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Electrical, structural and optical properties of SnO_2:F thin films: Effect of the substrate temperature

A.A. Yadav[a,*], E.U. Masumdar[a], A.V. Moholkar[b], M. Neumann-Spallart[c], K.Y. Rajpure[d], C.H. Bhosale[d]

[a] *Thin Film Physics Laboratory, Department of Physics, Electronics and Photonics, Rajarshi Shahu Mahavidyalaya, Latur, 413512 M.S., India*
[b] *Department of Materials Science and Engineering, Chonnam National University, 300 Yongbong-Dong, Puk-Gu, Gwangju 500-757, South Korea*
[c] *Groupe d'Étude de la Matière Condensée, C.N.R.S., 1 Place Aristide Briand, 92195 Meudon CEDEX, France*
[d] *Electrochemical Materials Laboratory, Department of Physics, Shivaji University, Kolhapur 416004, India*

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ABSTRACT

SnO_2:F thin films have been deposited from stannic chloride solution by simple and cost effective chemical spray pyrolysis technique onto glass substrates at different substrate temperatures. The as-deposited films were characterized by X-ray diffraction (XRD), SEM, optical and electrical characterization techniques. XRD analysis showed that, at lower substrate temperature; amorphous films have been obtained, while at higher temperatures ($T > 450\,^\circ C$); crystalline SnO_2:F films with preferential growth along (2 0 0) plane have been observed. The average transmission in the visible region has been found to vary from 60% to 87% depending upon the substrate temperature. In the visible region of the spectrum, the transmission is very high (high enough to observe interference). For films prepared at 475 °C, relatively higher transmittance of about 87% at 850 nm has been observed. A thickness was found to vary from 440 nm to 740 nm with substrate temperature. The direct optical band gap energy for the SnO_2:F thin film is found to be 4.15 eV. The films deposited at 475 °C substrate temperature were found to have relatively lower resistivity of $3.91 \times 10^{-4}\,\Omega\,cm$. Hall Effect studies reveal that the films exhibit n-type conductivity.

1. Introduction

Transparent conducting oxides (TCOs) are wide band gap semiconductors ($E_g \geq 3.1$ eV) whose properties strongly depend on the stoichiometric deviation, e.g. oxygen deficiency (SnO_{2-x}) and on the nature and quantity of impurities trapped into the host lattice, such as in In_2O_3:Sn and SnO_2:F materials [1]. Among the TCOs, SnO_2:F has many applications due to the unique combination of high transparency and low resistivity. The low resistivity value of SnO_2:F film is due to a high carrier concentration caused by both oxygen vacancies and substitutional fluorine dopants. The high transparency in the visible and near-IR region is also caused by wide band gap of a TCO. SnO_2:F is the most widely used transparent-conducting oxide material. The transparent-conducting thin films of this oxide are used as electrodes in a variety of optoelectronic devices, as heat-mirror coatings in 'smart windows' for buildings, in automobile and airplane windshields, in incandescent light bulbs, in flat-plate and concentrating solar collectors. Tin oxide and zinc oxide, in thin film configuration are technologically important materials because of their applications in photovoltaic cells [2], gas sensors [3], electrochromic devices, low-e windows [4], and photocatalysis [5], etc. In order to combine and to stabilize the properties of TCO films, an alternative strategy is proposed in the literature to fabricate TCO materials based on multicomponent oxides, which consist a combination of binary or ternary compounds such as: In_2O_3–ZrO_2, TiO_2:ZrO_2 [6], ZnO–SnO_2 [7], In_2O_3–Sc_2O_3 [8], and ZnO–V_2O_5 [9].

The TCO thin films can be prepared by a variety of methods such as chemical vapor deposition (CVD), sputtering, sol–gel process and spray pyrolysis. The sputtering and evaporation processes often prove to be too expensive for some industrial applications. The chemical spraying offers a competitive alternative for the massive production of these coatings. The spray pyrolysis method presents some noticeable advantages, such as: a wide possibility of varying the films properties by changing the composition of the starting solution (introduction of dopants and change the film microstructure) and low cost when large-scale production is needed. Previously the effect of quantity of spraying solution and concentration of $SnCl_4$ on the properties of spray deposited SnO_2:F thin films have been reported [10,11]. In continuation to our earlier work, the aim of this work is to study the effect of the substrate temperature on the structural, optical and electrical properties of spray deposited SnO_2:F thin films. Further it is investigated that the part of the film formed near the central area of the substrate yields relatively better optoelectrical properties due to uniformity of the film deposition within this area. We present results of SnO_2:F thin

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* Corresponding author. Tel.: +91 9975213852.
E-mail address: aay_physics@yahoo.co.in (A.A. Yadav).

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films deposited by an optimized spraying procedure which lead us to fix important parameters such as concentration of precursors in the solution, quantity of spraying solution, solution flow rate and nozzle-to-substrate distance. The effect of the substrate temperature on the physical properties of SnO_2:F thin films like thickness, crystal structure and resistivity has been reported and the results obtained are compared and discussed.

2. Experimental

To make 100 cc of 2 M solution of stannic chloride, the required quantity of stannic chloride is dissolved in double distilled water. Followed by calculated amount of ammonium fluoride (NH_4F) salt (to make 20% doping concentration in starting solution) is dissolved in prepared stannic chloride solution. To make the turbid solution clear, 24 cc of 0.01 M oxalic acid is added in it. It has been observed that the optical transmission of SnO_2 films could be increased by the addition of few drops of oxalic acid to the spray solution [11]. This was taken as a stock solution. For each deposition, 10 cc from stock solution was mixed with 10 cc of propane-2-ol to make the final spraying solution of 20 cc. The resulting concentrations of various ingredients in the spraying solution (20 cc) were as under; $SnCl_4{\cdot}5H_2O$: 0.806 M, NH_4F: 1.527 M, and oxalic acid: 0.96 mM. Glass microslides of the size 2.4 cm × 7.4 cm, obtained from Blue Star Polar Industrial Corporation, Mumbai and quartz plates of the size 2.0 cm × 2.0 cm were used as substrates. The substrates were washed with water, then boiled in concentrated chromic acid and kept in distilled water for 48 h; finally substrates were ultrasonically cleaned for 10 min prior to deposition, and the substrates were degreased with trichloroethylene, followed by rinsing with acetone, ethanol and water. Spray pyrolysis involved the spraying of a precursor solution through a pneumatic nozzle onto a substrate located on a temperature controlled heating plate. The apparatus has been described elsewhere in detail [12]. All the films were allowed to cool naturally to a room temperature on hot plate after deposition.

To identify the best suitable position for deposition in the chamber, a systematic study on the film properties (film thickness, refractive index and conductivity) as a function of distance in two-dimensional horizontal plane of the deposition area of the system was carried out. After locating the best suitable position for deposition, further experiments were carried out. The substrate temperature was varied from 450 °C, in the interval of 25–525 °C by keeping all other parameters at constant; especially total quantity of spraying solution at 20 cc.

The structural characterization of deposited thin films was carried out by analyzing the X-ray diffraction patterns obtained using a Philips X-ray diffractometer model PW-1710 ($\lambda = 1.54056$ Å for Cu-Kα radiation). For microstructure investigation with SEM (Cambridge Stereoscan 250-MK-3 unit), the film was coated with gold palladium by a Polaron sputter coating unit E-2500. The coating thickness was 15 nm. The transmission spectrum has been studied at room temperature and at normal incidence by Systronics makes UV–vis spectrophotometer (model 119) in the wavelength range 350–850 nm. Hall Effect setup supplied by Scientific Equipments, Roorkee, India was used for the measurements of electrical parameters like resistivity (ρ), sheet resistance (R_s), carrier concentration (n) and mobility (μ) at room temperature. Van der Pauw technique [13] was used for this purpose. Specially designed Hall probe on printed circuit board (PCB) was used to fix the sample of the size 10 mm × 10 mm. Silver paste was employed to ensure good electrical contacts.

3. Results and discussion

Organic solvents like methanol and ethanol are typically used in spray pyrolysis of TCO. There are few reports on the synthesis of TCO films wherein an aqueous/organic solvent mixture was used. As the complex formation mechanism in chemical reaction and pyrolytic decomposition process of the spraying solution at the substrate surface are different for different solvents used, the growth kinetics involved in the resulting film is different. It was observed that the films prepared using propane-2-ol exhibit the better opto-electronic properties than the films prepared using either methanol or ethanol as solvents. In the present investigation, propane-2-ol was used as solvent for the deposition of films. When $SnCl_4$ is dissolved in the solvent propane-2-ol, the hydrated SnO_2 molecule is formed. An exothermic reaction takes place with HCl is given off in which the part of the $SnCl_4$ in propane-2-ol ionizes into Sn^{4+} and Cl^-, and the rest reacts with alcohol to form complex molecules such as $SnCl_4{\cdot}2C_2H_7OH$ and $SnCl_4{\cdot}5C_2H_7OH$ [14]. These two complexes are neutral, and thus can react to form SnO_2 on the substrate.

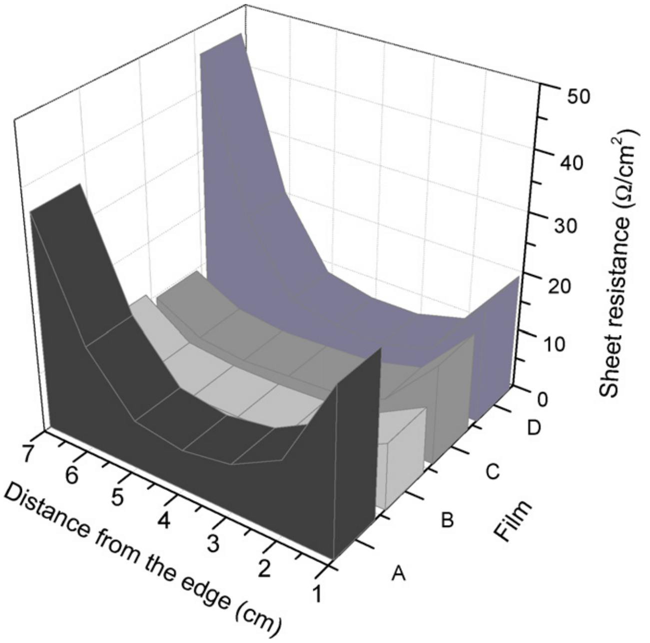

Fig. 1. Variations of sheet resistance as a function of distance on the hot plate.

The conductivity of undoped SnO_2 is attributed to a combination of chlorine ion and oxygen vacancies, produced due to the incomplete decomposition of $SnCl_4$ and incomplete oxidation of the films. These defects are considered to be electron donors. Since SnO_2 films have been prepared at various deposition temperatures ranging from 450 °C to 525 °C by spray pyrolysis of $SnCl_4$, this method is known to produce different numbers of oxygen vacancies. As the starting solution is $SnCl_4$, chlorine contamination is also expected.

Thicknesses and refractive indices are computed from transmission data and by using the following formula [15]:

$$T = \frac{t_1{}^2 t_2{}^2}{1 + 2r_1 r_2 \cos 2\delta_1 + r_1{}^2 r_2{}^2} \times \frac{n_2}{n_0} \tag{1}$$

where

$$r_1 = \frac{n_0 - n_1}{n_0 + n_1}, \qquad r_2 = \frac{n_1 - n_2}{n_1 + n_2}, \qquad t_1 = \frac{2n_0}{n_0 + n_1},$$
$$t_2 = \frac{2n_1}{n_1 + n_2} \qquad \text{and} \qquad \delta = \frac{2\pi n_1 d}{\lambda} \tag{2}$$

with $n_0 = 1$ the R.I. of air, n_1 = refractive index of thin film, n_2 = refractive index of the substrate, d = thickness of thin film, T = transmittance and λ = wavelength of incident radiation. By fitting the observed transmittance data with the theoretical data given by Eq. (1), one can search for a pair of thickness and refractive index [15–17].

3.1. Electrical and optical parameters mapping

Physical inspection of the films deposited in all experiments reveals that different coloured rings owing to different thicknesses appear on the film surface due to interference. This suggests that it has non-uniform distribution of film thickness, refractive index and conductivity. Thus it leads us to carry out the systematic study on these properties as a function of distance in two-dimensional horizontal plane of the deposition area of the system.

Fig. 1 shows the variation of sheet resistance as a function of distance on the hot plate. In a typical case, for film C and at 5 cm distance, relatively minimum sheet resistance of 4.532 Ω cm^{-2} and maximum thickness of 740 nm have been observed. It is observed

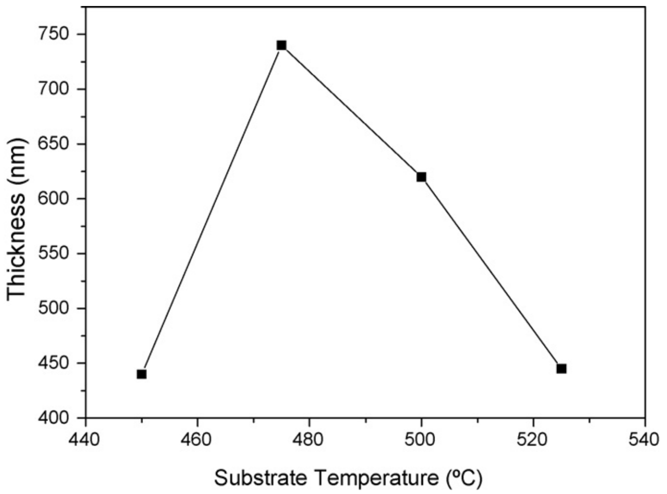

Fig. 2. Variation of the film thickness of SnO_2:F thin films with respect to substrate temperature.

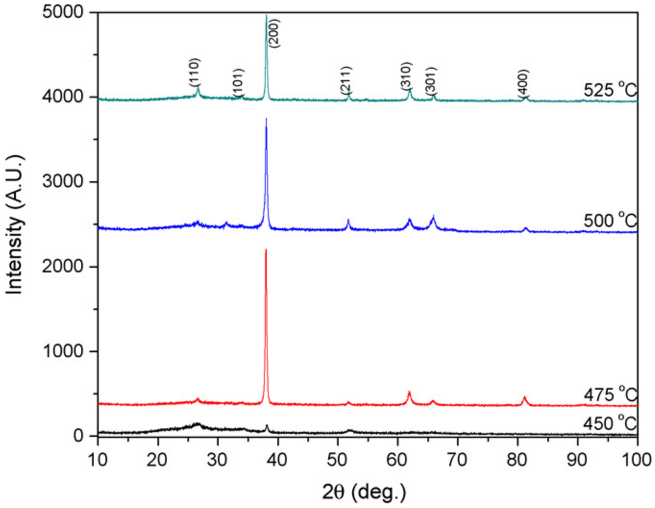

Fig. 3. XRD patterns of spray deposited SnO_2:F thin films at various substrate temperatures.

that resistivity goes on increasing and thickness goes on decreasing from the center of the plate to outward. This may be attributed to the production of different numbers of oxygen vacancies at different locations of the plane due to temperature gradient on the surface of hot plate.

From the mapping studies it is observed that the central part of the hot plate of area $\sim 3\,cm^2$ gives the good quality and uniform films in terms of thickness, refractive index and surface resistivity. The possible reason for such a kind of variation in film properties as a function of distance in two-dimensional horizontal plane is the difference in actual temperatures at various parts of the hot plate. From 3D variation of thickness it appears that film thickness is maximum at central zone of the plate and it decreases as one move towards the edges. The decrease in thickness towards the edges is due to relatively higher temperature in this region compared to central region. Thus it reflects that actual temperatures in various zones of the plate are different than that of measured value, supposing that it is constant over entire surface of the plate. Therefore in each experiment, the part of the film within this area only was chosen for further characterizations. By modifying the configuration and engineering of deposition unit, synthesis of uniform SnO_2:F thin films of large area, seems to be feasible after detailed investigation, which is mainly dependent on the uniform temperature of the plate. The efforts of improving the central area for maximum yield are still under study, by having different designs and arrangements of heating systems.

3.2. *Structural properties*

Fig. 2 shows the film thickness as measured interferometrically versus substrate temperature; the spray rate was kept constant at 4–5 cc/min Relatively higher thickness is found to be 740 nm for film deposited at 475 °C. It is seen that the film thickness increases with increasing substrate temperature from 450 °C to 475 °C, attains terminal value at 475 °C (740 nm), beyond which it decreases. This can be explained as follows: initially, at lower substrate temperatures, e.g. 450 °C, the temperature may not be sufficient to decompose the sprayed droplets of Sn^{4+} and F^- ions from the solution and this therefore, results in a low thickness. At a particular substrate temperature 475 °C, decomposition occurs at the optimum rate resulting in the terminal thickness being attained. A noticeable decrease of the film thickness with increasing the substrate temperature is observed after substrate temperature 475 °C. This decrease may be attributed to re-evaporation of film material after deposition or to thermal convection of the sprayed droplet during the deposition process or both. Another cause which may account for thickness decrease is water loss [18] or removal of interlayer water with consequent formation of the compact film. The decrease in film thickness at higher substrate temperatures may be due to a higher evaporation rate of the initial ingredients of the solution. Similar type of behavior was reported for nickel oxide films prepared by electrochemical technique [19].

The crystal structure of the as-deposited SnO_2:F thin films is determined by X-ray diffraction technique. The XRD pattern was recorded for different films deposited onto glass substrates kept at different substrate temperatures in 2θ range 10–100°. SnO_2:F thin films deposited at 450 °C show an amorphous nature with (1 1 0) and (1 0 1) peaks of very low intensity corresponding to the SnO_2 cassiterite structure according to the joint committee on powder diffraction standards (JCPDS) 41–1445 card [20], as shown in Fig. 3. When substrate temperature increases, the amorphous background diminished and the intensity of the diffraction peaks increased, this behavior is due to the fact that at low substrate temperature the starting materials ($SnCl_4$) and undesired by-products (HCl, CO_2, CO, etc.) are present in the films, suggesting that the substrate temperature is not enough for completing the chemical reaction, e.g. the droplet hit on the hot substrate removing a lot of heat causing a partial decomposition of the starting materials. SnO_2:F thin films prepared at and above 475 °C are polycrystalline materials and present a single phase with a tetragonal rutile structure (cassiterite). The unique feature of all the diffractograms is that they contain the characteristic SnO_2 orientations along the preferred (2 0 0) plane. This preferred orientation remains predominant irrespective of the substrate temperature. The presence of other peaks such as (1 1 0), (1 0 1), (2 1 1), (3 1 0), (3 0 1) and (4 0 0) has also been detected but with substantially lower intensities. Intensity of (2 0 0) peak increases progressively as the substrate temperature changes from 450 °C to 475 °C and then it decreases afterward for further increase in substrate temperature. The increase in (2 0 0) peak intensity from 450 °C to 475 °C temperature may be attributed to the continuous increase in film thickness from 440 nm to 740 nm. Decrease in (2 0 0) peak intensity after 475 °C might be ascribed to the fact that at these substrate temperatures, there is complete thermal decomposition of sprayed droplets before reaching the hot substrates due to unsuitable thermal energy (higher) than required for perfect decomposition and subsequent recrystallization. It has been reported that the preferred orientation of SnO_2 film on glass substrate is affected by source compounds, growth parameters such as solution concentration, solution feed rate, spraying gas pressure, substrate temperature [21] and solvents [22]. Similar XRD patterns have been reported for SnO_2:F [23,24] and high Sb doped transparent oxide films prepared by spray pyrolysis [25]. The enhancement of the crystal structure at temperatures above of 450 °C is due to the growth of the films carried out by the low temperature CVD process, e.g. the solvent vaporizes near to the surface substrate then the resulting solid melts and vaporizes and the vapor diffuses to the substrate which undergoes a heterogeneous reaction. It is known that at high substrate temperature, the mobility of the ad-atoms is high and this fact promotes that the ad-atoms be able to diffuse reaching a nuclei and that desorption of product molecules from the surface be carried out. A matching of the observed and the standard '*d*' values confirms that the deposited films are of SnO_2 having tetragonal structure [20]. Crystallite size is estimated by using Scherrer's formula given by equation [26]:

$$D = \frac{k\lambda}{\beta\cos\theta} \qquad (3)$$

where k varies from 0.89 to 1.39. But in most of the cases it is closer to 1. Hence for grain size calculation it is taken to be one, λ is wavelength of X-ray, β is the full width at half of the peak maximum in radians and θ is Bragg's angle. It is observed that crystallite size increases initially with increase in substrate temperature reaches the maximum 33 nm at 475 °C and thereafter it goes on decreasing with increase in substrate temperature. Increase in crystallinity and crystallite size with substrate temperature is due to the optimum rate of supply of thermal energy for recrystallization with substrate temperature [27]. The values of crystallite size for different substrate temperatures are shown in Table 1.

3.3. *Scanning electron microscopy (SEM) studies*

The micrograph (Fig. 4) of the typical SnO_2:F film deposited at 475 °C shows that the film is continuous with the continuous distribution of grains. It shows that the sample has grains with different sizes whereas intergranular regions appear dark. The fine grains of 200 nm, cover the entire surface of the substrate.

3.4. *Optical transmission studies*

Fig. 5 shows the variation of transmittance with wavelength of SnO_2:F thin films deposited at various substrate temperatures. The average transmission in the visible region has been found to vary from 60% to 87% depending upon the substrate temperature. An increase in transmission is observed with increase in temperature. At lower temperatures, i.e. at 450 °C, relatively lower transmission is due to the formation of whitish milky films due to incomplete decomposition of the sprayed droplets. In general, in the visible region of the spectrum, the transmission is very high (high enough to observe interference fringes). It is due to the fact that the reflectivity is low and there is no (or less) absorption due to transfer of electrons from valence band to conduction band owing to optical interference effects, it is possible to maximize the transmission of thin film at particular region of wavelengths. Relatively higher transmittance of about 87% at 850 nm for films prepared at 475 °C has been observed.

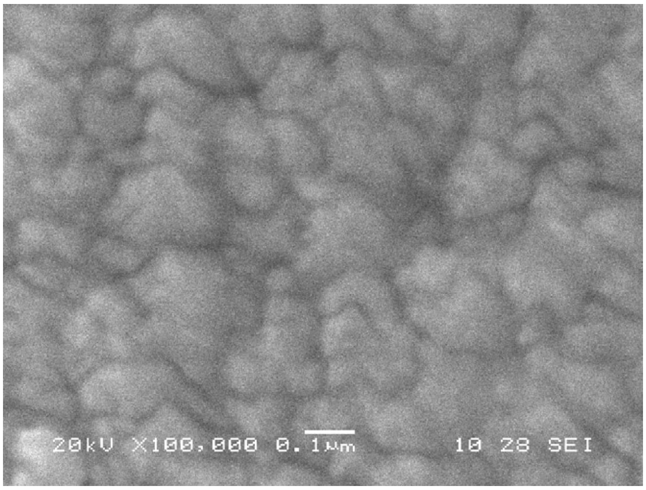

Fig. 4. The typical SEM micrograph of SnO_2:F films prepared at optimized substrate temperature of 475 °C.

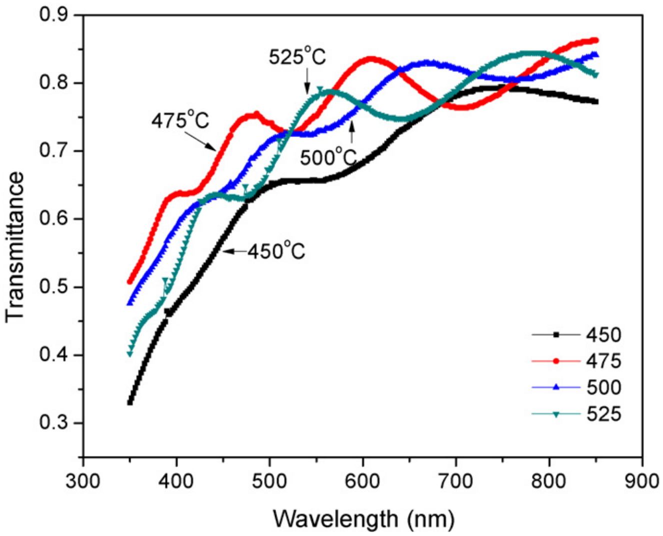

Fig. 5. Variation of transmittance with wavelength for spray deposited SnO_2:F thin films deposited at various substrate temperatures.

In order to estimate the band gap energy of the films, optical transmission of SnO_2:F films deposited on quartz substrates at 475 °C has been studied at room temperature in the wavelength of 200–850 nm. It is found that the optical absorption coefficient, derived from the transmittance, is a function of photon energy and the presence of an absorption edge. The optical absorption coefficient is of the order of 10^4 cm^{-1} supporting the direct and allowed band transition in the material. The variation of $(\alpha h\nu)^2$ with $h\nu$ has

Table 1
Values of various parameters of the sprayed SnO_2:F thin films deposited at different substrate temperatures.

Substrate temp. (°C)	t (nm)	R_s (Ω cm^{-2})	ϕ ($x \times 10^{-2}$ Ω^{-1})	ρ ($x \times 10^{-4}$ Ω cm)	n_D ($x \times 10^{20}$ cm^{-3})	μ (cm^2 V^{-1} s^{-1})	Crystallite size (nm)
450	440	9.0	0.43	3.96	3.63	11.98	28
475	740	5.3	1.25	3.91	4.71	10.96	33
500	620	9.8	0.59	6.07	1.84	15.41	22
525	445	22.6	0.38	10.07	0.98	17.49	17

t, film thickness; R_s, sheet resistance; ϕ, figure of merit; ρ, specific resistivity, n_D; carrier concentration; and μ, mobility.

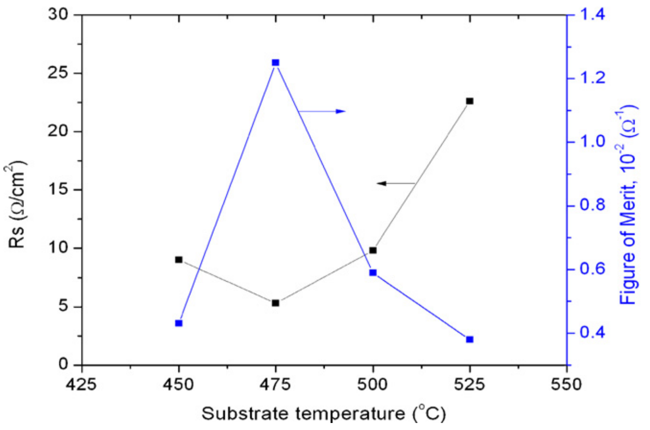

Fig. 6. Variation of sheet resistance and figure of merit with substrate temperature for spray deposited SnO_2:F thin films.

a straight line portion indicating that transition involved is direct allowed type [10]. The direct band gap, determined by extrapolating the straight portion to the energy axis to $(\alpha h\nu)^2 = 0$, is found to be 4.15 eV, which is higher than the value of $E_g = 3.57$ eV reported for single crystal SnO_2 [28] and is comparable to the value of $E_g = 4.3$ eV reported for SnO_x:F films [29,30]. This increase in band gap can be attributed to an increase in carrier concentration of the films due F doping. Similar optical band gap values have been reported for the SnO_2:F films [31,32]. Thin films often show a small band gap widening and the absorption edge moves to a shorter wavelength than that of the bulk material. However, when SnO_2 is doped with fluorine and the carrier concentration is of the order of 10^{20} cm^{-3}, a substantial band gap widening is expected [29] Similar results have been obtained for spray deposited SnO_x, SnO_x:F, and SnO_x:Sb films [33]. The fact that the onset of light absorption is shifted towards lower wavelengths in thin films, especially highly doped ones, turns out to be an advantage for optical applications in the UV.

3.5. *Electrical properties studies*

For SnO_2:F thin films, we have studied the effect of the substrate temperature on the electrical properties. The sheet resistance is calculated from measurements using the Van der Pauw technique [13]. For solar cell application of these films, the figure of merit (ϕ) of the film plays an important role. The device performance is determined from ϕ. It is calculated by using the formula [34]:

$$\phi = \frac{T^{10}}{R_s} \tag{4}$$

where T is the transmittance at $\lambda = 550$ nm and R_s the sheet resistance.

Fig. 6 shows the variation of figure of merit and sheet resistance with substrate temperature. It is seen that sheet resistance has its minimum value for the film deposited at 475 °C temperature, and it increases for further values of deposition temperature. This is due to relatively higher thickness and crystallite size of the deposits at 475 °C than other temperatures. From the Hall Effect measurements, it is observed that the SnO_2:F films exhibited n-type conductivity. Fig. 7 shows film resistivity (ρ) plotted against substrate temperature. Carrier concentration increases up to substrate temperature 475 °C and decreases for higher substrate temperatures. The increase carrier concentration observed at 475 °C can be related to both the observed improvement in crystallinity of the SnO_2:F thin films without any residual amorphous content and to a pin hole free surface due to the fact that these films grown at the optimal temperature mediated a CVD process. The decrease in carrier concentration at higher temperature can be related to the decrease in the oxygen vacancies due to at higher temperatures, there is a greater incorporation of oxygen to the films. The lowest resistivity (3.91×10^{-4} Ω cm) is found for the sample deposited at 475 °C, while the lowest resistivity reported for spray deposited SnO_2:F thin films is 5.8×10^{-4} Ω cm [35]. The values of carrier concentration and mobility for the sample deposited at 475 °C are 4.71×10^{20} cm^{-3} and 10.96 cm^2 V^{-1} s^{-1}, respectively. The value of various properties of SnO_2:F films deposited at various substrate temperatures is given in Table 1.

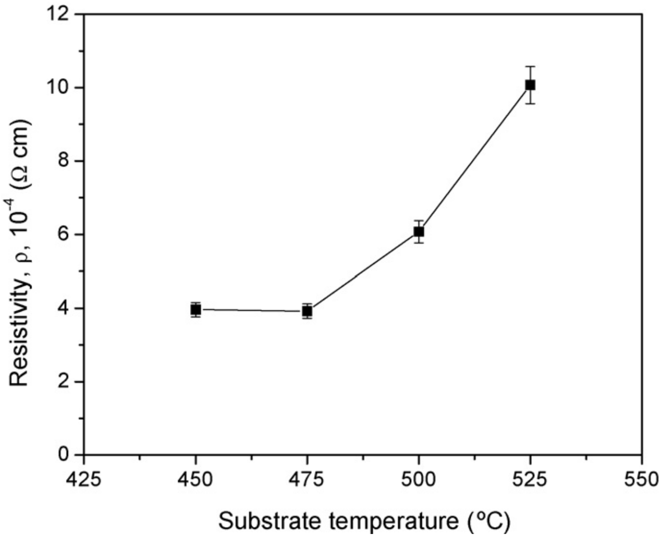

Fig. 7. Variation of resistivity (ρ) with substrate temperature for spray deposited SnO_2:F thin films.

4. Conclusions

SnO_2:F films have been prepared onto glass substrates from stannic chloride solution using spray pyrolysis technique. From the mapping studies it is observed that the central part of the hot plate of area ~3 cm^2 gives the good quality and uniform films in terms of thickness, refractive index and surface resistivity. By modifying configuration and engineering of deposition unit, synthesis of large area, uniform SnO_2:F thin films will be feasible. As revealed by XRD analysis, no SnO_2:F has been formed at substrate temperature less than 450 °C. The substrate temperature greater than 450 °C favors the pyrolytic reaction with tin oxide formation. The film deposited above substrate temperature 450 °C has been formed with preferential growth along (2 0 0) plane and crystallite size of 20–33 nm. The optical study revealed that band gap energy due to a direct transition is 4.15 eV. The detailed analysis of electrical phenomenon revealed that resistivity as low as 3.91×10^{-4} Ω cm is obtained for films prepared at substrate temperature of 475 °C. The efforts of improving the central area for maximum yield by testing different designs and arrangements of heating systems are still under study.

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