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Hydrothermal Synthesis of Tungsten Oxide (WO_3) for the detection of NO_2 gas

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Abstract

Inorganic materials play an important role in development of chemiresistive gas sensors. We have prepared tungsten oxide (WO_3) by hydrothermal method at 150°C and it used for gas sensing applications. The prepared sensor film was characterized by X-ray diffraction (XRD), Raman Spectroscopy, X-ray photoelectron spectroscopy, Field Emission Scanning Electron Microscopy (FESEM) and contact angle. Developed tungsten oxide (WO_3) sensor film works excellently at 200°C operating temperature and shows higher response to NO_2 gas. The chemiresistive metal oxide sensor is highly stable, reproducible and has short response and recovery time.

Key words: WO_3 sensor films; XRD; RAMAN; XPS; FESEM; NO_2 sensing

Introduction

Development of sensor for practically detection of poisonous gases below their hazardous limit is a major concern of recent researchers. To date variety of metal oxide based sensors are used for detection of such poisonous gases but some of the sensors detect gas at higher concentration. The sensor explained by waghule et. al shows response above 100ppm [1]. Detection of gas at higher concentration limits the use of material for sensing applications, therefore it is essential to fabricate a sensor which can detect gas below its hazardous limit decided by EPA and have long term stability. Tungsten trioxide (WO_3) is commonly known n-type semiconductor, it has many exceptional properties resulted in wide applications in many areas such as gas sensors, electrochromic devices, rechargeable lithium batteries, photocatalysts, information displays, field-emission devices, solar-energy devices and smart windows [2-4].

In technological applications and fundamental scientific interest one of the most challenging issues is to develop innovative methods for the preparation of nanomaterials, as well as the modification of their size and morphology. Varieties of methods are presently known for synthesis of nanomaterials. In this paper we are reporting the preparation and characterization of WO_3 sensor as active material among the metal oxides.

2. Experimental

2.1 Preparation of WO_3 sensor films

Initially 2.31 gm of sodium tungstate ($Na_2WO_4{\cdot}2H_2O$) was dissolved in 70 ml deionized water under vigorous magnetic stirring till get clear solution for 30 min. Then 3M HCl was added dropwise into the clear solution to obtain acidified solution of pH 2. A newly formed pale yellow suspension was transferred into 100 ml Teflon-lined stainless steel auto clave. Then the hydrothermal growth was carried out at 150°C in a sealed Teflon-lined autoclave by immersing the glass substrates (10 **X** 10 mm^2) in the precursor solutions. After 24 hours the reaction was finished, the autoclave was cooled to room temperature naturally. Subsequently, the deposited substrates were rinsed repeatedly with deionized water and dried in air for further characterization.

3. Results and discussion

The crystalline phase of prepared WO_3 film was determined using X-ray powder diffraction (XRD) analysis. X-ray pattern in Fig. 1 (a) shows sharp and strong peaks, signifying the high degree crystallization of the sample. All the observed diffraction peaks can be indexed to the hexagonal phase of WO_3 JCPDS card no. 75-2187 with lattice constants of a=b= 7.298 Å and c= 3.899 Å. No noticeable additional diffraction peaks arising from other phases, suggesting impurity free WO_3 produced during reaction.

Fig. 1 (b) shows the Raman spectrum of the WO_3. The intensity band at 931.41 cm^{-1} is assigned to stretching vibrations of W=O [5]. The band centered at 812.73 and 671.68 cm^{-1} is attributed to stretching vibrations O-W-O [6-7]. The peaks at 243.98 and 327.26 cm^{-1} can be ascribed to W-O-W bending vibration mode of the bridging oxygen. The peak at 109.44 cm^{-1} can be assigned to the lattice vibration of crystalline WO_3 lattice [8].

The XPS survey spectra showed in Fig. 1(c) exhibits the presence of W, C and O.

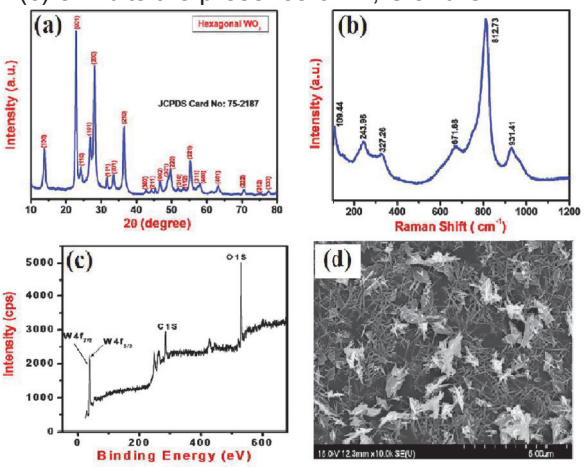

Fig. 1(a) XRD, (b) Raman, (c) XPS Survey spectra and (d)) FESEM of the WO_3.

The morphology of the WO_3 was demonstrated in Fig. 1(d). On account of FESEM check, randomly distributed nanorod morphology was estimated with average diameter of the uniform nanorods was about 80 nm and length upto 2 µm. Therefore one can conclude that aspect ratio of 25. Inset of

3.4 Gas sensing measurement

Fig. 2(a) shows the resistance vs time graph of WO_3 sensor measured at 200°C operating temperature. The response of WO_3 sensor was performed by measuring the resistance change on exposure to 100ppm of NO_2 gas. When the sensor is exposed to NO_2, amazingly the resistance of sensor increases rapidly reaches highest value and starts decrease immediately. WO_3 is n-type semiconductor majority charge carriers are electrons, NO_2 is an oxidizing gas and it have ability to accept electrons. When the WO_3 sensor is exposed to NO_2 gas, it donates electrons to NO_2, due to this charge carrier density decreases and consequently resistance of sensor increases. The sensor shows fast response and recovery times of 3s and 89s after exposure to NO_2 gas. This shows that there is fast rate of reaction on the surface of the WO_3 sensor. Fig.2(b) shows the response vs time graph of WO_3 sensor.

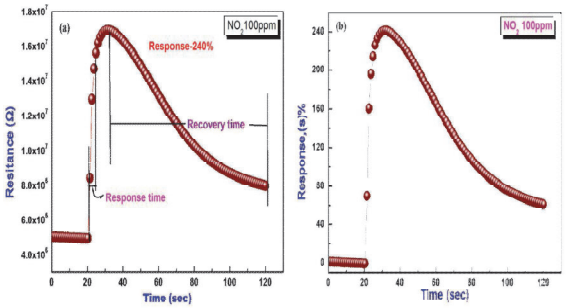

Fig.2(a) Resistance vs time and (b) Response vs time graph for 100ppm NO_2 gas

4. Conclusions

The WO_3 sensor was fabricated successfully on glass substrate by hydrothermal method. Prepared sensor was characterized by various techniques and sensing performance was measured to NO_2 gas. The WO_3 sensor shows excellent response of 240% with short response and recovery times.

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