Flourine Doped Tin Oxide Nano Films for Photo Voltaic Applications using Sol-Gel Process

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Abstract

Transparent Conducting Oxides (TCOs) have been widely used in Solar photovoltaic applications. Flourine doped Tin Oxide (FTO) has been extensively as TCOs as well as in solar thermal emissive coatings. Sol-gel dip coating (SGDC) technique is simple, yet cost effective way of obtaining highly cost effective way of obtaining highly uniform TCO coating. Further issues like formation of cracks and pores and alkaline contamination due to longer curing time at have temperatures have to be addressed. In the present work, FTO coatings has been developed using Sol-Gel process on glass substrates at temperatures 300^{0}C and 500^{0}C

1. Introduction

Transparent conducting oxides (TCOs) are the most commonly used materials to produce transparent conducting films. They are essentially based on oxides such as In_2O_3, SnO_2, ZnO and CdO. These materials are usually insulators and have a wide band gap (> 3 eV) and excellent transparency in the visible region. To provide them conductivity, either non-stoichiometry or appropriate dopants, like Sn for In_2O_3, Sb or F for SnO_2, and Al or Ga for ZnO, should be introduced in order to create electron degeneracy in the wide band gap. This property, in combination with high transparency to solar radiation, can be exploited for applications such asphotovoltaic cells [1]. As TCO must necessarily represent a compromise between electrical conductivity and optical transmittance, a careful balance between the properties is required. For Photo Voltaic (PV) applications, improvement in optical and conducting properties will eventually impact the performance of the complete device [2].

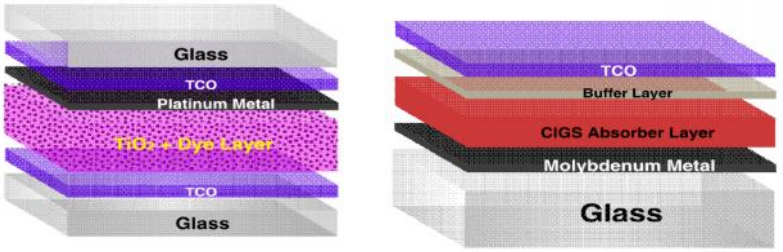

figure 1: configuration of different solar cells

TCO is an essential coating for all types of solar cells. The dominant polycrystalline thin film solar cells: CdTe Solar cells, CIGS solar cells, a-Si:H solar cells and Dye Sensitized Solar cells require TCO films to be deposited in various configuration as shown in figure.

Sol-gel Process and Dip Coating

The sol-gel process may be described as the formation of an oxide network through the poly condensation reactions of a molecular precursor in a liquid. The precursors for synthesizing these colloids consist usually of a metal or metalloid element surrounded by various reactive ligands. The starting material is processed to form a dispersible oxide and forms a sol in contact with water or dilute acid. Removal of the liquid from the sol yields the gel, and the sol/gel transition controls the particle size and shape. Calcination of the gel produces the oxide.

Dip coating is a popular way of creating thin and uniform films can be applied onto flat or cylindrical substrates.

In dip coating process the substrate is immersed in the solution of the coating material at a constant speed.The substrate is then remained inside the solution for a while and is started to been pulled up.The thin layer deposits itself on the substrate while it is pulled up. The withdrawal is carried out at a constant speed to avoid any judders. The speed determines the thickness of the coating (faster withdrawal gives thicker coating material). The excess liquid will drain from the surface. The solvent evaporates from the liquid, forming the thin layer.

For volatile solvents, such as alcohols, evaporation starts already during the deposition & drainage steps. In the continuous process, the steps are carried out directly after each other. To obtain the final coating material, normally a further curing or sintering step is then necessary.

The consolidation step represents the actual sol-gel transition with concomitant processes of draining, evaporation and hydrolysis. In this stage of deposition, any turbulence or variation in the atmosphere will inevitably lead to inhomogeneities in the film properties.

The process of film formation is based on a fluid mechanical equilibrium between the entrained film and the receding coating liquid. The two regimes are divided by a stagnation line, above which the liquid is entrained by the substrate whereas the liquid below is retained in the bath. The equilibrium is governed by several forces from which the most important are viscous drag and the gravity force, but other forces like surface tension, the inertial force can also play an important role.

Landau - Levich equation gives the relation between withdrawal speed and final thickness

$$h = 0.94 \cdot \frac{(\eta \cdot v)^{2/3}}{\gamma_{LV}^{1/6} (\rho \cdot g)^{1/2}}$$

Where, the constant (.94) is used for the Newtonian liquid,η- liquid viscosity, v- withdrawal speed, λ - surface tension of the liquid against air, ρ- liquid density and g -gravitational force.

In the above expression, it is seen that, the thickness of the coating is directly proportional to the withdrawal speed. However, in most cases, these theoretical treatments even with more complex extension do not apply in the practical situation and usually the calibration curve is determined by actual experimental trial.

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2. Experimental

2.1 Preparation of Flourine doped Tin Oxide (FTO) Sol:2- Iso Propanol (IPA) (purity 99.7%, Fisher Scientific), 2-Isopropoxy ethanol (IPE)(Purity 99.0%, Sigma Aldrich) and n-Butyl tin hydroxide (purity 95.0%, Alfa Aesar) are taken in a 200 ml glass bottle and stir for half an hour at roomtemperature. To this mixture addTrifluoro acetic acid (purity 99.0%, Alfa Aesar) drop by drop while stirring. The mixture is maintained at temperature 50 0C using hot water bath. The mixture is ultra-sonicated for 20 minutes in between. This process is carried out until a clear solution is obtained [7].

2.2 Dip Coating and Calcination:

The glass substrates of dimensions (10 cm x 10 cm) are cleaned with tap water. Then cleaning is done with alconox solution. The concentration of alconox is 0.3 g in 25 ml of distill water. Later they are cleaned with tap water. Further they are cleaned with distill water. Finally the substrates are washed with IPA. And then they are dried with drier. Before coating the glass surfaces are air blown to remove any adsorbed dust particles.

These glass substrates are dip coated with FTO sol. The dwelling time of the glass substrates is around 30-60s. The withdrawal speeds are 1mm/s, 2mm/s, 3mm/s and 4mm/s. After dip coating the glass dried in an oven at 100^0C for 1 h. Then the coated glass substrates are cured at 300^0C and 500^0Cin ambient atmosphere [8].

The coated glass substrates are characterized using UV-Vis-NIR spectrophotometer (Cary 5000).The thickness of the coating on glass substrates was measured using ellipsometer. The conductivity measurements are done using multi-meter.

3. Results and Discussion

TFA should be added slowly 1 ml by 1ml to prevent turbidity of the sol.Addition of more amount of TFA is not preferred as it will increase the acidity of the solution. Glass substrates are dip coated with FTO Sol at different withdrawal speeds 1, 2,3 and 4 mm/s. The higher withdrawal speeds increases more transmittance. This may be due to lesser thickness of the coating.

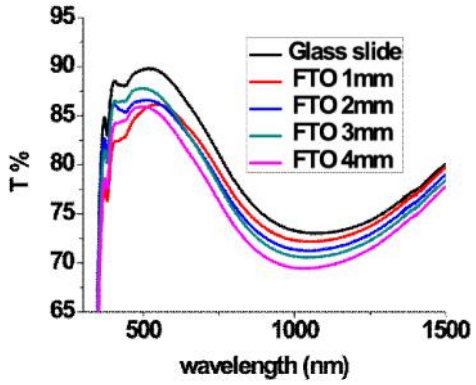

figure 2. Transmittance of FTO coated glass substrates at 300^0C

Table The transmittance values for FTO coated glass substrates calcined at 300 0C.

Sample	**300 ^{0}C %T (300-1500nm)**
Blank side	76.09
Glass FTO -1mm	81.42
Glass FTO -2mm	84.34
Glass FTO -3mm	85.03
Glass FTO -4mm	86.61

The thickness at 1mm/s and 4 mm/s are 35 nm and 75 nm. The results are measured using mathematical model Cauchy. The experimental curves and modelled curves exactly coincided which indicate the mathematical model exactly suitable for measurement of thicknesses by using ellipsometer. The coatings didn't show any conductivity when measured through multimeter.

To study the influence of curing temperature, theglass substrates are dip coated with FTO Sol at different withdrawal speeds 1, 2,3 and 4 mm/s and further heat treated at 500^{0}C for 4 h in air. The rate of heating is 5^{0}C/min and furnace cooled.

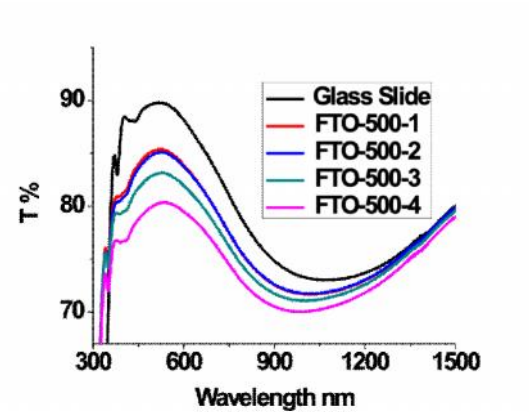

Figure Glass Slide FTO coated at 500^{0}C.

Table The transmittance values for FTO coated glass substrates calcined at 500 0C.

Sample	**500 ^{0}C %T (300-1500nm)**
Glass FTO -1mm	75.58
Glass FTO -2mm	78.63
Glass FTO -3mm	79.52
Glass FTO -4mm	80.93

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It is observed that compared to 300 0C, heat treatment at 500^0C showed lower transmittance.

Table The transmittance values for FTO coated glass substrates calcined 500^0C. The thickness of 5 mm/s glass substrate came around 85 nm. The thickness of the coating on glass substrates at 2 mm/s and 3 mm/s are measured using Ellipsometer.

2 mm/s - FTO - 500^0C: Thickness 474.13 A^0 , Refractive Index 1.7214

3 mm/s - FTO - 500^0C: Thickness 618.61 A^0 , Refractive Index 1.7211

These coated glass slides after heat treatment at 500 0Cshowed high transparency but didn't show any conductivity when measured with multimeter.

4. Conclusion

The non-detection of conductivity of TCO films at 300 0C and 500 0C can be due to non-uniformity of the film at microscopic level though visually they are uniform. It can be due to limitation of multimeter to detect low conductivity. So it is proposed to conduct measurements using four probe conductivity meter. It can also be due to Na+ ions presence in glass substrates. So it is proposed to conduct the experiments with quartz substrates.

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