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Research Article

Study of Various Characteristics of SWCNT Chemiresistive Sensor on Exposure of Ammonia (NH_3) at Room Temperature

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Abstract

In the present report, high quality randomly network of Single walled carbon nanotubes (SWCNTs) with diameter range of 1-2nm were grown on Fe based Si substrate at the growth temperature of (6500C) through plasma enhanced chemical vapour deposition (PECVD technique. The aim of this work was to fabricate highly sensitive and excellent SWCNT gas sensors which will operate at room temperature and overcome the deficiencies of metal oxide sensors which usually operate at elevated temperature. The surface morphology, structural quality and diameter distribution of as grown SWCNTs was observed by field emission scanning electron microscope (FESEM), high resolution transmission electron microscope (HRTEM) and Raman spectroscopy respectively. A comparative NH3 gas sensing study was done at different operating temperature and concentration range. The obtained sensor response at 400C, 1000C and 2000C was 25%, 20%, and 15% respectively showing a decreasing behavior with increasing temperature because adsorption prefers at low temperature. On the other hand, the obtained sensor response at 5ppm, 8ppm and 10ppm was 4%, 8% and 15% respectively showing almost a linear behavior with increasing target gas concentration. In addition of this, the SWCNT sensor had shown excellent performances towards repeatability, selectivity, resistance variation and long-term stability and is a step forward to monitor the environment at room temperature for security reasons.

Keywords: PECVD; RF-Sputtering; SWCNTs; NH_3; Sensor

Introduction

Gas sensing applications involves the environmental monitoring, food quality observation, defense area, fire detection and other safety measures [1,2]. Different pollutants releasing in the environment require continuous monitoring by different kind of sensitive sensors. Gas sensors are the indispensable part of our modern technology and are being much focused by scientific community [3,4]. Excellent sensors are being designed and fabricated in various ways for the environmental monitoring and mostly for industrial security. Due to the better development in industrial sectors, various dangerous chemicals are being released in the natural environment [5,6]. With much concentration of these dangerous pollutants like NH_3, NO_2, CO_2 and O_3 etc., the quality of environment degrades day by day [7-10]. Inspite of environmental pollution, animal, aquatic and plant life is also affected at a major scale [11,12]. Some chemicals are much harmful at trace level concentration.

To trace these dangerous chemicals, various excellent sensor devices are being designed by Nano-technological techniques. Nanotechnology is serving the mankind at the best level. New chemistry and physics can be generated by nanotechnology with reduced dimension [13,14]. The properties of the materials get changed as per the dimension is reduced so new and excellent devices can be fabricated with much purity [15,16]. Among these devices, chemical sensors are playing the major role in tracing these chemical species in trace level concentration [17-20]. However, as per the literature survey, most of the metal oxide sensors work efficiently but they lack the high quality of selectivity and operate at high temperature consuming much energy [21,22]. To overcome these limitations of metal oxide sensors, CNT based sensors are showing good performance in sensitivity and selectivity at moderate operating temperature and are also used in other applications like field emission displays devices [23] super capacitors [24], composites [25], transistors etc. [26].

Mostly CNT based chemical sensors are providing the excellent results due to their high aspect ratio supporting the surface phenomenon [27-30]. The sensitivity depends on various factors like operating temperature, target gas concentration, and functionalization [31,32]. The SWCNT sensor shows a decreasing behavior in sensor response with increasing temperature and it shows an increasing behavior with increasing target gas concentration showing almost a linear behavior [33,34].

There are various techniques for the growth of SWCNTs. Among these techniques, PECVD is the renowned technique for the growth of SWCNTs at quite lower temperature as compared to low pressure chemical vapor deposition (LPCVD) [35-38]. Excellent quality and vertical growth of SWCNT has been reported by PECVD technique by different groups of scientific community for different applications [39,40]. The growth is being done in the assist of high quality of plasma [41,42].

In the present work, SWCNTs were grown on Fe based Si substrate by PECVD technique at an operating temperature of 650^0C. SWCNT sensor was fabricated for NH_3 sensing at different operating temperature and concentration. The detailed gas sensing investigations were carried out. The sensing performances were done at 40^0C, 100^0C and 200^0C with achieving the sensor response 25%, 20%, and 15% respectively leading a decreasing behavior with elevation of temperature. The response was also investigated at varied concentration of NH3 (10ppm, 20ppm and 30ppm and the response was 4%, 8% and 15% respectively showing almost a linear behavior with increasing target gas (NH_3) concentration. The sensor characteristics like sensor repeatability, selectivity, resistance variation and long-term stability were also investigated and the sensor had shown excellent performances towards these characteristics.

Experimental

The experimental procedure for the growth of SWCNTs includes deposition of nano-catalyst particles on Si substrate by RF-sputtering technique and then it follows the growth of SWCNTs by PECVD technique. For the proper deposition of catalyst, Fe target with 2-inch diameter and 2mm thickness was used. The substrate was purely cleaned in an ultrasonic bath with acetone before loading in the RF- Sputtering chamber. The target and the substrate distance were kept 10cm. The RF-power and the vacuum level during the deposition of catalyst was 200 W and 10^{-5} torr respectively. Argon (Ar) was used as a sputtering gas into the chamber at a rate of 60sccm with mass flow controllers (MFC). The deposition time was about 3-5min and the film thickness was observed a few nanometers.

Then the growth of SWCNTs follows in the PECVD reactor. Simply the PECVD reactor consists two equal area parallel plate electrodes in the vacuum chamber (PECVD)/bell jar). Mostly the upper electrode is grounded and the lower electrode is used to hold the substrate on which the SWCNT growth occurs. Normally a mechanical pump is also associated with the PECVD reactor for the creation of adequate pressure for the operation and growth of SWCNTs. A proper cooling arrangement is provided beneath the substrate. The Fe catalyst deposited Si substrate was subjected into the chamber on a graphitic heater of PECVD reactor. An adequate vacuum of the order of 10-3 torr was raised in the chamber for the operation process. The catalyst film was then pre-treated with hydrogen at a temperature of 500^0C under the hydrogen (H_2) with flow rate of 750sccm. A temperature of 650^0C was raised for the growth of SWCNTs in the PECVD chamber. After achieving the proper pressure and temperature in the reactor, a selected amount of source gas (C_2H_2 20sccm) associated with hydrogen (H_2) and ammonia (NH_3 150sccm) was inserted in the reactor through a showerhead which is housed direct opposite to the Fe catalyst-based Si substrate.

The hydrogen works as the carrier gas and the NH3 works as the diluting agent. At this temperature the growth of SWCNTs occur in the assist of plasma. The source gas gets decomposed in the reactor in the assist of high quality of plasma and the carbon atom gets nucleated on the Si substrate and hence SWCNT growth occurred at lower temperature. The growth of SWCNTs was terminated by putting off the power supply of the heater and the gas flow. The as grown samples were then cool down for further characterization. The PECVD unit and the schematic illustration have been represented in figure 1.

SWCNT Characterization and their Property measurements

The as grown SWCNTs were characterized to investigate the growth, structure and morphology properly by using different techniques. The surface morphology of as grown SWCNTs was observed by the FESEM (Nova Nano). The structural information was gathered by Raman spectroscopy (HORIBA Jobin Yvon (LABRAM

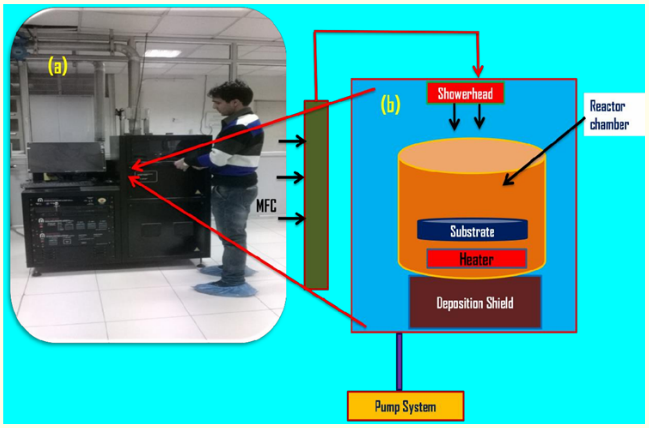

Figure 1: (a) PECVD unit and (b) the schematic illustration.

SWCNT sensor fabrication and gas sensing mechanism

The as grown SWCNTs were used for the fabrication of SWCNT sensors for environmental protection from harmful gases and chemicals. The designed sensor model of SWCNT and step by step configuration has been shown in figure 2. It usually consists a Si substrate covered with silicon dioxide film for electrical isolation. Best qualities of gold electrodes were made on the SWCNT network having electrode gap 2mm.

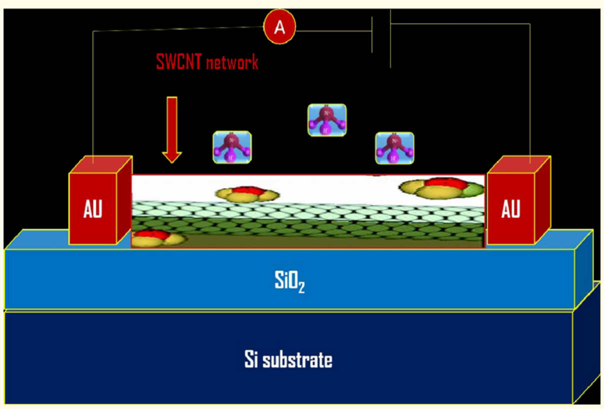

Figure 2: Schematic of SWCNT NH_3 gas sensor.

The fabricated electrodes on SWCNT network were dried in an oven for 1 hour at 1000C so that the good electrical contact may occur and other impurities may be reduced. The schematic model of SWCNT sensor shows how the interaction of target molecules takes place with SWCNT sensor surface. The gas sensing is a surface phenomenon, and the sensing mechanism involves the charge transfer between target gas and the SWCNT sensor surface. Depending on the nature of target gas (Reducing or Oxidizing) and the charge transfer, the change in resistance occurs accordingly. Reducing gases usually donate electrons towards the SWCNT sensor surface while oxidizing gases accept electrons from the SWCNT sensor surface during the interaction.

The as fabricated SWCNT sensor has been subjected into a self-assembled sensing unit for gas sensing measurements. The sensing unit consists a well-designed transparent cuboid glass chamber, a sample holder, temperature controlled resistive heater, a circulating fan for uniform distribution of target gas, an injection needle to insert the target gas in the chamber. There is a small hole for the target gas injection. The schematic diagram of sensing unit and data acquisition system has been shown in figure 3, [43].

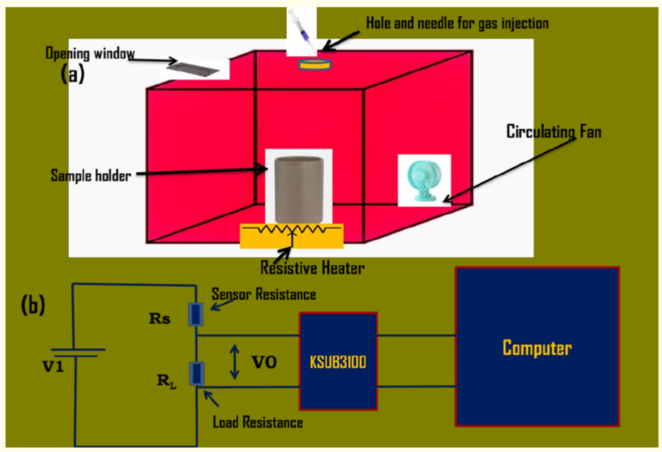

Figure 3: (a) Gas Sensing assembly (b) data acquisition system.

The whole sensing assembly is associated with Keithley Data Acquisition Module KUSB–3100 and a computer [44].

Results and Discussions

FESEM study

The surface morphology of as grown SWCNTs has been investigated by field emission scanning electron microscope (FESEM). From the FESEM micrograph of SWCNTs, it is evident that high quality randomly oriented branched network type of SWCNTs have been synthesized successfully. The figure itself indicates that very rare dense SWCNTs were grown on Fe deposited Si substrate by PECVD technique. The rare dense SWCNTs are much sensitive. These types of structures are very useful as per the gas sensing application point of view. The Fe catalyst particles are also visible in the micrograph.

The FESEM micrograph also shows excellent uniform growth of SWCNTs on Si substrate. The as grown SWCNTs have length in micrometer and the diameter is in 1-2 nanometer range. The obtained micrograph of SWCNTs has been shown in figure 4.

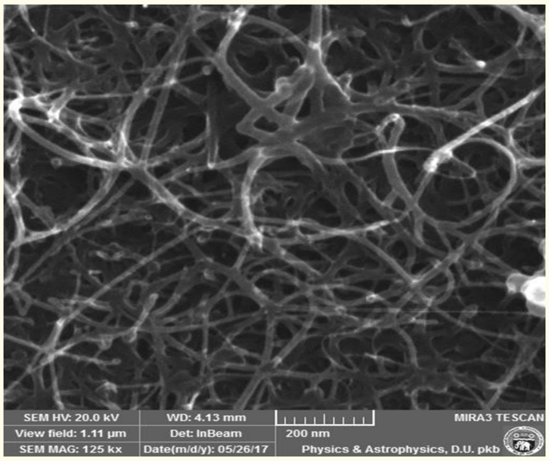

Figure 4: FESEM micrograph of as grown SWCNTs.

HRTEM study

As per the nanoscale range of SWCNTs, it is more difficult to make the best observation of SWCNTs and to distinguish them individually. The HRTEM can be regarded as the highly powerful technique for this purpose. It provides the wealth of information about the structural quality of as grown SWCNTs. The HRTEM observation micrograph of as grown SWCNTs has been shown in Figure 5. On observing this micrograph high quality defect free long network of SWCNTs were observed. Iron (Fe) catalyst nanoparticles in a circular shape were also visible in the obtained structure. The observation has been taken at high magnification.

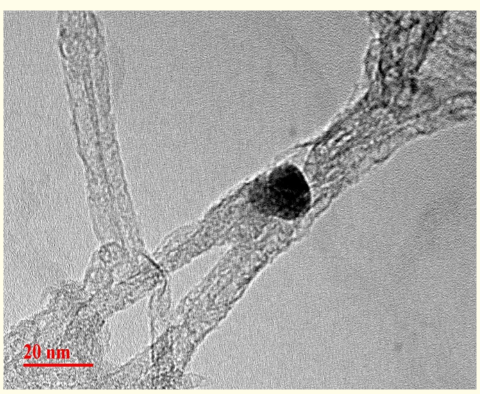

Figure 5: HRTEM micrograph of as grown SWCNTs.

Raman Spectroscopic study

To get the detailed study about the structural properties of as synthesized (SWCNTs), Raman investigation has been done by Raman spectrometer of HORIBA Jobin Yvon (LABRAM HR 800 JY) at a wavelength of 633 nm respectively. Raman investigation provides us complete information about the diameter distribution and crystallinity nature of SWCNTs.

From Raman spectra, we get the different bands which provide different information about the as grown SWCNTs. The G-band of Raman spectra stands for the graphitic nature of SWCNTs and the D-band provides the defective nature (disordered nature) of SWCNTs. The G-band usually stands between 1500-1650 cm-1 and D band stands between 1300-1360 cm-1. In the present case the G-band and D- band are occurring at 1582 and 1336 respectively. The ratio of the IG/ID has been calculated 1.18. The RBM modes have been observed at 220, 260, 268 and the diameter of as grown SWCNTs has been calculated by the relation $d = 248/\nu$, where d is the diameter of SWCNT in nm and ν is the Raman shift in cm-1. The observed diameter of as synthesized SWCNTs was in the range of 0.95-1.12.nm. The obtained Raman Spectra has been shown in Figure 6.

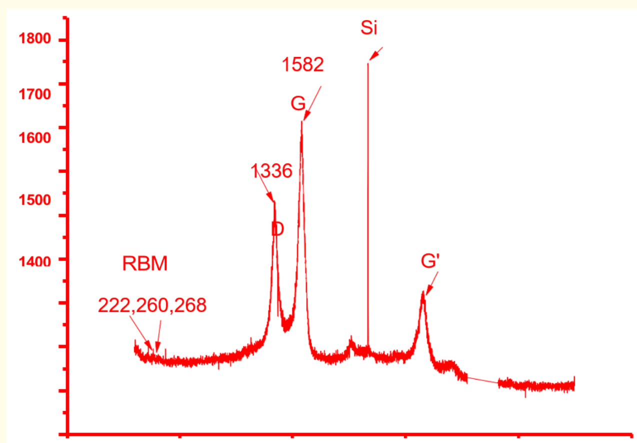

Figure 6: Raman spectra of as grown SWCNTs.

Gas sensing investigation of as fabricated SWCNT Sensors

Particularly the gas sensing mechanism involves the charge transfer and is based on the adsorption processes and is a surface phenomenon. It is also a reversible process (adsorption and desorption). Depending on the nature of the gas (Reducing or Oxidizing) the variation of resistance takes place accordingly. When the target gas molecules are adsorbed on the SWCNT surface during interaction, a charge transfer takes place and hence the variation in resistance occurs. Some target gases may be electron donating while others may be electron accepting.

Effect of Operating Temperature

The as fabricated SWCNT sensor was exposed to NH3 at a constant concentration of 10ppm, and the sensor response measurements were taken at 40^0C, 100^0C and 200^0C. The obtained sensor response was observed 25%, 20% and 15% for 40^0C, 100^0C and 200^0C respectively as shown in figure 7. The increasing operating temperature enhance the desorption rate because the adsorption always prefers at room operating temperature. Hence gas sensing response decreases as the operating temperature increases. The sensor response was calculated with the simple formula

S (%) = Ra/Rg

Where S is the sensitivity of the sensor, Ra is the resistance of sensor in air and Rg is the resistance of sensor in the presence of target gas. The response/recovery characteristics were observed very fast. However, the recovery percentage (%) at 400C was observed about 70-85% and for 100^0C and 200^0C it has been found about 90-99% because desorption enhances with increasing temperature. The response time was observed about 4-6 seconds and the recovery time was observed about 6-10 seconds indicating high sensitivity of as fabricated SWCNT sensor.

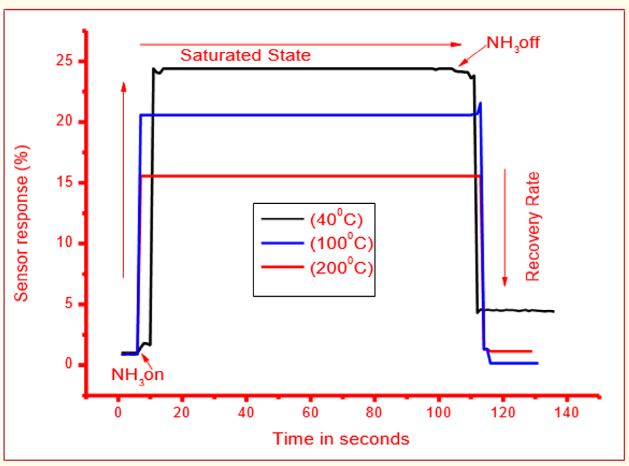

Figure 6: SWCNT sensor response at various operating temperature.

The behavior of the observed sensor response of as fabricated SWCNT sensor at different operating temperature has been also shown in figure 8. It is evident from the figure 8 that sensor response is largely dependent on the operating temperature. The as fabricated SWCNT sensor was tested for resistance measurement. The drastic variation in resistance has occurred on exposure of ammonia (NH3) at a concentration of 10ppm range and at temperature of 400C. As per the reducing nature of NH3 gas, the resistance was drastically increased and after some time it became saturated as is shown in figure 9. The gas has not been removed in this case and because of this, no recovery has been observed. It was observed that 26-ohm resistance change has occurred in this case.

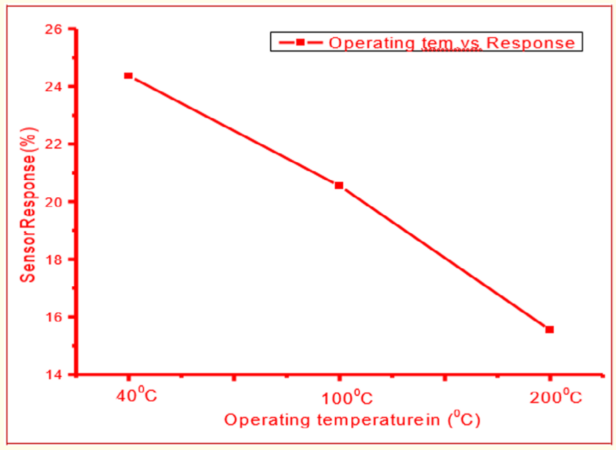

Figure 8: SWCNT Sensor response with increasing operating temperature.

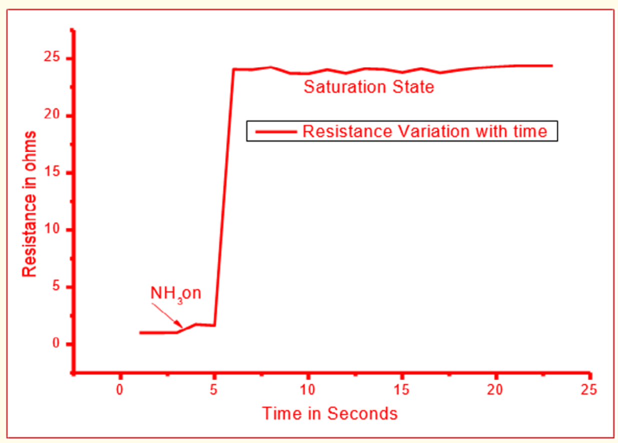

Figure 9: SWCNT sensor resistance variation with time.

Effect of NH_3 Concentration

The variation of sensor response with the effect of different NH3 concentration at room temperature has been shown in figure 10.

The sensor response was observed at 5ppm, 8ppm, 10ppm and the sensor response was observed 4%, 9%, and 15% respectively which directly indicate that the sensor response directly increases with the increase of target gas concentration. The highest sensor response was observed at 10ppm and the lowest sensor response was observed at 5ppm. With the increased target gas concentration, much charge transfer occur between the SWCNT sensor and the target gas molecules which directly leads to the large change in resistance and hence leading higher response.

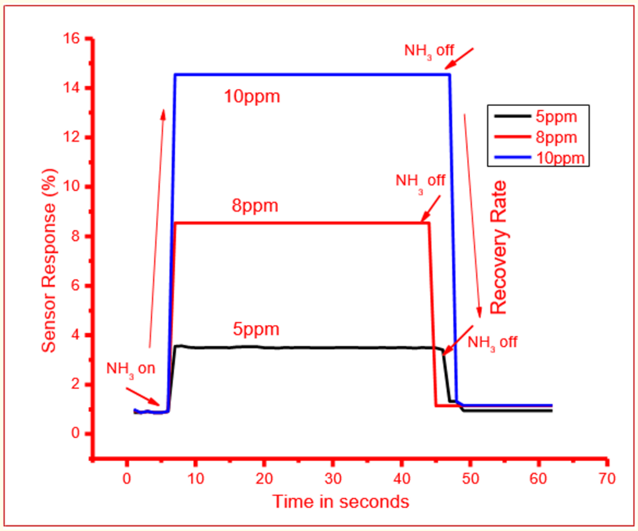

Figure 10: SWCNT sensor response variation with NH_3 concentration.

The sensor response shows a linear behavior with increasing target gas (NH_3) concentration as shown in figure 11. However, a new approach of thermal heating has been used to obtain the complete recovery. An excellent recovery was observed in this case by thermal heating. The electrons of the target gas mostly take place in defective sites of SWCNTs and hence require sufficient energy for desorption. The heating treatment provides the required amount of energy to the NH_3 for desorption.

Repeatability of the Sensor

The main important characteristic of a good sensitive sensor is its proper repeatability. In this case, the fabricated SWCNT sensor was tested under the processes of repeatability at a constant concentration NH_3 (5ppm) and at 400C. High quality and constant sensor response of the order of 1.8 % was observed for about four cycles. It is evident from Figure 12, that our fabricated SWCNT sensor possesses the excellent repeatable nature and is a good step for the application point of view.

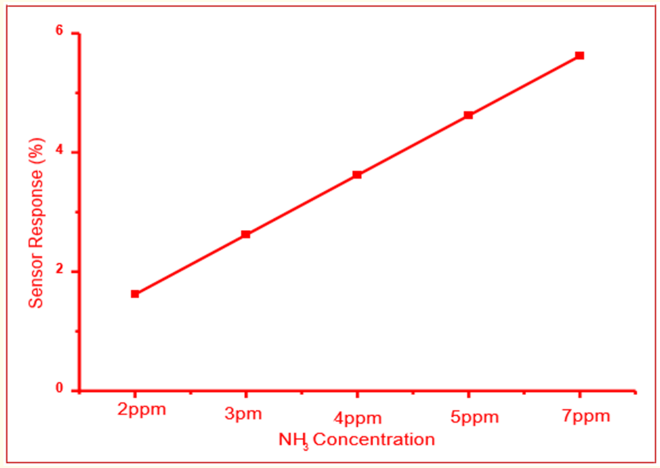

Figure 11: Linear behavior of sensor response with increasing concentration.

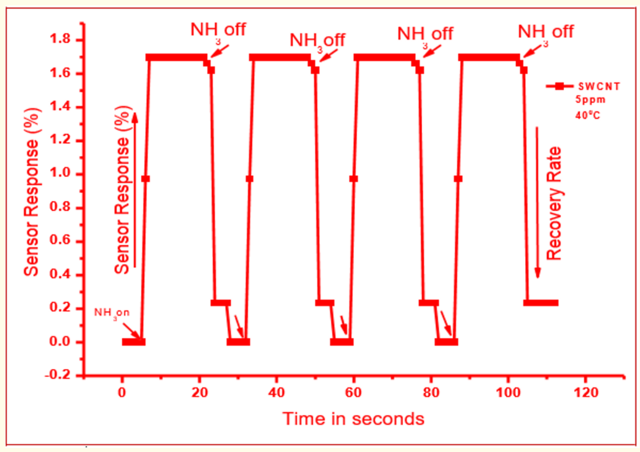

Figure 12: SWCNT sensor repeatability at constant concentration 5ppm.

However, the repeatability was also observed at different trace level concentration for about four cycles to check the proper sensitivity and performance of the as fabricated SWCNT sensor and it has been shown in figure 13.

In the field of sensor technology, stability is the important factor of concern and may be defined as the ability of the sensor to keep its good reproducible and excellent performance of response for many cycles and for a long period of time. Without stability, the

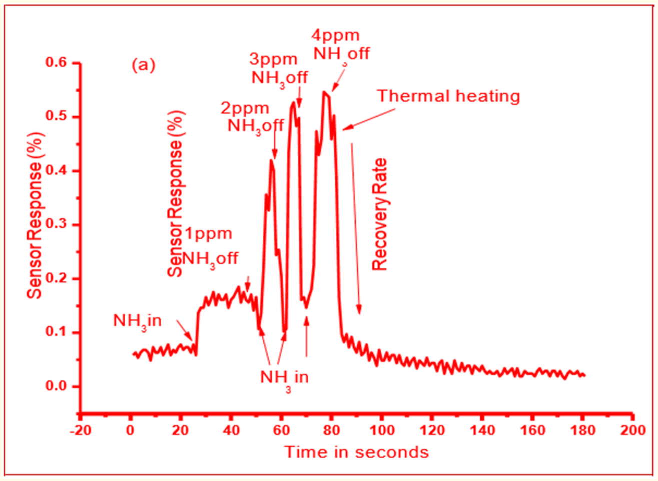

Figure 13: SWCNT sensor response repeatability at various trace level concentration.

fabrication of an excellent sensor is impossible. The stability of as fabricated SWCNT sensor has been recorded continuously for 25 days at a periodic interval of five days. The stability measurements were taken at room temperature and at a constant NH3 concentration of 5ppm. A slight fluctuation occurs but not much decline in sensitivity was observed. The stability graph of SWCNT sensor has been shown in figure 14. From the response curves, it is obvious that the fabricated SWCNT sensor showed very high stability for a long period of time and is applicable for long term use.

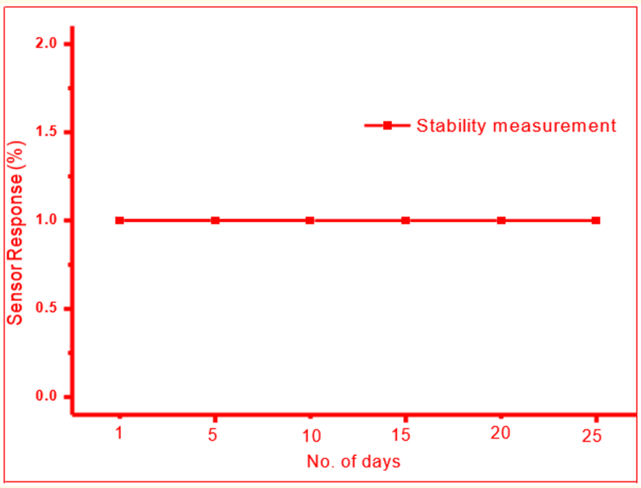

Figure 14: Response of gas sensing stability at a periodic interval of five days.

Selectivity of the Sensor

Selectivity of an excellent sensor usually determine the highly and selective response of the fabricated sensor towards a target gas in a group of gases. Selectivity plays an important role in the sensor technology. The fabricated SWCNT sensor was also tested for the different gases like ammonia, nitrogen dioxide, liquefied petroleum gas, acetone and methanol at a constant concentration (NH_3 20ppm) and at a constant temperature 40^{0}C. The obtained response of different gases has been shown in figure 15. The fabricated SWCNT sensor is highly sensitive towards the NH_3 as compared to other gases.

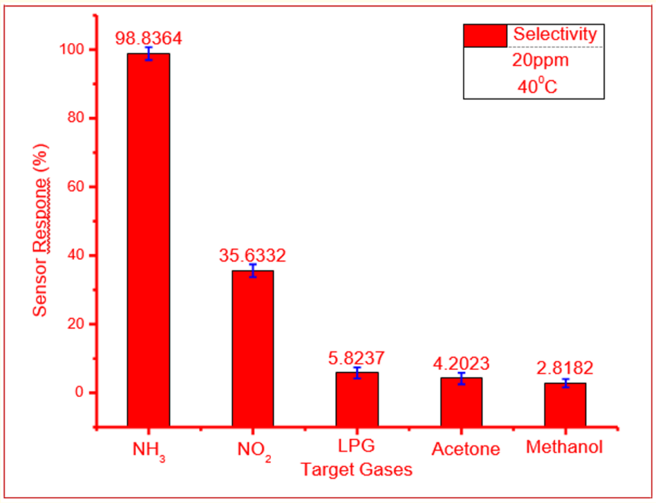

Figure 15: Selectivity of SWCNT sensor towards different gases at 20ppm.

The best sensor response (90-99%) was observed for NH3 and 5-40% sensor response was observed for other gases at constant concentration. This also indicates the poor interaction of other gases molecules with SWCNT sensor and hence the poor sensor response results.

Conclusion

High quality, rare dense and randomly oriented SWCNTs were successfully grown on Fe catalyst-based Si substrate by PECVD technique at an operating temperature of (650^{0}C) in the assist of plasma. The surface morphology and structural qualities of as grown SWCNTs were investigated by FESEM and HRTEM at a suitable magnification. Diameter distribution of as grown SWCNTs was calculated by Raman Spectroscopy at a wavelength of 633nm and it was in the range of 1-2nm. The effect on gas sensitivity with increasing target gas concentration and at different operating temperature was conducted.

The obtained sensor response at 40^0C, 100^0C and 200^0C was 25%, 20%, and 15% respectively showing a decreasing behavior with increasing temperature because adsorption prefers at low temperature. On the other hand, the obtained sensor response at 5ppm, 8ppm and 10ppm was 4%, 8% and 15% respectively showing almost a linear behavior with increasing target gas concentration. In addition of this, the SWCNT sensor had shown excellent performances towards repeatability, selectivity, resistance variation and long-term stability and is a step forward to monitor the environment at room temperature for security reasons. However, fast and complete recovery was obtained by thermal heating because desorption enhances with increasing temperature.

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