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Physical Properties of Nanocrystalline Tin Oxide Thin Film by Chemical Spray Pyrolysis Method

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Abstract: Tin oxide (SnO) thin films have been deposited using chemical spray pyrolysis on non- conducting glass substrates at temperature 250°C with solution concentration of 0.2M. The micro structural, surface morphological and compositional properties of deposited films were studied using X-ray diffraction (XRD), scanning electron microscope (SEM) and Energy dispersive spectroscopic analysis (EDX) techniques. The XRD studies reveal that films are crystalline and structure of tetragonal phase. Micro structural parameters such as crystallite size, micro strain, and dislocation density are calculated and found. SEM studies reveal that the prepared film exhibited the irregular grains over the surface. Energy dispersive spectroscopic analysis (EDX) confirmed the presence of Sn and O elements.
KEYWORDS: Spray pyrolysis, SnO Thin films, SEM, XRD, Structural studies.

I. INTRODUCTION

In recent years, the nano-structured metal oxides have been most widely studied materials owing to their many applications. Tin oxide (SnO) is the most important transparent conducting oxide (TCO) material among various TCO materials such as ZnO, CdO, In_2O_3 etc., due to their high transmittance, high reflectance, chemically inert, mechanically hard, not affected by atmospheric conditions. The tin oxide film were used in various applications such as window materials in solar cell [1], gas sensors [2], transistor [3], optoelectronic devices [4], lithium batteries [5] , flat panel display etc., Its splendid physical and chemical properties makes it one of the top-quality material used for detection of distinct types of gases. Different types of techniques have been used to prepare SnO thin film such as electron beam evaporation [6], sputtering [7], thermal evaporation [8], spray pyrolysis [9] and nebulizer spray pyrolysis [10]. The binary and ternary oxide thin film such as MgO [11], Cd doped SnO2 [12], and Tin oxide have been deposited using perfume spray pyrolysis technique. Among various techniques perfume spray pyrolysis is a simplified spray pyrolysis technique. This is cost effective, low volume of spray solution and time saving technique by which tiny droplets of particles can be deposited. In this study, the structural, morphological and compositional studies have been investigated and analyzed.

II. EXPERIMENTAL TECHNIQUE

Spray pyrolysis technique consists of a Perfume spray unit, temperature controller and a substrate heater unit as shown in Figure.1. Tin oxide thin film was deposited on amorphous glass substrate by spraying an aqueous solution containing 0.2M tin tetra chloride (Sigma-Aldridge) with spray pyrolysis technique. Well cleaned glass substrate of dimension 7.5 x 2.5 x0.25 cm^3 was kept on the preheated hot plate. The optimized substrate temperature was taken as 250 °C. The distance between nozzle and substrate was kept at 3cm. It does not require high quality target and vacuum. The thickness of the film and rate of deposition can be easily controlled. The thickness of the coated films was measured using weight gain method. X-ray diffraction analyses were obtained using the model X"pert PRO (Analytical) X-ray powder diffractometer with Ni filtered CuKα (1.54056 Á) radiation. The surface morphology and homogeneity of the deposited films were studied by SEM model JSM 35 CF JEOL.

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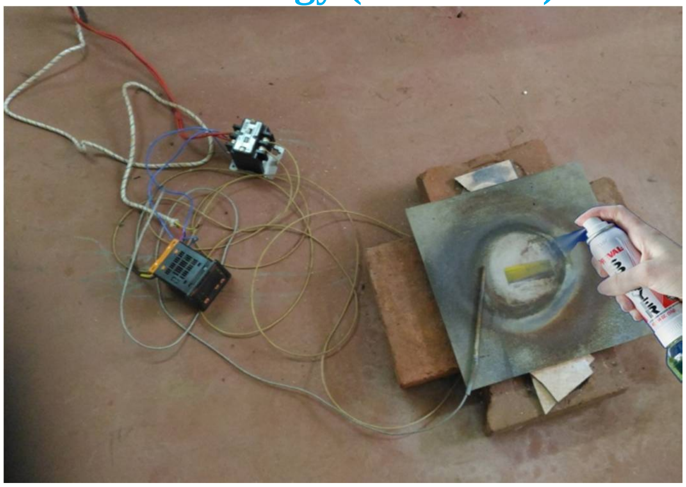

Figure: 1. Simple Spray Pyrolysis technique

Table .1: Optimized parameters applied to fabricate the TCO films.

Precursor molar concentration	0.2M
Solvent	Distilled water
Solvent volume	50ml
Substrate temperature	250°c
Substrate to nozzle distance	5cm
Spray angle	45°c
Spray interval	2 sec

III. CHARACTERIZATION STUDIES

A. Thickness Measurement

Thickness is the most important film parameter, which controls the film properties. Hence, precise knowledge of the film thickness is necessary for the intensive study of the properties of thin films. Thickness of the prepared film was estimated using weight method. The well-cleaned glass plate is weighted in a high precision microbalance. After the deposition the coated glass plate is weighted. The difference in weight gives the mass of the deposited film "m". The area over which the film deposited is measured as "A". The standard value of density "ρ" has been taken and the thickness is calculated using the expression.

$$t = m/ (A\rho) \text{ (microns)}$$

Where,

t – is the thickness of the film.

m – is the weight difference (Mass of the film)

A – is the area of the sample.

ρ – is the density of the film.

The calculated thickness of the prepared tin oxide film was 636 nm.

B. XRD Studies

Figure 2 shows the XRD pattern of the 0.2 M concentration of tin oxide thin films deposited using spray pyrolysis method. All the diffraction peaks of XRD pattern could be indexed to tetragonal phase crystal structure of SnO, which is in good agreement with the

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standard data for SnO (JCPDS data file no. 77-2296). The grown SnO have exhibited strong orientation along (020) plane and also other peaks like (002), (110), (111), (112), (115) and (225) are appears.

The structural parameters of SnO thin films are given in Table 2. The crystallite size is calculated using Debye - Scherrer's formula [13]

$$D = k\lambda / \beta \cos\theta \quad \text{------------}\rightarrow \ (1)$$

Where k is the shaping factor which takes value from 0.89 to 0.94, 'λ' is the wavelength of the Cu-k_α line, 'β' is the full width at half maxima (FWHM) in radians and 'θ' is the Bragg's angle.

Dislocation density (δ) for (020) plane is evaluated using the relation [13 -17]

$$\delta = 1/D^2 \quad \text{------------}\rightarrow \ (2)$$

The strain (ε) is calculated from the following relation

$$\varepsilon = (\beta \cos\theta) / 4 \quad \text{------------}\rightarrow \ (3)$$

The Number of Crystallites (Nc) is calculated from the following relation

$$Nc = t / D^3 \quad \text{------------}\rightarrow \ (4)$$

Lattice Distortion (LD) is calculated from the following relation

$$LD = \beta / (4\tan\theta) \quad \text{------------}\rightarrow \ (5)$$

Table 2. Microstructural Parameters of SnO Thin Films

MATERIAL	SnO
2θ	35.4286
Full width half maximum	0.1968
Crystallite size D (10^{-9}m)	42.194
Dislocation density (δ) 10^{14} lines/m	5.616
Micro strain ε x10^{-3} lines^{-2}m^{-4}	0.65
Number of Crystallites (Nc) x 10^{16}	8.38
Lattice Distortion (LD) x10^{3}	159.15

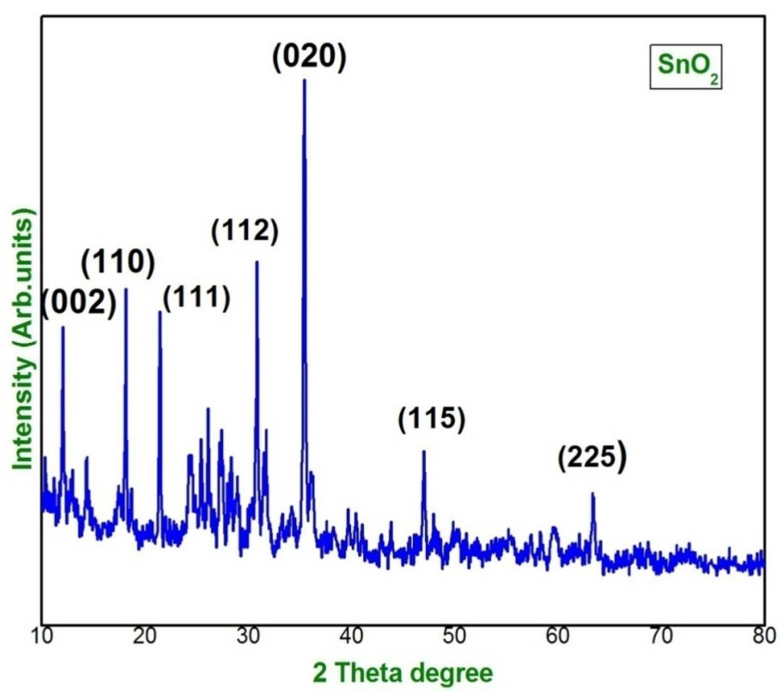

Figure 2. Typical X-Ray Diffraction patterns of SnO thin film

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C. Morphological studies

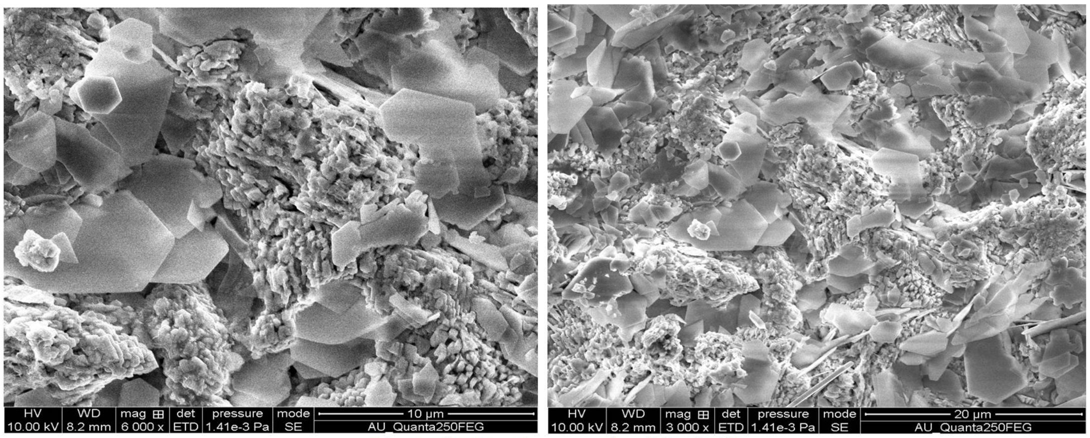

Fig 3: SEM micrograph of SnO films

The surface morphology of the SnO thin films was investigated by using scanning electron microscope. Figure 3 shows the various magnification like 10 and 20 µm SEM micrographs of SnO thin films prepared with 0.2 M of solution concentration. SEM micrograph reveals the presence of uniformly sized spherical nanoclusters distributed over the surface. The surface of the film is found to be heterogeneous, having porous surface with small pinholes. Those were formed by bigger particles agglomerated randomly and found that there is no uniform shape. Such image is compact and relatively not dense. In 10 µm magnification the observed micrograph the smaller particles are tightly connected. This kind of micro structure is desirable for sensor applications

D. Elemental Analysis

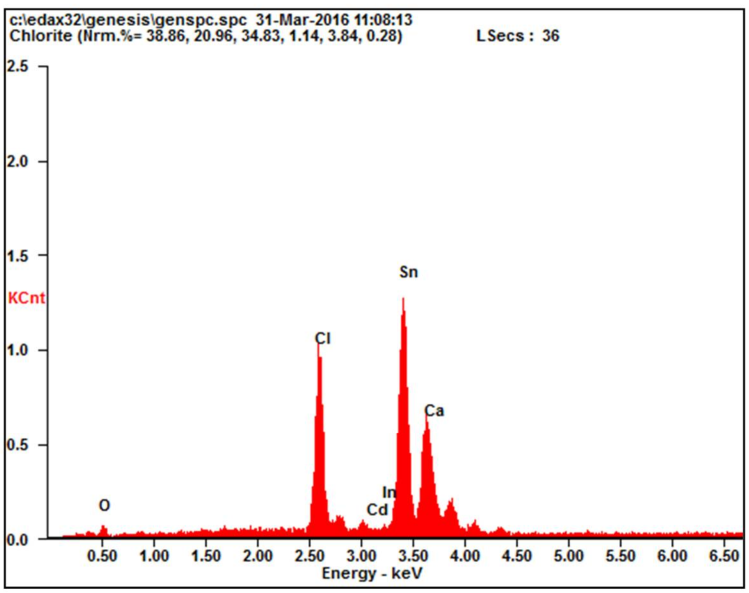

Figure 4: EDX Spectrum of SnO

Elemental analysis of prepared tin oxide thin films was carried out by EDX. Fig. 4 represents the EDX spectrum of SnO thin film. EDX analysis confirms the presence of Sn and O elements in the prepared SnO film.

IV. CONCLUSION

Nano crystalline tin oxide (SnO) thin film was prepared on micro glass substrate at 250 ^{o}C by simple spray pyrolysis technique. The x-ray diffraction pattern revealed that the as deposited film had tetragonal crystal structure with preferable orientation along (020) plane. The surface morphology studies SEM micrograph reveals the presence of uniformly sized spherical nanoclusters distributed over the surface. The surface of the film is found to be heterogeneous, having porous surface with small pinholes. Those were formed by bigger particles agglomerated randomly and found that there is no uniform shape. EDX analysis confirms the presence of

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Sn and O elements in the prepared SnO film. The investigated results of the SnO thin film deposited by spray pyrolysis technique ensure the stability of the film and their employability in gas sensor applications.

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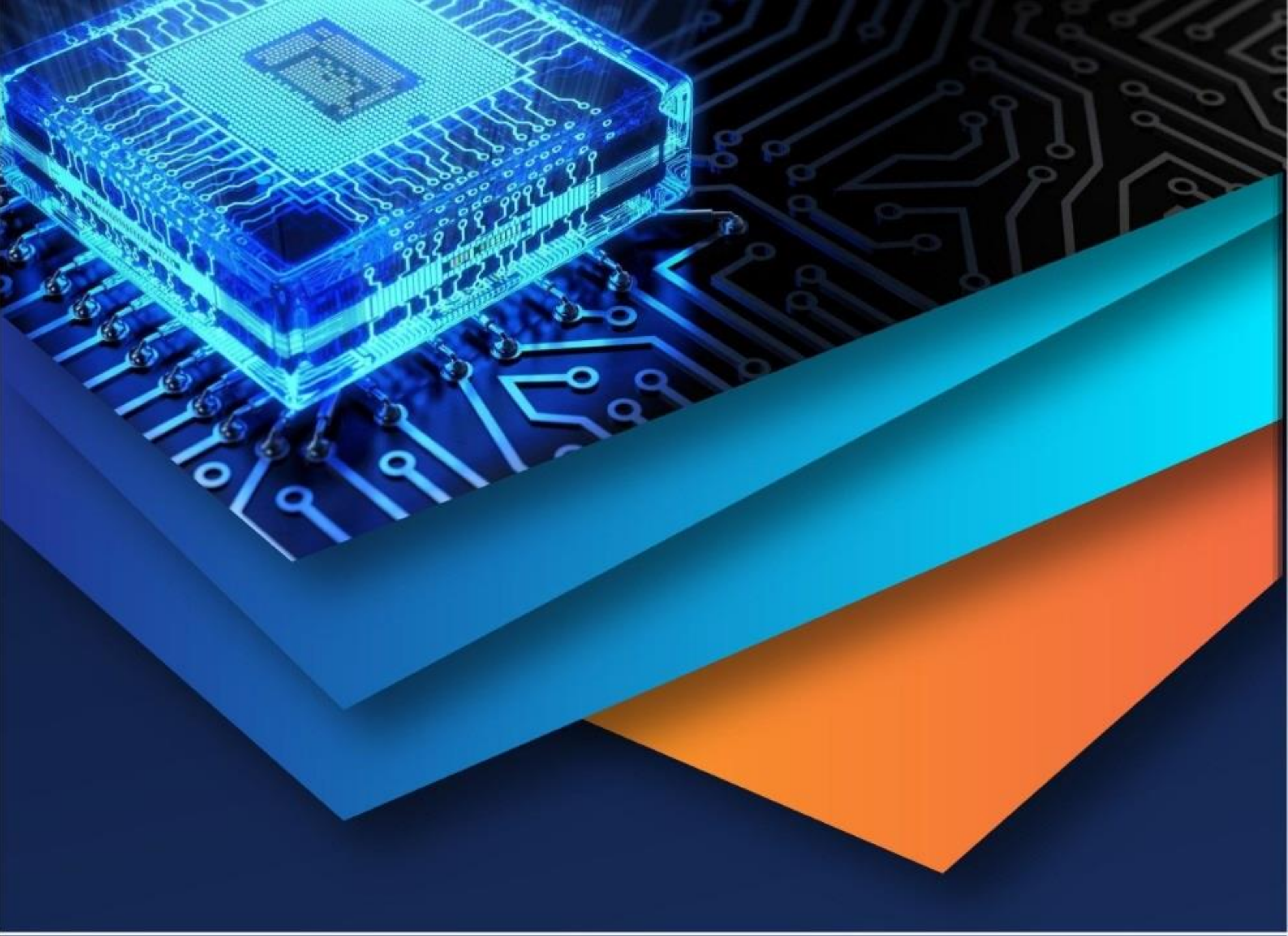

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