

Characterization of Pure and Al-Zn Doped Conducting SnO₂ Thin Films for Gas Sensors

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Abstract

The present study investigates the influence of Al-Zn co-doping on the structural, morphological, and optical properties of nanostructured SnO₂ thin films prepared by spray pyrolysis. Pure and 2 mol% Al-Zn co-doped SnO₂ thin films were deposited on glass substrates at 500 °C using a laboratory-developed spray pyrolysis system. Tin chloride pentahydrate (SnCl₄·5H₂O) was used as the precursor, while aluminum chloride and zinc chloride were employed as dopant sources. The precursor solution was prepared in ethanol and stirred continuously at 50 °C for 4 h. During deposition, the spray nozzle was maintained at a distance of 25 cm from the substrate and the solution was delivered at a spray rate of 10 mL min⁻¹. The films were characterized using X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), UV-Visible spectroscopy, and energy-dispersive X-ray analysis (EDAX). XRD analysis showed that the average crystallite size decreased from 38.2 nm for the undoped SnO₂ film to 27.3 nm for the Al-Zn co-doped film. The optical band gap decreased from 3.52 eV to 3.47 eV after co-doping, while good visible-region optical transparency was retained. EDAX confirmed the presence of the intended dopant elements in the co-doped film. These results demonstrate that Al-Zn co-doping modifies the structural and optical properties of SnO₂ thin films and indicates their potential for gas sensor applications. Future studies will investigate gas-sensing response, sensitivity, selectivity, response/recovery characteristics, and long-term stability under different operating temperatures, humidity levels, gas concentrations, and interfering-gas conditions.

Keywords: SnO₂ Thin Films, Spray Pyrolysis, Al-Zn Co-doping, Gas Sensors, Nanostructured Materials, Thin Film Characterization.

Introduction

Tin oxide (SnO₂) is one of the most widely investigated transparent conducting oxide (TCO) materials because of its excellent optical transparency, high electrical conductivity, chemical stability, and environmental durability [1-5]. Owing to its intrinsic nonstoichiometric nature, SnO₂ exhibits remarkable electrical and optical characteristics that make it suitable for a wide range of technological applications. Thin films of SnO₂ can be successfully deposited on different substrates, including glass, ceramics, and oxide materials, making them highly versatile for use in electronic and optoelectronic devices [1-5]. Its high melting point, superior transparency in the visible region, and resistance to oxidation and moisture further enhance its suitability for long-term applications. [3,4].

SnO₂ thin films have attracted considerable attention for use in gas sensors, transparent conducting electrodes, solar cells, flat-panel displays, and

optoelectronic devices owing to their high optical transmittance and excellent sensing characteristics [4,5,13,14,18,19]. The gas sensing performance of SnO_2 is particularly influenced by its surface morphology, crystallinity, and electrical conductivity, which can be significantly improved through suitable elemental doping. Among various dopants, aluminum (Al) and zinc (Zn) have been extensively investigated because they enhance carrier concentration, improve crystallinity, and modify the optical band gap, thereby improving the functional properties of SnO_2 thin films [2,10,16-19].

Several deposition techniques, including chemical vapor deposition (CVD), magnetron sputtering, sol-gel processing, pulsed laser deposition, and spray pyrolysis, have been employed to fabricate SnO_2 thin films. Among these methods, spray pyrolysis has emerged as an attractive technique because of its simplicity, low fabrication cost, ease of operation, high deposition rate, and capability for large-area thin-film deposition [1-5]. In addition, the technique allows precise control of deposition parameters, making it suitable for producing high-quality nanostructured oxide films for industrial and research applications [1,3,5].

In the present study, pure and Al-Zn co-doped SnO_2 nanocrystalline thin films were synthesized using a laboratory-developed spray pyrolysis system. The influence of Al-Zn co-doping on the structural, morphological, optical, and elemental properties of SnO_2 thin films was systematically investigated [2,10,13,16-19]. The deposited films were characterized using X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), UV-visible spectroscopy, and energy-dispersive X-ray analysis (EDAX). The specific objective was to determine how Al-Zn co-doping at 2 mol% affects the crystallite size, surface morphology, optical transparency, and optical band gap of SnO_2 thin films and to assess whether the resulting properties support their potential application in gas sensors.

Research questions and hypothesis: This study addresses the following questions: (1) How does 2 mol% Al-Zn co-doping affect the crystal structure and crystallite size of SnO_2 thin films? (2) How does co-doping influence the surface morphology, optical transmittance, and optical bandgap? (3) Do

the resulting structural and optical characteristics support the potential use of the films in gas sensor applications? The working hypothesis is that Al-Zn co-doping inhibits grain growth and modifies the optical band gap while retaining good visible-region transparency.

Review of Literature

Recent studies have demonstrated that SnO_2 is a promising transparent conducting oxide with excellent structural stability, optical transparency, and gas-sensing performance. The properties of SnO_2 thin films can be significantly enhanced by incorporating suitable dopants such as aluminum (Al) and zinc (Zn), which improve crystallinity, electrical conductivity, and optical characteristics [2,13,16,17].

Several researchers have reported the fabrication of Al- and Zn-doped SnO_2 thin films using techniques such as spray pyrolysis, sol-gel, and sputtering. These studies revealed that the dopant concentration and deposition conditions strongly influence the crystallite size, surface morphology, optical band gap, and sensing behavior of the films [1-5,13,16-19]. Co-doping with Al and Zn has also been shown to improve carrier concentration and transparency, making the films suitable for gas sensor and optoelectronic applications [10,19].

Although considerable work has been carried out on individually doped SnO_2 thin films, only a limited number of studies have investigated Al-Zn co-doped SnO_2 films prepared by spray pyrolysis while simultaneously examining structural, morphological, optical, and compositional properties. Recent studies also demonstrate the continued relevance of SnO_2 -based thin films for gas sensing and show that sensing performance is strongly related to surface characteristics, dopant incorporation, operating conditions, and film structure [14,18,19]. Thus, a systematic comparison of undoped and Al-Zn co-doped SnO_2 films prepared under identical conditions is useful for establishing the material-property changes relevant to future sensor development.

Therefore, the present study focuses on the preparation of pure and Al-Zn co-doped SnO_2 thin films using the spray pyrolysis technique and systematically investigates their structural,

morphological, optical, and elemental characteristics to evaluate their potential for gas sensor applications.

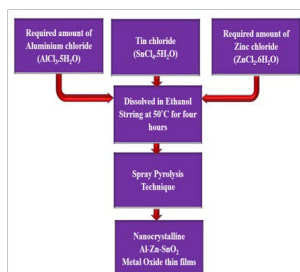
Methodology

Al–Zn co-doped SnO_2 thin films were deposited using spray pyrolysis deposition (SPD). In this process, a precursor solution containing tin, aluminum, and zinc salts was atomized and sprayed onto a heated glass substrate with the aid of a carrier gas. The precursor chemicals and dopant materials were dissolved in ethanol to prepare a homogeneous solution, which was continuously stirred at 50°C for 4 hours. Both the undoped SnO_2 solution and the Al–Zn co-doped SnO_2 solution was prepared with a dopant concentration of 2 mol%.

The cleaned glass substrates were placed on a thermostatically controlled hot plate and maintained at a deposition temperature of 500°C . The substrate temperature was continuously monitored using a digital thermometer to ensure uniform heating during the deposition process. During spraying, the distance between the spray nozzle and substrate was fixed at 25 cm, and the precursor solution was delivered at a constant spray rate of 10 mL min^{-1} . These deposition parameters were carefully maintained to obtain uniform and well-adhered thin films of the desired thickness. A schematic flowchart of the preparation procedure is presented to illustrate the experimental sequence.

To ensure the reproducibility of the experimental results, each sample was prepared under identical deposition conditions, and the optimized processing parameters were maintained throughout the study period. The deposited films were subsequently characterized to investigate their structural, morphological, optical, and compositional properties of the films.

Al–Zn– SnO_2 Nanostructure Metal Oxide Thin Films Preparation Flow Chart:



X-ray diffraction was used to investigate the crystalline structure using a DMA X2200 diffractometer with Cu-K α radiation ($\lambda = 0.1548\text{ nm}$) over a 2θ range of 20° – 70° . A field-emission scanning electron microscope (FESEM) was used to examine the surface morphology and cross-sectional film thickness. UV–Visible spectroscopy was performed using a Shima DZU UV-3101PC double-beam spectrophotometer to evaluate the optical characteristics.

Experimental Reliability and Measurement Conditions

To ensure the reproducibility of the experimental results, each thin-film sample was prepared three times under identical deposition conditions, including the substrate temperature, spray rate, precursor concentration, and nozzle-to-substrate distance. Similar structural and optical characteristics were obtained for all the prepared samples, confirming the repeatability of the deposition process.

The crystalline structure of the deposited films was analyzed using X-ray diffraction (XRD) over a 2θ range of 20° – 70° . The surface morphology was examined using field emission scanning electron microscopy (FESEM), while the optical properties were measured using a UV–Visible spectrophotometer in the wavelength range of 300–800 nm. The elemental composition was determined using energy-dispersive X-ray analysis (EDAX). All measurements were performed under ambient laboratory conditions using calibrated instruments.

The crystallite size was calculated using the Scherrer equation, with an estimated uncertainty of $\pm 2\text{ nm}$, whereas the optical band gap values were determined with an uncertainty of approximately $\pm 0.03\text{ eV}$. The reported values represent the average of repeated measurements, ensuring the reliability and consistency of the results.

Results and Discussion

Structural and Morphological Characterization of Al, Zn doped SnO_2 Thin Films

The XRD patterns of the pure and 2 mol% Al–Zn co-doped SnO_2 thin films deposited on glass substrates at 500°C are shown in Figure 1. The diffraction peaks were indexed by comparing the calculated

interplanar spacing (d-values) with the standard data reported for rutile SnO_2 . The XRD results confirmed the tetragonal rutile structure of SnO_2 , with the prominent diffraction peak corresponding to the (110) plane and weaker reflections from other planes, indicating the polycrystalline nature of the deposited films [3,7,8]. The average crystallite size was calculated using the Scherrer equation, $D = K\lambda / (\beta \cos \theta)$, where $K = 0.9$ is the shape factor, $\lambda = 0.1548$ nm is the wavelength of Cu-K α radiation, β is the full width at half maximum (FWHM), and θ is the Bragg diffraction angle [8]. The average crystallite sizes of the undoped and Al-Zn co-doped SnO_2 films were 38.2 nm and 27.3 nm, respectively. The reduction in crystallite size after co-doping suggests inhibited grain growth and is consistent with previous reports on doped SnO_2 thin films [13,16,17]. The smaller crystallite size may increase the surface-to-volume ratio, which is favorable for gas-sensing applications [14,18,19].

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$$D = k\lambda / \beta \cos \theta$$

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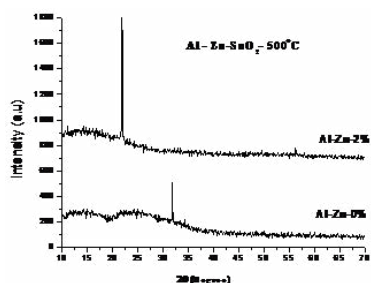


Figure 1 XRD Pattern of Undoped and Al-Zn Doped SnO_2 Thin Films Deposited on Glass Substrates at 500 °C

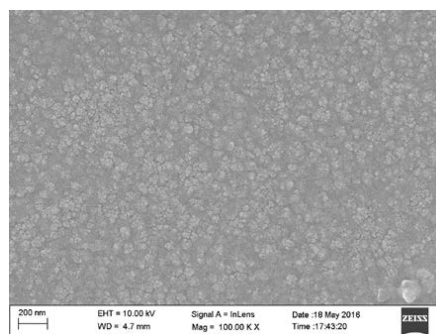


Figure 2 FESEM Image of Undoped SnO_2 Thin Film Deposited on Glass Substrate at 500 °C

The FESEM micrographs of the undoped and 2% Al-Zn co-doped SnO_2 thin films deposited at 500 °C are shown in Figures 2 and 3. The surface morphology revealed that the films consisted of densely packed nanocrystalline grains with uniform distribution. The addition of Al and Zn significantly influenced the grain size and surface texture. Compared with the undoped film, the co-doped film exhibited finer grains and a more compact morphology. These observations are consistent with the crystallite size obtained from XRD analysis and previous reports on Al- and Zn-doped SnO_2 thin films [7,13,16,17].

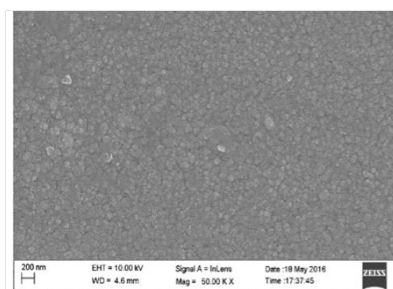


Figure 3 FESEM image of 2 mol% Al-Zn co-doped SnO_2 Thin Film Deposited on Glass Substrate at 500 °C

Optical Properties of Al-Zn- SnO_2 Thin Films

The optical transmittance spectra of undoped and Al-Zn co-doped SnO_2 thin films recorded in the wavelength range of 300–800 nm are shown in Figure 4. Both films exhibited high optical transparency in the visible region with a sharp absorption edge between 480 and 510 nm. The co-doped films maintained high optical transmittance, indicating that Al and Zn incorporation did not adversely affect the optical quality of the films [1,2,4,13,17].

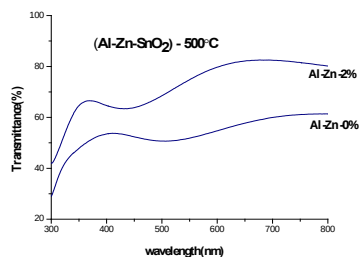


Figure 4 Optical Transmittance Spectra of Undoped and 2 mol% Al-Zn Co-doped SnO₂ Thin Films Deposited on Glass Substrates at 500 °C

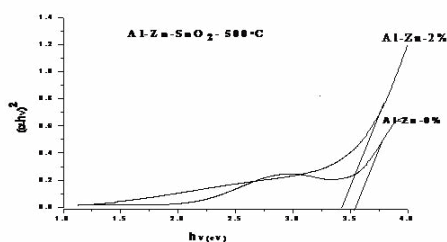


Figure 5 Tauc Plots for the Optical Band-gap Determination of Undoped and 2 mol% Al-Zn Co-doped SnO₂ Thin Films

The direct optical band gap of the undoped and Al-Zn co-doped SnO₂ thin films was determined from the Tauc plot shown in Figure 5. For a direct allowed transition, the relation is $(\alpha h\nu)^2 = A(h\nu - E_g)$, where α is the absorption coefficient, h is Planck's constant, ν is the photon frequency, E_g is the direct optical band gap, and A is a material-dependent constant. Extrapolation of the linear portion of the Tauc plot gave band-gap values of 3.52 eV for the undoped film and 3.47 eV for the Al-Zn co-doped film. The slight decrease in band gap indicates that Al-Zn incorporation modifies the optical properties of SnO₂ and is consistent with the dependence of the optical band gap on dopant incorporation and preparation conditions [1,2,13,16,17].

$$(\alpha h\nu) = (h\nu - E_g)^{1/2}$$

where h (J.S.) is Planck's constant, (HZ) is the photon frequency, E_g (eV) is the band gap, (m^{-1}) is the absorption coefficient, and A^* is a constant that varies depending on the material and the direct optical band gap (E_g). The plot shows that the optical gap is undoped at 3.52 eV and 3.47 eV. The literature values are well aligned. Depending on the dopants

and preparation technique, these values change. The AL-Zn-SnO₂ EDAX spectra are displayed in (Figures 5, 6). The growth of grains with distinct boundaries may be seen in the micrographs as a sign of the samples' high crystallinity. According to the theoretical calculations, the EDAX spectra verified that all of the elements in the samples were stoichiometric [1,2,16,19]

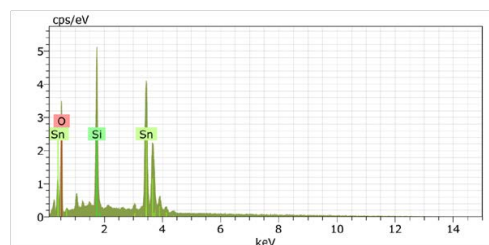


Figure 6 EDAX Spectrum of Undoped SnO₂ Thin Film Deposited on Glass Substrate at 500 °C

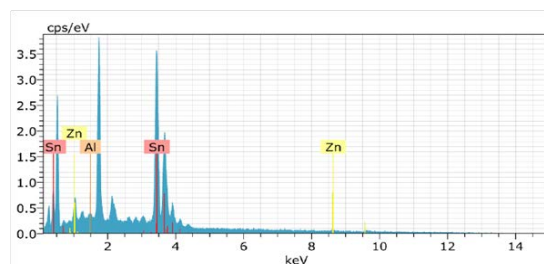


Figure 7 EDAX Spectrum of 2 mol% Al-Zn Co-doped SnO₂ Thin Film Deposited on Glass Substrate at 500 °C

Conclusions

Pure and Al-Zn co-doped SnO₂ thin films were successfully deposited on glass substrates using the spray pyrolysis technique at 500°C. XRD analysis confirmed the formation of a polycrystalline tetragonal rutile structure, while FESEM and UV-Visible studies revealed uniform morphology, high optical transparency, and a reduction in the optical band gap from 3.5 eV to 3.2 eV after Al-Zn co-doping. EDAX analysis verified the successful incorporation of the dopant elements. These results demonstrate that Al-Zn co-doping effectively improves the structural and optical properties of SnO₂ thin films, making them promising materials for gas sensor applications.

The present study was limited to a single Al-Zn dopant concentration (2%) and a fixed deposition

temperature of 500 °C. Future research should investigate different dopant concentrations, optimize the deposition parameters, and evaluate the gas sensing performance, selectivity, and long-term stability of the prepared thin films for practical sensor applications.

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