

# Effect Of Deposition Temperature On The optical Properties Of Iron-Doped Cadmium Stannate Thin Films Deposited By Spray Pyrolysis

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**ABSTRACT:** Spray Pyrolysis Technique (SPT) was used to deposit thin films (TFs) of cadmium stannate, doped with iron (Fe) for the first time. The precursor solutions that were used were prepared at a concentration of 0.1M, doped with Fe and then sprayed onto the pre-heated glass slides immediately at deposition temperatures of  $350 \pm 7^\circ\text{C}$ ,  $400 \pm 7^\circ\text{C}$  and  $450 \pm 7^\circ\text{C}$ . The optical properties of the deposited Fe-doped TFs were studied in the wavelength range of 300-1100nm. The X-Ray diffraction (XRD) studies showed that the TFs were orthorhombic with growth orientation along the (2 0 0) direction. Film thickness of  $85 \pm 10$  nm was obtained. It was found out that an increase in the deposition temperature increased transmittance and band gap, but decreased absorption coefficient, extinction coefficient and refractive index, which was attributed to the improvement in crystallinity of the deposited TFs. Doping reduced transmittance and band gap, but increased refractive index, absorption coefficient and extinction coefficient, which was attributed to the increase in free carriers.

**KEYWORDS:** cadmium stannate, deposition temperature, iron-doped, optical properties of cadmium stannate.

## I. Introduction

This article is divided into the following sections:

**Introduction:** An overview of transparent conducting oxides is given, including the previous research and their applications. The disadvantages of indium tin oxide, the most dominant transparent conducting oxide is also given, which gives the insight into the research for alternatives.

**Experimental details:** The procedure that was applied in preparing the TFs is given in this section. How deposition was carried out is also explained in this section. The section concludes with the measurements that were made on the deposited TFs.

**Results and discussion:** The results obtained from the experiment are presented, discussed and interpreted in this section. Comparisons of the optical properties obtained in this study with some of the already published work are also given in this chapter.

Transparent conducting oxide (TCO) TFs have extensively been used in a variety of electronic and optoelectronic applications because of their high transmission in the visible region, high infrared reflection and low direct current resistivity<sup>1</sup>. The great mass market of panel flat display is the main reason for TCO TFs research. The TCO film in practical applications nowadays is the tin-doped indium oxide, also known as indium tin oxide (ITO). However ITO has two main drawbacks: Firstly, it is rare and secondly, it is expensive<sup>2</sup>. The above drawbacks of ITO call for cheaper and readily available materials for TCOs. Cadmium stannate ( $\text{Cd}_2\text{SnO}_4$ ) has emerged as an excellent alternative candidate TCO material to the ITO due to its low cost as well as being abundant. Moreover,  $\text{Cd}_2\text{SnO}_4$  has comparable optical properties to ITO with a transmittance of up to 99% and a band gap of above 3.3 eV<sup>3</sup>, making it transparent over a large wavelength range. Because  $\text{Cd}_2\text{SnO}_4$  possesses good optical properties, its TFs have found a wide range of applications in transparent electrodes such as photovoltaic (PV) cells, liquid crystal displays (LCDs), window films and smartphone touch screens<sup>4,5</sup>. Most researches have focused on the improvement of electrical and optical properties of  $\text{Cd}_2\text{SnO}_4$  TFs by optimizing conditions like deposition temperature, annealing, addition of a second layer as well as doping it with non-metals like argon, nitrogen, hydrogen and chlorine; where the properties (electrical, electronic, optical and structural) have been found to improve. Doping  $\text{Cd}_2\text{SnO}_4$  with metals such as Zinc<sup>6</sup> and Yttrium<sup>7</sup> has also been done, although the studies did not consider the optical properties

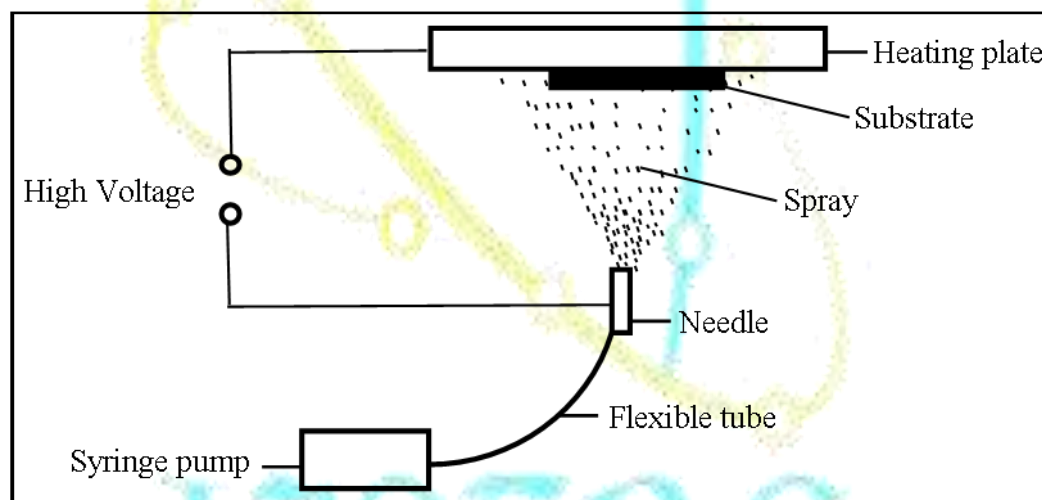
of the deposited TFs, but rather the structural, electrical and electronic. Fe has been studied to produce transparent TFs within the wavelength of 600-2000 nm<sup>8</sup>. Characterization of Cd<sub>2</sub>SnO<sub>4</sub> doped with Fe thus provides an opportunity of obtaining a variety of materials that can be used for the fabrication. The objective of the study is to investigate the effect of deposition temperature on the optical properties of iron-doped Cd<sub>2</sub>SnO<sub>4</sub> TFs.

## II. Experimental details

### 2.1 Film preparation

The microscope glass substrates used in the experiment were cleaned by dipping them in AquaRegia (3:1 of hydrochloric acid: nitric (V) acid) for 12 hours, after which they were removed and then rinsed using deionised (DI) water<sup>9</sup>. Whatman lens cleaning tissue, model number 105 (100 x 150mm) was used to wipe the glass substrates dry.

The precursors used were cadmium acetate dihydrate (99.99% pure), tin II chloride dihydrate (99.99% pure) and iron III chloride (98% pure), all obtained from Central Drug House (CDH) (P) Ltd. 7/28 Vardaan House, Daryaganj, New Delhi – 110002 (India). All the three compounds were dissolved separately in absolute ethanol (99.99% pure) at a concentration of (0.1M). The cadmium acetate solution was made by dissolving 6.67g of the compound in 250 ml of the ethanol. The tin II chloride solution was made by dissolving 5.64g of the compound in 250 ml of the ethanol. The iron III chloride solution was made by dissolving 4.06g of the compound in 250 ml of the ethanol. The cadmium acetate and tin II chloride solutions were then mixed at a ratio of 1:1 by volume, forming a white precipitate. (3ml) of dilute hydrochloric acid (2M) was then added to the precipitate, after which it dissolved to form a colourless solution of Cd<sub>2</sub>SnO<sub>4</sub><sup>6</sup>. The colourless solution (Cd<sub>2</sub>SnO<sub>4</sub>) was finally mixed with the dopant (Fe in the form of iron III chloride) at 0-8% by volume of Cd<sub>2</sub>SnO<sub>4</sub> solution. This solution mixture was then poured into the syringe (gun), then sprayed onto the preheated microscope glass substrates by SPT (model number HO-TH-04), that were placed on a hot plate at a constant temperature of 450±7°C (Fig. 1). The deposition temperature was then varied to 400 °C and 350°C in order to obtain different TFs.



**Figure 1: The process of depositing thin films**

### 2.2 Spectroscopic measurements

The measurement of the masses of cadmium acetate, tin II chloride and iron III chloride that were dissolved in ethanol to form the spray solutions were made using the Mettler Toledo PB303 analytical balance of accuracy of 0.001g and maximum capacity of 310g.

The measurements of transmittance and reflectance were made using the Shimadzu Solid Spec 3700 Deep Ultraviolet Spectrophotometer in the wavelength range of 300-1100 nm in steps of 1 nm. The spectrophotometer had an accuracy of ±0.3 nm in the UV-VIS wavelength region and ±0.32 nm in the NIR region, with a resolution of 0.1 nm. The sample was illuminated with light from a 50W halogen lamp.

The XRD analysis was done using Phillips X-Ray diffractometer system with  $CuK_{\alpha}$  source of wavelength of 1.540598 Å. The analysis was done through an angle of 2θ ranging from 5° to 90° in steps of 0.05°.

The chemical composition of the TFs was determined by XPS, ESCALAB 250 Xi, Thermo-Fisher Scientific with an accuracy of (1000 ppm). Monochromatic Al-K $\alpha$  radiation of energy of 1486.6 eV was used as the excitation.

The measurement of film thickness was made using the computerized KLA-tensor alpha-step IQ surface profiler with a resolution of 0.014  $\mu\text{m}$  and a thickness range of 8 nm-2 mm step heights with an accuracy of 0.75 nm. The profiler characterized the surface by scanning with a diamond-tipped stylus from the bare portion of the TF to the coated portion. A thickness of  $85 \pm 10$  nm was obtained.

### III. Results and discussion

#### 3.1 XPS and XRD studies

Fig.2 shows the results of XPS and XRD analysis done on the TFs. The intensity of the peaks increased with an increase in the deposition temperature. This can be attributed to the improvement in the crystallinity with deposition temperature. The TFs deposited at 350°C were amorphous and are not presented here. The crystalline TFs had growth orientation along (2 0 0) direction in the orthorhombic structure (ASTM 20-0188), which is in agreement with the findings of Krishna et al<sup>3</sup>, who observed that the TFs of Cd<sub>2</sub>SnO<sub>4</sub> are orthorhombic with growth orientation along the (1 1 1) direction. The peaks, diffraction angles and d spacing (interplanar distance) values are shown in Table 1.

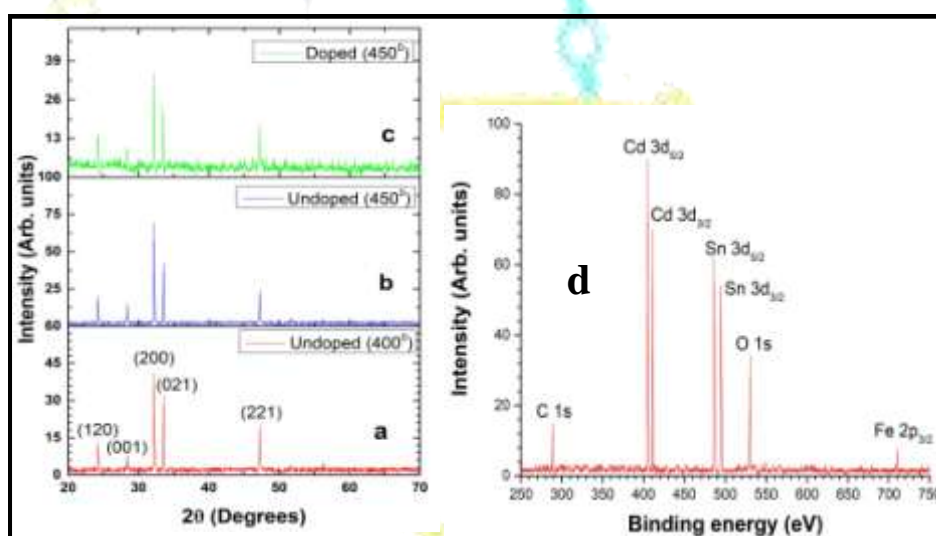


Figure2: (a, b and c) XRD studies and (d) XPS studies on the deposited TFs

Doping did not change the structure of the TFs as can be observed in Figs.2a, b and c, although the doped TFs produced weaker peaks compared to the undoped TFs at the same deposition temperature (comparing Figs.1b and c). The reduction in the intensity of the peaks upon doping implies that the crystallinity of the TFs decreased with doping, which implies that the crystallinity is deteriorated with poorly incorporation of Fe, which can be attributed to the Fe atoms being able to incorporate interstitially into the lattice of Cd<sub>2</sub>SnO<sub>4</sub> instead of occupying the proper lattice positions. The poor incorporation of foreign bodies into the lattice of Cd<sub>2</sub>SnO<sub>4</sub> has also been reported by Dislich and Hinz<sup>10</sup>. The poor incorporation causes a structural disorder, leading to a change in the interplanar distance  $d_{hkl}$  as can be observed in Table 1.

The undoped TFs gave a unit cell volume of 172.32  $\text{\AA}^3$ , while those of the 8% doped TFs gave 173.49  $\text{\AA}^3$ . The lattice parameters obtained in this study are in agreement with some of the previous work. Dawar and Joshi<sup>11</sup> obtained the following values:  $a = 5.568 \text{\AA}$ ,  $b = 9.887 \text{\AA}$  and  $c = 3.192 \text{\AA}$ , with *pbam* space group (number 55) and a unit cell volume of 175.722  $\text{\AA}^3$ . Setty and Sinha<sup>12</sup> obtained the lattice parameters of Cd<sub>2</sub>SnO<sub>4</sub> as  $a = 5.546\text{-}5.684 \text{\AA}$ ,  $b = 9.869\text{-}9.888 \text{\AA}$  and  $c = 3.189\text{-}3.193 \text{\AA}$ , with a unit cell volume of 174.545-179.457  $\text{\AA}^3$ .

The lattice parameters of the doped TFs are higher than those of the undoped TFs (Table 1), which confirms the interstitial incorporation of Fe into the lattice of Cd<sub>2</sub>SnO<sub>4</sub> instead of occupying the proper lattice positions<sup>10</sup>.

Fig. 2b shows that the XPS spectra contained characteristic peaks of Cd, Sn, O, Fe and a weak peak of C at 288 eV. The peak for C can be attributed to the contamination in the samples. The weak peak of C has also been reported by<sup>13</sup> at an energy of 289 eV.

The binding energy for Cd 3d<sub>5/2</sub> was found to be 404.71 eV while that of Cd 3d<sub>3/2</sub> was found to be 411.52 eV, indicating that Cd was in the Cd<sup>2+</sup> bonding state. The spin-orbit splitting between Cd 3d<sub>3/2</sub> and Cd 3d<sub>5/2</sub> was found to be 6.81 eV. The binding energy for Sn 3d<sub>5/2</sub> was found to be 495.18 eV while that of Sn 3d<sub>3/2</sub> was found to be 486.55 eV, indicating that the Sn was in the Sn<sup>4+</sup> bonding state. The spin-orbit splitting between Sn 3d<sub>3/2</sub> and Sn 3d<sub>5/2</sub> was found to be 8.63 eV. The binding energy for O 1s was found to be 530.05 eV. The values of binding energy obtained from the XPS analysis in this study are in agreement with those of Golestani-Fard and Mackenzie<sup>14</sup>, who obtained a value of 531.2 eV for O 1s, 404.7 eV for Cd 3d<sub>5/2</sub> and 486.2 eV for Sn 3d<sub>5/2</sub>. The binding energy values obtained in this study are typical of Cd<sub>2</sub>SnO<sub>4</sub><sup>7</sup>. The binding energy for Fe was detected with a weak intensity, which occurred only in the doped TFs, with a value of 710.2 eV. This indicates a bonding state of Fe<sup>3+</sup>. The weak Fe peak in the doped TFs is due to the low concentration of Fe in the starting solution.

**Table 1: d spacings for the strongest peaks and the calculated lattice parameters of Cd<sub>2</sub>SnO<sub>4</sub> films deposited at the three temperatures**

Sample	2θ (Degrees)	Peak	d <sub>hkl</sub> (Å)
Cd <sub>2</sub> SnO <sub>4</sub>	24.25	(1 2 0)	3.6673
	28.4	(0 0 1)	3.1401
	32.2	(2 0 0)	2.7777
	33.55	(0 2 1)	2.6690
	47.25	(2 2 1)	1.9221
Cd <sub>2</sub> SnO <sub>4</sub> : Fe	24.2	(1 2 0)	3.6748
	28.35	(0 0 1)	3.1456
	32.15	(2 0 0)	2.7819
	33.5	(0 2 1)	2.6728
	47.2	(2 2 1)	1.9241

Parameter	a	B	C
Undoped (Å)	5.555	9.765	3.1766
Doped (Å)	5.564	9.788	3.1456

### 3.2 Transmittance

The curves in Fig. 3 show the transmittance that was obtained from the deposited TFs. The TFs deposited at 450 °C gave the highest transmittance of 78.45%, followed closely by those deposited at 400 °C at 75.12%. The TFs deposited at 350 °C had the least transmittance of 74.33% at the upper end of the visible spectrum (700 nm wavelength). The TFs deposited at 450 °C gave the highest transmittance of 78.45%, followed closely by those deposited at 400 °C at 75.12%. The TFs deposited at 350 °C had the least transmittance of 74.33% at the upper end of the visible spectrum. Jianchao et al<sup>15</sup> also observed the increase in transmittance with an increase in the deposition temperature in deposited lanthanum titanate TFs. The increase in transmittance with the increase in the deposition temperature in this study can be explained by the fact that high deposition temperature enables the atoms to land on the substrate in an ordered manner, since high temperature enables them to rearrange themselves, thus improving the crystallinity as can be observed in Fig. 2a and b. With the improvement in crystallinity, the light wave scattering decreases, resulting in better transparency of the TFs<sup>16</sup>. The highest transmittance for the TFs deposited at 450 °C also implies that there was low absorbance or reflectance of the incident photons by the TFs.

All the curves in Fig.3 tend to converge at the origin (tend to zero) at short wavelengths below 300 nm. This is due to strong absorption of the incident light by the TFs in the short wavelength range (UV region)<sup>17</sup>.

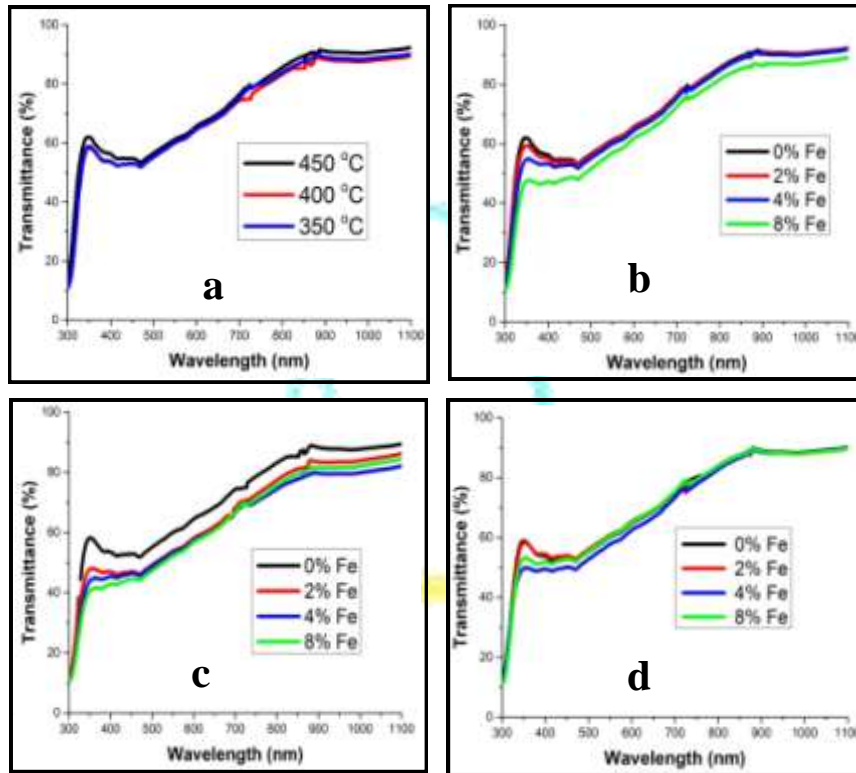


Figure. 3: Transmittance against wavelength for (a) undoped cadmium stannate thin films deposited at the three temperatures, (b) cadmium stannate deposited at 450°C with 0-8% iron, (c) cadmium stannate deposited at 400°C with 0-8% iron and (d) cadmium stannate deposited at 350°C with 0-8% iron.

Figs.3b, c and d show the doped TFs at 0-8% Fe at the three deposition temperatures. As can be observed, transmittance decreased with increase in the concentration of the dopant. This is due to the free charge carriers introduced by the dopant, which then absorb and reflect more of the incident photons, thus reducing transmittance. The decrease in transmittance of TFs upon doping is in agreement with the results obtained by Maity and Chattopadhyay<sup>18</sup>, who attributed it to the defects of the dopant (aluminium) in the deposited TFs.

### 3.3 Absorption coefficient

Absorption coefficients of the deposited TFs shown in Fig.4 were determined from the formula<sup>19</sup>:

$$\alpha = \frac{\ln\left(\frac{1}{T}\right)}{t} \quad (1)$$

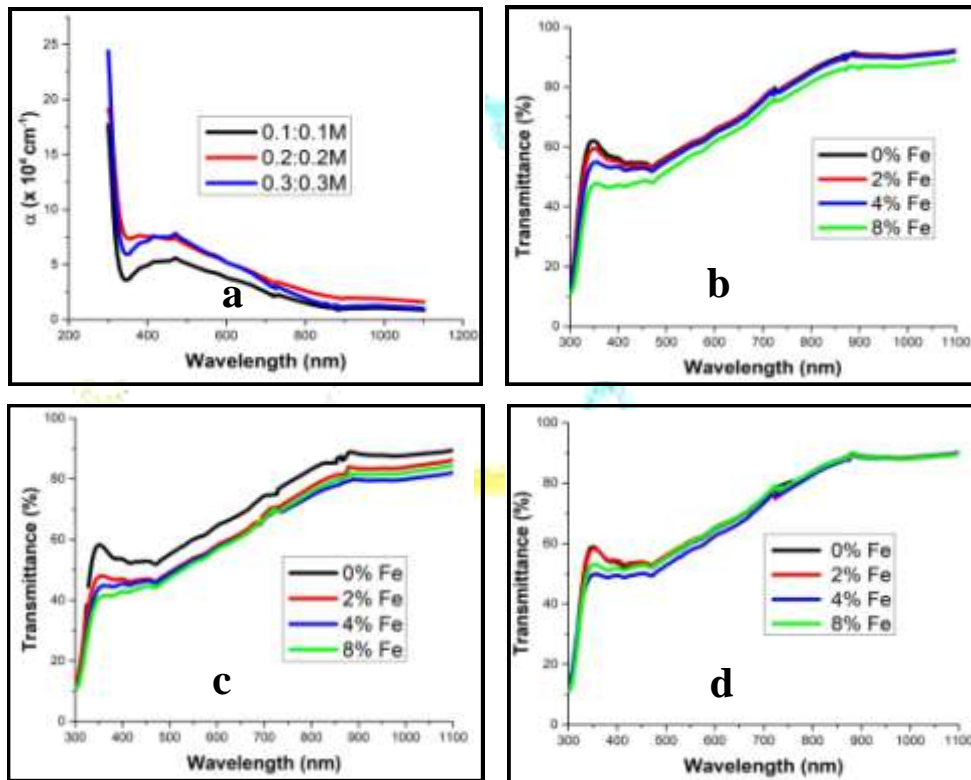
Where T is the transmittance and t is the film thickness in cm.

Absorption coefficient decreased with an increase in the deposition temperature. The TFs deposited at 350 °C produced the highest absorption coefficient of  $5.52188 \times 10^4 \text{ cm}^{-1}$ , those deposited at 400 °C produced  $5.50469 \times 10^4 \text{ cm}^{-1}$ , while those deposited at 450 °C produced the lowest of  $5.29065 \times 10^4 \text{ cm}^{-1}$ . The decrease in absorption coefficient with an increase in the deposition temperature is due to an increase in free carrier density<sup>16</sup>.

All the deposited TFs produced high absorption coefficient of the order of  $(\times 10^4) \text{ cm}^{-1}$ . This result is very important because the spectral dependence of the absorption coefficient is one of the important factors which influence the solar conversion efficiency<sup>20</sup>.

The absorption coefficient increased with increase in the concentration of the dopant at all the three deposition temperatures, with the 8% Fe giving the highest values. This can be attributed to the increase in carrier absorption with doping.

The absorption coefficients obtained in this study are high at short wavelengths, which is good for making front panels of TF solar cells<sup>21</sup>.



**Figure4:** Absorption coefficient against wavelength for (a) undoped cadmium stannate thin films deposited at the three temperatures, (b) cadmium stannate deposited at 450 °C with 0-8% iron, (c) cadmium stannate deposited at 400 °C with 0-8% iron and (d) cadmium stannate deposited at 350 °C with 0-8% iron.

### 3.4 Extinction coefficient

The extinction coefficients ( $k$ ) of the TFs shown in Fig.5 were obtained from the absorption coefficient ( $\alpha$ ) and wavelength ( $\lambda$ ) using the equation<sup>22</sup>:

$$k = \frac{\alpha\lambda}{4\pi} , \quad (2)$$

Extinction coefficient decreased with an increase in the deposition temperature, same as absorption coefficient, since the two are directly related by equation 2. The TFs deposited at 350 °C gave the highest value of extinction coefficient of 0.25834, followed very closely by the TFs deposited at 400 °C with 0.25754. The TFs deposited at 450 °C had the lowest extinction coefficient of 0.24753. Khalifa<sup>23</sup>, who studied titanium IV oxide, also found out that the extinction coefficient of the deposited TFs increased with a decrease in the deposition temperature. The extinction coefficient increased with increase in the concentration of the dopant, which is as a result of an increase in the absorption coefficient.

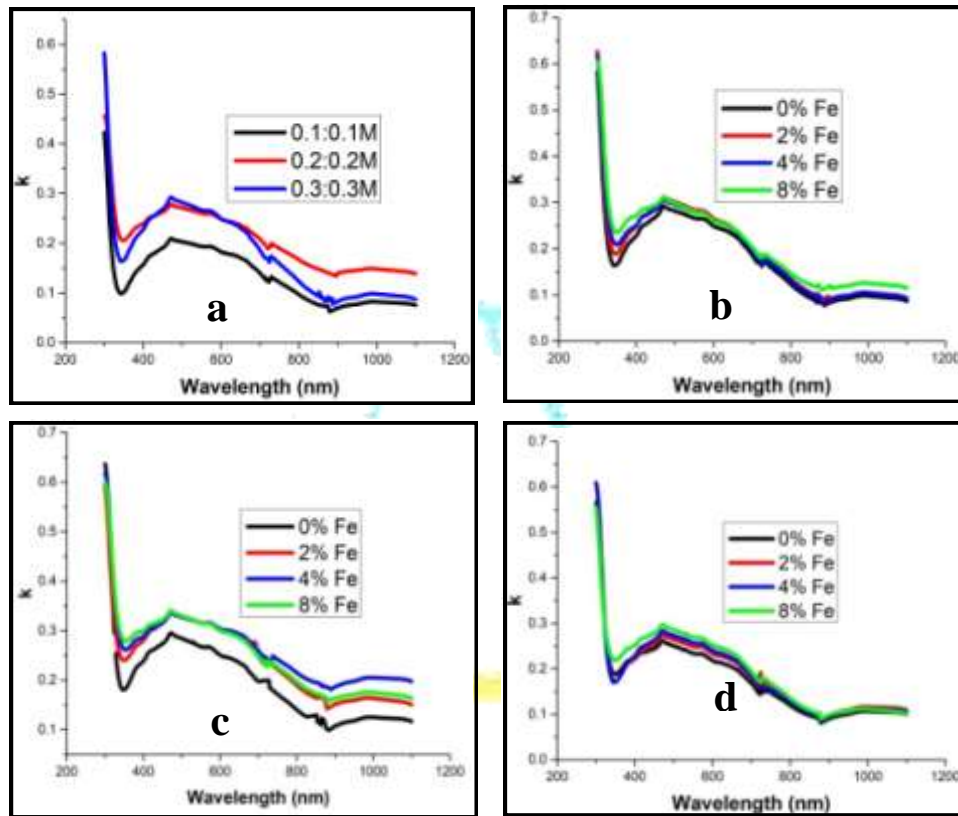


Figure 5: Extinction coefficient against wavelength for(a) undoped cadmium stannate thin films deposited at the three temperatures, (b) cadmium stannate deposited at 450 °C with 0-8% iron, (c) cadmium stannate deposited at 400 °C with 0-8% iron and (d) cadmium stannate deposited at 350 °C with 0-8% iron.

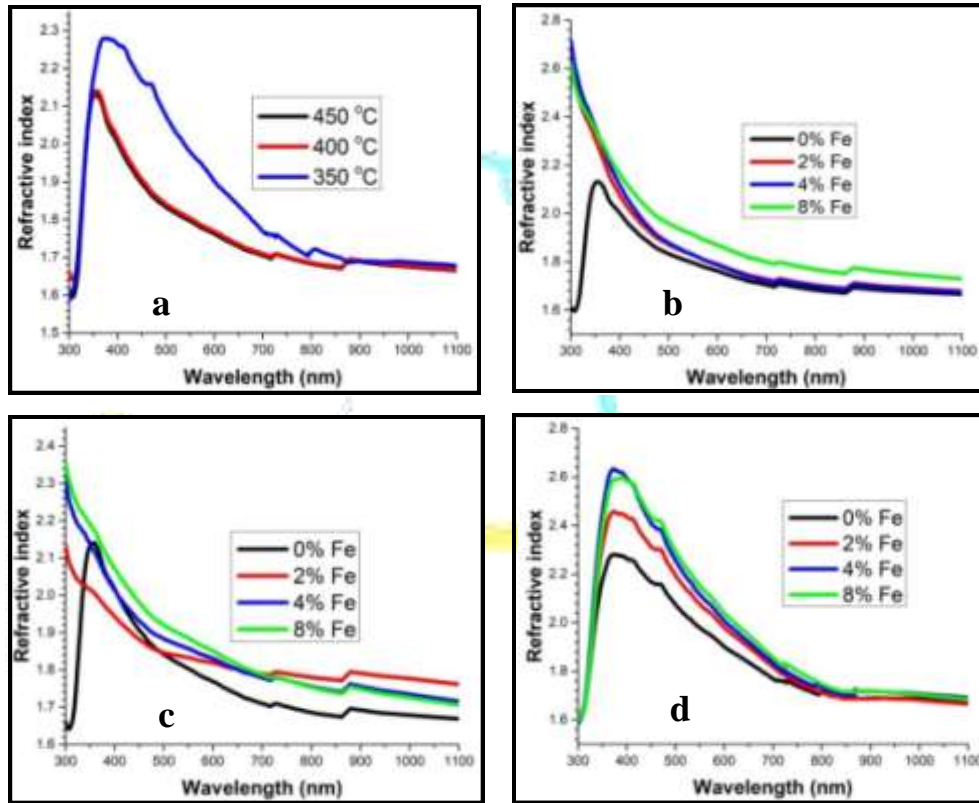
### 3.5 Refractive index

Refractive index of the deposited TFs in Fig.6 was obtained from the formula<sup>24</sup>:

$$n = \left( \frac{1+R}{1-R} \right) + \sqrt{\frac{4R}{(1+R)^2} - k^2} \quad (3)$$

It was observed that the TFs deposited at 450 °C had the lowest values of refractive index, while those deposited at 350 °C had the highest values. This is as was expected, since higher deposition temperatures are supposed to rearrange  $Cd_2SnO_4$  as it lands on the substrate, thus reducing reflectance<sup>3</sup>. Refractive indices of all the deposited TFs decreased exponentially with an increase in wavelength, which means low absorption of light at long wavelength range. Khalifa<sup>23</sup> observed that refractive index of titanium IV oxide TFs decreased with increase in deposition temperature, which they attributed to the decrease in packing density with increase in the deposition temperature. The TFs deposited at 450 °C gave a refractive index of 1.76974, those deposited at 400 °C gave 1.77628 while those at 350 °C gave 1.92305.

The refractive indices still reduced with an increase in deposition temperature upon doping (Figs.6b, c and d). The refractive indices were also observed to decrease exponentially with an increase in the wavelength. The doped TFs recorded higher values of refractive indices compared to the undoped TFs, which might have been brought about by the increase in reflectance due to the free carriers introduced by the dopant.



**Figure 6:** Refractive index against wavelength for (a) undoped cadmium stannate thin films deposited at the three temperatures, (b) cadmium stannate deposited at 450 °C with 0-8% iron, (c) cadmium stannate deposited at 400 °C with 0-8% iron and (d) cadmium stannate deposited at 350 °C with 0-8% iron.

### 3.6 Band gap

The optical band gap is the minimum energy needed to remove an electron from its bound state within an atom to its free state. It is the energy difference between the valence band and the conduction band. A direct band gap is given by<sup>25</sup>:

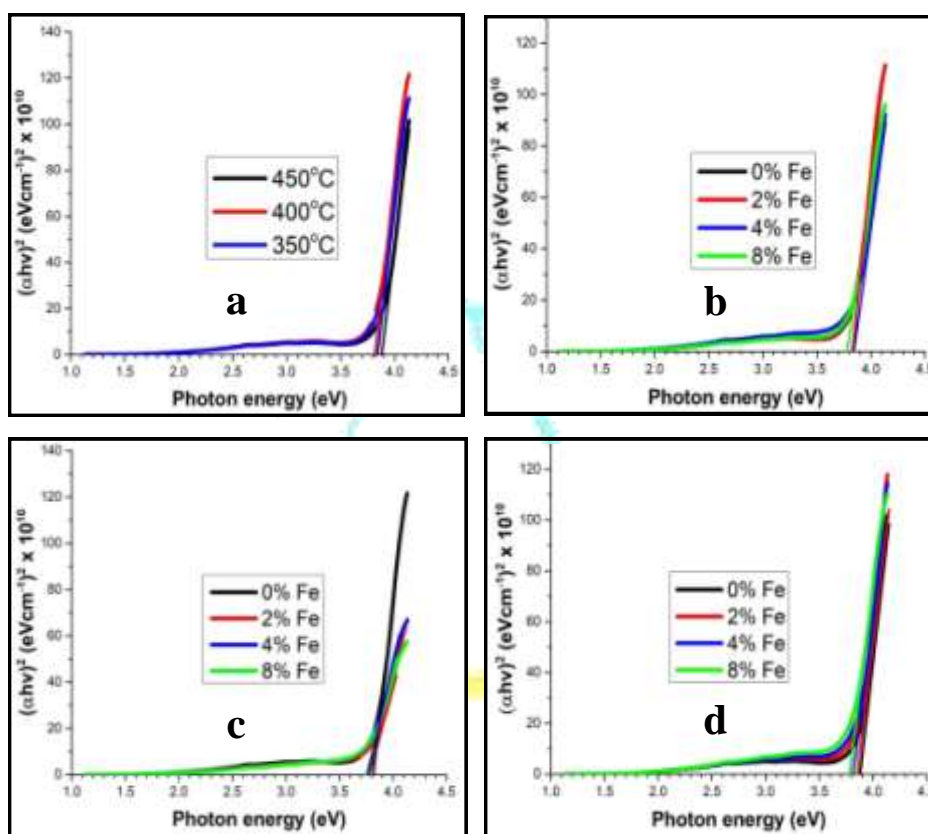
$$\alpha E = \chi(E - E_g)^{1/2}, \text{ from which we get: } (\alpha E)^2 = \beta(E - E_g). \quad (4)$$

where  $\chi$  is a constant and  $\beta = \chi^2$ ,  $h\nu$  is the energy of the incident photon ( $h$  is the Planck's constant and  $\nu$  is the frequency),  $E_g$  is the optical band gap and  $r$  is an index which can take different values according to electronic transition. The relation in equation 4 is known as the *Tauc* relation<sup>26</sup>.

Graphs of  $(\alpha E)^2$  were plotted against photon energy ( $E = h\nu$ ) in eV to obtain Fig.7. On extrapolating the graphs at the straight portions of the curves to intercept the energy axis<sup>27</sup>, the band gap was found to be 3.9 eV for the TFs deposited at 450 °C, while those deposited at 400 °C and 350 °C both gave a band gap value of 3.8 eV (Fig.7a). The highest value of band gap for the TFs deposited at 450 °C can be attributed to the crystallinity of the TFs, as can be observed in Fig.2.

According to Ashok et al<sup>28</sup>, the increase in band gap with an increase in the deposition temperature is a way of achieving band gap tuning in semiconductor materials and hence, the development of new thin films for efficient photovoltaic application. As the substrate temperature increases, the sharp absorption edge is formed because the grain size increases as TFs become more crystalline. It was observed that the potential energy was less at lower absorption.





**Figure7:** A graph of  $(\alpha E)^2$  against photon energy ( $E = h\nu$ ) in eV for (a) undoped cadmium stannate thin films deposited at the three temperatures, (b) cadmium stannate deposited at 450 °C with 0-8% iron, (c) cadmium stannate deposited at 400 °C with 0-8% iron and (d) cadmium stannate deposited at 350 °C with 0-8% iron.

The doped TFs registered lower band gaps than the undoped TFs(Figs.7b, c and d), with the value of band gap decreasing with increase in the concentration of the dopant. This is because the absorption edge shifts to higher wavelengths with an increase in the concentration of Fe in the starting solution<sup>18</sup>.The values of the band gap obtained in this study agree well with those found in the literature. Dawar and Joshi<sup>11</sup> obtained the band gap values of 3.9-4.6 eV.

#### IV. Conclusion

The XRD studies showed that the TFs were orthorhombic with a growth orientation along the (2 0 0) plane. The XPS studies showed the peaks for Cd, Sn, O, Fe and a weak peak of C. The TFs deposited at 450 °C recorded the highest transmittance of 78.45% at the upper end of the visible spectrum, while those deposited at 350 °C recorded the least at 74.77%. The TFs deposited at 400 °C recorded a transmittance of 78.04%. Doping the TFs reduced the transmittance at all the three temperatures, with the value of transmittance decreasing with increase in the concentration of the dopant. Absorption coefficient increases with an increase in the deposition temperature. The TFs deposited at 350 °C recorded the lowest value of  $5.291 \times 10^4 \text{ cm}^{-1}$ , while those deposited at 450 °C recorded a value of  $5.505 \text{ cm}^{-1}$  at the upper end of the visible spectrum. Doping improved the absorption coefficient. Extinction coefficient increased with an increase in the deposition temperature. The TFs deposited at 450 °C recorded the highest value of extinction coefficient of 0.258, while those deposited at 350 °C recorded the least value of 0.248. Doping increased the extinction coefficient. Refractive index decreased with an increase in the deposition temperature. Those deposited at 450 °C gave the lowest value of 1.770, while those deposited at 350 °C gave the highest value of 1.923. Doping increased the refractive indices at all the three deposition temperatures. The band gap decreased with increase in the deposition temperature, with a value of 3.9 eV for those deposited at 450 °C, while those deposited at 400 °C and 350 °C both gave a value of 3.85 eV. Doping reduced the band gap at all the three deposition temperatures, with the band gap value reducing with the increase in the concentration of the dopants. The results obtained in this study provide a good alternative

antireflective material for use in: front panel of TF solar cells, smartphone touch screens and television screens, since the TFs have high transmittance of ~ 80%, a high absorption coefficient of  $\times 10^4 \text{ cm}^{-1}$  and a large bandgap of up to 3.9 eV.

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#### **Author contribution statement**

Nicholas O. Ongwen wrote the main manuscript text

Andrew O. Oduor read the paper and made corrections

Elijah O. Ayieta supervised Nicholas O. Ongwen when he was performing the experiment.

All the authors reviewed the manuscript

#### **Additional information**

The authors declare no competing financial interests

The authors declare no competing non-financial interests

#### **Data availability statement**

Some of the data generated or analyzed during this study are included in this published work and its supplementary information files

Some of the datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

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