CHARACTERIZATION OF DOPED AND UNDOPED TIN OXIDE THIN FILMS PREPARED BY SOL GEL SPIN COATING TECHNIQUE

Dissertation Submitted

By

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In partial fulfillment of requirement for the

Award of the Degree of

M.Phil Physics

Supervised By

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April, 2017

CERTIFICATE

This is to certify that Amandeep Kaur has completed the M.Phil dissertation titled "Characterization of doped and undoped Tin Oxide thin films prepared by Sol Gel Spin Coating Technique" under my guidance and supervision. To the best of my knowledge, the present work is the result of her original investigation and study. No part of the dissertation has ever been submitted for any other degree or diploma at any University.

The dissertation is fit for the submission and the partial fulfillment of the conditions for the award of M.Phil Physics.

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DECLARATION

I hereby declare that the dissertation entitled **"CHARACTERIZATIONS OF DOPED AND UNDOPED TIN OXIDE THIN FILMS PREPARED BY SOL GEL SPIN COATING TECHNIQUE"** submitted for the M.Phil Degree is my original work and all ideas and references have been duly acknowledged. It does not contain any work for the award of any other degree or diploma at any other University.

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(Amandeep Kaur)

ABSTRACT

Optical and structural characterizations of the Cu-doped and pure SnO2 thin films were done as a part of these investigations. In this respect, the effects of changing the concentration of the precursors on the thin film properties were investigated.. SnO2 thin films both in pure and doped form were prepared by a sol gel spin coating technique. The metallic oxide (SnO2) films deposited were characterized using the UV Spectrophotometer and XRD studies. The average band gap for tin oxide thin films was found to be approximately 3.80eV. As for as direct band gap is concerned that the Cu doping appears to have no effect on the direct band gap which is in accordance with as reported in the literature. The indirect band gap for thin films was found to be approximately 3.48eV, 3.32eV and 3.20eV for different concentrations of the solutions respectively. The indirect energy gap of the Cu doped thin films decreases as 3.76 eV, 3.73 eV and 3.16eV respectively for different concentrations of the precursor solutions. The less value may be due to the addition of Cu- dopant and also may be due to growth of grain and improvement of the degree of crystallization.

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CHAPTER-1

THIN FILMS

1.1 INTRODUCTION

Semiconductor materials have many essential properties related to optics, electronics and magnetism. So the study of these semiconductor materials has great importance. Tin oxide (n-type semiconductor) has extremely good photoelectric properties. These semiconductor materials are used as gas sensing materials for the reduction of gases. If the grain size of the tin oxide thin films is very small, preferably less than the Debye length, then the sensitivity of the sensor are significantly increased. In order to have the better response of optical and electrical devices, the development of metal oxide semiconductor materials having optical and non linear optical properties is essential. In general, light is emitted from oxygen vacancies in oxides in luminescence process. These vacant oxygen sites are named as radiative centers and are responsible for the emission of light incident on the oxides. In order to study the optical properties of tin oxides such as luminescence, it is important to know about the participation of oxygen vacancies in this process [1].

Tin oxide thin films have excellent transparency is due to greater energy difference between valence band and conduction band. Free electrons in the vacant positions of oxides are responsible for high conductivity. [2]

The properties of thin films are greatly influenced by the structure of the crystal. Thus these properties are dependent on the method used for the preparation of thin films and also depend on the variables included in the preparation technique. Sol gel spin coating technique is the best technique for controlling the amount of dopant to prepare thin films. In this process tin is used as the starting material and low concentration of tin is used. The important parameters involved in the sol gel spin coating technique are the amount of solute, temperature of heating and rate and time for spinning process. These parameters help to obtain the better optical and electrical properties of thin films. For the better quality of devices, these parameters are being controlled. [3]

Transition metals exhibit more than one oxidation states. Compounds of most of the transition metals can be converted into one another between oxidation states so these transition metal compounds are used to increase the rate of the chemical reaction. These transition metal compounds act as catalysts that exist in same phase as the reactants. Electrons can be interchanged between reactants with the help of catalyst as intermediary. Transition metals are

used in enzymes for binding the substrate molecules and also responsible for the chemical reaction. The main requirement in the electro catalysis is that the transition metal catalyst should be changed into appropriate host material for the fabrication of thin film electrodes. Also the arrangement, type and ratio of atoms in molecules in chemical substances of the resultant thin films should be of the same kind. An electrode can have the ability to be act as catalytic. Because the addition of doping material is responsible for the change in electrical conductivity, transparency and energy band gaps. The preparation of thin film electrodes using tetravalent tin oxides is easier because of the low absorption, good electrical properties. Thus tetravalent tin oxides are used for the fabrication of thin film electrodes. [4]

The conduction level of tetravalent tin oxides can be raised to higher values due to the increase in n-type charge carriers because of the presence of doping agents such as Cl or Sb. The electrical properties of the thin films are affected by the deposition parameters such as thickness of the film and deposition temperature. In order to develop gas sensors having good sensitivity and reproducible properties, the deposition parameters should be absolutely controlled. There are many metal oxide electrodes available to be used in a wide variety of applications. The alloys of transition metals have great importance because their electronic structure. The rate of chemical reaction increases because of the electronic structure of the alloys of transition metals. The alloys of amorphous materials also have a strong catalysis because these alloys try to become same. The position of central atom, ions and molecules around the central atom is not constant. All these raise the rate of chemical reaction. A system of mixed metal oxides fabricated using tetravalent tin oxides helps in the completion of the degrading of phenol. The tetravalent tin oxides with Sb_2O_5 as doping agent are able to exhibit improved of catalytic process for phenol oxidation. In order to develop electrodes able to exhibit better catalytic property, the transition metal oxides are used as doping agent in tetravalent tin oxides. [4]

Tin oxide thin films increase the rate of chemical reaction. So these films act as best catalyst. Tin oxide thin films also have extremely good adhering properties to surfaces of objects. The optical and structural properties of tin oxide thin films can be changed by changing the doping material and concentration of precursor material. Thin films of oxides having homogeneity and reproducibility can be prepared by using RF-Sputtering method. These prepared thin films can be used for devices in industries. [5]

Sol gel process is preferred due to many favorable properties such as low temperature, controllable thickness. Also thin films prepared by this method are uniform and homogeneous. The thickness and transparency of thin films are greatly influenced by change in coating cycles. [6]

Tin oxide thin films with Cu doping have extensive applications due to their favorable optical and structural properties. The band gap of Fe and Mn doped tin oxide thin films prepared by vapor deposition technique was approximately 4.08eV. While the transmittance of tin oxide thin films was high for smaller amount of Sb dopant and low for larger amount of doping. Some changes in phase and size of the crystal were seen due to the addition of metal ions. So to obtain the best structural and optical properties of tin oxide thin films, Cu is preferred to be use as doping material. [9]

Tin oxide thin films have low electrical sheet resistance and greater area of surface. Due to these desirable properties, tin oxide thin films are used in various applications. In sol gel method, spin coating, dip coating and spray coating are used for the preparation of thin films. [10]

Due to excellent transparency of tin oxide thin films in the visible region; these thin films are used for semiconducting sensors for the detection of toxic gases. [11]

Tin oxide is the best material to be used as thin films because of its desirable properties. Tin oxide materials are more transparent. These types of materials have considerable conductance and reflectance. So these materials are preferred by the researchers for the formation of thin films. Sol gel method is the most suitable method for the preparation of thin films. By using this type of technique, thin films with greater uniformity and homogeneity over larger areas. It is more important to control the amount of material to be used as sol. Also the temperature of substrate has to be controlled. Annealing is the powerful process to achieve the fine crystalline properties. Thus, the structural and optoelectronic properties of tetravalent tin oxides are greatly influenced by the deposition temperature. [12]

Transparent conducting oxide based materials are used in both electrical and optical devices. A variety of thin film deposition methods are used for the fabrication of thin films of these types of materials. A transparent p-n junction is required for the fabrication of devices based on the transparent conducting oxides. But the stable p-type materials have less conductivity due to unfavorable electrical properties such as high hole mobility and hole density. In order to have better understanding of conductive properties for p type materials, further investigation is required. Tin oxide is the best material used for optical and electrical devices. Tin oxide materials are preferred for transparent devices applications because in the valence band and conduction band of tin oxides the density of 5s states and 5p states is high and delocalized. Tin oxide exists in +2 and +4 oxidation states. But the divalent state can be changed into tetravalent state by oxidation.SnO_2 exhibit rutile phase and orthorhombic phase depending upon the temperature and pressure. [13]

There are various deposition techniques for the preparation of tin oxide thin films as sputtering, hydrothermal deposition, pulsed laser deposition, electron beam evaporation, spray pyrolysis, ultrasound-assisted and microwave-assisted synthesis and sol gel methods. But sol gel spin

coating technique is preferred because it is a simple technique in which properties of the tin oxide thin films can be easily controlled. Ethanol, methanol and isopropanol solvents are to be used for the preparation of orthorhombic divalent and tetravalent tin oxide thin films. The properties of surface and crystal structure of the tin oxide thin films are studied by using X- ray diffraction and Atomic force microscopy. For the study of optical properties of tin oxide thin films, UV visible spectroscopy, Raman analysis, photoluminescence is used. [13]

SnO_2 is an n-type semiconductor having energy difference of 3.71eV between the energy levels of the valence band and conduction band .Due to this greater energy difference the large amount of light is transmitted in the visible spectrum. The vacant positions of oxygen help to produce electrical conductivity in a semiconducting material. The oxygen vacancies are n-type impurities which act as dopant in the semiconducting material. Due to all these favorable properties of tin oxide with doping and without doping, these are useful in research in the field of condensed matter physics. Tin (II) chloride is preferred as a precursor material because of its cost effectiveness**.** [14]

Thin films are used in different areas of technology and also in research due to their optoelectronic properties. Tin oxide thin films are useful due to their transparent and reflective nature for ultra violet and infrared light respectively. For sol gel spin coating technique of thin film preparation, a solution is prepared by using one of the three different solvents ethanol, methanol and isopropanol and by choosing tin((II) chloride as a precursor due to its cost effectiveness . Acetic acid is chosen to be used as a chelating agent. The prepared solution is deposited on a glass substrate. [14]

Tetravalent tin oxides have resistive nature for vapours, liquids and acids. Sol gel spin coating technique is also responsible for controlling the shape and structure of thin films. Thin films having different values of thickness are prepared. The optical and structural properties of thin films are greatly affected by varying the number of coating layers. The large variations have seen in the size of the crystal of thin films with the variation in the number of coated layers. [15]

The optoelectronic properties and the fine structure of tetravalent tin oxide thin films depend on the method used for the preparation of thin films. It has been proved that the nanostructure of sintered sample get removed due to the sintering of nanoparticles. The techniques having reasonable cost are expected in future for the composition of tetravalent tin oxide nanoparticles which have to be favorable microstructure. The arrangement, type and ratio of atoms in molecules of tetravalent tin oxide thin films can be controlled by sol gel technique for which minimum temperature is required. [16]

Tin oxide thin films are chemically and mechanically stable. Tin oxide thin films are used in gas sensors for sensing the toxic gases. With the increase in annealing temperature, tin oxide thin

films crystallize. As the thickness of thin film increases, the electrical sheet resistivity of thin film decreases. [17]

Tin oxides have rutile structure and also can be chemically changed in different shapes and sizes. Tin oxide thin films have large number of charge carriers which can exist freely. The vacant oxygen positions are responsible for it. Tin oxide nanoparticles can be successfully prepared by using sol gel process. In sol gel process, the particles of smaller size are dispersed in the liquid called sol and are converted into interconnected network of these small molecules to form gel. [18]

The defects in thin films depend on the environment in which thin films are prepared. These defects include the vacant positions of oxides. The physical properties of thin films depend on these defects. [19]

Thin films are needed to be highly transparent, electrically conductive, and stable at high temperature to be used in optoelectronic devices. Tin oxide is the best material to be used for optoelectronic devices. Tin oxide thin films are mechanically hard. The methodology is preferred depending upon the type of substrate and the material selected for the deposition. [20]

1.2 CLASSIFICATION OF THIN FILMS

Thin films can be classified into three basic categories:

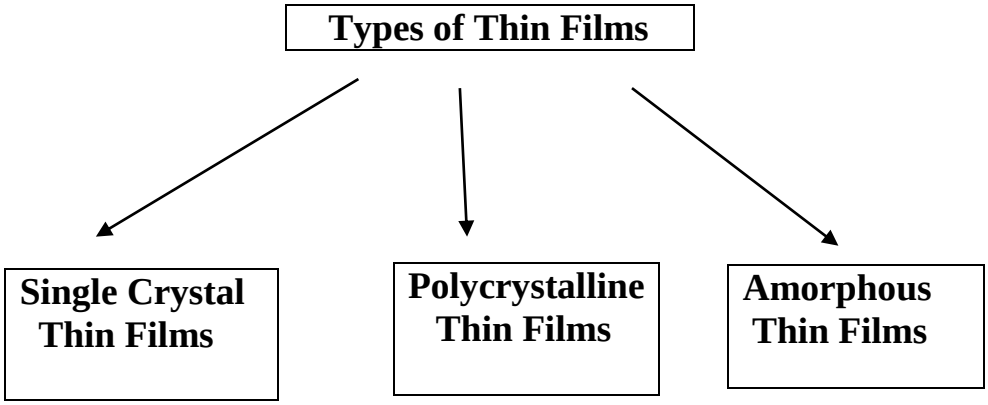

Fig 1.1 Classification of thin films

1.2.1 Single Crystal Thin Films

Single crystal thin films have regularity in crystal lattice absence of grain boundaries. The production of these types of thin films in the laboratory is complicated. Since the electrical and physical properties of these thin films are greatly influenced by the presence of grain boundaries so controlled conditions are the basic requirement for the production of these films. In order to

fabricate single crystal thin films, a clean glass substrate is needed. Also high temperature is the main requirement for the fabrication of these types of thin films for the mobility of growth species.

1.2.2 Polycrystalline Thin Films

The materials having alteration in the size and orientation of the crystals are required for the fabrication of polycrystalline thin films. Powder grains are produced due to the small polycrystalline grains. Polycrystalline thin films have grain boundaries between two grains. A moderate temperature of deposition is needed for the fabrication of these types of thin films. All the materials including common metals and ceramics form polycrystalline thin films.

1.2.3 Amorphous Thin Films

The deposited solid layers on the surface of substrates having thickness from few nanometers to micrometers are the amorphous phase of thin films. In these types of thin films, the positions of atoms are limited to small ranges. Low temperature is needed for the production of these types of thin films. There is no sufficient surface mobility of the growth species.

1.3 FABRICATION OF THIN FILMS

Thin films can be fabricated by depositing a thin film on to the surface of a substrate. This process of depositing thin films is referred to as thin film deposition. Thin films can be fabricated by using a variety of materials such as metals, semiconductors, insulators and dielectrics etc. The wide variety of deposition techniques is available for depositing the thin films. The classification of deposition techniques involves the nature of material used to be deposit thin film.

1.3.1 Thin Film Deposition By Using Liquid Materials

Spray pyrolysis and sol-gel deposition techniques are used for depositing thin films of liquid materials.

1.3.1.1 Spray Pyrolysis

This technique involves the deposition of metal oxides by spraying a solution of metal salt on the surface of a substrate. A chemical compound is formed due to the reaction of constituents. The processes may occur simultaneous or in a number of sequences in the spray pyrolysis. The aerosol generation and transport solvent evaporation are the significant processes. The aerosol generation process does not influenced by the deposition temperature. The temperature of substrate surface plays most significant role for the surface morphology and properties of the thin films.

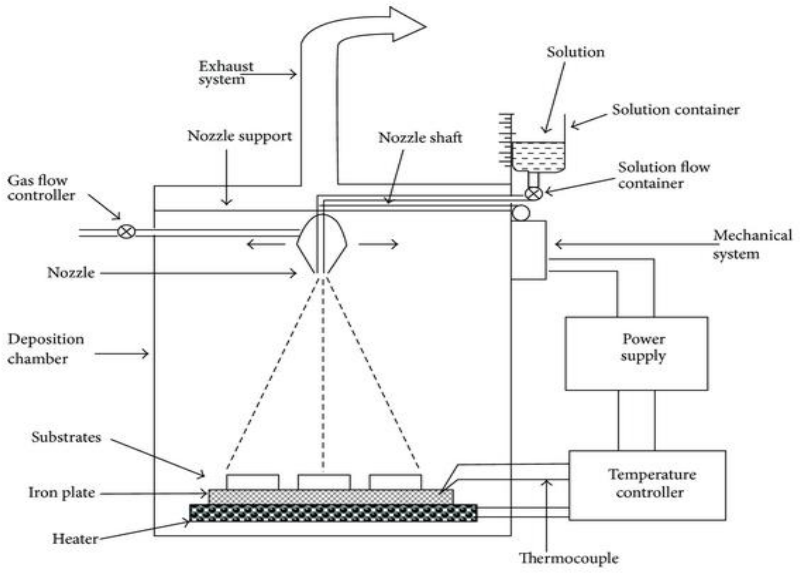

Fig 1.2 Spray Pyrolysis Technique For Thin Films Deposition Of Liquid Materials [21]

1.3.1.2 Sol-Gel Method

Sol-Gel method involves a process in which solid particles are dispersed in the liquid. The particles having size from 0.1µm to 1 µm are suspended due to the Brownian motions. A gel involves the dispersion of solid and liquid that represents a solid network having liquid components. The sol-gel technique of depositing thin films involves the following steps:

(1) A sol is a dispersion of solid particles in a liquid.

(2) The sol can be deposited on the substrates by spraying, dipping, spinning and drop casting methods.

(3) The particles in a sol combined to form a polymer and are responsible for the production of gel.

(4) Finally, the amorphous or crystalline deposition layer is obtained heat treatment. This decomposes the organic and inorganic components.

1.3.1.2.1 Dip Coating

The dip coating involves the immerging and withdrawing processes of any material or substance into a liquid. This process results in deposition of a layer of material. Thin film prepared by using dip coating technique depends upon various factors. Thin films having a large number of repeated structure and thickness can be obtained by adjusting the parameters such as withdrawal speed, composition of solution, number of dipping cycles, temperature and concentration. Thin films prepared by dip coating technique have uniformity and excellent quality for bulky and complex shapes.

1.3.1.2.2 Spin Coating

Thin films with excellent uniformity can be deposited on the surface of substrates by using spin coating technique. In order to deposit thin films, substrates are coated by applying a small amount of coating material. The substrate rotating at higher speeds is responsible for the spreading of coating material. The machine used for this process is named as spin coater or simply spinner. As the angular speed of spinning increases, the thickness of the film also decreases. The film thickness also influenced by the viscosity and concentration of solution and solvent.

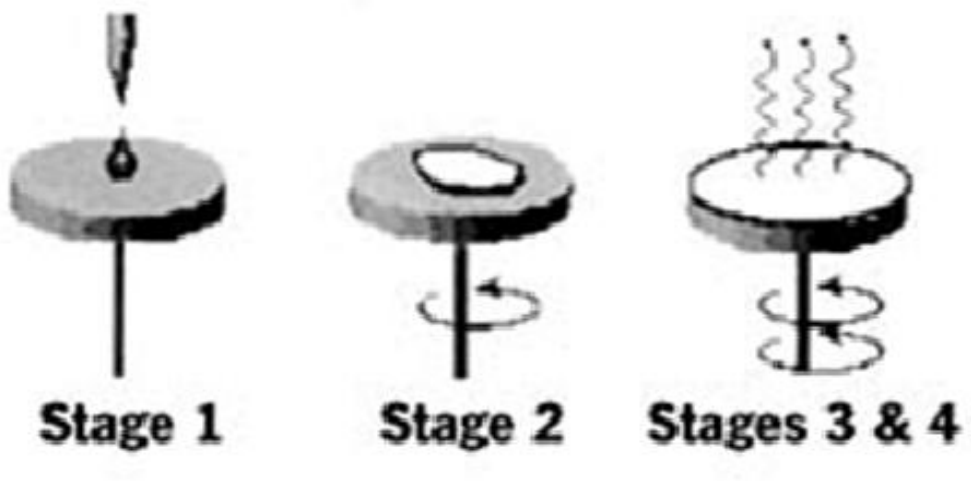

Fig 1.3 Spin Coating Technique For Depositing Thin Films of Liquid Materials [22]

1.3.1.2.3 Drop Casting

Drop casting technique requires stable, specific pressure, temperature and flow rate for depsiting thin films onto the substrates. Thin films are prepared by applying 100-200µg of sol per square millimeter over a width of several feet and lengths of many thousands of feet.

1.3.2 Deposition of Thin Films Using Gaseous Reagents or Vapor Deposition

Vapor deposition is the process in which the material in vapor phase condenses to a solid material. Thus the properties of the substance such as thermal, electrical and mechanical properties can be varied by using this type of method. A vacuum chamber is required in the vapor deposition process.

There are mainly two vapor deposition processes:

1.3.2.1 Chemical Vapor Deposition

This type of deposition technique is used to fabricate pure solid material thin films. The substrate is placed in the pressurized chamber filled with organometallic gas such as silicon dioxide, polysilicon, and silicon nitride. Thin film is deposited due to the reaction of gas with the surface of the substrate. The various materials such as silicon, carbon nanofibres, carbon fibre, carbon nanotubes are deposited by using this technique. And all these results in different forms like monocrystalline, polycrystalline and amorphous.

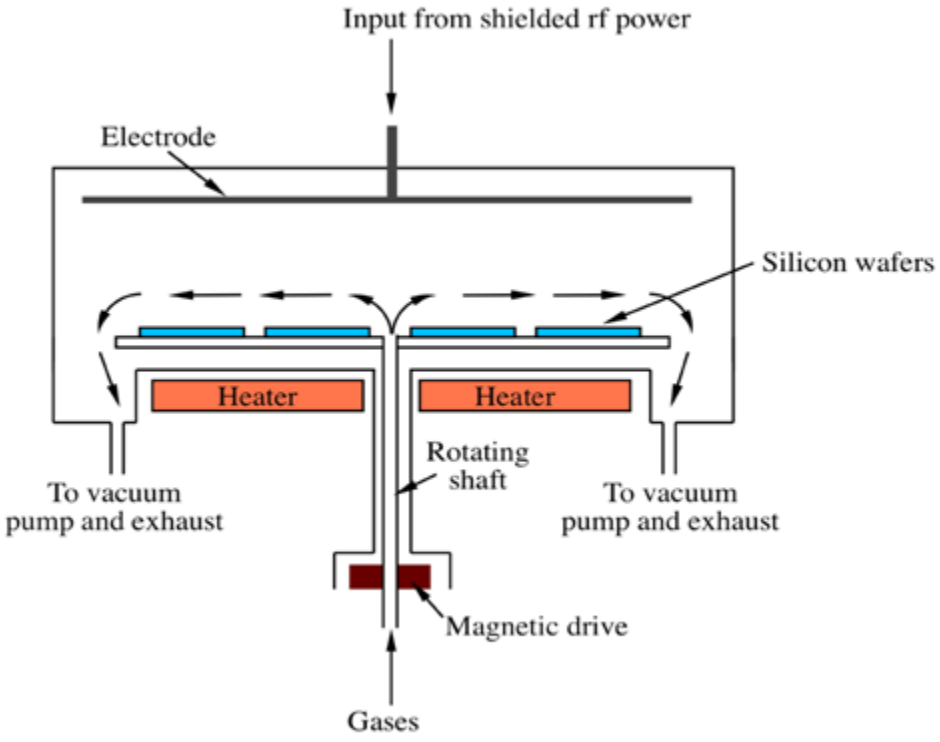

Fig 1.4 Chemical Vapor Deposition Technique For Depositing Thin Films of Gaseous Materials [23]

1.3.2.2 Physical Vapor Deposition

Physical vapor deposition process is used to deposit thin films of thickness from few nanometers to several micrometers. Physical vapor deposition technique involves three basic steps:

1. High temperature vacuum or gaseous plasma is required for vaporizing the solid material.

2. Transportation of the vapor in vacuum or partial vacuum to the substrate surface.

3. Materials are condensed on to the surface of substrates for the preparation of thin films.

The main physical vapor deposition techniques are sputtering and thermal evaporation. In thermal evaporation technique, the source material is vaporized by heating the source material. In sputtering the source material is vaporized by the bombardment of the material using high energy particles or ions. In both these techniques, the vapors of the material condense on the surface of the substrate and deposit there.

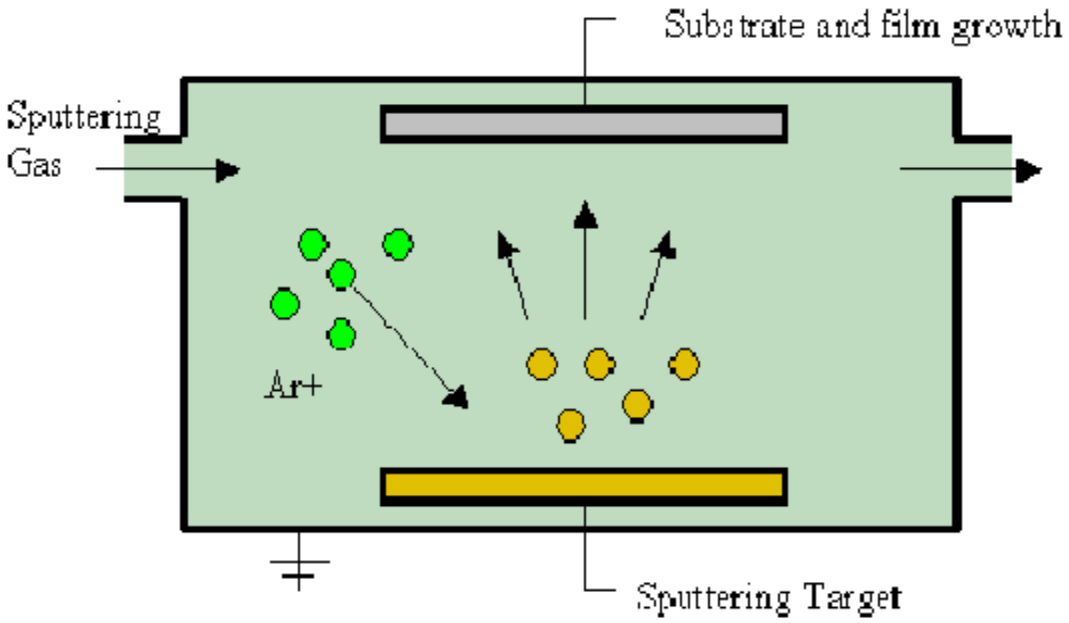

Fig 1.5 Physical Vapor Deposition Technique For Depositing Thin Films of Gaseous Materials [24]

1.4 CHARACTERIZATION OF THIN FILMS

Thin films of materials are characterized by using the following techniques:

1.4.1 X-Ray Diffraction

About 95% of all solid materials can be described as crystalline. The interaction of X-rays with a crystalline material results a diffraction pattern. The polycrystalline phases are characterized and identified by using powder diffraction method. The powder diffraction method is mainly used for identifying the components in a sample by a match procedure. Also there is a relation between the area under peak and amount of various phases in a sample. The concepts of X-Ray Diffraction by crystal structure and light diffraction by a ruled grating are same. There is equality between the angle of incident and angle of reflection. According to Bragg's law, the angle and wavelength of X-Ray beam are related as

$$\lambda = 2d\sin\theta$$

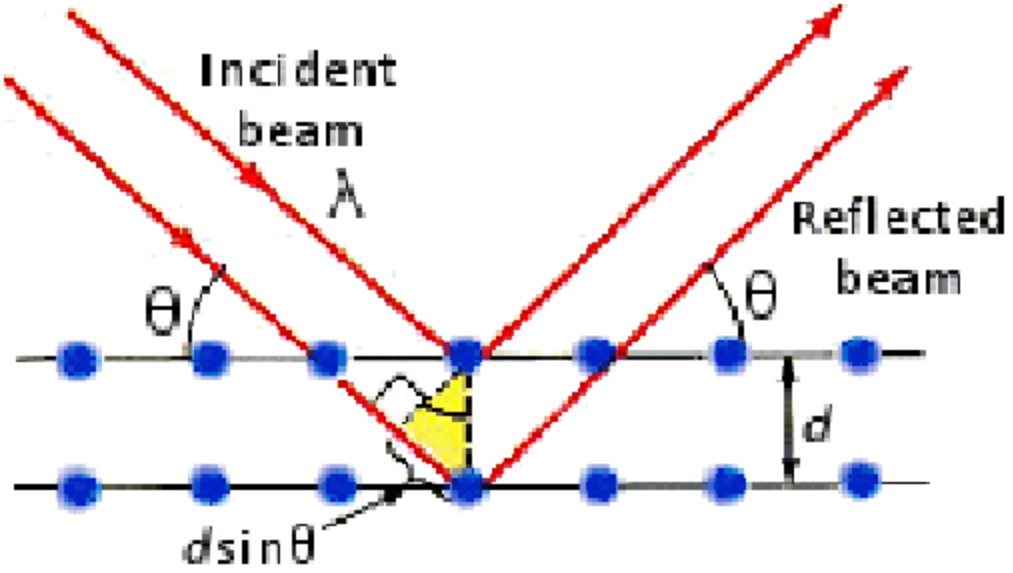

Fig 1.6 The diagram shows Bragg's Law For X-Ray Diffraction [25]

The following are the applications of X-Ray Diffraction

- XRD is a nondestructive technique
- For the identification of crystalline phases and orientation
- The structural properties such as lattice parameters, grain size, strain, phase composition and thermal expansion can be determined by XRD
- The thickness of thin films can be measured
- Arrangement of atoms can be identified

1.4.2 Ultraviolet-Visible Spectroscopy (UV-VIS Spectroscopy)

UV-VIS spectroscopy is used to characterize the optical properties such as absorption, transmission of thin films. Also the energy band gap and refractive index of thin films

can be measured by using UV-VIS spectroscopy. This technique also provides the qualitative and quantitative data of thin films.

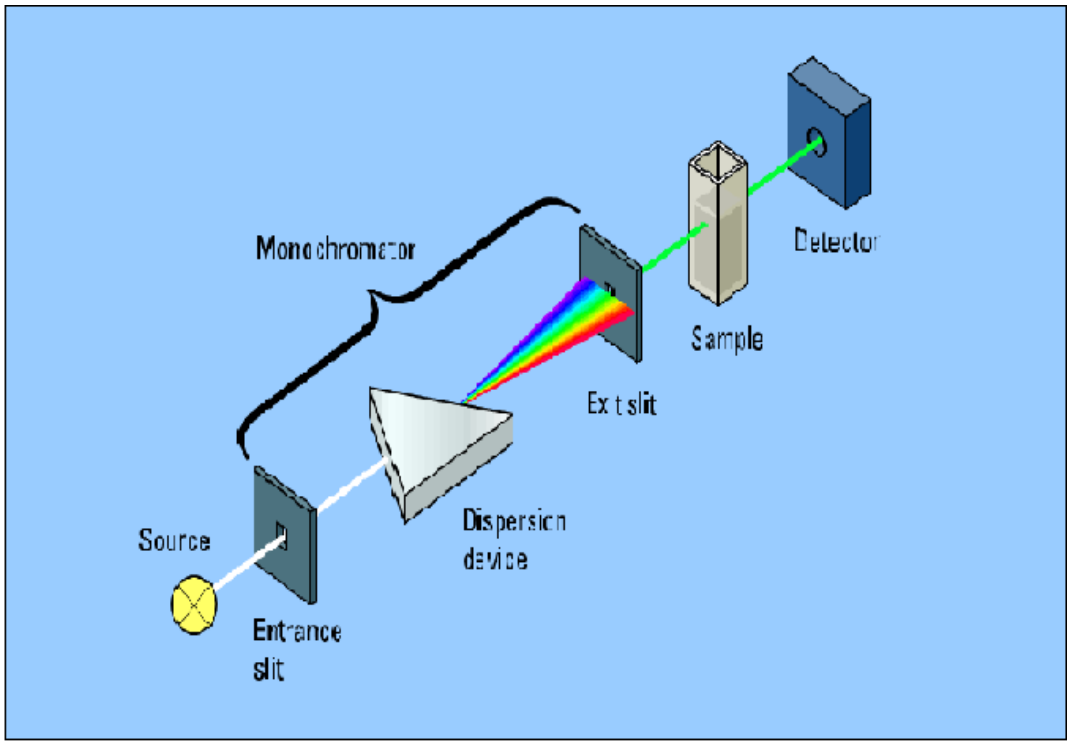

Fig 1.7 UV-VIS Spectroscopy For Determining Optical Properties of Thin Films [26]

1.5 OBJECTIVES

The main objectives of the study are:

a) Preparation of Cu doped and undoped tin oxide thin films by using Sol gel spin coating technique.

b) To determine the best structural and optical properties of the prepared thin films for various applications by XRD studies and UV-Visible spectroscopy.

c) Use of simple and cost effective technique such as sol gel spin coating for the preparation of thin films.

d) To stop UV light and admit visible light so that the homes do not get warm.

e) To prepare thin films for antireflective coatings.

f) To prepare thin films for optoelectronic applications.

CHAPTER-2

REVIEW OF LITERATURE

Feng Gu et al. (2004) had prepared tin oxide thin films by using sol gel spin coating technique. The prepared thin films were deposited on glass substrates. The surface properties of thin films were characterized by using Atomic force microscopy and X-ray diffraction methods. The various spectroscopic techniques such as UV visible spectroscopy and photoluminescence spectroscopy were used for the study of optical properties of thin films. From optical characterization, it was concluded that there were different maxima for emission. One maximum at higher wavelength was associated with the vacant positions of oxygen in tetravalent tin oxide thin films. However the maxima at lower wavelength were associated with the absence of tin atoms from the lattice positions. The energy difference between the valence band and conduction band of tin oxide was found to be 4.38 eV.

Seung-Chul Leen, Jae-Ho Lee, Tae-Sung Oh et al (2003) prepared tin oxide thin films by using sol gel spin coating and dip coating technique. Tin isopropoxide was used as the starting material and isopropyl alcohol was used as solvent. The thickness of thin films was varied by increasing the coating cycles. The prepared thin films had excellent transmittance and lower resistivity.

E. Savarimuthu et al. (2006) preferred sol gel spin coating technique for the preparation of tin oxide thin films. Many parameters such as amount of the solute to be used as starting material, rate and time of spinning process, temperature for heating the thin films and the deposition cycles had great effect on the thin film properties. The structural properties of prepared films were studied by XRD method, Scanning Electron Microscopy and Atomic Force Microscopy. Structural analysis showed that the films were consisted of many crystalline parts randomly oriented with respect to each other. Optical properties were characterized by using UV visible spectroscopy. The films prepared at minimum temperature had better transparency, low resistance and band gap 3.46eV.

P.G.L. Baker et al. (2007) had used the sol gel spin coating technique for the preparation of transition metal oxide thin films. The prepared thin films were deposited on the solid titanium

substrates. The structure properties of prepared metal tin oxide were studied by using Atomic force microscopy and Scanning electron microscopy. Also the arrangement, type and ratio of atoms in molecules of chemical substances were taken into account. The energy difference between the valence band and conduction band of thin films was determined by UV visible spectroscopy of the prepared thin films. Higher values of temperature were required for the stabilization of all the transition metal oxides except palladium tin oxide catalyst. UV visible spectroscopy of thin films concluded that there is no effect on the energy difference between the valance band and conduction band of tin oxides due to the addition of any doping agent. Atomic force microscopy of thin films concluded that there are very small holes on the surface of thin films. Thin films with plane surface were prepared by using spin coating technique.

S. Saipriya, M. Sultan, R. Singh (2011) used RF Magnetron Sputtering technique for the deposition of tin oxide thin films on quartz substrates in the presence of Ar and O_2. The structural characterization of tin oxide thin films was carried out by using X-Ray Diffraction analysis. It was found that the prepared thin films had amorphous nature. From the optical properties, the energy band gap of thin films was found out to be from 4.15eV to 4.3eV due to lowering the Ar gas content. Also the energy band gap of thin films varied due to annealing of the film.

Sumanta Kumar Tripathy, Bhabani Prasad Hota, P V Rajeswari (2012) prepared tin oxide thin films by using sol gel dip coating technique. Tin chloride and methanol was used as the starting material and solvent respectively. The optical properties were studied by UV visible spectroscopy. The thickness of thin films was determined from the transmission spectrum. It was found that the thickness of thin films was increased with the increase in coating cycles whereas the transmittance decreased.

Sumanta Kumar Tripathy et al. (2013) characterized the structural and optical properties of Cu doped tin oxide thin films prepared by thermal evaporation technique. The optical properties were studied by UV-Visible spectroscopy and resulted that the thin films were highly transparent for wavelength greater than 450nm. The optical band gap was about 3.55eV. The structural properties were characterized by XRD method and Scanning Electron Microscopy.XRD analysis showed that the structure of thin films was tetragonal rutile and SEM analysis resulted that thin films were consisted of many crystalline parts randomly oriented with respect to each other and some peaks were also seen.

Sumanta Kumar Tripathy, Bhabani Prasad Hota, P V Rajeswari (2013) preferred sol gel dip coating technique for the preparation of tin oxide thin films. Tin chloride and methanol was used as the starting material and solvent respectively. Optical properties of the prepared thin films were studied by UV Visible spectroscopy. Influence of coatings on the transmittance and refractive index of thin films were studied. With the increase in number of coating cycles, there was increase in transmittance whereas refractive index of thin films was decreased. The

structural properties of prepared thin films were studied by X-ray diffraction analysis. The crystalline phases occurred due to increase in number of coatings. The surface characterization was carried out by using Scanning Electron Microscopy. The thin films were found to be rough. The prepared thin films had tetragonal rutile structure.[13]

Sumanta Kumar Tripathy, R. Prabeena, V. Siva Jahnavi et al (2013) preferred thermal evaporation and sol gel techniques for the preparation of tin oxide thin films. The prepared thin films were used for the detection of gases. The structural properties of the prepared thin films were studied by X-Ray Diffraction analysis using SIEMENS diffractometer (Model) D 5000. The surface characterization of thin films was carried out by Scanning Electron Microscopy. The prepared thin films had tetragonal rutile structure. The optical properties such as thickness, refractive index and energy difference between valence and conduction bands of thin films were determined by UV-Visible Spectroscopy. The prepared thin films were more sensitive for CO gas at 200^0c and 235^0c for thermal evaporation and sol gel technique respectively.

B. Yarmand (2014) had examined the influence of temperature on the thin film properties such as transmittance, reflectance, electrical conductance and structural properties. Sol gel spin coating technique was used to deposit tin oxide thin films on the glass substrates. Optical properties were studied by UV-visible spectroscopy and Fourier Transform Infrared (FTIR) techniques. X- Ray Diffraction (XRD) and Scanning Electron Microscopy was preferred for the analysis of surface and structural properties. XRD analysis showed the improvement in the crystallization by increasing the temperature. Scanning electron microscopy of thin films showed the uniformity of thin films. There were small rough films without holes and breaks on the surface. As the temperature of deposits increased there was decrease in the band gap. Also with the increase in deposition temperature the sheet resistance was decreased.

M. Marikkannan et al. (2015) used tin oxides for the preparation of thin films. The prepared thin films were deposited on the glass substrates by using the sol gel spin coating technique. The prepared tin oxide thin films exhibit orthorhombic structure which was not stable. For the preparation of tin oxide thin films, $SnCl_2.\ 2H_2O$ and different samples such as SnO-E_tOH, SnO-MeOH and SnO-IPA were used. The techniques such as X-ray diffraction and Atomic force microscopy were used for the study of surface properties of the thin films. The optical properties of the prepared thin films were studied by using different techniques such as UV-vis spectroscopy and photoluminescence. The characterization results for structure properties showed that there are different diffraction peaks for the three samples which exhibit the orthorhombic structure of divalent tin oxide and tetravalent tin oxides. The orthorhombic structure of divalent tin oxide and tetravalent tin oxide thin films became stable by selecting methanol and isopropanol as the starting material for the preparation of thin films. Also large amount of light was absorbed and transmitted from thin films prepared by using these solvents. It had been seen that divalent tin oxide thin films have very small two dimensional defects in the

structure. From all these results, it was concluded that sol gel spin coating technique is most favorable for the preparation of tin oxide thin films. Further investigation is required to study the electrical properties of thin films so that these become able to be used in the transparent conducting oxide devices used for many applications.

Jonathan O. Ajayi et al. (2015) studied the structural and optical properties of tin oxide thin films by using the sol gel spin coating technique. Tin oxide thin films were prepared by using tin chloride and acetic acid. Methanol was used as solvent. Thin films were also affected by the concentrations of these materials. The structural properties of thin films were studied by scanning force microscopy. UV visible spectroscopy was used for the study of optical properties. The prepared thin films were less capability for absorbing the light. However the large amount of light was transmitted by thin films. The energy gap between the valance band and conduction band was broad. So these thin films were acceptable for optical and electrical devices. With the decreased concentration of materials, the reflectivity also decreases.

Mohamed Shaban et al. (2015) preferred the sol gel spin coating technique for the production of tin oxide thin films with different thicknesses. The thin films with different thicknesses were deposited on the glass substrates. Ethanol and tin chloride dihydroxide materials were used for the preparation of thin films. The properties related to structure of prepared thin films were studied by using X- ray diffraction method and Scanning electron microscopy. The difference between the energies of the valance band and conduction band was dropped. The variations in the size of the crystals of tin oxide thin films were seen due to varying the number of coating layers thin films. The surface and optical properties of the thin films were affected by the varied coating layers. There was increment in the size of the crystals as the number of coating layers increased. With the increased thickness of the film, less amount of light was transmitted. The localized density increased due to the enlarged number of coating layers. The films were preferred for anti reflection coatings to reduce the reflection.

G. S. Shahane (2015) had studied the optical, structural and surface properties of nanocrystalline tin oxide thin films. Sol gel spin coating technique was used to study all these properties. Thin films prepared by using this technique were thin in thickness and there was uniformity in thin films. The size of the crystals was 7nm showed by XRD analysis. Scanning Electron Microscopy showed that there were small holes on the thin films and small particles were also present. There was increment in the area of the surface of thin film due to these holes and grains. The band gap for tetravalent tin oxide thin film was 3.96eV.

]Imad H. Kadhim ,H. Abu Hassan (2015) prepared nanocrystalline tin oxide thin films by using sol gel spin coating technique on Si substrates. The structural analysis was carried out by X-Ray Diffraction and Field Emission Scanning Electron Microscopy. It was found that tin oxide thin films were crystallized at 400^0c. With the increase in annealing temperature, the

crystallization of tin oxide thin films was improved. Also the size of the crystals increased. For the fabrication of nanocrystalline thin films, 500^0c annealing temperature was preferred.

Ashok D. Bhagwat, Sachin S. Sawant, Bal aprasad G et al. (2015) used sol gel method for the fabrication of nanocrystalline tin oxide powder. The structural and optical characterization of tin oxide powder was carried out by X-Ray Diffraction analysis, Scanning Electron Microscopy and UV-Visible Spectroscopy respectively. From the structural studies, it was found that the tin oxide powder samples have rutile structure and some grains were uniformly distributed. The energy band gap was found to be 3.78eV.

S SujathaLekshmy, I John Berlin, L V Maneeshya et al. (2015) used sol gel dip coating technique for depositing tin oxide thin films on quartz substrates. The structural and surface characterization was carried out by XRD analysis and Scanning Electron Microscopy respectively. From these studies, it was found that thin films had amorphous nature due to annealing in air whereas in oxygen, thin films were crystalline. The band gap of thin films was raised due to annealing of thin films in oxygen. The prepared tin oxide thin films were found to be useful in transistors.

Sunday S. Oluyamo, David B. Agunbiade (2016) prepared tin oxide thin films by using spin coating technique. Optical properties were characterized by UV-Visible spectroscopy and obtained the results for transparency and reflectivity of deposited thin films. While the surface properties were studied by Scanning Electron Microscopy and concluded the presence of group small ring shaped particles. Thin films prepared by spin coating technique were highly transparent and also had band gap of approximately 3.78eV.

CHAPTER-3

EXPERIMENTAL DETAILS

This chapter deals with the experimental details for the preparation of undoped and Cu-doped tin oxide thin films deposited by using sol gel spin coating technique.

3.1 STANNIC OXIDE (SnO_2)

Tin oxide is the magnificent material because its desirable properties can be tailored by doping of materials for various applications.

Another name for tin oxide is stannic oxide. The IUPAC name for tin oxide is tin (IV) oxide. Tin oxide has white, off-white and grey crystalline solids. The formula for this material is SnO_2. The mineral form of this material is calledcassiterite. The band gap for tin oxide is 3.6eV. The refractive index and density of this material is 2.006 and 6.90-7.00g/cm^3 respectively. Tin oxide has rutile structure. The molar mass of tin oxide is 150.7g/mol. The melting and boiling temperatures are 1630^{0}C and 2500^{0}C respectively.

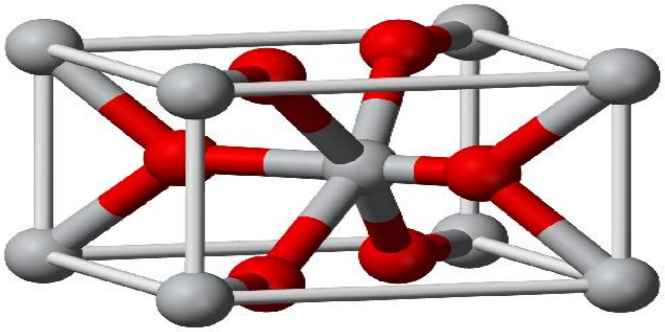

Fig 3.1 Structure of Tin Oxide [27]

Red ball represents tin atom and grey ball represents oxygen atom.

3.2 APPLICATIONS OF TIN OXIDE THIN FILMS

Tin oxide thin films are used in a wide variety of applications because of their excellent optical and electrical properties. Tin oxide is an n-type semiconductor material used as gas sensing materials for the reduction of gases. . The preparation of thin film electrodes using tetravalent tin oxides is easier because of the low absorption, good electrical properties. Thus tetravalent tin oxides are used for the fabrication of thin film electrodes. Tin oxide materials are more

transparent. These types of materials have considerable conductance and reflectance. So these materials are preferred by the researchers for the formation of thin films. Thin films are used in different areas of technology and also in research. Tin oxide thin films are also used in Flat panel displays, solar cells, antireflective coatings and many optoelectronic devices.

3.3 EXPERIMENTAL METHODOLOGY

3.3.1 Preparation of Sol-Gel

Tin Chloride Dihydrate ($SnO_2.2H_2O$) is used as the precursor for the preparation of sol gel. Methanol and acetic acid glacial are used as the solvent and chelating agent for dissolving the precursor material in a beaker. The solution is prepared for different concentrations 0.2M, 0.4M and 0.6M respectively. For the preparation of Cu-doped tin oxide thin films, 2.5%, 3.5% and 5% of copper acetate is taken. The stirring of prepared solution is done at room temperature for by using magnetic stirrer in order to achieve the transparent and homogeneous solution. Then heat the solution for 1 hour and then cool it for 20-25 minutes for achieving the sol gel.

3.3.2 Preparation of Undoped Tin Oxide Thin Films

The glass substrates are cleaned with cleaning soap, distilled water and acetone in order to remove the solution organic impurities. The cleaned glass substrates are dried in a furnace at 200^0 C for 10 minutes. After drying, the glass substrates are used for depositing the films from the prepared solution. The films were deposited by the use of a spin coater (Apex instruments make) model no. Spin NXG-P1. The thin films are deposited on the glass substrates by applying 1ml of the solution on the surface of glass substrates. The films are deposited with the spin coater having spin duration for each cycle of 60 s, Spin speed of 3000rpm and acceleration of $2m/s^2$.

3.3.3 Preparation of Cu-doped Tin Oxide Thin Films

The procedure for preparation of Cu-doped tin oxide thin films is same as for undoped tin oxide thin films. The glass substrates are cleaned with cleaning soap, distilled water and acetone in order to remove the solution organic impurities. The cleaned glass substrates are dried in a furnace at 200^0 C for 10 minutes. After drying, the glass substrates are used for depositing the films from the prepared solution having Copper acetate as a dopant. The films were deposited by the use of a spin coater (Apex instruments make) model no. Spin NXG-P1. The thin films are deposited on the glass substrates by applying 1ml of the solution on the surface of glass 60 s, Spin speed of 3000rpm and acceleration of $2m/s^2$.

3.1 CHARACTERIZATION OF THIN FILMS

The optical properties of thin films have been studied from UV spectrographs from a UV-visible spectrophotometer (Model no. UV-1800 SHIMADZU) in the wavelength range of 200- 800nm. The structural properties were analyzed by using X-Ray Diffraction (XRD) spectra using Cu Kα (wavelength = 1.5405 Å) radiation (model no. X′ Pert PRO, PANalytical Netherland).

UV-VIS SPECTROSCOPY

Fig 3.2 shows the UV-visible spectrophotometer (UV-1800 SHIMADZU) [28]

X-RAY DIFFRACTION

Fig 3.3 The above diagram shows the X′ Pert PRO, PANalytical apparatus for obtaining XRD patterns [29]

CHAPTER-4

RESULTS AND DISCUSSIONS

4.1 OPTICAL PROPERTIES

Absorption Spectra

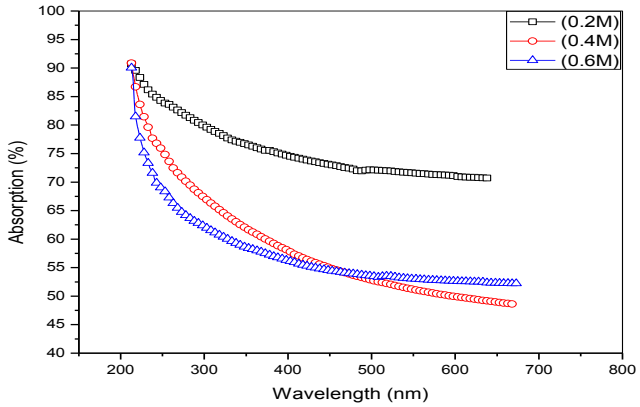

Fig.4.1 The Absorbance Spectra for SnO_2 thin films as a function of wavelength

Transmittance Spectra

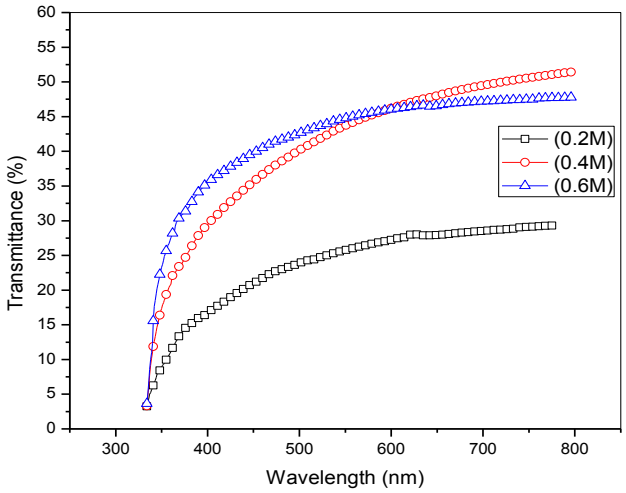

Fig.4.2 The Transmittance Spectra for SnO_2 thin films as a function of wavelength

Absorption Coefficient

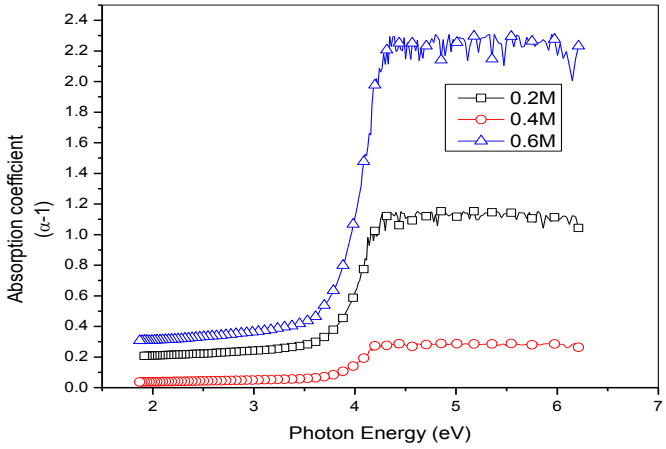

Fig. 4.3 The Absorption Coefficient of SnO_2 thin films as a function of Photon Energy

Direct Band gap

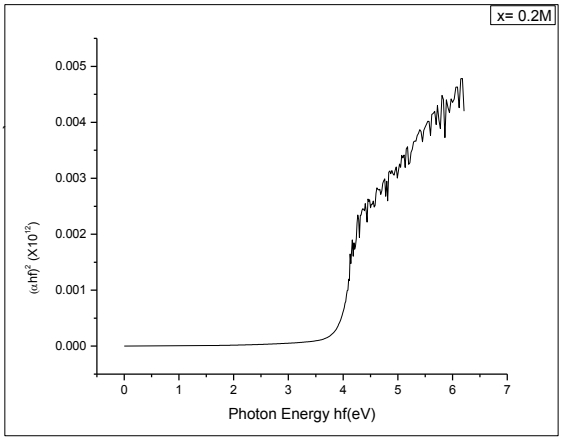

Fig. 4.4 The Optical Energy Gap for the Direct Allowed Transition of SnO2 films for the sample prepared by of 0.2 M concentration

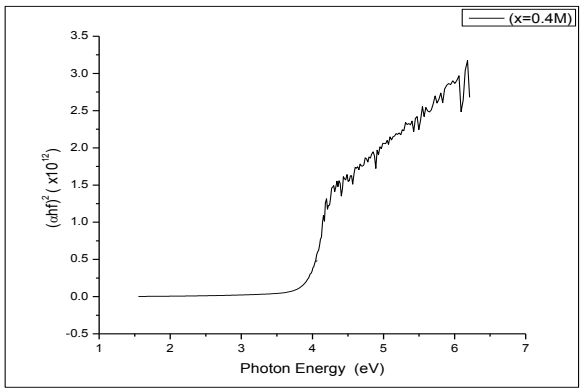

Fig. 4.5 The Optical Energy Gap for the Direct Allowed Transition of SnO2 films for the sample prepared by of 0.4 M concentration

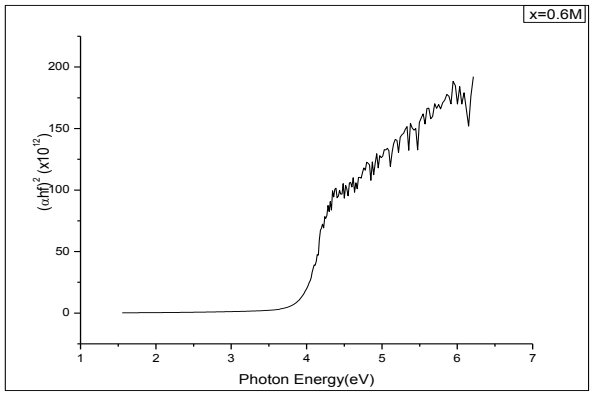

Fig. 4.6 The Optical Energy Gap for the Direct Allowed Transition of SnO2 films for the sample prepared by of 0.6M concentration

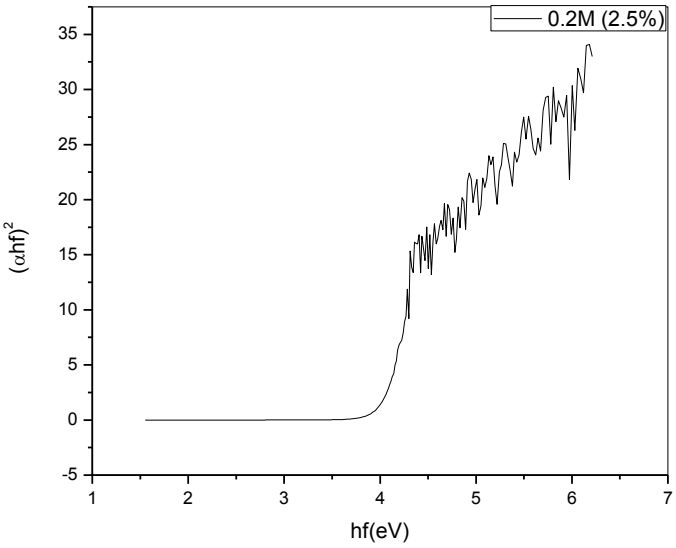

Fig. 4.7 The Optical Energy Gap for the Direct Allowed Transition of Cu-doped SnO2 films for the sample prepared by of 0.2M (2.5% Copper acetate doping) concentration

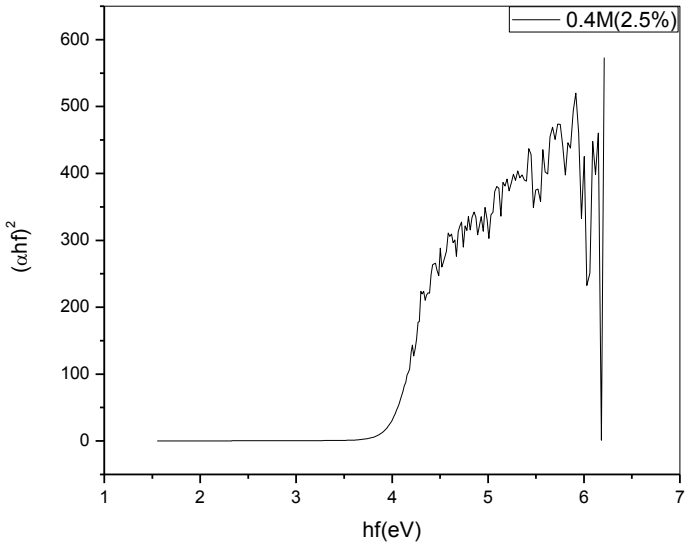

Fig. 4.8 The Optical Energy Gap for the Direct Allowed Transition of Cu-doped SnO2 films for the sample prepared by of 0.4M (2.5% Copper acetate doping) concentration

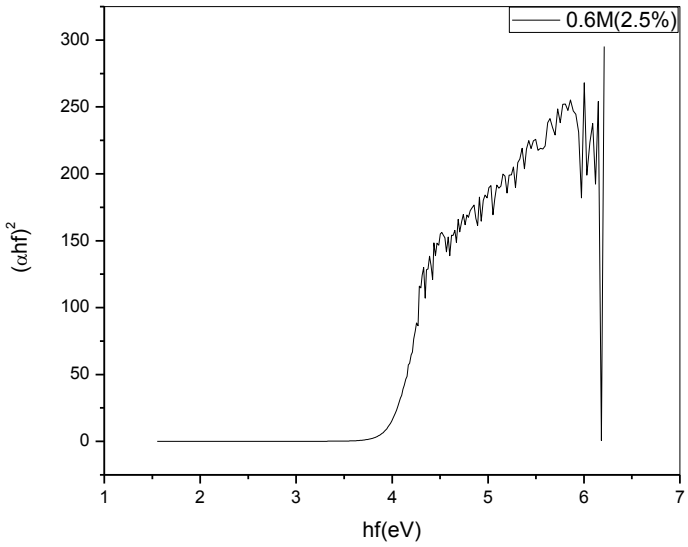

Fig. 4.9 The Optical Energy Gap for the Direct Allowed Transition of Cu-doped SnO2 films for the sample prepared by of 0.6M (2.5% Copper acetate doping) concentration

E. Indirect Band Gap

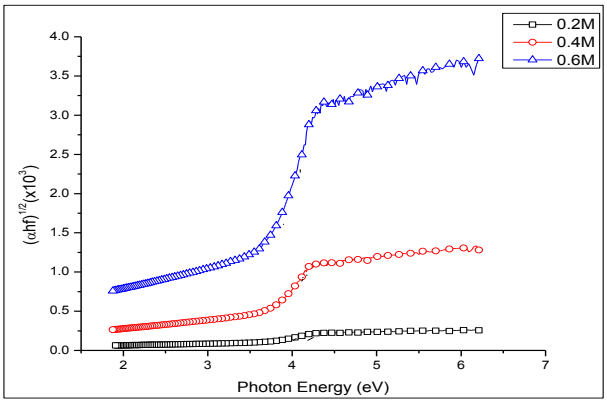

Fig. 4.10 The Optical Energy Gap for the indirect Allowed Transition of SnO2 films for the sample prepared for three concentrations

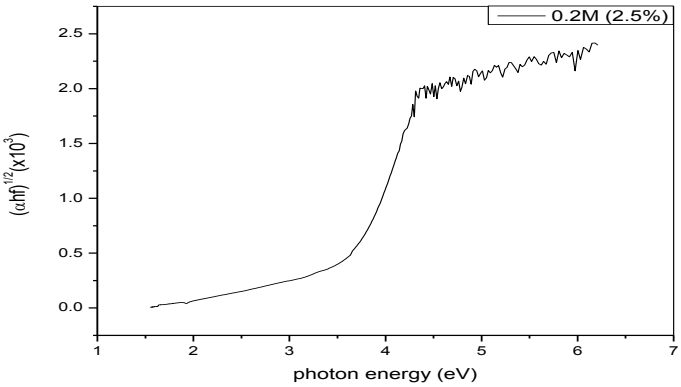

Fig. 4.11 Indirect Band Gap of Cu doped SnO_2 for 0.2M concentration

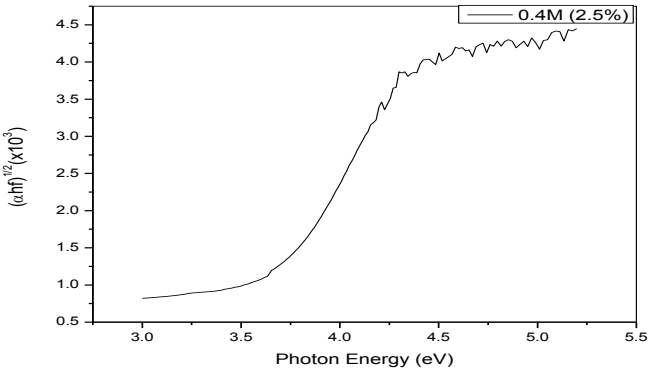

Fig. 4.12 Indirect Band Gap for 2.5% Cu doped SnO_2 (0.4M) concentration

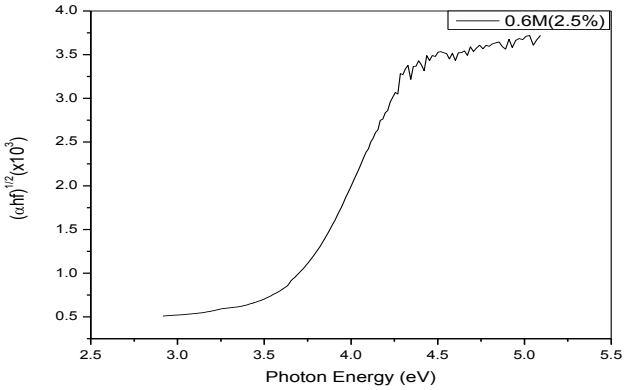

Fig. 4.13 Indirect Band Gap for 2.5% Cu doped SnO_2 (0.6M) concentration

The absorbance spectra of thin films with different concentrations are shown in Fig. 4.1. The trend of absorption spectra is same for all the grown thin films. Absorbance spectra show that absorbance decreases with the increase in wavelength for all the samples. The absorption decrease is almost same for 0.4 M and 0.6 M samples whereas it is comparatively greater for 0.2M sample. This shows that the thickness of 0.2M sample is higher than the other samples. Absorption is very high in the UV region, lowest absorbance in the visible region. In the NIR region, absorbance became steady. Absorbance decreases as precursor concentration increases for thin films. The transmittance spectra of films with different concentrations are shown in Fig 4.2. The transmittance spectra have the same trend for all the films. The transmittance is highest in the visible region for 0.4 and 0.6M solutions and is lowest for 0.2M solution. This is because lower thickness of 0.4 and 0.6M solution samples. In the NIR region, transmittance became steady. Transmittance increases with the increase in precursor concentrations. The absorption coefficient of SnO_2 thin films with different concentrations is shown in Fig.4.3. The absorption coefficient (α) of SnO_2 thin films were calculated using the following equation:

$$\alpha = \frac{2.303A}{t}$$

Where α is the absorption coefficient, t is the thickness of the film and A is the Absorbance. The thickness of thin films has been found to be 2μm-10μm.

The absorbance coefficient of SnO_2 thin films was plotted against the photon energy. The pattern is same for all the films with different concentrations. The films have lower absorption for longer wavelengths; absorption is highest for smaller wavelengths.

The optical energy gap for the direct allowed and indirect allowed transition between valence band and conduction bands of SnO_2 thin films are shown in fig. 4.4- 4.9 and Fig 4.10-4.13 respectively. The direct and indirect optical band gap energy of thin films was extrapolated from the plot of $(\alpha hf)^2$ and $(\alpha hf)^{1/2}$ against photon energy (eV) respectively. The band gap energy values were determined by plotting intercept with energy axis. The direct and indirect band gap energies are affected by precursor concentrations. The direct energy band gap for thin films is about 3.77eV, 3.81eV and 3.83eVrespectively. The wide direct band gap makes these films good material for potential applications in optoelectronic devices such as multilayer dielectric filters and solar cell. The indirect band gap for thin films is approximately 3.48eV, 3.32eV and 3.20eV respectively.

Figs. 5a-5c show the UV spectra for Cu doped SnO2 thin films with copper concentration of 2.5% in 0.2M, 0.4M and 0.6 M solutions. From these spectra, it is observed that the energy gap of the thin films decreases. These are 3.76 eV, 3.73 eV and 3.16eV respectively. The less value may be due to the addition of Cu- dopant and also may be due to growth of grain and improvement of the degree of crystallization. As for as direct band gap is concerned that Cu doping appears to have no effect on the direct band gap which is in accordance with as reported in the literature [3].

4.2 STRUCTURAL PROPERTIES

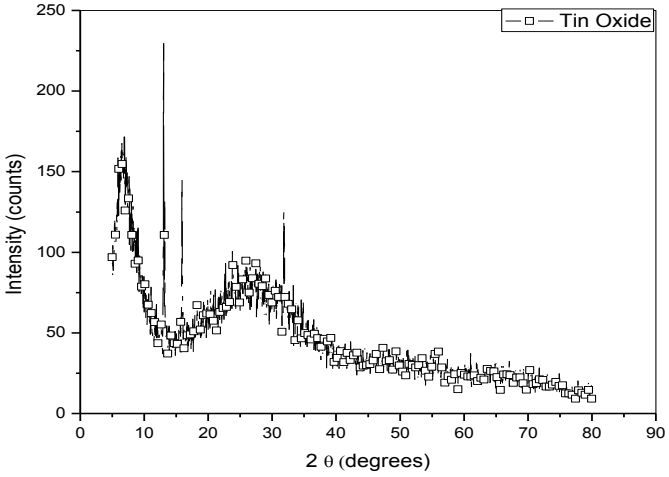

Fig 4.14. XRD patterns of Tin Oxide

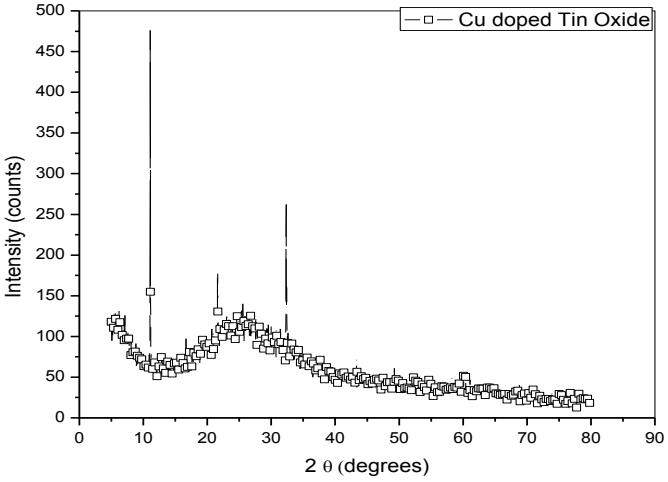

Fig 4.15. XRD patterns for Cu doped Tin Oxide thin films

Fig 4.14 and 4.15 shows the XRD patterns of tin oxide and Cu doped tin oxide thin films respectively. Fig.6a shows that tin oxide thin films have crystalline nature. The peaks at [101] and [022] planes were present. The peaks corresponding to angles 6.87^0 and 13^0 was unidentified. The lattice parameters for tin oxide thin films were calculated. It was found that the lattice parameter 'a' for tin oxide thin films was 6.75 and the value of 'c' came out to be 4.53 which are different from the values mentioned in the literature. However for Cu doped tin oxide thin films, the peaks having planes [022] and [101] were examined. The peak corresponding to

10.97 was unidentified. The lattice parameter ‘a’ and ‘c’ for Cu doped tin oxide thin films were 6.03 and 4.05 respectively. The tin oxide thin films had tetragonal rutile structure. The calculated XRD parameters for tin oxide and Cu doped tin oxide thin films are shown in Tables 4.1 and 4.2 respectively.

TABLE 4.1. XRD data for Tin Oxide thin films

hkl	**2θ**	**Intensity (I)**	**D**	**A**	**C**
(101)	15.85	145.110	5.58	7.891311678	5.302961448
(020)	31.73	124.078	2.81	5.62	3.77664

TABLE 4.2. XRD data for Cu doped Tin Oxide thin films

hkl	**2θ**	**Intensity (I)**	**d**	**A**	**C**
(022)	21.73	174.679	4.085	8.16	5.48352
(101)	32.25	260.447	2.772	3.917371568	2.632473694

SUMMARY

Tin oxide (n-type semiconductor) has extremely good photoelectric properties. Tin oxide is the best material to be used as thin films because of its desirable properties. Tin oxide materials are more transparent. These types of materials have considerable conductance and reflectance. So these materials are preferred by the researchers for the formation of thin films. The electrical properties of the thin films are affected by the deposition parameters such as thickness of the film and deposition temperature.

Tin oxide is the best material used for optical and electrical devices. Tin oxide materials are preferred for transparent devices applications because in the valence band and conduction band of tin oxides the density of 5s states and 5p states is high and delocalized. Tin oxide exists in +2 and +4 oxidation states.

Due to all these favorable properties of tin oxide with doping and without doping, these are useful in research in the field of condensed matter physics. Tin (II) chloride is preferred as a precursor material because of its cost effectiveness**.** Sol gel process is preferred due to many favorable properties such as low temperature, controllable thickness

For sol gel spin coating technique of thin film preparation, a solution is prepared by solvent methanol by choosing tin ((II) chloride as a precursor due to its cost effectiveness. Acetic acid is chosen to be used as a chelating agent and copper acetate is used as doping material. The prepared solution is deposited on a glass substrate. The prepared films are characterized by using UV-VIS spectroscopy for finding the absorbance, band gap and XRD analysis for the identification of structure of thin films.

CONCLUSION

Tin oxide (SnO_2) thin films have been successfully deposited on the glass substrates by using sol gel spin coating technique. Thin films were fabricated by taking the different concentrations of precursor solutions of $SnCl_4$ and Copper acetate. From the absorbance spectra, it has been concluded that the absorbance is low and transmittance is high. This is due to the greater thickness of the films which is in μm. The prepared thin films have wide energy band gap in the range of 3.77-3.83eV which is in agreement with the energy band gap values reported in the literature. Due to more absorbance in the UV region and low absorbance in the visible region, these films can be used to stop UV radiations and admit the visible light so that homes cannot get warm. These films appear to have potential to be used in optoelectronic devices.

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