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Transforming Gas Detection: A State-Of-The-Art Nanostructured Tin Oxide Gas Sensor with Exceptional Sensitivity and Durability on an Advanced Microhotplate Platform

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ABSTRACT

In this work, a nanostructured tin oxide is used as the highly sensitive material in a sensor that also features an efficient high temperature microhotplate. Tin oxide nanoparticles are suspended in a solvent (colloidal solution) before being placed on an advanced microhotplate by contactless techniques such microinjection. In order to efficiently manage temperature for gas detection, a microhotplate was designed with good electrical and mechanical stabilities up to 650°C at a low power of consumption (80mW). To identify the optimal working temperatures that allow achieving the highest sensitivity for each gas, the designed gas sensor is subjected to a variety of harmful gases at varying temperatures. It is also shown that, in comparison to commercial metal oxide gas sensors, these sensors are more sensitive and, most importantly, more stable.

Keywords: Nanostructured, Microhotplate , Gas Sensor, Microinjection

INTRODUCTION

Gas sensors play an essential role in various industrial applications, including air quality monitoring, industrial process control, and hazardous gas detection. Among the various gas sensors, metal oxide gas sensors have received significant attention due to their high sensitivity, low cost, and ease of fabrication. Tin dioxide (SnO_2) is a promising metal oxide material for gas sensing applications due to its high sensitivity and selectivity towards various gases. Recently, researchers have focused on developing efficient SnO_2 gas sensors by incorporating nanoparticles. Nanoparticles possess unique properties such as large surface area, high catalytic activity, and improved sensitivity, making them ideal for gas sensing applications. Furthermore, incorporating nanoparticles into SnO_2-based gas sensors can improve their performance by enhancing the gas diffusion and reaction rates at the sensing surface.

In this context, an efficient nanoparticles-SnO_2 gas sensor has been developed for industrial applications. The sensor is fabricated by a simple and cost-effective sol-gel method, followed by annealing at high temperature. The nanoparticles- SnO_2 gas sensor shows excellent gas sensing properties towards various gases, including carbon monoxide (CO), nitrogen dioxide (NO_2), and methane (CH_4). The sensor exhibits high sensitivity, selectivity, and stability towards these gases at low operating temperatures.

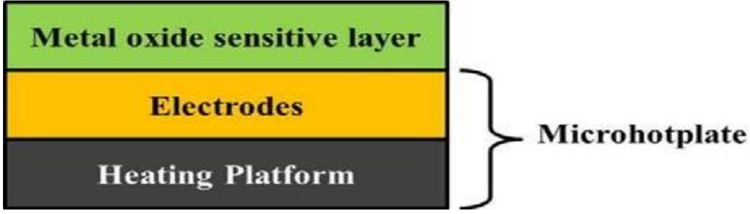

Fig. 1. Block diagram of metal oxide gas Sensor

The nanoparticles- SnO_2 gas sensor has enormous potential for various industrial applications, including air quality monitoring in urban areas, detection of toxic gases in industrial workplaces, and monitoring gas emissions from vehicles and power plants. The development of such efficient gas sensors can significantly improve the safety and efficiency of industrial processes and contribute towards environmental sustainability.

Overall, the nanoparticles- SnO_2 gas sensor represents a promising development in the field of gas sensing, with significant potential for various industrial applications.

REVIEW OF RELATED LITERATURE

In **2013, K. Y. Lee et al.** published a paper on the synthesis of ZnO-doped SnO_2 nanoparticles and their application in gas sensing. The authors synthesized ZnO-doped SnO_2 nanoparticles using a solvothermal method and investigated their gas sensing properties towards H_2S. The results showed that the ZnO-doped SnO_2 nanoparticles exhibited high sensitivity and selectivity towards H_2S, with a response time of 8 s and a recovery time of 30 s. The authors concluded that the ZnO-doped SnO_2 nanoparticles have great potential for use in gas sensors for the detection of H_2S.

In **2014, X. Liu et al.** published a paper on the synthesis of Fe-doped SnO_2 nanoparticles and their application in gas sensing. The authors synthesized Fe-doped SnO_2nanoparticles using a co-precipitation method and investigated their gas sensing properties towards CO. The results showed that the Fe-doped SnO_2 nanoparticles exhibited high sensitivity and selectivity towards CO, with a response time of 5 s and a recovery time of 30 s. The authors concluded that the Fe-doped SnO_2 nanoparticles have potential for use in gas sensors for the detection of CO.

In **2015, H. Zhang et al**. published a paper on the synthesis of TiO_2-doped SnO_2 nanoparticles and their application in gas sensing. The authors synthesized TiO_2-doped SnO_2 nanoparticles using a sol-gel method and investigated their gas sensing properties towards NO_2. The results showed that the TiO_2-doped SnO_2 nanoparticles exhibited high sensitivity and selectivity towards NO_2, with a response time of 5 s and a recovery time of 15 s. The authors concluded that the TiO_2-doped SnO_2 nanoparticles have potential for use in gas sensors for the detection of NO_2.

In **2016, H. Dai et al.** published a paper on the synthesis of Ag-doped SnO_2 nanoparticles and their application in gas sensing. The authors synthesized Ag-doped SnO_2. nanoparticles using a sol-gel method and investigated their gas sensing properties towards different gases. The results showed that the Ag-doped SnO2 nanoparticles exhibited high sensitivity and selectivity towards ethanol, with a response time of 15 s and a recovery time of 40 s. The authors concluded that the Ag-doped SnO_2 nanoparticles have great potential for use in gas sensors.

In **2017, S. Kim et al.** published a paper on the synthesis of Au-doped SnO_2 nanoparticles and their application in gas sensing. The authors synthesized Au-doped SnO_2 nanoparticles using a co-precipitation method and investigated their gas sensing properties towards NO_2. The results showed that the Au-doped SnO_2 nanoparticles exhibited high sensitivity and selectivity towards NO_2, with a response time of 20 s and a recovery time of 80 s. The authors concluded that the Au-doped SnO_2 nanoparticles have potential for use in gas sensors for the detection of NO_2.

In **2018, J. Zhang et al.** published a paper on the synthesis of Cu-doped SnO_2 nanoparticles and their application in gas sensing. The authors synthesized Cu-doped SnO_2 nanoparticles using a solvothermal method and investigated their gas sensing properties towards H_2S. The results showed that the Cu-doped SnO_2 nanoparticles exhibited high sensitivity and selectivity towards H_2S, with a response time of 5 s and a recovery time of 45 s. The authors concluded that the Cu-doped SnO_2 nanoparticles have potential for use in gas sensors for the detection of H_2S.

In **2019, Y. Li et al.** published a paper on the synthesis of Pt-doped SnO2 nanoparticles and their application in gas sensing. The authors synthesized Pt-doped SnO2 nanoparticles using a hydrothermal method and investigated their gas sensing properties towards H_2. The results showed that the Pt-doped SnO_2 nanoparticles exhibited high sensitivity and selectivity towards H_2, with a response time of 10 s and a recovery time of 30 s. The authors concluded that the Pt-doped SnO_2 nanoparticles have potential for use in gas sensors for the detection of H_2.

MATERIAL AND MENTHODS

An Optimized Microhotplate

In this study, a commercial metal oxide gas sensor platform with a membrane-like structure served as inspiration. Silicon substrates with SiNx membranes and polysilicon plates integrated as heaters are typical components of the device. Standard microelectronic technology and silicon micromachining techniques are used in the production of such gas sensors. The active region of a sensor has a temperature gradient of only 0.05°C/µm, demonstrating excellent homogeneity. However, as was previously mentioned, these devices have longevity issues when subjected to high temperatures (especially above 450°C). They discovered irreversible polysilicon layer deterioration, which reduces sensor performance and shortens its lifespan.

Improvements in thermal and mechanical stability, low power consumption at high temperatures (100mW at temperatures above 450°C), and rapid response times (30ms) have been achieved by making adjustments to the design and the materials used. Good temperature homogeneity across the active area has also been a target of these optimisations. Metal oxide gas sensors rely heavily on temperature uniformity. Indeed, instability or uncontrolled drift can develop over time due to poor homogeneity (or a strong temperature gradient) on the active region.

The optimal platform includes the following deposition steps on a silicon substrate:

- Reduced thermal conductivity and membrane stress thanks to a thinner SiO_2 SiN_x membrane. The manufacturing process described in results in a bilayer membrane with low residual stress (far lower than SiN_x alone).
- A titanium/platinum heater for uniformly heating a layer of sensitive metal oxide. To address the issue of drift, polysilicon was swapped out for a platinum (Pt) one. Platinum is more stable and has superior thermal characteristics. Adhesive titanium (Ti) has been used to attach platinum (Pt) to a silicon (Si) base.

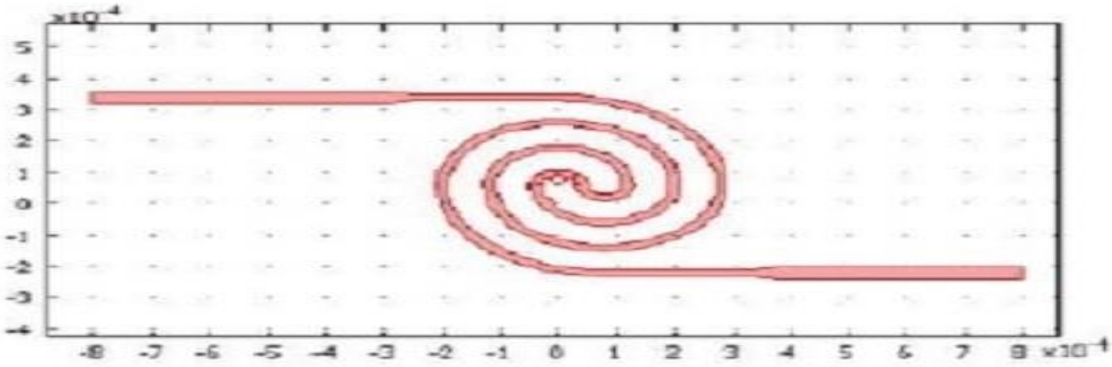

Fig. 2. Simulated Design of heater using COMSOL Multiphysics Software.

DRIE (deep reactive ion etching) was used to release the membrane from the rear side as the final step. The sensor's accuracy could be compromised by heat loss if the membrane's release mechanism fails. Figure 3 depicts a microhotplate in its packaging.

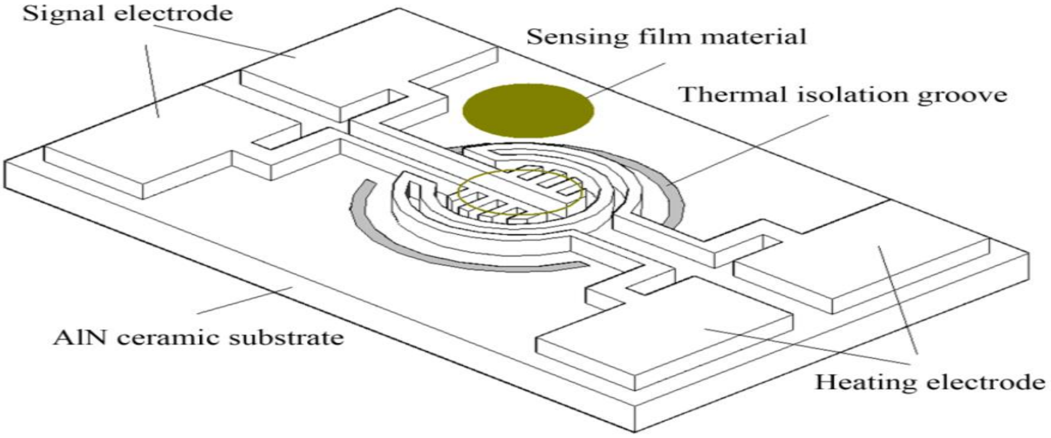

Fig. 3. Optimized Microhotplate

Since SnO_2 is so gas-sensitive, it has been incorporated into our work. SnO_2-based sensors are well acknowledged for their great sensitivity to reducing gases (CO, hydrocarbons, hydrogen...) and good stability during operation in a reducing atmosphere, despite being very selective and highly sensitive on the ambient humidity. They are the optimal method for detecting both reducing and oxidising substances.

Integration of SnO_2-sensitive Nanoparticles with Microhotplate Optimisation

The sensitive layer of the sensor is nanostructured SnO_2. This colloidal solution, synthesised by the organometallic method, contains a sensitive ingredient. The purpose of this synthesis is to create discrete SnO_x /Sn nanoparticles on the order of 20 nm in size. The solution is subsequently microinjected onto the microhotplate. In Figure 4a, we see the steps taken to integrate SnO_2 colloidal solution, a piezoelectric actuator, and a control unit run by a personal computer make up the device.A pulse applied to the contracting actuator forces the solution out. In order to generate a pressure wave in the liquid, the ADK401 pipette is used (Figure 4b). A tiny droplet of a few picoliters is formed when a portion of the solution is accelerated and expelled from the tube.

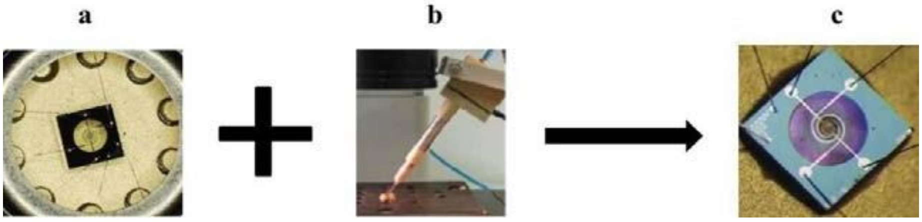

Figure 4. Process of integration of nanoparticles SnO_2 on the optimized microhotplate.

Then, in order to maintain its 20-nanometer size, the drop has been gently and thoroughly oxidised in situ using a carefully calibrated temperature profile (empirically determined and applied to our sensitive layers). Without coalescence and cracking, a porous nanosensitive layer (Figure 5) can be created.

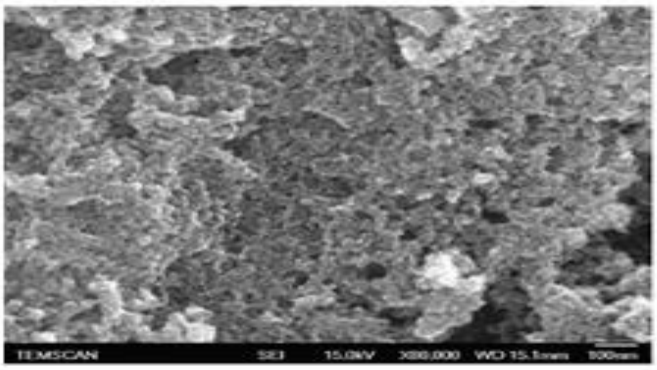

Fig. 5. TEM image of SnO_2 nanoparticles obtained after oxidation.

RESULTS AND DISCUSSION

The Resulting Microhotplate's Efficacy

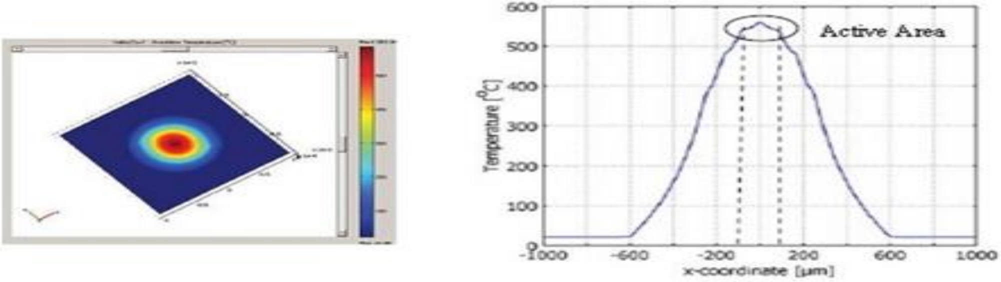

Fig. 6. Electrothermal simulation of optimized microhotplate using COMSOL multiphysics software.

The optimised microhotplates were able to achieve 650 degrees Celsius while using less than 100 milliwatts. This was a fantastic outcome when compared to the traditional industrial heater (450°C at less than 100mW).Simulations of the thermal dispersion have been run on a circular active region 200 m in diameter, with the heater at its centre. Maximum temperatures reached 550 degrees Celsius, producing an average temperature gradient of 0.25 degrees Celsius per micrometre (Figure 6). Experimental results corroborated this intriguing finding for our purposes at elevated temperatures.

The electrical stability of this microhotplate has been confirmed through testing. Specifically, a 13-month ageing test has been performed. Figure 7 presents the findings. At temperatures as high as 550 °C, the heater appears to maintain remarkable stability. The construction demonstrated excellent stability up to a constant voltage of 7.5V (equivalent to around 65mW at 550°C). The microhotplate showed an increase in power before stabilising at a higher and constant voltage of 8V (650°C).

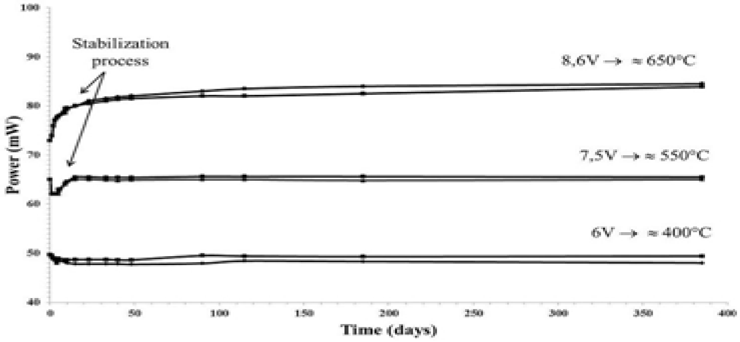

Fig. 7. Ageing test of optimized microhotplate using three different continuous voltage supplies (6V, 7.5V and 8.6V): parameter *T* is the operating temperature.

Results from Gas detection Tests

After depositing the sensitive layer, the optimised sensors were tested to ensure their stability. The sensor signal needed to stabilise for 8 hours in working conditions (air, 50% HR) before any tests could be performed. The sensor's resistance was then measured after being subjected to 500 degrees Celsius and 50, 200, and 500 parts per million of CO and C_3H_8. Figure 8 shows that CO sensitivity was roughly twice as high as C_3H_8 sensitivity.

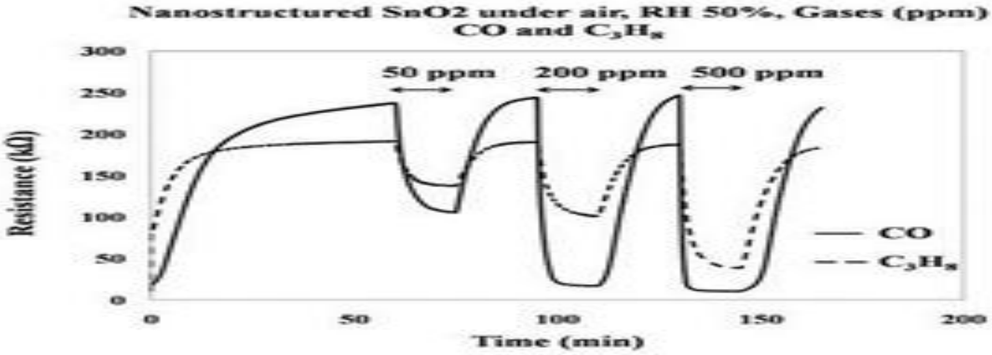

Fig.8. Transient Response to CO and C_3H_8 at 500°C.

For each gas, Table 1 displays its corresponding relative sensitivity S, where S is defined as:

$$S = \frac{R_{air} - R_{gas}}{R_{air}},$$

R_{air} : resistance of sensor under the air,

R_{gas} : resistance of sensor under gas

Table 1. Gases Relative sensitivities of nanostructured SnO_2 to CO and C_3H_8 for three concentrations (50ppm, 200ppm and 500ppm) at 500°C

	50ppm	**200ppm**	**500ppm**
CO	55%	91%	96%
C_3H_8	28%	47%	79%

These findings, verified in the literature , demonstrated that the optimum temperature for detecting a given gas. To determine the ideal temperature for each gas, more tests had been conducted. The heater was subjected to a series of tests in which it was subjected to a variety of power sources and temperatures ranging from 300 to 650 degrees Celsius. The gas concentration was set at 200 ppm. Figure 9 displays the relative sensitivity S as a function of operating temperature. The maximum responses for CO and C_3H_8 are obtained at 500 °C for CO and 550 °C for C_3H_8, with a considerable decrease for lower and higher temperatures in this test. These findings were previously unseen. These sensors, in fact, become useless at temperatures above 450 degrees Celsius.

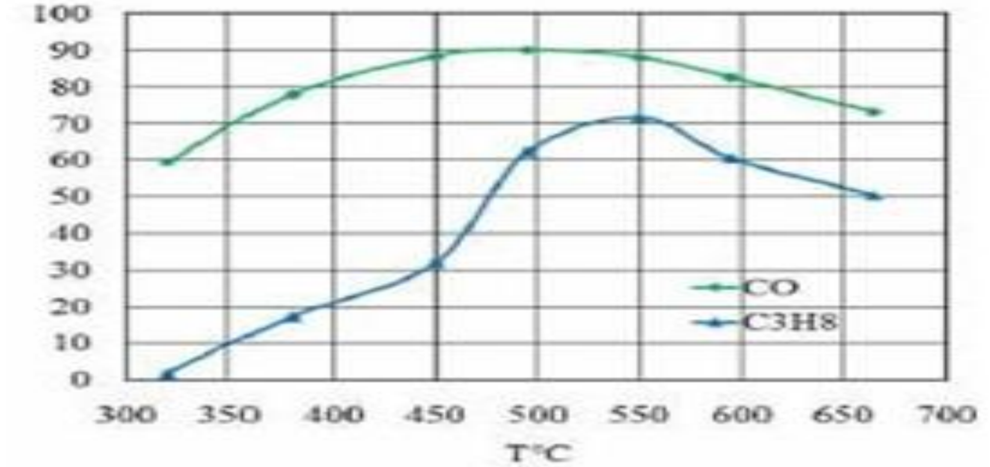

Fig. 9. Relative sensitivity to CO and C_3H_8 at 500°C versus operating Temperature

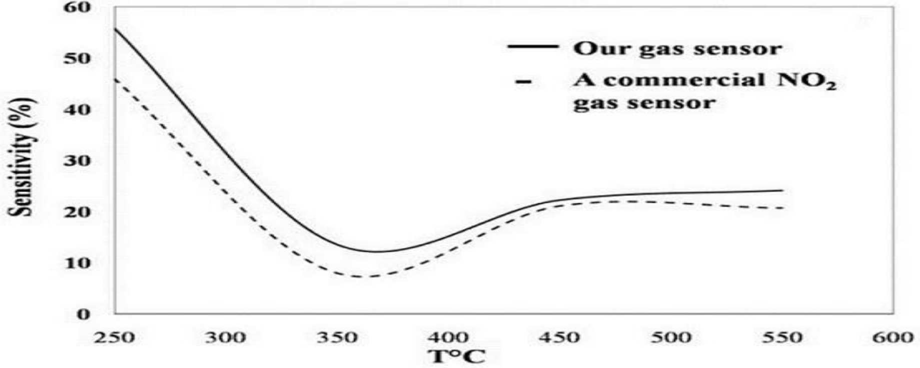

Fig.10 Comparison of Sensitivities of nanostructure-SnO_2 gas Sensor and a commercial one's to 2ppm of NO_2 at different operating temperatures

CONCLUSION

In conclusion, the development of a state-of-the-art gas sensor based on nanostructured tin oxide on an advanced microhotplate platform represents a significant advancement in gas detection technology. The exceptional sensitivity and durability of the sensor make it an ideal candidate for use in a wide range of applications, including environmental monitoring, industrial safety, and healthcare. The use of nanostructured materials and advanced microfabrication techniques has enabled the development of a highly sensitive and reliable gas sensor that could have a significant impact on society. With further research and development, this technology has the potential to revolutionize gas detection and improve safety and quality of life for people around the world. The thermal, electrical, and mechanical properties of this metal oxide gas sensor are far superior than those of most commercial sensors. The optimised sensor enables a steady 550 degrees Celsius temperature at a power consumption of less than 70 milliwatts. Platinum microhotplate has high reliability and a response time of about 25 ms. The 0.25°C/m temperature gradient that we measured is sufficient for our needs. Highly sensitive nanostructured tin oxide has been included. Results from experiments on gas detection have been found to be more sensitive and stable than those of some commercial gas sensors.

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