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TiO_2 Nanostructured Material-based Gas Sensor for Detecting Oxygen and LPG Gases

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Abstract

This Paper investigates the use of TiO_2 nanowires and nanotetrapods on glass and silicon substrates for sensing combustible gases such as LPG (Liquid Petroleum Gas) and Oxygen. The growth of Titanium dioxide (TiO_2) nanowires/nanorods/nanotetrapods was investigated using both physical and chemical methods. In thermal evaporation method, both with and without vacuum conditions, was employed to synthesize various TiO_2 nanostructures. A vacuum coating machine was utilized to create uniform TiO_2 thin films, which were subsequently employed in both thermal evaporation methods and chemical methods for the growth of TiO_2 nanowires. For the thermal evaporation method without vacuum, Thermal Gravimetric Analyzer equipment was used to facilitate the growth of TiO_2 nanowires on TiO_2-coated silicon thin films. The impact of using microwave radiation for heating and use of capping agent during the chemical synthesis of TiO_2 nanowires was studied in detail. The produced samples were characterized at various stages for UV absorption spectroscopy, particle sizing, structural and morphological analysis. Under structural analysis, XRD of TiO_2 nanowires/nanotetrapods samples on glass were obtained and the size of TiO_2 nanostructure was calculated using Scherrer's formula. Based on these properties, TiO_2 nanowires /nanotetrapods samples were extensively studied for different gas sensing applications. The gas sensing experiments were conducted using the TiO_2 nanowires and the TiO_2 nanowires/nanotetrapods samples for varying gas volume concentrations.

Keywords: Titanium dioxide (TiO_2) nanowires, Liquid Petroleum Gas (LPG), Recovery response, Sensitivity, Gas sensor applications

INTRODUCTION

One of the main challenges in detecting oxygen and LPG gases is ensuring that the sensor is both sensitive and selective to these specific gases. Additionally, other key challenges include factors like temperature and humidity during measurements, long-term stability, and the recovery time after exposure to high gas concentrations [1]. Timely detection of LPG, a flammable gas, helps prevent explosions, while oxygen monitoring in confined spaces is essential to avoid dangerous depletion for safety. Controlling LPG leaks is vital for minimizing environmental damage, and monitoring oxygen levels in combustion processes enhances efficiency and reduces emissions. For gas sensing applications, Titanium dioxide is a suitable candidate to replace the toxic and expensive materials like SnO_2 commonly used in gas sensors [2,3] TiO_2 is particularly suitable for gas sensors due to its properties such as resistivity control over a wide range, high electrochemical stability, non-toxicity, and abundant availability in nature [4,5]. TiO_2 gas sensors have been fabricated using powders, pellets, thick, and thin films. However, thin films are especially suitable for such sensors, as the gas sensing properties of metal oxides are related to the material surface, and gases are always adsorbed and

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react with the surface [6]. Thin films of TiO_2 are expected to exhibit a high degree of gas sensitivity and selectivity [7,8], as the sensing mechanism involves chemisorption followed by charge transfer at the surface, leading to a change in the resistance of the sensor element [9-11]. Furthermore, the gas sensing response depends on the surface area of the sensing materials. Therefore, the use of metal-oxide semiconductor sensors with nanostructures, such as TiO_2 nanowires/nanotetrapods, which have a higher surface area, can provide higher sensitivity compared to TiO_2 nanoparticles, as the interaction between the adsorbed gases and the sensor surface will be stronger for the former. TiO_2 nanostructured sensors are also well-suited for integration into advanced wireless technologies. The application of TiO_2 nanostructure-based sensors with other technologies, such as wireless sensor networks (WSNs) or smart systems has been discussed by Lee et. al [12]. The fabrication process outlined in the paper uses both physical and chemical methods to grow TiO_2 nanowires/nanotetrapods on glass and silicon substrates. The combination of vacuum and non-vacuum techniques allows for potential scalability in large-scale production. The scalability of this process will depend on optimizing the efficiency and consistency of methods for large-area deposition and nanostructure growth. If these methods are refined for mass production, long-term costs could decrease. Achieving uniformity in the growth of nanowires and nanotetrapods at scale remains a challenge. While TiO_2 is abundant and inexpensive, making TiO_2-based sensors more cost-effective than those using rare or toxic materials like SnO_2, the cost of fabrication methods (such as vacuum deposition or microwave-assisted synthesis) may increase overall production costs [13].

In our study, we explored the use of TiO_2 nanowires/nanotetrapods on glass and silicon substrates to detect combustible gases such as Liquid Petroleum Gas (LPG) and Oxygen.

METHODOLOGY

Thermal evaporation methods with and without vacuum conditions were used to synthesize different TiO_2 nanostructures. A vacuum coating machine was utilized to produce uniform TiO_2 thin films, which were later employed in both thermal evaporation and chemical methods for growing TiO_2 nanowires. Using chemical route, in first synthesis method the TiO_2 nanowires/nanorods were grown on silicon wafer by a method as proposed by Lori E. Greene et.al. [10].

The second synthesis method was repetition of first synthesis method with use of RF-Microwave radiations for heating in place of conventional heating. Third synthesis method was repetition of second synthesis method with addition of TEA(Triethanolamine) as capping agent in first step of synthesis process. Morphological studies using SEM images showed the growth of TiO_2 nanopowders using the first synthesis method and TiO_2 nanotetrapods and nanopowders using the second synthesis method. The formation of nanotetrapods was attributed to high titanium vapor pressure and oxygen concentration [14]. The TiO_2 nanotetrapods have diameters ranging from 140-190nm and lengths of about 750nm.

The experimental setup is illustrated in Figure 1. A sample, in the form of a thin film of TiO_2 nanowires/nanotetrapods on a glass, was placed on a heater chamber plate inside an airtight glass dome with a free space volume of 35 liters. The heater plate temperature could be controlled from room temperature up to nearly 550ºC using a copper-constantan thermocouple feedback temperature controller. The two-probe DC measurement technique was employed to measure the electrical resistance in air and in the presence of the target gas. For electrical measurements, silver paste contacts (1.5mm) separated by a 4mm gap were used to form ohmic contacts on the TiO_2 nanostructure sample. A DC regulated power supply source provided a constant voltage of 5V, and a programmable digital multimeter measured the current output in the range of milliamperes or nanoamperes. A specific quantity of the target gas was injected into the airtight chamber through a rotameter (volume measurement unit). This setup was extensively used for sensing LPG and oxygen. The gas sensing setup was used to study two response variations: (a) The variation in sensitivity response as a function of temperature. (b) The sensing and recovery responses.

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RESULTS

UV Absorption Spectroscopy and Particle Sizing Analysis

The UV absorption of TiO_2 colloidal samples prepared using chemical synthesis method resulted in band edge peak wavelengths (λ) of 400nm, 375nm with average particle sizes of 200nm and 175nm respectively (Figure 2). The results clearly indicate a decrease in particle size with the use of microwave radiation for heating in the second synthesis method. Additionally, using microwave radiation for heating reduced the synthesis time by half, from 2 hours to 1 hour, for the growth of TiO_2 nanoparticles.

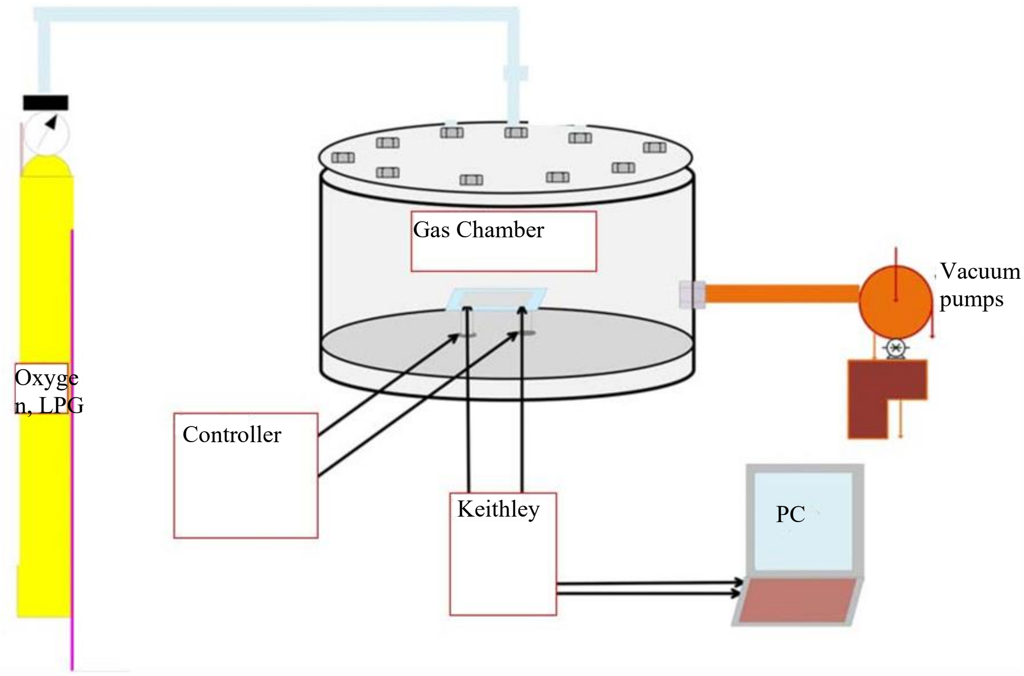

Figure 1. Gas Sensing Setup.

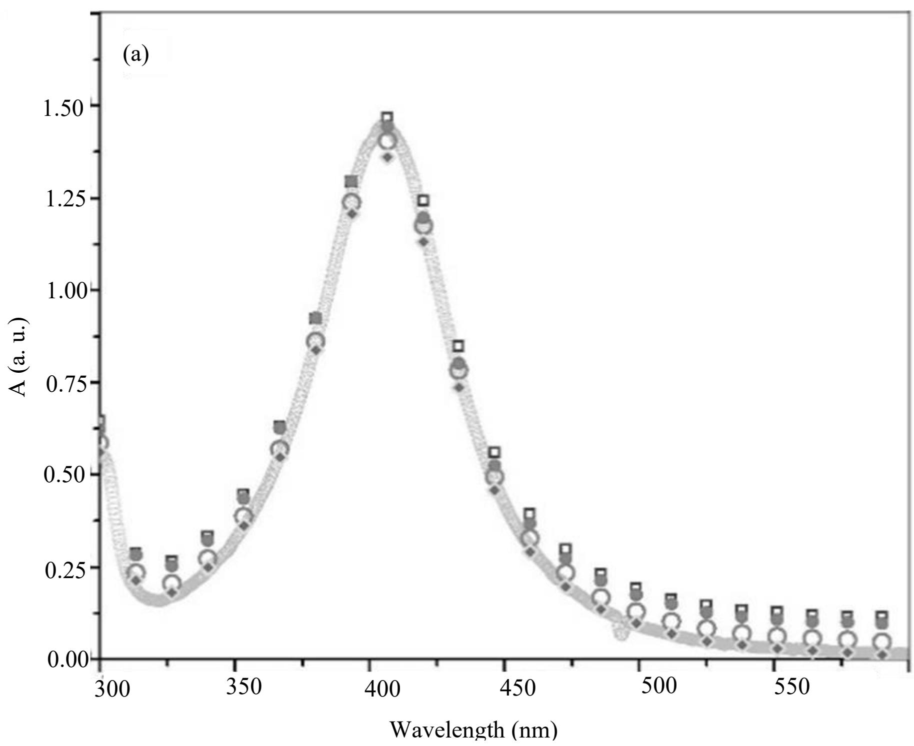

Figure 2. UV-Visible Data for Prepared TiO_2 Nanoparticles.

Structural and Morphological Analysis

Under structural analysis, XRD of TiO_2 nanowires/nanotetrapods samples on glass were obtained (Figure 3) and the size of TiO_2 nanostructure was calculated using Scherrer's formula. The size of the synthesized TiO_2 nanowires/nanorods/nanotertrapods were found to be between (10nm – 80nm), (35nm- 100nm) for the first and second chemical synthesis methods respectively. The hexagonal wurtzite structure of the TiO_2 nanostructure sample was also confirmed from XRD. The morophology analysis using SEM confirmed that TiO_2 nanowires/nanorods with diameter 150-300nm and length 1-3µm were synthesized using first chemical method, TiO_2 nanowires with diameter 30-150nm and length 0.5-1µm were synthesized using second chemical method (Figure 4).

OXYGEN DETECTION

Oxygen Sensing Mechanism

Oxygen vacancies on TiO_2 surfaces are both electrically and chemically active. When oxygen is introduced, it gets adsorbed onto the surface of the TiO_2 nanostructured thin film, along with the already adsorbed oxygen from the air. These adsorbed O_2 molecules are charge-accepting, depleting electrons from the conduction bands. This results in an increase in resistivity. The higher the O_2 concentration, the greater the depletion, leading to increased resistivity. Therefore, the sensitivity response increases with the volume concentration of O_2 due to the higher resistivity of TiO_2 nanostructured samples.

Sensitivity to Oxygen Gas as a Function of Temperature

The oxygen sensitivity response as a function of temperature was repeated using the different thin film samples: one with TiO_2 nanowires (sample I on glass) and the other with TiO_2 nanotetrapods and nanowires (sample II on glass). In our experiment, before exposing the TiO_2 nanostructure samples to O_2, they were allowed to equilibrate in open air at the operating temperature for 35-40 minutes. The stabilized resistance was recorded as the resistance in air (Ra). The response was measured over a temperature range from 303K (30°C) to 698K (425°C) at three different O_2 concentrations: 50ml, 75ml, and 100ml, corresponding to 0.2%, 0.3%, and 0.4% volume concentration, respectively.

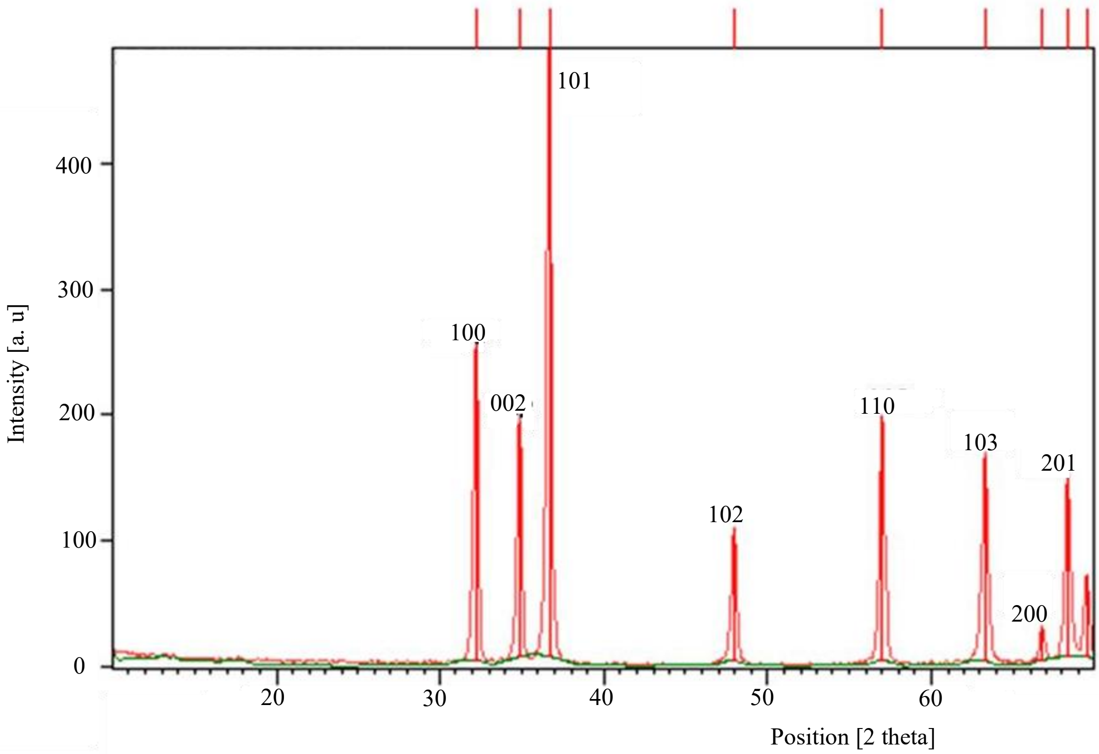

Figure 3. XRD of TiO_2 Nanowires/Nanotetrapods Samples on Glass Substrate.

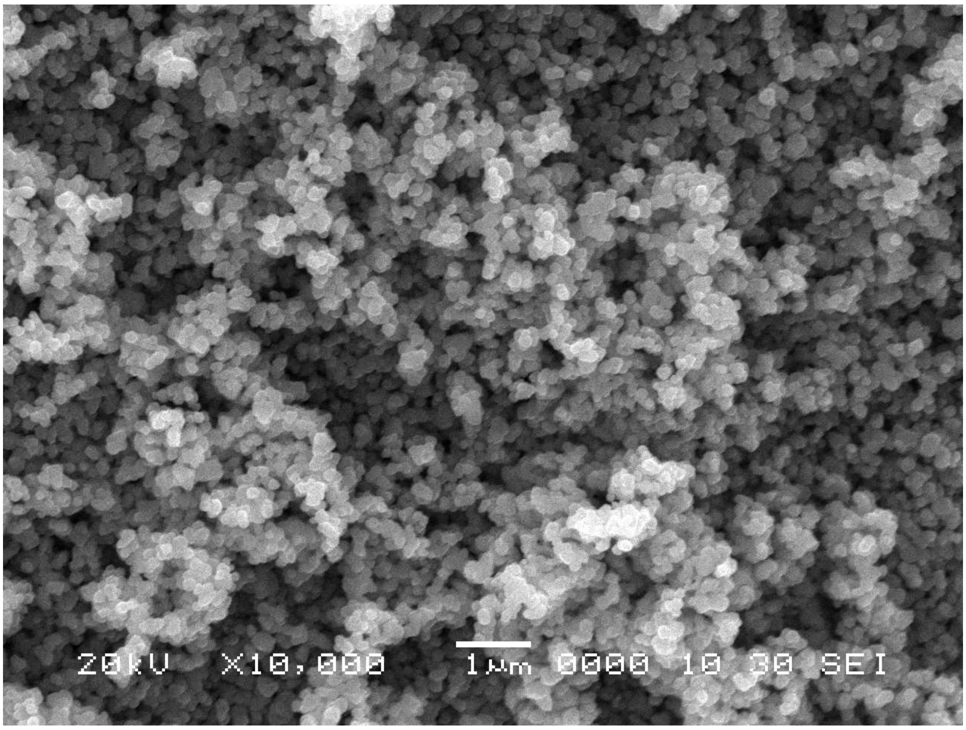

Figure 4. SEM Image of TiO_2 Nanorods Vertically Grown on the Substrate.

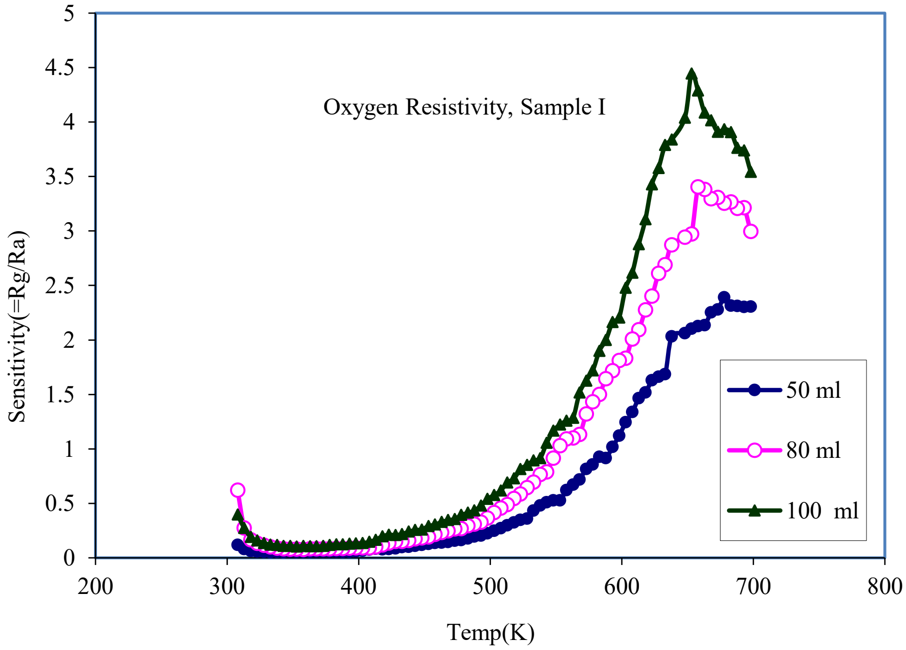

Figure 5. Oxygen Gas, Sensitivity (Rg/Ra) Response with Temperature, TiO_2 Nanowires Sample I.

The sensitivity response plots for the two different samples are shown in Figure 5 and 6, demonstrating that the peak sensitivity response for both TiO_2 nanowires (glass sample I) and TiO_2 nanowires/nanotetrapods (glass sample II) increases with the O_2 gas concentration. From Figure 2 for TiO_2 nanowires (glass sample I), the sensitivity response was found to be maximum at temperature values of 678K (405°C), 658K (385°C), and 653K (380°C) for 0.2%, 0.3%, and 0.4% volume concentrations of O_2, respectively. Meanwhile, Figure 3 shows that for TiO_2 nanowires/nanotetrapods (glass sample II), the sensitivity response was maximum and stabilized around the temperature values of 468K (195°C), 543K (270°C), and 548K (275°C) for 0.2%, 0.3%, and 0.4% volume concentrations of O_2, respectively.

Oxygen Gas Sensing and Recovery Response

The oxygen sensing and recovery response was repeated using two thin film samples: one with TiO_2 nanowires (sample I on glass) and the other with TiO_2 nanotetrapods/nanowires (sample II on glass). The experimental setup shown in Fig. 1 was used for each sample separately. In our experiment, before exposing the TiO_2 nanostructure sample to O_2, it was allowed to equilibrate in open air at the operating temperature for 35-40 minutes, and the stabilized resistance was recorded as the resistance in air (Ra). The response of thin films to 100ml (i.e., 0.4% volume concentration) O_2 exposure for 30 minutes in an airtight chamber and subsequent recovery in air was measured at different temperatures: 100°C, 150°C, 200°C, and 250°C as shown in Figure 7 and 8.

The percentage reduction in resistance and recovery time at different temperatures after 30 minutes of O_2 exposure is shown in comparison Table-1. At 250°C, the percentage reduction in resistance (S%) for both samples ranges between 75% and 85%, indicating a good O_2 sensing response with the shortest recovery time of 0.6 to 0.85 minutes compared to other operating temperatures for both samples. Therefore, 250°C can be considered the ideal operating temperature for oxygen detection using TiO_2 nanostructure samples to achieve high sensitivity and minimal recovery time.

Table 1. For Oxygen Gas, TiO_2 Nanowires and Nanowires/Nanotetrapods Samples Comparison Table.

Temperature (°C)	**TiO_2 Nanowires (Sample I)**		**TiO_2 Nanowires & Nanotetrapods (Sample II)**	
	Percentage reduction of resistance (S%)	***Time of recovery (min)***	***Percentage reduction of resistance (S%)***	***Time of recovery (min)***
100	55	13.5	73.5	15.5
150	72	5.65	65.7	4.5
200	86	2.75	86.4	1.4
250	75	0.85	84.3	0.6
300	78	0.95	--	--

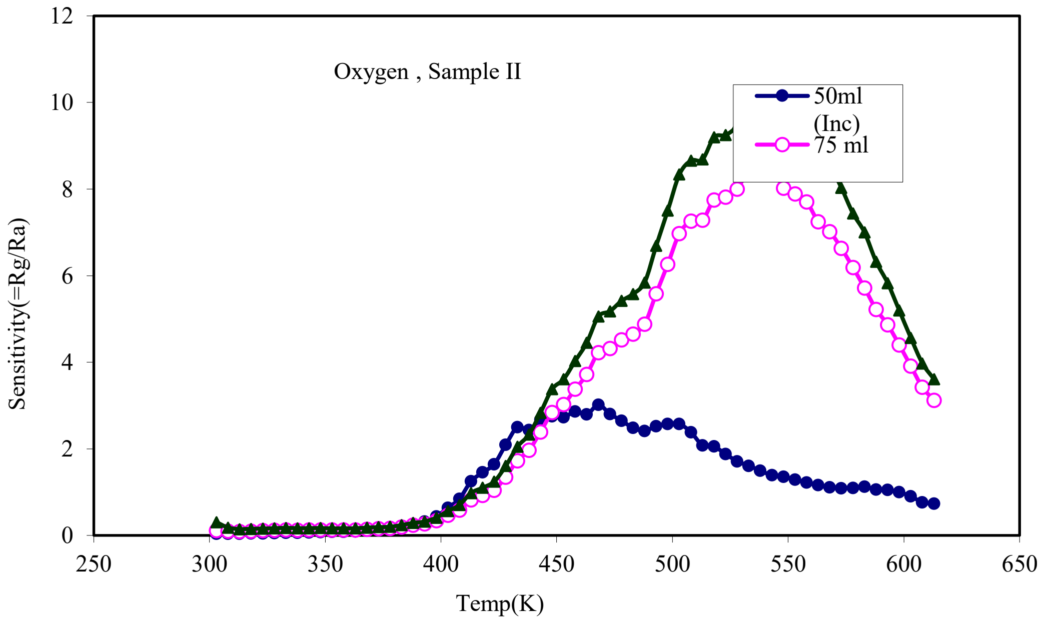

Figure 6. Oxygen Gas, Sensitivity (Rg/Ra) Response with Temperature, TiO_2 Nanowires/Nanotetrapods Sample II.

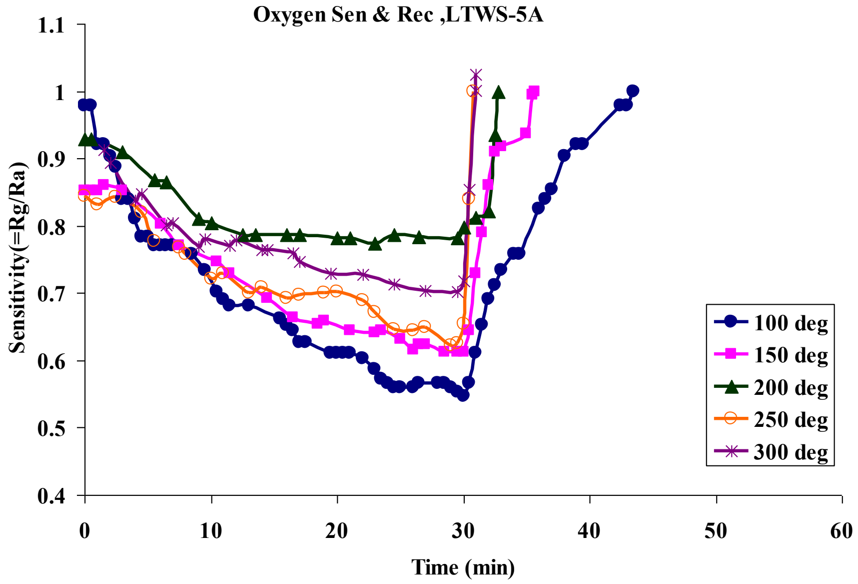

Figure 7. Oxygen Gas, Sensing and Recovery Response, TiO_2 Nanowires Sample I

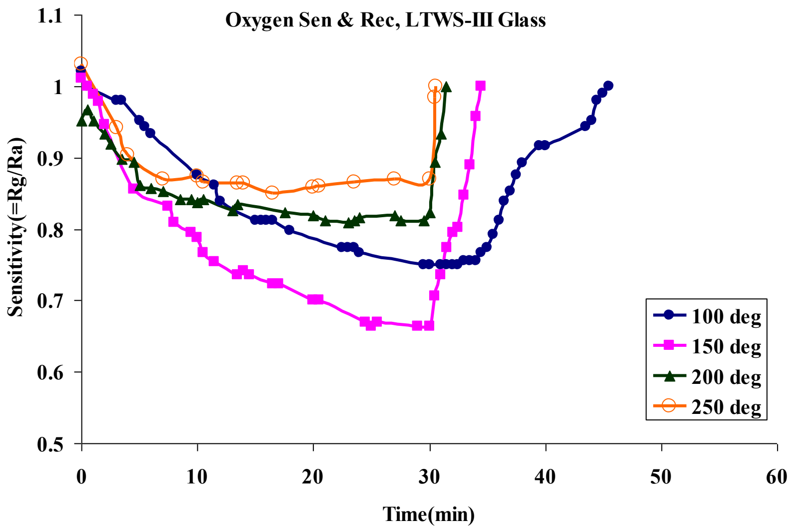

Figure 8. Oxygen Gas, Sensing and Recovery Response, TiO_2 Nanowires/Nanotetrapods Sample II.

LPG and Oxygen Sensing Response and Recovery Time Analysis

For studying gas sensing applications of TiO_2 nanowires/nanotetrapods, LPG and Oxygen gases were used as target gases. The gas sensing experiments was carried out using TiO_2 nanowires sample I and TiO_2 nanowires/nanotetrapods samples II for different gas % volume concentration. As LPG is a reducing agent, the sensitivity response peak decreased with increase in gas concentration and temperature. On the other hand, oxygen being an oxidizing agent, the sensitivity response increased with both temperature and gas concentration. The sensing and recovery response for LPG and Oxygen was carried, using fixed 0.2%volume gas concentration and at fixed temperatures (100°C, 150°C, 200°C, 250°C and 300°C). It showed that for both the TiO_2 nanowires samples the percentage reduction in resistance (S%) and recovery time reduced with increase in operation temperature. The comparison of data for both the samples showed, 250°C is an ideal operation temperature to achieve good percentage reduction of resistance (S%) and least recovery time for both the gases. It was also noticed that at operation temperature of 300°C, the recovery time slightly increases compared to the values at 250°C for both samples. Further comparison of results at 250°C between both the samples recommends TiO_2 nanowires/nanotetrapods sample II for sensing LPG and oxygen gas, as it has better percentage reduction in resistance (S%) and least recovery time.

Temperature significantly affects the gas-sensing performance of TiO_2 nanowires and nanotetrapods. Higher temperatures typically enhance sensitivity to both oxygen and LPG by increasing gas molecule adsorption and improving surface reaction rates [15]. The optimal temperature for oxygen detection was around 250°C, where sensitivity and recovery time were best, highlighting temperature's crucial role in controlling resistance changes during gas exposure. While the paper does not specifically address other factors like humidity or the presence of interfering gases, it is important to consider these for the sensor's practical, real-world applications.

CONCLUSIONS

In this paper, both physical and chemical synthesis methods were explored for the growth of TiO_2 nanowires. Non-uniform growth of TiO_2 nanotetrapods/nanopowders on silicon (100) substrates was achieved using physical methods. The effect of using microwave radiation for heating and a capping agent during the chemical synthesis of TiO_2 nanowires was studied in detail. Additionally, TiO_2 nanowires and TiO_2 nanowires/nanotetrapods samples were studied for their applications in sensing gases (LPG, oxygen). These samples exhibited good sensing sensitivity and low recovery time for both gases (LPG, oxygen).

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