PREPARATION OF FLUORINE DOPED NANOCRYSTALLINE TIN OXIDE THIN FILM

Khin Moh Moh Hlaing[1], Khin Than Yee[2], Hlaing Hlaing Oo[3]

Abstract

The increasing demand for energy that human beings are faced with the photovoltaic (PV) technology which converts solar radiation into electricity has undergone increasable development. Fluorine doped Tin oxide (FTO) thin film is widely used in various fields of electronic devices such as window layer in solar cell, substrates for electrode deposition and transparent contact in optoelectronic and so on. In this work, nanocomposites of FTO thin film was fabricated by sol-gel dip coating method and then it was applied as dye sensitized solar cell (DSSC) component. The effect of concentration of precursor solution, annealed temperature and heating time in the growth of FTO on glass substrates were studied and discussed. The prepared FTO thin film was analyst by FT IR. The surface morphology of FTO thin films was studied using Scanning Electron Microscope (SEM) to identify the distribution of grain and the growths of nanostructure with prefer orientation. The electrical properties of sample were analyzed by using four-point probe methods. The optical property was studied using UV-Vis spectrophotometer. The structural investigation of as-prepared film was performed using X-ray diffraction (XRD). The minimum value of sheet resistance was found to be 1.02 Ω sp^{-1} for the prepared SnO_2:F film at annealed temperature 400°C. The X-ray analysis confirmed the polycrystalline nature of FTO film with preferential orientation along 110 plane. The method was found to be economic and suitable for research and development. The prepared FTO was applied as an electrode in the dye sensitized solar cell (DSSC) electrode application and the photovoltaic effect was observed.

Keywords: SnO_2:F thin film, optoelectronic, nanocomposites, polycrystalline, solar cell

Introduction

Nowadays, the study and application of thin film technology is entirely entered into almost all the branches of science and technology due to brisk development of Nanotechnology. Fluorine doped tin oxide thin films belong to a special class of metal oxide thin films *i.e* transparent conducting oxide thin films which are a special part of nanostructure thin film solar cells. As it permits the transmission of solar radiation directly to the active region with little or no attenuation, these solar cells have improved sensitivity in the high-photon-energy portion of the solar spectrum and make thin film solar cells suitable for large scale application with high efficiency (Abdullahi *et al.,* 2014).

Many techniques have been employed to deposit SnO_2:F such as sputtering (Kang *et al.,* 2011; Singh *et al.,* 2013), inkjet printing technique (Samad *et al.,* 2011), aerosol assisted chemical vapour deposition (AACVD) (Jafar *et al.,* 2013), sol gel (Wu *et al.,* 2010), atmospheric-pressure plasma deposition system (Tsai and Huang, 2010), spray pyrolysis (Russo and Cao, 2007). Out of all the above methods sol-gel technique play an important role due to several advantages such as easy control on film thickness with a high porosity area which can improve the efficiency of the sensors, low processing cost, greater homogeneity and more purity. Generally, there are three methods used in corporate with sol-gel technique; they are spin coating, dip coating and spray coating. There are many reports regarding the preparation, characterization and application of the SnO_2:F thin films for dye sensitized solar cells (DSSC) (Alhamed *et al.,* 2012 ; Shelke *et al.,* 2013).

In the present work, an atmospheric chemical vapour deposition (APCVD) system was designed and fabricated for the deposition of fluorine doped tin oxide (SnO_2: F) thin films on glass with the aim of growing SnO_2: F thin films for fabrication of solar cells. Tin(IV) chloride

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and ammonium fluoride were used as the precursor and dopant. The characteristics of prepared transparent conductive films were investigated by FT IR, XRD, SEM, UV and also studied the various electrical and optical properties of the SnO_2:F thin film with number of coating, heat treatment temperature and heating time using four point probe methods. Furthermore, the structure and morphology characteristics for the prepared transparent conductive films were investigated. Finally, the prepared SnO_2:F thin films was tested in fabrication of dye sensitized solar cells.

Materials and Methods

Transparent conducting fluorine doped tin oxide thin films were prepared by sol-gel method accompanied with deposition methods. Structural, optical and electrical properties were studied under different preparation conditions like dopant concentration of ammonium fluoride, aging time, deposition methods, number of coatings, heat treatment temperature and heating time.

Preparation of fluorine doped tin oxide (SnO_2:F) solution

Fluorine doped tin oxide sol solution was prepared from tin(IV)chloride pentahydrate and ammonium fluoride by sol-gel method. About 13 g of tin(IV)chloride pentahydrate was added to 100 mL of ethyl alcohol in a beaker and stirred for 5 h. Then 10mL of ammonium fluoride solution (1 % w/w NH_4F) was slowly added to the beaker containing tin(IV) chloride solution. Five milliliter of 1 M hydrochloric acid solution was then added into the above solution drop by drop. Then the solution was stirred for at least 5 h. Finally, the solution was stirred and refluxed for one hour at 60 °C. Then the solution was cooled under room temperature and aged in open beaker for about 9 days. The same procedure was carried out with different concentrations of ammonium fluoride solutions (2.5 %, 5 %, 7.5 % and 10 %). The same procedure was carried out to prepare pure tin oxide thin film without ammonium fluoride. The prepared SnO_2:F solutions with various weight percent of ammonium fluoride (1 %, 2.5 %, 5 %, 7.5 % and 10 %) was taken and the viscosity was measured by Oswald viscometer.

Preparation of fluorine doped SnO_2 thin films

The preparation of fluorine doped tin oxide thin films composed of two steps (Yousaf, and Ali, 2008). For thin film deposition, glass slides (75 mm × 25 mm) were used as the substrates. The substrates were washed with detergent solution and with water. These substrates were boiled in chromic acid for five minutes and cleaned with distilled water. Then they were kept in sodium hydroxide solution to remove the acidic contamination and were again washed with distilled water. Finally, the substrates were dried in alcohol (methanol) vapour. And then, the prepared sol solution was deposited on cleaned glass substrate by the layer and layer deposition cycle (dip-, spin-, spray- coating) was done by alternatively between coating a thin layer and drying in air after each new layer (Figure 1). The coated glass substrate was annealed at temperature 100 °C, 200 °C, 300 °C, 400 °C and 500 °C for about 1 h, 2 h, 3 h, 4 h and 5 h in an electric furnace. In all, more than ten samples were produced simultaneously at each substrate temperature. Process was repeated several times for each parameter.

(a) (b) (c)

Figure 1 Deposition of thin films using various methods (a) Spray coating (b) Spin coating and (c) Dip coating

Characterization of the Prepared Fluorine Doped Tin Oxide Thin Film

Physicochemical properties of these prepared thin film were characterized by using modern techniques such as FT IR, XRD, SEM and UV.

Determination of electrical properties

The electrical measurements were carried out by LCR meter at room temperature by four-point probe methods. The electrical properties of the prepared fluorine doped tin oxide films were measured by LCR meter. The techniques employed were in accordance with the recommended standard procedure as reported in the company's catalogue. The resistance values of prepared SnO_2:F thin films with different dopant concentration of ammonium fluoride were measured by four point probe method.

Results and Discussion

In the present work, SnO_2:F transparent thin films were prepared by the Atmospheric Pressure Chemical Vapour Deposition (APCVD) method. Transparent conductors are needed as the front surface electrodes in all types of solar cells. Deposition of high quality, uniform thin films, is an intensive area of research which has yielded different deposition techniques. Each technique falls into one of three broad categories: wet chemical deposition; physical vapour deposition and chemical vapour deposition. The APCVD method was chosen due to its low cost of deposition and the optical and electrical characterizations of the deposited film were examined. SnO_2:F films were produced over a range of deposition temperatures, number of coatings and various weight percent of fluorine in the precursor solution from tin IV chloride pentahydrate and ammonium fluoride in ethanol.

The effect of fluorine concentration on the structural, surface morphological and characteristic properties of the SnO_2:F films were studied. The characteristic properties of the multi-coating have been investigated, including their electrical conductivity and optical transparency. The surface morphologies of the prepared thin films were characterized by scanning electron microscopy (SEM) and the crystalline phase of SnO_2:F thin films were determined by XRD (X-ray diffraction) using a diffractometer with CuK a radiation. At present, the main method of utilization of solar energy is the converse of solar energy into other energy sources.

Effect of Aging Time of the SnO_2:F Sol Solution

The fluorine doped tin oxide (SnO_2:F) thin films are a special kind of material that exhibit electrical conductivity and transmittance in visible region making it suitable for solar all application. The specific viscosity of the prepared SnO_2:F sol solution with the various weight percent of ammonium fluoride (1 %, 2.5 %, 5 %, 7.5 %, 10 %) at ambient temperature were measured by Oswald viscometer. The rheological behaviour of the sol solution using 7.5 % w/w NH_4F is shown in Figure 2. The viscosity of SnO_2: F sol solution gradually increased first two days. After two days, the viscosity breaks off the base line termed as break off point (viscosity markedly increased) and saturated on the sixth day called as the saturation point (viscosity slightly changed). It was evident that viscosity abruptly increase between the third and sixth day of aging (*i.e.* between the break off point and saturation point) and this rise was apparently due to the occurrence of polymerization, *i.e.* condensation, among the hydroxyl ligands attached to the tin ions. The saturation behaviour indicated the colloidal nature of the gelatinous suspension, which was devoid of strong cross-linking among the clusters in the solution. The physical observation of the solution during the entire period shows increasing cloudy nature of the solution, due to depolymerization.

According to the experimental evidence in this research, if the films were developed by using the gel aged lower than its break-off point, then the spreading and thinning mechanism of the sol becomes strong and as a result the evaporation process was also rapid. Above 7 days of aging time non-uniformity in films thickness and striation, resistance cannot be measured. If the films were coated with the solution above the saturation point, most quantity of the sol gel is thrown away from the substrate due to high viscosity, leading to poor adherence and also causes comet formation on the as-coated film. Among 2 to 6 days of aging, good and uniform coating can be obtained. Therefore, it can be inferred that the optimum coating period of the sol solution, so as to obtain good quality films, a time $t_{½}$ can be adopted, at which the viscosity reaches one-half of the sum of the break off and saturation viscosities. From Figure 2, $t_{½}$ value is found to be 4 days and this is regarded as the optimum coating time for the sol solution. But experimental observation revealed that sol solution coated between the period 4 and 6 days provided good quality films. According to weight of ammonium fluoride percent 5 to 10 give $t_{½}$ values nearly the same, weight of ammonium fluoride percent above 5 should be used for SnO_2:F film preparation.

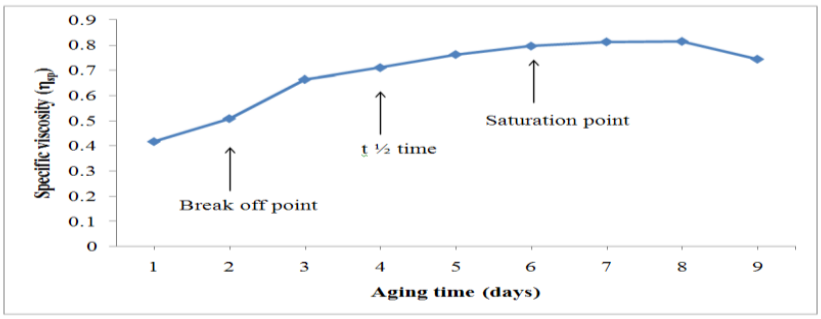


Figure 2 Variation of specific viscosity of the prepared SnO_2:F sol solution (7.5 % w/w NH_4F) with aging time

FT IR analysis for prepared fluorine doped tin oxide films

FT IR spectra of prepared SnO_2:F thin films on glass substrate at (300 °C and 500 °C) are shown in Figure 3. FT IR spectra have been assigned to the absorption peaks of Sn-O, Sn-O-Sn, Sn-OH or O-H bond vibrations. The absorption peaks between 400 cm^{-1} to 700 cm^{-1} can be assigned to Sn-O and Sn-O-Sn vibration of SnO_2. Small peaks between 1600 cm^{-1} to 1900 cm^{-1} are attributed to Sn-OH vibration mode. Since the precursor solution contains water, Sn-OH vibration mode appears in the spectrum. In the spectrum, an absorption and corresponding to the presence of adsorbed water (1630-1640 cm^{-1}) and hydroxide absorption bands in the range of 3500-3700 cm^{-1} were observed. The bonds nearly 1000 cm^{-1} was assigned to chloride contamination, which arises from chloride precursor. The peaks were assigned to O-Sn-O and Sn-OH in the range of 500 cm^{-1} to 900 cm^{-1} (Stretching vibrations and variations).

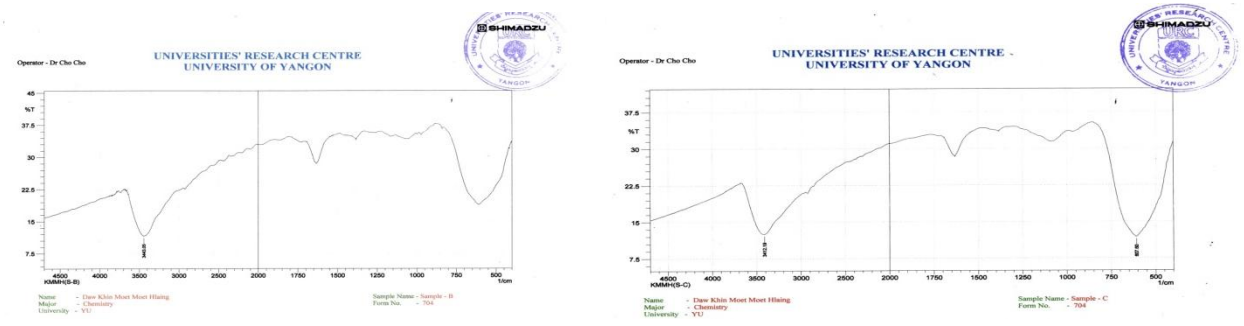
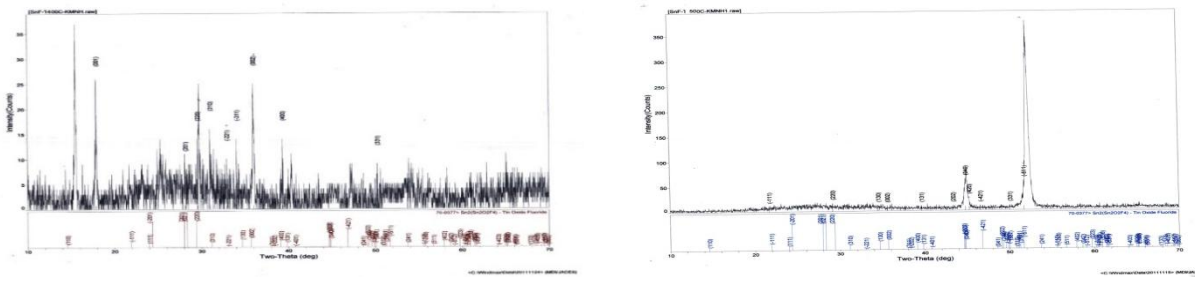


Figure 3 FT IR spectra of prepared SnO_2:F thin films at (a) 300 °C (b) 400 °C

X-ray diffraction analysis for prepared fluorine doped tin oxide films

The crystal structure of the prepared SnO_2:F thin films were identified by XRD diffractometer. The X-ray diffractogram of prepared undoped tin oxide (SnO_2) thin film and prepared 7.5% w/w NH_4F SnO_2 thin film at 400 °C and 500°C are presented in Figure 4. The crystal structure of fluorine doped thin films were studied in the 2q range of 10° to 70°. X-ray diffraction measurements were made to determine the d-values, crystallographic structure, lattice parameters and grain size. All the diffractograms contained the characteristic of SnO_2 orientations. The observed d-values are compared with the standard ones from the (JCPDS-no-70-0377) data files. The matching of the observed d-values and standard d-values confirms that the deposited films are of tin oxide with tetragonal structure.

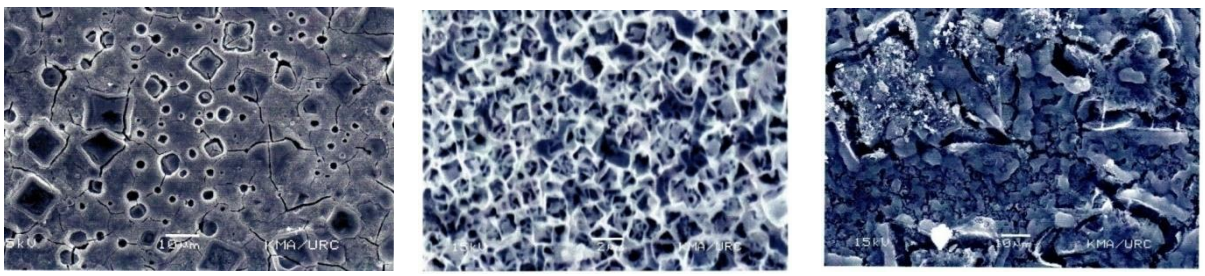


(a) (b)

Figure 4 XRD diffraction patterns of prepared SnO_2:F thin film 7.5% w/w NH_4F at (a) 400 °C and (b) 500 °C

SEM analysis

SEM micrographs showed agglomeration of the grain particles and flower like structure. The morphology of the prepared SnO_2:F thin films studied by SEM analysis indicated the films comprise of small grain size particles with less crack on the surface. SEM micrographs of prepared fluorine doped tin oxide films with different heat treatment temperature (300 °C, 400 °C and 500 °C) are shown in Figures 5. All of the images were found to be polycrystalline with various surface grain shapes and sizes. It was observed that the grain size become larger and the crystallinity was improved with the increase in the heat treatment temperature. The surface roughness was observed to be improved because the radical's mobility at the sample surface enhanced with substrate temperature. These results agree with the results of Tatar *et al.*, (2013). The fluorine doped tin oxide film characterized by uniform size grains with cubical shape at the substrate temperature 400 °C, which is on the average smaller than grains in the high temperature region. Consequently, the results were good agreement with XRD observation.



(a) (b) (c)

Figure 5 SEM micrographs of prepared SnO_2:F thin films (7.5% w/w NH_4F) at different temperature (a) 300°C (b) 400°C and (c) 500°C

Optical Properties of the Prepared Fluorine Doped Tin Oxide Thin Film

Optical analysis of fluorine doped tin oxide thin film on glass substrate at different temperatures was studied from transmission (%) vs wavelength curve in the wavelength range 400-1000 nm. The high optical transmittance for the film heat treated at the temperature 400 °C which is shown in Figure 6. Highest value of transmittance is observed to be 95.15 % for the film heat treated at 400 °C and this is attributed to the low scattering effect and uniform film thickness caused by smooth and good surface texture of the film. In the visible region of the spectra, the transmission of film was very high, due to fact that the reflectivity is low and there is less absorption due to excitation of electrons from the valence band to conduction band (Tripathy *et al.,* 2013). It may be concluded that the transmittance was more which may be due to high porosity and larger grain size and less absorption in the film. According to the transmittance data it was found that the deposition temperature was improved the optical transmittance between 85 % T and 95 % T.

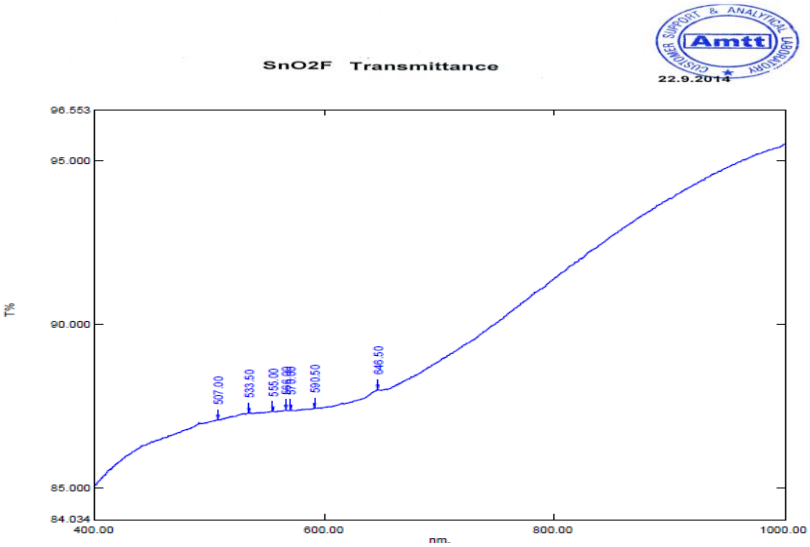


Figure 6 Transmission spectrum of SnO_2:F thin film (7.5% w/w NH_4F, 400 °C) as a function of wavelength

Electrical Properties of the Prepared Fluorine Doped Tin Oxide Thin Film

Electrical testing was done to assess the electrical performance of the best films produced from each deposition technique. Sheet resistance measurements were recorded using a linear four-point probe (Olopade *et al.,* 2012) method. Four-point probe method is an electrical resistance measuring technique that uses separate pairs of current-carrying and voltage-sensing electrodes to make more accurate measurements than traditional two-terminal (2T) sensing. Hall Effect measurements were done using the Van der Pauw technique in order to determine the dominant charge carrier type, charge carrier mobility and charge carrier concentration.

The change of sheet resistance of the SnO_2:F thin films coated on glass substrate for a substrate temperature of 200 °C and different weight percent of fluorine 1.0 % w/w to 10.0 % w/w NH_4F, the minimum sheet resistance value was obtained at 7.5 % w/w NH_4F. The increasing of sheet resistance after a specific level of fluorine content probably represents doping limit of fluorine in the tin oxide lattice. The excess fluorine atoms do not occupy the proper lattice position to contribute to the free carrier concentration and at the same time the increasing disorder leads to the increase of the sheet resistance (Miao *et al.,* 2010).

According to the literature, in sol-gel derived thin films, the highly porous with small crystal sizes is formed especially in the case of single layer coating. However, in multiple

coating, sheet resistance of SnO_2:F thin films can be increased (Gasparro *et al.*, 1998). The sheet resistance decreased gradually from 73.99 W sq^{-1} to the minimum 8.91 W sq^{-1} with the number of coating from 2 times to 10 times. Therefore, number of coating was optimized to 10 times, in order to get film of good electrical and optical properties.

Deposited films by sol-gel method, contain water and organic materials, and mechanical properties of the film are poor and must be improved. Therefore, the sample should be put in an oven for a suitable time and temperature. This improves the optical and mechanical properties of thin film and also removes water and organic materials from thin film. The sheet resistance value of SnO_2:F thin films at 250 °C was (9.85 W sq^{-1}) and its gradually decrease to 3.71 W sq^{-1} with the heat treated temperature of 400 °C. And then, the resistance value increased with the heat treated temperature of 450 °C and 500 °C. According the previous reports, the substrate temperature was over 400 °C, the amorphous structure transformed into crystal structure, and then the narrowing of grain boundaries which result in an decreased in the conductivity (Aldelkani *et al.*, 2007). The sheet resistance does not change more with the temperature increase from 400 °C to 500 °C, due to the slight change of the crystal structure. Therefore, the sheet resistance was slightly varied with the substrate temperature in the range of 400-500 °C. Therefore, the sheet resistance value and polycrystalline nature depends directly on the heat treated temperature. Thus, the sheet resistance of prepared SnO_2:F thin films at 400 °C was observed lowest value and this temperature is optimal temperature for that treatment process.

The change of sheet resistance of the SnO_2: F thin films coated on glass substrate with heat treated temperature (400 °C), number of coating (10) times, weight percent of (7.5 % w/w NH_4F) and heating time (1, 2, 3, 4 and 5) h, it was found that the sheet resistance value decreased when the annealed time was increased from 1 to 3 h and then increased when annealed time was increased from 4 to 5 h. The minimum sheet resistance value was 1.02 W sq^{-1} at the annealed time of 3 h. This is because the carrier concentration decreased with higher rate due to the loosing of fluorine atom. The optimum point was found at the annealed time of 3 h.

Table 1 shows the variation of sheet resistance value of prepared fluorine doped tin oxide thin film (7.5 % w/w NH_4F) with three deposition methods such as spray, spin and dip coating. Among them, a dip-coating deposition is one of the most promising ones, due to the simplicity of the apparatus, lost-effectiveness, good uniformity of the films and well suitability for large-scale production. It was also found that the minimum resistance value (1.7 W sq^{-1}) for dip-coating method. Therefore, based on their resistance value, dip-coating method is suitable for the preparation of fluorine doped tin oxide thin film.

Table 1 Sheet Resistance of the Prepared SnO_2:F Thin Films by Various Deposition Methods

Sr. No	Deposition Method	Sheet Resistance (Ω sq^{-1})
1	Spray	3.49
2	Spin	2.06
3	Dip	1.70

No. of coating = 10 times
Heated Temperature = 400 °C
Sample = 7.5 % w/w NH_4F

Conclusion

The present investigation revealed that transparent conducting fluorine doped tin oxide thin films (SnO_2:F) were prepared on glass substrate by atmospheric pressure chemical vapour deposition and sol gel dip-coating method. The precursor sol solution was prepared from tin(IV) chloride pentahydrate $SnCl_4.5H_2O$, ammonium fluoride and ethanol as solvent. Structural, optical and electrical properties under different preparation conditions like concentration of ammonium fluoride, viscosity of the sol solution, coating period, heat treatment temperature, heating time duration and deposition method. Then the prepared thin films were characterized by modern techniques such as FT IR, XRD, SEM and UV. Fluorine doped tin oxide thin film of excellent reproducibility, adherence and device quality was prepared by dip coating method via sol gel route under optimized conditions viz, dopant concentration 7.5 % w/w NH_4F, heat treatment temperature 400 °C, number of coating 10 times and heating time duration 3 hour. The rheological studies indicated that the optimum coating period for the sol solution lie between 4 and 6 days.

The X-ray diffraction studies confirmed the tetragonal structure with polycrystalline nature. The preferred directions of crystal growth in the diffractogram of SnO_2: F films correspond to the reflection from the (110), (200), (220) and (310) planes. The matching of the observed and standard d-values confirmed that the deposited films are of tin oxide with tetragonal structure. FT IR spectroscopy showed strong Sn-O and Sn-O-Sn bonding. The minimum value of resistance was 1.02 W for the film heat treated at 400 °C for 3 h (7.5 % w/w NH_4F). The nature of the films indicated that at lower temperature (100°C-400°C), chemi-sorptions mechanism predominates and grain boundary scattering become low, which reduced the film resistance. At higher temperature (above 400 °C) oxygen desorption phenomena become predominant with higher grain boundary scattering which causes an increased in resistance.

The SEM micrographs showed that the variation of substrate temperature results in different grain size and shapes for different orientations while the SnO_2:F film heat treated at 400 °C shows uniform surface pattern with evenly distributed fine grains. The film deposited by dip-coating showed clear flowerlike crystal shape on the surface and the presence of uniform and dense microstructure apparently devoid of any cracks and voids, although it was possible that some microscale porosity was present in the film. Besides that, there are some crystal shapes structures like as mostly tetragonal shape. This observation was approved to the XRD results as tetragonal structure of prepared SnO_2:F thin films.

The optical property of prepared SnO_2:F thin film showed that thin films are fully transparent in the visible region (400 to 1000 nm). All the SnO_2:F thin films showed good transmittance in the visible region and the fundamental absorption edge lies in the UV region. In the present work, highest value of transmittance is observed to be 95.15 % at 1000 nm for the film heat treated at 400 °C and this is attributed to the low scattering effect and uniform film thickness caused by smooth and good surface texture of the film.

Thus, this studies indicated that sol gel dip coated SnO_2:F films prepared under the optimized operating conditions could be potential for the solar cell application. Consequently, a large area of solar panel can be fabricated based on the preparation of dye sensitized solar cell using prepared SnO_2:F transparent conductive films. Further research will be much more necessary to develop these types of material in the future.

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