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# STRUCTURAL AND OPTICAL PROPERTEIS OF ZnS, PbS, $Zn_xPb_{1-x}S$ AND $PbZn_xS_{1-x}$ THIN FILMS

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## Abstract

In this work we used the chemical spray pyrolysis technique to prepare ZnS, PbS, $PbZn_{1-x}S_x$, and $Pb_xZn_{1-x}S$, thin films with (x = 0.01, 0.02) at glass substrate at 280˚C, 230˚C, 270˚C and 270˚C respectively. And also study the optical and structural properties.

The result of X-ray diffraction obtained that the films are polycrystalline and have cubic with hkl (111) and wurtize with hkl (100) phases for ZnS films, the phase for PbS films is cubic with hkl (200) and the phases for $Pb_xZn_{1-x}S$ and $PbZn_{1-x}S_x$. are cubic with hkl (200)

The lattice constants (a) which were calculated 5.939A˚, for PbS films, 3.36 A˚ and 5.44 A˚ for wurtize and cubic of ZnS films respectively, 5.92A˚for $Zn_xPb_{1-x}S$ films with x = 0.02, and 5.921A˚for $PbZn_xS_{1-x}$ films with x = 0.01

Also the absorption spectra used in the range (200 nm – 1100 nm) to calculate the optical energy gap which is decrease with increase the film thickness for all the films prepared by this technique.

The values of energy gap were determined between (1.58 – 1.78 eV) for PbS films, (2.9 – 3.1eV) for ZnS films, The energy gap of $Zn_xPb_{1-x}S$ films in the range (2.4 – 2.9) eV and (2.38– 2.75 eV for x = 0.02 and x = 0.01 respectively and The energy gap of $PbZn_xS_{1-x}$ (1.57 – 1.8) eV and (1.4 – 1.6) eV for x = 0.02 and x = 0.01 respectively.

The optical constants, such as refractive index (n) increase with increase the film thickness, while the extinction coefficient (k) decrease with increase the thickness for all the films except ZnS films which are inversely. And the dielectric constants ($\varepsilon_r$, $\varepsilon_i$) have the same behavior of refractive index (n) and extinction coefficient (k) respectively.

## Introduction

Semiconductor materials are always the focus in material science due to their outstanding electronic and optical properties and extensively potential application in various devices including light-emitting diodes single electron transistors and field-effect thin-film transistors In principle, the electronic and optical properties of semiconductor materials are tunable by varying their shapes and sizes so it is one of the desired goals in material science to realize precise control of the morphology of semiconductor materials.[1]

By selecting appropriate compound semiconductor materials, it becomes possible to realize various devices which can not be achieved using the main elemental semiconductor material, silicon.

A number of techniques have been examined in the search for more reliable and cheapest method of producing thin films. Physical and chemical depositions are the most common methods for transferring material atom by atom from one or more sources to the

growth surface of a film being deposited onto a substrate. So we have two general methods which are the physical and chemical depositions: [2-5]

There are many techniques that have been used to prepare thin films among the varies thin film deposition techniques chemical spray pyrolysis is one of the principle methods to produce large area and uniform coating [6, 7].

The chemical spray pyrolysis technique (CSPT), one of the major techniques to deposit a wide variety of materials in thin film form. The prime requisite for obtaining good quality thin film is the optimization of preparative conditions viz. substrate temperature, spray rate, concentration of solution etc

The interest to non-vacuum methods for thin films deposition has increased in the last decade. The solution based process has several advantages: simplicity of process, access to a wide range of metal oxide stoichiometries, precise composition control, and applicability to substrate at any size.

The optical properties of a sprayed film depend strongly on the technique of spray. Two of the most important optical properties are the refractive index n and the extinction coefficient k, which are generally called optical constants.

Using the fundamental relations of photon transmittance T and absorbance A,

$$I_t = I_o e^{-\varepsilon t} \quad \ldots\ldots\ldots \quad (1)$$

Where $I_t$ : is the incident photon energy at thickness (t) inside the material
and $I_o$: is incident photon energy at surface of material [8]
and $A = \text{Log } I_o/I_t$ … …(2)
We have [8]
□□□□□□□□□□□□□□□□□□□□ε = 2.303 (A/t) …… (3)
The reflectance (R) has been found by using the relationship

$$R+T+A = 1 \quad \ldots\ldots (4)$$

For normal reflectance, we have [9, 10]

$$R = (n-1)^2 / (n+1)^2 \quad \ldots\ldots (5)$$

By using the above relation, the refractive index can be determined from the relation

$$n = (1+\sqrt{R}) / (1- \sqrt{R}) \quad (6)$$

The extinction coefficient is related to the absorption coefficient ε by the relation [11].

$$k = \square\square / 4\square \quad \ldots (7)$$

Where □ is the incident photon wavelength

The absorption of radiation that leads to electronic transitions between the valence and conduction bands is split into direct and indirect transitions these transitions are described by the equation [51, 56].

$$\alpha(h\upsilon) = A^*(h\upsilon - E_g^{op})^r \quad \ldots\ldots\ldots\ldots \quad (8)$$

Where $$A^* = \frac{e^2(2\frac{m_h^* m_e^*}{m_h^* + m_e^*})^{3/2}}{nch^2 m_e^* h\upsilon} \quad \ldots\ldots\ldots \quad (9)$$

$m_e^*$ : is the effective mass of electron
$m^*_h$ : is the effective mass of hole.
$hv$ : is the incident photon energy, and $E_g^{op}$ : is optical energy gap.
r : is constant which takes the values (1/2, 3/2, 2, 3) depending on the material and the type of the optical transition whether it is direct or indirect,

The real and imaginary parts of dielectric constant ($\varepsilon_r$ and $\varepsilon_i$ respectively) can be calculated as follows [12]

Complex refractive index $N^* = n - ik$ ……. (10)

Complex dielectric constant $\varepsilon^* = \varepsilon_r - i\,\varepsilon_i$ ….. (11)

From the relation $N^* = \sqrt{\in^*}$ , there fore:

$(n - ik)^2 = \varepsilon_r - i\,\varepsilon_i$ (12)

So that $\varepsilon_r = n^2 - k^2$ (13)

$\varepsilon_i = 2nk$ ……..... (14)

**Experimental Part**

Before starting the deposition we must mix the solutions according to the film components, (after found their weight by this equation ($W=M\times M._{wt} \times V/1000$ , where M, is the molarity , $M._{wt}$, is the molecular weight ,V, is the volume ),the solutions can be prepared by solving the salts in the distill water as follows,

*** Thioria solution $CS(NH_2)_2$:** This solution can be prepared with molarities (0.1M), from solving (0.761gm) of thioria in (100ml) of distill water.

***Lead acetate solution $Pb(CH_3COO)_2\ 3H_2O$:** This solution can be prepared with molarities (0.1M), from solving (2.78gm) of lead acetate in (100ml) of distill water

***$ZnSO_4 7H_2O$ solution:**This solution can be prepared with molarities (0.1M), from solving (2.87gm) of $ZnSO_4 7H_2O$ In (100ml) of distill water

After getting the different amount of solutions according to the ratio and volume requirement, we put it on the magnetic stirrer about 15 minutes to be sure from mixing the solutions mixed properly. After cleaning the substrates, we place them on the electrical heater surface and leave them about 15minutes so as to their temperature reaches the electrical heater temperature. The solution must be put in solution container. After that we can start the deposition process,

The films were prepared on clean glass substrates, which were placed on the surface of a substrate heater when sprayed. The substrate heater was an electrically controlled block furnace. After the spray process complete we leave the substrate on the surface of heater about 20 minutes in order to chemical interactions and the crystallization process will be complete.

The UV/VIS/NIR spectroscopy type Shimadzu was used to measure the absorption of prepared films from wave length 200 nm up to 1100 nm.and from know thickness of thin films the energy gap and absorption coefficient can be calculated Where all these measurements under the condition Incidence: Normal, Temperature: Room temperature ,Reference: Uncoated glass slide. The extinction coefficient of prepared thin films was

measured by using the equation (3). Refractive index can be defied as the ratio between the speed of wave in space to its speed in a medium, and the refractive index measured by using the equation (6). The real part and imaginary part of the dielectric constant measured due to the equation (11) and (12) respectively.

## Result and Discussion

### Structural and Optical Properties of ZnS Films:

#### * X-Ray Diffraction Results for ZnS films

It is observed that for ZnS film of thickness (1500) nm was found to exhibit three diffraction peaks associated with (100) reflections for the hexagonal structure and (111) reflection for the cubic structure of which the intensity of the (111) orientation is predominant. And with unknown peaks at ($2\theta = 18.58^{\circ}$). The lattice constant (a) for ZnS thin films was (3.36 A˚) for wurtize and (5.44 A˚) for cubic phase which are agreements with the result obtained by Shabaan for ZnS films by thermal evaporation technique [9]. seen in the table (1)

**Table (1) Comparison of crystallographic data for ZnS thin films with standard data [13].**

| Standard Data | | Observed (d) and (a) value for ZnS films | | |
|---|---|---|---|---|
| d (A˚) | hkl | d (A˚) | hkl | a (A˚) |
| 3.309 | 100 | 3.360 | 100 | 3.360 |
| 3.128 | 111 | 3.201 | 111 | 5.440 |
| | | 4.769 | unkown | |

#### ** Optical Characteristics for ZnS Films:

As listed in Table (2), It is observed that the optical energy gap decreases with the increase of film thickness**.** This may be due to the possibility of structural defects in the films arisen during the time of their preparation, which could give rise to the allowed states near the conduction band in the bandgap. In the case of much thicker films, these allowed states could well merge with the conduction band resulting in the reduction of the energy bandgap [14] . This is similar to the results of many other workers like Nadeem and Ahmed for ZnS thin films which were prepared by Resistive Heating Technique [15]

The Refractive index was determined from the reflectance data, and As listed in Table (2), The increase of the film thickness causes an overall decrease in the refractive index. The decrease is due to the over all decrease in the reflectance with the increase of

film thickness. This behavior is similar to the work of Shahbaan for ZnS thin films by thermal evaporation technique [9].

The variation of extinction coefficient with the photon wavelength decreases as the film thickness increases which are due to the different effective thickness of the thin films [16]

Table (2) gives values of the real and imaginary parts of the dielectric constant at 600 nm wavelength. and have the same behavior of refractive index and extinction coefficient.

**Table (2) Values of Refractive index (n), Extinction coefficient (k), Real ($\varepsilon_r$) and Imaginary ($\varepsilon_i$) Dielectric Constant for ZnS films (at 600 nm wavelength)**

| **Thickness (nm)** | **n** | **K** | **$\varepsilon_r$** | **$\varepsilon_i$** | **Eg (ev)** |
|---|---|---|---|---|---|
| 797 | 1.304 | 0.0019 | 1.708 | 0.004 | 3.1 |
| 103.6 | 1.282 | 0.0012 | 1.64 | 0.003 | 3.0 |
| 130.6 | 1.274 | 0.0009 | 1.62 | 0.002 | 2.9 |

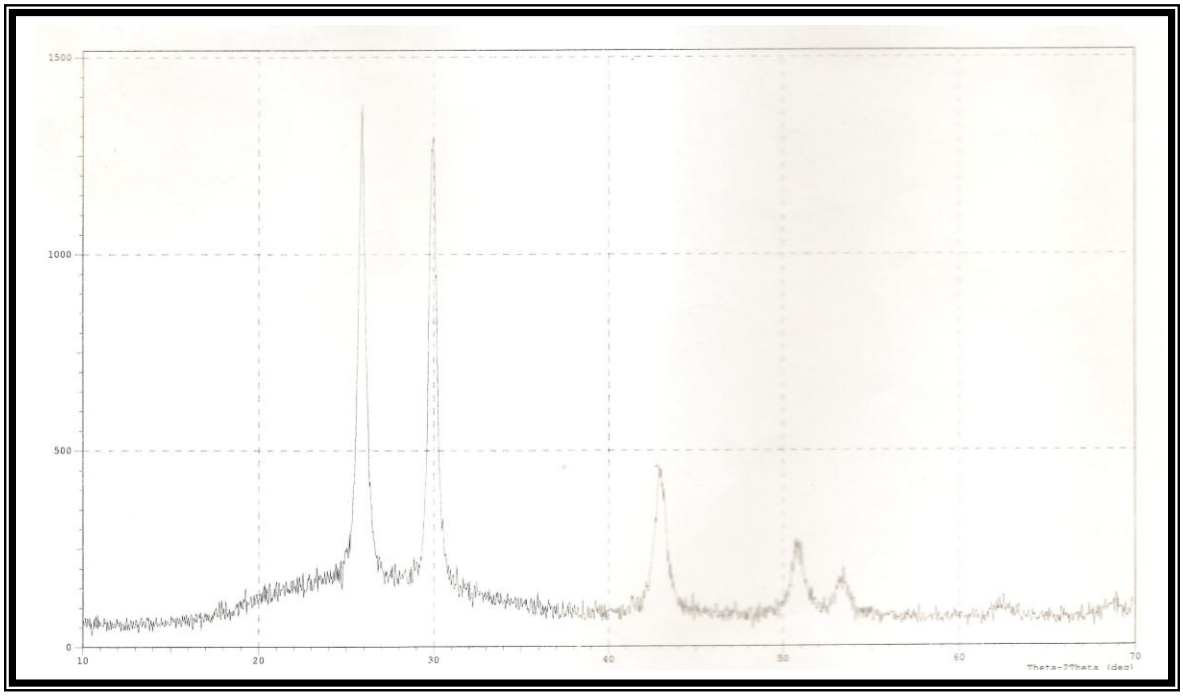
## **Structural** and Optical Properties of PbS Films:

* X-Ray Diffraction Results for PbS films

It is observed from figure (1) that for PbS film of thickness (1038) nm was found to exhibit five diffraction peaks associated with (111), (200), (220), (311), and (222) also there are another two weak peaks associated with (400) and (331). All the diffraction peaks can be indexed to a face-centered-cubic rock-salt-structured PbS. Which are coincides with the work of Ken-Tye Yong whose study PbS Nanowires [17].

The different peaks for PbS film are indexed in Table (3) as well as the corresponding values of the interplanar distance $d_{(hkl)}$ and compared with the standard values of ASTM data.

The lattice constant (a) for PbS thin films, was (5.939 A˚). This value is in a good agreement with the value estimated by Yongong [18], and also with the standard value as shown in the table (3) which is 5.94A˚. No obvious characteristic diffraction peaks from other impurities can be detected. The strong and sharp diffraction peaks suggest that the as-obtained products are well crystalline.




Figuer (1) : X-ray diffraction pattern for PbS thin film

Table (3) Comparison of crystallographic data for PbS thin films with the JCPDS card 5-592 [19].

| Standard data | | Observed d and a value for PbS film | | |
|---|---|---|---|---|
| d (A˚) | hkl | d (A˚) | hkl | a (A˚) |
| 3.429 | 111 | 3.429 | 111 | 5.939 |
| 2.969 | 200 | 2.974 | 200 | 5.948 |
| 2.099 | 220 | 2.100 | 220 | 5.939 |
| 1.790 | 311 | 1.792 | 311 | 5.943 |

**** Optical Characteristics for PbS Films**

It is observed from the table (4) that the optical energy gap decreases with the increase of film thickness this behavior is similar to result of Ubale [19] and his energy gap in the range (1.88-2.28) eV, while ours (1.58- 1.78) eV this difference refers to our thicker films and different technique.

From the table we can observed that the refractive index increase with increase the thickness but not systematically this is due to reflectance.

The extinction coefficient was decreases as the film thickness increases which is due to the different effective thickness of the thin films, but not systematically as given in Table (4). This behavior similar to the work of DEBNATH thin films using Electron Beam Evaporation Technique [20].

The real and imaginary parts of the dielectric constant for PbS thin films shows that $\varepsilon_r$ increase with increase thickness as given in table (4).this is due to the extinction coefficient .

**Table (4) Values of Refractive index (n), Extinction coefficient (k), Real ($\varepsilon_r$) and Imaginary ($\varepsilon_i$) Dielectric Constant for PbS films (at 540 nm wavelength)**

| Thickness (nm) | n | K | $\varepsilon_r$ | $\varepsilon_i$ | Eg (ev) |
|---|---|---|---|---|---|
| 550 | 1.424229 | 0.004478 | 2.028409 | 0.012756 | 1.78 |
| 710 | 1.424241 | 0.003445 | 2.02845 | 0.009812 | 1.68 |
| 880 | 1.484373 | 0.00352 | 2.203352 | 0.010451 | 1.58 |

## Structural and Optical Properties of $Zn_xPb_{1-x}S$ Films:

### * X-Ray Diffraction Results for $Zn_xPb_{1-x}S$ films

X-ray diffraction pattern for $Zn_xPb_{1-x}S$ film (x = 0.02) was carried out with X-ray diffraction in the range 2θ between 20˚ to 50˚ shown in Figure (2). It is observed from this figure that for $Zn_xPb_{1-x}S$ film of thickness (1500) nm was found to exhibit three diffraction peaks associated with (111), (200), (220), with d value 3.418A˚, 2.965 A˚ and 2.095 A˚ respectively and with obvious characteristic diffraction peak from impurities can be detected another unknown peak with its (2θ = 33.278) d value is 2.690 A˚ which is due to the ratio of Zn. All the diffraction peaks can be indexed to a face-centered-cubic.
The different peaks for $Zn_xPb_{1-x}S$ film are indexed in Table (5) as well as the corresponding values of the interplanar spacing $d_{(hkl)}$ and compared with the values of PbS film.

The lattice constant (a) for $Zn_xPb_{1-x}S$ thin films, was 5.92A˚ which is less than the lattice constant for PbS film due to existence of Zn. From the table (5) we can see that adding the Zn to the PbS leads to decrease the lattice constant

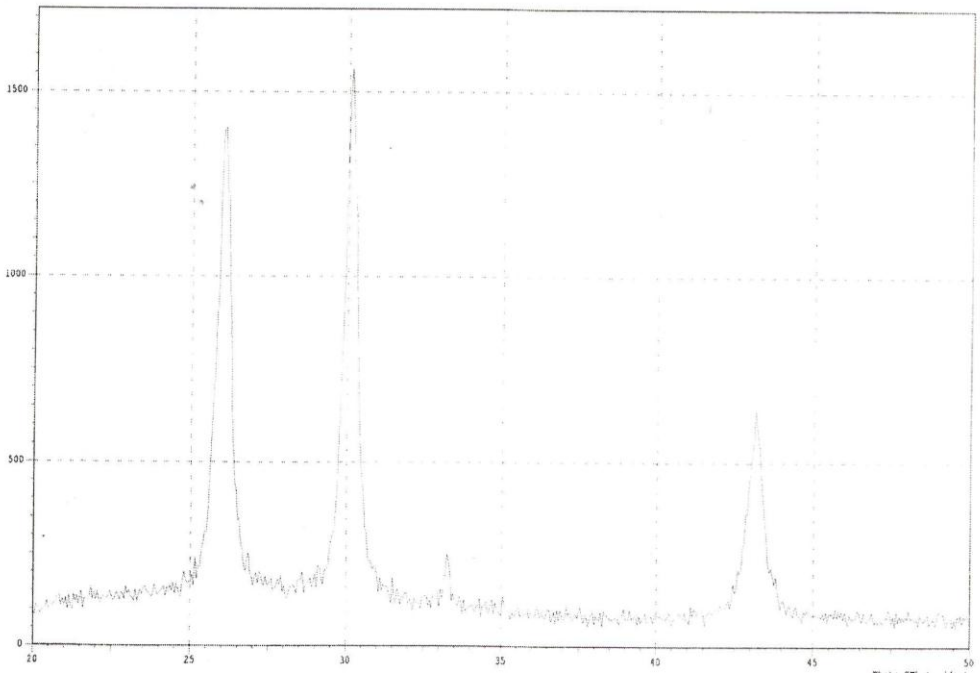



Figure (2).: X-ray diffraction pattern for $Zn_xPb_{1-x}S$ thin film (x = 0.02)

Table (5) Comparison of crystallographic data for $Zn_xPb_{1-x}S$ thin films with PbS film.

| **Observed d and a value for PbS film** | | | **Observed (d) and (a) value for $Zn_xPb_{1-x}S$ film** | | |
|---|---|---|---|---|---|
| **d (A°)** | **hkl** | **a (A°)** | **d (A°)** | **hkl** | **a (A°)** |
| **3.429** | **111** | **5.939** | **3.418** | **111** | **5.920** |
| **2.974** | **200** | **5.948** | **2.965** | **200** | **5.93** |
| **2.100** | **220** | **5.939** | **2.095** | **220** | **5.925** |
| | | | **2.690** | **unknown** | |

Optical Characteristics for $Zn_xPb_{1-x}S$ Films
*-at (x= 0.01)

There is the same behavior of optical constants for $Zn_xPb_{1-x}S$ with x = 0.01 as seen in the table (6) except decrease extinction coefficient and imaginary dielectric constant with increase the film thickness this is due to the effect of different thickness. And its energy gap decrease with increase the film thickness and it's in the range (2.38– 2.75 eV).

Table (6) Values of Refractive index n, Extinction coefficient k, Real $\varepsilon_r$ and Imaginary $\varepsilon_i$ Dielectric Constant for $Zn_xPb_{1-x}S$ films with x=0.01 (at 700 nm wavelength).

| **Thickness (nm)** | **n** | **K** | **$\varepsilon_r$** | **$\varepsilon_i$** | Eg (eV) |
|---|---|---|---|---|---|
| 790 | 1.411 | 0.0037 | 1.992 | 0.0107 | 2.75 |
| 1030 | 1.417 | 0.0029 | 2.009 | 0.0084 | 2.62 |
| 1300 | 1.421 | 0.0024 | 2.078 | 0.0068 | 2.38 |

From the table (5) and ( 6) we can see that due to decrease the ratio of Zn the optical constants increase. While the energy gap is decrease with decrease Zn ratio this behavior is a good agreement with the result of Al- Ajubory for $Zn_{x-1}Pb_xS$ films were prepared by spray pyrolysis technique [21].

**** at (x = 0.02)**

. Variation of $(\alpha h\nu)^2$ with $(h\nu)$ for $Zn_xPb_{1-x}S$ films is a straight line, indicating the presence of a direct transition.

It is observed from the table that the optical energy gap decreases with the increase of film thickness . The energy gap of $Zn_xPb_{1-x}S$ films in the range (2.4 – 2.9 eV) which is a good agreement with the work of Al-Ajubory for $Zn_{x-1}Pb_xS$ films by Spray pyrolysis technique [21].

The optical behavior of a material is generally utilized to determine its optical constants [i.e. refractive index (n), extinction coefficient (k), real ($\varepsilon_r$), and imaginary ($\varepsilon_i$) dielectric constants.

From the table we can observed that the refractive index increase with increase the thickness this is due to reflectance. as listed in table (7) this is similar the work of Sahay or ZnO Thin films by Spray Pyrolysis Technique [13].

The extinction coefficient was increases as the film thickness increases which is due to the different effective thickness of the thin films

The real and imaginary parts of the dielectric constant were showing in table (7). The variations of the real and imaginary parts of the dielectric constant with the incident photon wavelength shows that $\varepsilon_r$ increase with increase thickness

Table (7) Values of Refractive index n, Extinction coefficient k, Real $\varepsilon_r$ and Imaginary $\varepsilon_i$ Dielectric Constant for $Zn_xPb_{1-x}S$ films with x=0.02 (at 700 nm wavelength).

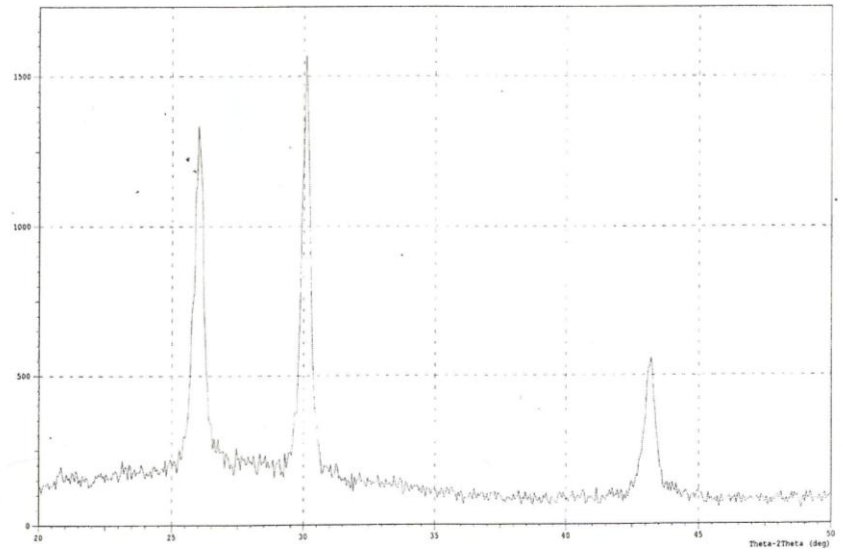
| **Thickness (nm)** | **n** | **K** | **$\varepsilon_r$** | **$\varepsilon_i$** | Eg (eV) |
|---|---|---|---|---|---|
| 790 | 1.31 | 0.0023 | 1.72 | 0.0061 | 2.9 |
| 1030 | 1.40 | 0.0028 | 1.96 | 0.0078 | 2.6 |
| 1300 | 1.53 | 0.0036 | 2.34 | 0.0112 | 2.4 |

**Structural and Optical Properties of $PbZn_xS_{1-x}$ Films:**

*** X-Ray Diffraction Results for $PbZn_xS_{1-x}$ films:**

X-ray diffraction pattern for $PbZn_xS_{1-x}$film (x = 0.01) was carried out with X-ray diffraction in the range 2θ between 20˚ to 50˚ shown in Figure (3). It is observed from this figure that for $PbZn_xS_{1-x}$ film of thickness (1500) nm was found to exhibit three diffraction peaks associated with (111), (200), and (220), with d value 3.419A˚, 2.965 A˚ and 2.094 A˚ respectively. All the diffraction peaks can be indexed to a face-centered-cubic rock. The different peaks for PbS film are indexed in Table (8) as well as the corresponding values of the interplanar spacing $d_{(hkl)}$ and compared with the PbS film.

The lattice constant (a) for $PbZn_xS_{1-x}$film, was (5.921A˚) which is close to the lattice constant of PbS film. Also from table (8) could be seen due to existence of Zn lattice constant would be decrease




Theta – 2Theta (deg)

Figure (3) :X-ray diffraction pattern for $PbZn_xS_{1-x}$ thin film (x = 0.01)

Table (8) Comparison of crystallographic data for $PbZn_xS_{1-x}$ thin films with PbS film.

| Observed (d) and (a) for PbS films | | | Observed (d) and (a) for $PbZn_xS_{1-x}$ films | | |
|---|---|---|---|---|---|
| d (A˚) | hkl | a (A˚) | d (A˚) | hkl | a (A˚) |
| 3.429 | 111 | 5.939 | 3.419 | 111 | 5.921 |
| 2.974 | 200 | 5.948 | 2.965 | 200 | 5.93 |
| 2.100 | 220 | 5.939 | 2,094 | 220 | 5.922 |

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**** The Optical Constants of $PbZn_xS_{1-x}$ Thin Films at (x= 0.01, 0.02):**

The optical constants for compound $PbZ_nxS_{1-x}$ for x = 0.01, 0.02 are shown in the table (9) and (10), as seen from the tables the refractive index increase with increase film thickness while extinction coefficient decrease with increase film thickness and both real and imaginary dielectric constant have the same behavior of refractive index and extinction coefficient respectively .The energy gap decrease with increase film thickness.

Table (9) shows the Values of Refractive index n, Extinction coefficient k, Real $\varepsilon_r$ and Imaginary $\varepsilon_i$ Dielectric Constant for $PbZn_xS_{1-x}$ films with x=0.01 (at 700 nm wavelength).

| **Thickness (nm)** | **n** | **K** | **$\varepsilon_r$** | **$\varepsilon_i$** | **Eg (eV)** |
|---|---|---|---|---|---|
| 790 | 1.324665 | 0.003642 | 1.754725 | 0.009648 | 1.4 |
| 1030 | 1.451133 | 0.00341 | 2.105761 | 0.014473 | 1.5 |
| 1300 | 1.465699 | 0.003607 | 2.148259 | 0.009605 | 1.6 |

Table (10) Values of Refractive index (n), Extinction coefficient (k), Real ($\varepsilon_r$) and Imaginary ($\varepsilon_i$) Dielectric Constant for $PbZn_xS_{1-x}$ films with x=0.02 (at 700 nm wavelength).

| **Thickness (nm)** | **n** | **K** | **$\varepsilon_r$** | **$\varepsilon_i$** | **Eg (eV)** |
|---|---|---|---|---|---|
| 790 | 1.355342 | 0.004248 | 1.836934 | 0.011515 | 1.57 |
| 1030 | 1.370304 | 0.003514 | 1.87772 | 0.00963 | 1.64 |
| 1300 | 1.467929 | 0.00429 | 2.154798 | 0.012596 | 1.8 |

Also from the table (9) and (10), we can see that increasing Zn ratio leads to increase the energy gap. And the energy gaps for $PbZ_nxS_{1-x}$ with x = 0.01, 0.02 are closed to the energy gap of PbS films because the ratio of Zn is very small compare to ratio of Pb & S.

**Conclusions :**

The following conclusions may be drawn from the results we obtained

1-XRD- studies showed that all films are polycrystalline with cubic phase except ZnS films have cubic and hexagonal phase.

2- Optical studies reveal that these films have a direct band gap and it's observed that the bad gap decrease with increase the film thickness.

3-The transmittance is high in VIS-NIR regions and the high transmittance properties makes film a good material for thermal control widow coating for cold climates and antireflective coating.

4- The refractive index (n) increase with increase the film thickness, while the extinction coefficient (k) decrease with increase the thickness for all the films except ZnS films which are inversely.

5- Spray pyrolysis method for production of the solid films is a good method for preparation of thin films suitable for scientific studies and for many applications in technology and industry.

4- Preparation of $PbZn_xS_{1-x}$ and $Pb_{1-x}Zn_xS$ due to another technique like electrical deposition, chemical deposition or thermal evaporation. And compare the results with this technique.

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