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ZNO THICK FILM GAS SENSOR FOR METHANE SENSING AT ROOM OPERATING TEMPERATURE

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ABSTRACT

Methane (CH_4) is a strong greenhouse gas and dangerous in industrial settings, so highly sensitive and reliable sensors are needed to detect it at low concentrations. In this study, ZnO-based thick-film gas sensors were fabricated using a screen-printing technique on Kapton substrates and evaluated for methane sensing at room temperature. The device consists of an interdigitated electrode (IDE) and a ZnO sensing layer, both deposited onto the Kapton substrate via screen printing. The prepared sensors were then tested with methane at 6700 ppm and 7500 ppm concentrations. Results show that both ZnO sensors have good linearity in I-V measurements meaning that they exhibit ohmic behavior. ZnO2 sensitivity was higher than ZnO1 probably due to some microstructural differences. Resistance changes during gas exposure were used as the sensor response.

KEYWORDS

Methane, ZnO Gas Sensor, Binder, Screen-Printing, I-V Characteristics.

1. INTRODUCTION

Methane (CH_4) is the simplest hydrocarbon and the primary component of natural gas. Methane's high energy content, relatively clean combustion, and abundance make it a valuable resource in both industrial and domestic settings. In climate impact assessments, methane is classified as a strong greenhouse gas, as its warming influence over a 100-year period is estimated to be about 28 times greater than carbon dioxide (CO_2) (Tsui etal, 2024). It is detected by gas sensors that operate on various principles, most commonly involving a change in the electrical or optical characteristics of the sensing material caused by the presence of the gas. Chemiresistive sensing is the most common method which measures the change in electrical resistance by the effusion of methane on the sensor surface (Goel et. al 2022). This change is typically noted by the variation of electrical conductivity in response to the interaction of electrons between the gas and the sensing material most commonly a metal oxide semiconductor like tin oxide (SnO_2) (Xu et. al 2016) or ZnO (Cai et. al 2023). Gas detectors, particularly methane sensors can be used in mining to check for possible explosive atmosphere for safety (He et. al 2023). The greatest problem with chemiresistive sensors lies in the fact that these sensors normally react to many other gases apart from those intended for sensing (Fu et. al 2023). In addition, most of these sensors require high operating temperatures; thus, increasing net power consumption and hence limiting their application in some areas. In gas sensing research, MOS-based devices are among the most explored technologies because they offer high response characteristics, reliable stability, and economical implementation.

Among the most widely applied metal oxides in building gas sensors, sensitivity properties for a certain target gas tend to vary are: SnO_2, ZnO, TiO_2, WO_3, and CuO (Kong et. al 2023). Shukur (2021) for example noted that the thickness substantially changes the crystallinity of CuO thin films—besides its optical band gap and semiconducting behavior hence showing how important deposition parameters are toward optimizing sensitivity performance. Advantages of MOS gas sensors include simplicity in design, low power consumption and compatibility with microelectronic fabrication procedures that make them suitable for miniature and internet-of-thing (IoT) devices (Pravas et. al 2023). In order to improve the performance of MOS gas sensors, the following techniques may be employed; nano-squencing, doping and formation of heterojunction, the improvement of sensitivity, selectivity and response rate (Singh et. al 2021). Due to its outstading chemical and physical characteristics including high sensitivity to numerous gases, stability, and high electron mobility, ZnO powder has garnered considerable interest in being used as the sensing layer in the fabrication and design of gas sensors (Kolodziejczak et. al 2014). ZnO is associated with a broad band gap and high exciton binding energy which is believed to be responsible for ZnO's superior gas sensing capabilities (Ozgur et. al 2005). Considerable gas sensing studies have been carried out for ZnO materials in the forms of nanoparticles, nanoflowers and nanowires, particularly due to the large surface to volume ratios present in these materials. Recent studies have also shown that the large surface area and small particle size of nano-ZnO, as seen in its effect on natural rubber composites, enables it to enhance the properties of materials (Ali & Abdulwahid, 2018). Furthermore, cross-disciplinary research, including in the field of construction, has demonstrated that ZnO and other oxide systems nanomaterials, when structured at the nanoscale, have unique surface reactivity and functional characteristics (Aljashaami & Mohammed, 2024).

For example, Wang et al have reported that ZnO nanorods give an excellent response to ethanol vapour (Wang et. al 2004) have shown that ZnO dumbbell-like ZnO microcrystals has high responsivity to acetone gas (Qi et. al 2008). Not only that, ZnO applicable for a broad range of target gases but it is also appreciable for immobilizing nitrogen dioxide (NO_2), carbon monoxide (CO) and ammonia (NH_3) (Yamazoe et. al 2005). The WO_3 sensors can be used effectively for NO_2 and H_2S gases (Li et. al 2024) however, the ZnO sensors provide better response recovery times (Ahmad et. al 2019). As discussed previously, decorating the graphene surface with ZnO nanoparticles enhances the sensor performance in terms of sensitivity, selectivity, and response speed. This improvement can be attributed to the synergistic contribution of graphene's high carrier mobility and large effective surface area, together with the catalytic activity of ZnO and its capability to modulate the electronic characteristics of graphene (Worachote et al., 2022).

Gas sensors based on ZnO have also demonstrates potential for methane (CH_4) detection, particularly when utilizing nanostructures such as ZnO nanorods and nanoflowers. These nanostructures offer higher sensitivity compared to bulk ZnO, attributed to the larger active surface area and greater quantities of oxygen vacancies (Bhati et. al 2019). The development of methane sensors that utilize UV-irradiated ZnO nanostructures at room temperature further highlights their potential use in low power electronics (Sun et. al 2023). This study seeks to extend the discourse on ZnO gas sensors with particular focus on improving the performance of the sensors in methane gas detection. This is due to the unparalleled response time and sensitivity the author highlighted in previous works (Amaniah et. al 2020).

The paper outlines the making of thick-film gas sensors employing screen-printing onto glass substrates made of ZnO. Unlike the fluctuating temperatures when testing sensors used in (Mohd Chachuli et. Al 2022), the sensors were designed to detect methane at room temperature. In this paper, the experimental data and response time of ZnO gas sensor samples were comparable. The response sensitivity of the ZnO_2 sensor compared to the responsiveness of the ZnO_1 sensor demonstrated that the gas sensor constructed from ZnO_2 was more proficient in the detection of methane gas. This likely is due to the varying microstructure of the sensor and the composition that was heterogeneous in the ZnO_2 sample.

2. METHODOLOGY

2.1. ZnO Paste preparation

The materials used in this study were obtained from different suppliers: ZnO powder from Chemiz, ethyl cellulose from Sigma-Aldrich, and α-terpineol from Acros Organics. All reagents were used as received without further purification. Fig. 1(a) illustrates the first step, where α-terpineol (95 wt.%), ethyl cellulose (5 wt.%), and ZnO powder (50 wt.%) were accurately weighed using a balance. In Fig. 1(b), the ethyl cellulose and α-terpineol were mixed together using a magnetic stirrer at 40°C for 24 hours to form a homogenous binder. Finally, Fig. 1(c) shows the prepared binder (50 wt.%) being gradually combined with ZnO powder (50 wt.%) to create a thick film paste. The mixture was stirred continuously with a magnetic stirrer for approximately 24 hours to achieve a homogeneous and viscous paste.

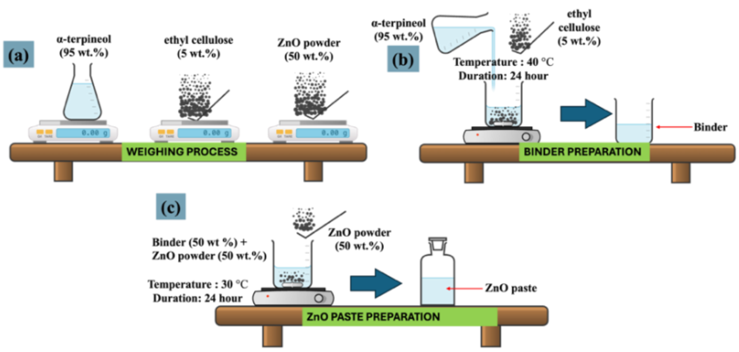

Fig. 1 Step by step of sensing layer preparation in the gas sensor (a) Weighing process of materials, (b) binder preparation and (c) ZnO paste preparation.

2.2. Gas sensor fabrication using screen printing

In this work, kapton film was used as the substrate for the gas sensor. The film was cut into 2 × 2 cm squares. The gas sensor consisted of an interdigitated electrode (IDE) and a sensing layer, both of which were printed onto the Kapton film. Fig. 2(a) shows the fabrication of the IDE using a screen-printing technique with a stencil mesh thickness of 10 μm, where silver paste was used. The printed electrode on the substrate was then annealed at 150°C for 30 minutes to enhance adhesion and conductivity. Fig. 2(b) illustrates the application of the previously prepared ZnO paste onto the interdigitated electrode as a 1×1 cm sensing layer, also using the screen-printing technique with a stencil mesh thickness of 3-4 μm. This layer was subsequently annealed at 200°C for 15 minutes to achieve optimal sensing properties and stability. Finally, a fine copper wire was attached to the gas sensor to establish an electrical connection by securing the wire to the leg of the interdigitated electrode with silver paste. ZnO paste was screen-printed onto a flexible substrate and annealed to enhance surface adhesion and conductivity. Thermal treatment plays a key role in improving crystalline structure and electrical pathways. Related work on Zn and Sn coatings has shown improved electrical contact and interfacial stability under heat, supporting the effectiveness of such treatments in sensor applications (Akkar & Alali, 2023).

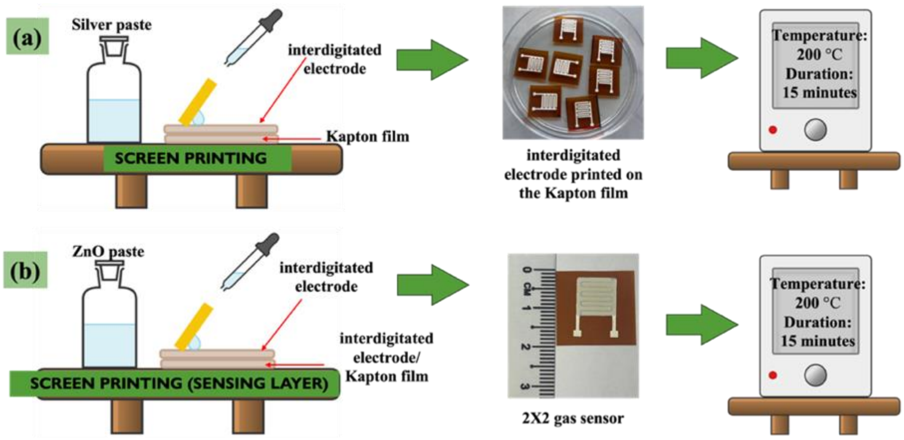

Fig. 2 Screen printing techniques (a) Fabrication of the interdigitated electrode via screen-printing with silver paste. (b) Application of a 1×1 cm ZnO sensing layer using screen-printing.

2.3. Current-Voltage (I-V) measurement setup

The electrical performance of the fabricated device was assessed through I–V testing, where the sensor current was monitored under different applied voltages. As the surrounding gas concentration influences the resistance of the sensing layer, the resulting resistance variation is manifested in the I–V profile. The presence of the target gas can result in an increase or a decrease in the current at a given voltage, depending on the material properties of the gas sensor and the interaction between the gas molecules and the gas sensor surface. The I-V curve measurement provides information on the conductivity of the gas sensor, which indicates the ease of current flow in the gas sensor when a voltage is applied. To explain the I-V curve measurement setup, Fig. 3 illustrates the setup, which includes a gas chamber containing the IDE, a source meter, and a LabView platform on the computer. In the setup, both legs of the IDE are connected to the source meter (Keithley 6482). The applied voltage, which ranges from -1 V to 1 V, is used to supply a voltage. The output current produced by the gas sensor is measured and recorded using the LabView platform. Therefore, an I-V graph is obtained for each IDE and recorded for analysis.

2.4. Gas Sensor Setup Methane Gas

Fig. 4 shows the gas sensor measurement setup designed to evaluate the sensor's performance in detecting methane. The system begins with two gas cylinders, one containing methane and the other a reference gas, both regulated by mass flow controllers (MFC) to precisely control the gas flow into the gas chamber. The gas sensor is housed within the sealed gas chamber, where it is exposed to various methane concentrations. A vacuum pump is connected to the chamber to remove residual gases between tests. The source meter applies a voltage to the sensor and measures the current response. This data is monitored and analyzed in real-time using LabVIEW software. Both ZnO_1 and ZnO_2 samples were prepared using the same materials and identical fabrication processes, including paste formulation, screen-printing conditions, and annealing process. These two samples were then subjected to gas sensing performance analysis, as described in the subsequent sections, to evaluate their response characteristics under identical test conditions.

Fig. 3 The I-V measurement setup, which includes a gas chamber containing the interdigitated electrode (IDE), source meter, and a LabView platform on the computer.

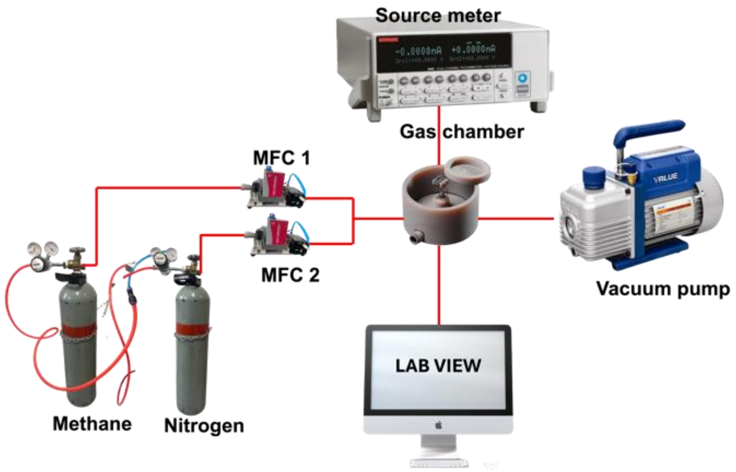

Fig. 4 Illustration of gas sensor measurement setup for methane detection, integrating gas flow control, vacuum pump, source meter, and real-time data acquisition using LabVIEW.

3. RESULTS AND DISCUSSION

3.1. Current-Voltage (I-V) characteristics of ZnO gas sensor

Fig. 5 represents the measurements were taken over a voltage range of approximately -1V to +1V. The current response ranges from about -1.2 µA to +1.2 µA for both samples. The graph shows a linear relationship between current and voltage for both ZnO_1 and ZnO_2 samples. This suggests that the materials are obeying Ohm's Law, as the current increases proportionally with the applied voltage. Since the graph demonstrates linearity, both ZnO_1 and ZnO_2 can be considered conductive. This linearity suggests that ohmic contacts were successfully formed between the ZnO sensing layer and the silver electrodes, ensuring that the current flow is not limited by contact resistance or non-linear junction effects. A linear I–V characteristic is crucial for reliable gas sensing, as it confirms that the sensor's current response during gas exposure is primarily influenced by changes in the sensing layer's resistance such as those caused by surface adsorption of methane molecules rather than interface-related distortions (Wang et. al 2022). The difference in resistance between the two lines is minor, suggesting that both samples were prepared with a similar ZnO deposition using the screen printing technique on each gas sensor.

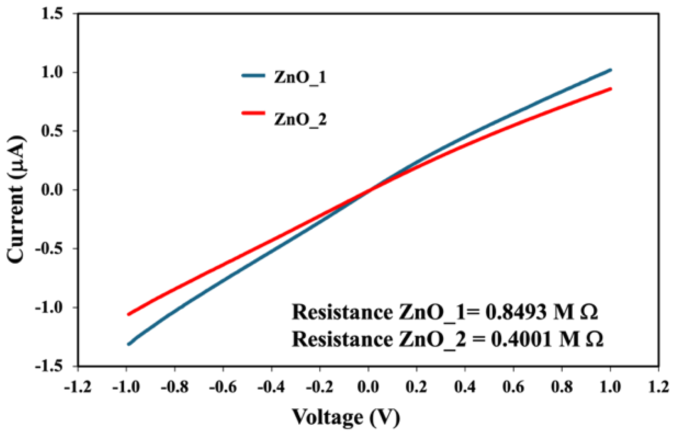

Fig. 5. I-V characteristic of ZnO gas sensor at -1.5 to 1.5 V

3.2. Response ZnO gas sensor to methane gas

Fig. 6(a) shows comparison of the methane sensing performance of ZnO_1 and ZnO_2 over a one-hour period, divided into three 20-minute phases of nitrogen, methane, and nitrogen exposure at methane concentration of 6700 ppm. Upon transitioning from nitrogen to methane, both ZnO_1 and ZnO_2 exhibit an increase in current, indicating their response to methane exposure. ZnO_1 shows a more pronounced increase compared to ZnO_2, suggesting higher sensitivity, while ZnO_2 demonstrates a smaller yet noticeable response as well as Fig. 6 (b).

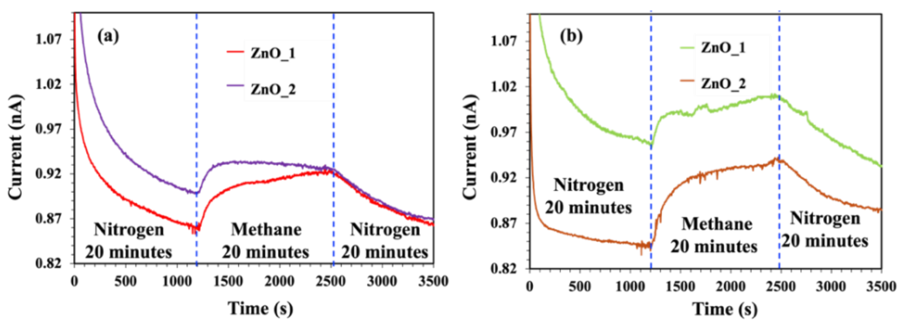

Fig. 6 The current measurement (nA) response of ZnO gas sensors to methane detections (a) Response of samples ZnO_1 and ZnO_2 at 6700 ppm methane concentration (b) Response of samples ZnO_1 and ZnO_2 at 7500 ppm methane concentration. Note: The figure shows response curves specific to methane exposure. Comparative plots with other gases or commercial sensors were not included in this work but are proposed for future studies.

In Table 1, the properties of ZnO based gas sensors for methane detection show marked differences in response and response time among the samples. ZnO_2 has good response compared with ZnO_1 at 6700 ppm (1.0571/1.0386) and at 7500 ppm (1.1060/1.0564) which demonstrates ZnO sensitivity. Additionally, ZnO_2 shows a faster response time at 6700 ppm (648 s vs. 672 s) but a slightly longer response time at 7500 ppm (1256 s vs. 1207 s). From all these investigations ZnO_2 has a better sensitivity to methane, especially at low ppm levels. The same effects have been reported and observed for the ZnO-based sensors incorporated with the nanostructures which have both higher sensitivity level and lower response time owing to the large number of active surface areas for the adsorption of the gaseous substances. Compared to recent optimizations, our ZnO_2 sensor exhibits strong performance at room temperature, achieving a response time of 648 s. For perspective, Co-doped ZnO nanowire sensors have shown CH_4 detection at concentrations as low as 1 ppm operated at 50 °C thanks to increased surface defect density (Shafiei et. al 2020). For comparison, Chen et al. reported 2D ZnO nanowall methane sensors that achieved a high response of ~810% to 3000 ppm CH_4 at 300 °C, with rapid response and recovery (~6 s / 21 s, 67% criterion), attributed to the enhanced surface area of the nanowall structure. On the other hand, Pd–Ag-enhanced ZnO films can achieve rapid response (16.3 s) and 74.3% response metrics but require 250 °C operation and involve expensive noble metals (Nanto et. al 2014). Moreover, the addition of noble metals such as palladium has been found to enhance the response of the sensor and its response time. A study showed that the Pd-Ag (26%) catalytic metal contact on ZnO thin films resulted in a maximum response of 74.3% and a response time of 16.3 s at 250°C for methane sensing (Ahmad et. al 2006). These results can be attributed to the variation in the electronic and structural properties, further emphasizing the potential of ZnO_2 in methane sensing.

Table 1. Characteristics of ZnO gas sensors on methane detection. Note: Table 1 presents the response characteristics of ZnO_1 and ZnO_2 to methane only. Additional gas types and benchmark sensors will be included in extended analysis

Sample Name	Concentration (ppm)	Response	Response time (s)
ZnO_1	6700	1.0386	672
	7500	1.0564	1207
ZnO_2	6700	1.0571	648
	7500	1.1060	1256

4. CONCLUSIONS

The present research confirmed the feasibility of producing ZnO thick-film methane sensors through screen-printing fabrication, followed by performance testing at room temperature. Both sensors, ZnO_1 and ZnO_2, were found to be ohmic in current-voltage (I-V) characteristics, with linear responses to varying voltages. The methane gas sensing characteristics of the sensors indicated that while both sensors have similar response times, the ZnO_2 sensor was found to be much more sensitive to methane gas, especially at higher concentrations. This is attributed to the minute differences in the microstructure and/or composition of the ZnO films, which enhanced the sensor's response to methane gas molecules. The findings of this study indicate the potential of ZnO gas sensors for methane gas detection, especially for environmental and industrial applications, where the detection of low concentrations of methane gas is of prime importance for safety and regulatory purposes. Future studies should aim at enhancing the performance of ZnO gas sensors through material modifications such as doping with noble metals like palladium or platinum, or through the incorporation of heterojunctions with other semiconductor materials like MoS_2 or TiO_2. ZnO gas sensors have vast potential for applications in methane gas detection for various industries.

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