



SnO₂ thin film synthesis for organic vapors sensing at ambient temperature



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ARTICLE INFO

Article history:

Received 25 September 2016

Received in revised form 28 October 2016

Accepted 8 November 2016

Keywords:

SnO₂ thin film

Sensitivity

XRD

SEM

AFM

UV–visible

ABSTRACT

The present work is a study of tin dioxide (SnO₂) based thin sensitive layer dedicated to organic vapors detection at ambient temperature. SnO₂ thin film was deposited by chemical spray pyrolysis technique. The glass substrate temperature was kept to 400 °C, using a starting solution of 0.1 M tin (II) dichloride dihydrate (SnCl₂ · 2H₂O). Films structural and morphological properties were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM) and atomic force microscope (AFM) respectively. Films optical characteristics were studied using UV–VIS spectrophotometer. XRD revealed the presence of pure SnO₂ polycrystalline thin film with a tetragonal rutile structure. The SEM and AFM observations confirmed the granular morphology with presence of pores in the film surface. The prepared film was tested in various organic vapors (ethanol, methanol and acetone) at ambient operating temperature (25 °C ± 2 °C). The obtained results suggested that SnO₂ is more sensitive to ethanol vapor with a maximum sensitivity of 35% higher than to methanol and acetone vapors (1% and 3%). The realized SnO₂ based sensor demonstrated fast response and recovery times as revealed by the values of 2 s to 3 s towards 47 ppm of ethanol vapor.

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1. Introduction

Almost all organic solvents and alcohols are among the most underrated hazards in the atmosphere. They are poisonous if swallowed or inhaled in sufficient quantity. They are anesthetics and irritants of the eyes and upper respiratory tract. In high concentrations, alcohols like ethanol, methanol and acetone can cause dizziness, intoxication, blurred vision, possible liver and kidney damage. If swallowed, they can cause blindness and even death. In this context, the environment protection has established the air quality standards defining an odor threshold limit for chemical alcohols at 3 ppm (ethanol), 13 ppm (acetone) and 84 ppm (methanol) [1–3]. The detection of these toxic gases in the environment requires versatile sensors with higher sensitivity, faster response and recovery time. Many metal oxide based gas sensors such as TiO₂, WO₂, In₂O₃, ZnO and SnO₂, show a large variation in conductivity since they are able to detect a wide range of chemical and toxic gases at trace ppm or sub ppm level [4–7]. Among them tin dioxide thin films (SnO₂) enable the detection of many toxic and inflammable gases such as ethanol, methanol and acetone. However, its working principle requires sensor heating at high temperatures (100 to 350 °C) [8]. These work conditions cause several damages in sensor structure and its operation principle like thermal stability, heating device development, thermal degradation of target compounds to be detected.

Extensive research is still underway to produce reliable SnO₂ sensors by various synthesis techniques. These include physical vapor deposition (PVD) [9], laser ablation, molecular beam epitaxy, sputtering [10, 11], chemical vapor deposition (CVD), sol-gel, ultrasonic spray pyrolysis, spin and dip-coating [12–14]. Among these techniques, spray pyrolysis is a simple and low cost-effective processing method. It is suitable for preparation of undoped and doped semiconductors thin films and provides uniform and homogeneous layers on various glass substrates [15]. This process enables to control many parameters such as the grain size, the porosity and the thickness of layer. To the best of our knowledge, there are no published studies of SnO₂ based sensor for the detection of alcoholic solvents at ambient temperature. The goal of this work is to investigate the properties and the applicability of spray pyrolysis deposited SnO₂ thin films as gas sensors working at ambient temperature. The main aim of the present study is to improve optimum experiment parameters deposition (the scanning nozzle speed, the distance between spray nozzle-substrate, pressure and flow rate) and optimum operating temperature where the response and recovery times as function of the sensitivity of SnO₂ thin films are performed.

2. Experimental details

2.1. SnO₂ thin film preparation

The SnO₂ thin film is deposited using the precursor aqueous isopropanol solution including tin (II) dichloride dihydrate (SnCl₂ · 2H₂O, 99.8%, Aldrich) in 100 ml deionized water and 25 ml of ethanol

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Table 1
Spray pyrolysis parameters for deposition of SnO₂ thin film.

Spray parameters	
Constant parameters	Values
Solution concentration	0.1 M
Substrate temperature	400 °C
Deposition time	30 min
Nozzle-substrate distance	18 cm
Scanning nozzle speed	5 mm/s
Solution flow rate	0.5 ml
Solvents	Deionized water, acetone and methanol
Carrier gas	Compressed air

(C₂H₅OH) by the spray pyrolysis technique. Well clean glass substrates (in acetone, methanol solution bath and rinsed in distilled water) were placed on hot plate heated at $T = 400\text{ }^{\circ}\text{C}$ and the deposition time was kept at 30 min. The scanning nozzle speed was done in the intervals 5 mm/s along X and Y axis, the spray nozzle is maintained constant at distance of 18 cm from the substrate and flow rate controlled through air compressor regulator was done 0.5 ml/min. The process parameters for SnO₂ thin films deposition are listed in Table 1.

2.2. Sensing measurements

For sensing measurements, inter-digitized Au electrodes were deposited on the film surface by DC sputtering technique as shown on the Fig. 1.

The vapor sensing studies were carried out by the way of a static gas chamber to alcohols vapors detection in air ambient. The SnO₂ thin film deposited at 400 °C was used as the sensing element. A thermocouple is mounted to measure the temperature. The experiments were performed at ambient temperature ($25\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$) with varying concentration from 23.4 ppm to 329 ppm of alcohols solution. The sensor response to reducing gas such as ethanol, methanol and acetone is detected through the sensors resistance, where the sensitivity is given by [15]:

$$S\% = ((R_a - R_g)/R_a) \times 100\% \quad (1)$$

Where R_a is the resistance of the film in air and R_g is the film resistance during gas exposure. The time variations in SnO₂ thin film resistance during gas vapor exposure were monitored by using a Keithley 617 programmable electrometer.

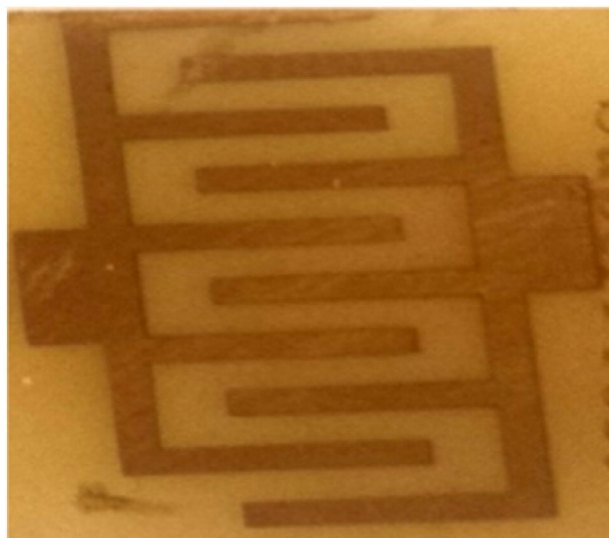


Fig. 1. Image of inter-digitized electrodes used for SnO₂ based gas sensors.

3. Results and discussion

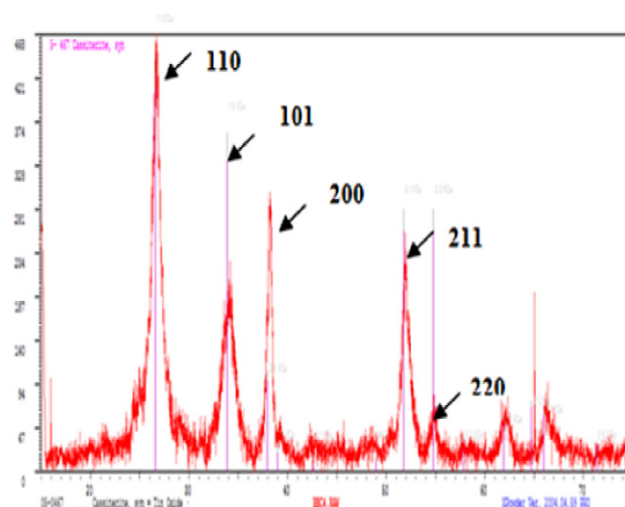
3.1. Structural properties

SnO₂ thin film was characterized using various analytical techniques. The XRD study was primarily carried to determinate its structure and its preferential oriented.

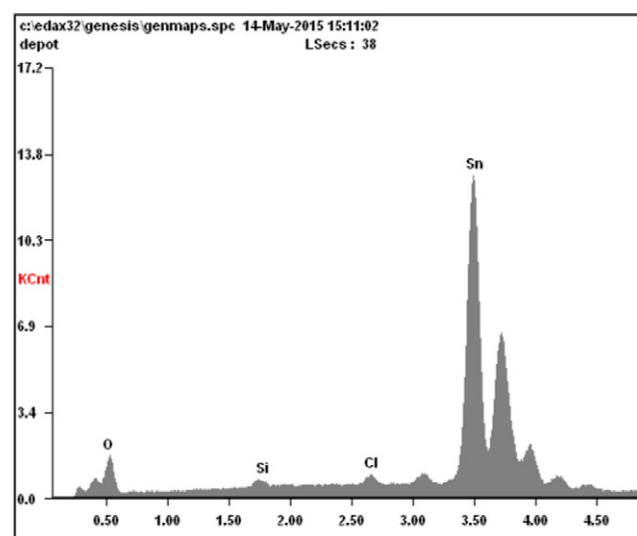
Fig. 2(a) shows patterns of the deposited SnO₂ thin film. The presence of intense peaks indicates that the deposited film has polycrystalline structure. The main peak can be clearly seen at 2θ value 26.5° corresponding to atomic plane (110), and the others peaks assigned to (101), (200), (211) planes confirmed that the deposited SnO₂ has a tetragonal rutile structure. No peaks corresponding to others phases of tin oxide have appeared in the XRD patterns, thereby indicating the formation of pure SnO₂ thin film.

The crystallites sizes were estimated from XRD results according to Scherrer's formula [16]:

$$d = \frac{0.9 \lambda}{\beta \cos \theta} \quad (2)$$



(a)



(b)

Fig. 2. (a) XRD patterns of the sensitive SnO₂ thin film (b) EDX spectrum of SnO₂ thin film deposited at 400 °C.

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