

Effect of Annealing Temperature on Structural And Optical Properties of SnO_2 Thin Films

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Abstract:

SnO_2 thin films were prepared by DC magnetron sputtering technique on glass substrates at 200 °C temperature. Both an X-ray diffractometer and a UV/VIS spectrophotometer were used to examine how the annealing temperature affected the structural and optical characteristics of the films. X-ray diffraction patterns indicated that the SnO_2 films showed a polycrystalline tetragonal structure. The optical properties and dispersion parameters of the films have been studied over a wavelength (300-900) nm. A high optical energy-gap of 3.72 eV was achieved at temperature 400 °C. Many optical parameters such as refractive index, dielectric constant, ratio of carrier concentration to the effective mass, single oscillator energy, dispersive energy, moments of the optical spectrum, the average operator strength, single operator wavelength, optical resistivity, thermal emission, and optical conductivity parameters were determined and examined in relation to the annealing temperature.

Keywords: SnO_2 , Optical properties, Dispersion parameters, DC. magnetron sputtering, Annealing, Structure.

Introduction:

Tin Oxide SnO_2 films has a high energy band gap (3.7 eV), strong optical transparency, and low electrical resistance within the electromagnetic spectrum's visible range. and low electrical resistance are widely used for many applications, particularly as transparent electromagnetic shielding materials, an electrode material in light emitting diodes, solar cells, etc [1].

The properties of SnO_2 films such as its, optical properties, structure and dispersion parameters ... etc can be varied by preparation method. SnO_2 has been created using several techniques such as chemical vapor deposition [2], electron beam evaporation [3], sol gel coating [4], spray pyrolysis [5], magnetron sputtering [6] etc. However, among these methods, magnetron sputtering is significant due to a number of benefits, including the ability to build films at low deposition temperatures, control over the deposition rate, excellent purity, and homogeneity of the coating over wide substrate surfaces [7]. Furthermore, numerous studies are presented with an emphasis on how various factors (such as substrate type and temperature, deposition rate, oxygen partial pressure, and annealing temperature) affect the optical, electrical, and structural characteristics of SnO_2 thin films.

The annealing procedure is one of the most significant variables that may influence SnO_2 thin-film characteristics. Mohamed and Hadia[8] examined The influence of post-thermal annealing on SnO_2 optical properties produced using the electron beam evaporation method. In this work, the investigation focused on studying how the annealing temperature impacts the optical dispersion and structural characteristics the DC magnetron sputtering method of growing SnO_2 thin films on glass substrates.

Many optical parameters such as carrier concentration to effective mass ratio, dielectric constant, and refractive index, single oscillator energy, dispersive energy, moments of the optical spectrum, the single oscillator's wavelength and average oscillator strength, optical conductivity, the values of optical resistivity and thermal emissivity were computed. and analysed with relation to the temperature of annealing.

Experimental

The SnO_2 were deposited by means of DC magnetron sputtering operating at a temperature of 200 °C On glass substrates. An oil-diffusion pump operating at 2×10^{-6} was employed to empty the vacuum chamber, while an Edwards 306 pumping system was employed to remove the vacuum. The experiment lasted 30 minutes and was carried out in a pressurized chamber with 95% argon and 5% oxygen. Here were the factors to consider: 40W direct current where there has to be sufficient energy, electron mobility, and a force that permits the gases to come into close contact with one another, 370

gauss magnetic field, and 30 mm gap between cathode and substrate holder. The glass base was treated with tin oxide that was expelled from targets. The target materials are 60 mm diameter, 2 mm thick SnO_2 powder plates. Thickness film measured by Figure (1) displays an image obtained using scanning electron microscopy (SEM). which was 800 ± 10 nm. Film was post-deposition annealed at (300, 400 and 500) °C for 1 hour. The SnO_2 films' structural characteristics were examined utilizing a $1.5406 \text{ \AA}$ wavelength Cu ($K\alpha$) X-ray diffractometer (XRD). At normal incidence, a double-beam spectrophotometer was used to record the spectrum of optical absorbance and transmittance of each film in the (300-900 nm) spectrum range.

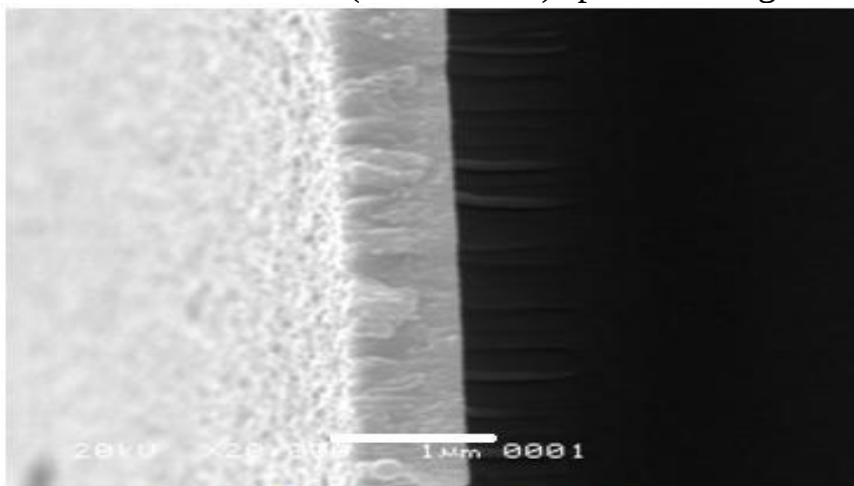


Fig.1. SEM picture of SnO_2 thickness

Results and discussion

Figure (2) displays the SnO_2 films X-ray diffraction patterns for samples that were heated to 200 °C as-deposited and for samples that were annealed in air for 1 hour at 300, 400, and 500 °C. According to Table 1, all of the films include one phase of SnO_2 and have a polycrystalline tetragonal structure. One possible reason for the emergence of strain relief at the crystal grains might be for the small changes in diffraction angles and interplanar of the crystal planes change slightly before and after annealing [9]. Crystalline size D values prior to and following annealing, which are around 14.4 nm and 26.3 nm, respectively, according to Table (1), derived from the famous Scherrer equation [10]:

$$D = k' \lambda / \beta \cos \theta \quad \dots\dots\dots (1)$$

Within this range, k' might be anywhere from 0.89 to 1.39, with 0.9 often being the closest figure. This is the reason behind selecting an X-ray wavelength of 0.9λ , where β represents the full-width at half of the most intense peak's maximum. (i.e. (110)) in radians, and θ is Bragg's angle for the purpose of calculating grain size. The calculated crystallite size values are also shown in Table (1). The film's average crystallite size is 24.6 nm when deposited, but it grows to 28.3 nm at annealing temperatures up to 400°C, but then decrease to 25.5 nm at 500°C. In other words, the maximum crystallinity occurs in the film annealed at 400 °C, the size of the crystallites in thin films is determined by the defect (fewer defects), where a low defect level indicates a high degree of crystallinity.

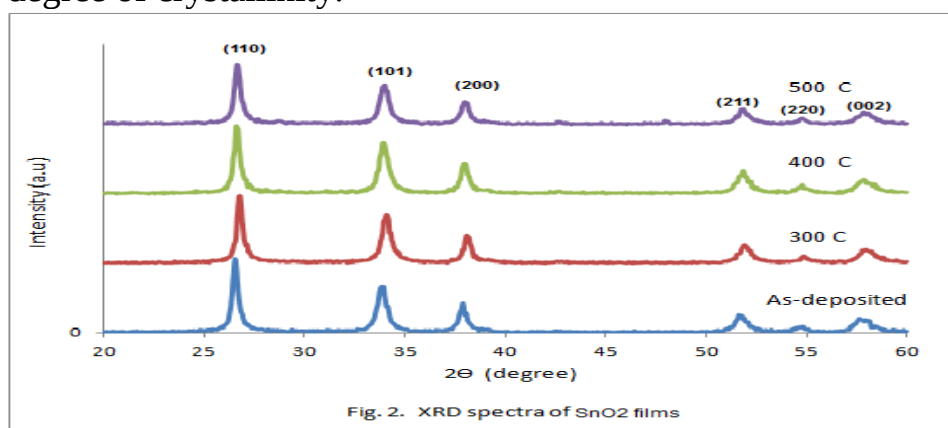
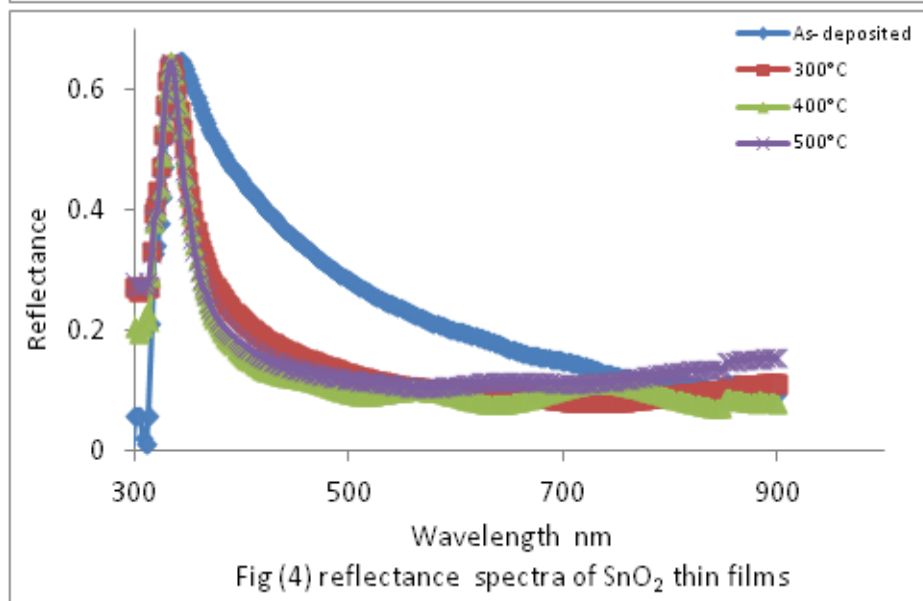
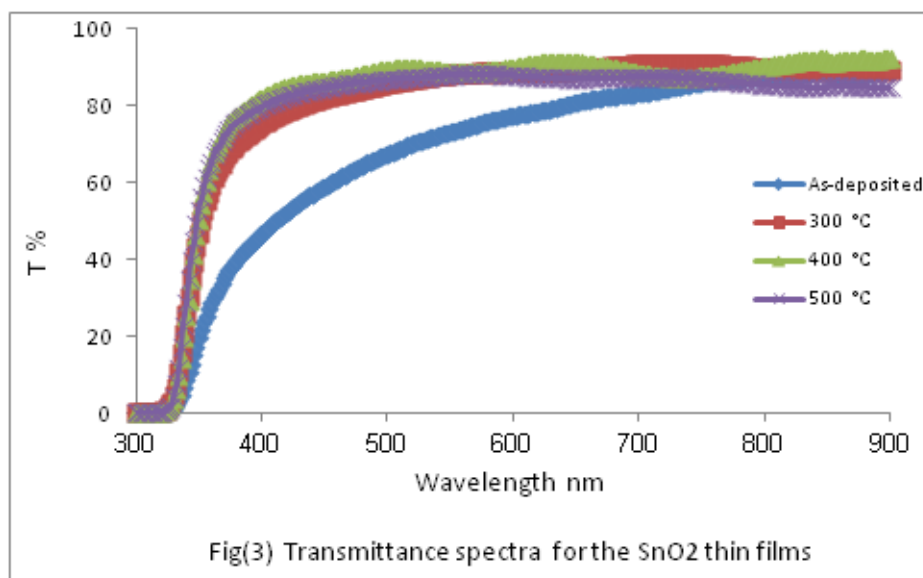


Table (1) Interplanar distance between different planes and crystalline sizes for the SnO₂ thin films

Samples	Standard values		As-deposited at 200°C		Annealed at 300°C		Annealed at 400°C		Annealed at 500°C	
(hkl)	2θ	d(Å)	2θ	d(Å)	2θ	d(Å)	2θ	d(Å)	2θ	d(Å)
(110)	26.61	3.351	26.58	3.350	26.81	3.323	26.67	3.339	26.69	3.336
crystalline sizes D nm			24.6		26.3		28.3		25.5	

The reflectance $R(\lambda)$ and transmittance $T(\lambda)$ spectra of SnO₂ films as-deposited and after they are annealed at 300, 400, and 500 °C with regard to wavelengths ranging from 300 to 900 nm are displayed in Figure (3) and Figure (4), respectively. Annealing films at higher temperatures results in more visible changes to the films' characteristics, such as an increase in

transmittance and a reduction in reflectance. This is particularly true for films annealed between 300 and 400°C.



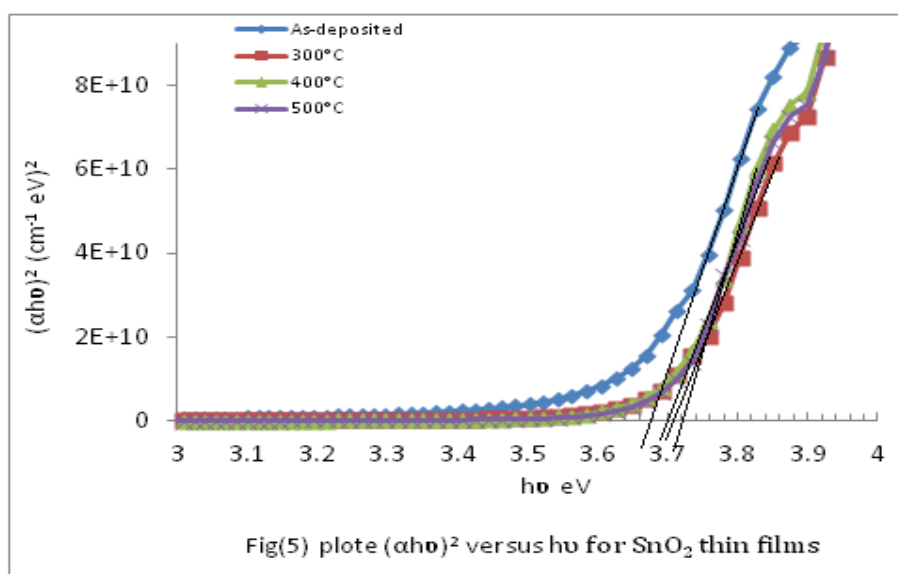
Using the measured values of transmission (T) The following equation was used to get the absorption coefficient α [5]:

$$\alpha = \frac{1}{t} \ln(1/T) \quad \dots\dots\dots(2)$$

Where t is the films thickness.

Using the following formula, the direct optical band gap E_g was calculated [5]:
$$\alpha h\nu = \beta_o (h\nu - E_g)^{\frac{1}{2}} \dots\dots\dots (3)$$

To determine the optical band gap of thin films, the relationship between photon energy ($h\nu$) and the square of the thickness ($\alpha h\nu$) was plotted, as seen in Figure 5, and the intercept of the curve with the $h\nu$ axis was calculated, where β_o is a constant. Band gap energies are expected and may be found in Table 2. The E_g of the films is 3.68 eV when deposited, 3.72 eV after annealing at 400 °C, and 3.7 eV after cooling to 500 °C. An expansion of the optical band gap When the annealing temperature is raised, unsaturated defects are gradually removed, increasing the number of saturated bonds. As a result, the optical gap widens and the density of localized states decreases [11]. However, as demonstrated by XRD, the energy gap decreases as annealing is raised to 500 °C, leading to a rise in defects and a decrease in crystallinity. The E_g values obtained by Camacho et al. are comparable to these [6]. While H. A. Mohamed and N. M. A. Hadia at 500 °C, an impressive 3.65 eV optical energy-gap was attained [8]. The band gap of the orthorhombic SnO_2 phase is reportedly greater than that of the tetragonal phase, according to certain studies. The band gap of orthorhombic SnO_2 that has been laser ablated was found to be 3.79 eV by Kong et al. [12]. Orthorhombic SnO_2 thin films produced by laser ablation were reported by Chen to have a value of 4.02 eV [13]. The band gap at 300 K for bulk tetragonal SnO_2 is 3.6 eV [14].



Optical parameters namely, refractive index n and extinction coefficient k , dielectric constant real part ϵ_r and imaginary part ϵ_i have been determined by using the flowing relations [8,11,15,16]

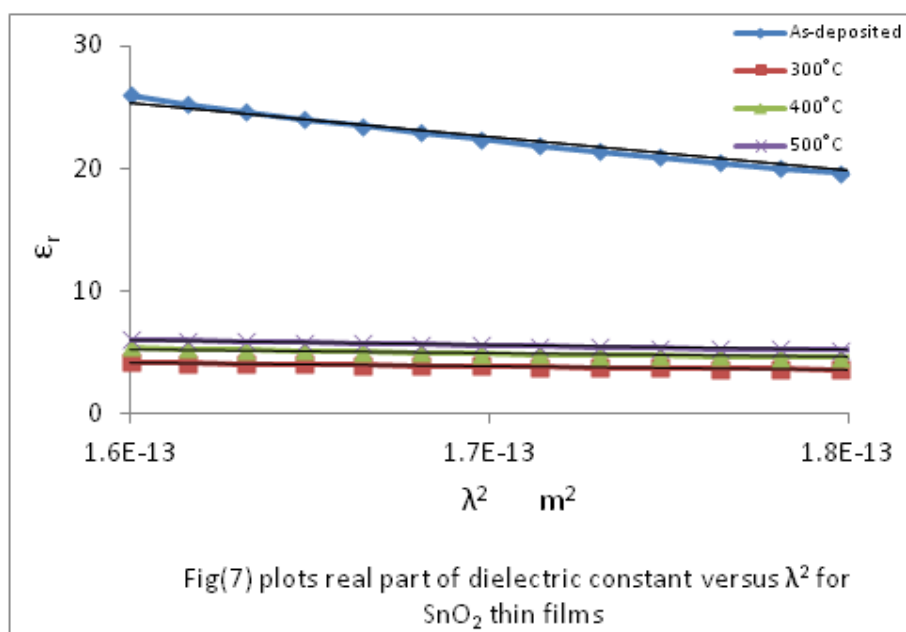
$$k = \frac{\alpha\lambda}{4\pi} \quad \dots\dots\dots(4)$$

$$n = \left[\left(\frac{1+R}{1-R} \right)^2 - (k^2 + 1) \right]^{\frac{1}{2}} + \frac{1+R}{1-R} \quad \dots\dots\dots(5)$$

$$\epsilon_r = n^2 - k^2 = \epsilon_\infty - \left[\frac{e^2 N}{4\pi^2 c^2 \epsilon_0 m^*} \right] \lambda^2 \quad \dots\dots\dots(6)$$

$$\epsilon_i = 2nk = \left[\frac{\epsilon_\infty \omega_p^2}{8\pi^2 c^3 \tau} \right] \lambda^3 \quad \dots\dots\dots(7)$$

Where R is reflectance, e is the electronic charge and ϵ_∞ is the lattice dielectric constant, often known as the high frequency dielectric constant, ϵ_o . It can be noticed that the dependence of ϵ_r on λ^2 is linear at longer wavelengths, from the slope of liner part the ratios N/m^* are computed and shown in Table (2). It is apparent that as the temperature at which annealing occurs increased up to 400°C, the values of the lattice dielectric constant ϵ_∞ and ratios N/m^* fall. After that, they slightly rise, which may be connected to the variations of the energy gap.



Equation (7) is utilized to compute the imaginary component of the dielectric constant, which is then shown as a function of λ^2 in Figure (7). From the slope of linear part, the values of ω_p and τ are calculated. Where ω_p is equal to [15]:

$$\omega_p = \left[\frac{e^2 N}{\epsilon_0 \epsilon_\infty m^*} \right]^{\frac{1}{2}} \dots\dots\dots(8)$$

The obtained ω_p and τ values are listed in Table (2). It is clear that the variation of optical relaxation time follows the same trend as the optical energy gap. Moreover, the plasma frequency decreases as the annealing temperature increases, which is directly correlated with the concentration of free carriers. [17].

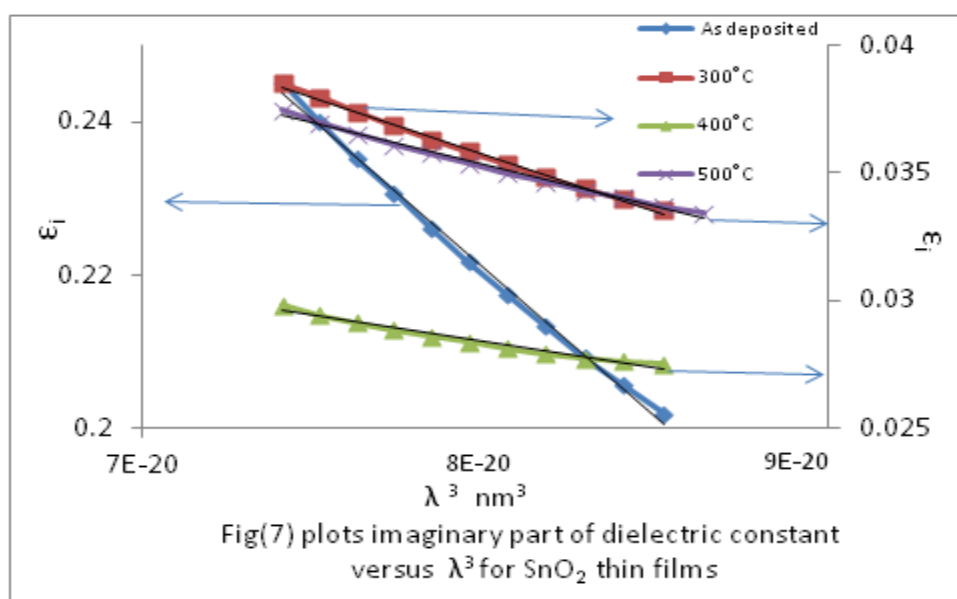


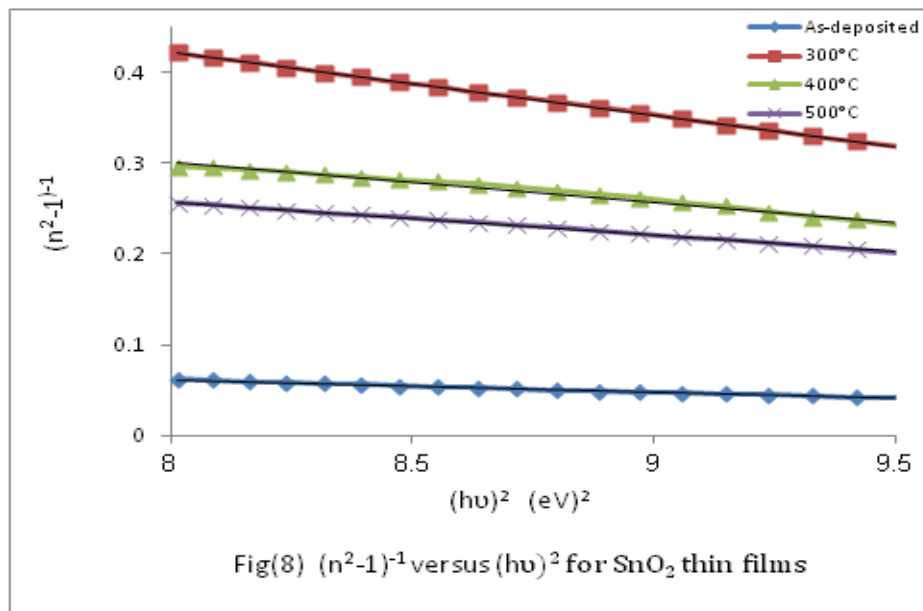
Table (2) Optical parameters for the thin films of SnO_2 at different temperatures of annealing

Parameters Samples	E_g eV	ϵ_∞	$N/m^* \times 10^{18}$ m^{-3}	ω_p $\times 10^{15}$ s^{-1}	$\tau \times 10^{-13}$ s
As deposited	3.68	68.87	65.00	5.2	2.2
Annealed at 300°C	3.71	10.37	6.45	4.7	2.7
Annealed at 400°C	3.72	8.32	6.40	4.2	4.4
Annealed at 500°C	3.7	11.68	8.54	4.6	3.9

The dispersion behavior affects optical communication and the design of devices for spectrum dispersion, it is a crucial aspect of optical material research. Wemple–DiDomenico (W–D) dispersion model, a single-oscillator model of the following form, is used to analyze experimental refractive index data, which typically yields physically meaningful quantities like the oscillator energy and dispersion energy in transparent region for different solids [8,15]:

$$n^2 - 1 = \frac{E_d E_o}{E_o^2 - (h\nu)^2} \quad \dots\dots\dots (9)$$

where the dispersion energy, or average intensity of the interband optical transitions, is expressed as E_d , and the oscillator energy with a single effective is expressed as E_o . The dispersion parameters (E_o and E_d) for every SnO_2 thin film are found by graphing $(n^2 - 1)^{-1}$ versus $(h\nu)^2$, as Figure (8) illustrates. Using the slope $(E_o E_d)^{-1}$ and the intercept on the vertical axis (E_o/E_d), and then can compute the principle of E_o and E_d .



Afterward, the following relationships can be used to derive the values of the optical spectrum's moments, M_{-1} and M_{-3} [8,18]:

$$E_o^2 = \frac{M_{-1}}{M_{-3}} \quad \dots\dots\dots (10)$$

$$E_d^2 = \frac{M_{-1}^3}{M_{-3}} \quad \dots\dots\dots (11)$$

Table (3) lists the principles of E_d , E_o , M_{-1} and M_{-3} . The optical energy gap and dispersion energy E_d behave in the opposite way, whereas the principles of E_o , M_{-1} and M_{-3} fall at an annealed temperature of 400°C and begin to grow at high annealing temperatures.

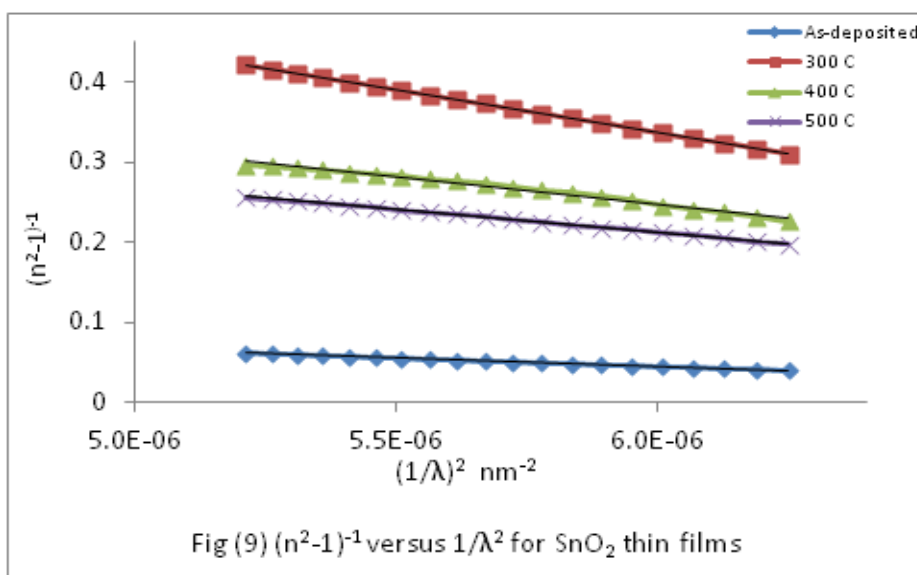
Table (3) Optical dispersion parameters

Samples	E _g eV	E _o eV	E _d eV	M ₋₁	M ₋₃ (eV) ⁻²	E _o /E _g
As deposited	3.68	4.35	15.56	4.35	0.23	1.18
Annealed at 300°C	3.72	4.78	15.36	3.21	0.14	1.29
Annealed at 400°C	3.71	4.92	14.61	2.96	0.12	1.33
Annealed at 500°C	3.70	4.59	15.63	3.41	0.16	1.24

The Sellmeier dispersion formula [19] may also be used to examine the refractive index dispersion data, and it is provided by:

$$n^2 - 1 = \frac{\lambda_0^2 S_0}{1 - \left(\frac{\lambda_0}{\lambda}\right)^2} \quad \dots\dots\dots (12)$$

Where λ_0 is the average oscillator wavelength, S_0 is the oscillator strength. Figure 9 illustrates the relationship between $(n^2 - 1)^{-1}$ and λ^2 for the SnO₂ films. From the slope ($1/S_0$) and intercept ($1/S_0\lambda_0^2$), which are stated in Table (4), we may estimate the values of S_0 and λ_0 . Table (4) displays the relationship between the annealing temperature and the average oscillator wavelength of SnO₂. In contrast to λ_0 , which exhibits the opposite pattern, S_0 values drop during annealing and reach a low of 0.51×10^{13} at an annealed temperature of 400°C. They slightly rise at higher temperatures, which might be explained by variations in the optical energy gap.



There has been a rise in interest in using transparent heat mirror coatings, which are made of transparent conducting oxides and have a low thermal emissivity, to reduce heat radiation loss through window panes. This is motivated by concerns about the environment and sustainability [20]. The following relation can be used to determine the thermal emissivity ϵ [21]:

$$\epsilon = 1 - (T_{IR} + R_{IR}) \quad \dots\dots\dots(13)$$

The relationship between the temperature during annealing and the thermal emissivity of SnO_2 films is shown in Table (4), where T_{IR} and R_{IR} in the near-infrared band ($\lambda=900$ nm) are the average transmittance and reflection, respectively. Obviously, the variance of thermal emissivity maintains a similar pattern to the average oscillator wavelength as shown in Table (3).

The optical resistivity ρ_{opt} of SnO_2 films are determined by applying the subsequent formula [22]:

$$R = 1 - 4(\epsilon_0 c / t) \rho_{opt} \quad \dots\dots\dots(14)$$

Wherever, t is film thickness. As seen from the Table (4) shows the optical resistivity of SnO_2 values increase with annealing temperature increasing to 400°C and then decreasing to 500°C . This variation due to the change in the carrier concentration ratio to the effective mass is known as a free carrier concentrations.

Table (4) oscillator strength, oscillator wavelength, optical resistivity and thermal emissivity of SnO_2 thin films

Parameters Samples	S_o m^2 $\times 10^{13}$	λ_o m $\times 10^{-7}$	ρ_{optical} $\Omega.m$ $\times 10^{-5}$	ϵ $\times 10^{-3}$
As deposited	4.74	3.22	6.37	12.74
Annealed at 300°C	1.47	3.51	6.84	0.39
Annealed at 400°C	0.51	4.49	6.94	0.14
Annealed at 500°C	1.77	3.20	6.72	0.44

Conclusion:

Thin films of SnO_2 deposited using the DC magnetron sputtering method on glass substrates and influence of annealing temperature on films' optical and structural characteristics was investigated. The thin-film XRD analysis indicated polycrystalline SnO_2 tetragonal structure with the best crystallinity obtained at 400°C. The results showed that as the temperature during annealing increased to 400°C, the following parameters decreased: high frequency dielectric constant, N/m^* , plasma frequency, dispersion energy, specific optical spectrum moments, oscillator strength, and thermal emissivity. On the other hand, optical energy gap, optical relaxation time, oscillator energy, oscillator wavelength, and optical resistivity all showed mild increases. Based on the results shown above, it might be inferred that the SnO_2 thin films produced using the DC magnetron sputtering technique and annealed at 400°C are of high quality. These films are ideal for use as transparent conducting oxides (TCOs) in various opto-electronic devices and can also be applied as heat mirror coatings.

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تأثير درجة حرارة التلدين على الخواص التركيبية والبصرية للأغشية الرقيقة SnO_2

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مستخلص البحث:

تم تحضير الأغشية الرقيقة لأوكسيد القصدير SnO_2 بواسطة تقنية الرش DC على ركائز زجاجية عند درجة حرارة 200 درجة مئوية. تم تلدين الأغشية المحضرة في الهواء عند درجات حرارة 300 و 400 و 500 درجة مئوية لمدة ساعة واحدة. تم دراسة تأثير درجة حرارة التلدين على الخواص التركيبية والبصرية للأغشية باستخدام مقياس حيود الأشعة السينية ومقياس الطيف الضوئي UV/VIS. أشارت أنماط حيود الأشعة السينية إلى أن الأغشية كانت أحادية الطور SnO_2 مع بنية رباعية البلورات. تمت دراسة الخصائص البصرية ومعايير التشتت للأغشية على طول موجي (300-900) نانومتر. تم تحقيق فجوة طاقة بصرية عالية تبلغ 3.72 إلكترون فولت عند درجة حرارة 400 درجة مئوية. تم دراسة العديد من المعلمات الضوئية مثل معامل الانكسار، ثابت العزل الكهربائي، نسبة تركيز الموجة الحاملة إلى الكتلة الفعالة، طاقة المذبذب المفرد، طاقة التشتت، لحظات الطيف البصري، متوسط قيم قوة المذبذب، الطول الموجي للمذبذب المفرد، الموصلية الضوئية، الانبعاث الحراري وتم حساب وتحليل المقاومة الضوئية كدالة لدرجة حرارة التلدين.

الكلمات المفتاحية:

SnO_2 ، الخصائص البصرية، معاملات التشتت، التيار المستمر. تقنية الرش DC، التلدين، الخصائص التركيبية.