

DC Magnetron sputtered V_2O_5 thin films for sensitive detection of hydrogen gas at room temperature

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Research Article

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Abstract

Hydrogen (H_2) gas sensing studies of direct current magnetron sputtered vanadium oxide (V_2O_5) thin films were sputtered at different partial pressures of oxygen to modify their stoichiometry, morphology, and grain size for efficient H_2 detection at room temperature. This drastically enhanced the H_2 adsorption sites of improved responsivity. The sample also showed good selectivity to H_2 and ammonia, acetone, ethanol, and toluene. The microstructure and morphology studies of materials were analyzed by Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and Raman analysis. Structural study of pure V_2O_5 thin films showed amorphous nature with the (100) direction orientation. SEM image surface morphology explored the small flower-like morphology. From the optical study, the transmittance and band gap has decreased due to increasing substrate temperature. Specifically, H_2 was measurable with concentrations ranging from 50–500 sccm (standard cubic centimeter) at room temperature in 1000 sccm dry N_2 . The results showed that a good sensitivity towards H_2 was obtained with a thin film sensor at the optimal operating temperature of room temperature ($27^\circ C$) and also showed good selectivity. The gas response, response time, and recovery time were measured.

1. Introduction

Hydrogen can easily diffuse to very small areas and although, its low concentrations level its detected to avoid any catastrophic damage to the system in human life. Metal oxide semiconductors (MOS) were mainly used as sensing materials for a few decades due to their rich features of high sensitivity towards volatile organic compounds [1]. V_2O_5 thin films have been initiated to have good gas-sensing responses of ammonia, methane, acetone, and other organic compounds due to their important response of electrical resistance [2]. Hence, various states of vanadium V^{2+} and V^{5+} is having useful applications like gas sensing, infrared detectors, and optical switches. Different types of gas sensors are semiconductor [3–5], solid electrolyte [6], contact combustion [7], optical [8–10], quartz vibrator [11], and surface acoustic wave gas sensors [12]. Among various types, MOS gas sensors are having high sensitivity, small size, and low cost [13].

Numerous thin film preparation techniques were available like electron beam evaporation [14], DC magnetron [15, 16], chemical vapor [17], spray pyrolysis [18, 19], and spin coating [20] methods. The first step is V_2O_5 deposition employing the DC magnetron technique with different oxygen flow rates during sputtering. The second step is the characterization study of the structural, morphological optical, and electrical properties. The final step is then evaluated for its H_2 sensing performance at room temperature under different gas concentrations.

2. Experimental

A V_2O_5 thin film was synthesized employing a DC magnetron using a vanadium target (3 inches diameter, 99.995% pure) with different oxygen flow rates with 30% of Ar. During sputtering, a cathode current of

300 mA and sputtered power of 66W is utilized. The substrate and target distance were nearly 80 mm. Details of these sputtering conditions had been described in Table 1.

Table 1 Different sputtering factor for preparation of V_2O_5 thin films

Substrate Temperature	Sputtering Power	Deposition time(min)	Base Pressure mbar	O_2 pressure mbar	Working pressure mbar	O_2 flow sccm	Sample code
Room temp	66W	30	2.5×10^{-5}	1.8×10^{-4}	1×10^{-2}	1	S1
Room temp	66W	30	2.5×10^{-5}	3.8×10^{-4}	1×10^{-2}	2	S2
Room temp	66W	30	2.5×10^{-5}	8.7×10^{-4}	1×10^{-2}	3	S3
350 °C	66W	30	2.5×10^{-5}	8.7×10^{-4}	1×10^{-2}	3	S4

The crystal structure was examined employing an X-ray diffractometer (Bruker D8 Advance) CuK_{α} wavelength of 1.5406 \AA . A surface morphology study was carried out using the SEM model of Quanta FEG 250. Argon-ion laser Raman spectroscopy with the wavelength of 200–2100 nm model of LAB RAM HR Horiba France. Optical transmittance and band gap energy of V_2O_5 were calculated at wavelengths between 300–3000 nm using UV-Vis spectrophotometer models (Varian5000). Electrical measurements were carried out microprobe station (Janis Microprobe Station) and data were taken with the Lab View program with the help of a Keithley digital multimeter.

For hydrogen gas sensing measurement, the measurement device was using placed inside a stainless-steel chamber equipped with a Keithley digital multimeter. The prepared V_2O_5 thin film dimension was $1 \text{ cm} \times 1 \text{ cm}$ with good electrical contacts of Aluminium. The V_2O_5 thin film was mounted in a 500 ml air-tight chamber where it is preheated using a temperature-by-temperature controller. Moreover, we have to wait more than three hours resistance of thin films to become stable. Using a rotary pump, the chamber was evacuated before testing the devices, and then target gas H_2 and dry air of N_2 1000 sccm was allowed and resistance change response of the device was monitored while waiting for a stable baseline to be attained.

The composition mixture of dry air with O_2 and N_2 gases is responsible for the fast recovery. Now we can be allowed to the examined gas with necessary concentrations onto V_2O_5 thin films. The change in the resistance of the sample was checked by employing a digital source meter. After successive measurements, pure air was passed into the chamber then target gas was injected into the box to obtain the desired concentrations. The selectivity studies were carried out on V_2O_5 thin film under different

concentrations of H₂ (50–500 sccm) in the presence of other gases with an operating temperature of 27° C.

3. Material characterization

3.1. Optical properties

Figure 1(a) shows that in V₂O₅ thin film deposited at room temperature, the optical transmittance was around 78% in the visible region. For the sample grown at 350° C, the transmittance increased dramatically, and colour of the film was modified from light yellow to yellow. This enhancement in transmission is due to the decrease in the optical band as evident from Tauc's plots. Using these plots, we have calculated indirect band gap energy from the linear extrapolations of the plots of $(Ah\nu)^{1/2}$ versus $h\nu$ as shown in Fig. 1(b). The S3 sample shows a band gap energy of 2.20 eV and substrate temperature increases to 350° C for the S4 sample of optical band gap energy decreases. The formation of the V₂O₅ phase is indirect from the calculated band gaps, range of 2.20 and 2.60 eV. The band gap energy is reported well [21–22] and is comparable to 2.3 eV, measured by optical spectroscopy [23]. In general, metal oxide semiconducting thin films have direct or indirect band gaps depending upon the growth conditions.

3.2. XRD characterization

The X-ray diffraction technique was to characterize sputtered V₂O₅ thin film. The XRD diffraction spectrum was analyzed from 10 to 80° and with different oxygen partial pressure and also with substrate temperature on the film composition was investigated. Figure 2 explored XRD patterns of as-grown V₂O₅ thin film and it was noticed that polycrystalline nature without any additional defeats. All the diffraction peaks are listed using Joint Committee on Powder Diffraction Standards (JCPDS card No, 85-06011). The diffraction peaks appeared around 20.27°, 27.26°, and 41.57° corresponding to (001), (110), and (200) miller index planes. With increasing substrate temperature, crystalline size also increased. Average crystalline size (D) = $0.89\lambda/\beta\cos\theta$, where λ - monochromatic wavelength of X-rays, β - full-width half maximum, and θ -diffraction angle. Among all V-O systems, V₂O₅ is a highly stable phase [25, 26].

3.3. Raman Spectroscopy

The structural information of V₂O₅ thin film is investigated using Raman spectroscopy. Figure 3 shows a Raman study of S3 of V₂O₅ thin film showing sharp peaks features which have active Raman active modes observed at a Raman shift of 141 and 990 cm⁻¹. The peaks that appeared are most likely attributed to prominently the V₂O₅ phase. The peak that appeared at 395, 523, 690, and 990 cm⁻¹ are amorphous in nature. The peaks are obtained at about 144 cm⁻¹ in the lower-level frequency region and 990 cm⁻¹ in the high-level frequency region in rigid layer-like mode and vanadyl mode respectively. The VO_x states together with V₂O₃, VO₂, V₆O₁₃, and V₂O₅ are previously reported and discussed using the Raman study [27].

3.4. Morphology studies

Figure 4a, b, and c explored scanning electron microscopy images of the S3 sample. The S3 sample was prepared at room temperature conditions and the image showed that a small flower-like morphology is observed. The morphology study S3 sample was most suitable for gas sensing because it has more porosity. Vanadium oxide fabricated by a two-step calcination process at 500°C tells very fine grains with irregular shapes between the wavelength range 220 nm to 380 nm sizes surrounded by an amorphous nature [28].

3.5 EDS Analysis

Figure 5 shows a typical EDS analysis of the V_2O_5 thin film which indicated that the material is contamination free with other elements during the sputtered process and also table inserted in the figure shows the details composition of the V_2O_5 thin film. From EDX analysis the following elemental composition contains V, O, Na, Mg, and Br the amount of 28.8, 33.9, 26.0, 5.0 and 1.1 A% respectively. V and O revealed V_2O_5 sputtered thin film.

3.6. Temperature dependence resistance studies

The temperature depends resistance measurement was done using Keithly digital multimeter. The initial resistance of the materials was recorded at room temperature conditions. The response was recorded with an increase in substrate temperature with a vacuum condition. The resistance changes of V_2O_5 film deposited at room temperature and of those heated from 27° C to 350 °C were measured. It was observed from Fig. 6 shows that both samples S3 samples show a decrease exponentially in resistance with increases in temperature. This is a very typical behaviour in semiconducting materials. V_2O_5 is an n-type semiconducting thin film whose resistance decreases when the state is reduced to V^{2+} during interaction with reducing gases. The properties of semiconducting V_2O_5 result mainly from oxygen non-stoichiometry due to the concentration of oxygen vacancies. The transition of metal oxide thin films was accounted for through semiconductor-to-metal transition [29].

Figure 7 shows the current-voltage characteristics of V_2O_5 thin films with and without hydrogen gas (100sccm) at room temperature in dry air. During hydrogen allows V_2O_5 thin film resistance decreases due to an increase in free charge carriers.

4. Gas sensing studies

V_2O_5 thin films were a vital application in gas sensing measurement because of numerous studies on vanadium oxide's chemical sensing properties. Vanadium oxide has shown hopeful results in the detection of ammonia [30] and ethanol [31]. In general, most of the journals published, major problems related to vanadium oxide thin films as gas sensing properties are narrated to vanadium oxides with good stability. The vacancies of oxygen in the material are created a vanadium-oxygen non-stoichiometry structure. The V_2O_5 thin film gas sensitivity was measured by observing the change in resistance when

exposed to hydrogen gas. During sensing of hydrogen gas, the capability of V_2O_5 thin films can be described employing surface adsorption and desorption kinetics of H_2 and O_2 gas. When oxygen is adsorbed on the V_2O_5 surface in the air atmosphere, the charge is transferred between the V_2O_5 and the oxygen molecules. Due to charge transfer, the oxygen molecule decays into negatively charged oxygen species [O^- , O^{2-} , and O_2^-] by attracting conduction band electrons of V_2O_5 .

5. Results and discussion

The V_2O_5 sputtered gas sensor undergoes H_2 gas sensing measurements at $27^\circ C$ with H_2 varying from 50 sccm to 500 sccm combined with 30% relative humidity. The advancement of change in resistance of the V_2O_5 sensor versus different concentrations of 50–500 sccm H_2 is shown in Fig. 6. The sensor response was allowed to maintain a constant value for each increment of the gas concentration. It was observed to gradually increase along with the increasing concentration of H_2 . The decrease in electron concentrations in V_2O_5 thin film is due to electron transfers from conduction to chemisorbed oxygen. In some reported papers, n-type metal oxides have increased in resistance [32]. Figure 8a-d shows the hydrogen transient response for a 50–500 sccm hydrogen gas flow at an operating temperature of room temperature ($27^\circ C$). A major change in the resistance can be viewed when the V_2O_5 thin films were exposed to hydrogen and dry air. During this time, charge flow increases with respect to H_2 , and it decreases when exposed to dry air, and response and recovery rates are remarkable. Figure 7a shows 50 sccm of H_2 concentrations along with 1000 sccm dry N_2 when exposed to H_2 in V_2O_5 thin films resistance was decreased rapidly after successfully completing four cycles. For the successive methods, we calculate the gas response of sensor 1.42, and response time and recovery time are the 70s and 132s. Similarly, Fig. 7b shows an H_2 concentration of 100sccm of H_2 flow gas response 1.60, and response and recovery time are 59s and 137s. The response time decreases from 70s to 59s whereas H_2 concentrations increase from 50 sccm to 100 sccm. Figure 7c shows 200 sccm of H_2 flows gas response of 1.866 and response time and recovery time are 87s and 218s respectively whereas H_2 concentration increases gas response and recovery time also increases. Figure 7(d) shows H_2 concentrations of 500 sccm flows with dry N_2 when exposed to H_2 resistance of S3 sensor decreases rapidly after successfully completing four cycles gas response of 1.90 and response time and recovery time are 65s and 202s respectively. Specifically, the sensor (S3) showed an extremely linear sensing performance with increases in gas concentration increases. Figure 9(a) explored the sensitivity of the V_2O_5 gas response of 50 sccm, 100 sccm, 200 sccm, and 500 sccm H_2 gas concentrations were measured around 1.42, 1.60, 1.86, and 1.90 respectively, and hence H_2 concentration increases sensitivity gas response also increases. Figure 8(b) explored response time and recovery time for different concentrations; their values are in Table 2. Meanwhile, adsorption and reaction occurred immediately at higher temperatures response time analysis apart from observing the sensing abilities of the different concentrations of H_2 . The sensor was exposed to gas of 100 sccm concentration at a constant flow rate at its operating temperature. The sensor response was observed to gradually increase along with increasing H_2 in gas flow.

Table 2 Variation of gas response, response and recovery time with different concentrations of H₂

H₂ concentrations (sccm)	Gas response	Response time (s)	Recovery time (s)
50	1.42	70	132
100	1.60	59	137
200	1.86	87	218
500	1.90	65	202

5.1. Stability and Selectivity

In the final step, the stability and selectivity of the V₂O₅ thin films were also evaluated. This study considers the stability of the sensor to keep its properties stable for a long time of 30 days as shown in Fig. 9.c, which is the best sensor action when kept at an H₂ concentration of 100 sccm significant resulting that no change of sensitivity remained at almost 1.62 after 30 days. Figure 10 shows the selectivity behavior of sensors based on V₂O₅ thin films by looking at the sensitivity of produced sensors to ammonia, acetone, ethanol and toluene, and hydrogen gases at room temperature was compared. The concentration of each gas was around 100 sccm and the gas response to ammonia, acetone, ethanol, and toluene gases was very low compared to hydrogen.

6. Conclusions

V₂O₅ thin films were sputtered using a DC Magnetron sputtering method at different substrate temperatures with various oxygen flow rates. The room-temperature sputtered V₂O₅ thin film of structural, morphological, optical, and electrical properties are systematically investigated. X-ray diffraction studies of V₂O₅ thin films have shown polycrystalline nature. The morphological investigations revealed small flower-like morphology in the film deposited at room temperature. The band gap energy of 2.2eV of V₂O₅ thin film was calculated and also decreased with increasing substrate temperature. Our results showed that V₂O₅ thin film samples of S3 for H₂ sensing at room temperatures with good sensitivity and selectivity. The response is approximately linear within a range from 50 sccm to 500 sccm. A V₂O₅ thin film which is a room-temperature sputtered thin film has exhibited better sensitivity towards hydrogen at room temperature. It showed better stability and good response and recovery time observed.

Declarations

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Author contributions

All authors contributed to the study conception and design, Material preparation, data collection, and analysis were performed by A. Paramesvaran and M. Balachandramohan. The first draft of the manuscript was written by A. Paramesvaran and all authors commented on the previous version of the manuscript. All authors read and approved the final manuscript.

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Availability of Data

The datasets analyzed during the current study are available from the corresponding author upon reasonable request.

Declarations

The authors declare that there is no conflict of interest.

Ethical Compliance

The authors declare that there is no ethical compliance.

Conflict of interest

The authors declare that there is no conflict of interest.

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Figures

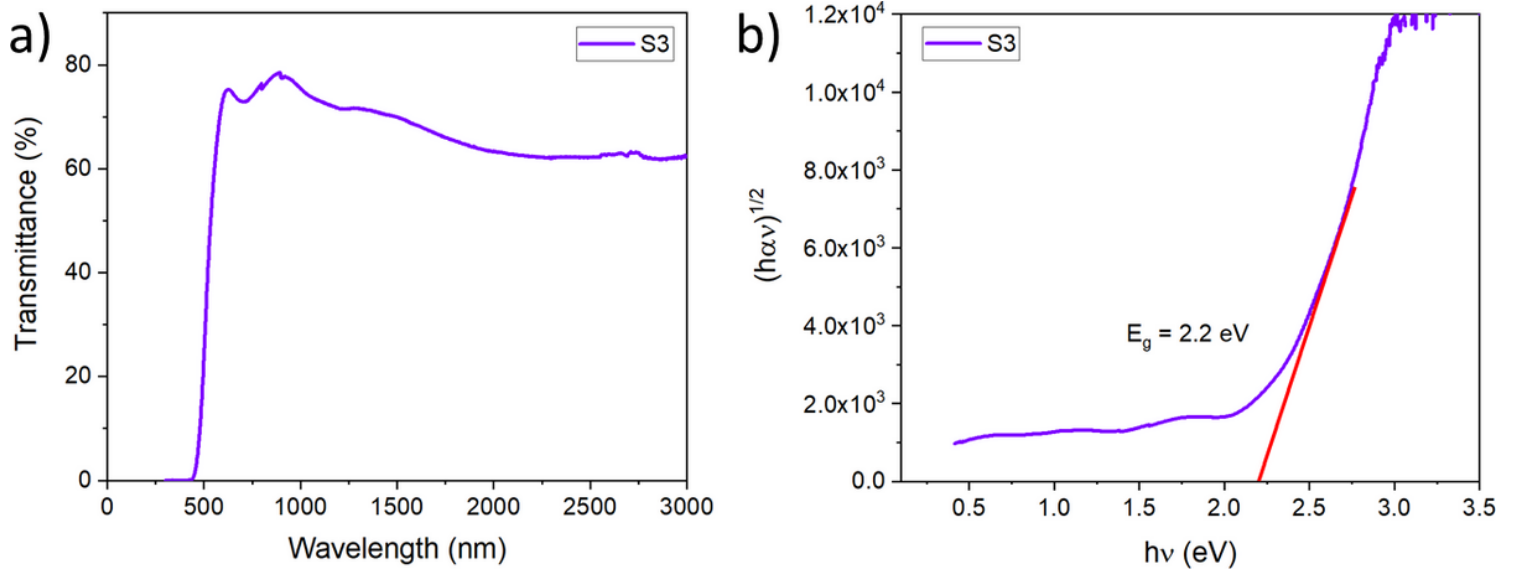


Figure 1

(a) optical transmittance spectra, and (b) Tau's plot calculated from the transmittance of the V₂O₅ thin film

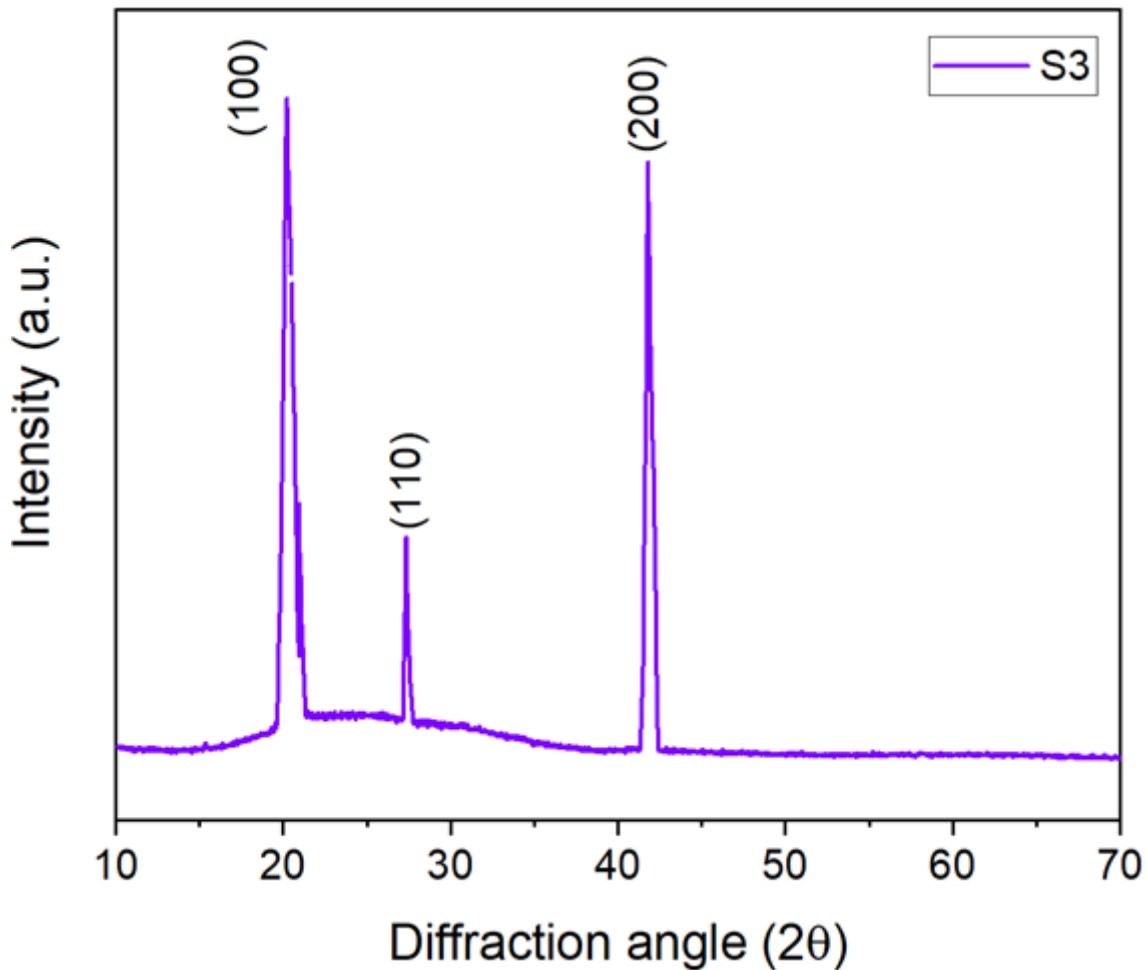


Figure 2

XRD pattern of the V_2O_5 thin film

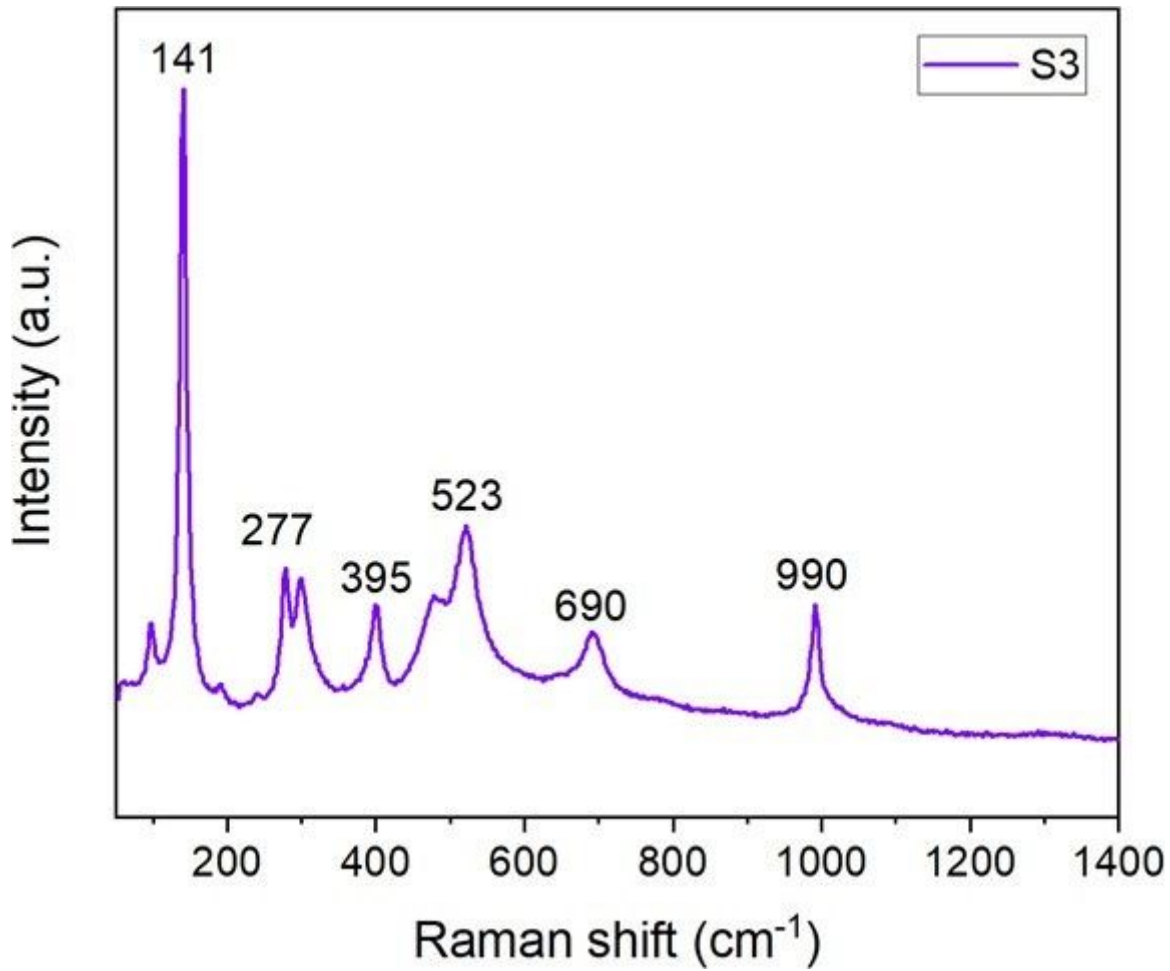


Figure 3

The Raman spectrum of the V_2O_5 Thin film

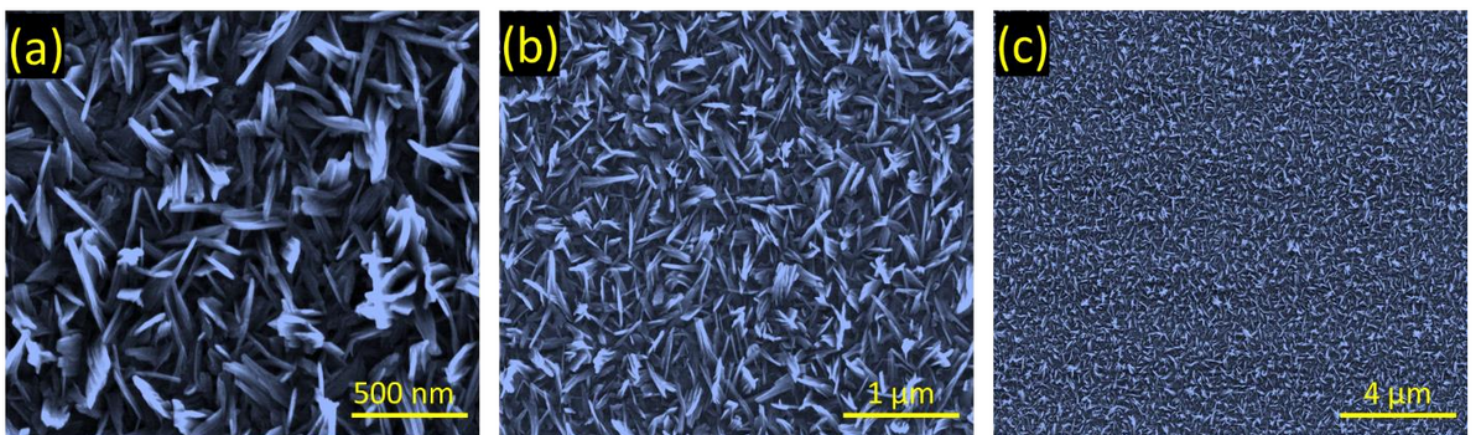


Figure 4

SEM image of the V_2O_5 thin film

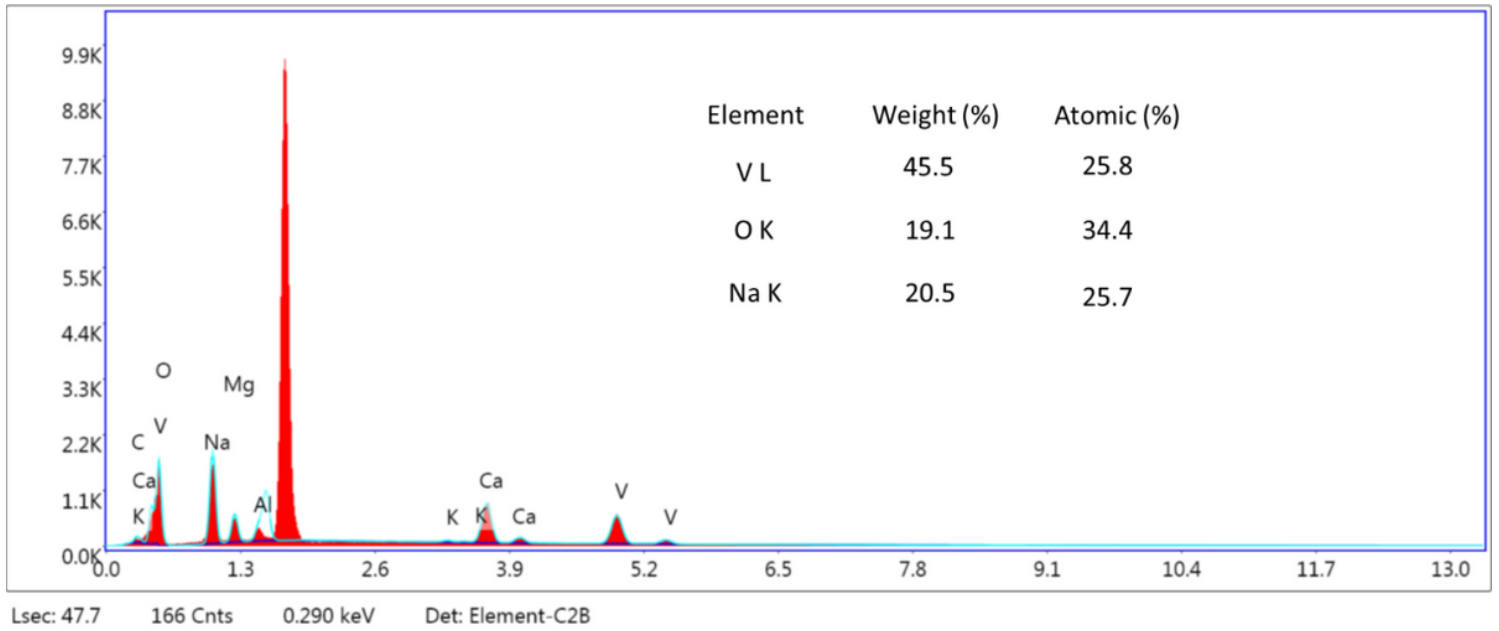


Figure 5

EDX -Compositional analysis of the V_2O_5 thin films

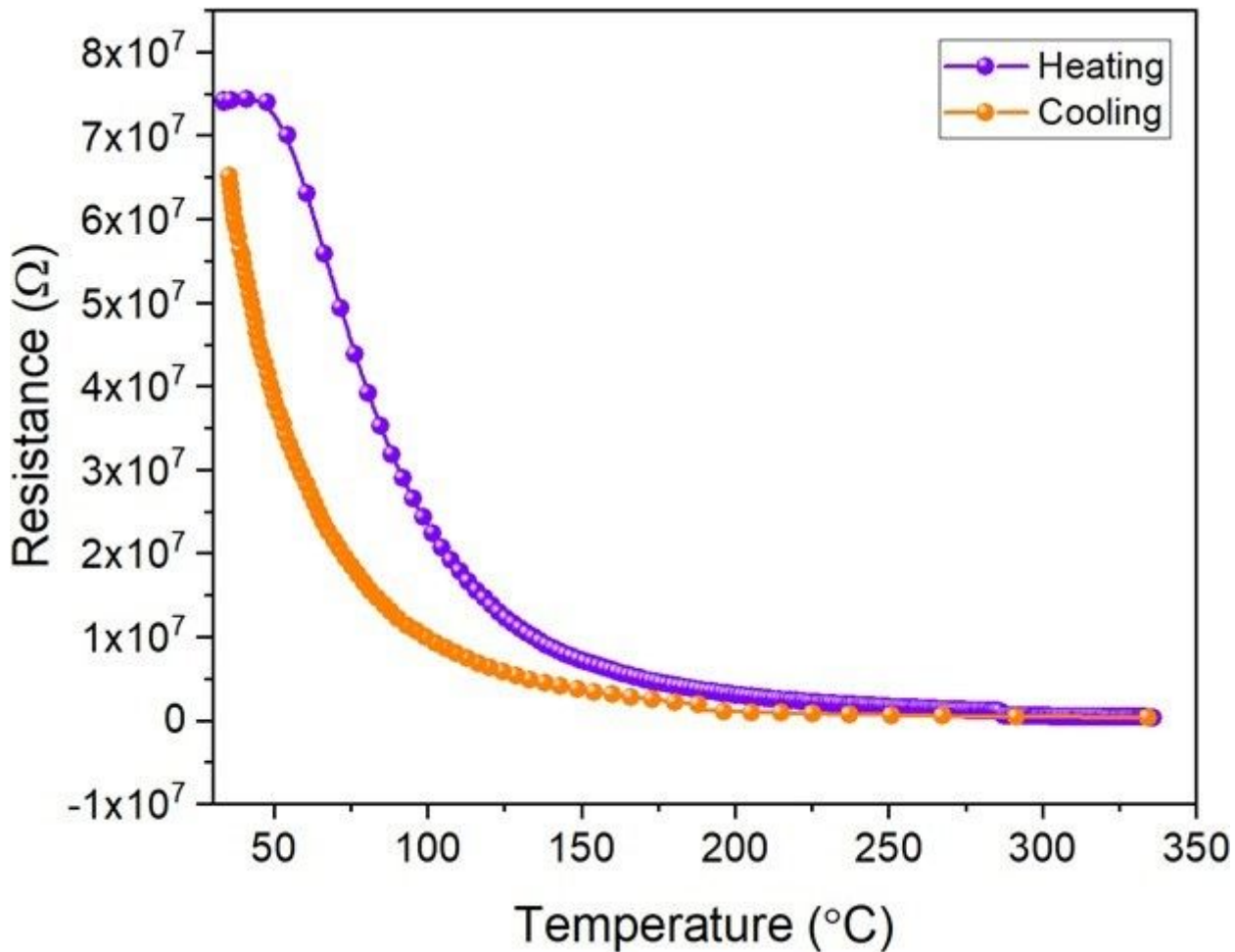


Figure 6

Resistance-temperature response of the V_2O_5 thin film

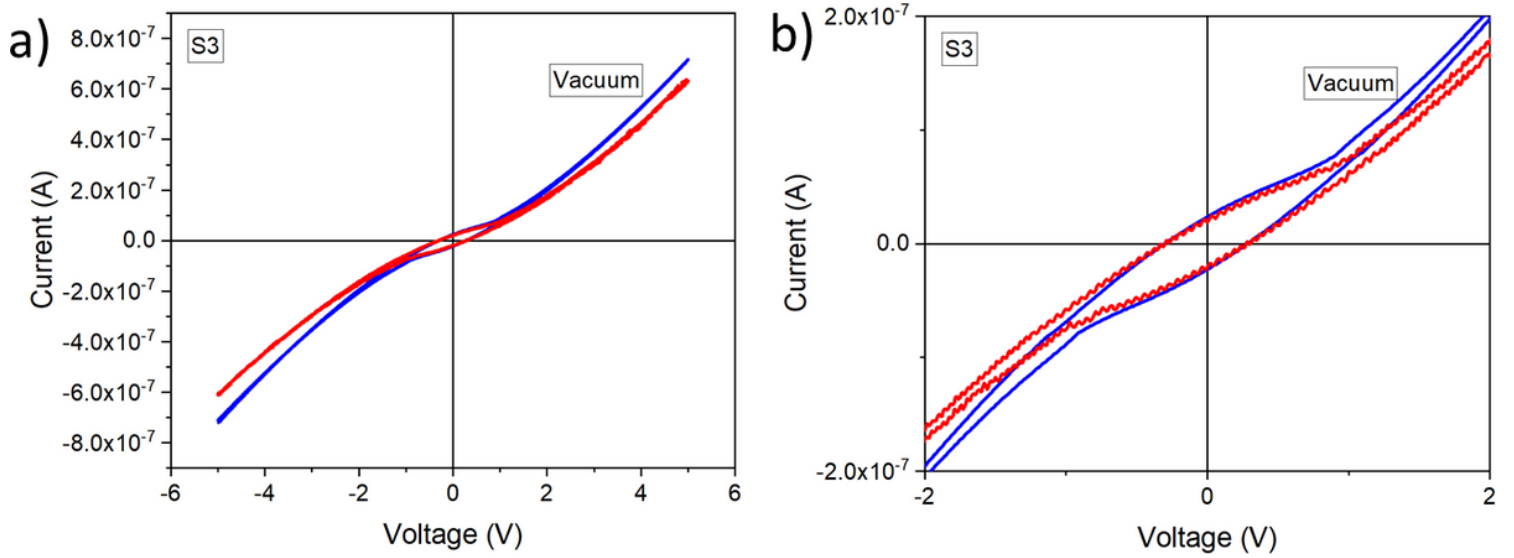


Figure 7

a & b are the V-I Characteristics of the V_2O_5 thin film

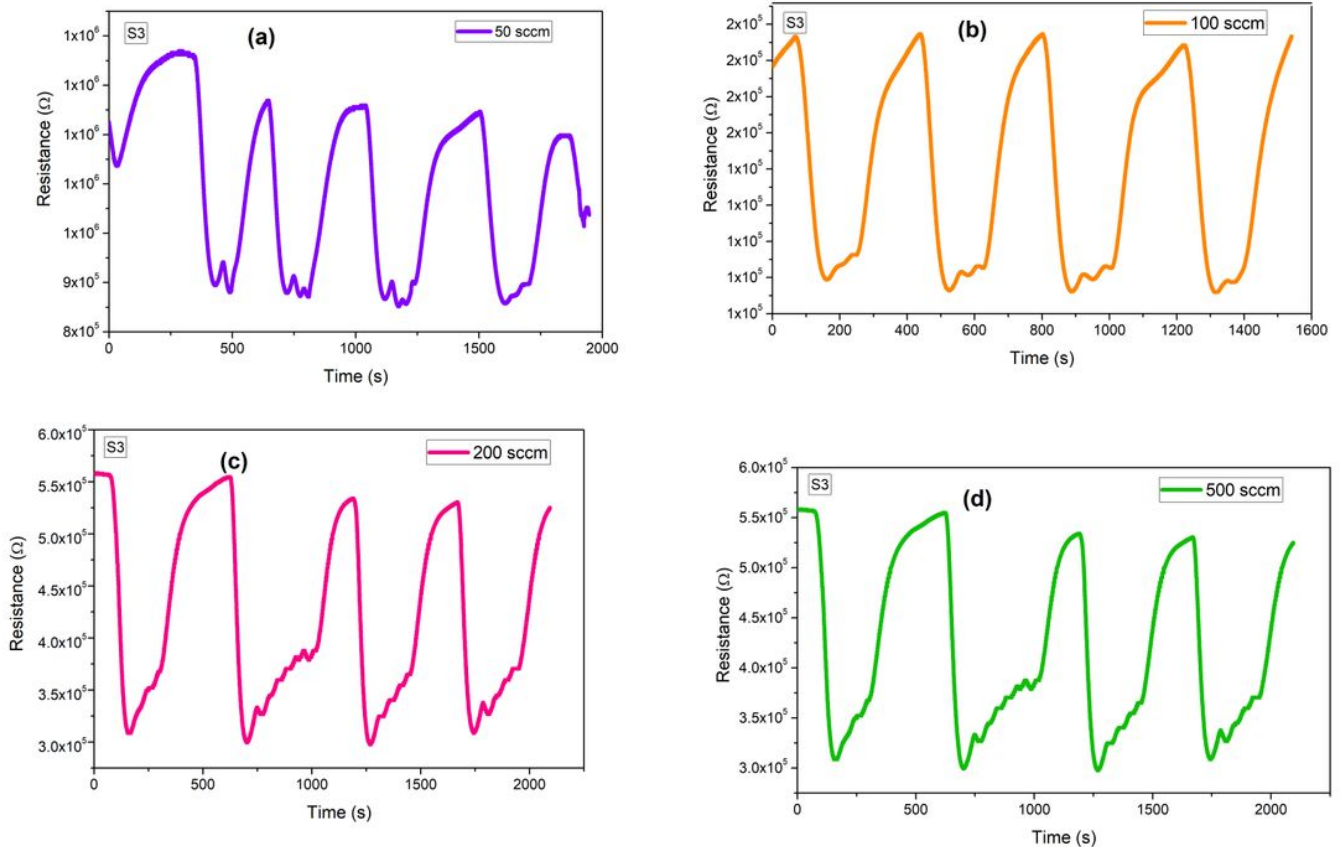


Figure 8

Hydrogen sensing response of the V_2O_5 thin film. Plots in Hydrogen flow rate of a) 50, b) 100, c) 200, and d) 500 sccm

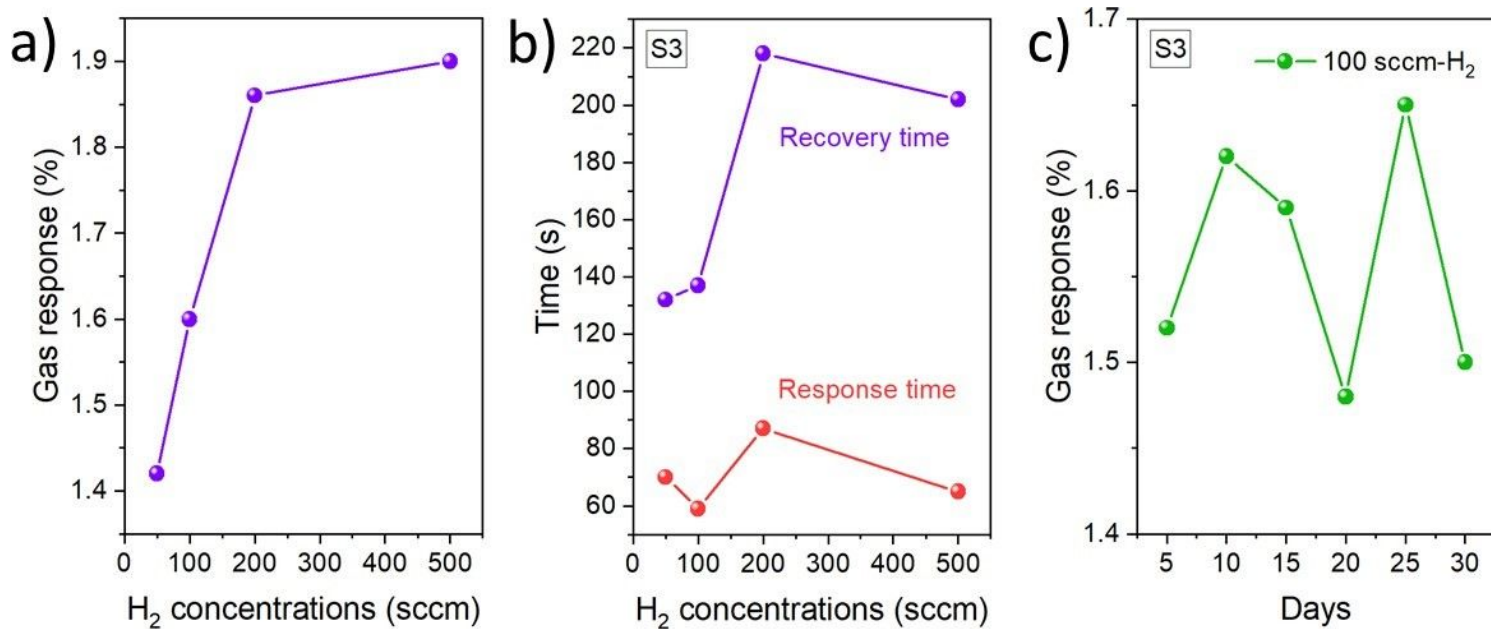


Figure 9

(a) Gas response for different concentrations of H_2 (b) response and recovery time for various concentrations of H_2 (c) Gas response of 100 sccm H_2 for 30 days

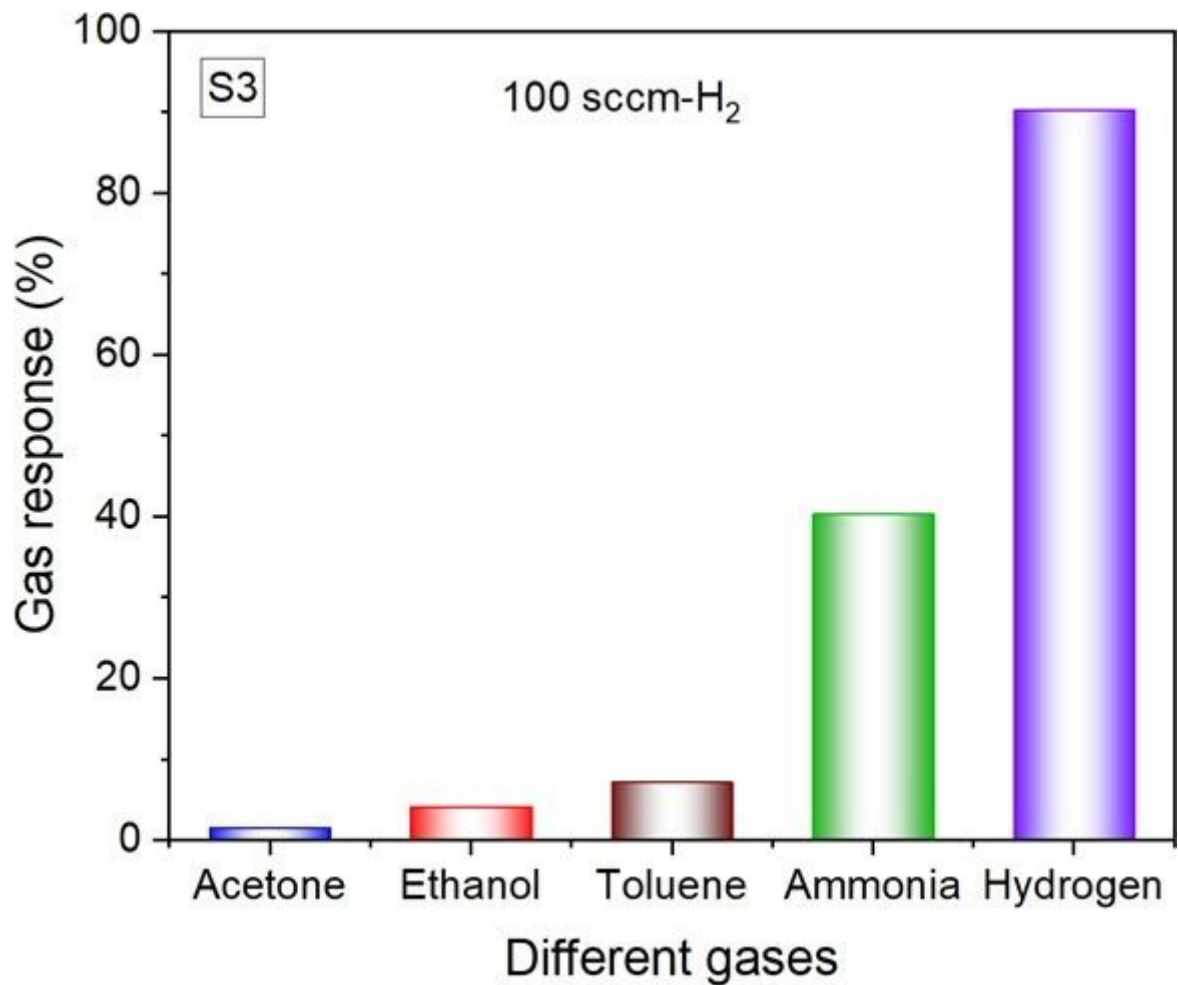


Figure 10

Selectivity behavior of the V₂O₅ thin films

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