International Journal of Leading Research Publication (IJLRP)

E-ISSN: 2582-8010 ● Website: www.ijlrp.com ● Email: editor@ijlrp.com

Fabrication and Gas Sensing Properties of WO_3 thin Films Prepared by Spin Coating Method

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Abstract:
In this work, the fabrication and gas sensing properties of tungsten trioxide (WO_3) thin film gas sensors prepared by the spin coating method are reported. WO_3 thin films were successfully deposited on glass substrates at 2000 rpm for 60 s, using spin coating method. The fabricated films were tested for their sensing performance towards various gases, including NH_3, H_2S, LPG, ethanol, and methanol. Among these, the WO_3 thin films exhibited the highest gas response (68.51%) to H_2S at an operating temperature of 120 °C with a concentration of 1000 ppm. The sensor also demonstrated good selectivity along with fast response and recovery times, highlighting the potential of spin-coated WO_3 thin films for efficient and reliable for H_2S gas sensing applications.

Keywords: Thin films, spin coating, gas sensors, gas response, recovery time.

1. Introduction

Gas sensors play a crucial role in detecting and monitoring toxic, combustible, and hazardous gases in environmental, industrial, biomedical, and domestic applications. The performance of gas sensors strongly depends on the nature, morphology, and microstructure of the sensing material used [1, 2]. Thin films have gained immense importance in the gas sensor domain due to their unique advantages compared to bulk or thick-film counterparts. Thin films offer a significantly high surface-to-volume ratio, which enhances the interaction between the gas molecules and the sensing material surface, thereby improving the sensitivity of the sensor. Since gas sensing is predominantly a surface phenomenon governed by adsorption and desorption of gas species, thin films provide a large number of active sites for these surface reactions [2, 3]. The thin films can be fabricated with controlled thickness, crystallinity, porosity, and grain size, which directly influence the sensor response, selectivity, and operating temperature. Another advantage of thin films is their compatibility with miniaturized electronic devices and integration into microelectromechanical systems (MEMS), making them suitable for portable and low-power sensing devices. Moreover, thin films often exhibit faster response and recovery times compared to bulk materials because the diffusion path of gas molecules into the sensing layer is shorter [4, 5]. This property is critical for real-time monitoring applications where rapid detection of toxic gases like H_2S, NH_3, or volatile organic compounds is essential. The thin films not only improve the efficiency and selectivity of gas sensors but also support the development of low-cost, reproducible, and scalable devices for diverse applications [3-5].

The fabrication of thin films for gas sensing applications can be achieved by a variety of physical and chemical methods, each offering distinct advantages and limitations. Broadly, thin film deposition techniques are categorized into physical vapor deposition (PVD), chemical vapor deposition (CVD), solution-based techniques, and thermal processes. In PVD methods such as sputtering, pulsed laser deposition, and thermal evaporation, the material is vaporized from a source and condensed on a substrate under high vacuum conditions. These methods yield high-quality films with controlled stoichiometry and crystallinity but require

expensive equipment and high-energy sources. In contrast, CVD techniques involve chemical reactions of precursor gases near or on the heated substrate, leading to the deposition of uniform and conformal films. CVD is widely used for semiconducting thin films but requires handling of toxic precursors and high process temperatures. Solution-based methods such as sol-gel, dip coating, spin coating, spray pyrolysis, and electrophoretic deposition have gained popularity for gas sensor fabrication due to their simplicity, low cost, and ability to produce porous nanostructured films. These techniques utilize a precursor solution that can be easily modified by changing the concentration, viscosity, or additives, thereby enabling control over film morphology and microstructure [8-10]. Among these, sol-gel and spin coating methods are particularly attractive because they allow the preparation of homogeneous films with fine control of thickness in a reproducible and scalable manner. Another class of methods includes thermal oxidation, anodization, and electrodeposition, where thin films are directly formed on a substrate by chemical or electrochemical processes. The choice of fabrication method depends on the desired film properties, cost, and application requirements. For gas sensors, solution-based deposition techniques are often preferred because they yield nanostructured, porous, and active films that enhance gas adsorption and response [10, 11].

Spin coating is one of the most widely employed solution-based thin film deposition techniques owing to its simplicity, low cost, and excellent control over film thickness and uniformity. The process involves the deposition of a liquid precursor solution onto a substrate, followed by rapid spinning to spread the liquid uniformly across the surface due to centrifugal force [11, 12]. Typically, the process begins with the preparation of a precursor solution containing the desired metal salts, solvents, and sometimes binding agents or stabilizers. A few drops of this solution are carefully dispensed onto a clean substrate, such as glass, quartz, or silicon wafer, which is mounted on a spin coater. The substrate is then rotated at a predetermined speed, usually ranging between 1000 and 5000 revolutions per minute (rpm). The centrifugal force spreads the solution radially outward, and simultaneously, the volatile solvents evaporate, leaving behind a uniform thin film of the solute material. The final film thickness depends on several parameters, including spin speed, spin time, viscosity of the solution, and concentration of precursors [12, 13]. Higher spin speeds and longer spin times generally result in thinner films, whereas more viscous solutions lead to thicker films. The spin coating process is often divided into three stages: deposition, spin-up, and evaporation. In the deposition stage, the precursor solution is dropped onto the substrate. During the spin-up stage, the substrate rapidly accelerates to the desired rotation speed, spreading the solution evenly. In the evaporation stage, most of the solvent evaporates while the film thins out and stabilizes. After deposition, the as-spun films are typically dried and subjected to annealing or calcination treatments at elevated temperatures to remove organic residues, improve adhesion, and promote crystallization of the sensing material. It offers several advantages over other techniques. It is simple, reproducible, and requires relatively inexpensive equipment compared to high-vacuum deposition systems. The method produces films with excellent thickness uniformity and smooth surfaces, which are critical for reliable gas sensing [13, 14].

The aim of this work is to fabricate tungsten trioxide thin films using the spin coating method and to investigate their gas sensing properties towards selected gases, with particular focus on gas response, selectivity, limit of detection and response/recovery characteristics of the gas sensor.

2. Experimental Procedure

2.1 Fabrication of WO_3 thin films

In this work all AR grade chemicals were used for the fabrication of WO_3 thin films as well as preparation of selected gases. For the fabrication of WO_3 thin films by the spin coating technique, tungsten hexachloride (WCl_6) was used as the primary precursor. A 0.1 N of WCl_6 was dissolved in distilled water (100 ml) under constant stirring to ensure complete hydrolysis. After the hydrolysis of tungsten hexachloride is highly exothermic and tends to form unstable intermediates, glacial acetic acid was added as a stabilizing agent to control the reaction and assist in gel formation [13, 15]. To adjust the pH upto 9 and promote condensation of

the tungstate species into a stable gel network, sodium hydroxide (NaOH) solution was slowly added dropwise under continuous stirring. This mixture was stirred for 3-4 hours until a clear and homogeneous sol–gel solution was obtained. The prepared sol was then used for thin film deposition. A few drops of the precursor sol were dispensed onto pre-cleaned glass substrates, which were mounted on a spin coater. The substrates were spun at 2000 rpm for 60 seconds to spread the sol uniformly across the surface by centrifugal action. The deposited films were initially dried under IR lamp for 20-30 minutes to remove excess solvents and then subjected to annealing at 300°C temperatures to decompose residual organics, improve adhesion, and crystallize the WO_3 phase [15, 16]. This process yielded uniform, adherent WO_3 thin films suitable for subsequent gas sensing studies.

3. Result and discussion

The gas sensing study was carried out using static gas sensing system (set-up) as shown in Fig. 1.

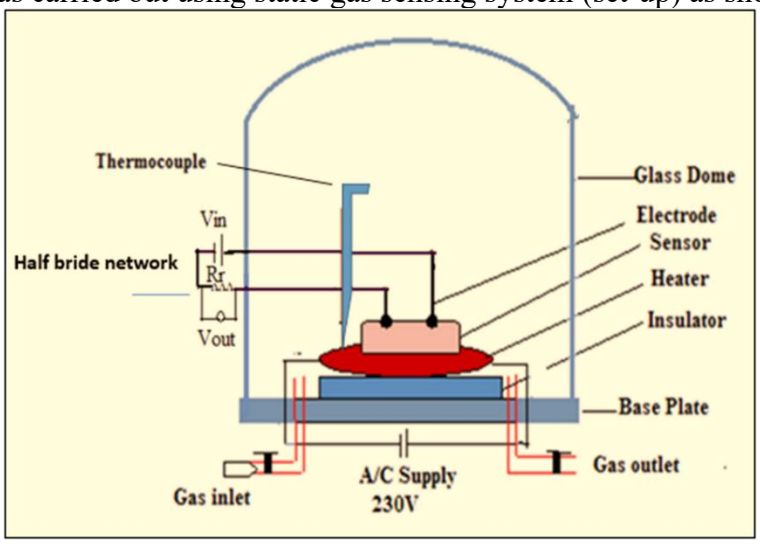

Figure 1. Schematic diagram of static gas sensing set-up

The gas sensing setup consists of a sealed glass dome mounted on a base plate, within which the WO_3 thin film sensor element is placed over a heater separated by an insulating layer. The integrated heater enables precise control of the sensor's operating temperature, while a thermocouple is employed to continuously monitor the temperature during measurements. The chamber is equipped with gas inlet and outlet ports, allowing the introduction of the test gases as well as purging with fresh air to restore baseline conditions [17, 18]. The sensor element is electrically connected to external electrodes incorporated into a half-bridge network circuit, which facilitates the measurement of resistance variations during gas exposure. When a target gas is introduced into the chamber, its interaction with the sensor surface modifies the charge carrier concentration in the WO_3 thin film, leading to a measurable change in resistance. This resistance variation is recorded as a voltage change across the bridge network and is directly correlated with the sensor's response to the analyte gas. The static gas sensing system ensures that the test gas remains confined within the chamber until complete adsorption and desorption take place, enabling accurate determination of gas response, selectivity, response, and recovery times [18, 19]. This setup provides a well-controlled environment for evaluating the gas sensing performance of WO_3 thin film-based sensors.

The gas response of the fabricated WO_3 thin films toward selected gases was evaluated using a resistance-based definition. The response is expressed as the ratio of the resistance of the sensor in the presence of the target gas (Rg) to its resistance in air (Ra), as shown in Equation (1) [20].

$$\text{Gas response (\%)} = Rg/Ra \times 100 \qquad \text{(Eq. 1)}$$

Where, Rg resistance in presence of the target gas and Ra resistance in air.

Selectivity in gas sensing is an important parameter that describes the ability of a sensor to preferentially respond to a specific target gas in the presence of other interfering gases. It is defined as the ratio of the gas response to the target gas (Starget) to the gas response obtained for a particular interfering gas (Sinterfering), as expressed in Equation (2) [20, 21].

Selectivity= Starget/Sinterfering (Eq. 2)

Where, Stargetdenotes the response of the sensor to the desired analyte, while Sinterferingcorresponds to the response towards other gases that may coexist in the environment.

Figure 2(a) shows the variation of gas response of WO_3 thin films as a function of operating temperature toward different gases including H_2S, ethanol, methanol, LPG, and NH_3. The results indicate that the WO_3 thin film exhibits the highest sensitivity to H_2S, with a maximum gas response of 68.51% at an operating temperature of 120 °C for a gas concentration of 1000 ppm. In contrast, the responses to other gases were relatively lower, with 12.06% for ethanol, 8.54% for methanol, 6.35% for LPG, and only 3.41% for NH_3, as shown in the Figure 2. This selectivity is further highlighted in Figure 2(b), where the selectivity plot clearly demonstrates that the WO_3 thin film sensor exhibits outstanding preference for H_2S compared to the other tested gases [19, 20]. The dynamic response behavior is illustrated in Figure 2(c), where the cyclic exposure of the sensor to H_2S and subsequent purging with air demonstrates reproducible response and recovery cycles. The sensor shows rapid response when the gas is introduced (ON time- 14 S) and quick recovery when the gas is removed (OFF time-53 S), confirming its suitability for real-time sensing applications. Figure 2(d) depicts the sensitivity of the WO_3 thin film as a function of H_2S gas concentration. The response increases steadily with increasing H_2S concentration, showing a nearly linear trend up to 1000 ppm, which indicates that the sensor is capable of detecting both low and high concentrations of H_2S with good accuracy and stability [21, 22].

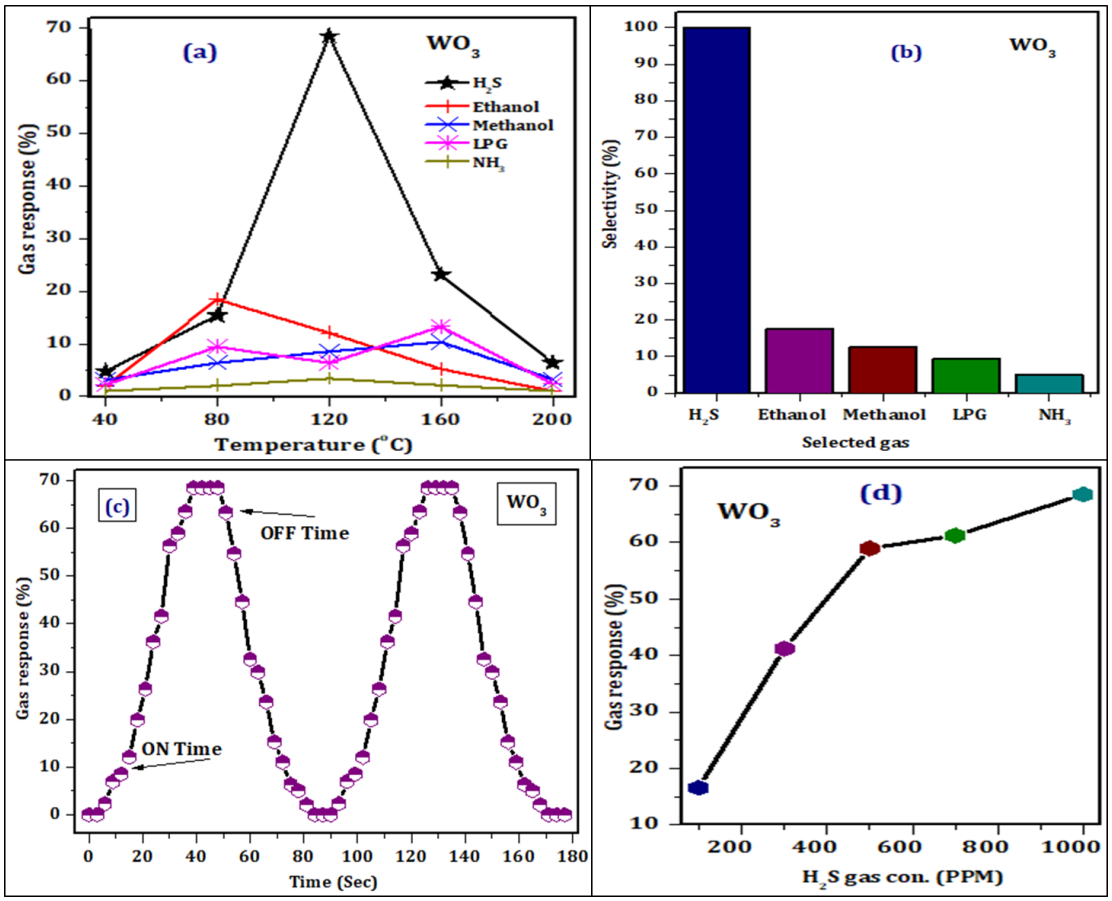

Figure 2. (a) Sensitivity versus operating temperature plot, (b) Selectivity, (c) Response and recovery time plot & (d) Sensitivity versus H_2S gas concentration plot of WO_3 thin films.

The gas sensing mechanism of WO_3 thin films is primarily governed by the surface adsorption and desorption of oxygen species. At the operating temperature, oxygen molecules from the air are adsorbed onto the WO_3 surface and capture electrons from the conduction band, forming oxygen ions such as O^- or O_2^-. This creates a depletion layer and increases the film resistance [20, 23]. When a reducing gas like H_2S is introduced, it reacts with the adsorbed oxygen species, releasing the trapped electrons back to the conduction band of WO_3. This reduces the depletion layer width and significantly decreases the resistance of the sensor. The magnitude of resistance change depends on the concentration and reactivity of the target gas. Since H_2S has strong reactivity with adsorbed oxygen, the WO_3 thin film shows maximum response toward H_2S compared to other gases [22, 23]. This mechanism explains the high sensitivity, good selectivity, and fast response/recovery behavior of the fabricated WO_3 thin film gas sensor.

CONCLUSIONS

The WO_3 thin films were successfully fabricated using the spin coating technique and evaluated for their gas sensing performance. The films demonstrated excellent sensitivity and selectivity towards H_2S gas, showing a maximum response of 68.51% at an optimum operating temperature of 120 °C with a gas concentration of 1000 ppm. In comparison, the response toward other tested gases such as ethanol, methanol, LPG, and NH_3 was relatively low, confirming the high selectivity of WO_3 thin films for H_2S detection. The sensor also exhibited fast response and recovery times, along with reproducible cyclic behavior, highlighting its potential for reliable real-time gas sensing applications. The observed sensing mechanism can be attributed to the surface adsorption of oxygen species and their interaction with reducing gases, which modulates the resistance of the WO_3 thin film. The study demonstrates that spin-coated WO_3 thin films are promising candidates for low-cost, efficient, and selective H_2S gas sensors suitable for environmental and industrial monitoring.

Acknowledgment

The author expresses sincere gratitude to the Head of the Department of Chemistry and Research Center in Chemistry, as well as to the Principal of Mahatma Gandhi Vidyamandir's Maharaja Sayajirao Gaikwad Arts, Science & Commerce College, Malegaon Camp, Dist. Nasik (423105), for their valuable support and for providing the necessary facilities to carry out this research work.

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