Fabrication and Characterization of SnO_2 Nanomaterial as CO_2 Gas Sensor

Faith C. Bancolo, Gil Nonato C. Santos, Reuben V. Quiroga

Abstract- Tin oxide nanomaterials were fabricated using the Horizontal Vapor Phase Growth (HVPG) Technique. The nanostructures formed were deposited on a silicon substrate to test the sensitivity of the material in a carbon dioxide gas environment operated at room temperature. The growth temperature was the same for all the various dwell times at 1200°C with a ramp rate of 10°C/minute. The surface topography, morphology and elemental composition of the synthesized SnO_2 nanomaterials were investigated using the SEM and EDX. The nanostructures were found to be sensitive to CO_2 gas exposure. This indicates that any nanostructures formed in the different dwell times were also found to be receptive in the gas exposure. Though remarkably all the different dwell times used have significantly responded to the CO_2 gas, it was noted that the sensor exhibited greatest response at the least dwell time which was grown at 4 hours. The best result sensitivity response has the average value of S = 1.142.

Keywords - Tin Oxide (SnO_2), Nanomaterials, Horizontal Vapor Phase Growth (HVPG) Technique, Carbon Dioxide (CO_2) gas sensor

1 INTRODUCTION

For over three decades, the development of thin and thick film techniques for metal oxide based gas sensors has enabled the fabrication of solid-state gas sensors due to their low dimension, price and power consumption. However, their working temperature and associated power supply requirement together with the long-term stability problems experienced have restricted the application of such devices in environmental, medical, domestic and industrial fields (Leghrib et al, 2009).

It is with this regard that nanotechnology and nanoscale sensing elements are believed to be of help. The nanostructure because of their peculiar characteristics and size effects often show some novel physical properties that are different from those of the bulk, and are of great interest both for fundamental studies and for potential nanodevice applications (Baratto et. al, 2005). Nanosensors play a major role in semiconductor processing, medical diagnosis, environmental sensing, and national security. Due to their higher sensitivity and high resolving power, nanosensors have potential applications in micro fluid detection, such as harmful gas molecules, individual bacteria and viruses etc. (Zhang, L. et al, 2007).

With the aim to control the air pollution and to detect toxic or smelling gases at low levels in the air, efforts towards the development of simple, inexpensive and reliable devices have been increased. Among a long list of materials, SnO_2 nanoparticles have been intensively studied for gas sensing applications owing to their sensitive conductivity changes upon gas reaction and adsorption. According to Marsal A. et al (2003), several oxides have been tested and SnO_2 is reported as one of the most reliable options for CO_2 detection.

The carbon dioxide gas is also considered as one of the primary greenhouse gases in the Earth's atmosphere. Due to human activities such as the combustion of fossil fuels and deforestation, the concentration of atmospheric carbon dioxide has tremendously increased since the beginning of the age of industrialization. This colorless gas when inhaled at low concentrations can be odorless but with much higher concentrations than the usual atmospheric levels, can produce a sharp and acidic odor. It can cause asphyxiation and irritation. Amounts above 5, 000 ppm are considered very unhealthy, and those above about 50,000 ppm are considered dangerous to animal life.

Surprisingly, given these facts, there were only a limited number of studies in the sensing materials of CO_2. Based from Xu, J. et al (2009), this was due to the high chemical stability of the gas. The high-power consumption for the bulky sensors of CO_2 is another significant issue that was addressed.

Because of these alarming effects in nature that human activities have created, it is with this reason that this study is conducted. A carbon dioxide gas sensor is needed to detect the increasing volume of such gas for environmental applications. It also aims to address low power consumption which was answered by operating the SnO_2 sensor in a room temperature.

Thus, from a method that was successfully adapted in synthesizing nanomaterials called the Horizontal Vapor Phase Growth (HPVG) technique, the researcher conducted a study on the SnO_2 nanomaterials as a carbon dioxide gas

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sensor. The effect of varying the dwell time on the sensor performance with growth temperature fixed at 1200°C was analyzed. Whereas, the surface topography and morphology of the SnO_2 nanomaterials was investigated through Scanning Electron Microscopy (SEM) and to identify the elemental composition of the grown nanostructures, the Energy Dispersive X-ray was used. The structured nanomaterials were deposited in a silicon substrate for the gas sensing application.

2 EXPERIMENTAL DETAILS

1. Synthesis of SnO_2 Nanomaterials

With the use of a blowtorch (mixture of oxygen and LPG), the quartz tube with an inner size diameter of 8.5 mm, outer size diameter of 11 mm, and length of 220 mm was sealed at one end. From its sealed end, a length of 80 mm was measured; at that marked, a dent was made in the quartz tube to hold the silicon substrate in place. The tubes were then cleansed for 1 hour in the Branson ultrasonic cleanser (sonicator) and allow it to dry in air.

A tin oxide (SnO_2) powder of high-purity (99.99%) amounting to 0.035 grams was then loaded onto the quartz tube, after which the substrate was dropped into it but also making sure that it would not pass through the dent made. It was connected to a Thermionics High-Vacuum System and sealed with an appropriate amount of LPG and oxygen gas to a pressure of 10^{-6} Torr. Fully sealed quartz tube was then detached from the vacuum.

The sealed sample tube was set in a Thermolyne Horizontal Tube Furnace programmed at 1200°C as its growth temperature with a varied dwell time of 4 hours, 6 hours, and 8 hours. A ramp rate of 10°C/minute was observed. . Moreover, to create a thermal gradient that would serve as the transport mechanism for the vapor during the deposition process, the completely sealed tubes was inserted halfway through the furnace in a horizontal position. After the set dwell time, the quartz tube was allowed to cool down to room temperature naturally before retrieving the SnO_2 nanomaterials.

2. Surface and Elemental Characterization

After the quartz tube had cooled down, it was covered with masking tape and then cracked. The retrieved fragment (silicon substrate) was sputtered with gold using a gold (Au) coater. The SnO_2 nanostructure deposits were then subjected for characterization through SEM and EDX. The Scanning Electron Microscopy (SEM) was used to determine the surface topography and morphology, Oxford EDX Link Isis was utilized to determine the elemental percentage composition of the material.

3. Experimental Set-up of CO_2 Gas Sensor

From the characterized SnO_2 nanomaterials grown by Horizontal Vapor Phase Growth Technique, SnO_2 nanomaterials were analyzed for CO_2 gas sensing. The silicon substrate was sputtered with gold to serve as the electrodes but leaving the middle line portion of the substrate unexposed to it. The nanomaterials which were not sputtered with gold will be the one to detect the CO_2 gas and shall measure the changes in its reading. The gap between the electrodes was 1 mm.

An improvised chamber was constructed using a 400 ml beaker (D = 77 mm) with cover. The top cover used had a diameter of approximately 90 mm. On the top cover, holes of approximately 2 mm were bored for the purpose of the different connecting wires. One hole (D = 5 mm) was connected to a CO_2 gas cylinder for the gas injection. The other wires were connected to a power supply and for the PascoScientific V-I Sensor. The retrieved substrate coated with gold on its sides served as the electrodes. When the chamber was completely sealed by covering it tightly with a parafilm, a carbon dioxide gas was infused into the system for five seconds and changes in its reading were noted.

In the experimental set-up, a Lodestar DC power supply was used as a voltage source. A 400 ml beaker with the top cover completely sealed by parafilm served as the chamber in this study. The source of the high concentration CO_2 gas was a commercially available gas cylinder commonly used in air guns. The constant resistance utilized in the circuit was a potentiometer set at its maximum resistance of 9.6 kΩ. Lastly, a Passport Interfaced PascoScientific V-I Sensor and DataStudio for data-acquisition was employed to measure the voltage response of the sensor. It is important to note that the experimental set-up was operated at room temperature during the data acquisition.

3 RESULTS AND DISCUSSION

1. Surface Morphology and Elemental Composition of SnO_2 Nanomaterials

The synthesis of SnO_2 nanomaterials using the Horizontal Vapor Phase Crystal Growth Technique had been a well-studied material as attested by the different researches in the past (Delos Reyes, R., et al, 2009; Sowl, S., et al, 2009; Santos, G. N. C., et al, 2008 and Ngo, et al, 2008). From the study of Delos Reyes, R., et al, 2009; it was

revealed that at a temperature of 1200°C and at a deposition time of 6 hours, respectively; various nanostructures had been observed. It was also found that most of the deposited nanomaterials were grown in zone 2 of the quartz tube. Therefore, the silicon substrate was placed in this zone area.

The following images are representations of structured nanomaterials grown at different dwell times: 4 hours, 6 hours and 8 hours.

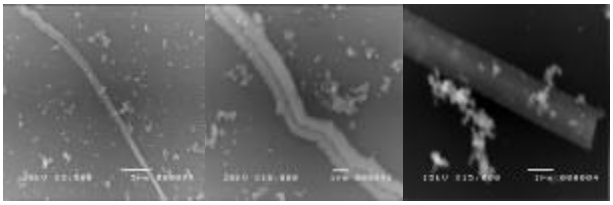

Figure 1. Samples grown at 1200°C for 8 hours.

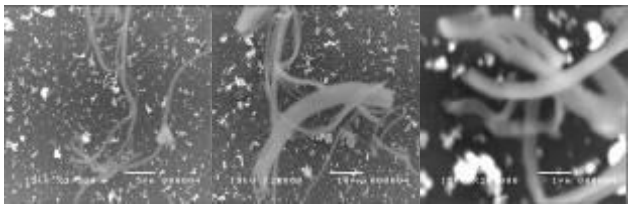

Figure 2. Samples grown at 1200°C for 6 hours.

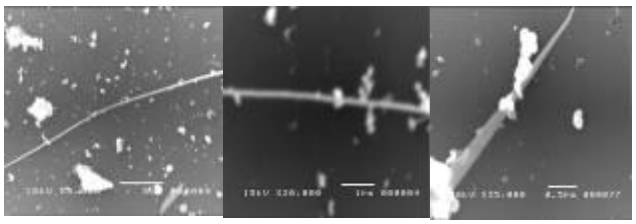

Figure 3. Samples grown at 1200°C for 4 hours.

From the representations of micrographs, various sizes of nanostructures were formed. Nanostructures grown in the silicon substrate for 8 hours of dwell time were thicker as compared to the nanostructures formed in 6 hours and 4 hours. The grown nanostructures in the 4 hours time showed smallest sizes and amount in the substrate. Interestingly enough, this verified the study conducted by Ngo, V. T., et. al (2008) where different geometric structures were grown at the 1200°C range. It was noted that as the growth time increases, the amount and sizes of the nanomaterials also increases. Since growth time is just the sum of the dwell time and ramp time, thus, an increase in the dwell time also increases the growth time.

The Energy Dispersive X-ray (EDX) analysis result also showed that there was a deposition of SnO_2 nanomaterials on the silicon substrate used for the CO_2 sensor as presented in figure 4.

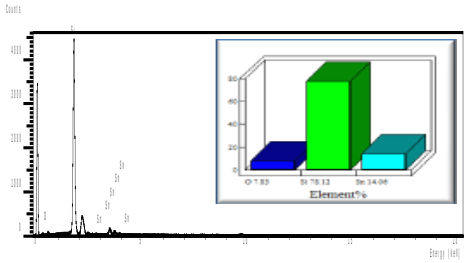

Figure 4. Representations of Energy Dispersive X-ray Spectrum and Elemental composition of SnO_2 nanomaterials

2. Gas Sensing Application

The electrical voltage of the sensor (voltage-time graph) was used to measure the detection of the CO_2 gas in this study. Any change in its voltage reading signifies a change in its resistance. The three silicon substrates grown at 1200°C but with different dwell times namely: 4 hours, 6 hours, and 8 hours were exposed to a target gas: CO_2. In the following experiments, a 5-second discharge time from the CO_2 gas cylinder had been utilized and a constant resistor was connected in parallel for the computations of sensor response since real time data in voltage was utilized.

Any variation in voltage reading across the parallel network corresponds to the change in the equivalent resistance. Since one of the resistances was constant, the change could be attributed to the sensor response. The sensitivity of the SnO_2 nanomaterial sensor was determined by calculating the ratio between its resistances before and after CO_2 gas exposure.

Table 1: Voltage readings and Sensor response for the sensor grown at 4 h dwell time

Initial Voltage	Final Voltage (PEAK)	$S = R_{gas}/ R_{air}$
9.33 V	9.60 V	1.145
9.33 V	9.59V	1.139

Table 2: Voltage readings and Sensor response for the sensor grown at 6 h dwell time

Initial Voltage	Final Voltage (PEAK)	$S = R_{gas}/ R_{air}$
6.55 V	6.72 V	1.028
6.55 V	6.75 V	1.033

Table 3: Voltage readings and Sensor response for the sensor grown at 8 h dwell time

Initial Voltage	Final Voltage (PEAK)	$S = R_{gas}/ R_{air}$
9.97 V	10.05 V	1.009
9.97 V	10.05 V	1.009

From the gathered results, it can be observed that before the exposure of the SnO_2 nanomaterial sensor to CO_2 gas, a constant voltage reading was obtained over time. However, upon gas injection an increase in voltage became evident signifying the sensitivity of the SnO_2 nanomaterial sensor to CO_2 gas. The change in voltage across the parallel connection corresponds to the change in the total resistance of the network. Since one of the loads has a constant resistance, the change in the equivalent resistance was attributed to the change in the resistance of the sensor.

In general, reducing gases increase the conductivity of the SnO_2 gas sensing material while the opposite is observed for oxidizing gases. In this study, the carbon dioxide gas acted as an oxidizing agent that caused the increase in the electrical resistance of the sensor (Batzill M. and Diebold U.,2005) signified by the increase in voltage reading. Adsorbed negatively charged oxygen species were considered to be responsible for this phenomenon. The negative charge trapped in these oxygen species caused an upward band bending on the SnO_2 nanomaterial thus increasing its resistance compared to the flat band situation before CO_2 gas exposure (Batzill M. and Diebold U.,2005).

Table 4: Average Sensitivity to the CO_2 gas of the

DWELL TIME (hours)	Average Sensitivity to the CO_2 gas
4	1.142
6	1.031
8	1.009

Table 5 reveals the average sensitivity of the nanomaterials grown at different dwell times. It shows that for nanostructures grown in 4 hours, a greatest response to the CO_2 gas upon exposure has been achieved as compared to the nanostructures grown at 6 hours and 8 hours. This suggests that in this study, the smallest dwell time for the synthesis of SnO_2 nanomaterials as gas sensor is highly sensitive to the CO_2 gas. From the study of Ngo, V. T., et al (2008), it was concluded that as the growth time increases, the amount and size of the nanomaterials also increases for a given growth temperature. Therefore, shorter dwell time produced SnO_2 nanomaterials with smaller dimensions. Lower dimensionality of the nanostructured SnO_2 resulted to an increase in the interfacial surface area of the nanomaterials. The increased interfacial surface area of the SnO_2 nanomaterials utilized as sensors made the exposed surfaces to CO_2 larger as the dwell time decreases, making them more sensitive to CO_2 gas. Though the other two dwell times can still detect the gas, it only proves that SnO_2 nanomaterials are capable and responsive to sense a carbon dioxide gas even if it is operated at room temperature.

4 CONCLUSION

The present work further verifies the effectiveness of Horizontal Vapor Phase Growth (HVPG) technique in growing nanomaterials particularly SnO_2. The SnO_2 nanomaterials grown in the silicon substrate using the Horizontal Vapor Phase Growth Technique were used for the gas sensing application operated at room temperature.

The nanostructures grown in the silicon substrate were subjected into the test chamber and upon the incorporation of the carbon dioxide gas; a change in its voltage reading was noted. The electrical voltage responded significantly indicating that resistance of the sensor has changed. The voltage-time graph showed that a change in its voltage is related to the change in its resistance. This change in resistance indicated that the electrical conductivity of the SnO_2 nanomaterial was altered by the adsorbed negatively charged oxygen species from the CO_2 gas. Hence, the nanostructures deposited in the seed were found to be sensitive upon CO_2 gas exposure. This indicates further that any SnO_2 nanostructures formed, as exhibited in the different dwell times, were also found to be receptive in the CO_2 gas exposure.

Though remarkably all the different dwell times used have significantly responded to the CO_2 gas, it was noted that the sensor exhibited greatest response at the smallest dwell time which was 4 hours. Based from the computations, the best result sensitivity response has the average value of $S = 1.142$. This suggests that in this study, the smallest dwell time for the synthesis of SnO_2 nanomaterials as gas sensor is highly sensitive to the CO_2 gas. This could be attributed to the fact that shorter dwell time produced SnO_2 nanomaterials with smaller dimensions (Ngo, V. T., et al 2008). The lower dimensionality of the nanostructured SnO_2 caused an increase in the interfacial surface area. The increased

interfacial surface area of the SnO_2 nanomaterials utilized as sensors increased the exposed area to the CO_2 gas, resulting to the highest sensitivity of the gas sensor.

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