

Tungsten Oxide Pellet Sensor to Detect H₂S Gas at Room Temperature

* **Rajendra. S. Khadayate and Pruthwiraj. R. Patil**

G. D. M. Arts, K. R. N. Commerce and M. D. Science College,
Jamner, Dist-Jalgaon, Maharashtra, India
Tel.: +91 02580 233081, +91 0257 2263437
E-mail: rskhadayate@yahoo.com

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Abstract: This paper presents H₂S gas sensing properties of WO₃ pellets at room temperature. In this work, the WO₃ pellets were prepared by standard solid state method. The prepared pellets were characterized by x-ray diffraction (XRD) measurements and scanning electron microscopy (SEM). The H₂S gas sensing properties of WO₃ pellets were investigated at room temperature and at different concentrations of gas. The WO₃ pellet exhibit excellent H₂S gas sensing properties in air atmosphere at room temperature with fast response and recovery time. *Copyright © 2012 IFSA.*

Keywords: WO₃ Pellet, Solid state method, H₂S gas

1. Introduction

In the recent years, sensors have attracted a great deal of attention from scientists and engineers. Even in the near future it is expected to gain importance in view of the construction subsystems. Detection of various gases using solid-state chemistry has generated a great deal of interest, both in academia and in industry for environmental monitoring [1-2].

Hydrogen sulfide is a toxic gas, often produced in coal, coal oil or natural gas manufacturing. Even at low concentration it produces severe effects on the nervous system. Therefore, reliable sensors with low cost, low energy consumption having high sensitivity, selectivity, and operable in ppm range of H₂S are in high demand for environmental safety and industrial control purposes.

Many H₂S gas sensors working on high temperatures are reported [3-9]. But very few are reported to work at room temperature. Gas sensors operating at room temperature are very important for electric power saving purpose. Hence research for new good gas-sensing materials operating at room temperature is continuously going on.

In this paper, we are reporting preparation of WO₃ pellets by solid state route and their H₂S gas sensing properties at room temperature.

2. Experimental

2.1. Preparation of Sintered Pellets of WO₃

The WO₃ powder (purity ~ 99.99 %) was mechanically milled in an acetone medium using Fisher type electric agate pestle and mortar for 24 hours. The powder was then dried at 200 °C for 20 min. After drying, pellets of the powder were prepared by using a die under pressure of 7 tons for 10 sec with the help of a hydraulic press. The prepared pellets were sintered at 800 °C for 1 hour to increase the strength and to have compactness. Fig. 1 shows flowchart for the preparation of WO₃ pellet by solid state route.

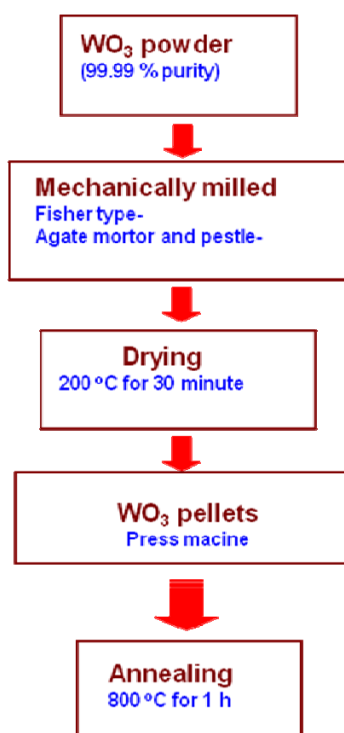


Fig. 1. Flow Chart for the preparation of WO₃ pellet by solid state Route.

2.2. Study of Structural Properties of Prepared Pellets of WO₃

The structural properties of the WO₃ pellets were investigated using X-ray diffraction (XRD) technique. The X-ray diffraction patterns were recorded with a Rigaku diffractometer (Miniflex Model, Rigaku, Japan) having Cu K ($\lambda = 0.1542$ nm). The thickness and diameter of the WO₃ pellets were measured by a standard vernier caliper and micrometer screw gauge.

2.3. Details of Gas Sensing System

Fig. 2 shows schematic diagram of gas sensing system. The gas sensing studies were carried out using a static gas chamber to sense H₂S vapour in air ambient. The WO₃ pellets were used as the sensing elements. The sensing element was kept directly on a base in the gas chamber. The known volume of the H₂S gas was introduced in to the gas chamber pre-filled with air and it was maintained at atmospheric pressure. A constant voltage was applied to the sensor element through a potential divider arrangement as shown. The prepared WO₃ pellet i.e. sensor element was act as a lower branch resistor of potential divider arrangement as shown in Fig. 3.

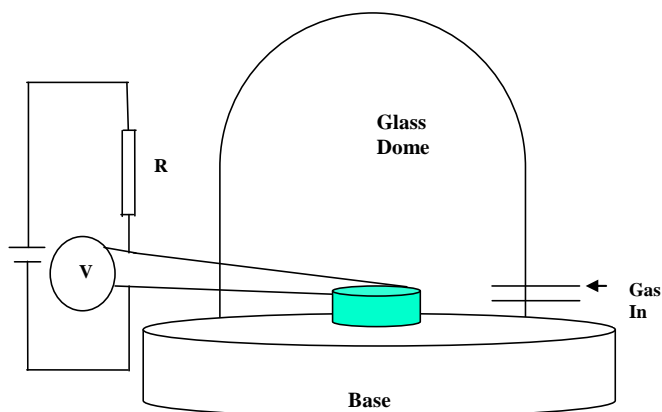


Fig. 2. Schematic diagram of gas sensing unit.

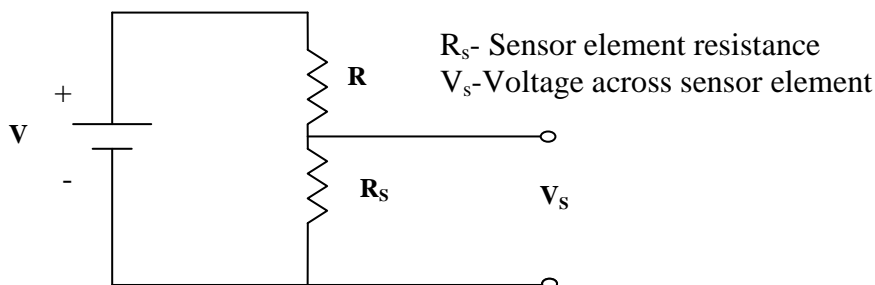


Fig. 3. Circuit used with sensor element.

The voltage across WO₃ pellet is taken as a base index for the measurement of sensitivity of the sensor element for the H₂S gas. The electrical voltage across the sensing element was measured by using a simple two probe configuration, before and after exposure to H₂S gas using a sensitive digital multi meter (METRAVI 603).

The sensitivity (S) of the sensing element is defined as:

$$S = \frac{V_a - V_g}{V_a} \times 100 \% ,$$

where V_a and V_g are the voltage values across the sensor element in air and in the presence of H₂S gas, respectively.

The sensing properties of the prepared WO_3 pellet for different regularly available gases in the atmosphere like LPG, NH_3 , ethanol, CO, CO_2 etc was also investigated.

3. Results and Discussion

The X-ray diffraction measurements performed on the prepared WO_3 pellets indicate that it is polycrystalline and the crystalline structure corresponds to the triclinic phase of WO_3 (JCPDS Data card 83-0948). The diameter and thickness of the prepared WO_3 pellets was 1cm and 2 mm respectively.

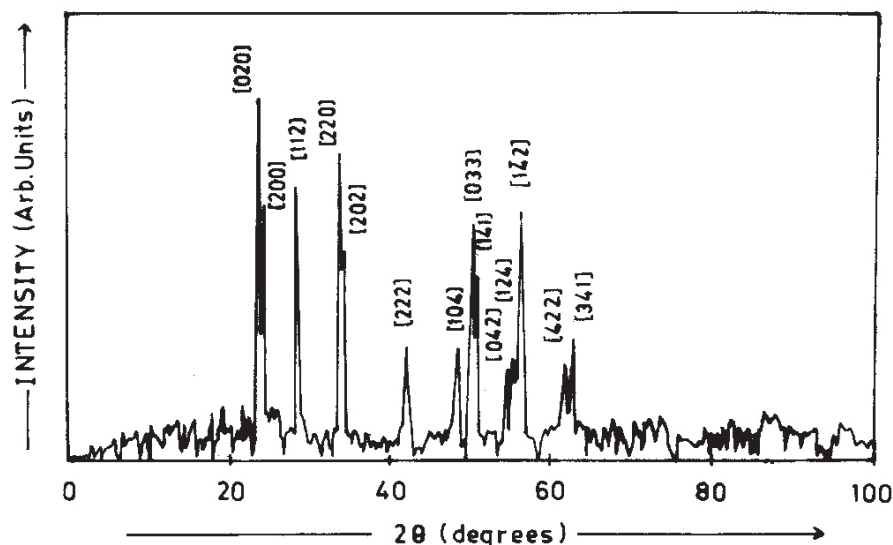


Fig. 4. XRD of the prepared WO_3 pellet.

Response and recovery time are the basic parameters of the gas sensors. They are defined as the time taken for the sensor to attain 90 % of maximum change in resistance on exposure to gas is the response time. The time taken by the sensor to get back 90 % of the original resistance is the recovery time. Fig. 5 shows response-recovery curve of the prepared WO_3 pellets at room temperature in response of H_2S gas (10 C.C.). It indicates that the WO_3 pellet operating at room temperature shows response within ~12 s after exposure to 10 c.c. H_2S gas and recovers back within ~45 s after removal of the H_2S gas. The sensitivity of WO_3 pellet to H_2S gas (10 c.c.) calculated from the response-recovery curve and the above mentioned formula is found to be ~ 10.49 %.

During the further H_2S gas sensing experiments with the prepared WO_3 pellet, the time dependence of the voltage across sensor element was monitored for the repeated exposure and removal of H_2S gas. The exposure of the WO_3 pellet to the H_2S gas followed by the removal of the gas constitutes a single cycle of gas response study. The variation of voltage across the WO_3 pellet operating at room temperature with the time when exposed to 10 c.c. H_2S gas shown in Fig. 6. As can be seen from Fig. 6, the voltage value across the WO_3 pellet decreases from decreases from 9.05 V to 8.1 V, when exposed to 10 c.c. of H_2S gas and after removal of the H_2S gas it shows the change in the voltage value back to 9.05 V. This constitutes the first cycle of the H_2S gas response study. On exposing again the same WO_3 pellet to H_2S gas, it is observed that the voltage value decreases from 9.05 V to 8.1 V and after removal of the H_2S gas it changes to 9.05V, which constitutes the second cycle of H_2S gas response study. The similar changes in the voltage value were observed for further four cycles as

shown Fig. 6. It is clear that the response and recovery characteristics are almost reproducible and rather quick when exposed to H₂S gas.

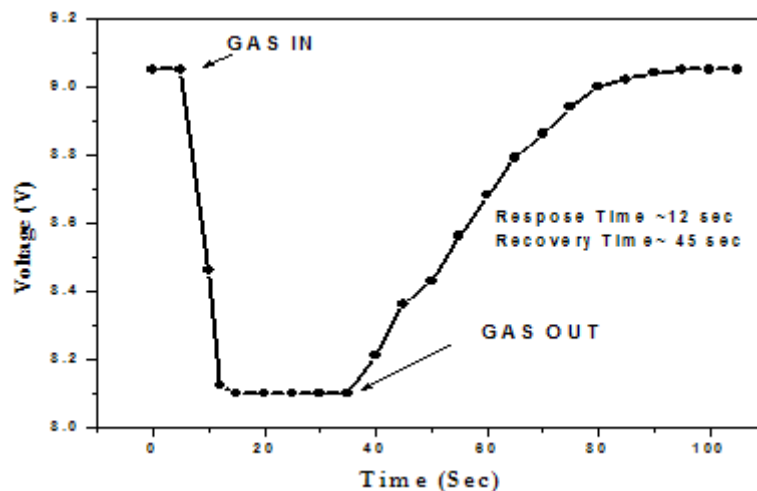


Fig. 5. Response-Recovery Curve of the prepared WO₃ pellet at room temperature in response to H₂S gas (10 c.c.)

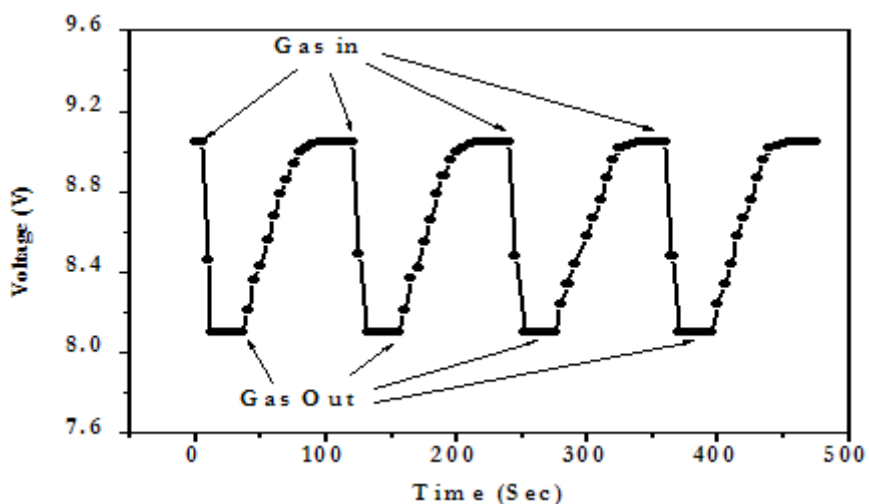


Fig. 6. Repetitive Response of the prepared WO₃ pellet at room temperature upon exposure to H₂S gas (10 c.c.)

Fig. 7 shows sensitivity verses concentration curve for WO₃ pellet in response to H₂S gas in c.c. The prepared WO₃ pellet was exposed to varying concentrations of H₂S gas (2-10 c.c.). It observed that the sensitivity increases linearly from 2-8 c.c. after that it remains constant.

The sensing properties of the prepared WO₃ pellet for different gases like LPG, NH₃, ethanol, CO, CO₂ etc was also investigated at room temperature. It is observed that the WO₃ pellet sensor element was not shown any response to other gases. Thus the prepared WO₃ pellet sensor element shows highest selectivity towards H₂S gas at room temperature.

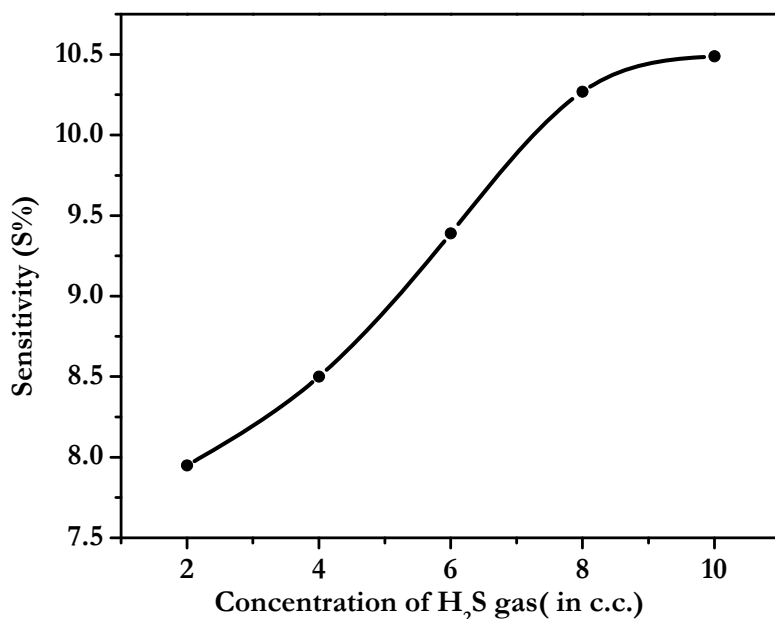


Fig. 7. Sensitivity versus Concentration of H₂S gas in c.c.

5. Conclusions

Following conclusions can be drawn from the experimental results

1. In this work pellets of WO₃ can be prepared by solid state route and characterized by XRD.
2. The H₂S gas sensing properties of prepared WO₃ pellets was investigated at room temperature and at different gas concentration.
3. The WO₃ pellet operating at room temperature exhibit excellent sensitivity, selectivity and repeatability towards H₂S gas as compared to other gases like LPG, NH₃, ethanol, CO, CO₂ etc.
4. This study demonstrates the possibility of utilizing WO₃ pellet as a sensor element for the detection of H₂S gas.

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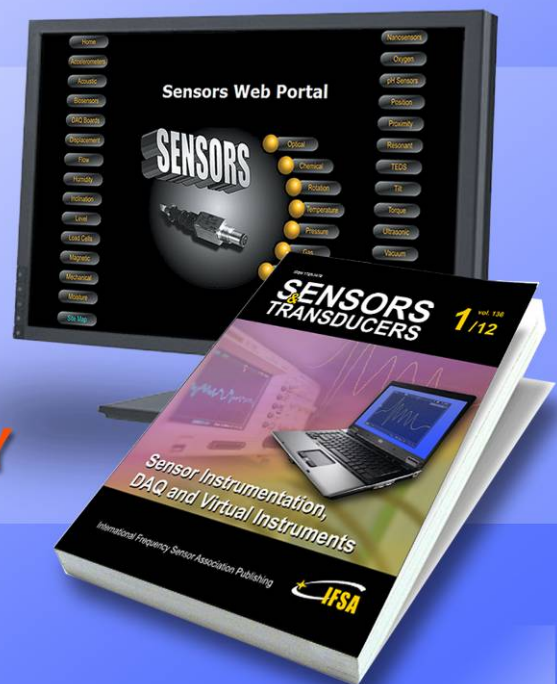
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