Recent Perspectives in the Synthesis, Properties, and Applications of Thin Conducting SnO_2 films

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Abstract. Tin oxide is one of the most extensively exploited metal oxides. Tin oxide thin film is one of the widely studied metal oxide thin film materials, thanks to its commendable optoelectronic properties. Varied techniques, including sol-gel dip-coating, spray pyrolysis, and chemical vapor deposition, have been used for obtaining tin oxide thin films. SnO_2 has already achieved some niches in the marketplace; it is used as a coating on the glass that is now being most widely utilized to impart structural rigidity to the surface of bottles and as a functional coating to decorate. The doping with antimony and indium is also frequent in these thin films that enhance their properties, due to which they can be used as semiconductors. These semiconductors are mechanically hard, chemically inert, and high transparency of oxide combined with good environmental stability and high resistance to temperature have opened up numerous applications.

This review aims to throw light on the current situation of SnO_2 conducting thin films giving particular emphasis on antimony doped thin films and a concise idea of how these are synthesized from their precursors using techniques that involve chemical vapor deposition(CVD), spray pyrolysis, sol-gel dip-coating (SGDC) methods and about their properties which are studied using various characterization techniques that include X-Ray photoelectron spectroscopy(XPS) and secondary ion mass spectrometry(SIMS) and very importantly their applications. This work also consists of the comparison made between the above-stated synthesis methods.

Introduction

Tin oxide is one of the most widely studied metal oxides. They have been of high utility due to their various properties. It has been used for years as a ceramic glaze, and some of its recent applications include being used as a catalyst and thin conducting films, and many more. Usually, a thin film is a layer of material (ranging from a smaller fraction of nanometres to various micrometers) in terms of thickness. The primary step in many of the applications includes the controlled development of thin films (a process referred to as deposition). During the 20th century, a betterment in thin-film development techniques has enabled a broad range of technological breakthroughs in various fields of science and technology. Tin oxide thin films are among the widely studied metal oxide thin film materials compared to other metal oxide thin films, all due to their exceptional optoelectronic properties [1]–[3]. Transparent conducting SnO2 thin films are extensively used as electrodes for numerous optoelectronic applications, like electroluminescent devices and solar cells [1]–[7]. They are also used as catalysts and gas sensors [8], [9]. Dopants like antimony, chlorine, cadmium, fluorine, and molybdenum are introduced to enhance tin oxide thin films' optical and electrical properties. In this review work, we have considered Sb as the main dopant along with fluorine [10][11] and sol-gel dip-coating (SGDC)method [11]–[13], Plasma enhanced chemical vapor deposition (PE-CVD) [14][15]and spray pyrolysis[16] as the techniques for synthesizing thin films. Antimony atoms are incorporated easily in the SnO2 matrix by substitution with tin atoms directing to a low lattice parameter modification when compared to that

of fluorine atoms [11], [12]. The properties of the thin conducting antimony doped tin dioxide films involving the doping concentration, oxidation states of the dopant atom, etc. are studied using characterization techniques such as secondary ion mass spectrometry (SIMS) & X-ray photoelectron spectroscopy (XPS) [11], [12]. The TEM images of the doped thin films are also considered for analyzing the effect of doping on the measured mean size of the crystallites [12]. This work also includes the correlation between the two synthesis techniques, which are the organometallic chemical vapor deposition (OMCVD) method and sol-gel dip-coating method (SGDC) [13]. Last, this review article also includes the various applications of conducting tin oxide thin films in a variety of fields [17]–[30]

Scope of the Review

Thin films of tin oxide are studied in the thin-film category with a wide range of applications. Different synthetic strategies can be used for thin films of desired quality. The objective of this review is to shed light on the current situation of SnO_2 focusing on antimony doped films and an idea of how these are synthesized from their precursors using advanced techniques and, most importantly, their applications. Moreover, this review also includes a comparison between the above methods in various prospects which would be helpful for researchers to adopt the right way as per their requirements.

Synthetic strategies

The thin film deposition is a way of preparing a thin film onto a surface called substrate or onto previously deposited layers. Several techniques are used to deposit thin films onto the surface of the substrate. The various deposition techniques employed can control the thickness of thin films. Thin films usually vary in thickness ranging from a few hundred angstroms to tens of micrometers. A brief idea of essential thin film deposition techniques is given in this section.

Sol-gel dip-coating (SGDC) method for the synthesis of antimony doped thin conducting SnO_2 films. The Sol-gel method is one of the easiest and most widely used ways to develop thin films with a wide variety of inorganic and nanocomposite materials. It allows a reasonable degree of control of the various critical parameters, introducing flexibility that cannot be obtained using other conventional techniques. Different wet thin film coating techniques include spray-coating, flow-coating, spin-coating, and dip-coating techniques. This review concentrates on the dip-coating method of sol-gel thin film synthesis. The following are the two methods of the dip-coating process of sol-gel film synthesis.

The antimony doped SnO_2 arrangements were set up from chloride precursors directly in the laboratory. Solution of SnO_2 was acquired by dissolving $SnCl_2.2H_2O$ in absolute ethanol. The solution with antimony was produced from SbCl3 and broke up in absolute ethanol. The two blends were independently mixed and warmed in a closed vessel. The vessels were then opened and blended, and heated again until the solvents were evaporated. They at long last got two powders wholly mixed in a specific volume of absolute ethanol. The doped blend was last mixed and heat-treated at a temperature of 50^oC for two hours. The substrates for this synthesis method were cleaned dried plates of pyrex glass, and afterward, they were covered up. The dip-coating apparatus is set up inside a covered plexiglass chamber to oversee the pulling stage's environment. The plates were dunked vertically and cautiously into the solution and were left for a brief timeframe, pulled back from the bath at a consistent rate. The lifting arm used to bring down the plates into the solution and lift them is engine controlled and structured so that it can forestall any vibration. After dipping, Sb: SnO2 gel films were dried at a temperature of 150◦C for around 40 minutes and heat-treated at 500◦C for 15 minutes in consistent progression of pure and dry oxygen for densification. The entire cyclic procedure can be rehashed to acquire layers of various thicknesses. It is possible to superimpose up to 5 layers of around 100 nm each, and the thickness can be controlled and taken care of using a Tencor "Alpha advance" device. The ratio between the initial quantities of antimony chloride and tin chloride determined the dopant level present in the solution. The influence of more extended aging of the doped solutions was not explored.[11], [12]

The materials used to direct the trial are triethanolamine (TEA), Sn(O-i-Pr)4, and isopropanol moving forward without any more contamination. The strategy for the experiment involves conducting the investigation at room temperature and preparing the reaction mixture that includes the addition of 1 molar proportionate triethanolamine to an isopropanol solution of the alkoxide (0.1-0.7M). The blend was mixed for 2 hours at room temperature. At that point, deionized water weakened with isopropanol was added, and the blend was mixed for 2 hours at room temperature. This was utilized for the dip-coating of the SnO_2 thin films by dipping the glass substrate to the blend, fetching up at a consistent speed (6-18 cm/min), and desiccating for around 30 minutes in the air to shape the thin films. The SnO_2 thin films were developed by warming the thin films for 1 hour at 600^0C. The antimony-doped SnO_2 film is prepared by utilizing a mixture of antimony triethoxide$Sb(OEt)_3$and tin alkoxide to set up the solution. [13]

Synthesis of antimony doped thin conducting SnO_2 films using Plasma Enhanced Chemical vapor deposition method (PE-CVD). PE-CVD is a widely accepted method for transparent conductive film development. The essential purpose behind its acknowledgment in thin-film creation is its capacity to work at lower temperatures than the thermally determined Chemical Vapor Deposition. PE-CVD uses plasma, an ionized gaseous species or particles, electrons, and some impartial species in the ground and the excited states. PE-CVD uses reactive species like electrons and free radicals to develop thin films at lower temperatures. The following is a method of thin-film development using PE-CVD.[14]

Sb doped tin oxide films were set up by PE-CVD strategy utilizing $SbCl_5$, $SnCl_4$, and O_2 as antecedents. A mass flow controller is used to control the flow rate of the gas phase mixture separately. The pre-cleaned substrate corning glass was mounted on an alumina susceptor within the reaction chamber. K-type thermocouple and mass flow controller will monitor the substrate's temperature and partial pressure. Condensation during the flow of reacting gas was prevented by the presence of the heating string that kept outlet tubes from the bubbler at a temperature of 60^oC. After antimony doped tin oxide films were saved onto the substrate, the samples were permitted to come down to a lower temperature. Antimony doping is usually possible by keeping an optimum partial pressure $PSnCl_4$ 0.0038 torr; PO2, 0.56 torr; and the total pressure, one torr. The $SbCl_5$ partial pressures were in the range of zero to 0.01 torr. The deposition temperatures differ from 250^oC to 500^oC, changing the RF power from the range of 15 to 90 W.[14]

Spray pyrolysis technique to synthesize fluorine-doped thin SnO_2 films. SnO_2 thin films with high crystallinity, conductivity, and transparency can be synthesized using tetra-n-dibutyltin (IV) as the precursor and spray pyrolysis as the deposition technique. Highly oriented and ordered SnO_2 films are obtained, and it occurred through the arrangement of SnO_2 cores, crystal growth with favored direction, and the development of crystals on the substrate. F- ions are used as active dopants; therefore, high-quality SnO_2: F films can be created on glass substrate at a generally low temperature, improving the figure of merit at low synthesis temperatures and increasing the chemical stability and heat resistance.[16]
Tetra-n-dibutyltin (IV) is added to ethanol solution and additives of H_2O_2 and NH_4F of molar ratio of 0-2.5($[H_2O_2]$ /[TBT]) and $[NH_4F]$ /[TBT] are prepared. The solution is then sprayed by using a spray gun fixed 30 cm above the substrate holder with a glass substrate and heated to a temperature of about 299°C to 490°C. It is compressed by air until the substrate temperature is cooled by mist. This process is repeated as many times as desired, thus forming a film thickness of 120nm. The solution is sprayed into the air by using a perfume atomizer cleaned with organic solvents. Thickness increases linearly with the increase in the cycles. The crystal structure is determined by CuKα radiation. By adding H_2O_2 to the source solution, the substrate temperature was decreased to 340^oC. The job of H_2O_2 is to advance SnO_2 arrangement not just by actuating an oxygen

environment during the pyrolysis of tetra-n-butyltin(IV) yet additionally by delivering tin peroxide complex in the source arrangement.[16]

Sol-Gel Technique to synthesize Fluorine-Doped Tin Oxide Thin Films. To utilize SnO_2 thin film as a conducting layer, the resistivity of SnO_2 should be brought down. In this manner, fluorine has also been used as a dopant for SnO_2. The fluorine particle replaces the O_2- atoms and goes about as an electron contributor, which brings about an n-type semiconductor. Fluorine is a perfect replacement for oxygen because the anionic sizes of both fluorine and oxygen are comparable. The vitality of separation of the Sn−F bond is like that of the Sn−O bond. The sol-gel process is more advantageous over different techniques because of its simplicity, ease, and the chance of film deposition onto huge and complex substrates.[10]

Fluorine was doped just as the undoped SnO_2 films were saved on microscopic glass substrates using a sol-gel strategy utilizing a dip-coating procedure. The antecedent ($SnCl_2 \cdot 2H_2O$) was made to dissolve into 5 ml of HCl acid and was heated for 10 minutes at 60˚C. A solution of ethanol was prepared of suitable concentration to upgrade the gel development, and a few drops of Triethanolamine were added to the readied mixture. The ammonium fluoride included in the pre-blended solution is the origin of the fluorine doping molecules. The subsequent solution was mixed at 60˚C for 2 hours; a clear and homogeneous arrangement was obtained; it was left for a day at room temperature resulting in a gel-like structure. The dipped glass substrates were released vertically with a consistent rate of 8 cm per minute from the solution. For the development of film with the desired thickness, fifteen dippings were rehashed; after each dipping, the deposited films were dried at a temperature of 100˚C and then annealed for 1.5 hours at 450˚C in air.

Comparison between the sol-gel dip coating and the chemical vapor deposition. In dip-coating (sol-gel method), batch-like film development occurs. Uniform film thickness is obtained from a single dip drying heat cycle. Multiple coating is required to get the thick film, yet progression between the layers is questionable. Fine crystals are acquired without pores, and a smooth and homogenous surface makes the film transparent. This method shows magnificent film reproducibility because of the steady viscosity gained. Multicomponent films are delivered by dissolving the applicable compound to the solution for a polymer network arrangement by controlling the composition. The film stress is malleable, and any alterations cannot bring it down as it might cause breaks in the film. An optimum temperature of 6000C is maintained to get a thick film. A lower temperature may cause crystallization, and a higher temperature can bring cracks in the movie. Another significant advantage is utilizing even the large substrates to make conductive transparent thin films. [13]

In OMCVD, it shows a continuous growth procedure, and there is the formation of uniform thick films, and it was acquired in a brief timeframe period. The volatile starting material is required for the process. They aggregate as large size crystals with large pores. On surface and cross-sectional area examination, uneven surfaces are observed. Numerous boundary conditions, including the temperature of carrier gas and vapor pressure of source, need to be controlled, or it may result in thick films. An ideal temperature of 6000C must be maintained throughout. A temperature above 6000C may bring some homogeneous vapor phase reactions that will result in powdery film development, and a temperature below 600oC may result in films that are hard to prepare. In this process, composition control is not dependable and straightforward.

Examination of OMCVD films suggests that dip-coating films were less crystallized, denser, and more uniform than OMCVD films. So, it is unrivalled in the synthesis of thin films, while OMCVD has an incentive in developing thicker films.

In contrast to other synthetic strategies included in this review, spray pyrolysis approaches have a few unique points of interest. Firstly, due to the simple model of apparatus and the procedure. It is a procedure mainly used to prepare tin oxide thin films as it is direct, cheap, low-cost equipment and raw materials. In addition to this, the films' thickness and the deposition rate can be easily

guarded over a broad range by changing the spray parameters such as the nature of added substances, flow rates, and the convergence of reactants in the antecedent solution. The spray pyrolysis method has been applied to store a broad scope of thin films utilized in different gadgets, such as photovoltaic cells, solid oxide fuel cells, and sensors.

Analysis of properties of Sb and F doped SnO2 thin conducting films

Analysis of antimony doped thin films using X-Ray Photoelectron Spectroscopy. The extent of antimony doping was analysed primarily using XPS analysis as per the previous reports [11], [12]. The first set of doped samples was analyzed with theoretical doping levels (Dpsol) as 3, 6, 8, 9, 10, and 12%. The antimony peaks were not detected on the corresponding XPS spectra. So, it may be concluded that the antimony had not been incorporated, but the detection limits of the XPS method must also be taken into account. In fact, for all actual doping Dpcoat ≤ 4.5%, the XPS method appears to be not sensitive enough to give evidence of the antimony species in the deposited SnO2 layers. Therefore, we need the SIMS characterization to prove that these layers are effectively doped but with relatively weak doping concerning those expected theoretically from the starting solutions.

The main result of these experiments is that the actual doping Dpcoat is less than the doping levels in the initial solutions. The weakest Dpcoat detected by XPS in the films is about 3.8%, corresponding to a theoretical doping Dpsol of 20%. The greatest Dpsol of 45% corresponds to 22% in the layers. This fact shows the difficulty in transferring the antimony from the solution to the gel.

The lack of doping atoms in the resulting film is not yet understood. Several hypotheses have been proposed: (a) an incomplete disintegration of the antimony chloride in C2H5OH; (b) a possible development of Sb-based precipitates in the Sol-Gel Dip-Coating solution; (c) a formation of Sb-based volatile compounds evaporating from the meniscus during the pulling process or annealing. Together, each of these procedures may contribute to this "loss" of dopant and are somewhat hard to confirm experimentally.

As the Sb content increments, and as revealed in different works identified with varying elaboration strategies, a more exceptional, somewhat bluish coloration of the films is observed.

Analysis of antimony doped tin oxide thin films using TEM and SIMS Technique. SIMS has also studied all the doping concentrations quoted in the above analysis. It was observed by C. Terrier et al. on all spectra obtained that the antimony presence is noted even for the lower doping concentrations where the XPS technique did not detect its presence. The SIMS characterization is much more sensible at lower doping concentrations. [11][12]

The thinnest samples are analyzed using Transmission Electron Microscopy (TEM) according to the various doping levels. All films obtained are polycrystalline, and the amorphous zones are not observed. The diffraction pattern for each doping gives the same diagram. It is evident from the TEM images obtained that the crystallite size decreases when the doping concentration increases.

The oxidation states of antimony in SnO_2 films [12][11]

When the actual doping Dpcoat expands, the binding energy changes show the nearness of two oxidation states of the antimony. The two oxidation states are Sb3+ and Sb5+. According to some authors, the transfer of charge from Sb5+ and Sb3+ was the reason for the bluish coloration observed on the thin film layers. It is found that with the increase in the concentration of antimony doping, the +3 oxidation state of Sb dominates over the +5 oxidation state, which is evident from the deconvoluted spectra of Sb 3d3/2 line as a function of Sb content in the SnO_2 layers. The XPS measurement indicates that the antimony has two oxidation states, Sb^{3+} and Sb^{5+}.

Application of tin oxide films [17]–[30]

SnO_2 films have intensively investigated applications in the field of transparent electrodes, catalytic support materials, transparent heat reflecting films [25], heterojunction solar cells, gas sensors [8], [24], [27], and protective coating solar cells owing to its exquisite properties. Mainly SnO_2 is extensively studied in the field of microsensor devices. SnO_2 transparent conductive films raised enthusiasm because of their broad scope of significant commercialization in the field of electroluminescent display, liquid crystal displays, touch display panels, and organic light emitted diodes [17], [18], [28]. SnO_2 is a semiconductor that is mechanically hard, chemically inert, and highly transparent oxide with good environmental stability and high resistance to temperature, have opened up numerous applications not only limited to research laboratories but also in low and high technology applications, including instrumental panels, antistatic coatings, optical coatings, and environmental monitoring. Significant utilization of thin-film innovation from the worldwide vitality crunch is the photovoltaic cell, which changes over the vitality of the photovoltaic radiation into helpful electrical energy. As of late, usage of the pioneer transparent conductor SnO_2 thin film as conducting solar materials created an eagerness for the commercialization of solar cells. Among the various conductive oxides, antimony doped SnO_2 thin films give off an impression of being the most legitimate in solar cells [5], [6] attributable to its large optical conveyance with less specific electrical resistance.

P-N junction diode

Transparent Conductive Oxides (TCOs) with wideband gaps are useful substances because of their tunability to effectively consolidate the high and low conductivity alongside the high visual transparency. TCO substances showing high optical transparency and electrical conductivity can be developed as thin film and are utilized considerably for various capacities comprising design windows, optoelectronic units, photovoltaic cells, ion storage, and defrosting windows in fridges and planes, and flat panel displays. TCOs have a legitimate total of optical and electrical properties that bring about their expansive mechanical property. N-type material of SnO_2 is very notable and generally utilized. Contrasting with some different oxides, SnO_2 shows more potential for stable p-type conducting oxides because of its weak ionization. The chemical nature of the potential barrier considered had been encouraged by the more homogeneous microstructure of tin oxides. The non-linear properties are a direct result of the physical heart. Numerous endeavors have been made with p-SnO2 and n-SnO2 films to blend p-n junction diodes.

Semiconductor Nanowires [29]–[32]

Over the most recent couple of years, semiconductor nanowires have pulled in expanding enthusiasm for the field of novel gadget applications because of their rich properties. They can be manufactured on various substrates, including plastic and glass. On examination with the traditional thin films, nanowires separate the device fabrication stages, with no concern about the compatibility and crystalline materials obtained at the high growth temperature. Synthesis of nanowires is first done under the optimum condition to yield top quality single crystal materials, in which structure, composition, and size of material will oversight the optoelectronic properties. Large quantities of SnO_2 nanowires with high optical conveyance with minimal development and the simplicity of acquiring Ohmic contacts are combined utilizing a simple vapor transport. Doping procedures of SnO_2 nanowires show a sensational impact on its electric property. The best thin films that show metal-like conductivity and low resistivity are antimony doped SnO_2 nanowires. Various sorts of SnO_2 nano-thin films are explored as nanoparticles [29], [30], nanosheets[33], nano rods[31], and nanowires[32] has been utilized in numerous applications, for example, lithium battery [19], photovoltaic vitality converter [23], directing electrodes [34], aqueous salt arrangement [25] harmful gas sensors [9], optoelectronic gadgets [20]–[22] and heat-reflecting mirrors [24].

Conclusions

The thin tin oxide films are extensively studied in the thin films category with many applications. Their various properties account for this vast tin oxide thin film application spectrum. Different synthetic strategies can be used for thin films of desired quality. The Sol-Gel dip-coating method of antimony doped SnO_2 thin films and its further development prove that while transmitting antimony to the gel from the solution, there is a considerable loss in dopant concentration. This dopant loss has not been studied extensively, but several hypotheses have been proposed to support the same. The amount of antimony in the SnO2 films can be reviewed by Secondary Ion Mass Spectrometry and X-ray Photoelectron Spectroscopy. As the Sb amount increases, the TEM observation showed a decrease in the mean size of crystallites. The elaboration of thin films by (PE-CVD) is a widely accepted method for producing thin films. It is widely accepted because it is lower temperature compared to thermally driven CVD. The spray pyrolysis deposition technique of tetra-n-butyltin (lV) has developed SnO_2 thin films with a high order of crystallinity, conductivity, and transparency. The resistivity of SnO_2 needs to be reduced to use SnO_2 thin films as a conducting layer. The N-type semiconductor can be prepared when O_2- of SnO_2 is replaced by Fluorine atom, where Fluorine is used as a dopant and acts as an electron donor. Thin films having high constancy and compactness were obtained by the dip-coating method by doping Sb in SnO_2, in which the films have increased transparency and conductivity. The Sol-gel process undoubtedly has advantages over other methods because it has possibilities of film deposition to large complex substrates, low cost, and simplicity. SnO_2 films find several applications in transparent electrodes, catalytic support materials, transparent heat reflecting films, heterojunction solar cells, gas sensors, and protective coating solar cells, owing to their exquisite properties. In conclusion, tin oxide thin films are essential candidates for recent and future research due to their easy and cost-effective synthetic strategies and exciting properties.

Conflict of Interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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