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Influence of pH on Tin Doped Zinc Selenide(SnZnSe) via Electrochemical Deposition Technique

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Abstract : Synthesisof Tin doped Zinc Selenide (SnZnSe) thin filmsmaterials was carried out using the cationic precursor, which was an aqueous solution of 0.035 mol solution of $ZnSO_4.7H_2O$while the anionic precursor was 0.1mol solution of selenium metal powder was prepared by dissolving with 4ml of Hydrogen chloride (HCl and Ammonium (NH_3) was use to varies the pH of the solution. The XRD of the films deposited on FTO substrates at different pH shows the reflection peaks at (220), (221), (300), (310), (311), (222) and (320) with the lattice constant of a = 7.189Å. SEM shows random distribution of tiny nano-grains on the substrate, the nano-grains were observed to agglomerate due to the presence of large free energy characteristic of small particles. The optical band gap of the deposited material increases from 2.0 eV – 2.3 eV as the pH increases from 8, 9, 10 and 11.
Key word : Electrochemical deposition, FTO, pH, Thin films, $ZnSO_4.7H_2O$and $SnCl_2.2H_2O$.

1. Introduction

Thin films and its devices play an important role in the development of modern technology [1-3]. Thin films are three dimensional form of solid material, whose one dimension, called the thickness, is much smaller than the other two dimensions[4]. Thin films are formed by atom to atom or molecule to molecule condensation process[5-6]. Semiconductor thin films have been the basis of the development of a wide variety of applications such as high-speed transistors, solar cells, solid-state lighting devices, sensors, information storage devices [7-9], etc. Fundamental knowledge of the basic properties of compound semiconducting thin films and understanding their interfaces with other device are very important for the recent technological progress [10-11]. In industrial, scientific and technical applications of thin films, their physical properties such as optical, chemical, electrical properties are investigated which results in variety of devices such as solar energy devices, xerography, switching devices, high resolution lithography, optic memories, photo-detectors [12] etc. Thin films deposition for optical, electronic and optoelectronic device application has become an industry in most advanced countries using highly technological, sophisticated and very expensive techniques [13-14]. However,

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in third world countries, the high technological and sophisticated techniques are not easily achieved because of their complexity and the poverty of the third world countries [15]. Hence, considerable efforts are put into developing simple and cheap techniques of depositing the films [16-17]. The solution growth technique offers the simplest, cheapest, most economical and affordable method of depositing thin films like Halide and Chalcogenides[18, 6, 31].Zinc selenide (ZnSe) shows unique optical properties exhibiting some potential applications, such as blue-green light emitting diodes, photo-luminescent and electro-luminescent devices, lasers, thin film solar cell, nonlinear optical crystal and infrared optical material [19]. ZnSe and its lattice matched ternary alloys have been regarded as useful II–VI compound semiconductors for optoelectronic and photo electronic devices. For the energy ranges from visible to ultraviolet.ZnSe based materials are structured the first manifestation of the blue-green laser in 1991 [20]. ZnSe has been a material of choice for blue diode lasers and photovoltaic solar cells since its bulk band gap is 2.7eV which can be tuned by adding impurities[36]. ZnSe thin films have been obtained by serval preparation approaches including sputtering [22-23], molecular beam epitaxy [24], pulsed laser deposition [25], chemical vapordeposition [26], successive ionic layer adsorption and reaction [27], spray pyrolysis [28], chemical bathdeposition (CBD) [22, 29] and electrodeposition [10,36]. Among these techniques, electrochemical deposition offers several advantages: it is relatively economical; it can be used on a large scale.

In this research, we reported on influence of pH on tin doped zinc selenide (SnZnSe) via electrochemical deposition technique, in order to study kind of transitions on the structural, morphological and optical properties and the elemental composition of the deposited material for photovoltaic application.

2 Experimental procedure

2.1 Materials and Methods

The chemicals used for this research were analytical grade and they were purchased from Sigma-Aldrich. The growth of SnZnSe thin film semiconductor material included Zinc tetraoxosulphate (VI) heptahydrate ($ZnSO_4.7H_2O$), Tin chloride ($SnCl_2.2H_2O$), Selenium metal powder (Se), Hydrogen Chloride (HCl) and Ammonium (NH_3). Electrochemical deposition technique (ECD) was used in this work which involves the deposition of any substance on an electrode as a result of electrolysis which is the occurrence of chemical changes owing to the passage of electric current through an electrolyte. This process involves oriented diffusion of charged growth species through a solution when an external field is applied and reduction of charged growth species at the growth or deposition which also serves as an electrode. The electrochemical bath system is composed of a source of cation (i.e $SnCl_2.2H_2O$, $ZnSO_4.7H_2O$ for Sn^{2+}, Zn^{2+}), a source of anion (i.e Selenium metal powder for Se^{2-}), deionized water all in 100ml beaker, magnetic stirrer was used to stir the reaction bath. A power supply was used to provide electric field (DC voltage), a conducting glass was used as the cathode while the anode was carbon and fluorine electrode . Finally, uniform deposition of thin films by electrochemical deposition technique was achieved.

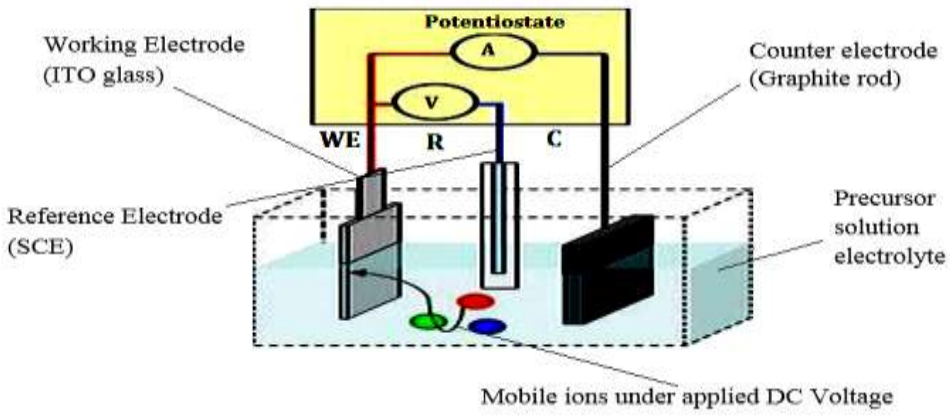

Fig. 1: Schematic diagram of electrochemical deposition technique [36]

2.2 Substrate cleaning procedure

We attention paid on the cleaning and activating the substrate surface. The selection of the substrate and methods used for the cleaning of the surface of the substrate are very important in the formation of thin films with reproducible process. Nature of the material, size, surface roughness and cleanliness of the substrate play an important role in the formation of the film and its properties such as adhesion, pin-hole density, porosity, film microstructure, morphology and mechanical properties. Substrate cleaning usually involves removing surface contaminations like greasy particles, dust, metals etc. The cleaning process varies with the substrate being cleaned. The contaminants present in the surface of the substrate can be broadly categorized into two types, namely organic and inorganic. Organic contaminants can be easily removed by emulsifying the surface of the substrate with washing solutions. However, for the removal of inorganic contaminants a direct mechanical approach is needed when they are in particle form. A number of procedures are available for this purpose such as immersing in solvents, ultrasonic cleaning, electronic discharge etc. The systematic procedure used for cleaning the substrates is; Transparent Fluorine doped tin oxide (FTO) coated glass substrates with a sheet resistance of 10Ω/m was used as substrate for deposition. The glass substrates were coated only on one side and this conducting side of the glass was detected by the use of a digital multimeter which gave reading as the non-conducting side does not give any reading for the deposition of thin films materials. Hand gloves were used to handle the substrates to avoid contamination. The substrates were dipped in acetone, methanol, rinsed with distilled water and later ultrasonicated for 30min in acetone solution after which they were rinsed in distilled water and kept in an oven to dry. All the prepared substrates were kept in air-tight container.

2.3 The Synthesis of SnZnSe thin films

The synthesis of tin doped Zinc selenide (SnZnSe) thin films materials was carried out using the cationic precursor which was an aqueous solution of 0.035 mol solution of $ZnSO_4.7H_20$while the anionic precursor was 0.1mol solution of selenium metal powder was prepared by dissolving with 4ml of Hydrogen chloride (HCl) while Ammonium (NH_3) was use to varies the pH of the solution. This was to ensure a uniform deposition. The electrochemical deposition bath system is composed of a source of cation$SnCl_2.2H_2O$, $ZnSO_4.7H_2O$ for Sn^{2+}, Zn^{2+}), a source of anion (i.e Selenium metal powder for Se^{2-}), distilled water all in 100 mL beaker, and a magnetic stirrer which was used to stir the reaction bath. A power supply was used to provide electric field (DC voltage), a Fluorine doped tin oxide (FTO) was used as the cathode while the anode was carbon and fluorine electrode. The molar concentration of the solution were varies in the process of the experiment (See table 1) for detailed.

Table 1: Variations of growth materials

Sample	SnCl2.2H2O (mi)	ZnSO4.7H2O (ml)	Se (ml)	pH	Time (Sec)	Voltage (V)
PT1	10	20	20	8	25	10
PT2	10	20	20	9	25	10
PT3	10	20	20	10	25	10
PT4	10	20	20	11	25	10

2.4 Characterization Of The Films.

The synthesizer films were characterized for their optical properties, electrical properties, Scanning electron microscope and structural properties. The structural characterization of the films was carried out using Bruker D8 Advance X-ray diffractometerwith Cu Kα line (λ= 1.54056Å) in 2θ range from 10° - 90° the instrument helped in determining the type of lattice crystal and intensities of diffraction peaks, with the help of data base software supplied by the international center of diffraction data. The quantitative analysis of the films were carried out using Energy Dispersive X-ray Analysis (EDX) for the thin films to study the stoichiometry of the film. This unit is attached to the Zeiss Scanning electron microscope (SEM). When a beam of electrons strikes the specimen, some of the incident electrons excite the atom of the specimen which emits X-ray on returning to the ground state. The energy of the X-ray is related to the atomic number of the excited element. Lithium drifted Si-diode, held at liquid nitrogen temperature is used as a detector of the X-rays. JEOL-JSM 7600F Japan was employed in the present investigation. The electrical characterizations of the films were measured using a four point probe. The absorbance spectral of the films was obtained in UV- visible NIR using

UV-1800 visible spectrophotometer. UV-visible spectrophotometer uses the principle that when a beam of electromagnetic radiation of initial flux I is incident on a transparent object, it is transmitted. Some part of the incident flux could be absorbed for an absorbing medium while some part could be reflected. Various other parameters from the absorbance include: Transmittance, Reflectance, Refractive index, Optical Thickness, Coefficient of absorption, Extinction coefficient, Optical conductivity and dielectric constants were derived using the formula below

(a) From the law of conservation of energy we obtained,

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

Where A is the absorbance, R is the Reflectance, and T is the transmittance.

$$d = \frac{n\lambda}{2sin\theta} \quad (2)$$

$$D = \frac{0.94\lambda}{\beta Cos\theta} \quad (3)$$

$$K = 2(\frac{In}{\lambda})^{\frac{1}{2}} = 0.94 \quad (4)$$

3. Results and Discussions

3.1 Structural Analysis Of Tin doped Zinc Selenide (SnZnSe) Thin Films

The XRD pattern of SnZnSe thin films deposited on FTO substrates at different pH of 8, 9, 10 and 11. From fig. 4.11, the reflection peaks shown at (220), (221), (300), (310), (311), (222) and (320) which correspond to the following angle respectively (26.00^0), (31.00^0), (34.00^0), (37.03^0), (52.00^0), (61.00^0) and (66.05^0) are indexed to face-centred cubic structure of SnZnSe [JCPDS card no. 01-088-2345] reported by [5, 9-13, 16, 18-19, 29-30, 35-36] and there is no reported research findings on SnZnSe before this report. The un-indexed peaks could have possibly resulted from the FTO substrates used for deposition. The lattice constant a = 7.189Å was obtained using (equ. 2). From Fig. 2 the higher peaks could be resulted to the fact that the films thickness increases with increase in pH, thus creating larger surface area for photovoltaic devices and solar cell activities. The average crystallite size of the films was determined using the Debye-Scherer's formula (equ.3 and 4). See table 2 for the calculated crystallite or grain sizes and dislocation density for the films deposited at different pH of 8, 9, 10 and 11

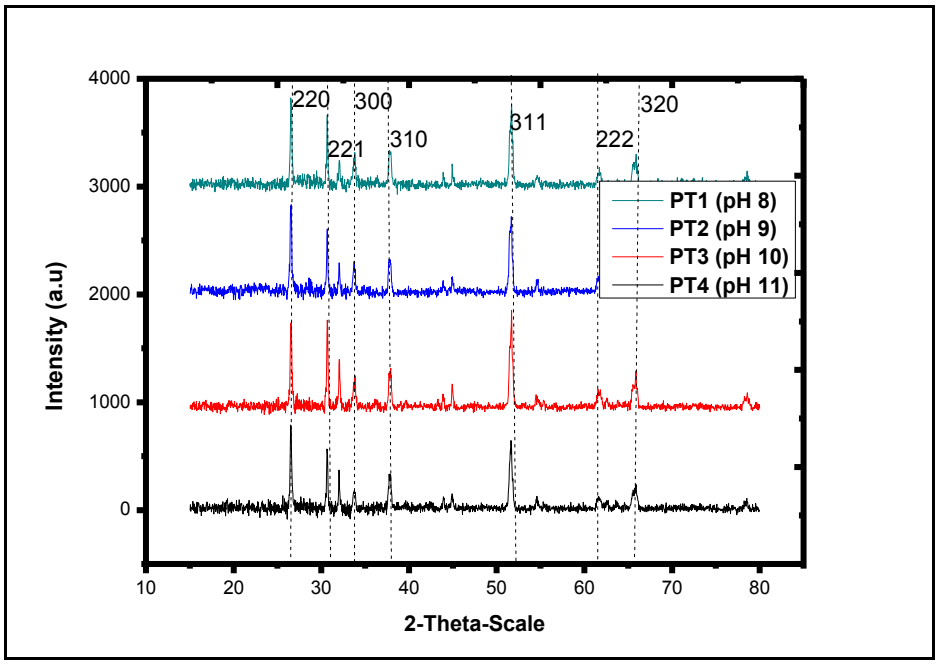

Fig.2: XRD pattern of Tin doped Zinc Selenide (SnZnSe) Thin Films deposited at different pH

Table 2: Structural parameters of Tin doped Zinc Selenide (SnZnSe) Thin Films deposited at different pH

Sample	**2θ (degree)**	**d (spacing) Å**	**Lattice constant (*Å*)**	**(β) FWHM**	**(hkl)**		**Grain Size(D) nm**	**Dislocation density,σ lines/m^2**		
PT1 (pH 8)	26.00	3.52076	7.189	0.49450	220		3.00556	0.11069		
	31.00	2.96364		0.49450	221		3.03906	0.10827		
	34.00	2.70888		0.49450	300		3.06234	0.10663		
	37.03	2.49407		0.49450	310		3.08838	0.10484		
	52.00	1.80668		0.49450	311		3.25829	0.09419		
	61.00	1.56047		0.49450	222		3.39883	0.08656		
	66.05	1.45319		0.49450	320		3.49286	0.08196		
PT2 (pH 9)	26.00	3.52076	7.189	0.47835	220		3.10703	0.10358		
	31.00	2.96364		0.47835	221		3.14166	0.10131		
	34.00	2.70888		0.47835	300		3.16573	0.09978		
	37.03	2.49407		0.47835	310		3.19265	0.09810		
	52.00	1.80668		0.47835	311		3.36829	0.08814		
	61.00	1.56047		0.47835	222		3.51358	0.08100		
	66.05	1.45319		0.47835	320		3.61079	0.07669		
PT3 (pH 10)	26.00	3.52076	7.189	0.38931	220		3.81765	0.06861		
	31.00	2.96364		0.38931	221		3.86020	0.06710		
	34.00	2.70888		0.38931	300		3.88977	0.06609		
	37.03	2.49407		0.38931	310		3.92285	0.06498		
	52.00	1.80668		0.38931	311		4.13867	0.05838		
	61.00	1.56047		0.38931	222		4.31718	0.05365		
	66.05	1.45319		0.38931	320		4.43662	0.05080		
PT4 (pH 11)	26.00	3.52076	7.189	0.45460	220		3.26936	0.09355		
	31.00	2.96364		0.45460	221		3.30580	0.09150		
	34.00	2.70888		0.45460	300		3.33112	0.09011		
	37.03	2.49407		0.45460	310		3.35945	0.08860		
	52.00	1.80668		0.45460	311		3.54427	0.07960		
	61.00	1.56047		0.45460	222		3.69714	0.07315		
	66.05	1.45319		0.45460	320		3.79943	0.06927		

3.2 Surface Morphological and EDX Analysis of Tin doped Zinc Selenide (SnZnSe) Thin Films deposited at different pH

The surface morphology of SnZnSe thin film grown on fluorine doped tin oxide (FTO) substrates at different pH of 8, 9, 10 and 11 as shown in fig. 3. It shows random distribution of tiny nano-grains on the substrate, the nano-grains were observed to agglomerate due to the presence of large free energy characteristic of small particles. As the pH of the solution increases from 8, 9, 10 and 11 the nano-grain become more densely packed. The grown films were homogenous and without cracks [29-30, 35]. Fig. 4&5 shows the energy dispersive X-ray spectra of SnZnSe thin film grown at pH of 8 and 11 respectively.

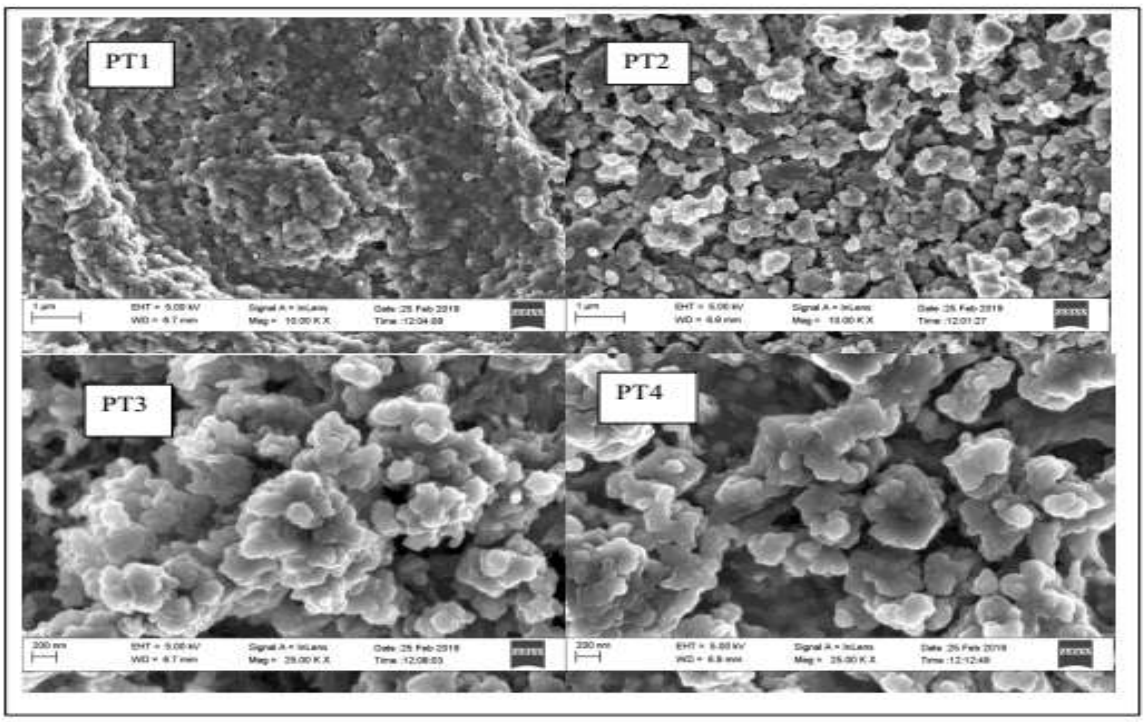

Fig. 3: SEM micrograph of Tin doped Zinc Selenide (SnZnSe) Thin Films deposited at different pH

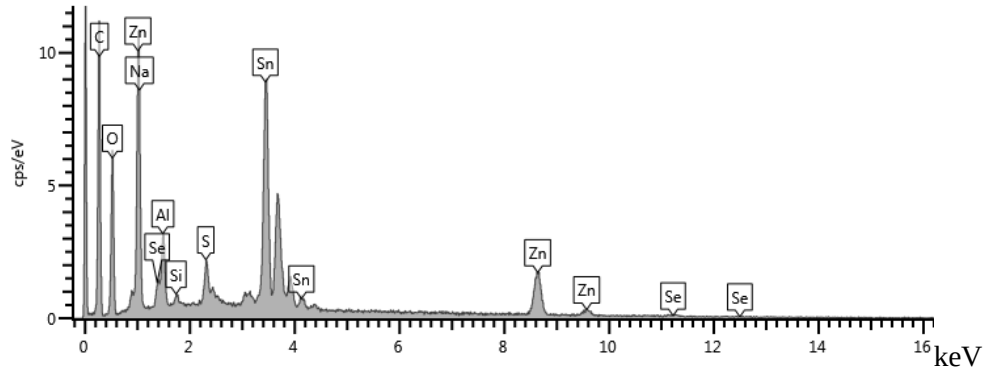

Fig. 4: Energy Dispersive X-ray Spectra (EDX) of Tin doped Zinc Selenide (SnZnSe) Thin Films deposited at pH of 8

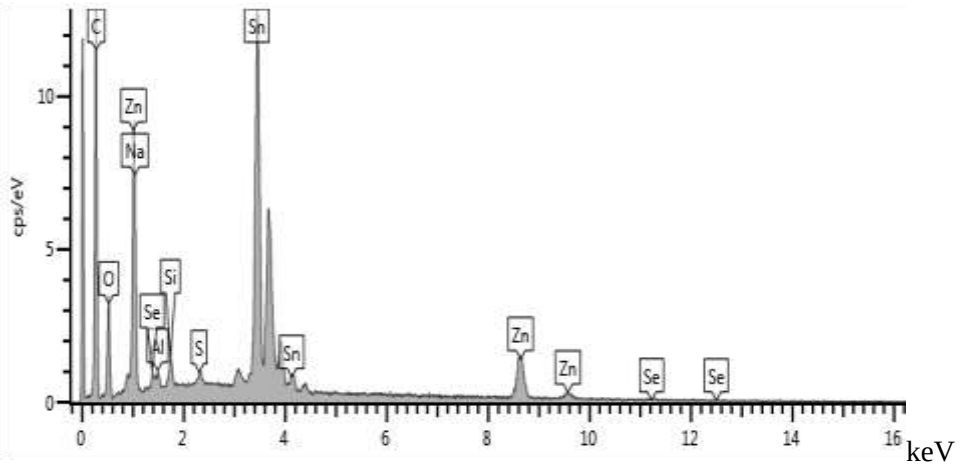

Fig. 5: Energy Dispersive X-ray Spectra (EDX) of Tin doped Zinc Selenide (SnZnSe) Thin Films deposited at pH of 11

3.2: Electrical Analysis of Tin doped Zinc Selenide (SnZnSe) Thin Films deposited at different pH

The electrical properties of Tin doped Zinc Selenide (SnZnSe) thin filmsfrom table 3, shown that the material deposited with pH of 8, 9, 10 and 11 reveal increase in thickness from 209.23 – 224.33nm with increases in the resistivity of the deposited material from $4.0741x10^{3}$–$6.5511x10^{3}$(Ω/cm) which result to the decrease in the conductivity of the deposited material from $2.4545x10^{11}$ – $1.5264x10^{11}$. The high resistivity value makes this thin film useful in solar cells application to improve the conversion efficiency as this could reduce the inevitable defects in solar cells fabrication during the actual production. As a result, SnZnSe thin films resistivity is quit suitable for a buffer layer in Solar cell, PV panel and photovoltaic devices [30-31].

Table 3: Electrical Properties of Tin doped Zinc Selenide (SnZnSe) Thin Films deposited at different pH

Samples	**Thickness, t (nm)**	**Resistivity, $\rho(\Omega m)^{-1}$**	**Conductivity, $\sigma(\Omega m/cm)^{-1}$**	
PT1 (8)	209.23	$4.0741x10^{3}$	$2.4545x10^{11}$	
PT2 (9)	217.21	$5.4981x10^{3}$	$1.8188x10^{11}$	
PT3 (10)	219.26	$6.2121x10^{3}$	$1.6097x10^{11}$	
PT4 (11)	224.33	$6.5511x10^{3}$	$1.5264x10^{11}$	

3.3: The Optical Analysis of Tin doped Zinc Selenide (SnZnSe) Thin Films deposited at different pH

The optical absorbance of SnZnSe thin films shown in fig. 6 spectral shown that the films grown under the same parametric conditions at room temperature and at varying deposition pH of 8, 9, 10 and 11 for sample PT1, PT2, PT3 and PT4 reveal that as the wavelength of the incident radiation of the material increase the absorbance of the material decreases. It was observe that as pH of the grown material increase from 8 - 11 the absorbance of the materials increases which reveals the highest absorbance of SnZnSe cells grown will be a good material that will absorb energy from the sun (Imran*et al.,* 2018, Rajesh *et al.,* 2017, Agawanea *et al.,* 2013, Ikhioya and Ekpunobi., 2015 and 2014, Senthilkumar, *et al.,* 2012, Mehta *et al.,* 2009) and there is no reported research findings on SnZnSe before this report. SnZnSe cells can also serve as a photovoltaic device and others application in the electronic industry. It was also notices that SnZnSe can be use in mass production of solar cells for the fabrication of lasting solar panel for alternative energy supply.

The optical transmittance of SnZnSe thin films is shown in fig. 6 spectral shown that the films grown under the same parametric conditions at room temperature and at varying deposition pH of 8, 9, 10 and 11 for sample PT1, PT2, PT3 and PT4 reveal that as the wavelength of the incident radiation of the material increase the transmittance of the material increases. It was observe that as pH of the grown material increase from 8 - 11 the transmittance of the materials decreases and all the grown material transmit above 55% of transmittance which reveals the highest transmittance of SnZnSe cells grown will be a good material that will absorb energy from the sun (Imran*et al.,* 2018, Rajesh *et al.,* 2017, Agawanea *et al.,* 2013, Ikhioya and Ekpunobi., 2015 and 2014, Senthilkumar, *et al.,* 2012, Mehta *et al.,* 2009) and there is no reported research findings on SnZnSe before this report. SnZnSe cells can also serve as a photovoltaic device and others application in the electronic industry. It was also notices that SnZnSe can be use in mass production of solar cells for the fabrication of lasting solar panel for alternative energy supply.

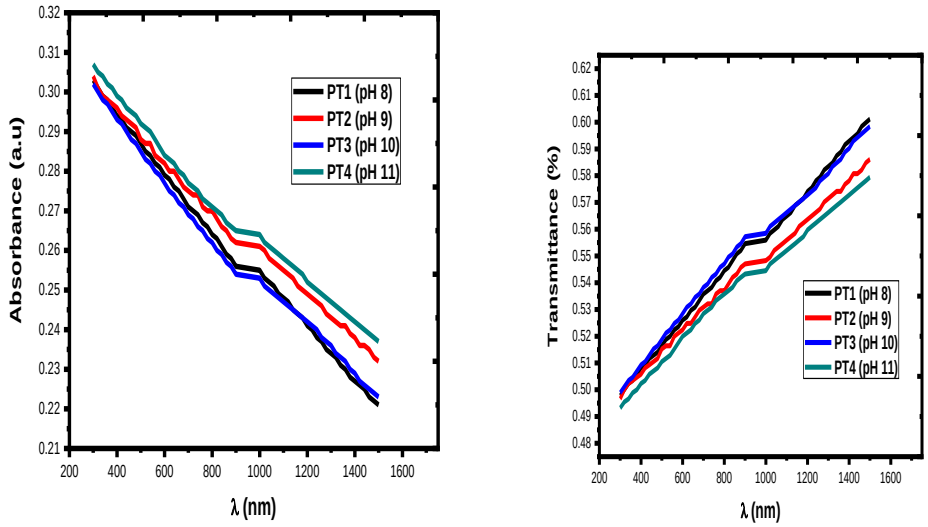

Fig. 6: Plot of absorbance and transmittance versus wavelength

The optical reflectance of SnZnSe thin films is shown in fig. 7 spectral shown that the films grown under the same parametric conditions at room temperature and at varying deposition pH of 8, 9, 10 and 11 for sample PT1, PT2, PT3 and PT4 reveal that as the wavelength of the incident radiation of the material increase the reflectance of the material decreases. It was observe that as pH of the grown material increase from 8 - 11 the reflectance of the materials increases which reveals the highest reflectance of SnZnSe cells grown will be a good material that will absorb energy from the sun (Imran*et al.*, 2018, Rajesh *et al.*, 2017, Agawanea *et al.*, 2013, Ikhioya and Ekpunobi., 2015 and 2014, Senthilkumar, *et al.*, 2012, Mehta *et al.*, 2009) and there is no reported research findings on SnZnSe before this report. SnZnSe cells can also serve as a photovoltaic device and others application in the electronic industry. It was also notices that SnZnSe can be use in mass production of solar cells for the fabrication of lasting solar panel for alternative energy supply.

The refractive index of SnZnSe thin films is shown in fig.7 spectral shown that the films grown under the same parametric conditions at room temperature and at varying deposition pH of 8, 9, 10 and 11 for sample PT1, PT2, PT3 and PT4 reveal that as the photon energy of the material increase the refractive index increases. It was observe that as pH of the grown material increase from 8 - 11 the refractive index of the materials increases which reveals the highest refractive index of SnZnSe cells grown will be a good material that will absorb energy from the sun (Imran*et al.*, 2018, Rajesh *et al.*, 2017, Agawanea *et al.*, 2013, Ikhioya and Ekpunobi., 2015 and 2014, Senthilkumar, *et al.*, 2012, Mehta *et al.*, 2009) and there is no reported research findings on SnZnSe before this report. SnZnSe cells can also serve as a photovoltaic device and others application in the electronic industry. It was also notices that SnZnSe can be use in mass production of solar cells for the fabrication of lasting solar panel for alternative energy supply.

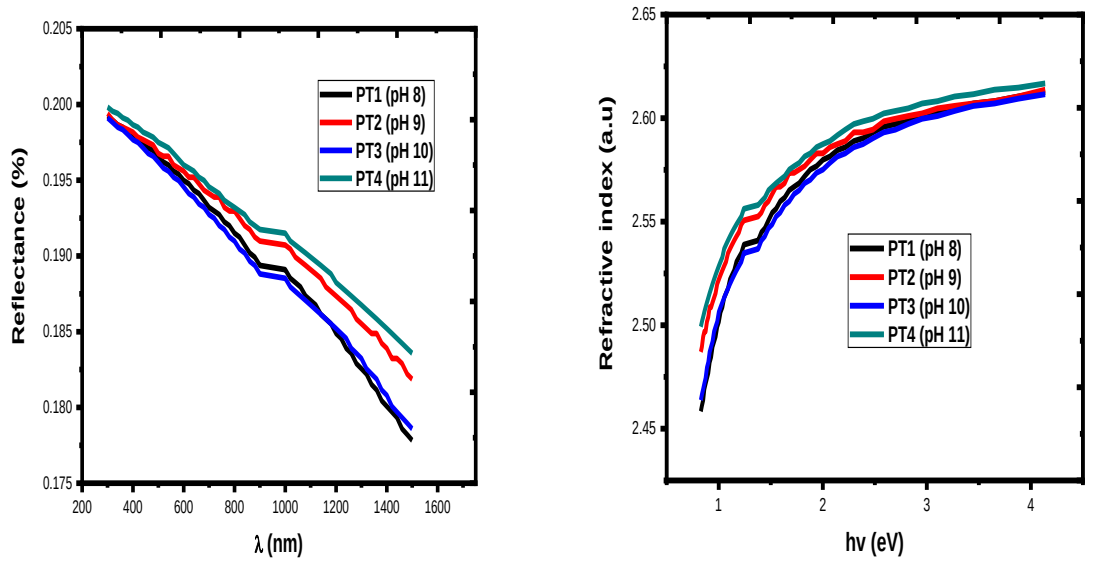

Fig. 7: Plot of reflectance and refractive index versus wavelength and Photon Energy

The optical band gap energy of SnZnSe thin films is shown in fig.8 spectral shown that the films grown under the same parametric conditions at room temperature and at varying deposition pH of 8, 9, 10 and 11 for sample PT1, PT2, PT3 and PT4 reveal the band gap energy for the grown SnZnSe thin films which was determine by extrapolating the straight part of the graph of absorption coefficient square against the photon energy to the photon energy axis. From the plot band gap energy is 2.0-2.3eV (Imran*et al.*, 2018, Rajesh *et al.*, 2017, Agawanea *et al.*, 2013, Ikhioya and Ekpunobi., 2015 and 2014, Senthilkumar, *et al.*, 2012, Mehta *et al.*, 2009) and there is no reported research findings on SnZnSe before this report.

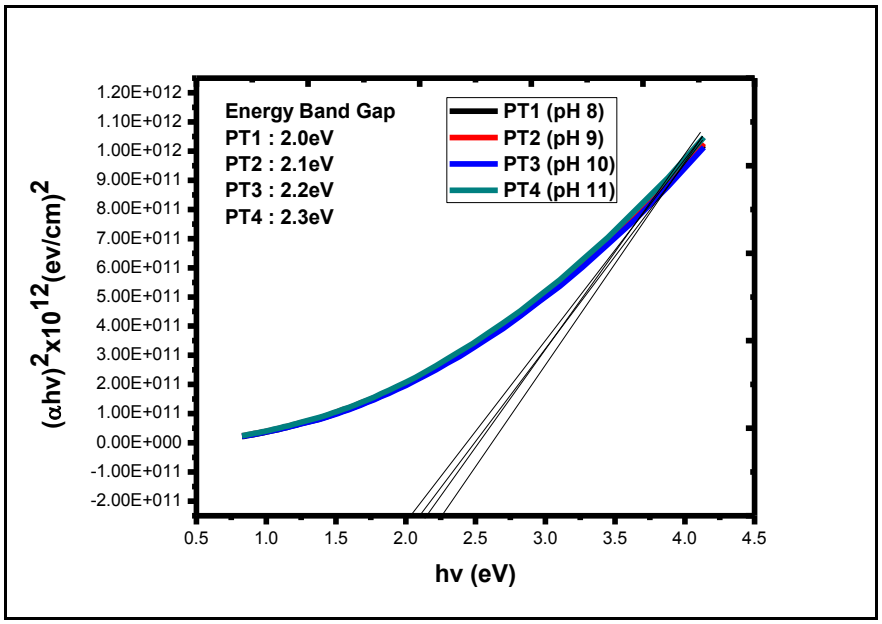

Fig. 8: Plot of absorption coefficient square versus photon energy

The extinction coefficient and optical conductivity of SnZnSe thin films is shown in fig. 9 spectral shown that the films grown under the same parametric conditions at room temperature and at varying deposition pH of 8, 9, 10 and 11 for sample PT1, PT2, PT3 and PT4 reveal that as the photon energy of the material increase the extinction coefficient and optical conductivity increases. It was observe that as pH of the

grown material increase from 8 - 11 the extinction coefficient and optical conductivity of the materials increases which reveals the highest extinction coefficient and optical conductivity of SnZnSe cells grown will be a good material that will absorb energy from the sun (Imran*et al.,* 2018, Rajesh *et al.,* 2017, Agawanea *et al.,* 2013, Ikhioya and Ekpunobi., 2015 and 2014, Senthilkumar, *et al.,* 2012, Mehta *et al.,* 2009) and there is no reported research findings on SnZnSe before this report. SnZnSe cells can also serve as a photovoltaic device and others application in the electronic industry. It was also notices that SnZnSe can be use in mass production of solar cells for the fabrication of lasting solar panel for alternative energy supply.

The real and imaginary dielectric constant of SnZnSe thin films is shown in fig. 10 spectral shown that the films grown under the same parametric conditions at room temperature and at varying deposition pH of 8, 9, 10 and 11 for sample PT1, PT2, PT3 and PT4 reveal that as the photon energy of the material increase the real and imaginary dielectric constant increases. It was observe that as pH of the grown material increase from 8 - 11 the extinction coefficient and optical conductivity of the materials increases which reveals the highest real and imaginary dielectric constant of SnZnSe cells grown will be a good material that will absorb energy from the sun (Imran*et al.,* 2018, Rajesh *et al.,* 2017, Agawanea *et al.,* 2013, Ikhioya and Ekpunobi., 2015 and 2014, Senthilkumar, *et al.,* 2012, Mehta *et al.,* 2009) and there is no reported research findings on SnZnSe before this report. SnZnSe cells can also serve as a photovoltaic device and others application in the electronic industry. It was also notices that SnZnSe can be use in mass production of solar cells for the fabrication of lasting solar panel for alternative energy supply.

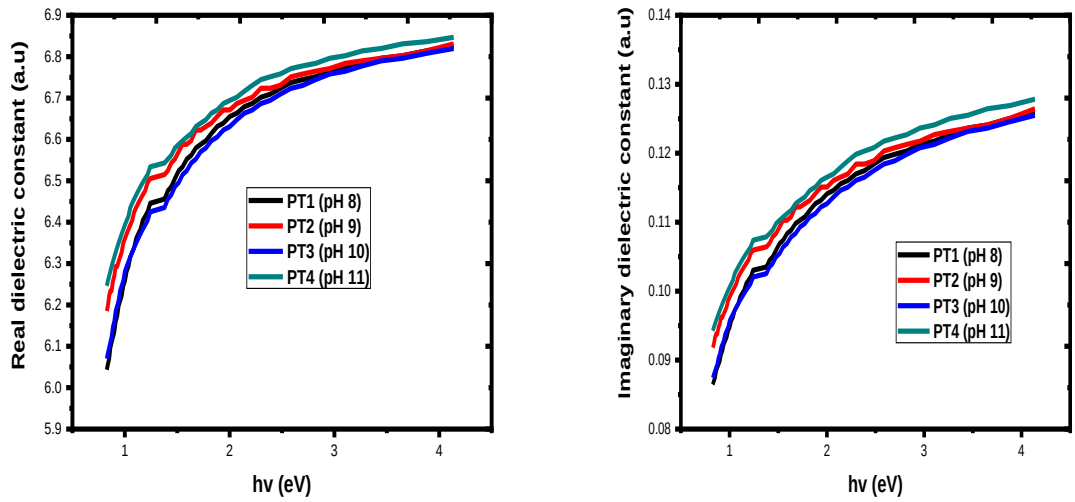

Fig. 10: Plot of real and imaginary dielectric constant versus photon energy

Electrochemical deposition technique have been successfully used to grown thin films of SnZnSe.The XRD of the films deposited on FTO substrates at different pH shows the reflection peaks at (220), (221), (300), (310), (311), (222) and (320) with the lattice constant of a = 7.189Å. SEM shows random distribution of tiny nano-grains on the substrate, the nano-grains were observed to agglomerate due to the presence of large free energy characteristic of small particles. The optical band gap of the deposited material increases from 2.0 eV – 2.3 eV as the dopant concentration increases. The optical absorbance of SnZnSe thin films grown under the same parametric conditions at room temperature and at varying deposition pH of 8, 9, 10 and 11 for sample PT1, PT2, PT3 and PT4 reveal that as the wavelength of the incident radiation of the material increase the absorbance of the material decreases. It was observe that as pH of the grown material increase from 8 - 11 the absorbance of the materials increases which reveals the highest absorbance of SnZnSe cells grown will be a good material that will absorb energy from the sun and cells can also serve as a photovoltaic device and others application in the electronic industry. It was also notices that SnZnSe can be use in mass production of solar cells for the fabrication of lasting solar panel for alternative energy supply.

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