Sensing characteristics of tin-doped ZnO thin films as NO₂ gas sensor

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Abstract

NO₂ gas sensor was fabricated by successive ionic layer adsorption and reaction (SILAR) technique and rapid photothermal processing (RPP) of the Sn-doped ZnO film. The experimental results shows that tin doping of zinc oxide thin films improve the sensor element sensitivity to 1.5 ppm NO₂ in air and downshift the operating temperature. The influence of variation of Sn concentration in the chemical bath and the RPP temperature on NO₂ sensitivity of thin film sensor elements was investigated in this work. Higher sensitivity was obtained at 5–10 at.% tin concentration in the solution of ions and RPP temperature of 550–650 °C. Increasing the Sn concentration in doped ZnO samples more than 10 at.% as well as decreasing the oxygen partial pressure leads to the NO₂ gas sensitivity decrease. It looks promising to use the inexpensive tin-doped zinc oxide thin films obtained by SILAR method and RPP in smart gas sensing devices that are able to recognize gas species in low concentrations and are demanded for continuous environmental monitoring.

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Keywords: Successive ionic layer adsorption and reaction (SILAR); Rapid photothermal processing (RPP); Sn-doped zinc oxide film; NO₂ sensor

1. Introduction

Recently, semiconductor gas sensors based on the control of electrical conductivity upon the expose to gases has attracted considerable attention owing to their compact size which facilitate miniaturization required in electronic circuits and simple sensing method. For this reason numerous materials have been developed for gas sensing where the metal oxides that are physically and chemically stable have been extensively investigated.

The electrical characteristics of semiconducting metal oxide materials depend on the composition of the surrounding gas atmosphere. Based on this effect, the development of the conductometric gas sensors using thin films of n-type zinc oxide is in progress. However, this type of gas sensor show a lack of selectivity due to an unspecific gas detection mechanism and more or less many types of reducing gases are detected. The development of selective sensors is based on the use of zinc oxide thin films doped with different impurities as Sn, Fe, Cu, Al, etc. [1–8], and specific interactions between the molecules to be detected and ions in the sensing materials.

Undoped ZnO films show n-type electrical conductivity and have poor stability in humid environment or corrosive medium and properties are altered by adsorption of O₂, CO₂ and water. Doping of the sensing zinc oxide thin films can produce the defects that increase their influence on the sensors conductivity in nocive gas ambient and enhance their properties with impurities, such as Sn, Cu, Al, etc. Different methods have been utilizing to obtain pure and doped ZnO films: magnetron sputtering, spray pyrolysis, thermal oxidation, reactive evaporation, vapour phase epitaxy, electrodeposition, sol–gel, solid-state reaction, chemical deposition, etc. [5–12]. Among them, successive ionic layer adsorption and reaction (SILAR) has attracted special interest for preparing the zinc oxide thin films during the last years due to its simplicity and low cost processes, capability to achieve large area coatings. SILAR is an alternative to vapour-phase and chemical precursor techniques and has been used to produce a large variety of semiconductor materials, multicomponent films and single-oxides like CdS, Zn(O, OH, S), SiO₂, SnO₂, CdSnO₄ and Cu₂O [11–15].

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The present work is a first report describing the formation of conductive tin-doped zinc oxide thin films by SILAR technique and rapid photothermal processing (RPP) and their NO₂ gas sensing applications. Also a procedure that improves the efficiency of the deposition method of the Sn-doped ZnO thin films growing is presented. The experimental results of growth kinetics, morphology, resistivity, sensing characteristics after post-deposition RPP have been presented. It was expected to find out the optimal temperature and ambient of RPP to obtain the best tin-doped zinc oxide films for NO₂ gas sensor applications. The results of the influence of the toxic NO₂ gas onto doped ZnO films electrical properties and sensitivity characteristics are presented. It is shown that using the rapid photothermal processing (RPP) which include the thermal and quantum effects\[16,17\], the higher sensitivity to toxic NO₂ gas was obtained.

2. Experimental

2.1. Preparation

The tin-doped zinc oxide (TZO) films were deposited onto corning glass substrates by a relatively new and less investigated successive ionic layer adsorption and reaction (SILAR) process first reported by Nicolau\[12\]. Before the deposition, the substrates were cleaned in HCl, rinsed in deionised water (DI) and ethanol:acetone (1:1) mixture, and dried in a nitrogen flux. The cleaned substrates were immersed in an aqueous solution bath for a time in order to fabricate the films of desired thicknesses. The aqueous solution comprises a mixture of zinc sulphate, sodium hydroxide, and Na₂SnO₃ mixed until complete dissolution. The concentration of complex solution was diluted to obtain 0.04, 0.08, 0.12 and 0.15 M zinc concentration for deposition process by adding respective quantities of deionised water. Adding the Na₂SnO₃ in the aqueous solution 5 or 10 at.% Sn concentration according to doping level the impurification was performed. The first reaction vessels with aqueous solution were held at room temperature and the beaker with water in a temperature interval from 95 to 98 \( ^{\circ} \text{C} \) during the deposition. The growth cycle of doped TZO thin film consist in immersion of the substrate in aqueous complex solution for 5 s, then rinsing the substrate with deionised water for 10 s, then dipping in a beaker with water in a temperature range 95–98 \( ^{\circ} \text{C} \) for 5 s and washing the substrate for 10 s in DI water. The processes of coating cycle were repeated until the desired thickness was obtained. Finally the films were dried in air at 150 \( ^{\circ} \text{C} \) for 5 min.

After the deposition process, the films were subjected to rapid photothermal processing at the temperatures 350, 550 and 650 \( ^{\circ} \text{C} \) in a low vacuum (10\(^{-1}\) Pa) using an IFO-6 RPP system, the set up illustrated in [17,18]. The duration of RPP was chosen as 15 s for all temperatures, in accordance with preliminary investigation results and report\[7\]. A typical wafer temperature schematic profile during the post-deposition processing at 650 \( ^{\circ} \text{C} \) is shown in Fig. 1. The schedules of the RPP processed TZO film based sensor elements are summarized in Table 1.

2.2. Measurements

The properties of the TZO thin films have to be well analyzed in order to optimize their characteristics for the NO₂ gas sensing element application of the films.

The film thicknesses have been measured from the cross-section image of the scanning electron microscope (SEM). The deposition kinetics was studied by measuring the dependence of the film thickness versus the deposition time—the number of successive cycles at the respective temperature. For the surface morphology studies of the tin-doped ZnO films deposited on glass substrate was used a VEGA TS 5130MH scanning electron microscope (SEM). The quantitative element composition analyses of the films were performed using energy dispersion X-ray spectrometer (EDX) unit.

After rapid photothermal processing of TZO thin films at 350, 550 and 650 \( ^{\circ} \text{C} \) in the air and in vacuum the electrodes

<table>
<thead>
<tr>
<th>Sensor element</th>
<th>9.1 (5 at.% Sn)</th>
<th>2.1 (10 at.% Sn)</th>
<th>As grown</th>
<th>As grown</th>
<th>15</th>
<th>15</th>
<th>15</th>
<th>15</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duration of RPP (s)</td>
<td></td>
<td></td>
<td>9.2</td>
<td>2.2</td>
<td>9.3</td>
<td>2.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Temperature of RPP (°C)</td>
<td></td>
<td></td>
<td>350</td>
<td>350</td>
<td>650</td>
<td>650</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
were deposited onto film surface by thermal evaporation of aluminum in vacuum system VUP-5.

To study the electrical characterization of the tin-doped ZnO films, electrical resistivity measurements were performed using two point probe method in the temperature range of 300–550 K. The type of conductivity exhibited by TZO film was determined by thermo-electromotive measurements. The polarity of thermal generated voltage at the hot end is negative indicating the n-type for tin-doped zinc oxide films.

Gas sensing measurements have been carried out using a gas flow system as described in [19]. The sensor element prepared as described above was exposed to 1.5 ppm NO₂ gas in air. The temperature of the sensor element was precisely controlled from the room temperature to 250 °C.

During the sensing measurements using a computer based testing apparatus the gases concentration was controlled by mass flow controllers, 10 V dc was applied across the circuit and the output voltage across the dropping resistor was recorded. A computer with suitable interfaces handled all control and acquisition data and functions.

Gas sensitivities as a function of the operating temperature were obtained by alternating the dry air and the gases at each temperature with 50 °C increments. To measure reproducibility and stability, sensors were exposed to NO₂ gas and dry air at 150 °C for four repeated cycles alternately. The dry air and the NO₂-containing gases were maintained at a constant flow rate, 500 sccm, using mass flow meters and a mass flow controller during the measurements. Calibration curves, a relation between sensor resistance and NO₂ concentration, were obtained for TZO sensor elements by changing the NO₂-containing gas concentration from 0.5 to 1.5 ppm at a fixed temperature.

3. Results and discussion

3.1. Film growth

The dependence of tin-doped ZnO film thickness with increasing number of successive cycles for four concentrations of aqueous complex solution 0.04, 0.08, 0.12 and 0.15 M zinc concentration is shown in Fig. 2.

The thickness increases linearly with number of dipping steps and for the samples prepared from 0.08 and 0.12 M complex solution the film was uniform. This behaviour is explained as follows [20]: during the deposition took place the adsorption and reaction on the surface of the substrate successive dipped into the two precursor solutions; initially the cations are adsorbed on the surface of the glass substrate. On the next step the excess adsorbed ions are rinsed away from the diffusion layer and during the reaction step the anions react with the adsorbed tin and zinc ions and form a solid substance on the interface. During the last step of a growth cycle the excess and unreacted species are removed [15]. By successive growth cycles, a thin layer of material has been grown with a good adhesion to the glass substrate. Films obtained from the complex solution 0.04 and 0.15 M were nonuniformly distributed on the substrate.

The growth rate of tin-doped zinc oxide thin film on glass substrate was determined by dividing the measured overall film thickness by number of successive growth cycles and is higher for the deposition process with complex solution concentration of 0.15 M and has the value of 2.85 nm/cycle till 150 complete dipping cycles, respective for the solutions 0.04, 0.08, and 0.12 M is 1.4, 2.4, and 2.8 nm/cycle of growth. The behaviour during SILAR growth was found to be a three dimensional island growth mode at the beginning [20]. There is a dispersion of 10% in the film thickness measurements because of nonuniformity of the substrate handling procedure.

3.2. SEM and EDX investigation results

Characterisations by scanning electron microscopy (SEM) and quantitative element composition of the thin films using energy dispersion X-ray spectrometer (EDX) indicate the formation of Sn–ZnO film.

SEM micrographs in Fig. 3a and c shows the morphology of the as-grown 5 and 10 at.% Sn-doped ZnO films grown by SILAR method. The crystallites of the deposited material are visible in the as-grown film and have the sizes around 200 nm. The influence of rapid photothermal processing on the surface morphology of tin-doped zinc oxide films has been investigated.

In the 100–300 °C RPP temperature range the morphology of TZO films is the same that without rapid photothermal processing (Fig. 3a and c) which looks to have more voids. Increasing the RPP temperature up to 650 °C decreased the voids in the film with changes in the surface roughness and porosity of the film. In Fig. 3b and d are shown micrographs of the films processed at 650 °C looking more dense and uniform than Fig. 3a and c.
The observed particle size varies between 0.2 and 0.5 μm. It seems that the homogeneous crystal growth occurred during the rapid photon annealing at 500–650 °C.

In the case of long time treatment (more than 50 s) and at higher temperatures (higher than 650 °C), the process of grain growth and nonuniform surface roughness of tin-doped zinc oxide particles was found remarkable.

The Sn and Zn at.% in the TZO film was analyzed by energy dispersive X-ray (EDX) measurement in the plan detection mode. The size of electron spot is about 3 μm in diameter and the spot scanned two regions on the film. The penetrated depth of electrons into the film is around 3 μm. The Sn content in the film was calculated with an experimental error of 10% in the EDX analysis. The Sn:Zn ratio surveyed is 4:96 and 8:92 (at.%) in different scanned regions on samples of sets 9 (5 at.% Sn) and 2 (10 at.% Sn), respectively.

3.3. Electrical resistivity characteristics

In order to study the effect of rapid photothermal processing on the electrical properties of TZO films, the films were processed at various RPP temperatures for 15 s in low vacuum and in air. Fig. 4 shows the electrical resistivity of Sn-doped ZnO films dependence on the annealing temperature in low vacuum and in air. The films resistivity decreased as RPP temperature raises in vacuum, but in air, the resistivity increases about one order of magnitude after annealing, resulting the influence of the air oxygen and the formation of hydroxyl species on the surface of ZnO crystallites during the cooling performed in air.

The room temperature (20 °C) resistivity of tin-doped ZnO film was determined to be in order of 10 Ω cm and notably decreased when adding Sn, which is attributed to the increase of the electrons concentration caused by tin ions substituting the Zn2+ ions. Fig. 5 shows the variation of resistivity with temperature in air for the TZO film on the glass substrate without RPP and with RPP in vacuum and air ambient.

In air, at the temperatures from 300 to 550 K, the electrical resistivity of TZO-based sensor element decreases with the temperature increase indicating the semiconducting behaviour of Sn-doped ZnO film. This variation is nonlinear corresponding to the two regions of low and high temperatures.

The fresh prepared zinc oxide films contained a large number of chemisorbed oxygen species at the grain boundaries and also on the surface [15,20]. The electrical resistivity of tin-doped zinc oxide decreases when increasing the temper-
temperature of RPP, whereas the lost of adsorbed oxygen increases the donor concentration of the surface and thereby enhances the conductance. It were observed the irreversible changes in the electrical resistivity when the films were subjected to RPP at 300 °C in vacuum and at 350 °C in air for 15 s duration. Higher RPP temperature (550–650 °C) improves the quality and stability of TZO samples; the measurements have been carried out during one year and it was determined the good stability of pure ZnO conductance in air and higher stability of the Sn-doped ZnO films. According to our investigation results obtained by described method films ensure a stable zero level for gas sensor applications.

3.4. Gas sensing properties

The TZO sensor elements gas sensitivity and response–recovery characteristics have been evaluated at room temperature, 100, 150 and 250 °C for different NO2 gas concentrations.

A typical gas sensing measurement sequence consisted of 1 h sensors exposure to the toxic gas. After completion of one sequence the sensors operation temperature was changed and the measurement sequence was repeated. The resistance response of each sensor structure was transformed into a sensitivity value using commonly used formula for the oxidizing gases:

\[ S = \frac{R_g - R_0}{R_0} \cdot \frac{100\%}{C}, \]  

where \( R_g \) is the sensor resistance influenced by the NO2 gas, \( R_0 \) the sensor resistance in the air and \( C \) the NO2 gas concentration (in ppm).

The sensitivity of zinc oxide films doped with Sn, Cu, Al and Pd grown by SILAR method and rapid photothermal process have been studied. Fig. 6 shows the sensitivity to NO2 concentration of the 10 at.% Sn–ZnO based sensor element (sample 2.3), also for Cu, Al and Pd-doped zinc oxide films.

The sensitivity of Sn-doped zinc oxide-based sensor to NO2 was higher than that of Cu, Al and Pd-doped ZnO films for all the concentrations studied at room temperature and 150 °C. The sensitivity of TZO sample increases with increasing the concentration of NO2 till 1 ppm and then is saturated.

In Fig. 6, the sensitivity of the 10 at.% Sn–zinc oxide film (sample 2.3, Table 1) to NO2 increases rather steeply with increasing the gas concentration. The large change in curve shapes is transformed into the large gas sensitivity. The ZnO films doped with Al, Cu, and Pd showed slight sensitivity to NO2 gas with small changes of electrical resistivity values when this toxic gas was introduced into the testing chamber. It is shown that only tin-doped zinc oxide films have the higher sensitivity in these gas sensing experiments.

Fig. 7a shows the dynamic response of 5 at.% Sn-doped zinc oxide gas sensor elements (sample 9.3, Table 1) to 1.5 ppm NO2 gas in air measured at different operating temperatures 100 and 150 °C. The reaction time is 10 and 20 min, respectively.

Fig. 7b shows the dynamic response of 10 at.% Sn-doped zinc oxide gas sensor element (sample 2.3, Table 1) to 1.5 ppm NO2 at 150 °C operating temperature and the reaction time is 60 min.

The Sn–ZnO sensor element has a fast response time and higher sensitivity compared to Cu–ZnO and Pd–ZnO-based elements. The obtained experimental results showed that increasing the Sn concentration from 5 to 10 at.% in the ZnO film leads to the sensitivity decreasing of the sensor element and increasing the response time.

Fig. 8 shows the sensitivity of Sn-doped zinc oxide-based gas sensor element toward 1.5 ppm of NO2 versus the operating temperature. The composition of 5 at.% Sn in ZnO thin film shows a maximum sensitivity of 11 at 150 °C and a sensitivity of 1.5–2 at room temperature 25 °C toward 1.5 ppm of NO2 gas. There was a decrease of sensitivity when the
operating temperature becomes higher than 250 °C. A composition of 10 at % Sn in ZnO thin film shows a maximum sensitivity of about 6 at 150 °C and a sensitivity of 1.5–1.8 at room temperature 25 °C toward 1.5 ppm of NO₂ gas in air.

Another experiment shows that sensitivity of Sn–ZnO decreases with the increase of humidity as a result of the decrease of sensors element electrical resistivity: the increase of humidity from 20 to 80% lead to the resistivity decrease from 100 to 1.0 Ω cm. In this case the decrease of the oxygen concentration lead to the increase of the electrons concentration which increases the conductivity and respectively decrease the sensitivity.

The presented above results shown that the maximum sensitivity is obtained at the relative low temperature (150 °C) compared to reported ~400 °C [21–22]. The Sn doping of the ZnO sensor element has enhanced the sensitivity toward 1.5 ppm NO₂ gas compare to pure ZnO and Cu, Al and Pd-doped films.

The grain size was responsible for the determination of the operating temperature where the maximum sensitivity is obtained.

3.5. Gas sensing mechanism

The surface layer of the TZO-based gas sensors is responsible for the sensors sensing properties. There is a lot of adsorbed oxygen Oₐd on a TZO surface at room temperature. The chemical reactions in the oxides surface layer change the concentration of the conduction electrons providing the sensing property. Chemisorptions modify the defect states of the oxide’s surface layer and the working temperature of the TZO-based NO₂ sensor is usually lower than that of the bulk conduction based gas sensor structures [23].

RPP determine the desorption of the large number of chemisorbed oxygen species at the grain boundaries and on the surface of freshly prepared Sn-doped zinc oxide sensor samples. The loss of adsorbed oxygen increases the electron concentration of the surface and improves the films conductance. Desorption is an irreversible process thereby resistivity stabilizes after few seconds RPP and remain stable for a long time. The detection of an oxidizing gas such as nitrogen dioxide can be associated with reaction in which conduction electrons are consumed and the subsequent detection reaction lead to increase the barrier height and decrease the surface conductance. The response to NO₂ can be explained by the reaction:

\[
\text{NO}_2 + e^- \rightarrow \text{NO} + \text{O}_{\text{ads}}^{-}.
\]

The electrons consumed in these reactions are extracted from the conduction band thus raising the resistivity of tin-doped zinc oxide film. It is necessary to mention that in the last case the reaction products emerging from the primary detection reactions escape further detection within the sensing layer.

The improvement of sensing properties of doped zinc oxide sensor structure towards hazardous gas is determined by
the use of a particular dopant for the detection of a specific gas species. The presence of chemical adsorbed molecules (e.g. NO₂) could cause electron depletion in the thin film surface and the building up of a Schottky surface barrier; consequently, the electrical resistivity of the thin film increases to a maximum [24]. This could be a method to monitor the gas sensing property of the film.

At the same time the observed changes in the sensitivity of pure and doped sensor elements may be attributed to the porosity and particle size variations with the RPP of the thin films and respectively to the number of adsorbed species, which are directly determined by the surface morphology of the TZO films. The Sn doped zinc oxide, which has shown high sensitivity to different NO₂ concentrations, has average low particle size compared to the other doped ZnO films.

4. Conclusions

A selective NO₂ gas sensor, based on ‘molecular recognition’, can be designed using Sn-doped ZnO thin films obtained by SILAR method and rapid photothermal processing. Chemical deposition processes were studied to realize the sensors with lower operating temperature. Gas sensor elements based on doped zinc oxide films were prepared and characterized. Among all tested devices the Sn-doped zinc oxide sensor is sensitive to NO₂ at room temperature. Readability of the electrical and sensing characteristics of Sn-doped zinc oxide-based gas sensor element was monitored during 1 year.

In all cases, doped thin films present the stable low-temperature Sn-doped ZnO structure and the sensor sensitivity is related to the film morphology. It was experimentally demonstrated that tin impurities in ZnO films improved sensors gas-sensing properties to NO₂ and produce a shorter response time. It is possible to tune these parameters by varying the film porosity and Sn concentrations in the solution: highly porous film sensors have a high sensitivity, but a longer response time. The selectivity of the sensors is consistent with the molecular recognition concept. The detection can be associated with reaction in which conduction electrons are consumed and the subsequent detection reaction leading the increasing of the barrier height and decrease the surface conductance.

These experimental results confirm that conductometric gas sensors based on tin-doped ZnO as sensitive layer are of great interest for NO₂ detection. Moreover, chemical processes and RPP allow the production of inexpensive sensors in a simple manner.

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References


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Currently, his research is focused on physics and nanotechnology with RPP for gas sensors based on semiconductor oxides (ZnO, Cu2O, SiO2); shallow and ultra-shallow p–n junctions on Si, GaAs and InP for solar cells, sensors and other micro-electronic devices. He has published over 200 papers and 5 books, over 60 conference presentations, 3 patents in the field of semiconductor technologies. He has supervised 14 graduate students for PhD, 32 for MSc, more than 140 Engineering Degree Diploma in microelectronics. He is fellow of The Institution of Electrical Engineers (UK). Chairman of the Coordination Council Reliability of Semiconductor Devices Moldova. Vice-President of the Scientific Council for DSc and PhD Degree in Moldova. He received the national Award for the scientific merit “Dimitrie Cantemir” from the Academy of Sciences of Moldova in 2003.

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