Effect of PEG Addition on an SnO₂ Gas Sensor Fabricated Using Spin Coating

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Abstract

Tin dioxide (SnO₂) and tin dioxide: polyethylene glycol (SnO₂: PEG) nano-composite thin films, at 10 and 20 wt % PEG, were fabricated by spin coating techniques from a mixed solution of ethanol and water. The structural, morphological, and optical properties of the prepared samples were examined to study the effect of PEG addition on SnO₂ thin film properties. X-ray diffraction shows a polycrystalline structure for all samples, where the crystalline size reduced from 8.3 nm (for the pure sample) to 7.2 nm (for the 20% PEG sample). The addition of PEG with the precursors significantly affected the growth behaviour of SnO₂ nanoparticles, where the nanoparticle diameter reduced and the connection between them was enhanced forming a continuous structure with pores that enhance gas sensitivity. The hydrogen (H₂) gas sensitivity, for the prepared SnO₂ and SnO₂:PEG samples, increased by increasing the PEG content from 0 to 20% by about four times. The optimum sensitivity was at the working temperature of 100 °C. The gas sensitivity was exponentially dependent on H₂ gas concentration across a concentration range of 100 to 4000 ppm.

Keywords

gas sensor, tin dioxide thin film, spin coating, PEG

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1. Introduction

Hazardous gases include those that are highly toxic or flammable. To ensure occupational safety on work sites, any gas leakage must be detected at the lowest possible concentrations. Hydrogen (H_2) is an odourless and colourless gas, and consequently it is not possible to detect it by human senses. In addition, indoor leaks of H_2 gas accumulate at the ceiling, away from the places to which people are typically exposed [1].

The minimum concentration of H_2 at which an explosion can occur in the air is 4% [2], which is below the concentration of hydrogen which would cause asphyxiation. Therefore, the hazard point for hydrogen concentrations depends on the lowest concentration of hydrogen in the air at which an explosion can occur. H_2 gas is naturally present in ambient conditions at a concentration of 0.5 ppm [3]. It can also be released as a by-product of charging batteries or by passing water vapour over heated iron. It is also found in aircraft at about 100 ppm concentrations [1]. For these reasons, hydrogen sensors are used to detect hydrogen leaks at job sites and, due to their critical nature in terms of safety, researchers are constantly studying new devices for H_2 gas sensing [4].

Tin oxide (SnO_2) attracts a lot of attention with regard to its use in various fields as gas sensors for different gases such as acetone [5], ammonia [6], and hydrogen [7]. SnO_2 is usually an n-type semiconductor. It has a rutile crystal structure and a large energy gap of 3.6 eV. There are numerous methods to fabricate SnO_2 thin film, for example, hydrothermal [8], chemical vapor deposition [9], and sol-gel techniques [10]. Among these methods, the sol-gel technique has been widely explored for its advantages such as low reaction temperature, low cost, and for being a straightforward process [11]. However, the thin films produced by the sol-gel technique are subject to the formation of cracks, so the initial components are mixed with certain materials to improve the final properties [12]. SnO_2 nanostructure can be fabricated as different structures such as nanofibers [13], nanobelts [14], etc.

If a metal oxide semiconductor is exposed to a reducing gas, due to its catalytical combustion with the adsorbed oxygen species, it releases electrons back to

the conduction band of the semiconductor surface [15]. This process increases the conductivity of n-type semiconductors due to the increment in the charge carrier concentration or narrowing of the depletion layer near the surface, which in turn increases the mobility of charge carriers [16]. The gas sensor sensitivity (S) is measured using the equation $(R_{air}-R_{gas})/R_{air}$, where R_{air} and R_{gas} are the sample resistance in clean air and on exposure to the mixed gas at the investigated concentration, respectively [17]. SnO_2 can be functionalized or doped with different substances to enhance its gas-sensing properties, such as silver nanoparticles [18], carbon nanotubes [19], copper [20] nitrogen [21], etc.

Numerous research efforts have confirmed the improvement in gas sensors by means of “nanoscale engineering”. For example, Al-jawad et al., (2003) studied the effects of annealing temperature on the nanocrystalline structure of SnO_2 thin films prepared by sol-gel and chemical bath deposition for carbon monoxide (CO) sensors [22]. Shoyama and Hashimoto (2003) studied the effects of the addition of PEG on the microstructure and CO sensing of SnO_2 thin films also prepared by the sol-gel method [23]. Sakthivel and Shameer (2015) examined the effects of adding PEG on the H_2 gas-sensing behaviour of composite coatings of magnesium oxide and aluminium oxide [24].

Nunes et al. (2019) reviewed recent metal oxide nanostructures as used for sensor applications for different gasses, humidity sensors, UV sensors, and biosensors. The research mentioned various studies concerned with hydrogen gas sensors based on different metal oxides such as $Au-TiO_2$, TiO_2-SnO_2 , $Pb-WO_3$, Cu_2O , and V_2O_5 [25].

Adding PEG to SnO_2 plays a vital role in nucleus formation and in directing crystal growth [26]. In this paper, we examine the effects of adding PEG to the initial material at different ratios with regard to subsequent structural properties, surface morphologies, and optical properties of the SnO_2 thin films prepared by spin coating. Also, the effects of this ratio on the H_2 gas sensitivity were studied. The H_2 gas-sensing mechanism will also be discussed.

2. Experimental

SnO_2 thin films were synthesized on corning glass via spin coating using tin (II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$) with a purity $\geq 99\%$ (as a precursor) and

polyethylene glycol 600 (as a surfactant) from Sigma–Aldrich Co. with no further purification. $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ powder was dissolved in 1:1 absolute ethanol and distilled water at a 0.2 M concentration by stirring vigorously at 30 °C until a clear solution was formed. An alcoholic solution of PEG was added to the solution at different weight ratios (0, 0.1, and 0.2 PEG/ $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$). The sol was aged for 5 h at a temperature of 60 °C to transform into a gel. The films were deposited as three layers by spin coating on glass slides at 3000 rpm and were then dried at 60 °C for 3 min after each coating had been deposited. The films were then thermally treated at 400 °C for 1 h. Three samples were prepared for each PEG ratio to ensure the reliability of the obtained results. The prepared films were homogeneously distributed over the surface, especially for samples with a 20% PEG content. The prepared films were examined by X-ray diffraction (Shimadzu XRD 6000), atomic force microscopy (CSPM contact mode spectrometer), scanning electron microscopy (JSM-7600F by JEOL Ltd), and UV–visible absorption (SP-8001 spectrophotometer).

To fabricate gas sensor devices, silver paste electrodes were screen printed in a comb-like pattern on the sample's surface in the dimensions illustrated in Fig. 1. Gas sensor specifications were tested in a homemade chamber on a thermo-controlled hot plate. The gas mixed at different concentrations in a normalized mixing chamber of 2000 ml volume, as

illustrated in the schematic view in Fig. 2. The H_2 gas was injected into this chamber by means of a syringe in specific quantities to achieve the different concentrations required. The gas-air mixture was pumped into the test chamber over the sample. The change of sample resistance with exposure to the gas was observed using an electronic circuit connected to a computer. The gas sensitivity measurement was repeated for the three batches, where very little difference was observed between each. The gas sensitivity results listed in the search refer to the average values.

3. Results and discussions

Figure 3 illustrates the X-ray diffraction patterns recorded for the as-deposited SnO_2 thin film prepared on glass slides and annealed at 400 °C. It seems that the as-prepared sample has low crystallinity when converted to a polycrystalline form via annealing at 400 °C that corresponds to a SnO_2 tetragonal crystal structure identical to standard card No. 96-210-4744. The peaks appeared as broad features located at diffraction angles of $2\theta = 26.569^\circ$, 33.859° , 37.939° and 51.759° , corresponding to the (110), (101), (200), and (211) crystal planes, respectively.

Figure 4 illustrates the typical X-ray diffraction curves for the SnO_2 samples prepared on glass with different PEG ratios (pure, 10%, and 20% wt. PEG) and sintered at a temperature of 400 °C. Polycrystalline structures appeared for all samples. The peak broadening was calculated via Lorentzian fitting using the X Powder software (a software package for X-ray diffraction analysis, version 2004.04.50) after subtracting the background, as illustrated in the inset figure. Peak broadening was found to decrease with increasing PEG ratio, indicating the decrease in the crystallite size, as revealed by Scherrer's formula.

Table 1 shows the diffraction angles, inter-planer spacing (d_{hkl}) calculated using Bragg's law compared with the standard d_{hkl} values, full width at half maxima (FWHM), crystalline size (C.S), and corresponding Miller indices for the XRD peaks in the three samples. There is a good match between the calculated and standard d_{hkl} values. Likewise, the calculated crystallite size for the preferred direction, with the highest intensity, was found to reduce from 8.3 nm (in the sample prepared without PEG) to 7.2 nm (at the 20% ratio).

Figure 5 shows the AFM images for the SnO_2 samples prepared at different PEG ratios and the associated histogram of the particle diameter distribution (calculated using the ImageJ software). The pure

Fig. 1. The components and the size of the sensor with the electrodes.

Fig. 2. Schematic of the chemoresistance gas sensor testing setup. The sensor is placed on a heater and attached to a thermal controller via a thermocouple. The gas under test is injected into the mixing chamber, which is mixed fully using a fan. The mixed gas flow is cycled over the sample using a small pump controller by a gas timer controller. The variation in sample resistance is transmitted by pins to a multimeter connected to a computer.

sample has a particle diameter with a wide distribution ranging from 85 to 243 nm and an average diameter of 141.55 nm. Adding the PEG at 10% and 20% ratios with the initial material causes a reduction in the

average diameters for each to 89.9 nm and 67 nm, respectively. The particle size appeared with narrow and homogeneous distribution after adding the PEG. Table 2 shows the range of minimum and maximum

Fig. 3. XRD patterns for SnO_2 thin film prepared (a) without annealing and (b) after annealing at 400 °C.

Fig. 4. XRD curves for SnO_2 thin film prepared (a) without PEG (b) with 10% wt. PEG (c) with 20% wt. PEG.

particle diameters and the average diameter for the three thin-film samples prepared at different PEG ratios. The average particle diameter significantly decreased with increasing the PEG ratio due to the difference in growth mechanism [27]. Such changes enhance the gas sensor specification, which has been confirmed in previous studies [28].

Figure 6 shows the scanning electron microscopy images for the surface of the pure SnO_2 film, and SnO_2 films with 10 and 20 wt.% PEG. There is a difference

in the particle size between samples prepared without and with PEG. The pure thin film appeared as nanoparticles with irregular shapes and sizes with an average diameter of 125 nm (as calculated by the ImageJ software) with low connectivity between each other and which may have low charge carrier mobility. On adding 10% and 20% PEG by weight to the precursor solution, nearly uniform spherical nanostructures appeared, and whose average size was decreased to about 60 nm. Grain aggregation was

Table 1

XRD peaks parameters for SnO_2 thin film deposited without PEG, and at 10% and 20% wt. PEG.

Sample	2θ (Deg.)	d_{hkl} Exp.(Å)	d_{hkl} Std.(Å)	FWHM (Deg.)	C.S (nm)	hkl
without PEG	26.569	3.3522	3.3498	0.981	8.3	(110)
	33.859	2.6453	2.6439	0.893	9.3	(101)
	37.939	2.3697	2.3686	0.903	9.3	(200)
	51.759	1.7648	1.7642	0.936	9.4	(211)
10% wt. PEG	26.580	3.3509	3.3498	1.072	7.6	(110)
	33.870	2.6445	2.6439	0.914	9.1	(101)
	37.950	2.3690	2.3686	0.925	9.1	(200)
	51.770	1.7645	1.7642	0.958	9.2	(211)
20% wt. PEG	26.591	3.3495	3.3498	1.129	7.2	(110)
	33.881	2.6436	2.6439	0.997	8.3	(101)
	37.961	2.3684	2.3686	0.978	8.6	(200)
	51.781	1.7641	1.7642	0.991	8.9	(211)

Fig. 5. The 3-D AFM images and related cumulative distribution for SnO_2 prepared (a) without PEG, (b) with 10% wt. PEG, and (c) with 20% wt. PEG.

Table 2

Minimum, maximum, and average diameters for the SnO_2 thin films.

Sample	Minimum diameter (nm)	Maximum diameter (nm)	Average diameter (nm)
Pure SnO_2	85.20	243.72	141.55
10 wt % PEG	57.26	144.42	89.93
20 wt % PEG	49.02	93.27	67.00

observed on addition of PEG, especially at 20 wt.%, and the particles were connected to each other via neck connections, forming continuous structures with cavities that increased the surface exposed to the gas sample. Such an arrangement between the nano-particles enhances the gas sensitivity, as the depletion

Fig. 6. SEM images for SnO₂ prepared (a) without PEG, (b) with 10% wt. PEG, and (c) with 20% wt. PEG.

layer of the charge carrier that forms near the surface, which increases and decreases in thickness due to gas adsorption and desorption, leads to the narrowing and widening of the channels between the nanoparticles at contact points, causing an obvious change in sample conductivity.

Figure 7 shows the initial absorption curves for the SnO₂ samples with different PEG ratios. The absorption edge appeared at about 350 nm and blue-shifted after adding PEG to the initial material. The optical band gap E_g^{opt} for the SnO₂ samples was measured via the Tauc formula the relation between $((\alpha h\nu)^2$ versus $h\nu$) as revealed in Fig. 8. The E_g^{opt} was 3.8 eV for the

sample prepared without PEG and increased to 3.9 and 3.95 eV with 10 and 20 wt.% PEG, respectively. E_g^{opt} is larger than that for bulk SnO₂ due to the oxygen vacancies [29]. The increment in band gap on addition of PEG is caused by the quantum confinement effect due to reducing the crystalline size to the nano-range [30].

The gas sensitivity was studied with the working temperature for SnO₂ and SnO₂:PEG samples against H₂ gas at a concentration of 1000 ppm, as illustrated in Fig. 9. The sensor shows a low sensitivity at room temperature. The sensitivity increased to 5.5%, 11.7%, and 20.7% at 100 °C for the three samples (Pure SnO₂, 10, and 20 wt.% PEG) as a result of the variation of

Fig. 7. Absorption curves for the SnO₂ thin films prepared (a) without PEG, (b) with 10% wt. PEG, and (c) with 20% wt. PEG.

oxygen ion species adsorbed on the active layer [31]. Increasing the temperature to 150 °C reduces the sensitivity of the samples to 3.5, 6.4, and 7.5%, respectively.

The resistance variation with time of the SnO₂ and SnO₂:PEG nanocomposites against hydrogen gas at different concentrations (100–4000 ppm) at a 100 °C operating temperature are revealed in Fig. 10. The samples exposed to H₂ gas show resistance signals of a height that is dependent on gas concentration.

Response time is the time taken for the sensor to reach 90% of the minimum resistance when exposed to H₂ gas. The recovery time is the time required to

recover to within 10% of the original baseline, when the H₂ flows off and the sensor is again exposed to clean air [22], as shown in the inset figure in Fig. 10. The response time was 23 s, while the recovery time was 34 s.

Figure 11 shows the variation in gas sensitivity of the three samples versus H₂ gas concentrations at a 100 °C working temperature. The sensitivity increased exponentially from 100 to 4000 ppm as $y = 0.0693x^{0.662}$, $y = 0.0665x^{0.754}$ and $y = 0.0795x^{0.812}$, for 10% and 20% PEG, respectively (with R² greater than 0.9). The sample prepared with 20% PEG was found to have the highest sensitivity. It

Fig. 8. $(\alpha hv)^2$ vs. $h\nu$ for spin-coated SnO₂ thin film prepared (a) without PEG, (b) with 10% wt. PEG, and (c) with 20% wt. PEG.

Fig. 9. Variation of gas sensitivity with working temperature for spin-coated SnO_2 :PEG at (a) 10% wt. PEG and (b) 20 wt. % PEG.

Fig. 10. Time variation of resistance for SnO_2 and SnO_2 :PEG sensors exposed to different H_2 concentrations for samples prepared (a) without PEG, (b) 10 wt. % PEG, and (c) 20 wt. % PEG.

Fig. 11. Variation of sample sensitivity with H_2 concentration for the SnO_2 and SnO_2 :PEG samples at prepared at 10 and 20 wt. % PEG.

is obvious that the deposition conditions influence the gas sensitivity for metal oxide thin films. This result agrees with that found by Nunes et al. [32]. Table 3 illustrates the gas sensitivities for the three samples at different H_2 concentrations compared with the average grain size calculated by AFM. It is obvious that the sensitivity is inversely related to the grain size.

Figure 12 illustrates the stability of the sensor prepared with a 20 wt.% PEG content at an operating temperature of 100 °C, showing the consistent resistance change when exposed to 1000 ppm hydrogen gas and clean air sequentially over several such cycles. The H_2 sensitivity mechanism can be assumed as follows. SnO_2 is an n-type semiconductor due to oxygen vacancies within the crystal lattices, and its sensing behaviour mainly depends on surface resistance [5]. The oxygen molecules are adsorbed on the SnO_2 surface from the surrounding air which are in turn ionized by extracting electrons and forming a surface depletion layer to a certain depth that can form potential barriers

that block the channels between the nanoparticles, hindering the movement of the charge carriers. When the sample surface is exposed to H_2 gas it reacts with these ions according to the reaction [33]:

The re-release electrons increase the charge carrier concentration and reduce the surface depletion layers, which leads to an increase in carrier mobility by reducing the barrier potential between the nanoparticles, thus reducing the resistance of the samples. The enhanced H_2 gas response of the SnO_2 /PEG-based sensor may be attributed to an enhancement to the connections between the nanoparticles, which in turn increases charge carrier mobility.

Table 4 compares the specifications of SnO_2 -based H_2 gas sensors which were optimized with the addition of different materials and prepared via sol–gel spin coating with the results of the current study. Although the sensitivity is lower than that reported previously, the optimum working temperature is significantly lower, and in addition operates over a very wide range of gas concentrations (100–4000 ppm) with lower detection limits when compared to other studies in the literature.

4. Conclusions

H_2 gas sensor devices were fabricated from an SnO_2 :PEG nanocomposite using the low-cost spin

Table 3

Gas sensitivity for H_2 at different concentrations for the SnO_2 and SnO_2 :PEG samples at 10 and 20 wt. % PEG compared with the average grain size calculated by AFM.

Sample	Grain size (nm)	Gas concentration (ppm)				
		100	500	1000	2000	4000
		Sensitivity $\pm$ 5%				
Without PEG	141.6	1.9	2.8	5.5	13.5	18.7
10%PEG	89.9	2.3	6.4	11.7	23.7	33.1
20%PEG	67.0	3.6	11.2	20.7	39.9	69.8

Fig. 12. Time variation of resistance for SnO₂:PEG sensors at 20 wt. % PEG exposed to 1000 ppm H₂ gas.

Table 4

Comparison between the specification of sol–gel spin-coated SnO₂-based H₂ gas sensors, at different addition, with the current study.

Metal oxide-addition material	Deposition technique	Temp. (°C)	Detection range (ppm)	sensitivity%	Resp. time (s)	Rec. time (s)	Ref.
SnO ₂ -carbon nanotube	Spin- coating	250	300–1500	147% at 1500 ppm	2–3	4–5	[34]
SnO ₂ -glycerin	Spin- coating	125	150–1000	195% at 1000 ppm	102.5	39.7	[12]
SnO ₂ -PEG	Spin- coating	100	100–4000	20.9% at 1000 ppm	23	34	This work

coating technique. Structural, morphological, and optical properties show clear benefits to the use of mixed ratios of PEG on the crystalline structure and surface morphology. Adding PEG played a significant role in determining the size of SnO₂ nanoparticles, which caused the reduction of particle size and which is effective at increasing the connection between nanoparticles, forming a continuous structure with cavities that enhances gas sensor specification. The energy gap increases with increasing PEG ratio. The gas-sensing examination shows that the sensitivity increased with increasing PEG content, as ranging from pure SnO₂ to 20% due to the associated reduction in grain size and change in surface morphology. The maximum sensitivity was observed at a working temperature of 100 °C. The results of the gas sensor specification, such as the sensitivity, detection limit, response time, and recovery time, were quite reasonable compared to the literature concerned with hydrogen gas sensing.

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