great repeatability and excellent precision. Therefore, this sensor array shows great promise to become a Smart Air Quality Sensor, and can be used in household, subway, and many other public places where safety monitoring is very important.

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