American Journal of Materials Research
2019; 6(1): 1-10
http://www.aascit.org/journal/ajmr
ISSN: 2375-3919

AASCIT American Association for Science and Technology

Synthesis of ZnO:SnO_2 Alloyed Thin Films for Surface Coatings, Solar Energy Conversion and Optoelectronic Applications

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Citation

Joseph Ijeoma Onwuemeka, Azubuike Josiah Ekpunobi, Peter Ifeanyi Ekwo. Synthesis of ZnO:SnO_2 Alloyed Thin Films for Surface Coatings, Solar Energy Conversion and Optoelectronic Applications. *American Journal of Materials Research.* Vol. 6, No. 1, 2019, pp. 1-10.

Received: January 11, 2019; **Accepted**: March 17, 2019; **Published**: April 9, 2019

Abstract: The synthesis of ZnO:SnO_2, alloyed thin films have been prepared on glass substrates under the elevated temperature of 60°C of NaOH solution, using two solution based methods: successive ionic layer adsorption and reaction (SILAR) and solution growth technique. The deposited alloyed samples of A_{24} and A_{25} were annealed at 200°C and 250°C respectively using Master Chef Annealing Machine. The structural properties and morphology of the samples were done using X-ray diffractometer (XRD) and scanning electron microscope (SEM) respectively. The XRD pattern of ZnO:SnO_2 alloyed thin films of sample A_{24} has two diffraction peaks at 2θ = 19.522° and 22.811°, which indicates the crystalline nature of the alloyed thin films. The grain sizes were found to be 40.522nm and 92.41nm. Rutherford backscattering Spectroscopy (RBS) analysis confirmed the percentage of the elements of Zn, Sn and O in the alloyed thin films. The alloyed thin films of samples A_{24} and A_{25} ZnO:SnO_2 show optical transmittance of 42%-58% in the ultraviolet region, 60%-61% in the visible, and 62%-58% in the near-infrared regions of electromagnetic spectrum for sample A_{24} annealed at 200°C. Sample A_{25} annealed at 150°C has an optical transmittance of 59% -62% in the ultraviolet region, 62%-65% in the visible and 65%-68% in the near infrared regions of electromagnetic spectrum. The two samples, have equal direct wideband gap of 3.34±0.05eV. Other properties that were investigated are; absorbance, reflectance absorption coefficient, extinction coefficient, refractive index, optical conductivity, and dielectric constants. These compound alloyed thin films may be found useful in heat mirror applications, galvanization, active layer in various types of solar cells and passive layer in solar-selective surfaces, and are also found interesting as semiconductor materials for opto-electronic applications.

Keywords: Absorbance, Reflectance, Absorption Coefficient, Extinction Coefficient, Refractive Index, Optical Conductivity

1. Introduction

The need for energy in our lives and in nation building cannot be over emphasized. This cuts across the socio-economic and political lives of the citizenry both in rural and urban areas. In spite of the fact that energy lies around us in vast quantities within the dynamic forces of nature - the sun, winds, tides, and waves; there must be a technological approach of harnessing them from a low grade form to a high one in order to achieve a desired purpose. Several studies have been made that indicate that the peak year of oil production has gone already [1].

The problems caused by the existing energy source (i.e. fossil fuel) have encouraged researchers to continue to search for most efficient and economic mode and device for tapping the huge solar energy available on earth. The standard of living of a nation currently depends on the energy consumption. Any reduction in its supply may lead to abrupt change in the lifestyle of the people and equally affect national security.

Recently, most researches on solar energy focus on finding

conversion systems that have the simultaneous promise of efficiency, durability, and low cost as well as solar devices whose absorption of solar radiation are wavelength dependent. To achieve this goal, photovoltaic cells made of polycrystalline thin films such as ZnO, SnO_2, CdO, PbO, In_2O_3, and Cu_2O as well as non-silicon materials that are relatively inexpensive and readily available for use. The usefulness of thin films and their applications cut across microelectronics, magnetic and gas sensors, optics, corrosion protection, wear resistance, and solar control devices among others.

These films are the transparent conducting oxides (TCOs) which play important roles in many large and small scale applications in selective window coatings, solar cells andflat panel displays (FPDs). The transparent conducting films have been prepared from a wide variety of materials. They are characterized by the simultaneous occurrence of high optical transparency (> 80%) in the visible region, low effective mass, high carrier mobility and high electrical conductivity (> $10^5\Omega^{-1}m^{-1}$). This is usually achieved by creating an electron degeneracy in a wide band gap (>3eV) semiconductor through the controlled introduction of non-stoichiometry or appropriate dopant [2] while others have band gaps less than 3eV. The primary n-type TCOs have remained virtually unchanged in the last two decades of simple oxides such as ZnO, CdO, SnO_2, Ga_2O_3 and In_2O_3 [3].

Alloys are defined by a metallic bonding character. An alloy may be a solid solution of metal elements (a single phase) or a mixture of metallic phases (two or more solutions). An alloy is distinct from an impure metal in that, with an alloy, the added elements are well controlled to produce desirable properties, while impure metals such as wrought iron, are less controlled, but are often considered useful. Alloys are made by mixing two or more elements, at least one of which is a metal. This is usually called the primary metal or the base metal, and the name of this metal may also be the name of the alloy [4].

Constituents of alloys may or may not be metals but, when mixed with the molten base, they will be soluble and dissolve into the mixture. The mechanical properties of alloys will often be quite different from those of its individual constituents. Although the elements of an alloy usually must be soluble in the liquid state, they may not always be soluble in the solid state. If the metals remain soluble when solid, the alloy forms a solid solution, becoming a homogeneous structure consisting of identical crystals, called a phase. If as the mixture cools the constituents become insoluble, they may separate to form two or more different types of crystals, creating a heterogeneous microstructure of different phases, some with more of one constituent than the other phase has. However, in other alloys, the insoluble elements may not separate until after crystallization occurs. If cooled very quickly, they first crystallize as a homogeneous phase, but they are supersaturated with the secondary constituents. As time passes, the atoms of these supersaturated alloys can separate from the crystal lattice, becoming more stable, and form a second phase that serve to reinforce the crystals internally. Naturally occurring alloys, such as electrum consisting of silver and gold [4].

The base material is called the matrix, or the solvent. The secondary constituents are often called solutes. If there is a mixture of only two types of atoms (not counting impurities) such as a copper-nickel alloy, it is called a binary alloy. If there are three types of atoms forming the mixture, such as iron, nickel and chromium, then it is called a ternary alloy. An alloy with four constituents is a quaternary alloy, while a five-part alloy is termed a quinary alloy. In this respect, all the various forms of an alloy containing only two constituents, like iron and carbon, is called a binary system, while all of the alloy combinations possible with a ternary alloy, such as alloys of iron, carbon and chromium, and is called a ternary system [5]. In this present work, the synthesis and characterization of ZnO:SnO_2 alloyed thin films have been studied.

2. Materials and Methods

2.1. The Deposition of ZnO:SnO_2 Alloyed Thin Films by SILAR Method

The synthesis of the alloyed thin films using SILAR method constituted: 3ml of 3M solution of ammonia used as complexing agent, 20.45g of 1M solution of $ZnCl_2$ dissolved in 150cm^3 water and 16g of 2M solution of NaOH dissolved in 200cm^3 of water.

3ml of 99% of 3M solution ofammonia used as complexing agent was measured with a syringe and added to beaker containing 1M solution of $ZnCl_2$, in separate beakers. $ZnCl_2$, produced white precipitates which dissolved in excess NH_3, forming zinc tetra-amine complex ion as given in equations (1).

$$ZnCl_2+4NH_3\rightarrow[Zn(NH_3)_4]^{2+}+2Cl^- \quad (1)$$

De-ionized water was added up to 50ml and the solution was stirred vigorously in order to achieve uniformity in the mixture.

ZnO thin films were deposited on substrates in cycles, by dipping the substrates into the beaker containing the cation precursor of zinc tetra-amine complex ion as depicts in Figure 1a and then rinsed in a beaker of de-ionize water, then immersed into the third beaker, containing the anion precursor, which is 2M solution of NaOH, at elevated temperature of 60°C as shown in Figure 1c. The substrates were rinsed in de-ionized water after successive immersion as depicts in Figure 1b and 1d and this is repeated based on the number of cycles. The reaction is given in equation (2). The parameters for SILAR deposition are given in Table 1.

$$[Zn(NH_3)_4]^{2+}{}_{(aq)}+2NaOH_{(aq)}\rightarrow ZnO_{(s)}+2Na^+{}_{(aq)}+H_2O_{(l)}+4HN_{3(l)} \quad (2)$$

Table 1. *The deposition parameters of ZnO thin films.*

Sample	Dip-time(s) in each reactant	No. of cycle	Dip-time(s) in eachBeaker of H_2O
A_{24}	8	22	3
A_{25}	8	22	3
A_{26}	8	22	3
A_{27}	8	22	3
A_{28}	8	22	3

2.2. The Deposition of ZnO:SnO_2 Alloyed Thin Films by Solution Growth Technique

Figure 2, shows the constituent reagent that make-up the deposited samples of$ZnO:SnO_2$ alloy on the substrates: 20ml of 0.2M solution of tin chloride, 3ml of 3M solution of NH_3 and 15ml of 2M solution of NaOH and the substrates containing freshly deposited samples of suspected ZnO thin films prepared by SILAR method. Ammonia ($NH_{3(aq)}$) in this reaction is the complexing agent. It controls the rate ofion – by – ion interaction, thereby moderating the rate of formation of precipitate. It also creates an alkaline medium for good formation of deposits. The reaction mechanisms that led to the deposition of the required samples is given in equation (3).

Several bath compositions were employed as depicts in Table 2, but the optimum result was achieved with the specification noted above with the pH value of 11.

$$2ZnO_{(s)} + [Sn(NH_3)_4]^{2+}{}_{(aq)} + 4OH^-{}_{(aq)} \rightarrow Zn_2SnO_{4(s)} + 2H_2O_{(l)} + 4HN_{3(aq)} \quad (3)$$

The samples were annealed at temperature range, 100°C-250°C in order to remove the water of crystallization, thereby obtaining adherent transparent deposit on the substrates as given in equation (4).

$$Zn_2SnO_{4(s)} + 2H_2O_{(g)} \xrightarrow[100°C\text{-}250°C]{\text{Heat}} Zn_2SnO_{4(s)} \quad (4)$$

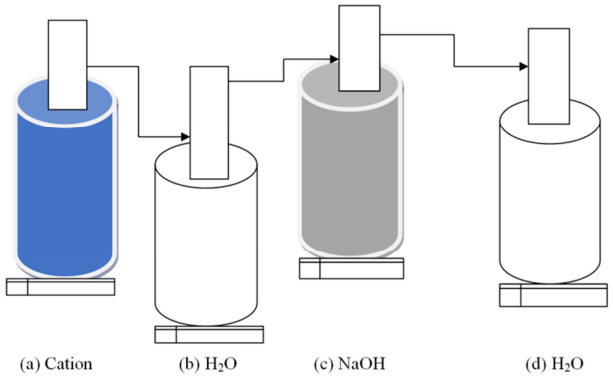

Figure 1. *The stages of SILAR deposition process.*

Cationic precursorb. Ion exchange waterc. Anionic precursor d. Ion exchange water.

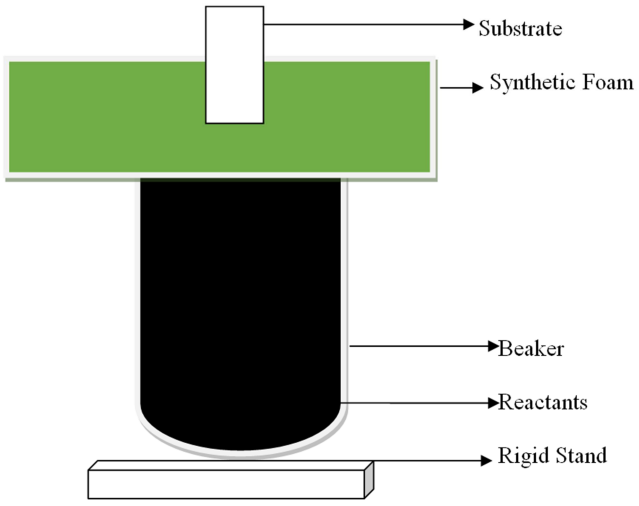

Figure 2. *Set-up ofSolution Growth Technique [7].*

Table 2. *The different parameters for the deposition of ZnO:SnO_2 thin films of 0.2M solution of $SnCl_2$.*

Samples	Annealing Temp. (°C) (for 1 hour)	$ZnCl_2$ Conc. (mol)	NH_3 Conc. (mol)	NaOH Conc. (mol)	$SnCl_2$ Vol.(ml)	NH_3 Vol. (ml)	NaOH Vol. (ml)	Dep. Temp (NaOH) (°C)	Dep. Time (hr)
A_{23}	100	1.00	3.00	2.00	20.00	4.00	10.00	60.00	8.00
A_{24}	200	1.00	3.00	2.00	20.00	4.00	10.00	60.00	8.00
A_{25}	250	1.00	3.00	2.00	20.00	4.00	10.00	60.00	8.00
A_{26}	200	1.00	3.00	2.00	20.00	4.00	10.00	60.00	8.00
A_{27}	250	1.00	3.00	2.00	20.00	4.00	10.00	60.00	8.00
A_{28}	200	1.00	3.00	2.00	20.00	4.00	10.00	60.00	8.00
A_{29}	150	1.00	3.00	2.00	20.00	4.00	10.00	60.00	8.00

3. Results and Discussion

It was observed that when 5ml of 3M solution of 99% NH_3 was allowed to react with 20ml of 0.2M solution of $SnCl_2$, a white precipitate was initially formed, which dissolved in excess NH_3 solution forming a colorless transparent solution of $[Sn(NH_3)_4]^{2+}$ (tetra-ammine complex tin ion).

When 15ml of 1M solution of NaOH at 60°Cwas added to the solution of $[Sn(NH_3)_4]^{2+}$, the white precipitate reappeared. When the substrates containing ZnO, thin films deposited by SILAR method were inserted into the mixture, it took 8 hours for $ZnO:SnO_2$, to deposit optimally depending on the deposition parameters such as pH, volumes of

reactants and the concentrations.

It must be stated clearly here that the chosen parameters such as concentration, volume, temperature, pH and time of deposition were found largely by trial and error and we have no means of guaranteeing that they lead to the best possible optical and electrical properties [8].

From literature, zinc salts have the capabilities of forming complex solutions with ammonia. This makes ammonia solution a suitable complexing agent for the deposition of the above named oxide thin films.

The concentration of Zn^{2+} decreases with increasing concentration of complexing agents.

Thus, the rates of reaction and the formation of precipitates are reduced, leading to a larger terminal thickness of the films. It should be noted that the deposition of these oxide thin films is pH-dependent. OH^- from NaOH, did not take part in the complex formation therefore, the addition of OH^- precipitated the corresponding hydrous oxides of the individual ions which were deposited on the substrates. In the case of OH^- ions taking part in the reaction processes, its addition increases the pH value, making the complex more stable, thereby reducing the concentration of free radicals.

The suitable pH value for this work is 11 as detected by the piston pH meter for the four deposited alloyed sample. The deposited samples were subjected to heat treatment from 100°C-250°C. using Master Chef Annealing Machine. The transmittance of the samples of the deposited oxides were measured using UV-1800 series double beam spectrophotometer.

3.1. Elemental Composition and Thickness Measurement

It is often necessary to determine the elements and the thicknesses of the thin film samples. In this work, elemental compositions and the thicknesses of the samples were determined using Rutherford Back Scattering equipment: 2.2MeV alpha beam, obtained from CERD Ion Beam Analysis (IBA) Facility With Model: NEC 5SDH 1.7 MV Pelletron Tandem Accelerator equiped with a Radio Frequency Charge Exchange Ion Source Alphatron.

The samples A_{24} and A_{25} of ZnO:SnO_2 thin films annealed at 200°C and 150°C respectively have 6.68% of zinc, 13.60% of tin, 79.72% of oxygen with thickness of 190.0nm and 5.14% of zinc, 13.42% of tin, 81.45% of oxygen with thickness of 388.52nm. These are oxygen reach films which may be due to surface hydration or hydrolysis [9]. These are shown in Figure 3 and Figure 4 and respectively itemized in Table 3 and Table 4.

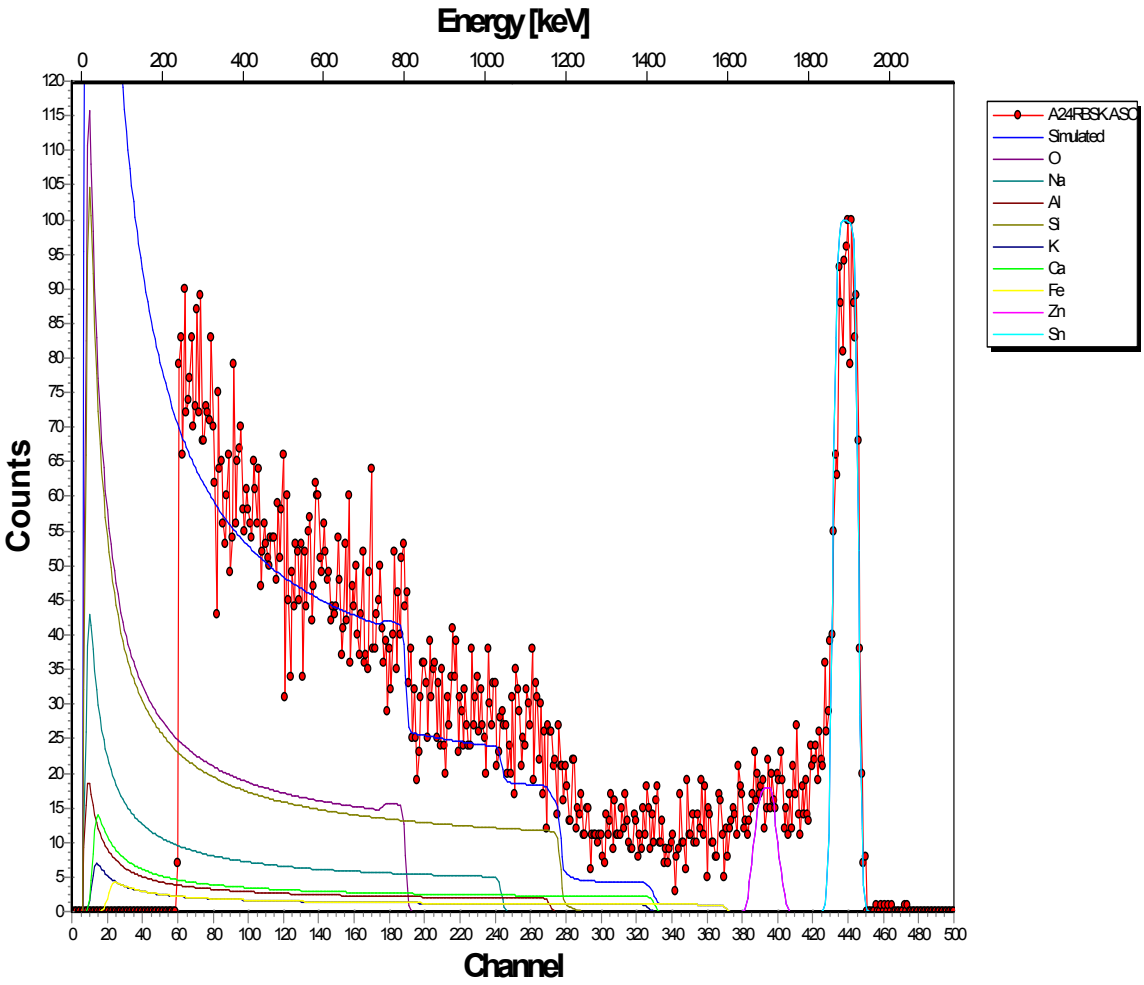

Figure 3. *The composition of sample A_{24} with thickness, 190.0nm as measuredbyRutherford backscattering spectroscopy.*

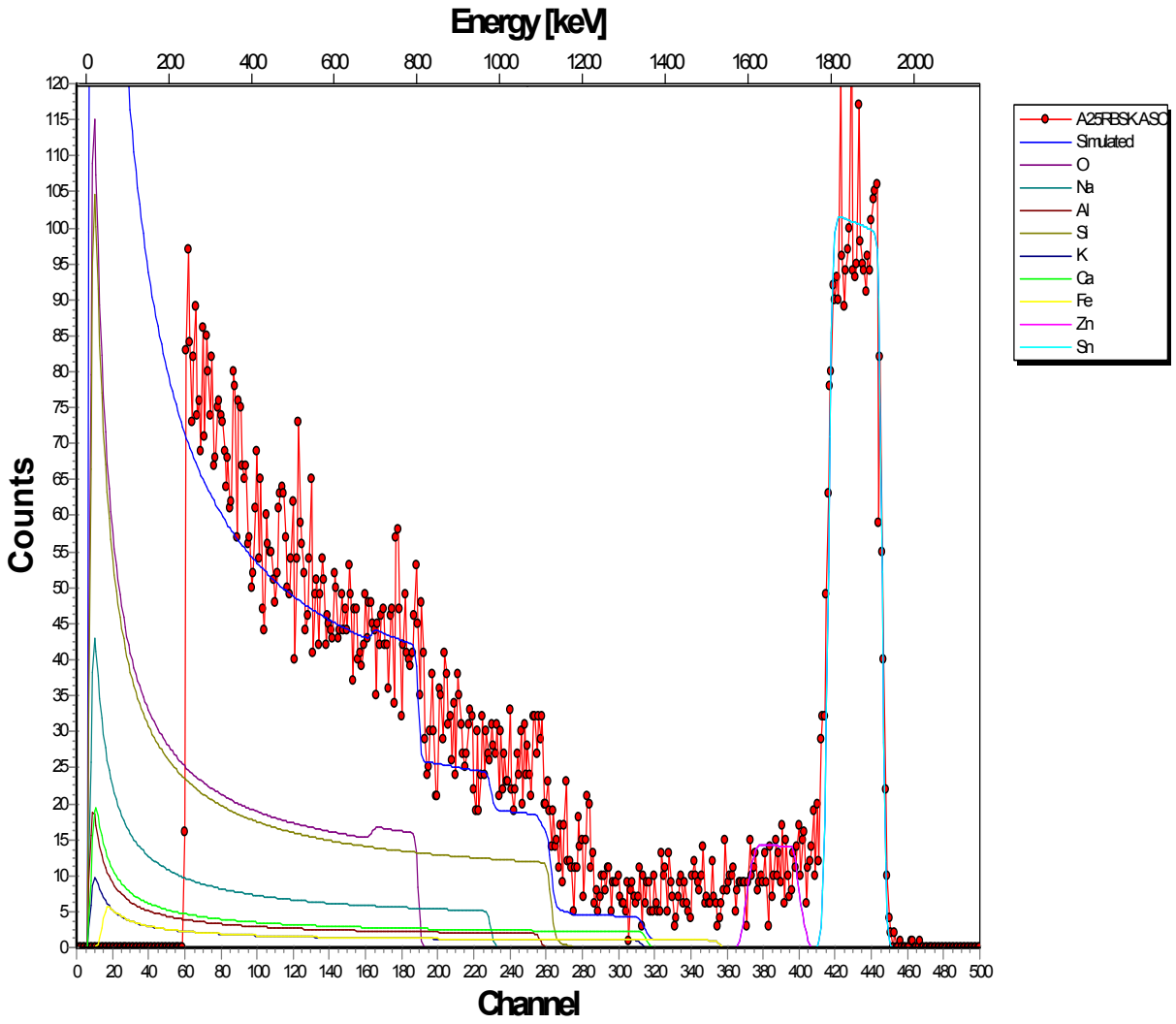

Figure 4. *The composition of sample A_{25} with thickness, 388.52nm as measured by Rutherford backscattering spectroscopy.*

Table 3. *The elements in deposited sample A_{24} of $ZnO:SnO_2$ alloyed thin films.*

Elements	Layer(1)%Comp.	Layer(2)%Comp.
O	79.72	56.00
Ca	-	1.83
Fe	-	0.52
Na		12.60
Al	-	0.53
Si	-	28.00
Zn	6.68	-
Sn	13.60	

Table 4. *The elements in deposited sample A_{25} of $ZnO:SnO_2$ alloyed thin films.*

Elements	Layer(1)%Comp.	Layer(2)%Comp.
O	81.45	56.00
Ca	-	1.83
Fe	-	0.52
Na		12.60
Al	-	0.53
Si	-	28.00
Zn	5.14	-
Sn	13.42	

3.2. Structural Properties of the Deposited Alloyed Thin Films of ZnO:SnO_2 of Sample A_{24}

The XRD analysis was carried out using X-ray diffractometer modeled GBC Enhanced Mini Material Analyzer (EMMA). XRD pattern gives information relative to the nature and structure of the alloyed thin films of ZnO:SnO_2 prepared at 60°C of sodium hydroxide solution. The crystallite size given in Table 5 is obtained using Debye-Scherers equation in (5) [10].

$$D = \frac{k\lambda}{\beta Cos\theta} \quad (5)$$

where k is the shape factor (k= 0.9), D is the grain size or average crystallite size, λ is the wavelength of CuKα radiation used (λ = 1.54Å), β is the experimentally observed diffraction peak width at half maximum intensity (full width at half maximum FWHM) and θ is the Bragg's diffraction angle.

Table 5. *X-rayDiffraction Results of ZnO: SnO_2 alloyed thin films sample A_{24}.*

Sample A_{24}	2θ (degree)	d-spacing (Å)	FWHM (radian)	Grain size (nm)	Count
ZnO: SnO_2	19.552	4.537	0.350	40.18	45
	22.811	3.895	0.153	92.41	130

Figure 5, shows that the XRD pattern of ZnO:SnO_2 alloyed thin films of sample A_{24} has two diffraction peaks at 2θ = 19.522° and 22.811°, which indicates the crystalline nature of the alloyedof ZnO:SnO_2 at 60°C of NaOH solution. From the observed peaks, the grain sizes were found to be 40.522nm and 92.41nm. From the XRD results, a new compound alloyed thin film known as di-zinc tin (IV) oxide with cubic crystal system was formed similar to spinel structure [11]. It has a chemical formula; Zn_2SnO_4.

From the data of the XRD peaks the lattice parameters of ZnO:SnO_2 alloy thin films of sample A_{24} are a = b = c = 8.610Å.

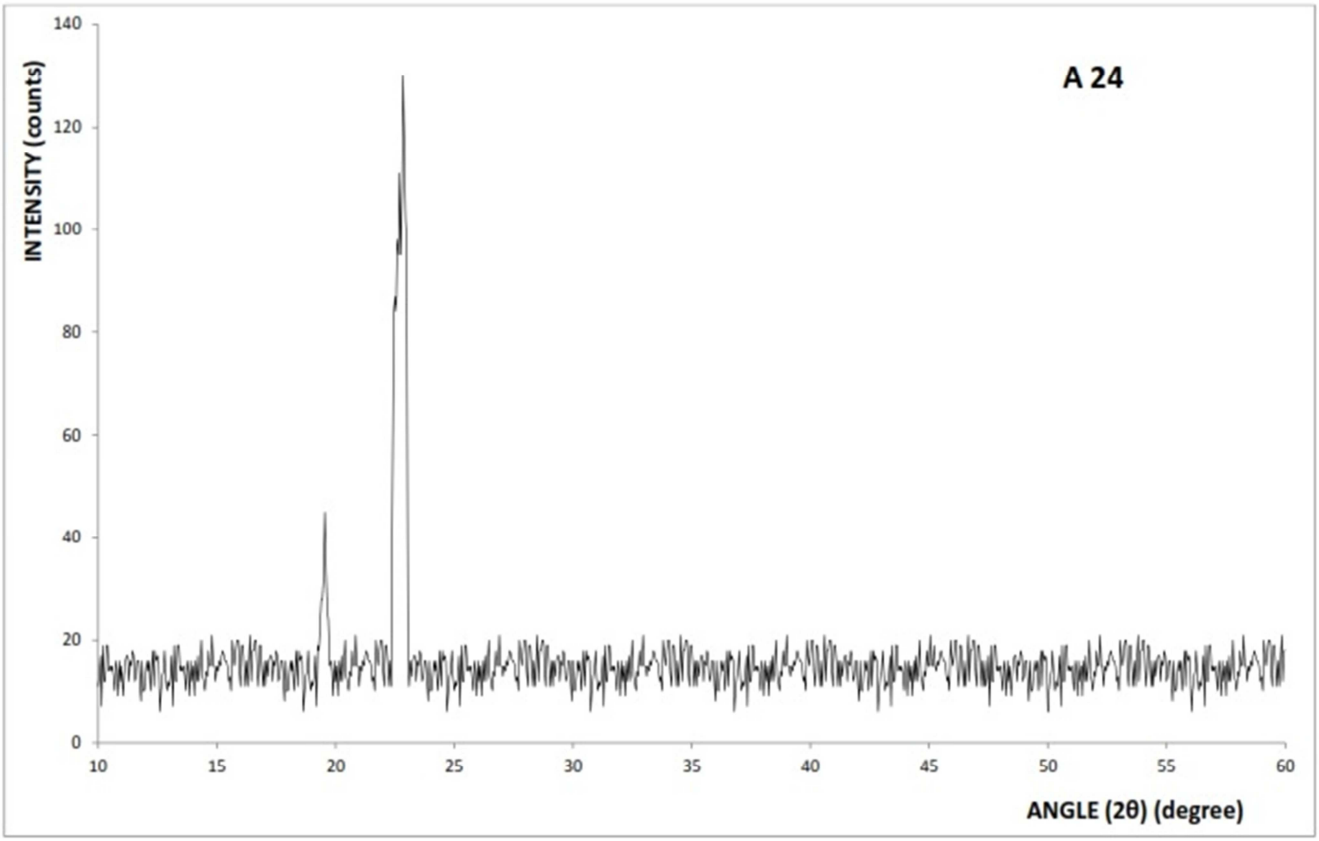

Figure 5. *XRD pattern of ZnO:SnO_2 alloyed thin films of sample A_{24} at 60°C of NaOH solution.*

3.3. Microstructure of ZnO:SnO_2 of the Deposited Sample A_{24}

Microstructure of the alloyed thin films of ZnO:SnO_2 were determined using electron microscope Phenom Prox, Model number MVEO16477830 manufactured by Phenom World Eindhoven Netherland. The process of analysis is through back scattering electron imaging method. Figure 6 shows that sample A_{24} of ZnO:SnO_2 contains non-agglomerated spherical morphology. It have defined coated thin films which is due to reduction in hydrolysis and condensation of the sample. It has a unity structure [12]. Sample A_{24}, has rough texture and granular microstructure.

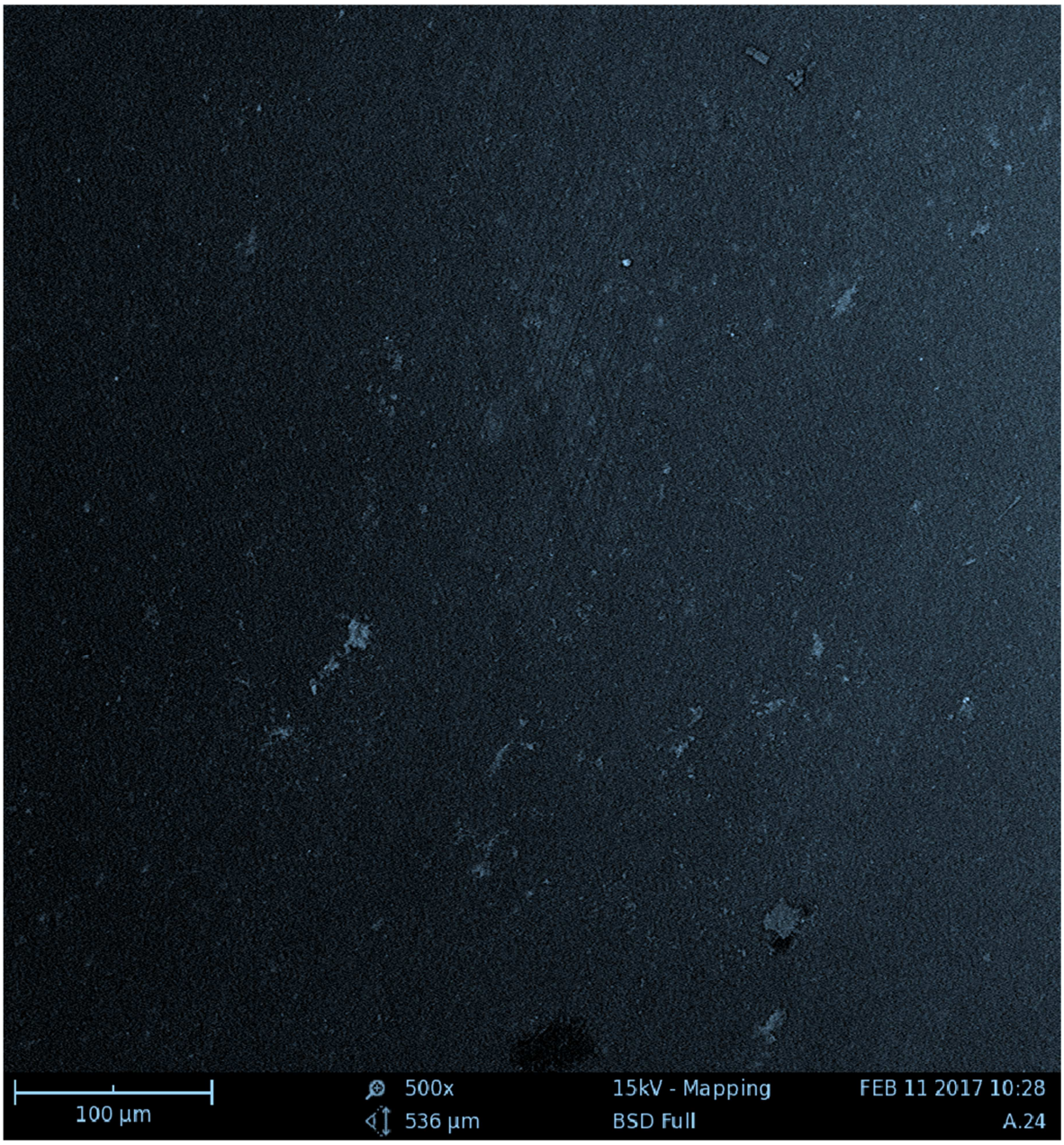

Figure 6. *Scanning microscopy of sample A_{24} of $ZnO:SnO_2$ alloyedthinfilms.*

3.4. Optical Properties

The optical properties of the deposited films were carried out using UV-1800 double beam spectrophotometer of wavelength range 190nm-1200nm. The transmittance spectra were measured directly from the spectrophotometer, other optical properties were determined using their appropriate equations.

The transmittance spectrum shows that the films of samples A_{24} and A_{25} have good transparency in the UV, visible and near infrared regions of electromagnetic spectrum. Sample A_{24} has transmittance range 42%-58%at the wavelength between 320nm-360nm in the UV region. Sample A_{25} has transmittance range 59%-62% at the wavelength between 320nm-400nm in the UV region. The transmittance of sample A_{24} increases to its maximum value of 61% at wavelength, λ=450nm in the visible region. The transmittance of sample A_{25} increases as wavelength increases up to a certain value 63% at wavelength λ=480nm after which it falls and rises up to maximum value of 68% at wavelength, λ=1080nmin the near infrared region of electromagnetic spectrum. The transmittance of sample A_{24} increases to its maximum value of 61% at wavelength, λ=450nm in the visible region.

Its transmittance falls as wavelength, increases within the visible and has linear characteristics in the near infrared region of electromagnetic spectrum as shown in Figure 7. This makes these alloyed films good materials for UV filter [13]. It is also a good material for solar cell application and equally a good material for transparent electrode in optoelectronic applications.

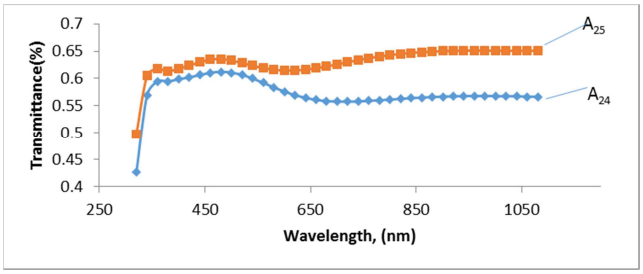

Figure 7. *Graph of transmittance against wavelength for ZnO:SnO_2 alloyed thin films of samples A_{24} and A_{25} at constant temperature of 60°C of NaOHsolution.*

The graph of the absorbance in Figure 8, is obtained from the equation

$$A = log_{10}\frac{1}{T} \quad (6)$$

where A is the absorbance and T is the transmittance

Samples A_{24} and A_{25} of ZnO:SnO_2 have decreasing absorbance from the UV, 0.356 and 0.31 respectively at wavelength 320nm which are their maximum values. The minimum absorbance of 0.21 at the visible region at wavelength of 480nm and 0.153 is obtained at the near infrared regions of electromagnetic spectrum at wavelength 1080nm. The absorbance of sample A_{24} in the visible region decreases continually from 400nm to near infrared region of electromagnetic spectrum. This makes the material veritable in the area of optical window applications [14].

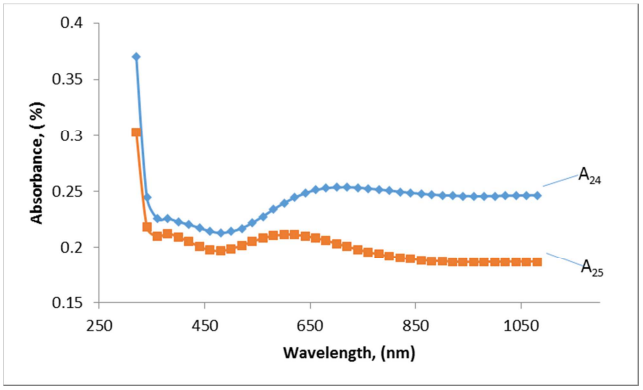

Figure 8. *Graph of absorbance against wavelength for ZnO:SnO_2 alloyed thin films of samples A_{24} and A_{25} at constant temperature of 60°C of NaOH solution.*

The graph of thereflectance in Figure 9, is obtained from the equation

$$R = 1 - (T + A) \quad (7)$$

where T is the transmittance, A is the absorbance and R is the reflectance.

Samples A_{24} and A_{25} have low reflectance of 0.175 to 0.204 and 0.165 to 0.20 respectively. It decreased as the wavelength increases from the UV through the visible to the near infrared regions of electromagnetic spectrum.

This makes ZnO:SnO_2 alloy thin film useful in the area of multilayer solar control coating. The coating allows the visible part of the spectrum in, but either reflects the infrared (IR) radiation back into the room (energy-saving) or does not allow the infrared radiation into the room (heat-protection) depending on which side of the window has the coating.

This material can serve as front contact for solar cells or liquid crystal displays, flat panel displays for optoelectronic applications.

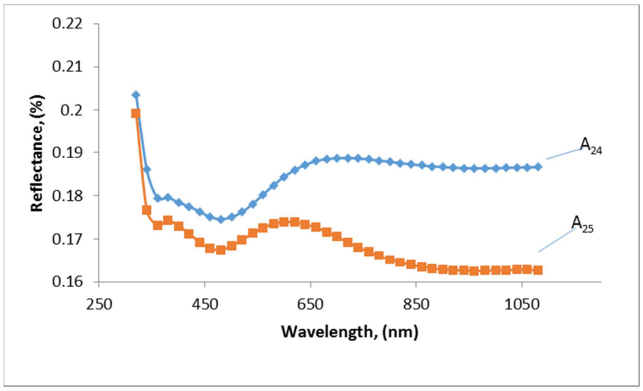

Figure 9. *Graph of reflectance against wavelength for ZnO:SnO_2 alloyed thin films of samples A_{24} and A_{25} at constant temperature of 60°C of NaOH solution.*

The graph of the refractive index in Figure 10 is obtained from the equation,

$$n = \frac{1+\sqrt{R}}{1-\sqrt{R}} \quad (8)$$

where n is the refractive index and R is the reflectance.

The refractive index, of sample A_{24} falls from its maximum value of 2.65 at λ= 320nm continuously to its minimum value of 2.45 in the visible region at wavelength, λ=460nm and rises as wavelength increases until it reaches a wavelength, ʎ=1080nm.

Sample A_{25} decreases from 2.63 at λ= 320nm and falls sharply to the visible region at wavelength equal to 2.40 and rises, then falls as wavelength increases to its minimum value of 2.35 at ʎ=1080nm.

The behaviour of these two samples of ZnO:SnO_2 makes it possible for use in the area of multiplier solar control coating applications where materials with high refractive index are required.

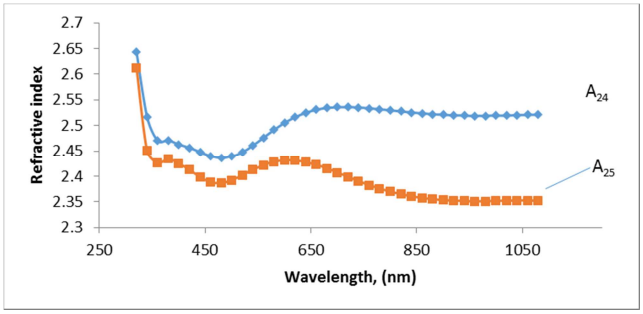

Figure 10. *Graph of refractive index against wavelength for ZnO:SnO_2 alloyed thin films of samples A_{24} and A_{25} at constant temperature of 60°C of NaOH solution.*

The optical energy band gap is obtained in kspace using equation 3.2, the equation of the absorption coefficient relationship for the direct transition, given by [15]. By extrapolating the linear portion of the plot $(\propto h\upsilon)^2$ against photon energy hυ at $(\alpha h\upsilon)^2 = 0$ energy band gap of the samples are shown in Figure 11.

$$(\propto h\upsilon)^2 = A(hv - E_g) \quad (9)$$

∝is the absorption coefficient, E_g is the energy band gap, hv is the photon energy and A is the constant that depends on the deposited sample.

A direct band gap value of 3.34 ± 0.05eV are obtained for samples A_{24} and A_{25}

The wide band gap obtained in this work makes the ZnO:SnO_2 a good material for the production of laser diodes and light emitting diodes (LEDs)

It can also be used to produce field effect transistors where P-n junction may not be required. If produced for solar application, it will serve as absorption layer [14].

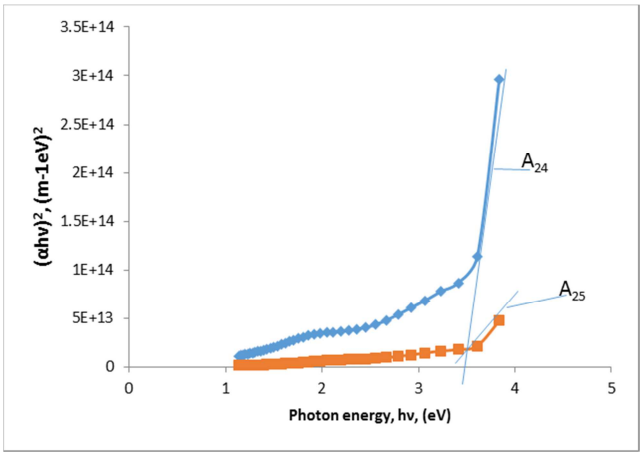

Figure 11. *The Graph of $(\alpha h\upsilon)^2$ against photon energy hυ for ZnO:SnO_2 alloyed thin films of samples A_{24} and A_{25} at constant temperature of 60°C of NaOH solution.*

4. Conclusion

ZnO:SnO_2 alloyed thin filmswere deposited on glass substrates using two solution based methods: successive ionic layer adsorption and reactionand solution growth technique at constant temperature of 60°C of NaOH solution while zinc complex ions were kept at room temperature of 20°C. NH_3 solution was used as complexing agent.

The deposited samples were annealed between 100°C-250°C.

The alloyed thin films exhibited appreciable good transmittance from the ultraviolet region, through the visible to near infrared regions of electromagnetic spectrum. Other optical properties of the samples were determined using appropriate equations. Direct average energy bandgap of 3.34±0.05eV was obtained for ZnO:SnO_2 alloyed thin films. The other properties investigated are absorbance, reflectance, optical conductivity, optical constants and absorption coefficient.

These material alloy thin films prepared under this condition with wide energy band gap, high transparency in the visible region can be found useful in passive applications as dazzling coating galvanizing, electroplating, cold and heat windows, ceramics, solar thermal-energy collector, selective absorbing layer and active solar cell applications, semiconductor materials, for optoelectronic applications, UV light emitting devices, laser diodes, sensors, and optical communications etc.

This alloyed thin film, has higher breakdown voltage, ability to sustain large electric field, low electronic noise, stable at higher temperature and high power operation. Due to their thermal stability, they can be found useful in the area of ceramics production, anti-corrosion material in iron and other metallic materials.

Acknowledgements

I am grateful to the laboratory officials of Obafemi Awolowo University Ile-Ife in the person of Prof. E. I. Obiajunwa, the Director Centre for Energy Research and Development and Mr. Akinola E. A., of Central Science Laboratory in the successful characterization of this work.

Also I wish to give a big thanks to Mr. Ofiwe, C. U., of National Agency for Science and Engineering Infrastructure (NASENI) Akure and Mr. Isa Abu of Chemical Engineering Department of Ahmadu Bello University, Zaria for their various roles in realizing this work..

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