# Analytical Study of Aluminum Doped ($SnO_2$) Tin Oxide Thin Film Deposited by Chemical Bath Technique: Enhancing the Morphological and Structural Properties

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## ABSTRACT

This work centers on the study of the impact of aluminum element on the properties of Tin Oxide deposited using chemical bath deposition technique, especially the structural and morphological properties which are the determinant of the optical and solid-state properties of the material when it is dope with aluminum element.

Tin Oxide is a well-known Perovskite oxide based thin film, and the interest is to know if the properties will be optimized for effective capabilities in solar energy harnessing and optoelectronics application by the dopant element. These properties were to be analyzed using Scanning electron microscope, X-ray diffraction machine, while the other aspects like crystal size and lattice parameters would be computed using well known formulas. The effect on the band gap due to the aluminum dopant would be analyzed.

(Keywords: *chemical bath deposition, tin oxide, analytical study, enhancement, properties*)

## INTRODUCTION

Perovskite material-based nanocrystals are material films with crystal structure whose structure is associated with these kinds of materials they are derived from. $TiO_3$, ZnO, and $SnO_2$ are generally among some the Perovskite oxide based thin films which have been some of the metal oxide thin film that has attracted attention of many researchers [1]. In some cases, they have been doped with some other transition metal in order to ascertain if their properties could be enhanced just as in $BaTiO_3$, $PbTiO_3$, etc. The general formula for such material for such formation is $ABO_3$ in which B is a small transition metal cation and A is a layer s-, d- or f- block cation [2]. Such materials can be classified into alkaline metal halide, inorganic oxide, and metal halide perovskites [3-5]. There are basically three groups of Perovskite oxide-based nanocrystal materials that may appear as binary, $TiO_2$, $TiO_3$ , ZnO, $SnO_2$ , etc. [1].

The functionality of these types of materials can be modified by the dopant which may be the result of their being in different forms and possessing various types of properties and thereby making them amenable to a wide range of applications and this is more rationale behind why there are lots of eyes and interest on Perovskite nanocrystals [6]. Based on the numerous applications of these materials towards technological and energy harnessing, especially in recent dispensation, more elements are being explored to be used as a dopant to these oxide-based materials in order to see if their properties could be further enhanced for other applications beyond than the ones already mentioned.

These and other applications of these materials are what has drawn the attention of material science researchers over decades, coupled with the vulnerability in manipulating growth materials into various applications to so many other domestic usages. For instance, the use of varieties of dopants on this type of material oxides results in following other types of very important items such as ferroelectric ($BaTiO_3$,$PbTiO_3$), dielectric ($(Ba,Sr)TiO_3$), piezoelectric ($Pb(ZrTi)O_3$), eletrostrictive ($Pb(MgNb)O_3$), magnetoresistant ($(LaCa)MnO_3$), and multiferric ($BiFeO_3$) [7-10] as has been well known especially when it comes to areas of applications with computers, memories, and electronic appliances such for ferroelectric

random access memories, multilayer ceramic capacitor, transducers, sensors and actuators, magnetic random access memories and other potential new types of multiple – state memories and spintronics devices that are typically controlled by electric and magnetic fields [1-7].

All these varieties of applications have created a quest for advancement in probing into Perovskite oxide nanoscale based electronic devices that are amenable to miniaturization of conventional CMOS, complementary metal oxide semiconductor devices. These and many other reasons had led researchers in nanoscience to have the urge to focus more on the area of production and development of high volume Multilayer Ceramic Capacitors (MLCCs) with good purity and uniform shape that would be useful in fabrication of next generation of the same MLCCs which will be of tens of nanometers for enhancement of miniaturization of the aforementioned devices [2].

## Deposition Mechanism

Various deposition techniques have been used in the deposition of $SnO_2$ though generally it is clear that any of the deposition methods that may be preferred, two principles are involved viz; Chemical or Physical processes. The chemical process involves a process in which (a) the chemical precursor is directed to form on a substrate surface with the product layer growth on the surface during which the reaction is kick started. In this case the growth of the film involves heterogeneous nucleation and growth while (b) the materials are meant to dispersed into solvent either as molecular precursor or nanoparticles "ink" or are deposited and the coating is formed on solvent evaporation (and potentially annealing) and its own case, viscous fluid is deposited on the substrate.

In the case of physical process, precipitation of vaporized material on the substrate under reduced pressure (vacuum) is first stage with the formation mechanism involving heterogeneous nucleation which follows the growth of thin film.

The chemical deposition techniques include Chemical Vapor Deposition (CVD), anodization, electroless deposition, and Chemical Bath Deposition (CBD). Successive Ionic Layer Adsorption and Reaction, SILAR, Spin/Dip Spray Coating, or ink jet printing of sol-gel solution or nanoparticle ink [9-17] and Atomic Layer Deposition (ALD) [18-36].

The principle of chemical bath deposition is based in the utilization of controlled chemical reaction by ligand to induce the formation of thin film layer of a material by means of heterogeneous precipitation [37] formed from an aqueous solution of a metal salt acting as a bath for the substrate with the addition of counter ion in the form of $OH^-$ for oxides/hydroxides or $S^{-2}$ for sulfides being governed by the solubility product of the target compound. The coating is deposited on the surface of the substrate with the required ligand to facilitated easy deposition processes [2-5].

SILAR for depositing thin film layer on the substrates operates within the combined ALD and CBD mechanisms as it makes use of physically separated layer approach similar to ALD in combination with some flexibility features and CBD in conjunction with the ambient deposition technique from aqueous solutions of metal salts without the need of high-vacuum equipment [30-37]. Though in contrast to CBD, it requires the application of alternating baths of chemical precursors and also additional features of pure solvent in between the deposition cycles in order enable removal of excessive precursor molecules which makes its process unique such that it has the ability to deposit ultra-thin film of highly conformal coatings on a desired substrate [38-41].

On the other hand, it is worth mentioning here all the chemical deposition processes especially CBD and SILAR deposition techniques have a unique inherent simplicity, low cost, effectiveness, and above all, the ability to operate in an ambient environment. These are the characteristics that placed them in an advantageous position over all the techniques used for deposition. Thus, this work is centered on growth of Tin Oxide $SnO_2$ thin film with aluminum element having various molarities using chemical bath deposition technique in order to ascertain the effect on the morphological and structural properties on $SnO_2$ as a Perovskite oxide-based thin film that to observe if using aluminum which is known for its corrosive resistance element as a dopant would enhance the desired properties of $SnO_2$ thin film or whether it would decrease the properties.

## MATERIALS AND METHODS

The substrates were scrubbed thoroughly with clean cotton wool and soap solution which were rinsed with water. Then the substrates were placed inside a bath container containing a solution of HCl acid (30%) for some time which were later rinsed with deionized water. The substrates were dried in an open furnace at a moderate temperature. All these cleaning processes were done to remove possible impurities (organic or inorganic) from the surface of the substrates which is very important for adherence of the films to the surface of the glass substrate.

The thin films of Tin Oxide ($SnO_2$) were deposited onto the chemically cleaned glass substrates using a simple deposition technique, chemical bath. The chemicals used were prepared sequentially, 0.5M of Tin Chloride Di-hydrate ($SnCl_2.2H_2O$); 0.5M of Sodium Hydroxide (NaOH) to neutralize the acidity of the solution, 0.5M of Triethanolamine (TEA) ($C_6H_{15}NO_3$) into three different baths (T, $AT_1$ and $AT_2$). Aluminum Chloride (AlCl) which is the source of Aluminum (Al) which is dopant was added into the second and third bath ($AT_1$ and $AT_2$) and the concentration was varied from 0.2M to 0.4M.

Distilled water was added until the volume of the solution reached 70ml after which the pH of the solution was the found to be 9. The bath solution was heated and kept to a temperature of 300K and then thoroughly stirred on a magnetic stirrer for a specific amount of time to aid homogeneity.

The prepared substrates were clamped vertically using retort stand, clipped and then lowered into the three beakers (100ml) containing the CBD solution of which the aluminum foil was used as a cover at the top of each beaker in order to prevent dust or unwanted particles from entering the solution. The deposition time was kept constant throughout for each of the samples labelled; T, $AT_1$ and $AT_2$.

After that the three samples were taken out of the bath solution, rinsed in distilled water and dried in an open furnace at moderate temperature at a specific time to remove residual water content and other possible adsorbed surface impurities and allowed to dry up.

The structural and morphological properties of the deposited films were analyzed using the following techniques such as; X-Ray Diffraction (XRD), Scanning Electronic Microscopy (SEM), and Energy Dispersive X-ray Diffraction (EDX),

## RESULTS AND DISCUSSION

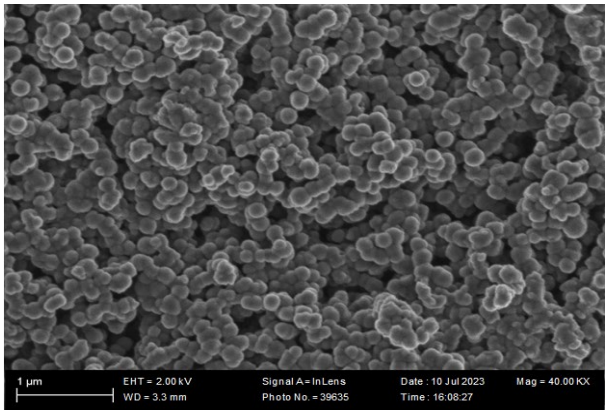


**Figure 1:** SEM Image of the Sample of As-Deposited $SnO_2$ Thin Film (T).

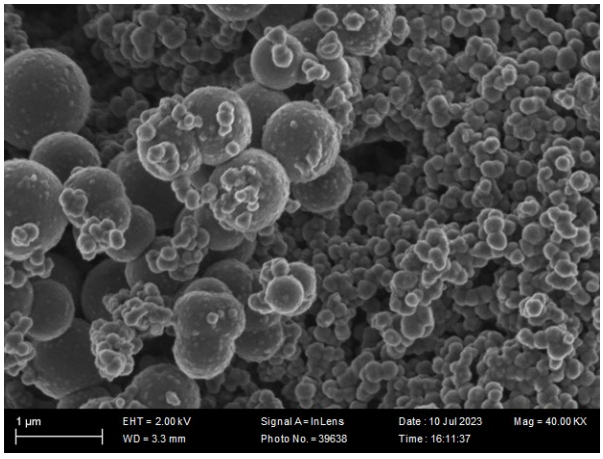


**Figure 2:** SEM Image of $SnO_2$ Thin Film Doped with 0.2M of Aluminum ($AT_1$).

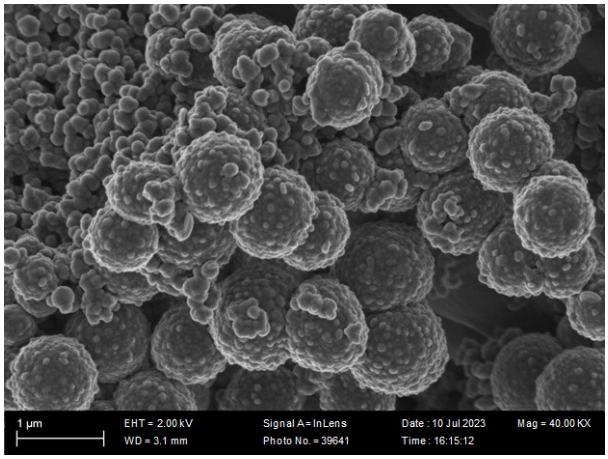


**Figure 3:** SEM Image of $SnO_2$ Thin Film Doped with 0.4M of Aluminum ($AT_2$).

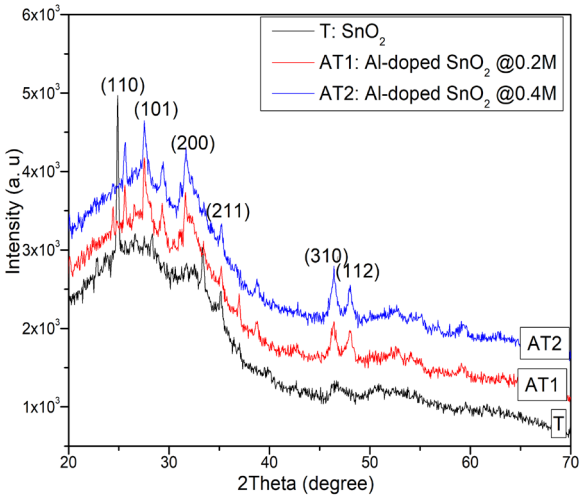


**Figure 4:** X-Ray Diffraction Pattern of As-Deposited $SnO_2$ and Al-Doped $SnO_2$ Thin Films.

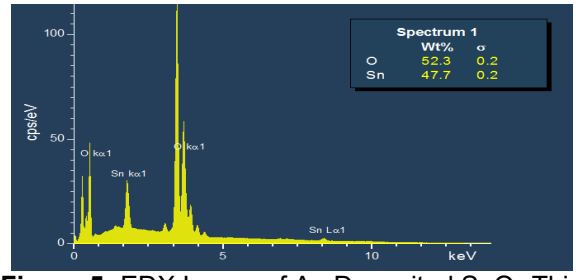


**Figure 5:** EDX Image of As-Deposited $SnO_2$ Thin Film.

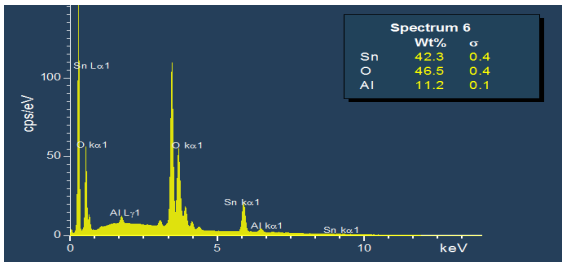


**Figure 6:** EDX Image of Al Doped $SnO_2$ Thin Film.

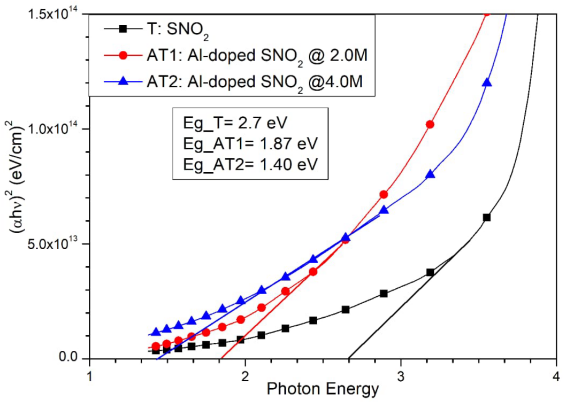


**Figure 7:** The Plot of Energy Band Gap of Samples of $SnO_2$ Thin Films by Photon Energy.

**Table 1:** The Obtained Values of Structural Properties of the Thin Film Samples from the XRD showing Preferred Plane (hkl), Grain Size (D), Dislocation Density (δ), Microstrain (ε), FWHM (β), Lattice Parameter (A) and Lattice Parameter (C).

| S/N | Thin Films Samples | Bragg's Angle (2Θ) | Preferred Plane (hkl) | Grain size (D) (Å) | Dislocation Density (δ) (Å) | Microstrain (ε) | FWHM (β) | Lattice parameter (a) (Å) | Lattice parameter (c) (Å) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | $SnO_2$ (T) | 24.9042 | 211 | 5.9051 | 0.0287 | 0.2725 | 0.2407 | 2.8132 | 1.8229 |
| 2 | Al-doped $SnO_2$ ($AT_1$) @ 0.2M | 25.6227 | 211 | 6.7039 | 0.0223 | 0.2334 | 0.2123 | 2.7342 | 1.7716 |
| 3 | Al-doped $SnO_2$ ($AT_2$) @ 0.4M | 25.6545 | 211 | 7.8847 | 0.0161 | 0.1982 | 0.1805 | 2.7308 | 1.7694 |

**Table 2:** Percentage Chemical Composition of Tin, Oxygen, and Aluminum in the Sample.

| S/N | Element | Wt.% for Pure Sample | Wt.% for Aluminum-doped Sample | σ for Pure Sample | σ for Aluminum-doped Sample |
|---|---|---|---|---|---|
| 1 | Sn | 47.7000 | 42.3000 | 0.2000 | 0.4000 |
| 2 | O | 52.3000 | 46.5000 | 0.2000 | 0.4000 |
| 3 | Al | 0.0000 | 11.2000 | 0.0000 | 0.1000 |

The morphological features of both as-deposited and annealed $SnO_2$ thin oxide thin film are shown in Figures 1 - 3 as obtained from SEM indicates that the morphological features of the material was clearly affected by the magnitude of the aluminum molarity used in doping because it is evidently observed that the coarseness of morphological features increased with the increase in the concentration of the aluminum dopant. This collaborates with Table 1 showing the grain size and other lattice parameters such as microstructure and dislocation density for both as-deposited and aluminum doped $SnO_2$ thin film.

The XRD patterns of the pure Tin Oxide thin film for (T) which represents as-deposited and the Aluminum-doped Tin Oxide thin films (i.e., AT1 and AT2) which represent different concentration of Aluminum doping is shown in Figure 4. The XRD pattern of Tin Oxide thin film was taken within the range of Bragg's angle (2Θ), it is clear as shown in the figure that the material has preferred orientation at as from 20º to 70º, the peaks in the spectra are identified as originating from reflection from (110) and (211) planes showing that $SnO_2$ film had tetragonal structure. Therefore, it showed strong orientation in (110) plane and from $D = \frac{0.9\lambda}{\beta \cos\theta}$

Where D is the grain-size,

λ is the wavelength of X-ray used,

where (λ = 1.54184Å) as regards the XRD data,

β is the diffraction peak's FWHM

and Θ is the Bragg's diffraction angle the estimated grain-size.

Some other structural data are presented as in Table 1, indicates that the D values of the films were dependent on the dopant molar concentration. Similarly, the micro-strain of the films is calculated using the equation: $\varepsilon = \frac{\beta}{4\tan\theta}$

where ε is the micro-strain,

β is the diffraction peak,

Θ is the Bragg's angle.

The dislocation density of the films was also calculated using the equation $\delta = {}^{1}\!/_{D^2}$

where δ is the dislocation density

and D is the grain-size.

Tin Oxide film is found to be a tetragonal structure, the lattice constants was computed using $\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2 + c^2}$

where (d) is the interlunar distance or lattice spacing,

(h, k, l) are the miller indices,

(a) and (c) are the lattice constants for the film structure.

However from the results as observed in table1, it was seen that at the same preferred orientation plane, which occurred at different Bragg's angle diffraction, the grain size of the particle varied in accordance with the variation of concentration of

the dopant just as it was depicted in the morphological diagram as analyzed by SEM in Figures 1 - 3.

On the other hands, the dislocation densities, micro-strain and lattice parameters decreased with increase in the concentration of the aluminum dopant

Based on EDX analysis, it was clearly observed that the elemental constituents of the deposited $AlSnO_2$ thin film in made up of $SnO_2$ and Al which indicates that there was no impurity element and this invariably explains the fact that chemical bath deposition technique is a good method of depositing relatively pure Perovskite oxide-based thin films.

In a similar manner, the energy band gap of the material seemed to get narrower as the concentration of the aluminum increased indicating that doping $SnO_2$ increases the features of the material to solar energy harnessing and opto-electronic applications [42].

## SUMMARY

The study of the impact of aluminum element on the properties of Tin Oxide deposited using Chemical bath deposition technique, especially the structural and morphological properties which are the determinant of the optical and solid state properties of material has been carried out here were the focus was to ascertain if the aluminum element which was used as a dopant will optimize the properties of the material for effective capabilities in solar energy harnessing and optoelectronics application.

From the analysis, it was found that these properties were grossly effected as obtained from the electron microscope, X-ray diffraction machine, and also from other parameters as computed because it was clear that both the crystal size and lattice parameters including the band gap of the material were influenced by the aluminum dopant. This work revealed two issues, firstly how the aluminum dopant affected the properties of Al $SnO_2$ thin film deposited by chemical bath deposition technique and secondly another salient information that the use of CBD produces pure thin film of $AlSnO_2$ with negligible or no impurity elements.

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## SUGGESTED CITATION

Ugwu, E.I. 2024. “Analytical Study of Aluminum Doped ($SnO_2$) Tin Oxide Thin Film Deposited by Chemical Bath Technique: Enhancing the Morphological and Structural Properties”. *Pacific Journal of Science and Technology*. 25(2): 12-19.