



An Analysis of Spray-Pyrolyzed Tin Dioxide's Optical and Structural Properties

Mohammed Nouri Yuonis¹ and Hiba M. Ali^{2*}

^{1,2} Department of Physics, College of Education for Pure Science (Ibn Al-Haitham), University of Baghdad, Baghdad, Iraq

*Corresponding Author

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Abstract

In this work, we used a pyrolysis spray method at a substrate temperature of 673 K to create SnO₂ thin films. After that, the films were annealed for one hour and then one and a half hours at 773 K. XRD, AFM, and UV-VIS-NIR spectrometers were used to analyse the samples. Without annealing, the XRD pattern revealed that the SnO₂ films exhibited an amorphous structure prior to annealing. After annealing, the film transformed into a tetragonal cassiterite structure with distinctive orientations (110, 101, 211). The study found that mean diameter, surface roughness, and particle size changed as the annealing temperature increased. The transmittance and absorption spectra were analysed within the wavelength range of 325-1100 nm. The one hour and one and a half hour treatments yielded the highest transmittance values for the sample, according to the results. According to the findings, optical band gap energies are 3.22, 3.54, and 3.64 eV, respectively.

Keywords: SnO₂, Spray pyrolysis, AFM, Transmittance.

1. Introduction

Many techniques, such as chemical vapour deposition, sol-gel, spray pyrolysis vapour deposition, thermal evaporation, and sputtering, can be used to create pure thin film^{1,2}. Metal oxide thin films exhibit high optical transparency in the visible spectrum and conductivity comparable to that of metals^{3,4}. Because of its superior transparency and relatively large optical band gap of 3.6 eV, SnO₂ is a common n-type oxide semiconductor with a wide range of applications, including solar cells and gas sensors^{5,6}. Along with other benefits, it has strong thermal stability, is insoluble in strong acids and alkalis, and has excellent chemical stability⁷. Spray pyrolysis is the technique used in this investigation. This preparation method is simple and cost effective, and it does not require advanced substrates or expensive chemicals. Strong dedication to a chemical and physical spraying method that can withstand environmental impacts on a variety of substrates^{8,9}. Spray pyrolysis has a set of adjustable parameters to change the thin film's surface morphology. Substrate temperature, solution concentration, volume of solution, flow rate, solvent type, carrier gas ratio, nozzle-substrate distance, and deposition time are the parameters^{10,11}.

2. Materials and Methods

Tin dioxide thin films were prepared by dissolving tin chloride dihydrate in a 1:4 methanol and deionised water solution at a concentration of 0.055 M. To enhance homogeneity and obtain a transparent solution, two to four drops of hydrochloric acid were added, followed by stirring with a magnetic stirrer^{12,13}. The thin film was deposited via spray pyrolysis using a homemade device equipped with a heating element, a rotating substrate, and a timer. The substrate rotated automatically (three seconds on to fifty-seven seconds off) to maintain a constant temperature. The solution was sprayed using an easily accessible airbrush that was connected to a compressor with a pressure of roughly (10⁵) N/m². There was a 24 cm gap between the substrate and the

spray gun. The thin film was applied to (2.5 x 2) cm glass slides at 673 K. After that, it was annealed twice at 773 K for one and one and a half hours. The resulting films had a thickness of 300 ± 50 nm. The film's crystal structures were ascertained by X-ray diffraction research. Conducted using an X-ray diffractometer. The Scherrer equation can be used to determine the crystal size (C.S.) according to **Equation 1**^{14,15}.

$$C.S = \frac{0.9\lambda}{FWHM \cos \theta} \quad (1)$$

Bragg's angle (θ), the primary peak's full width at half maximum (FWHM), and the X-ray wavelength of 0.154 nm (λ) were the symbols used. Atomic Force Microscopy (AFM) was used to examine the roughness and morphological characteristics of the film. The optical characteristics were examined using the UV-Visible-NIR spectrometer.

3. Results

3.1. Structural Analysis

3.1.1. XRD Analysis

Two annealing time results are displayed in **Figure 1**. Cu-K α radiation ($\lambda = 0.154$ nm) generated by X-ray diffractometer was used to analyse the X-Ray diffraction pattern of tin dioxide (SnO₂) deposited on glass slides at a substrate temperature of 673 K and annealed for one and one and a half hours at 773 K. The films were amorphous upon as-deposited, then after annealing for one hour, and then for one and a half hours the patterns of these films exhibit the SnO₂ crystalline phase's (110), (101) and (211). These patterns were used to analyses the crystal size. The artificial tin dioxide XRD patterns were measured between 20° and 80°.

Table 1. X-ray diffraction of SnO₂ at various annealing times (as-deposited, 1 h and 1.5 h) at 773K

Time (hour)	2 θ_{exp}	2 θ_{ASTM}	FWHM (deg)	d _{exp} (Å)	d (Å) ASTM	Planes (hkl)	C.S (nm)
1	26.299	26.59	2.270	3.385	3.348	110	3.75
	33.575	33.87	0.999	2.667	2.644	101	8.67
	51.580	51.78	0.866	1.770	1.763	211	10.36
1.5	26.483	26.59	1.973	3.362	3.348	110	4.31
	33.781	33.87	0.757	2.651	2.644	101	11.44
	51.753	51.78	0.659	1.764	1.763	211	13.96

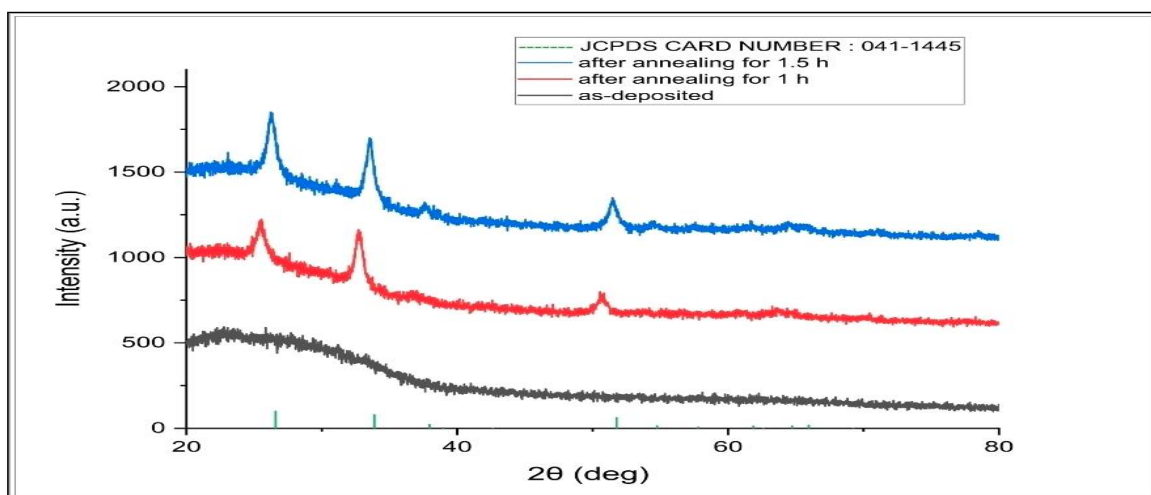


Figure 1. XRD diffraction patterns of SnO₂ thin films at different annealing times (as-deposited, 1 h, and 1.5 h).

3.1.2. The Analysis of Surface Morphology

Three-dimensional (3D) atomic microscopy images and a cumulative particle size distribution plot of tin dioxide thin films before and after annealing at different times can be observed in **Figure 2**. The particle diameter expanded from 46.51 nm in the as-deposited film to 70.73 nm after annealing at 773 K for one and a half hours, accompanied by a broader distribution range.

This indicates a variation in particle diameters in the sample over the longest annealing time. The average diameter, surface roughness and RMS roughness are listed in **Table 2**.

Table 2. AFM parameters (Average Diameter, Roughness Surface, and RMS roughness) for SnO₂ samples (As-deposited, 1 and 1.5 hours)

Time (hour)	Average diameter (nm)	Surface roughness (nm)	RMS (nm)
As-deposited	46.51	6.43	10.21
1	56.23	5.25	7.41
1.5	70.73	5.04	7.11

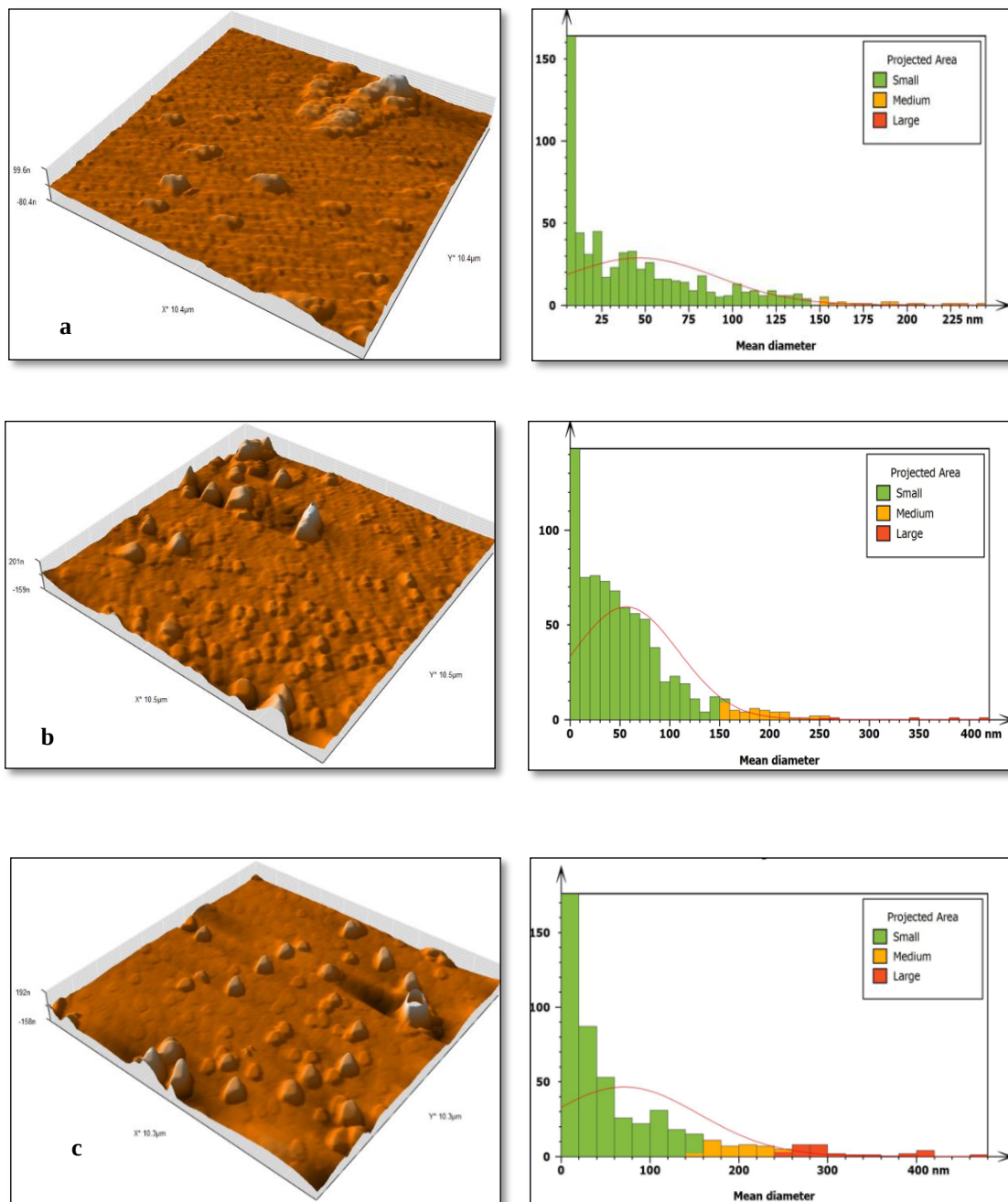


Figure 2. Three-dimensional AFM images and mean particle diameters of SnO₂ thin films at different annealing times: a. as-deposited, b. annealed for 1 hour, and c. annealed for 1.5 hours.

3.2. Optical Analysis

The optical transmittance spectra Of prepared SnO₂ films and annealed at Different annealing time in the wavelength range of (325–1100) nm are transparent in the visible region, as shown in **Figure 3**.

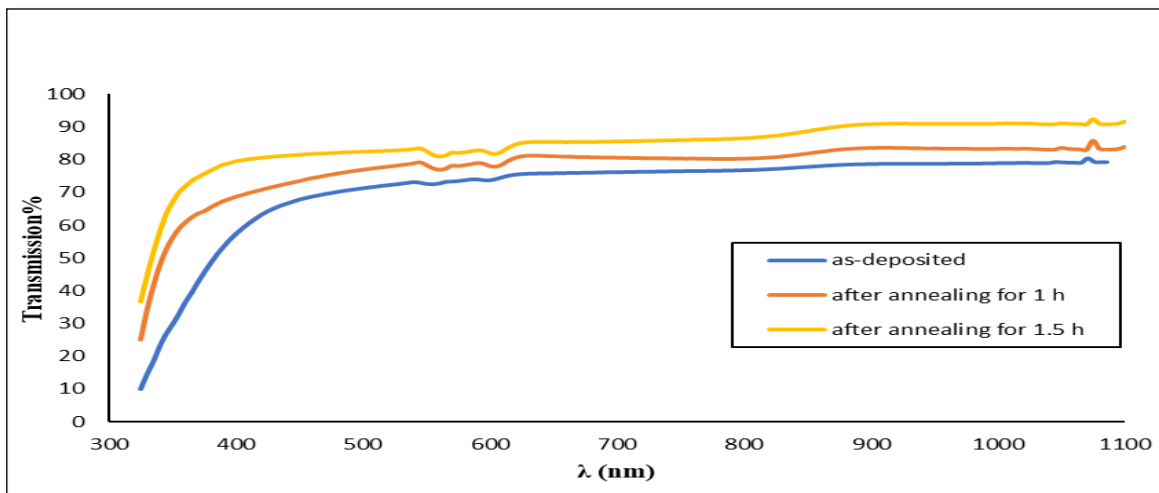


Figure 3. UV-VIS optical transmission of SnO₂ thin films annealed at various times and deposited on glass substrates.

The absorption rate is significantly influenced by the band gap and photon energy. **Figure 4**. shows how photon energy affects the coefficient of optical absorption (α) for the annealed SnO₂ films, which can be expressed as **Equation 2**.

$$\alpha = (2.303) \frac{A}{t} \quad (2)$$

where (A) is the absorbance of film and (t) is the film thickness .

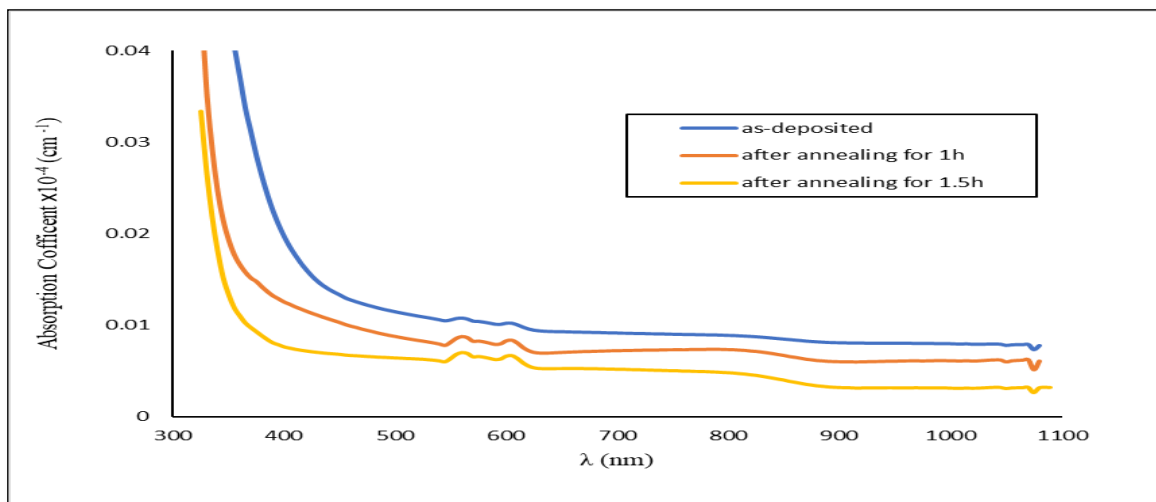


Figure 4. The absorption coefficient of SnO₂ particle thin films as a function of wavelength at two annealing time

The plots of $(\alpha h\nu)^2$ are shown in **Figure 5**. The optical energy gaps of the resulting SnO₂ thin films were found to be 3.22, 3.54, and 3.64 eV for the as-deposited films and for films annealed for one hour and one and a half hours, respectively. The direct optical energy gap was found to increase as the annealing time rose.

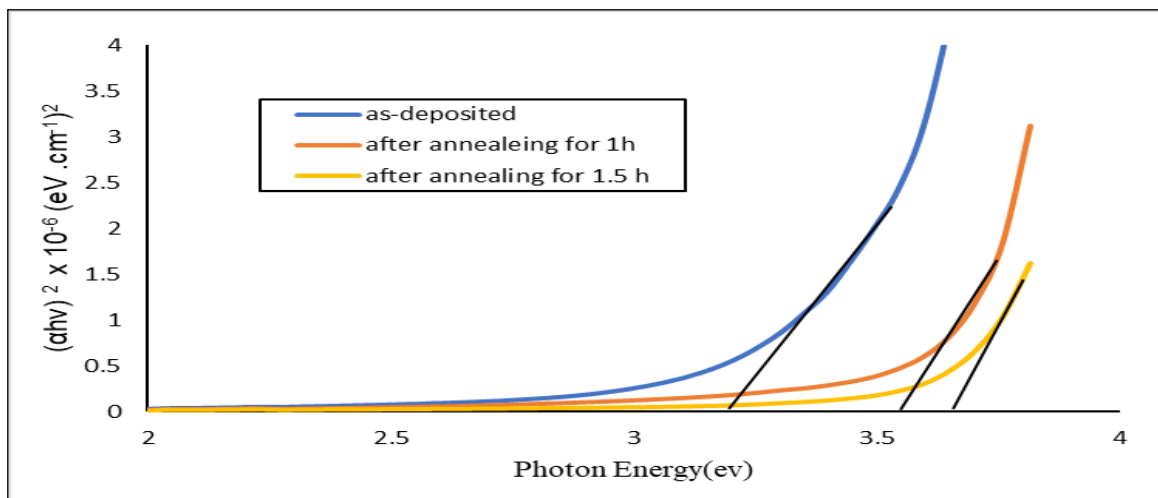


Figure 5. Variation of absorption coefficient with photon energy for SnO₂ thin films under different annealing conditions: as-deposited, annealed for 1 hour, and annealed for 1.5 hours at 773 K

4. Discussion

Annealing induced the transformation of SnO₂ thin film from an amorphous state in to a polycrystalline tetragonal cassiterite structure. No additional diffraction peaks of impurity phases were observed in the XRD patterns, confirming the pure phase of synthesized SnO₂. The grain size of SnO₂ increased with longer annealing times, as the high-temperature treatment promoted the coalescence of smaller grains in to larger ones¹⁶. Moreover, the concentration of non-bridging oxygen defects rapidly decreased with annealing time, facilitating the formation of SnO₂ grains^{17–19}. XRD peaks further confirmed that the applied thermal energy reduced grain boundaries, thereby enhancing crystal growth as annealing time increased^{20,21}. AFM images revealed the surface morphologies of SnO₂ thin films from the as-deposited state to those annealed for 1.5 hours. After annealing, surface protrusions merged laterally, indicating grain growth and a reduction in surface roughness. When the annealing time exceeded one hour, grain coalescence reduced the vertical distance to the grain tops, leading to decreased roughness²². As a result of fewer defects, the transmittance increased, improving crystallinity and reducing light scattering²³. The absorption coefficient (α) of SnO₂ thin films was measured before and after annealing at different times. All films exhibited a high absorption coefficient at short wavelengths, suggesting an allowed direct electronic transition, particularly in this region²⁴. The optical band gap (E_g) was determined using Tauc's formula²⁵, and was found to increase with annealing time^{26,27}. This widening of the direct optical band gap can be attributed to the enhanced crystallisation of the films upon annealing. Additionally, the Burstein–Moss effect provides a plausible explanation for the observed band gap shift, as it is related to changes in carrier concentration^{28,29}.

5. Conclusion

Tin oxide (SnO₂) thin films were deposited using the spray pyrolysis deposition technique. Two distinct annealing times were applied to the films. X-ray diffraction patterns revealed that all of the deposited samples exhibited a tetragonal structure, and vertical growth enhanced the optical and structural properties of annealed films at 773 K. As the annealing times increased, the average particle diameter also grew, contributing to the improved crystallinity of the thin film and enhancing their transmittance. The optical band gap was measured to be 3.22 eV for the as-deposited film, and increased to 3.54 eV and 3.64 eV after annealing, respectively

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Conflict of Interest

The writers affirm that they have no competing interests.

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None.

Ethical Clearance

The University of Baghdad's local ethical committee gave the project approval.

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