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Characteristics of Single- Wall and Multi-Wall Carbon Nanotubes Hydrogen Gas Sensor

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ABSTRACT: Two types of carbon nanotubes (MWCNTs and SWCNTs) films have been utilized to the manufactured hydrogen gas sensor. CNTs films have been deposited on porous silicon substrate by drop method. The two sensors show high sensitivity to hydrogen gas at different concentrations. Test results demonstrated that the SWCNTs have a better performance than MWCNTs. The SWCNTs gas sensor shows high sensitivity at 150^{o}C with response time 29sec, whereas MWCNTs gas sensor shows best sensitivity at 200^{o}C with response time 24sec.

KEYWORDS. SWCNT, MWCNT, Hydrogen sensor, Sensitivity, response time.

I. INTRODUCTION

Chemical gas sensors have been widely used to recognize and monitor various gaseous analytes in numerous applications including flammable and toxic gas detection, environmental monitoring, space exploration, industrial emission ,public security, medical diagnosis and food quality control[1,2]. Gas sensors with high sensitivity and selectivity are required for leakage detections of explosive gases such as hydrogen, and for real-time detections of toxic or pathogenic gases in industries [3,4]. In gas sensors, the dynamic component that is sensitive to the target gas molecules is the key part and thus receives intensive attention. For example, after realizing the resistance of some semiconductor materials can be drastically changed by the presence of gas molecules, many works have been directed to research them in gas sensors [5]. Carbon nanotubes have attracted tremendous interest as a novel material in various nanoelectronic devices since it was discovered, they have been examined as materials of chemical sensor because they can detect small concentrations of molecules with a high sensitivity under ambient conditions (thelarge surface area of CNT provides a very large gas absorptive capacity), the CNT sensor has a fast response and the special geometry of the CNT makes it electrochemically active. Further, the electrical properties of a CNT network to be modulated by various chemicals such as H and NO2. [6,7].

Carbon nanotubes are rolled-up graphene sheets occurring as a single - wall (SWCNT) or multi-wall (MWCNT) cylinders. They have diameters from 0.4 up to a few nm, and their lengths range from a few nanometers up to several millimetres. [8].

The present work study the sensing performance of the CNTs Hydrogen gas sensor by using two types of the carbon nanotubes (MWCNT and SWCNT) and make comparisons between them to get the best CNTs type and best performance.

II. EXPERIMENTAL WORK

A. Preparation of the samples

In this work, (2x2 cm^2) dimensions primary n-type silicon wafer substrates were thoroughly cleaned to de-contaminate their surface from any available stains and dirt. A porous silicon layer (PS) was prepared via photochemical wet etching. This process is carried out by using ordinary light source. Its main apparatus consists of a Quartz Tungsten Halogen lamp (250W) integrated with dichroic ellipsoidal mirror, a focusing lens, and the diluted etching HF acid poured in a Teflon container.To prepare CNT sample, 0.01 g of CNT was dispersed in Dimethylformamide (DMF). A magnetic stirrer was incorporated for this purpose for 15 minutes, followed by 1 hour sonication. The obtained solution was used for film deposition on porous silicon by the drop casting method.

B.Gas sensor testing system

The detail of the gas sensor testing unit which was used in the current tests is described elsewhere [9] A steel cylindrical test chamber of diameter 163 mm and of height 200 mm with the bottom base made removable and of O – ring sealed. The effective volume of the chamber is 4173.49 cc; it has an inlet for allowing the test gas to flow in and an

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air admittance valve to allow atmospheric air after evacuation. Another third port is provided for vacuum gauge connection. A multi-pin feed through at the base of the chamber allows for the electrical connections to be established to the sensor and the heater assembly. The heater assembly consists of a hot plate and a k – type thermocouple inside the chamber in order to control and set the desired operating temperature of the sensor. The thermocouple senses the temperature at the surface of the film exposed to the analyte gas. The PC – interfaced multi meter, of type UNI-T UT81B, was used to register the variation of the sensor conductance (reciprocal of resistance) exposed to predetermined air – hydrogen gas mixing ratio. The chamber can be evacuated using a rotary pump to a rough vacuum of 2×10-2 bar. A gas mixing manifold was incorporated to control the mixing ratios of the test and carrier gases prior to being injected into the test chamber. The mixing gas manifold is fed by zero air and test gas through a flow meter and needle valve arrangement. This arrangement of mixing scheme is done to ensure that the gas mixture entering the test chamber is premixed thereby giving the real sensitivity.

III. RESULT AND DISCUSSION

Hydrogen is the lightest and most abundant element in the universe. At standard pressure and temperature, it occurs as diatomic gas. Hydrogen absorption into carbon nanotubes samples at room temperature had no significant change in the resistance of carbon nanotubes. The carbon nanotubes need to be doped with other atoms or operated at higher temperature for them to be a good gas sensor for hydrogen.

The response of a sensor upon the introduction of a particular gas species is called the sensitivity (S). The most general definition of sensitivity applied to solid state chemi-resistive gas sensors is a change in the electrical resistance (or conductance) relative to the initial state upon exposure to a reducing or oxidizing gas component.
It is calculated using the equation:

$$S = \frac{|\Delta R|}{R_o} \times 100\% = \left| \frac{R_{gas} - R_{air}}{R_{air}} \right| \times 100\% \quad \ldots\ldots\ldots\ldots\ldots\ldots 1$$

Or $$S = \frac{|\Delta G|}{G_o} \times 100\% = \left| \frac{G_{gas} - G_{air}}{G_{air}} \right| \times 100\% = \left| \frac{R_{air} - R_{gas}}{R_{gas}} \right| \times 100\% \ldots 2$$

Where R is the electrical resistance and G is the electrical conductance and the subscript “air” indicates that background is the initial dry air state and the subscript “gas” indicates the analyte gas has been introduced.
Fig.1 shows the representative real – time electrical response of a thin film CNT sensing element to H2 gas concentration of 3% in air. The test was performed at various sensing temperatures with 2.5 v and 5 v bias voltage.
The sensitivity S% increased with increasing operating temperature T, where the maximum sensitivity of MWCNT is11.93% and 20.50 % were obtained at 2.5 and 5 volt respectively at 200°C testing temperature, after which it began to drop with increasing T and the test was terminated.
While the sensitivity of SWCNT is 21.84 % and 52.53 % were obtained at 2.5 and 5 volt respectively at 150°C testing temperature, after which it began to drop with increasing T and the test was terminated.

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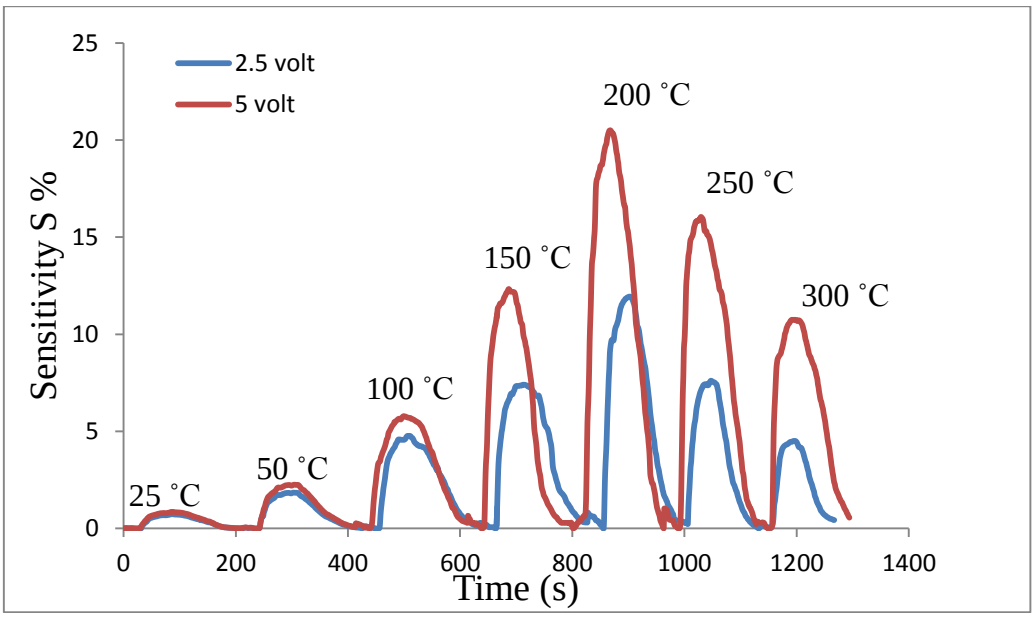
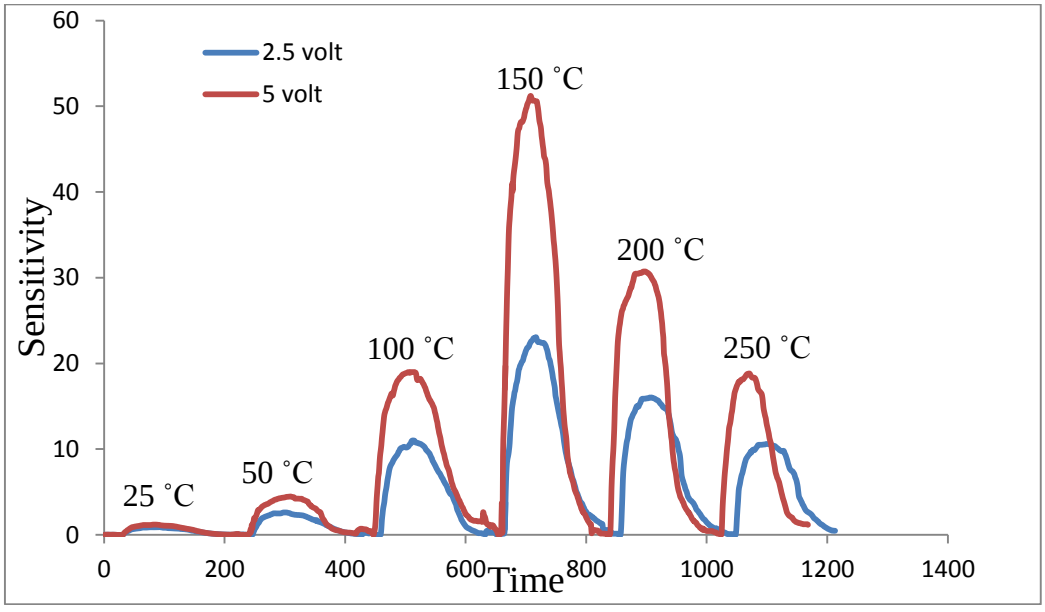


(a)



(b)

Fig.1 Transient response of CNT thin film at various testing temperatures upon exposure to 3 % H2: air gas mixing ratio and 2.5 and 5 bias voltages a) for MWCNT and b) for SWCNT.

We observed that the sensitivity of the gas sensor at 5 volt bias voltage has relatively higher than that of 2.5 volt.

This dependence of sensing response to the operating temperature of the MWCNT film is exhibited in Fig.2. It is seen that the film maximum resistance goes through a maximum on changing T, with the best operating temperature

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at around 200 ºC. Roughly speaking, the increase of Rmax(the left side of the maximum) results from an increase in the rate of surface reaction of the target gas, while the decrease of Rmax(the right side) results from a decrease in the utility of the gas sensing layer. At the temperature of the maximum conductance (response), the target gas molecules have optimum penetration depth into the gas sensing grains (large utility) i.e., optimum reactivity for the diffusion in the whole sensing layer, as well as for exerting sufficiently large interaction with the surface (large gas response coefficient).

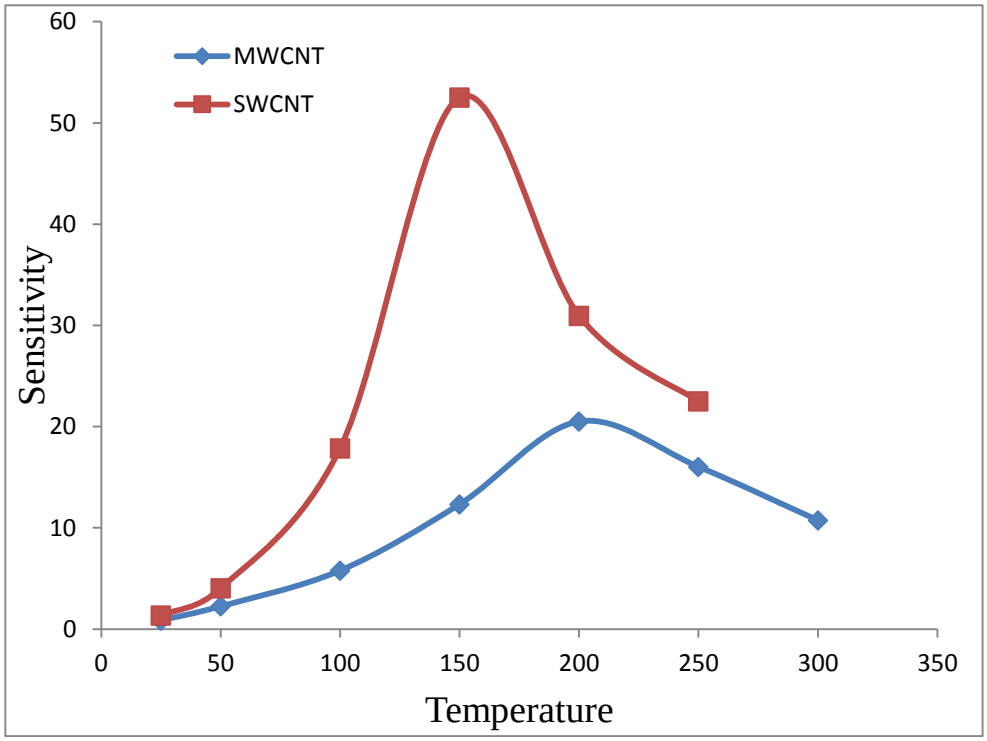

Fig.2 Sensitivity variation with the operating temperature of the MWCNT and SWCNT gas sensor at 5 bias voltage and 3 % H2: air gas mixing ratio.

Fig.3 shows the sensitivity S variation with hydrogen gas concentration. Figure (3.a) shows the sensitivity S% increased with increasing hydrogen gas concentration for MWCNT, where the sensitivity of 14.27 %, 17.28 % and 20.50% was obtained in 1%, 2 % and 3% hydrogen: air gas mixing ratio respectively after which the current tends to saturate with increasing the analyte gas. A relatively short response time of about 24ses and recovery time of about 79ses was noticed at 3% hydrogen gas concentration.

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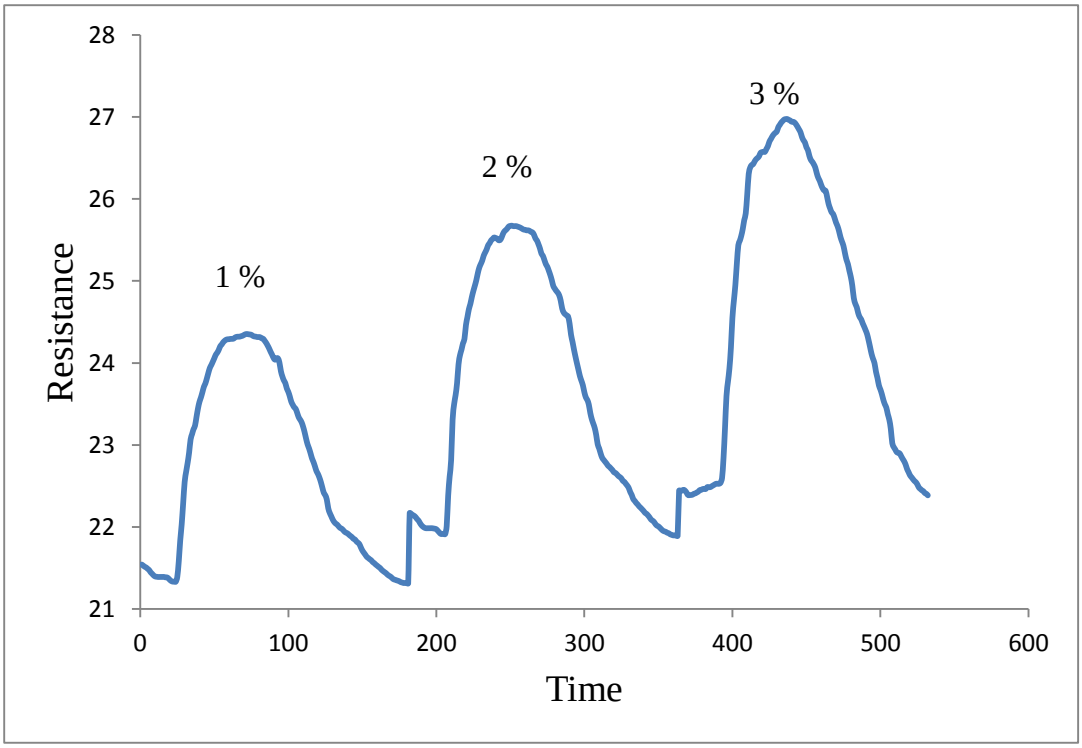

Fig.3-a Transient response of MWCNT gas sensor to different H2: air mixing ratios. The test performed at 200 degrees temperature and 5 v bias voltage

In fig.3-b shows the sensitivity S% increased with increasing hydrogen gas concentration for SWCNT, where the sensitivity of 31.07 % , 43.27 % and 52.53% was obtained in 1% , 2 % and 3% hydrogen: air gas mixing ratio respectively after which the current tends to saturate with increasing the analyte gas. A relatively short response time of about 29 sec and recovery time of about 89 sec was noticed at 3% hydrogen gas concentration.

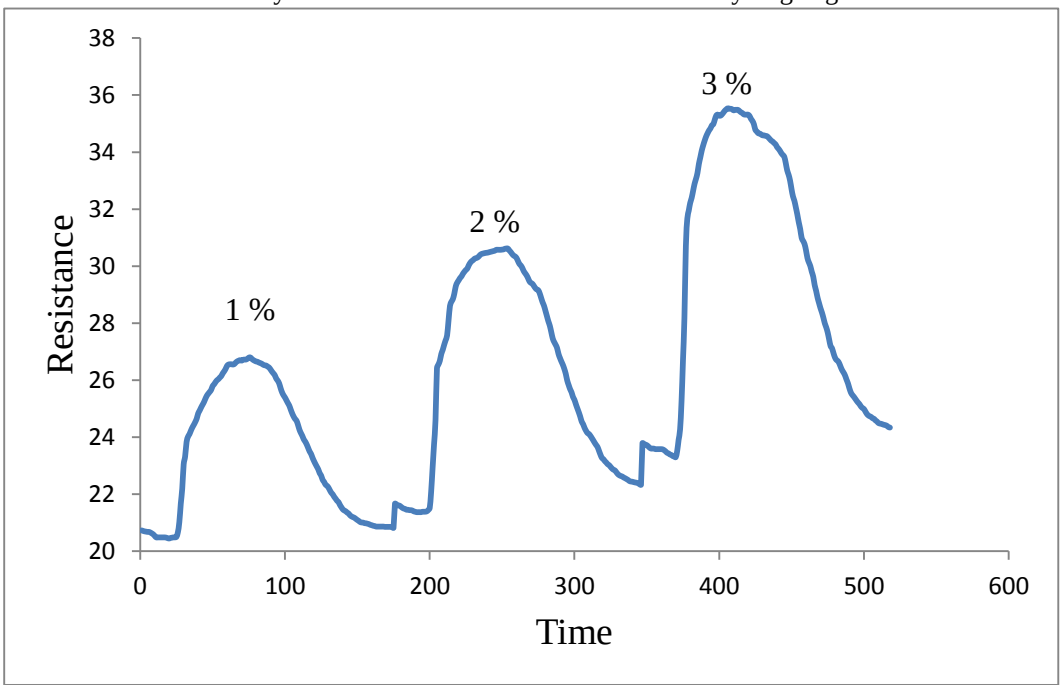

Fig.3-bTransient response of SWCNT gas sensor to different H2: air mixing ratios. The test performed at 150 degrees temperature and 5 v bias voltage

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From the gas sensing data,the result was expected and can be explained by the following: an H_2 molecule is reduced agent that donates an electron to another species. Therefore, H_2can play a role as an electron-donor and/or hole-acceptor. When the CNT gas sensor was exposed to H_2, electrons were transferred from H_2 molecules to the CNTs. H_2 then donated its electrons to the valence band of the CNTs, thereby decreasing the number of holes by recombination, reducing the conductivity of CNTs, and increasing electrical resistance. Therefore, it is reasonable to assume that CNTs are a p-type semiconductor.

The sensitivity of the sensor increased as the concentration of H_2 increased. CNTs have four possible sites to which gas molecules can be adsorbed: internally, in interstitial channels, in external grooves, and on external surfaces. At a low concentration, the adsorption sites were not entirely filled by H_2 molecules. However, once the concentration of H_2 reached a critical level of 30%, the adsorption sites became completely filled, leading to saturation. When the concentration exceeded 30%, the adsorbed H_2 molecules became tightly packed on the CNT surfaces, the resulting is no more interactions between H2 and CNTs.

IV. CONCLUSIONS

The best performance of two types CNTs (MWCNTs and SWCNTs) as hydrogen sensing were realized in the study. SWCNTs film provides good sensitivity to hydrogen gases more than MWCNTs (52.53% and 20.5%) respectively. The sensor temperature (150C^o for SWCNT and 200C^o for MWCNT) reduces the response to hydrogen gases from (29-24 Sec).

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