Design and Testing of a Wireless Portable Carbon Nanotube-Based Chemical Sensor System

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Abstract—The design and preliminary testing results of a wireless portable carbon nanotube-based chemical sensor system are presented in this paper. The overall sensor system consists of a chemical sensor module, a microcontroller-based data acquisition module, a multiplexer and constant current source module, and a wireless communication module. The chemical sensor module is based on use of an interdigitated electrode (IDE) configuration. A layer of single-walled carbon nanotubes (SWNTs) is laid on the IDE electrodes. Chemical sensing is based on detection of changes in the conductivity of the SWNT network. The system has 32 channels of chemical sensing elements. It is self-contained and portable, and wirelessly transmits measurement data to a PC via an 802.11b wireless LAN. Preliminary results on chemical detection and chemical selectivity are described.

Keywords—Chemical sensor, carbon nanotube, wireless

I. INTRODUCTION

Chemical sensors have been developed for decades to detect various concentration levels of gases and vapors for deployment in a wide range of applications in industry, space mission, environment monitoring, military, and others. The detection usually centers on changes of a particular property or status of the sensing material (such as temperature, electrical, optical, etc) upon exposure to the species of interest. The sensing material itself has ranged across the periodic table from inorganic, semiconducting elements to organic compounds, either in bulk or thin film form. One of the most investigated classes of chemical sensors in the past is the high-temperature metal oxides sensors due to their high sensitivity at low ppm to ppb concentration level, with tin oxide thin films as an example [1]. Polymer sensors have been studied in recent years because they can be operated at room temperature with low power consumption and are easily fabricated. While commercial sensors based on these materials are available, continued research is making progress with sensing technologies using new sensing materials and new transducer platforms. New sensing technologies, such as nanotechnology-based sensors, are developed to overcome the huge power consumption and poor selectivity of metal oxides sensors, and to improve the poor sensitivity and narrow detection spectrum of polymer sensors.

Typical figures of merit expected from a chemical sensor include sensitivity (even down to single molecule level), selectivity, low power consumption, rapid response time and rapid sensor recovery time. Sensors based on the emerging nanotechnology promise to provide improved performance on all of the above aspects compared to current micro and macro sensors. Nanomaterials exhibit small size, light weight, very high surface-to-volume ratio, and increased chemical reactivity compared to bulk materials; all these properties are ideal for developing extremely sensitive detectors. The potential of nanomaterials for not only detection but also for protection and remediation has been outlined in a recent report by the National Science and Technology Council [2].

One of the most exciting nanomaterials that has captured the attention across the world is carbon nanotube (CNT). It exhibits extraordinary mechanical, electrical, and optical properties. These interesting properties have prompted wide range investigations for applications in nanoelectronics, high strength composites, field emitting devices, catalysts, etc. The single-walled carbon nanotube (SWNT) has all the atoms on the surface and therefore would be exposed to the environment, allowing a change in its properties sensitively. The first demonstration of a SWNT-based chemical sensor took the form of a chemical field effect transistor or a CHEMFET [3]. In [3], a single semiconducting SWNT was used as the channel material and the conductivity was shown to change upon exposure to NO₂ and NH₃. Thus the potential for using CNTs in chemical sensors was first recognized. However, it still is a challenge to make practical sensors due to difficulty in fabrication complexity, low sensor yield and poor reproducibility [4].

A different configuration of carbon nanotube-based chemical sensors with much easier fabrication process was introduced in [5]. First, an interdigitated electrode (IDE) configuration is fabricated using conventional photolithographic method with a nominal finger width of 10 μm and gap size of 8 μm. The fingers are made of thermally evaporated Ti (20 nm) and Au (40 nm) on a layer of SiO₂ thermally grown on top of a silicon wafer. Second, a thin layer of carbon nanotubes forming a network is laid on the electrodes of IDE by solution casting process. The conductivity of the CNT network changes upon exposure of gases or vapors. Such a process is significantly simpler and produces consistent sensors based on statistical properties of the CNT network with high yield (~100%). The IDE configuration facilitates effective electric contact between SWNTs and the electrodes over large areas while providing good accessibility for analytes in the form of gas/vapor adsorption or contaminants adsorption/extraction in/from liquid to all SWNTs including semiconducting tubes. This paper presents the design and preliminary testing results of a wireless, self-contained,
portable chemical sensor system, based on the IDE configuration.

II. CHEMICAL SENSOR SYSTEM DESIGN

While carbon nanotube-based chemical sensors using the IDE configuration have been prototyped in laboratory environment [5], the objective of the effort presented in this paper was to design, build, and evaluate a self-contained, wireless, portable, multi-channel chemical sensor system using the IDE configuration. A schematic diagram of the sensor system is depicted in Figure 1. It consists of a chemical sensor module, a multiplexer and constant current source module, a microcontroller-based data acquisition module, and a wireless communication module. The chemical sensor module contains 32 sensing elements, each of which has an IDE configuration laid with pure SWNTs, with polymer coated SWNTs, or with metal nanoclusters doped SWNTs. At the heart of the data acquisition system is a microcontroller (PIC16F688) from Microchip Technologies Inc. that samples each sensor element through a set of four Texas Instruments CD4051 multiplexers. Each multiplexer reads from a group of eight chemical sensor elements. The LM234 constant current source from National Semiconductor was used to provide a constant 100 μA to each sensing element. Four of these devices were used to excite each group of eight sensing elements. Measuring conductivity or resistance is achieved by supplying a constant current and then measuring the corresponding voltage across the sensor. Also included in the data system was the AD22100K temperature sensor from Analog Devices. The microcontroller reads all 32 chemical sensor and temperature data. It then generates an RS-232 serial data output that can be connected directly to a PC for data logging or to a wireless serial device server for wireless data transmission. In this design, a W2150 serial server from Moxa Technologies, Inc. was used to wirelessly transmit data to a laptop or desktop PC via an 802.11b wireless LAN. A prototype of the chemical sensor system is shown in Figure 2.

Figure 1. Schematic of the chemical sensor system.

Figure 2. Photo of the chemical sensor system.

III. TESTING RESULTS

Preliminary testing results of the chemical sensor system are presented in this section. Typical raw measurement data and processed data are first provided and discussed. The simultaneous use of all 32 sensing elements for chemical discrimination is then described.

A. Sensor Response

Figure 3 shows typical sensor responses that were observed over a period of 25 hours from three sensing elements. The plot shows how sensor resistance changes in response to the introduction of an analyte at approximately 19 hours and 20.5 hours. Data from the sensors was collected at one sample per second. For this experiment, a cotton swap soaked in a cleaning solution with sodium, ammonium and Triethanolammonium as its main ingredients was placed approximately 2 cm above the sensor surface, but the concentration of the analyte was not quantified. The sensor resistance for sensors x, y, and z increased approximately 10% from the sensor’s resistance just prior to the detection of the analyte. However, it was also observed that the sensor resistance varies in a wide range over the time without the introduction of analytes. With this in mind, statistical properties of the measurement data were investigated to extract the information that presents the detection of analytes. Such information can be drawn from measurement data of a single sensor or multiple sensors.

Figure 4 shows the variance of the measurement data from sensor x computed over a moving window of 1000 samples of the data. This window represents 16.7 minutes of measurement data. Small variance indicates small changes in measurement data over this period of time. Large variance indicates possible presence of analytes. It is seen from Figure 4 that variance is substantially larger at two instants of time when analyte was introduced.

In addition to computing variance of individual sensors, statistical correlation of two sensors can be computed to confirm the presence of analytes. Figure 5 shows the
correlation coefficient for two sensors y and z, computed over the same moving window. It indicates an agreement between two sensors that both sensors were exposed to the presence of analytes. An enlarged view of Figure 5 around the two instants of analyte introduction is shown in Figure 6. Similar results were also observed for sensor pairs (x,y) and (x,z).

Figure 3. Typical sensor response from three sensing elements plotted in the form of the normalized relative resistance change with respect to the initial resistance R₀.

Figure 4. Variance of data from one sensor.

**B. Chemical Discrimination**

A sensor system with 32 channels is built to study the feasibility of chemical discrimination. The nanomaterials laid on this array of 32 sensing elements were individually studied for different gases. They can be classified in three different categories: 1) pristine single walled carbon nanotubes, 2) carbon nanotubes coated with different polymers (chlorosulfonated polyethylene and hydroxypropyl cellulose), 3) carbon nanotubes loaded with Pd nanoparticles and monolayer protected clusters of gold nanoparticles. When exposed to a vapor-phase analyte, each sensing element in the array responds uniquely. There will be a reproducible combination of resistances, or “smellprint”, for each vapor/gas (Figures 7 and 8). The sensor response is measured as a bulk relative resistance change (it can be current change or voltage change as well): ΔR/Δt (see Figure 3). To calculate the relative sensor response (SR), the initial sensor resistance (or current/voltage), (Ro) and the resistance (or current/voltage) during the exposure (Rt) are measured. Looking at the histogram of sensor responses from 32 sensors to different gases/vapors, patterns for each gas/vapor can be established for discrimination among tested gases/vapors. Pattern recognition algorithms can be applied to deal with such a large set of data. For example, the principal component analysis (PCA) was applied to the data set that was collected from this carbon nanotube based sensor array. The PCA is to express the main information in the variables X = {x_k, k = 1, 2...K} by a lower number of variables {i_1, i_2...i_K} (A < K), the so-called principal components of X [6]. In this case, the X is a matrix of 32 sensor responses to these gases and vapors from...
the sensor array. Figures 9 and 10 show the first three principal components for the data of 32 sensor responses to various tested gases and vapors. Here the three principal components are labeled as F1, F2, and F3, corresponding to \( i_1 \), \( i_2 \), \( i_3 \) above. The gases and vapors were completely separated in principal component space, which indicates that this sensor array provides a high discrimination power to these gases and vapors. Since these gases were tested by this sensor array at different concentration levels in the range of 5ppm to 45ppm, it is unique that this sensor array can be used for high sensitive gas and vapor detection and discrimination. It is noticed that this sensor array can discriminate the pair of HCl and HCN, both having proton, and the pair of HCl and Cl2 gases, both having chlorine. This sensor array discriminates the gases and vapors by their chemical nature rather than their concentrations. Also, it can discriminate the gases that have some similarity in their chemical nature, which shows a high discrimination power.

![Figure 9. Chemical detection using principal component analysis.](image)

![Figure 10. Chemical detection of four analytes.](image)

**REFERENCES**


**IV. Conclusions and Summary**

An integrated chemical sensor system based on use of SWNTs as sensing elements was presented. The overall design and system components were described. The chemical sensor system is easily fabricated using the existing technologies. It is wireless, portable, and readily deployable in the field. Preliminary testing results presented the paper validate the operation of the sensor system. Further testing and performance analyses under varying environment conditions are currently ongoing.