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Regular Article

Improved sensing response of photo activated ZnO thin film for hydrogen peroxide detection

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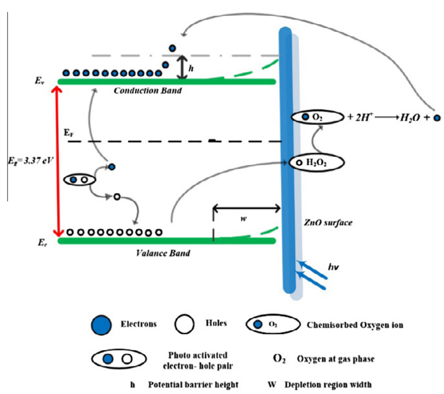
HIGHLIGHTS

- ZnO thin film was prepared by spray pyrolysis technique.
- The formation of spherical shaped nanoparticles was observed.
- The active detection range of hydrogen peroxide was found to be 10–50 ppm.
- The minimum detection limit of hydrogen peroxide vapour was found to be 10 ppm.
- The response and recovery times for 50 ppm were 115 s and 130 s, respectively.

GRAPHICAL ABSTRACT



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ABSTRACT

The nanostructured ZnO thin films were deposited using spray pyrolysis technique. Formation of polycrystalinity with hexagonal wurtzite structure was observed from the structural study. Highly dense spherical shaped nanoparticles with fine crystallites were observed from the surface morphological studies. The light induced hydrogen peroxide vapour sensing was done using chemi-resistive method and its effect on the sensing response was studied and reported.

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1. Introduction

Hydrogen peroxide is a colourless liquid with a bitter taste. It is also being manufactured in chemical industries since it is a strong oxidizer and which has been used as a bleaching agent and disinfectant [1]. Occupational Safety and Health Administration (OSHA) provides the human permissible exposure limits towards hydrogen peroxide is 1 ppm as eight-hour time weighted average (TWA) (or) 1.4 mg of hydrogen peroxide per cubic meter of air (mg/m^3) [2]. If hydrogen peroxide is inhaled or comes in contact with the eye or skin, it induces oxidative stress in the body leading to cancer, asthma, atherosclerosis and diabetes. Hence, hydrogen peroxide should be detected and monitored in a real time ambient environment. Numerous methods such as titrimetry [3],

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spectrophotometry [4,5], fluorimetry [6], chemiluminescence [7] and electrochemical biosensors [8,9] have been used to detect hydrogen peroxide. Metal oxide semiconductor based gas/vapour detections are widely used because of its real time detection. Nanostructured metal oxide semiconductor (MOS) thin films such as CdO [10], ZnO [11], TiO_2 [12], SnO_2 [13] and WO_3 [14] are being used for detecting the volatile organic compounds because of its variation in electrical conductivity towards oxygen and other species. ZnO has unique properties such as good optical transparency, high electron mobility, strong room-temperature luminescence, high refractive index, high thermal conductivity and UV protection [15]. In this work, the sensing behaviour of the spray deposited ZnO film was studied for various concentrations of hydrogen peroxide vapour under the light assistance to improve the photoelectric response.

2. Materials and methods

2.1. Preparation of sensing element

The precursor solution was sprayed on the glass substrates which were pre-heated at 230 °C by home-build spray pyrolysis unit [16]. Prior to a deposition, the chemically cleaned glass substrates were placed over the hot plate. Substrate temperature was kept at 230 °C controlled by a chrome-nickel thermocouple connected to the thermostat. A 0.1 M of zinc acetate dihydrate (Zn $((CH_3COO)_2)\cdot 2H_2O$, Sigma Aldrich, 99% Purity) was dissolved in 50 mL of deionized water and the precursor solution was sprayed over the pre-heated glass substrate by air-blast method. The spray gun was fixed at an angle of 45° with respect to solution reservoir and substrates kept at a distance of 40 cm from the spray gun. Air from the compressor was passed to air filter-cum regulator and it was used as the carrier gas in order to transform the precursor solution into fine mist. The spray rate of the solution was maintained at 3 mL/min. The spraying time was fixed as 5 s and the successive spray interval was maintained at 45 s for the formation of thin films. Paraguay et al. [17], reported the formation of ZnO through the pyrolytic reaction as denoted in the Eq. (1).

$$\begin{aligned} &Zn(CH_3COO)_{2_{(solid\ near\ substrate)}} \rightarrow 4Zn(CH_3COO)_{2_{(solid\ near\ substrate)}} + H_2O \rightarrow \\ &\quad Zn_4O(CH_3COO)_{6_{(adsorbed/substrate)}} + 2CH_3COOH_{(gas\ near\ substrate)} \\ &\quad and\ \uparrow Zn_4O(CH_3COO)_{6_{(adsorbed/substrate)}} + 3H_2O \rightarrow \\ &\quad 4ZnO_{(film/substrate)} + 6CH_3COOH_{(gas)} \uparrow \end{aligned} \tag{1}$$

2.2. Characterization

The structural analysis of spray deposited ZnO thin film was done by X-ray diffractometer (XRD, X'Pert PRO) with the scanning angle of 20–80° using $CuK\alpha_1$ radiation. The surface morphologies of the film were carried out using field-emission scanning electron microscopy (FE-SEM, JEOL-6701) and field-emission transmission electron microscopy (FE-TEM, JSM 2100, JEOL). The prepared ZnO thin film was scratched as a powder and dispersed in ethanol for TEM analysis. Optical studies were done by using UV–vis spectrophotometer (Lambda 35 with scan speed of 480 nm/min). Electron Paramagnetic Resonance (EPR) spectra was recorded using (JEOL-FE1X) with an operating frequency of 9.205 GHz (X-Band) at room temperature. For this analysis, the prepared ZnO thin film was scratched as a powder and analysed. Also, field modulation frequency of 100 kHz and microwave power of 5 mW were maintained throughout the EPR measurement. Further, XPS spectra were recorded using X-ray Photoelectron Spectroscopy (Thermo Fisher Scientific Inc., K Alpha, USA). The Light Emitting Diode (LED) (white light) was utilized as the light source in photoelectric based hydrogen peroxide vapour sensing studies.

2.3. Sensing setup

The home-build sensing setup was used to study the sensing characteristics of the spray deposited sensing element [18]. The details about the sensing chamber and the establishment of electrical contacts were reported earlier [18]. Calibrated volume of liquid hydrogen peroxide was introduced into the sensing chamber and the concentration of the injected vapour was obtained using the following Eq. (2)

$$C_{(ppm)} = \frac{\delta \times V_\Gamma \times R \times T}{M \times P_b \times V_b} \times 10^6 \tag{2}$$

where C is the concentration of the liquid ammonia (ppm), δ is the density of liquid ammonia (g/mL), V_Γ is the volume of injected ammonia (μL), R is the universal gas constant (8.3145 J/mol K), T is the absolute temperature (K), M is the molecular weight, P_b is the chamber pressure (atm), V_b is the volume of the chamber (L). By using LabVIEW controlled data acquisition system (DAQs), the variation in the electrical resistance of sensing element was recorded. The steady and stable resistance of the sensing element before the exposure of hydrogen peroxide was taken as baseline resistance (R_o). The stable resistance change after the exposure of the hydrogen peroxide was taken as R_g. The sensing response was calculated from Eq. (3)

$$\%S = \frac{(R_g - R_o)}{R_g} \times 100 \tag{3}$$

3. Results and discussion

3.1. Structural and morphological studies

The polycrystalinity of spray deposited ZnO thin film was investigated by comparing the observed XRD pattern with the standard JCPDS card [36-1451] as shown in Fig. 1(a). The formation of hexagonal wurtzite structure with the lattice parameters a = b = 3.24 Å and c = 5.21 Å was also observed. The crystallites were predominantly grown on the c-axis (0 0 2). The other weak intensity peaks such as (1 0 1), (1 0 2) and (1 0 3) were also observed. Majority of the crystallites were oriented in the c-axis and insufficient growth in all other orientations might be due to the morphological defects like agglomerated particles.

It was evident that no other characteristic peaks were observed for any impurities, which indicated the formation of ZnO without impurities. The interplanar distance d was calculated from Bragg's relation as shown in Eq. (4). Moreover, all the peaks were slightly shifted at higher angles due to the lattice contraction. The percentage of lattice contraction was calculated from the variation in standard interplanar distance d_{std} and observed interplanar distance d_{obs}. The relation between the interplanar distance d and percentage of lattice contraction (ε) was obtained from the relation shown in Eq. (5). Further the presence of contraction in all crystallites orientation was clearly observed from Table 1. This lattice contraction might be due to the presence of dangling bonds at the surface of ZnO and electrostatic interaction between the Zn^{2+} and O^{2-} [19].

$$d = \frac{\lambda}{2\sin\theta} \tag{4}$$

$$Lattice\ contraction\,(\varepsilon) = \frac{(d_{std} - d_{obs})}{d_{std}} \tag{5}$$

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