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Characterization of Optical Properties, X-Ray Diffraction and Fourier Transform Infrared Spectroscopy of Undoped Zinc Sulphide and Cu, Ni, Co-Doped Zinc Sulphide (ZnS) Thin Film Prepared By Chemical Bath Method.

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Abstract

In this work, a film of ZnS was deposited on a glass substrate using chemical bath technique. The bath was made up of Zinc Chloride, Potassium hydroxide, Ammonium nitrate, and thiourea. Where the Zinc Chloride dissociate to give Zinc ion, Ammonium nitrate serves as the complexing agent. Thiourea dissociates to give Sulfur ion, potassium hydroxide is to control the pH. The effect of variation in volume or capacity. (5mil, 10mil, 15mil) of the doped impurities (Cu, Co, Ni) was studied where copper was obtained from Chalcopyrite and bornite. Cobalt was obtained from cobaltite (metallic ore) and Nickel was obtained from ion-nickel-sulfides such as pentlandite, after their variations were studied, their optical properties were also examined using optical spectrophotometer, their physical properties were also examined using the X-ray diffractometer, and their infrared spectrum of absorption were also examined using the Fourier transform infrared spectroscopy. By using the optical spectrophotometer, we observed that the film has high absorbance (Co(10ml)) ZnS thin film, high transmittance (Co(15ml)) ZnS thin film, and a peak reflectance (Co(5ml)) ZnS thin film which can be used to produce solar panels, glass plate and glass mirror respectively. By using the x-ray diffraction analysis, amorphous type crystalline structures were found in films, and by using the Fourier transform infrared spectroscopy, the films infrared spectrum was known, obtained ZnS films were optically transparent with band gap energies values between 1.0-3.5 ev while the critical parameter of the bath for obtaining high quality films is the pH, with 11.5 as initial value.

***KEYWORDS**: ZnS (Zinc Sulfide), Thin Film, Chemical Bath Deposition, Doping (Cu, Co, Ni), Absorbance, Transmittance, Reflectance, Annealing, X-ray Diffraction, Fourier Transform Infrared Spectroscopy.*

1.0 Introduction

ZnS is a promising and alternative material used for thin film solar cells fabrication. Its band gap energy E_g at room temperature of 3.7 eV means a transparent material with a wide range of wavelength as compared with CdS which absorbs below 515 nm (E_g=2.42 eV). The high efficiency of 18.6% achieved with solar cells of ZnO/ZnS/CIGS (Cu(In, Ga)Se_2 has attracted the scientific attention. Chemical bath deposition (CBD) is an attractive, low cost and simple method to prepare large-area semiconductors films with good quality and controllable thickness.

Several kinds of chemical reagents have been used in the literature to prepare ZnS films by the CBD technique. For example, Liu *et al.*, used $ZnSO_4$, thiourea ($SC(NH_2)_2$) and ammonia as chemical reagents to prepare ZnS films at 80 ^oC; Noikeau *et al*., used, beside before components, an additional reagent (hydrazine) as complex agent to produce the ZnS films at 60 ^oC. Gode *et al.*, used Zn $(CH_3COO)_2$ triethanolamine (TEA), ammonia/ammonium chloride (NH_3/NH_4Cl, pH = 10.55) buffer solution, tri-sodium citrate ($C_6H_5Na_3O_7$), and thiourea, with the chemical mixture heated at 80 ^oC. The chemical reactants for ZnS preparation and reported by Long *et al*., were zinc acetate dihydrate ($Zn(CH_3COO)_2.(H_2O)_2$), thiourea, and complexing agents (tartaric acid and tri-sodium citrate).

In this work, we propose a chemical process to prepare ZnS thin film by the chemical bath technique. The chemical bath is composed by 40ml of 0.02m Zinc chloride ($ZnCl_2$), 40ml of 1.50m ammonium nitrate (NH_4NO_3) (as complexing agent), 40ml of 0.20m thiourea (Cs(NH2)2) and potassium hydroxide (KOH) to control the pH. Deposited films were annealed and studied to observe possible effects on their properties.

ZnS thin film optical properties, crystalline structure and infrared spectrum were reported.

2.0 Literature Review

Thin Film Deposition is the process of creating and depositing thin film coatings onto a substrate material. These coatings can be made of many different materials. From metals to oxides to compounds. Thin film coatings also have many different characteristics which are leveraged to alter or improve some element of the substrate performance. For example, some are transparent; some are very durable and scratch-resistant; and some increase or decrease the conductivity of electricity or transmission of signals.

2.1. Thin Film Deposition

Thin film deposition is an important manufacturing step in the production of many opto-electronic, solid state and medical devices and products, including consumer electronics, semiconductor lasers, fiber lasers, LED displays, optical filters, compound semiconductors, precision optics, microscopy & microanalysis sample slides, and medical implants. There a few different technologies and methods that can be used to apply film coatings, and array of tools and equipment that can be used to streamline or enhance the thin film deposition process, types of Thin Film Deposition are:

2.1.1. Physical Vapor Deposition (PVD)?

Physical vapor deposition (PVD) describes a group of thin film deposition techniques that involve vaporizing a solid material in a vacuum, then depositing that material onto a substrate. Coatings created in this manner are highly durable, and resistant to scratching and corrosion. PVD is useful in the production of devices ranging from solar cells to eyeglasses to semiconductors, Examples of PVD

1. Evaporation
2. Magnetron sputtering
3. Ion beam sputtering
4. Pulsed laser deposition

2.1.2. Chemical Vapor Deposition (CVD)?

Chemical vapor deposition (CVD) describes a group of thin film deposition techniques in which a substrate is placed into a vacuum chamber, two chemical precursors are heated, which causes them to vaporize. When they meet on the substrate surface, a chemical reaction occurs to form a high-performance thin film coating. CVD is useful in creating coatings for a wide variety of applications including medical devices, automotive components and silicon wafers, Examples of CVD

I. Atmospheric Pressure CVD
II. Low Pressure CVD
III. Ultrahigh Vacuum CVD
IV. Atomic Layer Deposition
V. Plasma-Enhanced CVD

2.1.3 Chemical bath deposition technique

Chemical Bath Deposition, also called Chemical Solution Deposition and CBD, is a method of thin-film deposition (solids forming from a solution or gas), using an aqueous precursor solution. Chemical Bath Deposition typically forms film using heterogeneous nucleation (deposition or adsorption of aqueous ions onto a solid substrate), to form homogenous thin films of metal chalcogenides (mostly oxides, sulfides, and selenides) and many less common ionic compounds. Chemical Bath Deposition produces films reliably, using a simple process with little infrastructure, at low temperature (<100 degree centigrade), and at low cost. Furthermore, chemical bath deposition can be employed for large-area batch processing or continuous deposition. Films produced by CBD are often used in semiconductors, photovoltaic cells, and super capacitors, and there is increasing interest in using chemical bath deposition to create nanomaterials.

3.0 Methodology

(A) *Chemical Reaction:* The used chemical process to prepare ZnS films is similar than the process used for Cds film (calcium sulfide). The growth of ZnS by chemical bath deposition is given by the decomposition of the thiourea ($CS(NH_2)_2$) in presence of a zinc salt in a basic solution bath improves the chemical reaction and accelerates the rate deposition. The importance of the formation of the tetra-amino-zine complex ions $[Z_n\ (NH_3)_4]^{2+}$ is determined for the liberation of the Z_n^{2+} and a further recombination

with S_2- ions to produce ZnS. The chemical process can be described through the following chemical reactions.

1. $Z_n^{2+} + 20H \longrightarrow Zn\ (0H)_2$

2. $NH_4^+ + OH \longrightarrow NH_3 + H_2O$

3. $Zn\ (0H)_2 + 4NH_3 \longrightarrow [Zn(NH3)4]^2 + 20H$

4. $CS\ (NH_2)_2 + 20H \longrightarrow S_2 + CN_2H_2 + 2H_20$

5. $[Zn\ (NH_3)_4]^{2+} + S^{2-} \longrightarrow ZnS\ (s) + 4NH_3$

(B) *Film Preparation:* Z.S films were deposited on commercial glass substrates before deposition. Substrates were cleaned following the next steps: soap thrichloroethene, acetone and isopropylic alcohol.

Substrates were rinsed with distilled water in between each step. The chemical bath was composed of 100ml of 0.1m potassium hydroxide (KOH), 40ml of 1 50m ammonium nitrate (NH_4NO_3), 40ml of 0.20m thiourea ($CS\ (NH_2)_2$) and 40ml of 0.02m Zinc Chloride ($ZnCl_2$). The total volume of the chemical bath was 220ml., the chemical solutions were prepared with distilled water without further purification and fresh solutions were used for every experiment. During deposition, the chemical bath was agitated with a magnetic stirrer covered with Teflon, meanwhile, the substrates remain vertically positioned with a Teflon-holder to avoid contamination. The bath temperature was maintained at 90±1°C and the deposition time varied from 60 to 120mins. At the beginning of the CBD process, the pH of the chemical bath was 11.5 bath after 2h of deposition its value diminished to 11.0.

(C) *Impurities (Cu, Co, Ni) were deposited into the undoped ZnS thin film at (5mil, 10mil, 15mil):*

i. For 5mil, after the deposition of the undoped ZnS on the (250ml) beaker containing KOH (100ml), NH_4NO_3 (40ml), Thiourea (40ml) and ZncI (40ml), cobalt (5mil) impurity was added and the temperature maintained at 90 ± 1°C and the deposition time varied for about 2hrs. Another measurement of KOH (100ml), NH_4NO_3 (40ml), thiourea (40ml) and ZncI (40ml) was added into the 250ml beaker and then the second impurity Cupper (5mil) was also added on top of the magnetic hot plate with the magnetic stirrer. Inside, we have the thermometer and substrates vertically positioned with a Teflon-holder to avoid contamination, maintained the temperature at 90 +1°C the time (deposition) was also varied for about 2hrs.

The same procedure was also followed for the third impurity. Nickel (5mil) was also added after a measurement of 100ml of KOH, 40ml of NH_4NO_3, 40ml of thiourea and 40ml of ZnC1 was added into a 250ml beaker with the substrates and thermometer. Vertically, positioned within Teflon-holder to avoid contamination also maintained the temperature at 90 1°C and the time (deposition time) range varied for about 2hrs.

ii. For 10mil, after the deposition of the undoped Z.S on the (250ml) beaker containing KOH (100ml), NHN0; (40ml), thiourea (40ml) and ZCI (40ml), cobalt (10mil) impurity was added and the temperature maintained at 90±1°C and the deposition time varied for about 2hrs. Another measurement of KOH (100ml), NH_4NO_3, (40ml), thiourea (40ml) and 2.CI (40ml) was added into the 250ml beaker and then the

second impurity Cupper (10mil) was also added on top of the magnetic hot plate with the magnetic stirrer Inside, the thermometer and substrates vertically positioned with a Teflon- holder to avoid contamination, maintained the temperature at 90±1°C the time (deposition) was also varied for about 2hrs. The same procedure was also followed for the third impurity. Nickel (10mil) was also added after a measurement of 100ml of KOH, 40ml of $NHN0_3$, 40ml of thiourea and 40ml of ZCI was added into a 250ml beaker with the substrates and thermometer. Vertically, positioned within Teflon-holder to avoid contamination also maintained the temperature at 90 1°C and the time (deposition time) range varied for about 2hrs.

iii. For 15mil, after the deposition of the undoped ZnS on the (250ml) beaker containing KOH (100ml), NH_4NO_3 (40ml), thiourea (40ml) and Zncl (40ml), cobalt (15mil) impurity was added and the temperature maintained at 90 ± 1°C and the deposition time varied for about 2hrs. Another measurement of KOH (100ml), NH_4NO_3 (40ml), thiourea (40ml) and Zncl (40ml) was added into the 250ml beaker and then the second impurity Cupper (15mil) was also added on top the magnetic hot plate with the magnetic stirrer Inside the thermometer and substrates vertically positioned with a Teflon- holder to avoid contamination, maintained the temperature at 90 ± 1°C the time (deposition) was also varied for about 2hrs.

The same procedure was also followed for the third impurity Nickel (15mil) was also added after a measurement of 100ml of KOH, 40ml of NHN03, 40ml of thiourea and 40ml of Zncl was added into a 250ml beaker with the substrates and thermometer. Vertically, positioned within Teflon-holder to avoid contamination also maintained the temperature at 90 ± 1°C and the time (deposition time) range varied for about 2hrs.

The chemical reactions will then be:

1. $[Zn(NH_3)_4]^{2+} + S^{2-} \longrightarrow ZnS\ (s) + 4NH_3 + CO$

2. $[Zn(NH_3)_4]^{2+} + S^{2-} \longrightarrow ZnS\ (s) + 4NH_3 + Cu$

3. $[Zn(NH_3)_4]^{2+} + S^{2-} \longrightarrow ZnS\ (s) + 4NH_3 + Ni$

Where Co, Cu and Ni are the three impurities deposited at (5, 10 and 15mil).

4.0 Data and Discussions

4.1. Results

(A) Optical Study of Cobalt- Doped Zinc Sulfide Material.

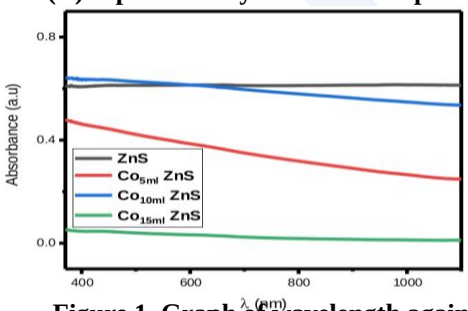


Figure 1. Graph of wavelength against absorbance of ZnS thin film annealed at different mil of cobalt

The spectral absorbance

Fig 1 shows the variations in spectral absorbance of ZnS, thin films deposited at different mil. The spectral absorbance of the undoped ZnS showed absorbance of 0.6, Co (5mil) ZnS showed absorbance value of 0.5 Co (10 mil) ZnS showed peak absorbance value of 0.7, Co (15 mil) ZnS showed peak absorbance value of 0.1, all corresponding to the wavelength range of 400 to 1000 nm of VIS IF region of Electromagnetic Spectrum.

The result shows that Co (10mil) which has the peak absorbance value, can be used to produce materials with a high absorbance of optics like solar panels, coal, black paint, carbon nanotubes, arrays, etc.

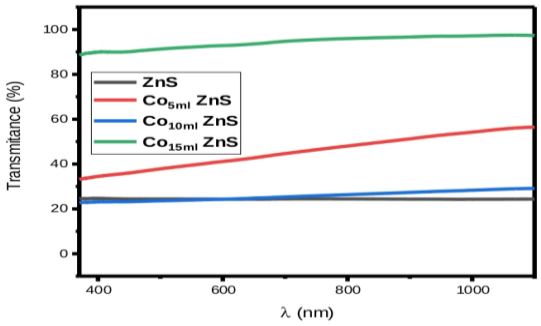


Figure 2. Graph of wavelength against percentage transmittance of ZnS thin film annealed at different mil of cobalt

The percentage transmittance

Figure 2. shows the percentage transmittance value of chemical both deposited ZnS thin film measured in the wavelength range of 400 to 1000 nm of visible infrared region of the electromagnetic spectrum. The undoped ZnS thin film showed transmittance value of 25%, Co (5 mil) ZnS thin film showed transmittance value of 35%, Co (10 mil) ZnS thin film showed transmittance value of 24% and Co (15 mil) ZnS thin film showed peak transmittance value of 89% all in the range of 400nm to 1000nm of this VIS IF region This result showed that Co (15 mil) ZnS thin film, which has the peak transmittance value, can be used to produce high transmittance materials like UV Quartz, glass plates, Borosli-cut glass, etc.

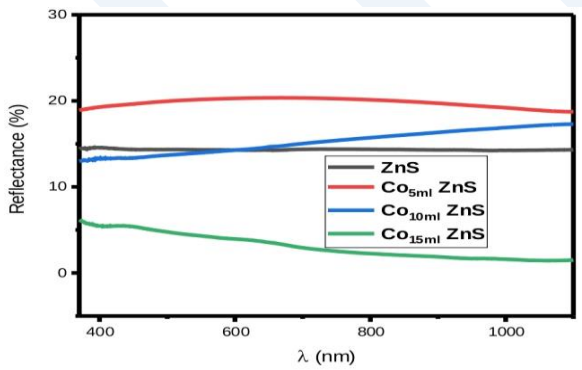


Figure 3. Graph of wavelength against percentage reflectance of ZnS thin film annealed at different mil of cobalt.

The percentage reflectance.

Figure 3., shows the spectral reflectance value of ZnS doped at different mil, all corresponding to the wavelength of 400 nm to 1000 nm between visible infrared region of the electromagnetic spectrum. The

undoped ZnS thin film showed reflectance value of 15%, Co (5 mil) ZnS thin film showed peak reflectance value of 19%, Co (10 mil) ZnS thin film showed reflectance value of 14% and Co (15 mil) ZnS thin film showed reflectance value of 6% and all corresponding to the wavelength of 1000 nm of the electromagnetic spectrum. The result showed that Co (5 mil ZnS) thin film which has the peak reflectance can be used to produce high reflectance materials like Astros foil, Glass mirror, Acrylic mirror, Can lids, Mylar, and Aluminum foil.

(B) XRD Results

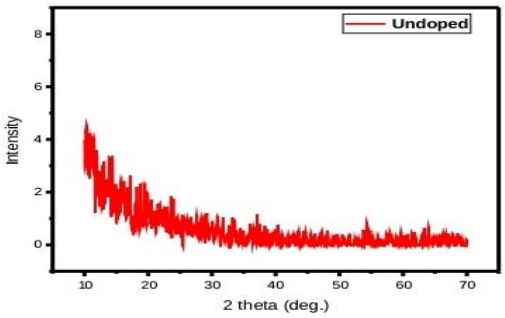


Fig 4: Graph showing undoped ZnS thin film

The undoped ZnS thin film which composed of the Potassium hydroxide (KOH (100ml)) Ammonium nitrate (NH_4NO_3 (40ml)) Thiourea ($Cs(NH_2)_2$ (40ml)) and Zinc chloride ($ZnCl_2$ (40ml)) in the absence of the impurities (Co Cu Ni) after film preparation on a glass substrate the film was finally analyzed using the x-ray diffractometer which gave us amorphous solid matter.

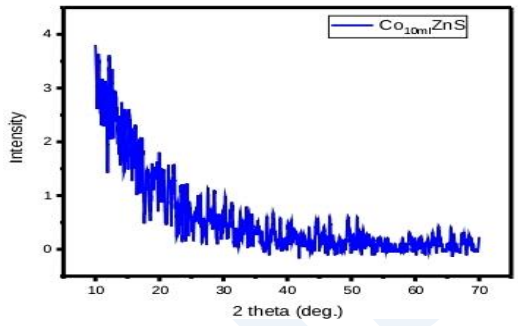


Figure 5. Graph showing cobalt doped ZnS thin film at 10ml (Co)

The cobalt doped ZnS thin film which composed of Potassium hydroxide (KOH) Ammonium nitrate (NH_4NO_3) Thiourea ($Cs(NH_2)_2$) and Zinc chloride ($ZnCl_2$) with added impurity of cobalt (Co) at 10ml after film preparation on a glass substrate was finally analyzed using the x-ray diffractometer which gave us amorphous solid matter.

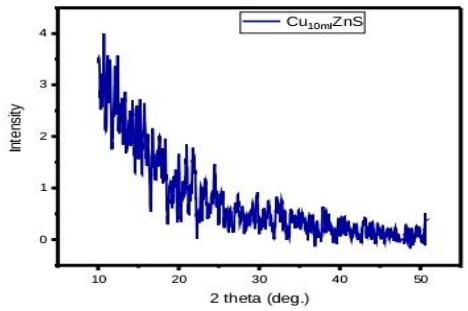


Figure 6. Graph showing Copper doped ZnS thin film at 10ml (Cu)

The Copper doped ZnS thin film which composed of Potassium hydroxide (KOH) Ammonium nitrate (NH_4NO_3) Thiourea ($Cs(NH_2)_2$) and Zinc chloride ($ZnCl_2$) with added impurity of Copper (Cu) at 10ml after film preparation on a glass substrate, the film was finally analyzed using the x-ray diffractometer which gave us amorphous solid matter.

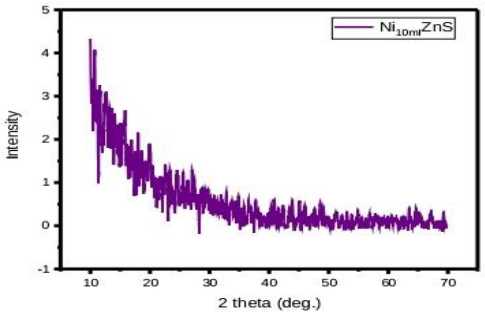


Figure 7. Graph showing Nickel doped ZnS thin film at 10ml (Ni)

The Nickel doped ZnS thin film which composed of Potassium hydroxide (KOH) Ammonium nitrate (NH_4NO_3) Thiourea ($Cs(NH_2)_2$) and Zinc chloride ($ZnCl_2$) with added impurity of Nickel (Ni) at 10ml after film preparation on a glass substrate, the film was finally analyzed using the x-ray diffractometer which gave us amorphous solid matter.

(C) FTIR Results

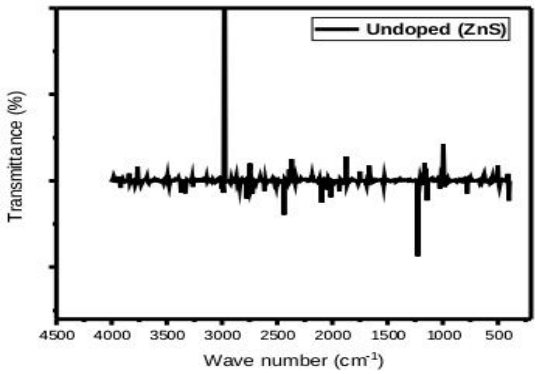


Figure 8. Graph showing the Undoped ZnS thin film

The undoped ZnS thin film which composed of the Potassium hydroxide (KOH) Ammonium nitrate (NH_4NO_3) Thiourea ($Cs(NH_2)_2$) and Zinc chloride ($ZnCl_2$) in the absence of the impurities (Co Cu Ni) after film preparation on a glass substrate the film was finally analyzed using the Fourier transform

infrared spectroscopy to obtain the infrared spectrum of the film which can be used in different cases like (i) To produce night vision devices. (ii) To produce telescopes. (iii) Infrared thermal imaging cameras, etc.

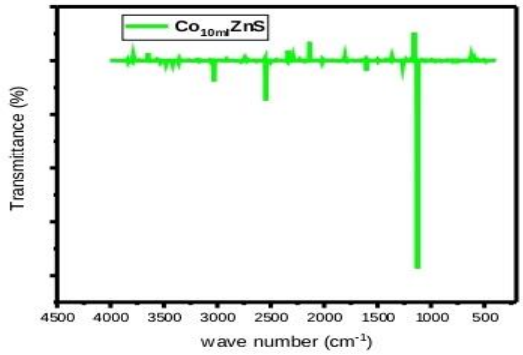


Figure 9. Graph showing Cobalt doped ZnS thin film at 10ml (Co)

The undoped ZnS thin film which composed of the Potassium hydroxide (KOH) Ammonium nitrate (NH_4NO_3) Thiourea ($Cs(NH_2)_2$) and Zinc chloride ($ZnCl_2$) with added impurity of Cobalt (Co) at 10ml after film preparation on a glass substrate the film was finally analyzed using the Fourier transform infrared spectroscopy to obtain the infrared spectrum of the film which can be used in different cases like (i) To produce night vision devices. (ii) To produce telescopes. (iii) Infrared thermal imaging cameras, etc.

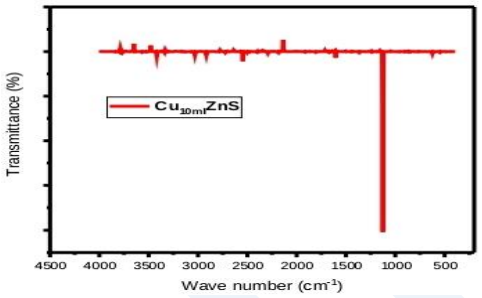


Figure 10. Graph showing Copper doped ZnS thin film at 10ml (Cu)

The undoped ZnS thin film which composed of the Potassium hydroxide (KOH) Ammonium nitrate (NH_4NO_3) Thiourea ($Cs(NH_2)_2$) and Zinc chloride ($ZnCl_2$) with added impurity of Copper (Cu) at 10ml after film preparation on a glass substrate the film was finally analyzed using the Fourier transform infrared spectroscopy to obtain the infrared spectrum of the film which can be used in different cases like (i) To produce night vision devices. (ii) To produce telescopes. (iii) Infrared thermal imaging cameras, etc.

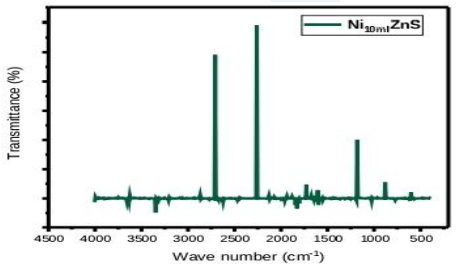


Figure 11. Graph showing Nickel doped ZnS thin film at 10ml (Ni)

The undoped ZnS thin film which composed of the Potassium hydroxide (KOH) Ammonium nitrate (NH_4NO_3) Thiourea ($Cs(NH_2)_2$) and Zinc chloride ($ZnCl_2$) with added impurity of Nickel (Ni) at 10ml

after film preparation on a glass substrate the film was finally analyzed using the Fourier transform infrared spectroscopy to obtain the infrared spectrum of the film which can be used in different cases like (i) To produce night vision devices. (ii) To produce telescopes. (iii) Infrared thermal imaging cameras, etc.

4.2. Discussion

From the work done, ZnS films produced by CBD technique are rarely prepared in a single phase. For instance, it is reported that the ZnO and Zn(OH)2 phases are usually mixed with the ZnS phase. Thus, some properties of the deposited CBD-ZnS films are reported showing dependence with the formation of the other phases into the films. For this reason, different authors report variations of the optical properties, crystalline structure, and infrared spectrum given the different chemical reagents and conditions used during the CBD process. These mixed-phases can produce different effects on films, which sometimes, are not easy to identify and to control. Thus, the repeatability of the reported method is another challenge to obtain ZnS films with high quality for CBD technique.

By the other hand, the role of the pH value in the chemical bath, adjusted with KOH in this work, resulted a key factor, because concentrations below 1.0 M do not produce conditions for ZnS films deposition. This effect was also reported by Antony *et al*., for a chemical bath with Zn(NO3)2 and NH4OH as the salt reagent and the source of OH radicals, respectively. However, by controlling the chemical bath conditions, CBD can be a simple and low-cost method to produce buffer layers of ZnS which can increase the efficiency of the CIGS solar cells.

5.0. Conclusion

In this work, a methodology to prepare ZnS thin films on glass substrates by chemical bath deposition for solar cells applications was proposed. An optimal pH=11.5 value of the chemical bath was established with the KOH reagent. High quality ZnS films were obtained at this condition. The pH value plays an important role for the films formation.

Reproductivity of the films preparation was possible with this method, given the similar properties obtained on the films deposited under similar conditions. Annealing treatments were applied on ZnS films at 200 °C and 400 °C in order to study their effects. Small changes on the optical properties of ZnS films with the annealing treatment were obtained, as demonstrated by the reduction of the band gap energy value for higher annealing temperatures.

However, we found that the annealing process did not produce important changes on the crystalline structure of the films. The optical transmittance of the ZnS was measured 25-89% in the visible light spectrum range. The band gap energy values of the deposited films changes from 3.1 ev to 200 ev for as-grown and for annealed films.

The chemical bath deposition method employed for the growth of zinc sulfide (ZnS) thin film doped with cobalt, copper and nickel was successful. The band gap of the film suggested that the material (film) is a semi conducting thin film.

A. The optical and solid-state properties such as reflectance (R), absorbance (A), transmittance (T), refractive index (n), Extinction coefficient (K), dielectric constant (E), and optical conductivity (Õ) which were determined using the spectrophotometer showed that the material (film) could be used to;

1. Produce Astor foil, glass mirror, Aluminum foil, Mylar, all which have high Reflectance.
2. Produce curtains, wool, all which has high absorption coefficient.
3. Produce solar panels.
4. Produce automobile materials, example steel, plastic, Aluminum, Rubber etc.
5. Produce Pvc, distil water, Glass, all which has high dielectric constant.
6. Produce wool fiber, glass fiber, all which has high Absorbance.

B. The material (film) is an Amorphous solid matter, which was determined while using the x-ray diffractometer which then implies that the film has no sharp boiling point, the film can be used to produce tubes, tires, shoe soles, etc.

C. The materials (film) Infrared spectrum was also determined using the Fourier transform infrared spectroscopy showing that the film can be used in optical construction companies to produce night vision devices, lens, projectors, etc.

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