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<sup>6</sup>Sami Salman Chiad Physical Characterization of sprayed nanostructured Fe2O3 thin films with Tindoped Prepared by Spray Pyrolysis Method



*Abstract:* - The Spray Pyrolysis Method (SPM) was utilized to make thin films of nanostructured Fe2O3 and nanostructured Tin-doped Fe2O3 with a volumetric concentration of 0.01%. (2 and 4. percent). The strongest peak, as determined by X-ray diffraction, corresponds to the (200). The average particle size values in the AFM imaging for the deposited films decreased from 58.46 nm to 31.13 nm, revealing a smooth surface morphology. The average roughness was also observed to drop from 9.29 nm to 4.68 nm. Roughness ratings range from 16.70 nm to 3.66 nm. The strain decreases from 26.64 to 23.68. We determined optical characteristics such as transmittance and optical constants using a UV-Visible spectrophotometer. For Tin Undoped Fe2O3 and 3 percent Sn doping, the optical transmittance is outstanding, with 80 and 75 percent in the visible zone. It was also discovered that as the concentration of Tin dopant was raised, the absorption coefficient increased. The Fe2O3 bandgap was reduced from 2.71 eV for Fe2O3 to 2.54 eV for Fe2O3: 4 % Sn film.

Keywords: Fe2O3, Sn, thin films, XRD, AFM, Optical Properties, bandgap.

# I. INTRODUCTION

The transition metal  $Fe_2O_3$  has a band gap of 2.2 eV, making it crucial. Due to its beneficial inherent physical and chemical characteristics, including cheap cost, stability under natural conditions, and kindness to the environment are all attractive qualities. [1], it has attracted a lot of interest. Photoelectrodes, gas sensing, catalysts, and medical sectors all use (Fe<sub>2</sub>O<sub>3</sub>). Iron oxide is hence materials that have the most promise for various optical applications and technologies, such as telecommunication, electrochromic applications, and magnetic devices. [3]. Microelectronic devices also use thin layers of Fe<sub>2</sub>O<sub>3</sub> as a dielectric material [4-6]. Hematite, a stable form of iron oxide, can be utilized in photoelectrodes, photovoltaic applications and devices, solar energy conversion, magnetic and nonlinear optical devices, sensors, and other objects [7-9]. Researchers prepare hematite nanostructures using a variety of methods to study the impact of these methods on sensing [10-17]. Chemical deposition [18], sol-gel [19, 20], and PLD [21] are only a few of the Fe<sub>2</sub>O<sub>3</sub> depositing methods that have been studied. CVD [22], thermal evaporation method [23], DC reactive magnetron sputtering [24], and SPM [25-29] are some of the techniques

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used. Because of its simplicity, ease, and low cost, this work seeks to investigate various physical properties of nanostructured undoped  $Fe_2O_3$  and  $Fe_2O_3$ : Sn films deposited using the Spray Pyrolysis Method.

# II. EXPERIMENTAL

The Spray Pyrolysis Method was used to make undoped  $Fe_2O_3$  and  $Fe_2O_3$ : Sn thin films. The initial stock solution was made using Fe chloride in deionized water at a concentration of 0.1 M. SnCl<sub>2</sub> was added to a stock solution of H<sub>2</sub>O (0.1) M, and a few drops of acetic acid were added to make a transparent solution that was agitated for 14 minutes. Tin had a volumetric ratio of (2, 4) percent, and the substrate temperature was 400 degrees Celsius. The layers were placed onto glass substrates that had previously been chemically and ultrasonically cleaned. To optimise the deposition, the following parameters were used: 0.2 ml/spray spray rate, 28 cm base to injector distance, 10 sec of spraying duration per cycle, 2 min between sprays, and carrier gas (filtered air) at a pressure of  $10^5$ Nm<sup>-2</sup>. The thickness was determined to be roughly 310 nm using the gravimetric method. An X-ray diffractometer (Shimadzu, model:) was used to examine film structure that were deposited. X–ray diffractometer (XRD) with monochromatic CuK radiation was applied to specify the composition of the structure. The surfaces of the films were studied using AFM (AA 3000 Scanning Probe Microscope). The optical properties of Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>: Sn films were carried out with a double beam spectrophotometer UV-Vis-NIR Shimadzu corporation.

### **III. RESULTS AND DISCUSSIONS**

XRD patterns of  $Fe_2O_3$  and  $Fe_2O_3$ : Sn thin films produced by SPM are shown in Fig. 1. All of the patterns had diffraction peaks around (228.41o, 33.10, 56.32, and 64.27), which correspond to the (111), (200), (211), and (321) favored directions. These findings match the card number 42-1340 (JCPDS) [30,31].

Using Scherer formula given in Eq. 1, the grain size (D) of Fe<sub>2</sub>O<sub>3</sub> thin films was estimated [32-34]:

$$D = \frac{0.9\lambda}{\beta \cos\theta} \tag{1}$$

Where  $\lambda$  is the x-ray wavelength,  $\theta$  is Bragg's angle, and k=0.9. *D* for Undoped Fe<sub>2</sub>O<sub>3</sub> particle is (13.01-14.63) nm with Fe<sub>2</sub>O<sub>3</sub>: 4% Sn [35-36]

The dislocation density ( $\delta$ ) is gained by [37-39]:

$$\delta = \frac{1}{D^2}$$
 (2)

Table 1. It is seen that  $\delta$  suffer a diminution from 58.89 to 46.72 [40, 41].

The strain ( $\epsilon$ ) was offered via Eq. 3 [42-44]:

$$\varepsilon = \frac{\beta cos\theta}{4} \tag{3}$$

Table 1. It depicted that  $\varepsilon$  reduces from 26.64 to 23.68 [45, 46]. Structural parameters S<sub>para</sub> are offered in Fig. 2.



Fig.1. XRD styles of grown films.

The surface abrasiveness and root-mean-square of the planned films are examined using AFM images. Crystallites with regular sizes and dense packing are visible in Fig. 3's AFM scans. The average particle size  $P_{av}$  was of (87.5), (49.7), and (41.6) nm for the required films. According to AFM, undoped NiO and NiO: Sb films have surface roughness (Ra) and root mean square roughness (Rms) in the (10.71-5.26) nm range (8.76-3.32). The decrease in  $R_a$  is due to the more extensive grain production [35-37]. Figure 3 displays  $R_a$  and Rms. Table 2 shows the  $R_a$  and Rms with Antimony content.

Sample	2 q (°)	(hkl) Plane	β (°)	E <sub>g</sub> (eV)	Grain size (nm)	$\begin{array}{l} \text{dislocation} \\ \text{density} \left( \delta \right) \\ \left( \times \ 10^{14} \right) \\ (\text{lines/m}^2) \end{array}$	strain (ε) (× 10 <sup>-4</sup> )
Fe <sub>2</sub> O <sub>3</sub>	28.41	200	0.63	2.71	13.01	58.89	26.64
Fe <sub>2</sub> O <sub>3</sub> : 2% Sn	28.38	200	0.6	2.62	13.66	53.59	25.37
Fe <sub>2</sub> O <sub>3</sub> : 4% Sn	28.35	200	0.56	2.54	14.63	46.72	2.68

TABLE 1. D,  $E_g$  and Spara of grown films.



Fig.2. S<sub>para</sub> of the grown films.

TABLE 2.	PAFM o	f grown films
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Samplag	Pav	Ra	Rms	
Samples	nm	( <b>nm</b> )	( <b>nm</b> )	
Fe <sub>2</sub> O <sub>3</sub>	58.46	9.29	6.78	
Fe <sub>2</sub> O <sub>3</sub> : 2% Sn	36.23	7.94	4.85	
Fe <sub>2</sub> O <sub>3</sub> : 4% Sn	31.13	4.68	3.80	



Fig. 3.AFM images, granularly distributed and diversity of PAFM.

The optical transmission (T) spectra of  $Fe_2O_3$  and Tin doped  $Fe_2O_3$  are offered in Figure 3. For wavelengths of 750 nm, the undoped  $Fe_2O_3$  and  $Fe_2O_3$ : Sn films show strong transmission (average > 70%), which is one of the requirements for optoelectronic devices, particularly for solar cell window layers [49, 50].

The transmission and reflectance spectra, as well as the film thickness(t), were used to calculate the absorption coefficient ( $\alpha$ ). [51-53]:

$$\alpha = (2.303 \times \text{A})/\text{t} \quad (4)$$

It was observed that  $\alpha$  increased as the content of Tin increased. A considerable increase in the transitions from the bonding molecular orbit to the nonbonding molecular orbit may be the cause of the significant rise in the absorption coefficient at higher energy [54, 55].



Fig. 4 Transmittance of the deposited Undoped Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>: Sn thin films versus wavelength.



Fig. 5  $\alpha$  Vs hv of grown films.

The optical band gap  $(E_g)$  is measured using Tauc 's equations [56-58]:

$$(\alpha h\nu) = A \left( h\nu - E_g \right)^{\frac{1}{2}}$$
 (5)

Plots were obtained using A as the constant and  $(hv)^2$  vs. photon energy (hv) is demonstrated in Fig. 6, the  $E_g$  values for Undoped Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>: Sn were quite near to each other.  $E_g$  was reduced from 2.71 eV for Fe<sub>2</sub>O<sub>3</sub> to 2.54 eV for Fe<sub>2</sub>O<sub>3</sub>: 4 % Sn film [59, 60].



Fig. 6 Direct bandgap of grown films.

The extinction coefficient (k) using the following relation [61-63]:

$$k = \frac{\alpha \lambda}{4\pi} - \dots (7)$$

Where  $\lambda$  is the wavelength, Figure 7 shows the fluctuation in K via wavelength when the concentration of tin dopant is increased [64, 66].

The refractive index (n) is obtained from the reflectance (R) data via the relation [67-69]:

$$n = \left(\frac{1+R}{1-R}\right) + \sqrt{\frac{4R}{(1-R)^2} - k^2} \quad \dots \quad (8)$$

As seen in Fig. (8), n is affected by the concentration of the Tin dopant, with an increase in Tin dopant causing a drop in its value, which may be linked to an increase in film compactness [70, 71].



Fig. 7 k of the intended films.



Fig. 8 n of the intended films.

## **IV. CONCLUSION**

Spray Pyrolysis Method was used to grow undoped  $Fe_2O_3$  films doped with Tin. The shape of  $Fe_2O_3$  thin films changed dramatically as the Tin dopant was increased from 0% to 4%. According to X-ray diffraction, the optimal orientation (200) for Undoped  $Fe_2O_3$  films at 4 percent Sn corresponds to the peak of greatest intensity. With  $Fe_2O_3$ : 4 % Sn, the grain size for undoped  $Fe_2O_3$  particles is around (13.01- 14.63) nm, but the strain increased from 26.64 to 23.68. With Undoped  $Fe_2O_3$  and  $Fe_2O_3$ : 4 % Sn nm,  $P_{av}$  was in the area of 58.46 nm to 31.13 nm, respectively. The transmittance spectra are set by UV-Visible spectrophotometer. With increasing Tin doping, the optical energy gap dropped to 2.54 eV for the  $Fe_2O_3$ : 4 % Sn film, but the absorption coefficient increased. The optical constants were also calculated.

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