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RESEARCH ARTICLE

EFFECT OF PRECURSOR CONCENTRATION ON THE PREPARATION OF IN_2O_3 THIN FILMS PREPARED BY SPRAY PYROLYSIS

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ABSTRACT

Polycrystalline In_2O_3 thin films have been deposited on glass substrate by spray pyrolysis technique using indium acetate as a precursor solution. The influence of precursor concentration was studied in the range of 0.025 – 0.15 M with substrate temperature 450 ˚C. The deposited films were characterized by using X-ray diffraction (XRD), Scanning Electron Microscope (SEM) with EDS, Atomic Force Microscope (AFM), UV-Visible spectrophotometer and Photoluminescence (PL). The XRD analysis indicated that the films were highly preferential grain orientation along (222) plane with cubic crystal structure. The surface morphology, grain size and roughness of the films were observed by SEM and AFM. The UV-Visible spectroscopy revealed that the films had high transmittance in the visible region above 75% and the optical band gap values were found to be in the range of 3.3 – 3.68 eV. The optical constants such as refractive index (n), and extinction coefficient (k) were evaluated from transmittance data. The photoluminescence (PL) was studied at room temperature with a 325 nm as excitation wavelength.

INTRODUCTION

Transparent conducting oxide (TCO) thin films are very important material due to their high transparency in the visible region, good electrical conductivity, and high infrared reflectivity. They form essential components in enormous number of optoelectronic devices such as light emitting diodes, photo detectors, transparent window, flat panel displays, solar cells, gas sensors and anti-reflecting coating (Mihaela, 2004). Among various TCOs, In_2O_3 is a promising material for solar cell and gas sensing applications. It has n-type semiconductor behavior of 3.7eV optical band gap and high electrical conductivity (Gagaoudakis *et al.,* 2001). Several techniques have been used for preparing In_2O_3 films such as chemical vapor deposition (Girtan and Folcher, 2003), thermal evaporation (Mihaela *et al.,* 2000), spin coating (Chunga *et al.,* 1998), spray pyrolysis (Korotcenkov *et al.,* 2002), sol–gel (Bel *et al.,* 1997), electron beam evaporation (Rozati *et al.,* 1993), pulse laser deposition (Choi *et al.,* 2001), and Dc magnetron sputtering (Subramanyam *et al.,* 2001). Among these, spray pyrolysis is one of the most widely used methods which is the most reliable and economic one. Spray pyrolysis has been developed as a powerful tool to prepare various kinds of thin films such as metal oxides, superconducting materials, and nanophase materials. In comparison with other chemical deposition techniques, spray pyrolysis has several advantages such as low cost of the source materials, high purity, excellent control of chemical uniformity, and stoichiometry in multi-component system. The other advantage of the spray pyrolysis method is that, it can be adapted easily for production of large-area films. In most cases, indium chloride ($InCl_3$) has been used as the starting material and ethanol as the solvent mixed with water (Prathap *et al.,* 2008; Manoj *et al*., 2005; Bagheri *et al.,* 2012; Mirzapour *et al.,* 1992; Korotcenkov *et al.,* 2004; Sathyapriya *et al.,* 2010).To the best of our knowledge, there is no experiment reported elsewhere, describing the deposition of indium oxide thin films using indium acetate as precursor in spray pyrolysis method. In this paper, we report on the influence of precursor concentration on sprayed In_2O_3 thin films, at different molar concentrations using indium acetate as precursor solution. The optimum temperature was already fixed by depositing films at various substrate temperatures and subjecting them to various studies like XRD and SEM with EDAX, in order to obtain highly conductive transparent oxide films. The aim of the study is to optimize the molarity by keeping the substrate temperature as constant.

Experimental procedure

Indium (III) Acetate trihydrate [$In(CH_3COO)_2.3H_2O$] salt was dissolved in deionized water and sprayed onto the microscopic glass substrates with dimension of 75x25 mm^2, at different molar concentrations (0.025-0.15M) by keeping substrate temperature at 450^{o}C. The substrates were first cleaned with water bath, followed by dipped in conc. HCl, acetone and finally rinsed with deionized water and allowed to dry in a hot air oven. In spray unit, the substrate temperature was maintained with help of a heater and controlled by a feedback circuit. During the spray, the substrate temperature was kept constant with an accuracy of ±5^o C. Spray head and substrate heater were kept inside a chamber, provided with an exhaust

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fan, for removing gaseous by- products and vapors from the solvent. The spray head was allowed to move in the X-Y plane using the microcontroller stepper motor, in order to achieve a uniform coating on the substrate. The spray head could scan an area of 200 × 200 mm with X-movement at a speed of 20 mm/sec and Y-movement insteps of 5 mm/sec simultaneously. In the spray unit, there was a provision for controlling the spray rate of the solution as well as the pressure of the carrier gas. The microcontroller device was communicated with PC through the serial port in which the data of each spray could be stored.

The optimized parameter values are given bellow:

Spray time = 20 min
Solution flow rate = 2 mL min^{-1}
Air flow rate = 1kg cm^{-2}
Spray nozzle to substrate distance = 20 cm

After the deposition, the films were allowed to cool slowly to room temperature and washed with deionised water and then dried. The structural characterization of the deposited films were carried out by X-ray diffraction technique on SHIMADZU-6000 (monochromatic Cu Kα radiation, λ=1.5406 Å). The XRD patterns were recorded in 2θ interval from 10 to 80˚ with the steps of 0.05˚ at room temperature. Fourier transform infra red (FT-IR) spectra were recorded in the range 4000-400cm^{-1} using a BRUKER: RFS 27. FT-Raman spectra were recorded at room temperature using same instrument using 100nm excitation laser over wave number range 50-3700cm^{-1}The thicknesses of the films were then measured using Stylus profiler (Mitutoyo SJ-301). The surface morphological and topological studies were carried out using SEM (HITACHI, S-3400N) at a magnification of 10K and Atomic force Microscope (NANONICS MV 1000) respectively. Optical absorption spectrum was recorded in the range 300-1200 nm using JASCO V-670 spectrophotometer. The photoluminescence spectrum (PL) was studied at room temperature using plorolog 3-HORIBAJOBINYVON with an excitation source wavelength of 325 nm.

RESULTS AND DISCUSSION

Structural studies

Figure 1. Shows the XRD spectra of indium oxide films prepared at different molar concentrations (0.025–0.15M). As seen from the XRD spectra, all films are polycrystalline in nature with a cubic structure in bcc phase (JCPDS No-06-0416). For the low molar precursor concentration (0.025M), the deposited films are very thin and have a low intensity for the (222) reflection. For further increase in molarities, the films show improved crystalline structure and the intensity of (222) plane increases to a maximum up to a molarity of 0.1M. Other planes (400), (440), and (622) are also present, but their intensities are lesser than that of (222) plane. The intensity of the (222) plane decreases for higher molar concentration (above 0.1M) were as the intensity of (400) plane increases. Agashe *et al. (*1991) reported the similar behavior of change in the growth direction of SnO_2 films prepared by spray pyrolysis and reported that with increase of molar concentration, tin occupies the regular lattice sites at low concentration. When the molar concentration increase certain limit, Sn occupies regular interstitial rather than lattice vacancies, which change in the preferred growth of the films. Hence the present result correlated well with reported observation. Elangovan *et al.*, (2004) reported the similar behavior of for spray deposited SnO_2: F films prepared at different precursor concentrations and explained the variations of preferred orientation due to the variation of precursor concentration that alter the nucleation and growth process, which decides the orientation of the film. From this study, It was observed that the preferred orientation of the film was found to vary with precursor concentration.

The crystallite size is evaluated from the Scherer formula (Klung *et al.,* 1974)

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

where θ is the Bragg's angle and β is the full width at half maximum of the peaks and λ is the X-ray wave length. The micro strain (ε) is calculated using the relation

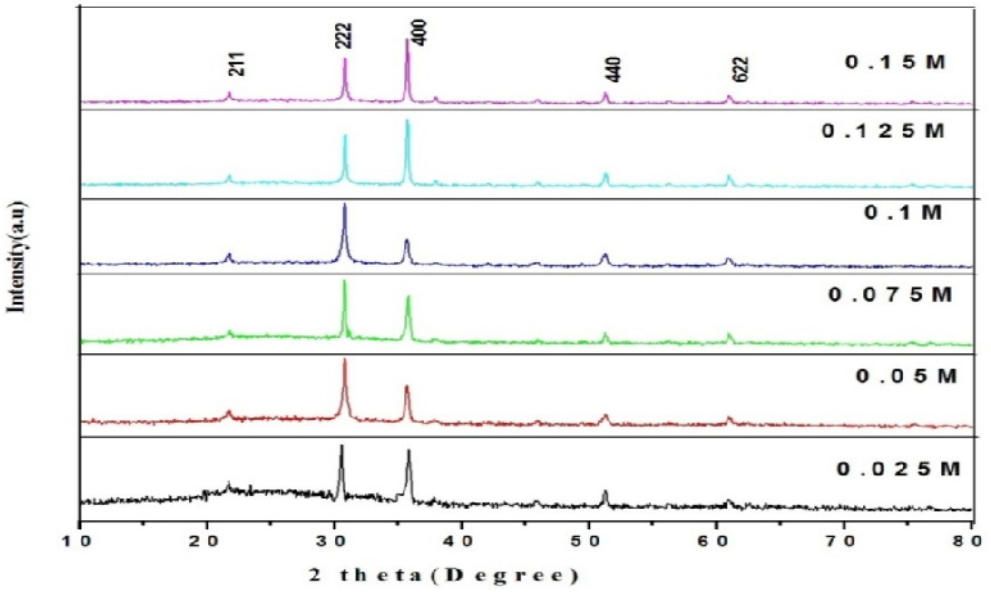

Figure 1. XRD spectrum of In_2O_3 films deposited at different molar concentrations

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

The value of dislocation density (δ) is calculated using the relation

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

From the full with half maximum (FWHM) values of the peaks, crystallite size of the films was determined using the Debye-Scherer formula (Table 1). With increasing molar concentration, crystallite size increased from 22.54-30.08nm. From the Table, it is evident that dislocation density and micro strain of (222) plane decreased with increase of molar concentrations.

boundaries in indium oxide thin films (Islam and Podder, 2009). The FT-IR transmittance spectrum of In_2O_3 films prepared under the optimized condition at 0.1M concentration is shown in Figure 3. The spectrum contains five peaks at 654, 614, 536 and 428, 322cm^{-1} corresponding to the In–O phonon vibration mode was characteristic of cubic In_2O_3. This result was in well agreement with the previous work reported in the literature (Korotcenkov *et al.*, 2005). Figure 4 displays Raman spectrum of pure In_2O_3 thin film for the 0.1M precursor concentration. Five bands were observed, these bands are situated at 126, 300, 360, 489 and 580 cm^{-1}, (with range 100-800cm^{-1}) corresponds to phonons associated with the bcc-structured In_2O_3, and also matched with the reported values (Chandradass *et al.*, 2011). The indium oxide belonged to cubic

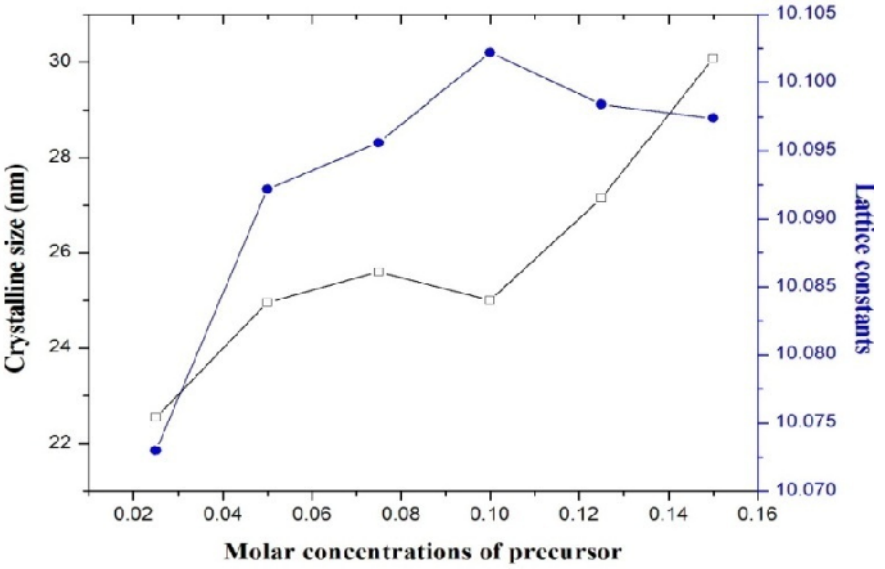

Figure 2. Variation of crystallite and lattice constant of In_2O_3 films prepared at 0.1M

Table 1. Microstructural parameters, thickness and optical band gap measurements value for different molar concentrations

Molar concentrations	2θ (degree)	Crystallite size (nm)	Lattice parameter (Å)	Dislocation density (x10^{14})	Micro strain (x10^{-3})	Thickness (nm)	Eg (eV)	Molar concentrations
0.025	30.7225	22.54	10.0730	1.9683	1.1772	175	3.68	0.025
0.050	30.6318	24.96	10.0922	1.6051	1.4721	211	3.64	0.050
0.075	30.6522	25.60	10.0956	1.5258	1.0521	225	3.63	0.075
0.100	30.6093	25.00	10.1022	1.6151	1.4520	260	3.62	0.100
0.125	30.6433	27.15	10.0984	1.3566	0.8255	307	3.42	0.125
0.150	30.6464	30.08	10.0974	1.1052	0.9534	297	3.30	0.150

Figure 2 shows the variation of lattice constant with molar concentration. The lattice constant of In_2O_3 films was found to be lower than the reported value of 10.118Å (Pramod *et al.*, 2012) for the cubic In_2O_3. The lattice constant increased from 10.07 to 10.15Å as the molarity of the precursor was increased upto 0.1M and the decreased to10.09Å for further increase of molarity. Thickness variation of In_2O_3 films deposited at different molar precursor concentration is shown in Table 1 As thickness depends on the band gap film deposition at higher molar concentrations are thinner as compared to films deposited at lower molar concentrations. This can be explained in terms of quantum size effect. Since in thin films, the average grain size is proportional to thickness of films, it is expected that the band gap of thin semiconducting films will increase quadratically, if charges are accumulated at grain boundaries. The decrease in the band gap with the increase in film thickness in our study indicates that there is no charge accumulation at grain

C-type rare-earth oxide structure and for this type of structure, the factor group analysis predicts 4Ag (Raman) + 4Eg (Raman) + 14Tg (Raman) + 5Au (inactive) + 5Eu (inactive) +16Tu (i.r.) modes. All the observed modes corresponded well with the band positions reported in the literature of cubic indium oxide (Vigreux *et al.*, 2001).

Surface morphology studies

Figure 5 shows the typical SEM image of In_2O_3 thin films obtained at Ts= 450^{o}C with different molar concentrations. Due to conducting character of all the films, no metal coating on surface of the samples was needed in order to obtain clear and very definit micrographs.It was found that the films showed large number of uniform spherical grains over the entire surface. It is observed that as the molar concentration increases the grain size also increases. From the XRD and SEM observations,the sample prepared with 0.1M were taken

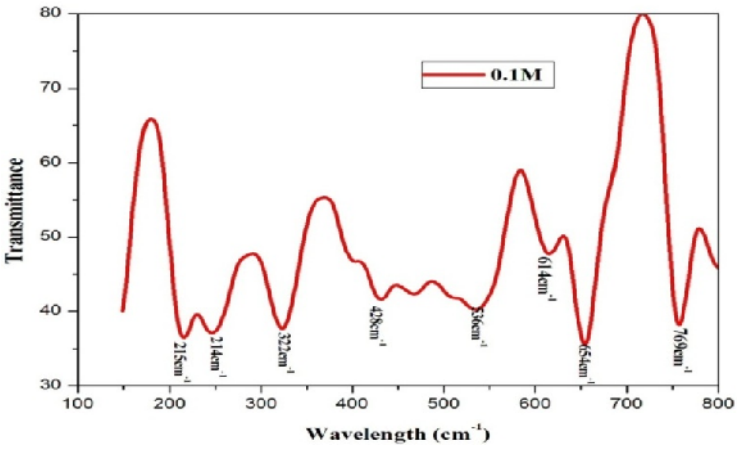

Figure 3. FT-IR spectra of In_2O_3 thin film prepared at 0.1M

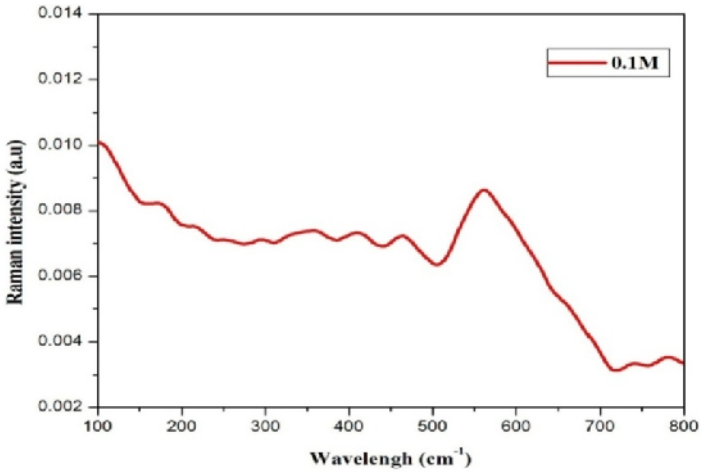

Figure 4. FT-Raman spectra of In_2O_3 thin film prepared at 0.1M

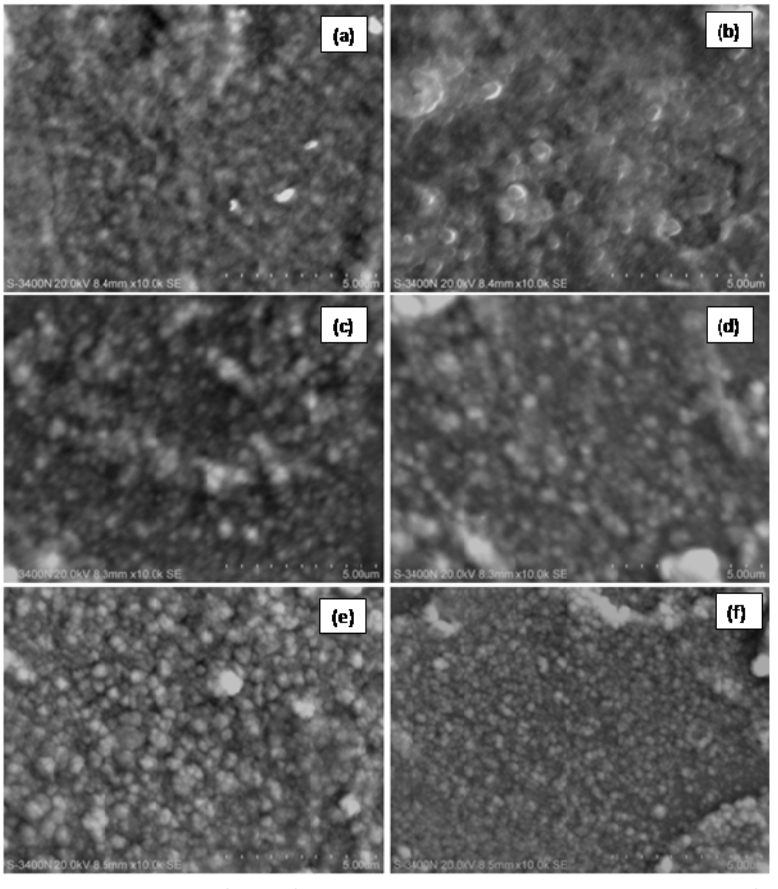

Figure 5. SEM images of In_2O_3 films prepared at various molar concentrations (a-f)

into consideration for further optoelectronic charactrizations. The chemical composition analysis was carried out using energy dispersive analysis of X-rays. Energy dispersion X-ray spectrometer (EDS), specra of the films are given in Figure.6. The atomic concentration of the films formed at different precursor concentration is given in Table 2. The films deposited at lower concentration of 0.025M showed atomic ratio of O to In of 0.83, which indicate the indium rich in the films.The atomic ratio of O to In of the film deposited at 0.1M was about1.03 showing that the film is in good stoichiometric In_2O_3 (see in Figure 6). Above this, the precursor concentration (0.125-0.15M), the atomic ratio of O to In of the films decreased to 0.86 due to excess of oxygen compared with lower concentration.The near stoichiometric was observed for the films prepared with tconcentration of 0.1M.

Table 2. Chemical composition of the In_2O_3 films formed at different molar concentration

Molar concentration	Oxygen	Indium	Oxygen/indium
0.025M	45.4	54.6	0.83
0.050M	46.4	53.6	0.86
0.075M	50.2	49.8	1.01
0.100M	50.8	49.2	1.03
0.125M	52.6	51.2	1.02
0.150M	54.6	53.2	1.02

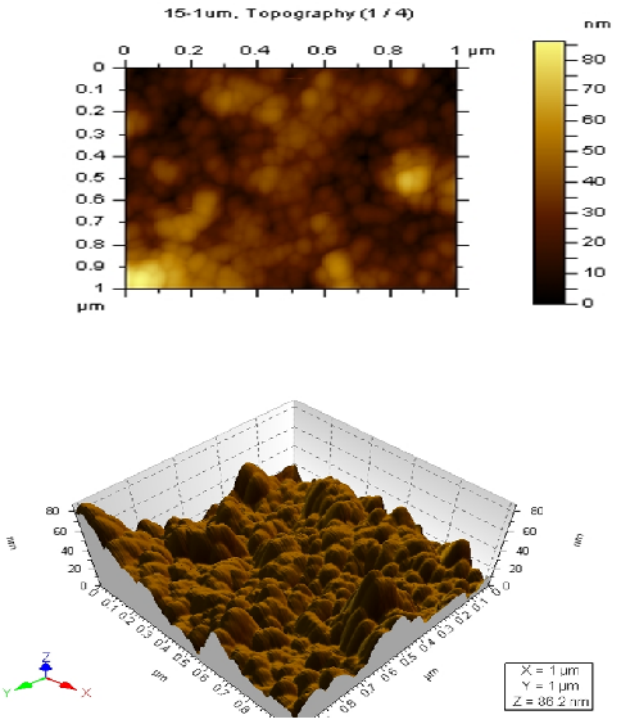

Figure 7. 3D-AFM topography of In_2O_3 film at 0.1M *Optical* studies

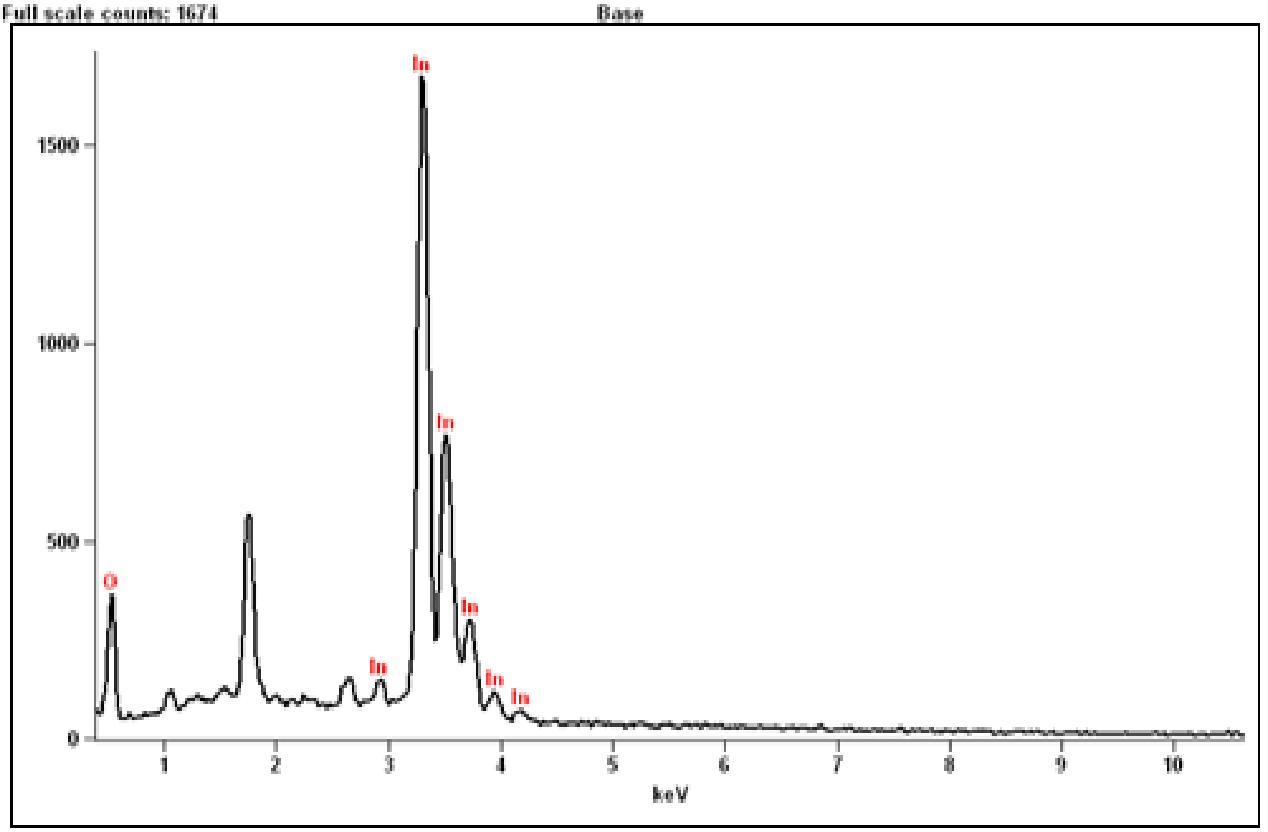

Figure 6. EDS spectrum of In_2O_3 film prepared at 0.1M

A study of the surface morphology by AFM gives a 2D and 3D presentation of the grain arrangement. Figure 7 shows the AFM picture of the In_2O_3 film prepared under the optimized condition at 0.1M. It can be seen that the film has better uniformity and crystallographic structure. The average roughness value was found to be 11.5nm.

Figure 8 shows the transmitance spectra of In_2O_3 films measured in the wavelength range between 300nm and 1200nm, deposited at 450^{o}C substrare temperatures.The deposition temperature exhibits a significant impact on the optical transmittance of the films.The optical trasmittance of the films decreased from 93 to 60% with the increase of molarity concentration from 0.025 to 0.15M. The higher

transmittance observed in the films was attributed to less scattering effects, structural homogeneity and better crystallinity, whereas low transmittance observed in the layers might be due to the less crystallinity leading to more light scattering (Bagheri *et al.*, 2012). The optical band gap of the In_2O_3 films is estimated from the relation (Bardeen *et al.*, 1956)

$$(\alpha h\nu)^2 = A(h\nu - E_g) \quad (4)$$

Where E_g is the optical band gap and A is a constant.

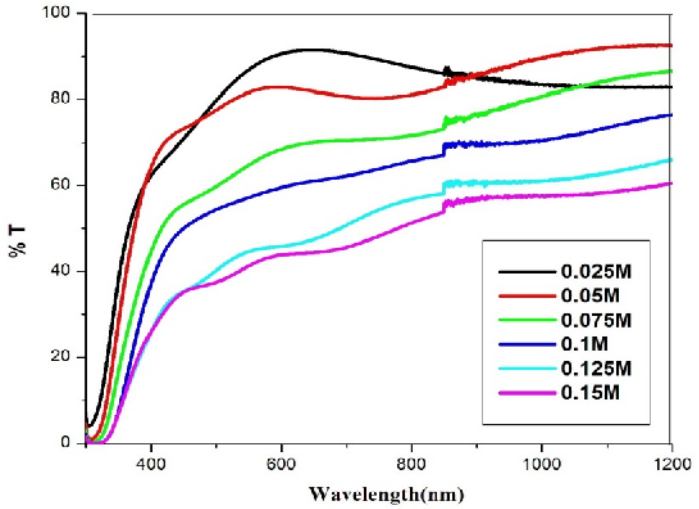

Figure 8. Optical transmission spectra of In_2O_3 films prepared at different molar concentrations

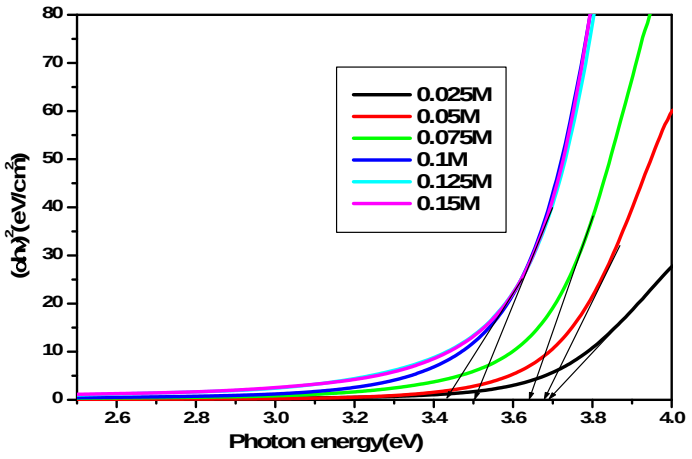

Figure 9. Optical bandgap spectra of In_2O_3 films prepared at different molar concentrations

A plot $(\alpha h\nu)^2$ as a function of photon energy ($h\nu$), was shown in Figure 9. The extrapolation of the linear part of the curves onto energy axis indicates the optical band gap, which decreases from 3.68-3.3eV with increase of molarity concentration from 0.025 to 0.15M (Sakthivelu *et al.*, 2011). The observed higher value of the energy band gap over the bulk value for the films deposited at lower precursor concentration might be due to the smaller crystallite size of the films. This could be explained on the basis of three-dimensional quantum size effect, which leads to an increase in the band gap with the decrease of crystallite size. The extinction coefficient(k) can be obtained from the relation (Prathap *et al.*, 2006)

$$K = \frac{\alpha\lambda}{4\pi}$$

The variation of extinction coefficient with wavelength is shown in figure.10. Extinction coefficient is high in the wave length range of 350-400nm and low in the wave range of 400-1200nm.The rise and fall in the extinction coefficient is directly related to absorption of light.

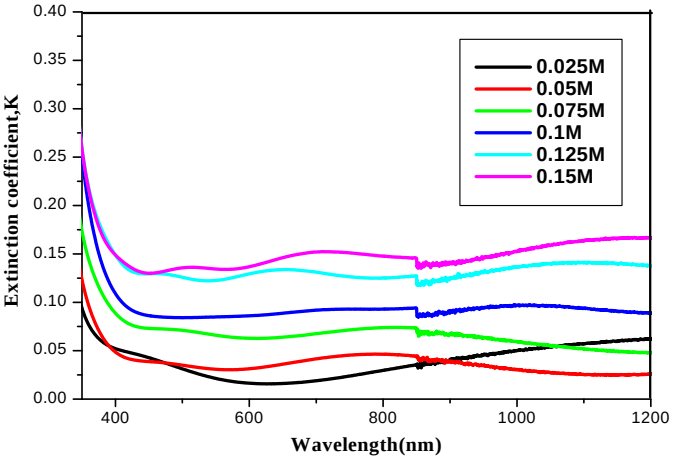

Figure 10.Variation of Extinction coefficient (k) versus wave length with various molar concentrations

The refractive index,n of the film is determined using the relation(Gielo *et al.*, 1987)

$$n = \frac{1 + R^{1/2}}{1 - R^{1/2}}$$

where R is the normal reflectance.

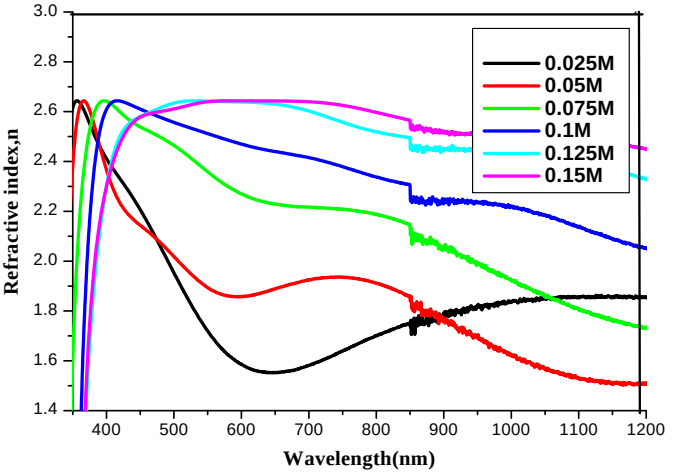

Figure 11.Variation of refractive index (n) versus wave length with various molar concentrations

Figure 11 shows the variation of refractive index of the In_2O_3 films with different wavelengths. From the result, it is learnt that the refractive index increases in the UV region and decreases gradually from 2.62 to 1.4 in the higher transmission range. Photoluminescence measurements were performed to investigate the optical properties of In_2O_3 thin films. Figure 12 shows the PL emission spectra of films deposited at different molar concentrations (0.025-0.15M) with an incident beam wave length of about 325 nm. The PL spectra are found to be dependent on the molar concentration. At the lower molar concentration (0.025M), a high strength violet emission (396nm) is observed. However, the intensity of the peaks reduces with the increase of molar concentration and also the

peaks are slightly shifted to blue emission. When the indium oxide films were deposited at 0.1M concentration two distinct shoulder peaks in the PL spectrum were observed in blue region at wavelength of 418 and 440nm. Therefore, the intense blue light emission can be attributed to oxygen vacancies and indium-oxygen vacancy centers (Kaleemulla *et al.*, 2008).

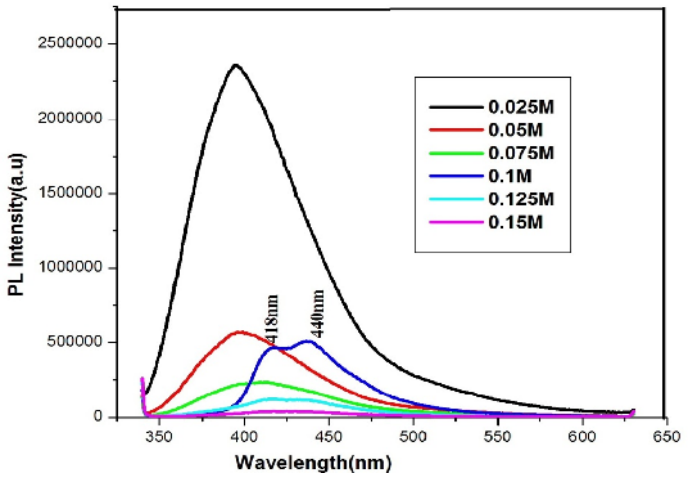

Figure.12. PL emission spectra of the films at different molar concentrations

Conclusions

In_2O_3 thin films have been prepared onto glass substrate with different molar concentrations of Indium acetate precursor keeping the substrate temperature as 450°C by spray pyrolysis method. XRD pattern confirm the polycrystalline structure nature of the films. The X-ray diffraction profiles of the deposited films showed a strong (222) and (400) reflection and a weak (211), (400), (411), (440) and (622) reflections. It was also observed that the preferred orientation of the films was found to vary with the precursor concentration. But the films prepared at molarity 0.1 M were strongly crystallized along the (222) orientation. The SEM and AFM analysis revealed that films prepared with precursor concentration 0.1M, the exhibited very smooth surfaces and good crystallographic structure. The optical band gap was found to vary from 3.3-3.68 eV. The band gap could be tuned by controlling the molar concentration of the precursor solutions. The higher values of optical constants such as extinction coefficient (k) and refractive index (n) are observed. The relative intensity of PL spectra of the In_2O_3 films decreased with increasing molar concentration. Therefore, it seems that the intense blue light emission (centered around 440 nm) was ascribed to the near band edge. In conclusion, the films prepared at optimized precursor concentration of 0.1M are most suitable for the optoelectronic applications.

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