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Article

Effect of Tin Concentration on The Structure and Color Characteristics of ITO Thin Films Synthesized by Sol–Gel at 400 °C

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Abstract: Annealed at 400 °C. Different tin concentrations were employed. X-ray diffraction revealed a cubic crystal structure with dominant peaks at (222), (400), (320), (622) and (440), With the prevailing trend of growth towards (222). Optical measurements showed that pure indium oxide films exhibited the lowest transmittance in the investigated spectral range, while the addition of 20% and 40% tin significantly increased the transmittance, exceeding 90% in the wavelength range of 550–700 nm, The results showed that the energy gap values increased with increasing tin concentration. The results of the Hall effect test show that the conductivity is good at a high tin concentration 20%. The highest color saturation was obtained at 20% Sn with a dominant wavelength of 660 nm and high a color purity of 0.366 according to the CIE 1931 system.

Keywords: Indium Tin Oxide, Spin Coating, CIE 1931, Sol gel, X-ray diffraction.

1. Introduction

Tin-doped indium oxide (In_2O_3:Sn, commonly known as ITO) has high electrical conductivity and great ability to transmit visible light, making it one of the most popular transparent conductive films. It is widely used in various industrial applications and is of great importance in the field of high technology and scientific research[1]. Thin film technology has witnessed remarkable development as the urgent need to manufacture integrated circuits has increased. The great advantage of thin films is their low cost and small size compared to the material in its bulk form[2]. The casting method is one of the most common and easiest methods for preparing thin films. Its basic idea is based on forming a thin layer of a solution containing the material to be deposited on the surface of the substrate, and then converting this layer into a solid, homogeneous film by drying or thermal annealing. There are two basic forms of this method: dip casting and spin casting[3], [4]. Indium tin oxide (ITO) films were prepared using a gelatinous ink solution via spin-coating technique on glass substrates. Processing conditions were evaluated by studying the effect of rotation speed and solution concentration[5]. The Sol-Gel method is an ideal method for preparing thin films on solid, flat substrates. This method relies on the formation of chemical colloids, where solid raw materials are dissolved in suitable solvents to form a homogeneous solution that later turns into a thin film after drying and heat treatment processes[6]. In this research, the structural, optical and electrical properties were studied, and the colorimetric properties were studied using two systems CIE 1931 and CIE LAB [7]. The samples were characterized using various techniques, including X-ray diffraction (XRD), atomic force microscopy (AFM), field-emission scanning electron microscopy (FESEM), and UV–visible spectrophotometry.

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2. Materials and Methods

Materials

1. $InCl_3 .3H_2O$ (CDH, India).
2. $SnCl_4 .5H_2O$ (LOBA Chemie, India).
3. $[CH_2(OH)CH_2]_2NH$ (THOMAS BAKER, India).
4. Deionized water as solvent.
5. HCL

Experimental Part

Thin films were prepared at three In:Sn ratios (100:0, 80:20, and 60:40). A 0.3 M solution of indium and tin chlorides was dissolved in 35 ml of deionized water and stirred magnetically at 50 °C for 30 minutes. A small amount of diluted HCl was added to eliminate any undissolved particles and impurities. Ethanolamine was then introduced as a stabilizer at a 2:1 molar ratio, and the mixture was stirred for an additional 20 minutes until a homogeneous, light-yellow solution was obtained. The prepared solution was sealed and kept isolated from air for 24 hours, followed by filtration through filter paper to remove residual undissolved materials before deposition. Glass substrates were cleaned thoroughly with deionized water and dried in an oven at 100 °C for 10 minutes to remove surface contaminants that could hinder adhesion and film formation. The deposition was carried out using a spin-coating technique, where drops of the ITO solution were placed at the center of the substrate and spun at 1000 rpm for 30 seconds. After each coating, the films were dried in a convection oven at 180 °C for 10 minutes to remove solvents. This process was repeated three times to achieve the desired film thickness. Finally, the coated substrates were annealed in a convection oven at 400 °C for 30 minutes to enhance film crystallinity and stability.

3. Results and Discussion

The structural properties of indium oxide thin films were investigated using XRD. The films were prepared by the sol–gel technique with varying tin concentrations (0, 20, and 40%) and annealed at 400 °C, as illustrated in Figure (1). The diffraction patterns revealed several characteristic peaks corresponding to the (222), (400), (440), (521), and (622) planes. With increasing tin content, a noticeable decrease in peak intensity was observed, indicating a tendency of the films toward an amorphous state at higher tin concentrations. These findings are in good agreement with previously reported results [8], [9]. The XRD patterns were indexed according to the standard JCPDS cards No. 00-044-1087 and 00-039-1058, confirming that all prepared thin films exhibit a cubic crystal structure.

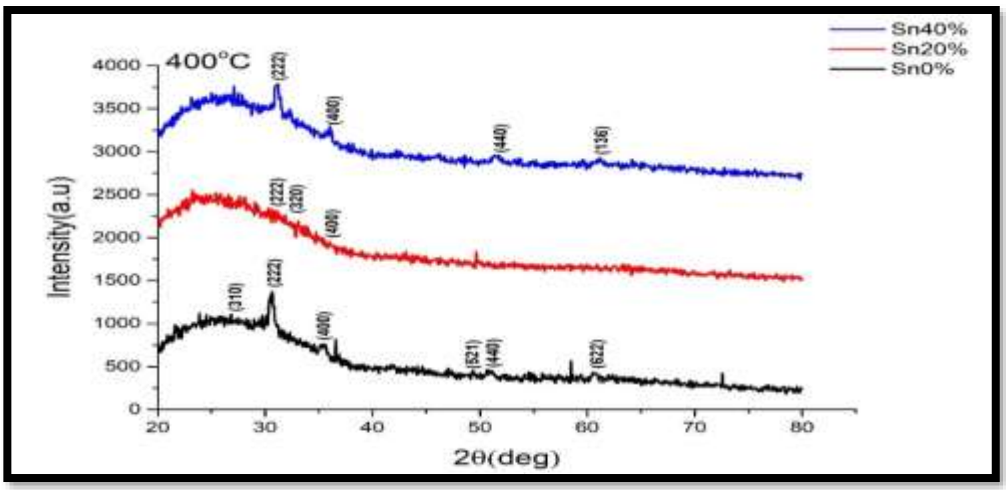

Figure.1 Results XRD with different tin concentrations at annealing temperature 400°C

Table (1) presents the calculated lattice constants of the ITO thin films, which were compared with the reference values from the standard JCPDS data, using the appropriate lattice parameter equation[10].

$$\frac{1}{d_{hkl}^2} = \frac{h^2+k^2+l^2}{a^2} \quad (1)$$

where d is the inter-planer distance and (hkl) are Miller indices.

Table 1. Lattice constants for ITO films

tin ratios(%)	a (Å)	c (Å)
JCPDS ITO	10.118	10.118
100:0	10.102	10.102
80:20	10.219	10.219
60:40	10.271	10.271

The average grain size was also calculated using the Scherrer's formula[11].

$$D_{av} = \frac{k\lambda}{\beta cos\theta} \quad (2)$$

Dav represents the average grain size in nanometers (nm). The constant k is the shape factor, taken as 0.9 depending on the grain morphology. The parameter λ denotes the X-ray wavelength (1.5406 Å), while β refers to the full width at half maximum (FWHM) of the diffraction peak in radians. Finally, θ corresponds to the Bragg diffraction angle at the peak position, expressed in degrees. Figure (2) showing the average grain size for ITO thin films, When indium oxide (In_2O_3) is pure Sn 0% , the grains are of relatively small size due to poor crystal growth due to the absence of catalysts. The addition of tin at a moderate rate, about 20%, leads to the replacement of Sn^{4+} ions with In^{3+} sites in the crystal lattice. This modification enhances the regularity of the crystal lattice and reduces internal defects, which encourages grain growth and increases their size. However, when the tin percentage exceeds the optimum of 20%, the properties of the material change significantly. These results are consistent with the results of researchers[8], [12].

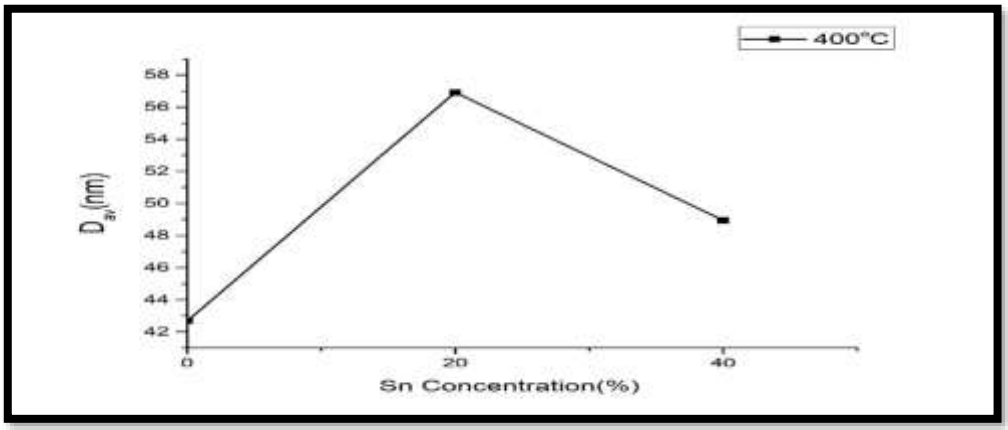

Figure 2. The average grain size for with different tin concentration ratios ITO thin films

AFM was employed to examine the topography of deposited surface films and to analyze the impact of varying mixing ratios on their properties. Figure (3) shows the AFM images. From the Table (2) and Figure (4), At an annealing temperature of 400^{o}C, When indium oxide is in its pure state, the root mean square (RMS) and surface roughness values are relatively low due to the small and uniform grain size distribution. With the addition of 20% tin, both the RMS and surface roughness increase significantly, attributed to enhanced crystalline grain growth, resulting in a more prominent structure on the film surface and, consequently, a higher roughness value, Surface roughness measurements show that when tin is added at a rate of 40% and the annealing process is performed, the surface roughness decreases due to the homogeneous distribution of tin particles within the film. This homogeneity is attributed to the diffusion of heat within the structure, which allows for atomic rearrangement and improved crystal lattice regularity, thus reducing surface roughness This is consistent with the researcher's findings[8].

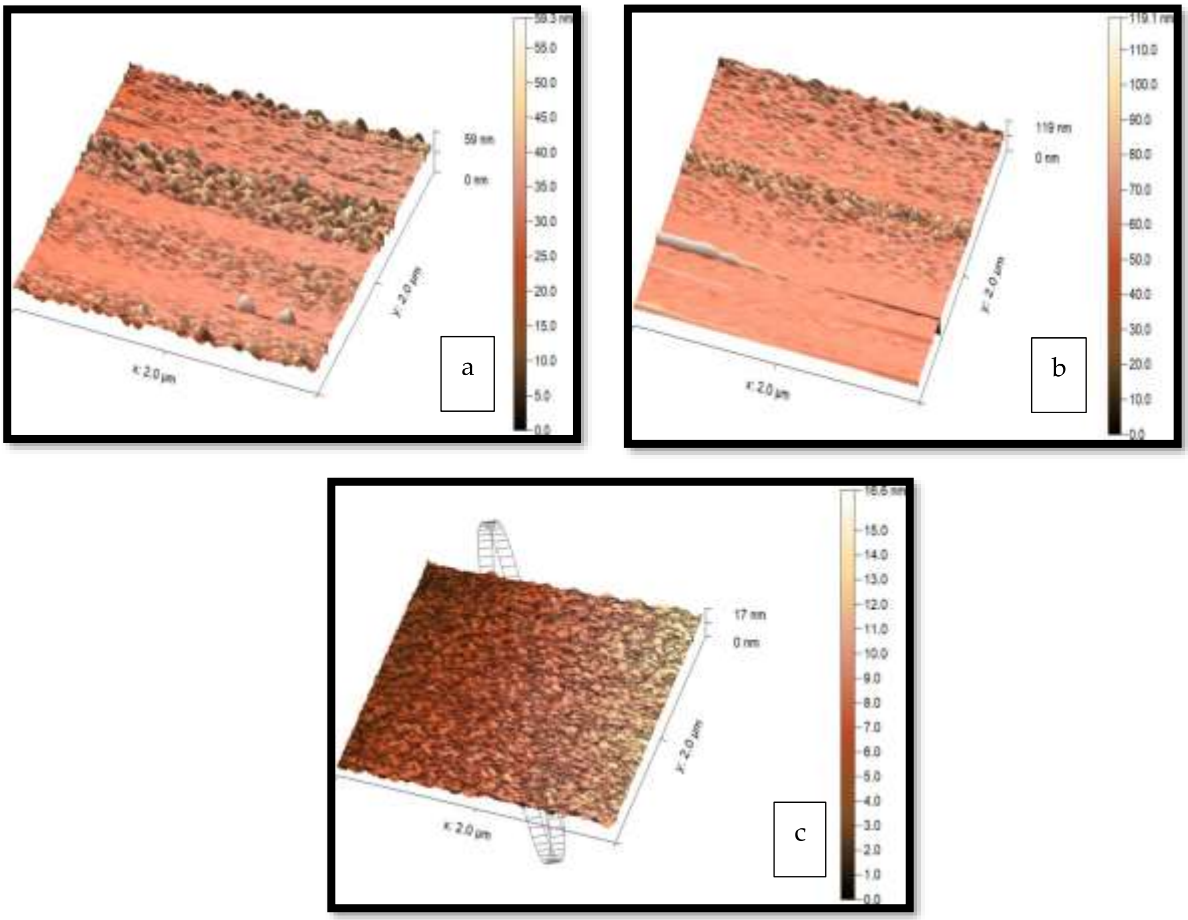

Figure.3. 3D AFM images for ITO thin films with ratios (a) Sn0% (b) Sn20% (c) Sn40%.

Table 2. AFM results of the prepared ITO thin films

tin ratios(%)	Root Mean Square Sq(nm)	Surface roughness Sa(nm)
Sn0%	3.934	2.938
Sn20%	6.409	4.154
Sn40%	2.513	2.077

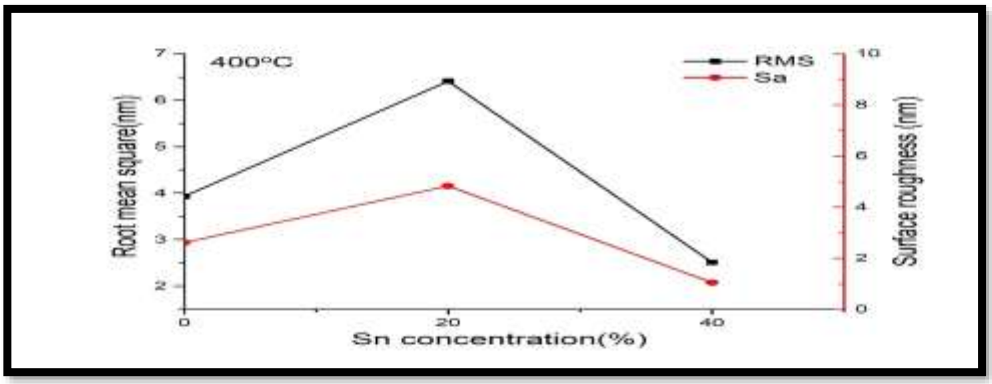

Figure.4. Effect of surface roughness (Sa) and root mean square (RMS) of ITO films

The surface topography was studied using a FE-SEM for ITO films with different mixing ratios and an annealing temperature of 400°C, as shown in Figure (5).

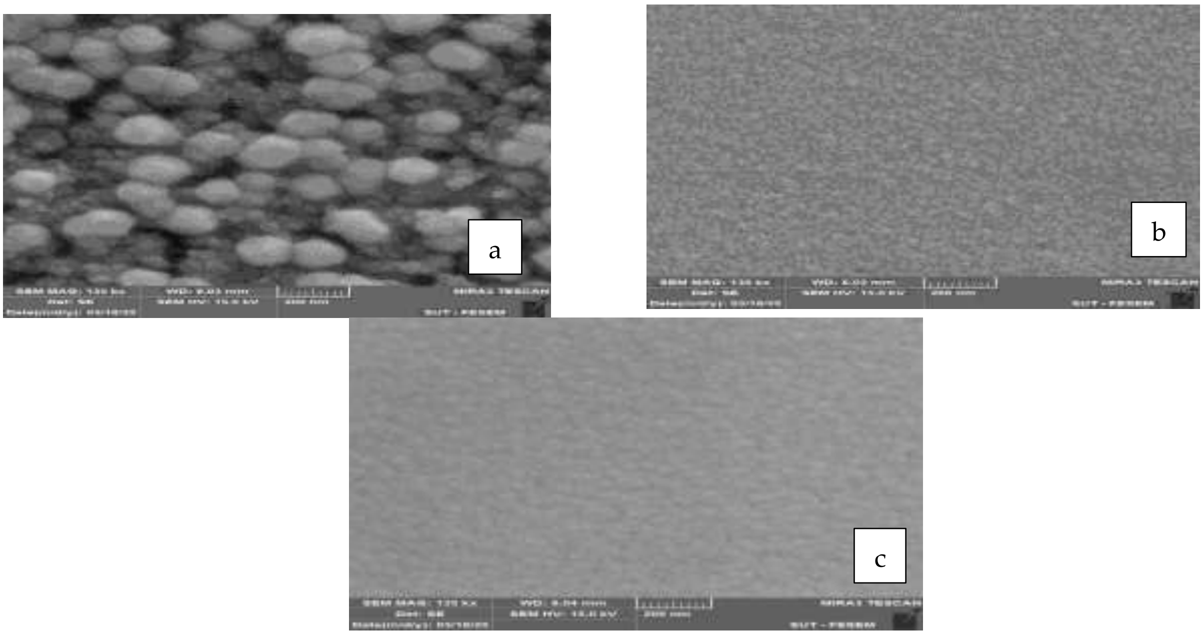

Figure.5. Surface images of ITO thin films using the technique FE-SEM with ratios (a) Sn0% (b) Sn20% (c) Sn40%

An EDX analysis was carried out on the indium tin oxide (ITO) films to identify the elemental composition. The results confirmed the presence of indium (In), tin (Sn), and oxygen (O_2) within the films. Table (3) presents the atomic and weight ratios of the ITO films prepared with different mixing proportions.

Table 3. Values of the Atomic and weight ratios of ITO thin films

tin ratios(%)	**Element**	**Weight (%)**	**Atomic (%)**
Sn 0%	O	55.58	90.03
	In	37.16	8.39
	Sn	7.26	1.59
Sn 20%	O	56.16	90.28
	In	30.59	6.85
	Sn	13.25	2.87
Sn 40%	O	40.72	83.33

	In	33.70	9.61
	Sn	25.58	7.06

The results of the electrical tests showed the Hall effect that the electrical conductivity is at its highest when the tin concentration is 20% due to the presence of abundant charge carriers. It was found that the prepared films of the n-type These results are consistent with the researcher's results[13]. Figure (6) show a change in conductivity with increasing tin concentrations, as shown in the Table (4).

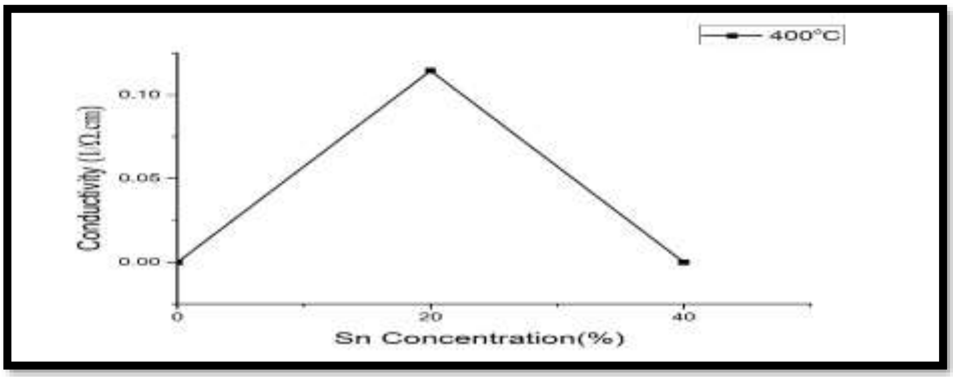

Figure 6. Change in electrical conductivity with tin concentrations in of ITO films

Table 4.Hall effect results with tin concentrations in of ITO films

tin ratios(%)	Resistivity (Ω.cm)	Conductivity (δ) (1/ Ω.cm)	Carrier concentration(cm^{-3}) n-type	Hall Coefficient (R_H)(m^3/C)
Sn0%	$9.93*10^{4}$	$1.01*10^{-5}$	$1.5*10^{6}$	$-4.17*10^{10}$
Sn20%	8.74	$1.14*10^{-1}$	$8.6*10^{17}$	$-7.27*10^{-2}$
Sn40%	$9.73*10^{4}$	$1.03*10^{-5}$	$8.56*10^{6}$	$-7.31*10^{9}$

The optical transmittance curves of the thin films Figure (7) and (8) show a clear change with the different tin concentrations of indium tin oxide. The optical transmittance curves of the prepared thin films show that transmittance values reach their lowest levels in the wavelength region corresponding to the optical energy gap, i.e., in the ultraviolet part of the electromagnetic spectrum. With increasing wavelength within the visible range, particularly in the range (330–400 nm), a gradual increase in transmittance values was observed. It is also evident that different mixing ratios of indium tin oxides (ITO) clearly affect the transmittance curves, with pure ITO films exhibiting the lowest transmittance values within the studied range, starting at about 30% and gradually increasing with wavelength. This decrease in transmittance is attributed to the poor degree of crystallinity and the presence of some internal defects and impurities in the structure. With the addition of tin at 20% and 40%, the optical transmittance values increased significantly, exceeding 90% in the wavelength region (550–700 nm). These results are consistent with what was reported by a number of researchers in previous studies[9]. Figure (9) show the results of energy gap.

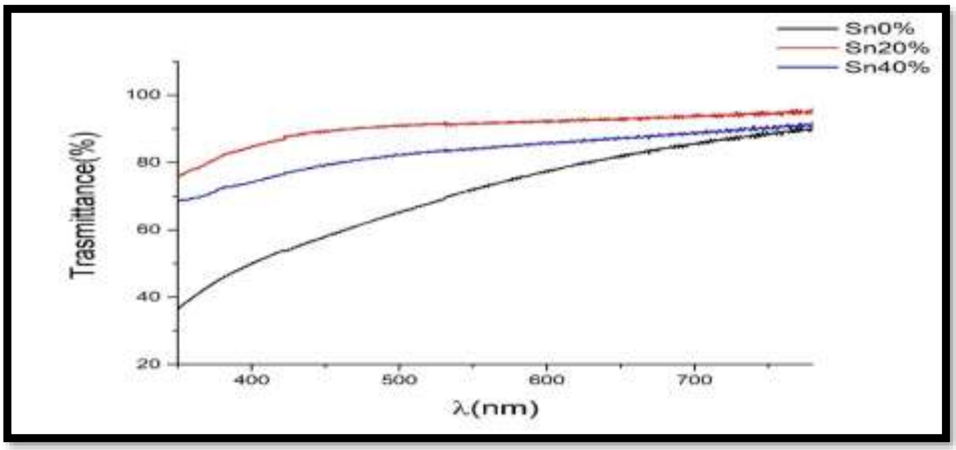

Figure.7. Transmittance as a function of wavelength for ITO thin films with varying tin concentrations

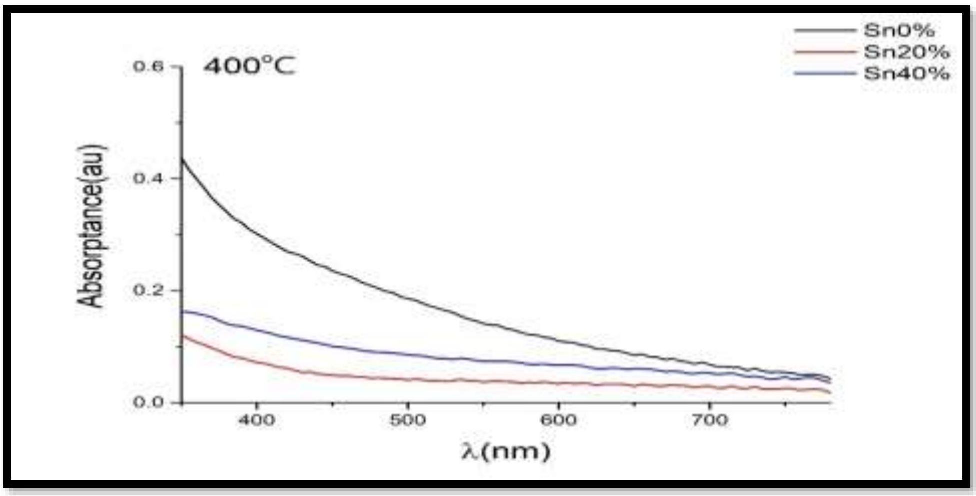

Figure 8. Absorptance as a function of wavelength for ITO thin films with varying tin concentrations

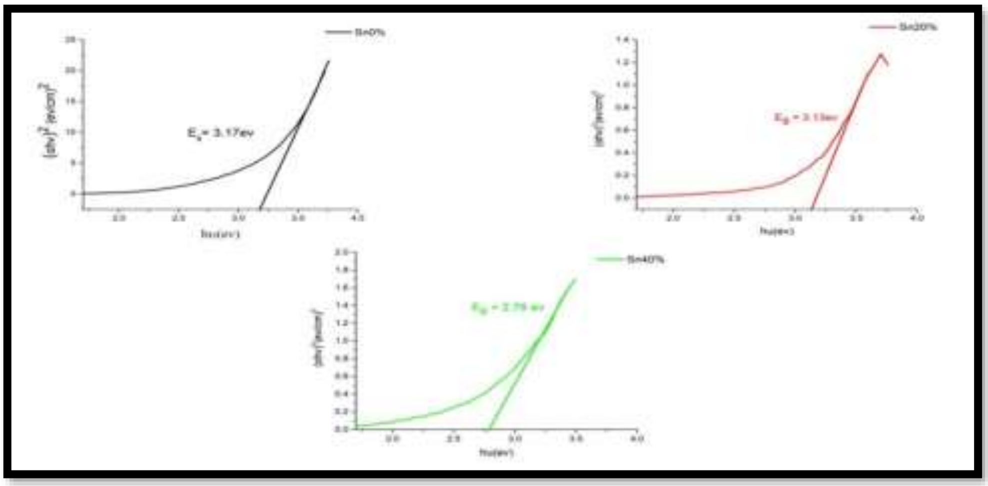

Figure 9. Energy gap values for ITO thin films with varying tin concentrations

These data are obtained by measuring the light spectrum with a UV-Visible Spectrophotometer, which generates the spectrum curve for the transmittance of materials like tin oxide within the visible wavelength range of 380 nm to 770 nm. From this analysis, a table containing the spectral transmittance (T_λ) values at specific, equally gradual wavelengths is derived, To calculate the values and chromaticity coordinates (x, y, z) of the transmitted light, a series of calculations are followed, based on the reference values defined by the CIE system[14], [15].

$X_T = \sum P_\lambda T_\lambda X_\lambda$ (3)
$Y_T = \sum P_\lambda T_\lambda Y_\lambda$ (4)
$Z_T = \sum P_\lambda T_\lambda Z_\lambda$ (5)

P_λ: is the power distribution curve of the light source used. X_λ, Y_λ, Z_λ: are the values of the distribution coefficients. It is noteworthy that the CIE1931 system defines three color values: brightness, purity, and dominant wavelength. Table (5) shows these values, and the Figures (10-11) show the change in purity and brightness values with increasing Tin concentration. It is clear from the figures that the highest brightness is when the tin concentration is Sn20%, while the color purity is higher when the indium is pure Sn0% without adding any tin concentration.

Table 5. The CIE1931 color system for ITO thin films

tin ratios(%)	**Dominant Wavelength**	**Brightness**	**Purity**
Sn0%	615	74.15	0.366
Sn20%	660	91.99	0.239
Sn40%	640	84.92	0.266

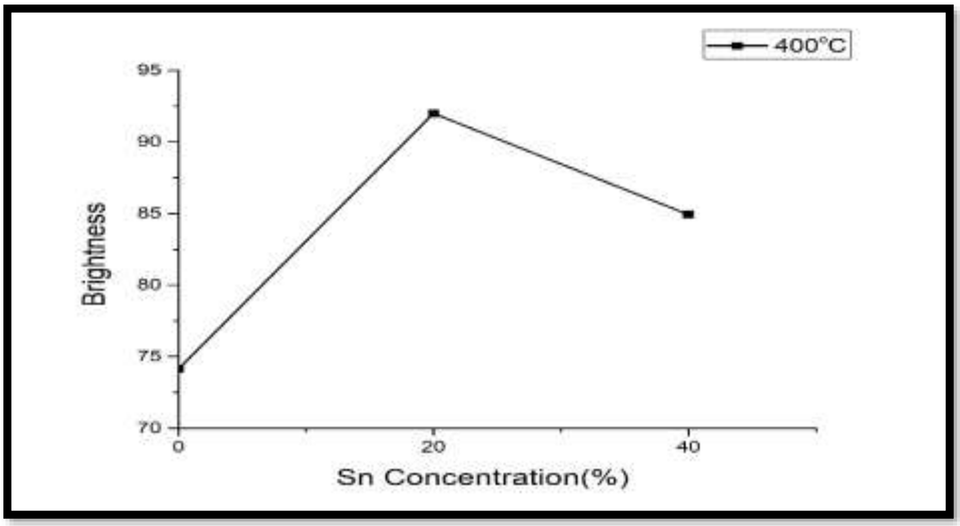

Figure.10. The color brightness for ITO thin films with various tin Concentration

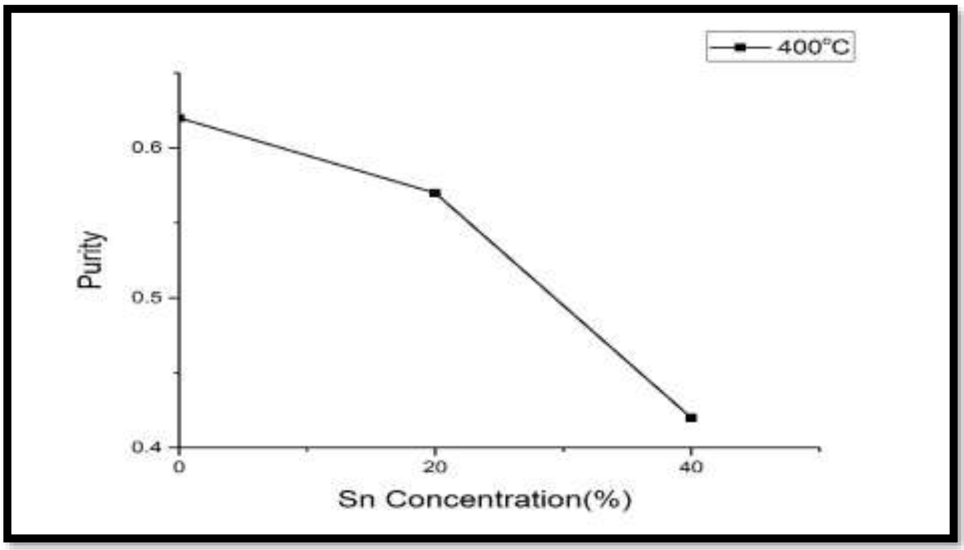

Figure.11. The color purity for ITO thin films with various tin Concentration

In the CIE LAB system, there are three basic color values. The first value is (a*). The second value of the system is (b*). The third value is (l*), which represents the lightness, after performing its mathematical transformations. Two color values are also derived in the CIE LAB system, namely the color angle (h_{ab}*) and the color amount (C_{ab}*). Their value increases counterclockwise during the color coordinate process, while the color amount (C_{ab}*) shows how dark or lighter[16], [17].

$$l^* = 116\left(\frac{Y}{Y_n}\right)^{\frac{1}{3}} - 16 \quad (6)$$

$$a^* = 500\left[\left(\frac{X}{X_n}\right)^{\frac{1}{3}} - \left(\frac{Y}{Y_n}\right)^{\frac{1}{3}}\right] \quad (7)$$

$$b^* = 200\left[\left(\frac{Y}{Y_n}\right)^{\frac{1}{3}} - \left(\frac{Z}{Z_n}\right)^{\frac{1}{3}}\right] \quad (8)$$

$$h_{ab} = tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (9)$$

$$C^*_{ab} = ({a^*}^2 + {b^*}^2)^{\frac{1}{2}} \quad (10)$$

The Figure (12) show the amount and angle of color with different Sn Concentration and the Table (6) shows the values for the CIE LAB system.

Table 6. The values for the CIE LAB system for ITO thin films

tin ratios(%)	**(h_{ab}) Hue angle**	**(C^*_{ab}) Chroma**	b^*	a^*	l^*
Sn0%	21.81	65.53	24.53	60.84	88.99
Sn20%	13.90	56.79	13.64	55.13	96.81
Sn40%	15.59	57.77	15.52	55.64	93.85

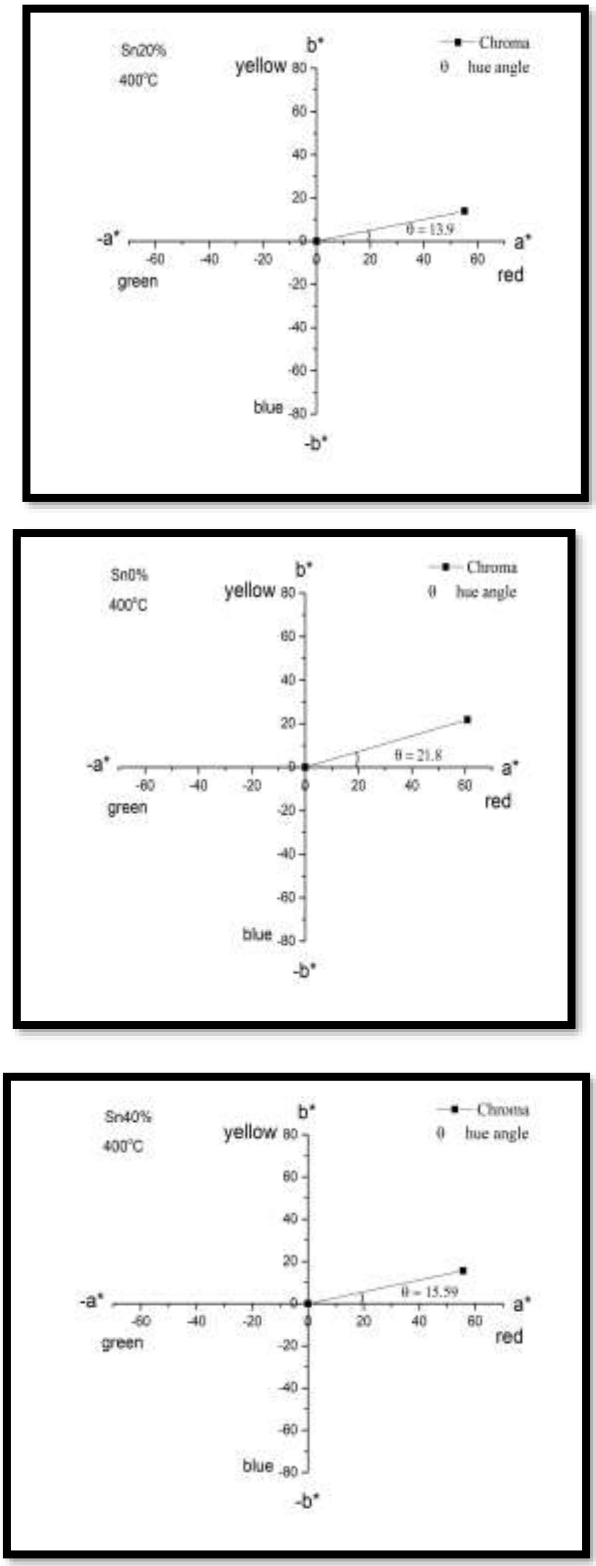

Figure 12. Metric Chroma and metric hue angle of the CIE LAB system for ITO thin films

4. Conclusion

Indium tin oxide (ITO) thin films were fabricated using the spin-coating technique and deposited onto glass substrates with varying tin concentrations, followed by annealing at 400 °C. XRD analysis confirmed that the films exhibited a cubic crystal structure. A slight increase in tin concentration resulted in larger average grain sizes, indicating improved crystallinity at lower doping levels. However, further increasing the tin concentration led to a reduction in grain size. The pure indium oxide sample showed the lowest transmittance within the investigated spectral range, while the incorporation of 20% and 40% tin significantly enhanced the optical transmittance, reaching values above 90% in the wavelength range of 550–700 nm. Colorimetric calculations demonstrated that the highest color intensity was obtained at 20% Sn concentration, with a dominant wavelength of 660 nm and high Brightness 91.99 , a color high purity of 0.366 at Sn0%, according to the CIE 1931 chromaticity system.

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