

Preparation and Study of the Optical Properties of ZnS Zinc Oxide Films Used in the Manufacture of Solar Cells Prepared by the Chemical Spray Pyrolysis Method

Sara Abbas Muslim Abbas

Department of medical physics , AL-Mustaqbal university College

Sarah jassim radhi sabih

Misan university College of science Department of physics

Ream sabah Habeb obed

Mustansiriya University College of Science Department of Physics

Zahraa Ali Mohammad Abboud

University of Babylon / College of Science / Department of General Physics

Sraa tariq sami saleh

al anbar university _college of science _Department of physics

Abstract:

In this research, copper oxide (ZnS) films used in solar cells were prepared by thermal chemical spraying method using aqueous copper chloride ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$) as a starting material at a concentration of (0.1M), and the films were prepared on glass substrates heated to a temperature of (350°C).

The optical properties of the (ZnS) thin film were studied (transmittance, absorbance, absorption coefficient, energy gap, reflectivity, refractive index, extinction coefficient and finally optical conductivity) and for the wavelength range (190-1100) nm, the transmittance recorded high values in the visible spectrum region, while the absorbance spectrum, which is high at short wavelengths, decreases at long wavelengths. In addition, the optical energy gap (Eg) of the thin film for direct

transition of both allowed and forbidden types was calculated and found to be equal to 2.113 eV and 2.095 eV respectively.

The results of optical tests showed that the absorption coefficient values of the films are greater than (10^4 cm^{-1}), which indicates the occurrence of direct electronic transitions.

As for the reflectivity and refractive index, it was found that they increase significantly at photon energies corresponding to the energy gap of all films. It was also found that the extinction coefficient increases at photon values less than the absorption edge and is followed by a clear increase. The optical conductivity was also studied and found to increase rapidly at photon energies close to the absorption edge until it reaches its maximum value.

Introduction

Semiconductors have been an important factor in the technical development of the world we live in now. These electrical and electronic devices and others are mainly based on transistors and diodes that are made of these materials, Semiconductors are important because their optical and electrical properties can be changed by changing the molar and weight ratios of the primary components of these materials

Interest in semiconductors began in the early nineteenth century to identify their properties and composition and the extent of their practical benefit due to the availability of these materials in nature in a very large way due to the unique features of these materials, as their physical properties are affected by heat, light, magnetic fields and the presence of small amounts of impurities.

Semiconductors are characterized by several properties, including:

- They have a specific resistance ranging between ($10^6 - 10^{-3}$) $\Omega \text{ cm}$.
- They have a resistance with a negative temperature coefficient.
- They have two types of charge carriers: electrons and holes.
- They are highly sensitive to light.
- The electrical conductivity can be increased by adding impurities, whether trivalent or pentavalent, etc.

In order to study the properties of semiconductor materials, it has become necessary to use new techniques to prepare these materials in the form of a thin film, as the behavior of these materials differs greatly when prepared in the form of a thin film than in their bulk form ((Bulk

The term thin films is to describe a layer or several layers of solid material atoms deposited on a solid substrate so that the thickness of the film is less than $1 \mu\text{m}$. Thin films are characterized by a high surface area compared to their size, so they exhibit properties that differ from bulk materials.((

Thin films were previously used to coat some materials to prevent their corrosion and oxidation or to make them appear beautiful with a metal coating or to show cheap alloys with valuable ones to increase their aesthetic and material value. Then the field of thin films developed and entered military applications when it was adopted in World War II to remove the effect of clouds in reflections from the surface of aircraft. Thin films are of great importance at the present time, as they are used in a large number of special fields, such as photoelectric sensitive panels, dry film manufacturing, photography, solar cells, and detectors. The semiconductor material prepared in the form of thin films has very wide uses and applications, especially in electronic industries such as the

manufacture of transistors, rectifiers, integrated circuits, and capacitors. It has also been used in optical fields such as the manufacture of mirrors and filters.

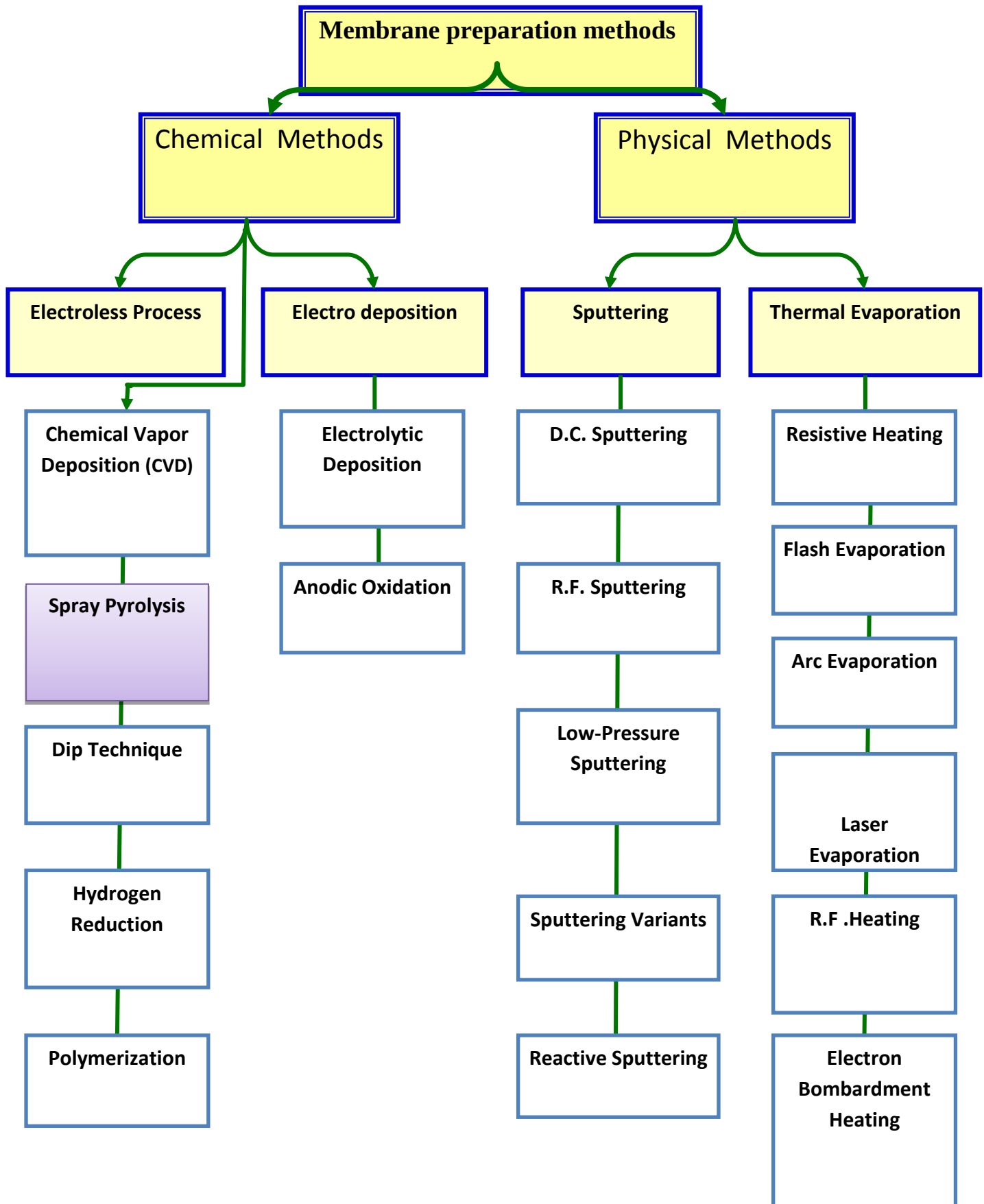


Figure (1-1) Thin film deposition methods

2-1:Description of the equipment used in preparing thin films:

The thermal chemical spray deposition system consists of several simple devices arranged in such a way that they can be used to prepare thin films on various substrates[16].

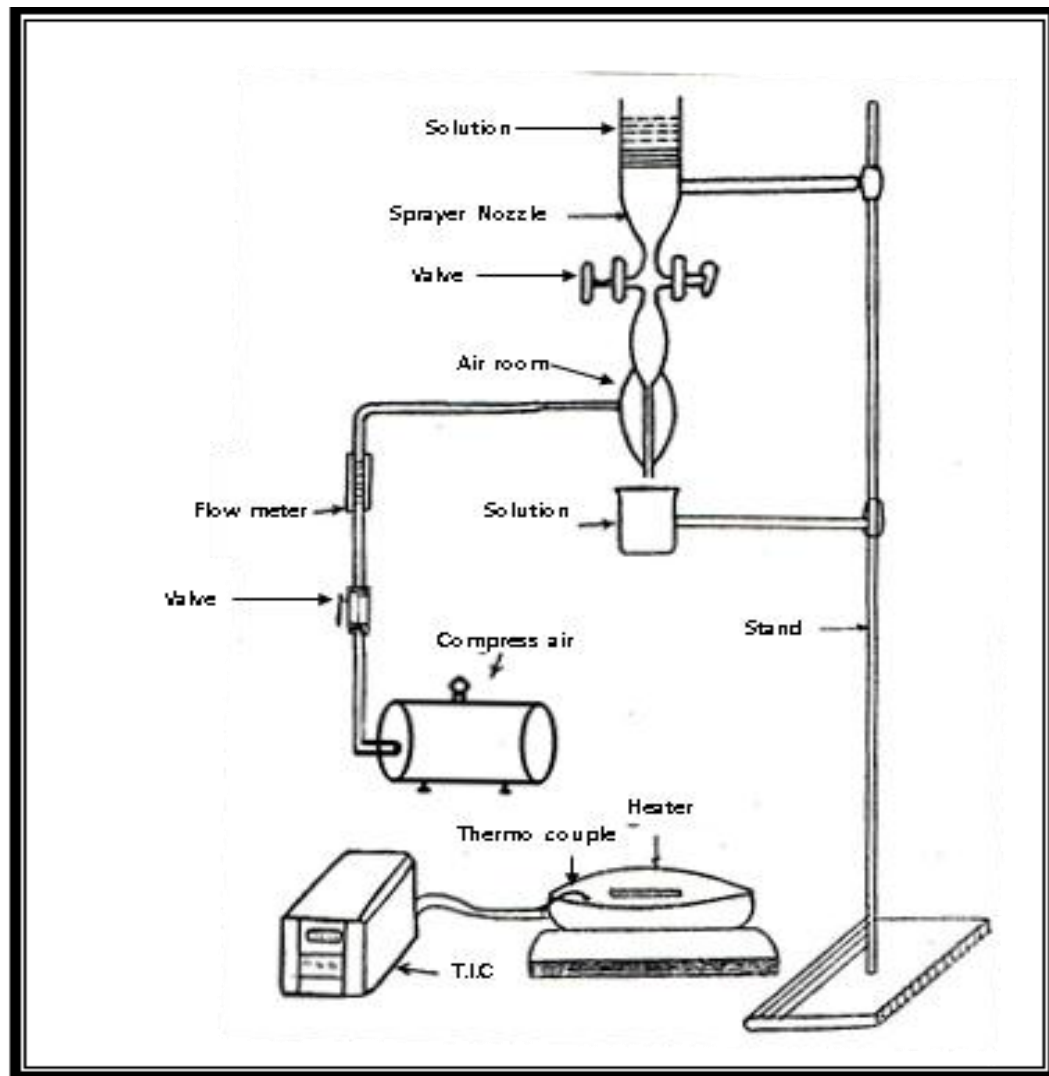


Figure (1-3) shows the thermal chemical spray system.

This system consists of the following devices:

1.Sprayer nozzle:[16].

It is one of the most important parts of the thermal chemical spray system, and through it the spray efficiency is controlled. It is made of chemically resistant glass and contains a tank of a suitable size in which the solutions to be sprayed are placed. It is connected from the bottom to a valve (tap) to control the amount of solution descending into a capillary tube with a diameter of (1 mm) and a length of approximately (60 mm) surrounded by a closed inflated glass chamber from the top due to its connection to the capillary tube and open from the bottom. The opening of the inflated glass chamber surrounds the opening of the capillary tube so that the two openings are at the same horizontal level and at the same center. The glass chamber contains a side opening through which compressed air passes to exit from its lower opening surrounding the sides of the opening of the

capillary tube. The length of the glass spray device consisting of the tank, valve, capillary tube and the surrounding chamber is approximately (25 cm). Figure (3-2) shows the parts of the spray device.

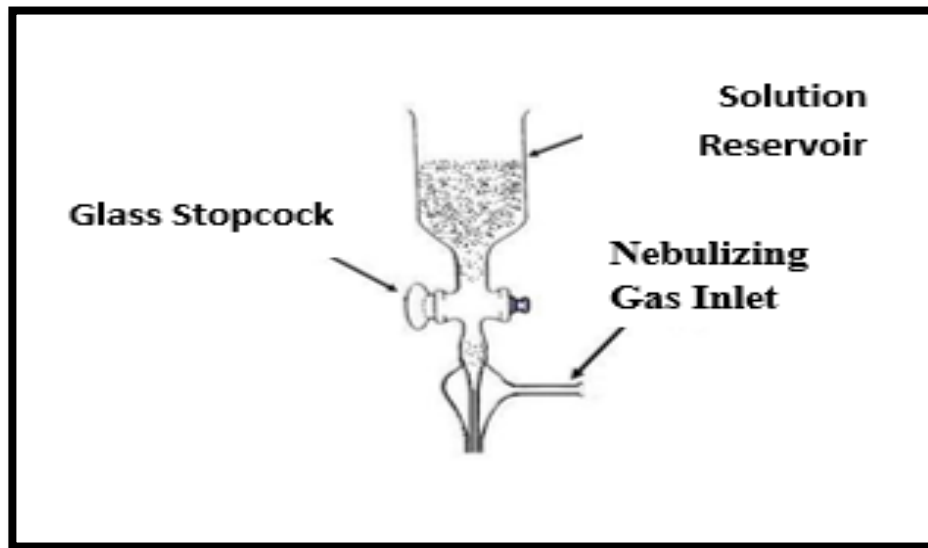


Figure (3-2) Spraying device

2 .Electrical heater: To raise the temperature of the base (on which the thin film is deposited), an electric heater (Hot plate) is used, with a temperature ranging between (80-700)°C.

3 :.Thermometer and Thermo couple: To know the temperature of the base surface, a thermocouple is used. It consists of a sensitive thermal probe placed on the base surface and connected to a digital meter that indicates the temperature in degrees Celsius, i.e. a thermometer. Figure (3-3) shows the electric heater and the thermocouple. Figure (3-4) shows the thermometer and the thermal controller.



Figure (3-3) Electric heater and thermocouple



Figure (3-4) Thermometer and thermal controller.

4. Compress Air : The air pump is used to push compressed air into the tube surrounding the capillary tube through the side opening, so that the air stream comes out at a certain pressure that leads to drawing the solution from the sprayer tank through the capillary tube to the surface of the hot base in the form of a cone-shaped spray with its bottom down and its head at the end of the capillary tube. The pump has a regulator that controls the air pressure entering between the sprayer tubes. Figure (3-5) shows the air pump.



Figure (3-5) Compressed air pump.

5 .Rotary pump

In order to preserve the prepared samples from damage due to various impurities present in the laboratory atmosphere, the samples are placed after preparation in a glass container with a tight lid connected at the top by a tube equipped with a valve to close and open the container and control the emptying of the container from air by means of the vacuum pump. Figure (3-6) shows the vacuum pump and the sample container. It is worth noting here that the spraying process of the liquid inside the spraying device is controlled in terms of the spraying time as well as the time period between one spray and another, as the system is equipped with an electronic timer that controls the pumping and stopping of air pumping to the spraying device. After each spray of the solution on the hot base,

we need a break period for the base to regain its temperature, which decreases due to the spraying of the cold solution on it.



Figure (3-6) Vacuum pump and model container.

1-3 Preparation of the solution used in preparing thin films- :

The solution used in preparing thin films was prepared from zinc chloride (ZnCl_2) at a concentration of (0.1M) with thiourea ($\text{Cs}(\text{NH}_2)_2$) at a concentration of (0.2M) according to equation [16].

$$M = \frac{W / M_w}{V(\text{litre})} \dots\dots\dots (3-1)$$

M: Molarity of solutions.

V: Volume in liters.

W: Weight of the substance in grams.

MW: Molecular weight of the substance g/mol.

The weight of the dissolved substance was about 4.262 gm)) from the equation

$$g_m = M_w * M * V / 1000 \dots\dots\dots (3-2)$$

The substance was dissolved in 100 ml of distilled water using a glass stirrer first, then using a magnetic mixer to ensure homogeneity and thus obtain a clear solution. It was prepared at laboratory temperature and stored in a volumetric flask.

1-4 Selection and cleaning of the bases: - The membranes were prepared on glass bases (Cover glass) with dimensions of (2.68X2.35 cm) and the triangle dimensions of (1.5, 0.2 cm) are added to them. These are thin bases that use very high temperatures, as there are glass bases with thicknesses of (1-1.2 cm). Here we differentiate that glass bases with a thin thickness of (0.1 cm) are better than bases with thicknesses of (1-1.2 cm) because the glass bases cannot be lowered in temperature when spraying, thus reducing the possibility of breaking them. Likewise, the possibility of the membrane cracking is almost non-existent, resulting from the difference in temperatures between the lower surface and the upper surface of the base. This base also has a property that quickly recovers the heat it lost during the deposition process (due to the cold solution and the air current), which ensures that the deposited material does not change as a result of the change in temperature. There is another property of glass bases with a thickness of (0.1 cm) which is their low absorption of light due to their thinness.

The process of cleaning glass bases includes several stages:

1. Then the base is cleaned using dilute sulfuric acid.
2. Then it is cleaned using ultrasonic waves using acetone and distilled water for 20 minutes.
3. Drying using a hot air stream in an air dryer.

These stages ensure that we obtain clean bases free of impurities and have a great impact on the prepared membrane and its composition

5-1 Thickness Measurement. The thickness of the film is the most important factor for the film to determine its suitability for study and measurement of its optical and electrical properties and constants. To measure the thickness of the film, homogeneous films free of defects and gaps are chosen. There are many methods for measuring the thickness of the film, including electrical, mechanical and optical. In this research, the gravimetric method was used to measure the thickness of the prepared thin films due to their ease and availability of their requirements. The following is a comprehensive description of this method:

- **Gravimetric Method:** After the completion of the cleaning process of the selected glass slides for the purposes of deposition, they are weighed using an electronic balance with a sensitivity of (10-4 gm) type (HR-200A&D Company). After the completion of the deposition process, the base is weighed to determine the weight difference between the two cases (Δm). After calculating the area of the film (A) deposited on the base, these variables can be known. The thickness of the prepared film can be measured using the following relationship:[2]

$$t = \frac{\Delta m}{\rho \cdot A} \dots\dots\dots (3-3)$$

Where:

t: Film thickness (cm)

m: Difference in mass before and after deposition (gm)

ρ : Density of the deposited film (gm/cm³)

A: Surface area of the film (cm²).

1. **6-Deposition of thin films:** After the glass bases have been cleaned and dried, and according to their thickness, they are placed on the electric hot surface and left for (20-15 min) before the deposition process to ensure that the temperature of the base surface reaches the required temperature, which is measured by a thermocouple that is placed on the path of the glass base surface. Here in our research, we used a temperature of 450 degrees Celsius, where when it reaches this temperature, the spray solution descent valve is opened and according to the required spray rate, as well as the air pump is opened. The spraying process continues for 15 sec)) after which the spraying process stops due to the decrease in the temperature of the device due to the cold solution, so we resume the spraying process after the temperature reaches the required degree of 450 degrees Celsius, and thus the spraying process continues periodically and according to the required thickness. Upon completion of the deposition process, the film is left on the surface of the heater after turning it off until it cools without trying to lift it to avoid breaking the film when trying to lift it suddenly from the surface of the heater due to the difference in temperature.

1-7. Results This chapter deals with the presentation and discussion of the results of the study of the optical properties of copper oxide films and their analysis by measuring the absorption and transmittance spectrum within the wavelengths (190-1100 nm) and calculating the values of the direct and indirect energy gap of both the allowed and the disallowed types and calculating the optical constants which included the absorption coefficient (α), the extinction coefficient (K) and the refractive index (n).

8-1(Optical Measurement) :

Transmittance & Absorbance -:Transmittance is defined as the ratio of the intensity of radiation transmitted through the material to the initial intensity of the radiation incident on the material. Information about the optical properties of the material can be obtained through the transmittance and reflectance spectra of different wavelengths, and through them many optical constants of the material are calculated. The transmittance spectrum of the prepared films was studied for a wide range of wavelengths (190-1100 nm). The transmittance increases with increasing wavelength, as it starts to increase from (400 nm) and continues to increase with increasing wavelengths of the prepared films, as shown in Figure (4-1), and the transmittance can be calculated from the relationship.(16-2) , As for the absorption spectrum, which is high at short wavelengths while it decreases at long wavelengths, it is known that the absorbance of materials is affected by several factors, including thickness, wavelength of the incident radiation and color of the material. The absorbance of ZnS films can be measured using the relationship (2-6). It was also noted that the absorption spectrum generally begins to decrease in the wavelength range between (400-800) nm and continues to decrease with increasing wavelengths, as shown in Figure.(1-4)

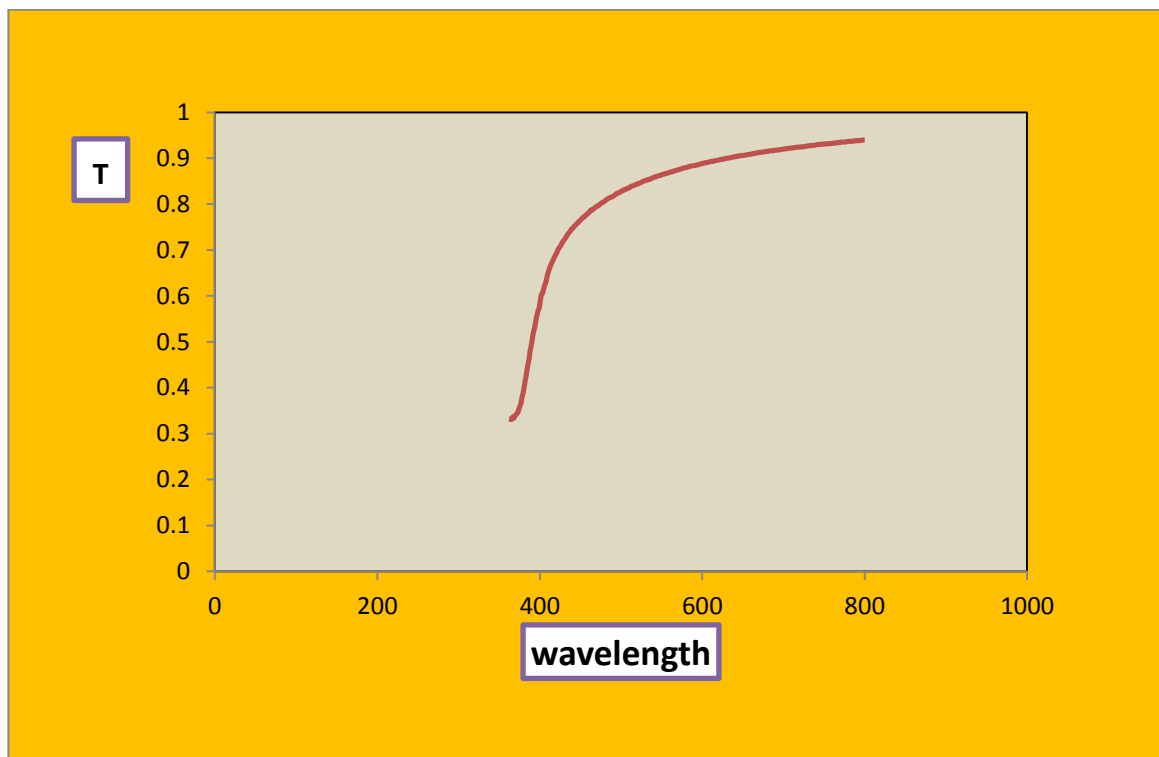


Figure (4-1) shows the spectrum of absorbance and transmittance as a function of wavelength.

Absorption Coefficient: The absorption coefficient (α) is defined as the percentage of decrease in the flux of radiation energies per unit distance in the direction of wave propagation within the medium. The absorption coefficient depends on the energy of the incident photons and on the properties of the semiconductor (energy gap) and the type of electronic transitions that occur between energy bands. The absorption coefficient also depends on the transmittance spectrum, and

is affected by the topography of the membrane surface (which affects reflectivity), as well as the chemical and crystalline composition of the material. When drawing the relationship between α as a function of photon energy ($h\nu$), it is noted that the absorption coefficient values in the high absorption region are greater than 10^4 , which indicates a high probability of direct electronic transitions. It also indicates that the point of rise in the absorption coefficient is the energy gap. Directly, that is, the charge carriers move from the extended levels in the valence band to the extended levels in the conduction band, thus generating an electron-hole pair. This process is called the fundamental absorption process (Fundamental Absorption). It is noted that the value of the absorption coefficient increases slightly in the low energy region (long wavelengths), while the increase becomes rapid in the higher energy region (shorter wavelengths). That is, this change becomes greater and increases rapidly near the edge of optical absorption in the energy range (1.5-3.39 eV). The absorption coefficient can be calculated from Equation (2-14). The absorption coefficient represented by the range before the absorption edge is attributed to the absorbance by impurities or transitions within the band and occurs at wavelengths where the energy of the incident photon is less than the forbidden energy gap.

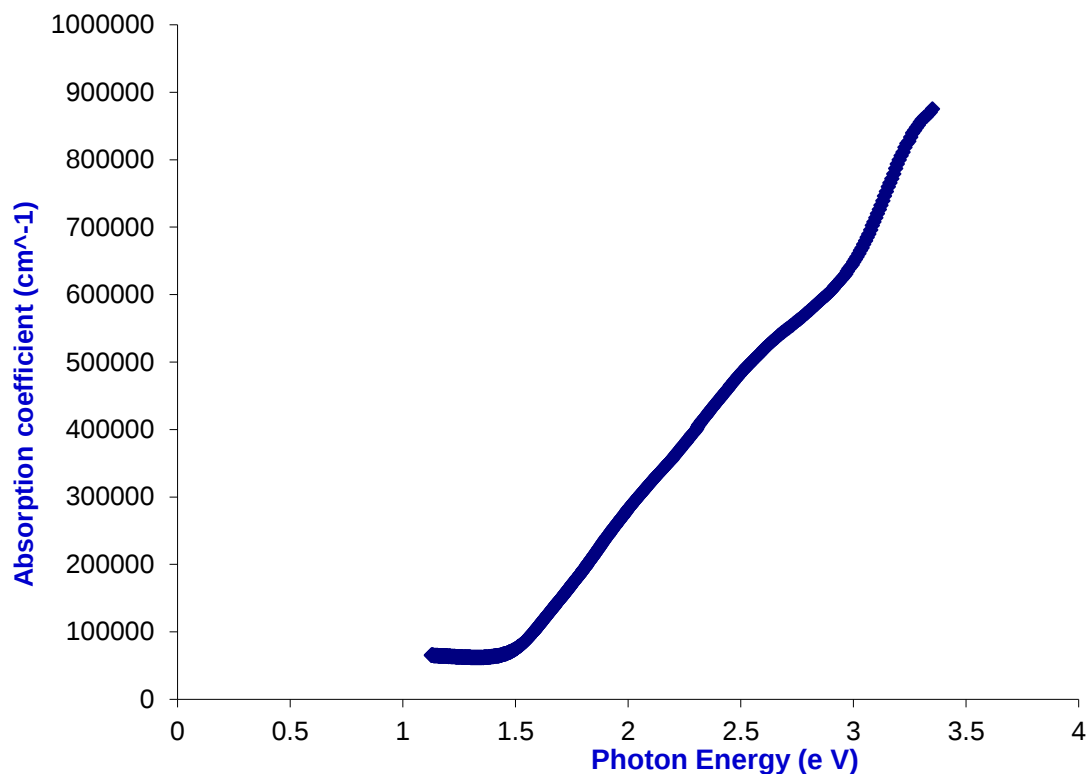


Figure (4-2) Change in absorption coefficient with incident photon energy.

Energy band gap: The optical energy gap is of great importance in determining the possibility of using thin films in the manufacture of solar cells, photovoltaic cells, display screens and other uses, as it gives a clear idea of optical absorption, as the film is transparent to radiation whose energy is less than the energy gap ($h\nu > E_g$) and absorbs radiation whose energy is greater than it ($h\nu < E_g$). The energy gap can be defined as the lowest energy required for the electron to move from the top of the valence band to the bottom of the conduction band, and is one of the most important optical properties of semiconductors. The values of the energy gap and its type depend on the type of material and the way the atoms are organized and distributed within the material, and are also affected by the type of impurities present within the structural composition. In order to calculate the value of the direct optical energy gap, the relationship between $2(\alpha h\nu)$ was drawn as a function of photon energy ($h\nu$) where the intersection of the straight part of the curve with the energy axis ($2 =$

$0(\hbar\omega)$ represents the value of the direct optical energy gap. A linear relationship was obtained for zinc sulfide (ZnS) material, which confirms that the films have a direct energy gap and that the basic direct absorption process allowed is dominant. Figures (4-3) and (4-4) show that the difference in the results of the energy gap depends on the factors specific to the thermal chemical spraying technique, as these factors affect the deposition rate and the variation in the crystal structure of these films, which leads to a change in the energy gap.

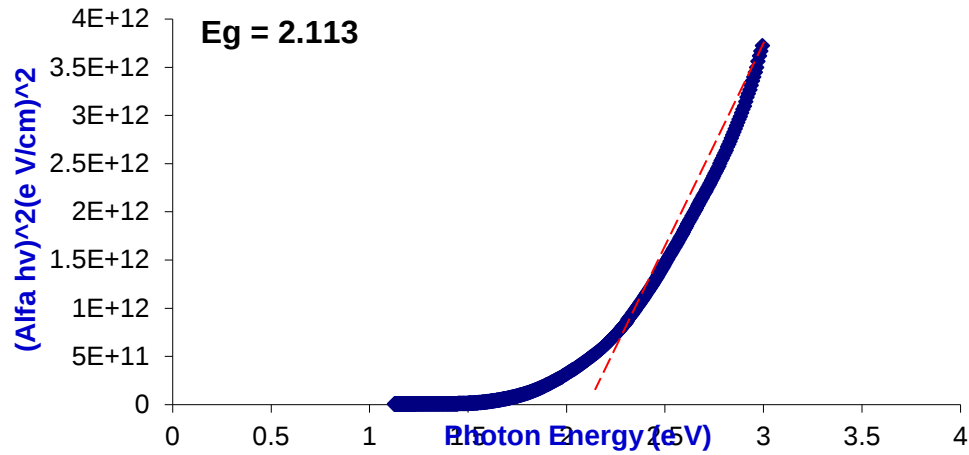


Figure (4-3) represents the value of the optical energy gap for the allowed direct transmission of the (ZnS) film.

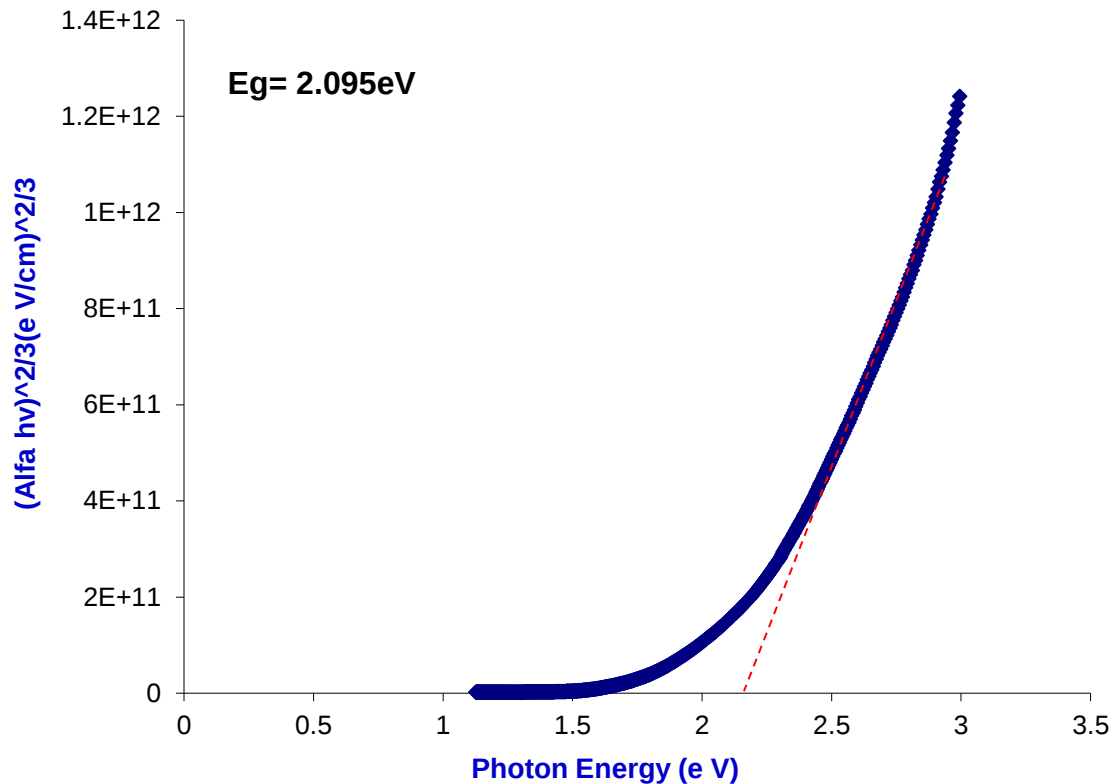


Figure (4-4) represents the value of the optical energy gap for the forbidden direct transition of the ZnS film.

Table (1-4) shows the values of the optical energy gap for the ZnS films.

Eg (eV)	Preparation Method	Year	Researcher(s)
2.65 – 2.05	Chemical Spray Pyrolysis	2011	Ban Khalid and her team
2.05 – 1.95	Chemical Spray Pyrolysis	2011	Ban Khalid and her team
1.40 – 1.30	Chemical Deposition	2009	Keilan Asaad and his team
2.1	Chemical Spray Pyrolysis	2012	Mustafa Aamer Hassan
1.4	Chemical Spray Pyrolysis	2012	Muhammad Muhibbullah
1.8	Chemical Spray Pyrolysis	-	Hanan Reda Abdul Ali
1.85 – 1.5	Chemical Spray Pyrolysis	2011	Issam Mohamed Ibrahim
1.8	Pulsed Laser Deposition	2010	Muslim Fadel and Heba Salam
1.7	Laser Deposition	2009	Muslim Fadel and Heba Salam [17]
1.73	Chemical Deposition	2011	Mohd Rafie and his team [9]
1.90	Sol-Gel-Like Dip	2001	Sekhar C. Ray [21]
1.7	Chemical Bath	2012	Ezenwa I. A. [20]
2.113	Chemical Spray Pyrolysis	2013	Current Study
2.095	Chemical Spray Pyrolysis	2013	Current Study

Reflectance (R): Reflectivity is defined as the reflected portion of the incident rays on the surface of the membrane. Reflectivity is calculated based on the transmittance spectrum (T) and the absorbance spectrum (A)] and according to the relationship (2-17). The reflectivity of the membrane increases rapidly at low energies, then a peak is formed at energies corresponding to the energy gap values, followed by a decrease in the reflectivity of the membrane at photon energies greater than the energy gap. This has been explained on the basis that absorption is negligible or very little at photon energies smaller than the energy gap of the material, accompanied by an increase in reflection from the surface of the membrane. When the photon energy becomes greater than or equal to the energy gap, a clear value of absorption appears and increases, resulting from the interaction of the electrons of the material with the incident photons with sufficient energy to cause electronic transitions, which causes a noticeable decrease in the reflectivity of the membrane .It was found that the maximum reflectivity of the membrane is at its energy gap of (2.113) eV.

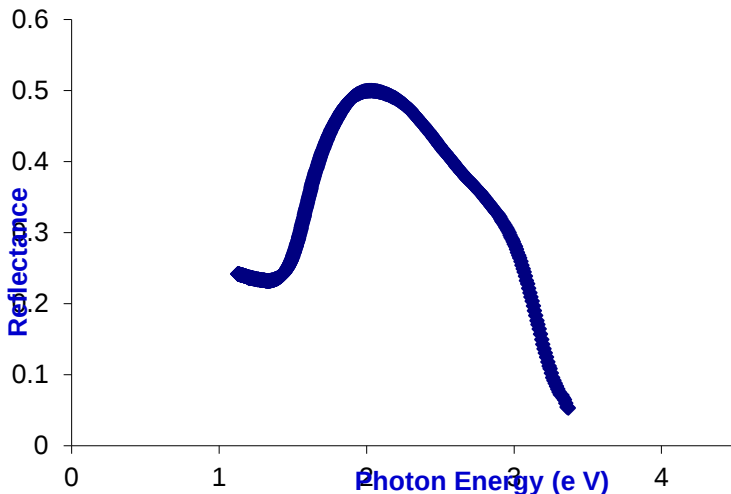


Figure (4-5) represents the change in spectral reflectivity as a function of photon energy for ZnS films.

Refractive Index(n): The refractive index is defined as the ratio of the wave speed in a vacuum to its speed in the medium, and the refractive index of the films was calculated from the relationship (23-2).

The refractive index (n) depends on several factors, including the type of material and its crystal structure, and that the refractive index changes according to the change in the particle size even if the crystal structure is the same. It is noted from Figure (4-6) that there is a peak in the refractive index curve at the energies corresponding to the energy gap, and the reason for the presence of this peak is the increase in the values of the refractive index of the film at low photon energies until it reaches the maximum value at the values of the energies corresponding to the energy gap, and then it suffers a decrease in its value, and this decrease is due to the increase in direct electronic transitions at those energies. We note that the nature of the refractive index curve is similar to the nature of the reflectivity curve due to the association of reflectivity with the refractive index.

The highest value reached by the refractive index curve is at (2.11eV), which approximately corresponds to the energy gap.

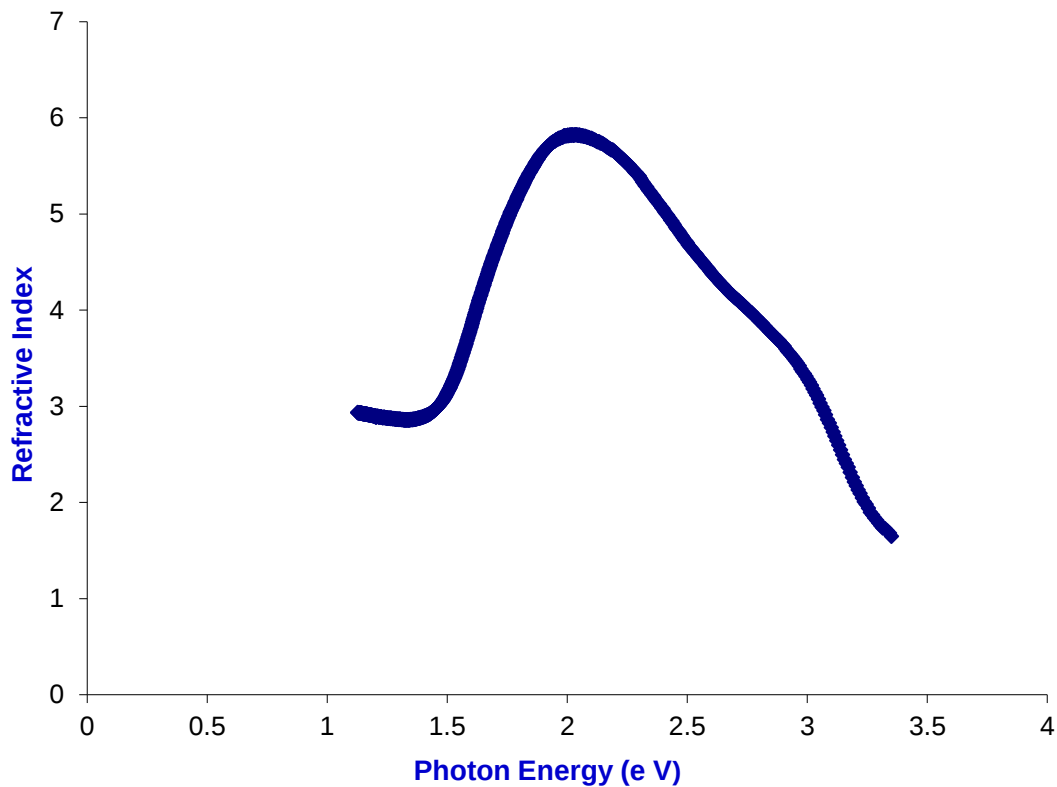


Figure (4-6) shows the change in the values of the refractive index as a function of photon energy.

Extinction Coefficient(K_0)

The extinction coefficient represents the imaginary part of the complex refractive index and is defined as the amount of energy loss due to the interaction between light and the charges of the medium or the amount of energy absorbed in the thin film, i.e. the amount of extinction of the electromagnetic wave inside the material. The extinction coefficient values (K_0) depend on the density of free electrons in the material and on structural defects. The extinction coefficient is affected by many factors, including the loss in the energy of the incident wave due to the absorption

process. This behavior is similar to the behavior of the absorption coefficient due to the clear dependence of the extinction coefficient on the absorption coefficient, and is calculated from Equation (2-15). We notice from Figure (4-7) that there is a gradual increase in the extinction coefficient values at photon values less than the absorption edge, followed by a clear increase. This increase indicates the occurrence of electronic transitions between the valence and conduction bands, which led to an increase in the absorption coefficient and then a clear increase in the extinction coefficient.

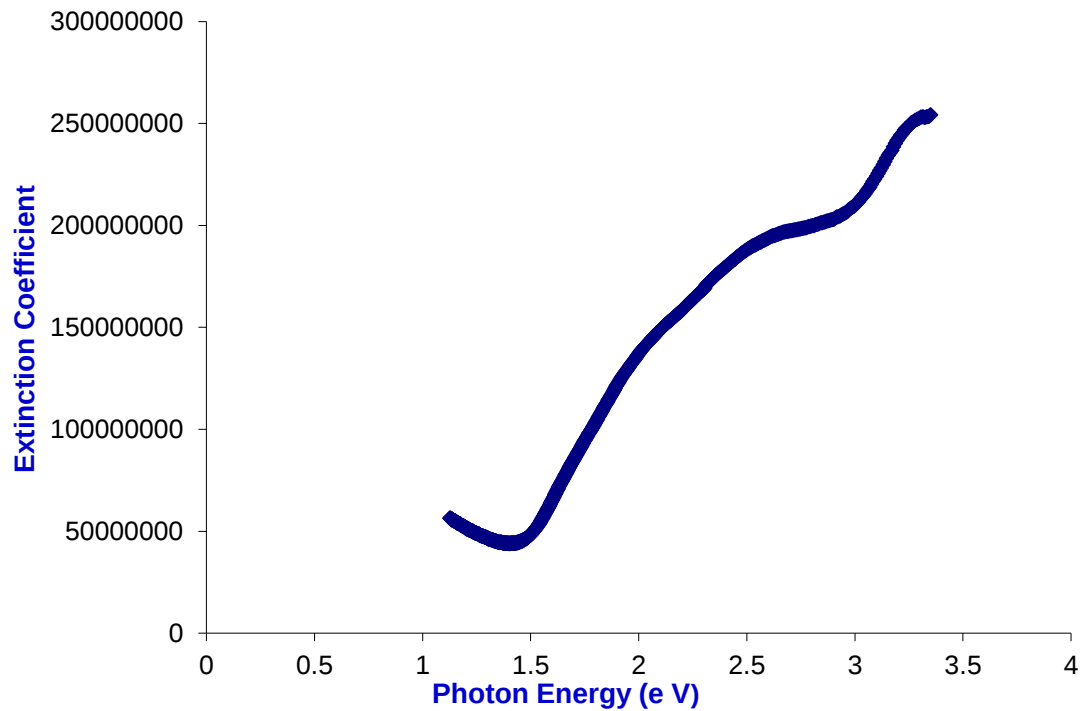


Figure (4-7) represents the extinction coefficient as a function of photon energy.

It is noted from Figure (4-8) that there is a peak in the photoresistance curve and the reason for the presence of this peak is the increase in the photoresistance values of the membrane at low photon energies until it reaches the maximum value at the energy values after which it suffers a decrease in its value and this decrease is the result of the increase in direct electronic transitions at those energies.

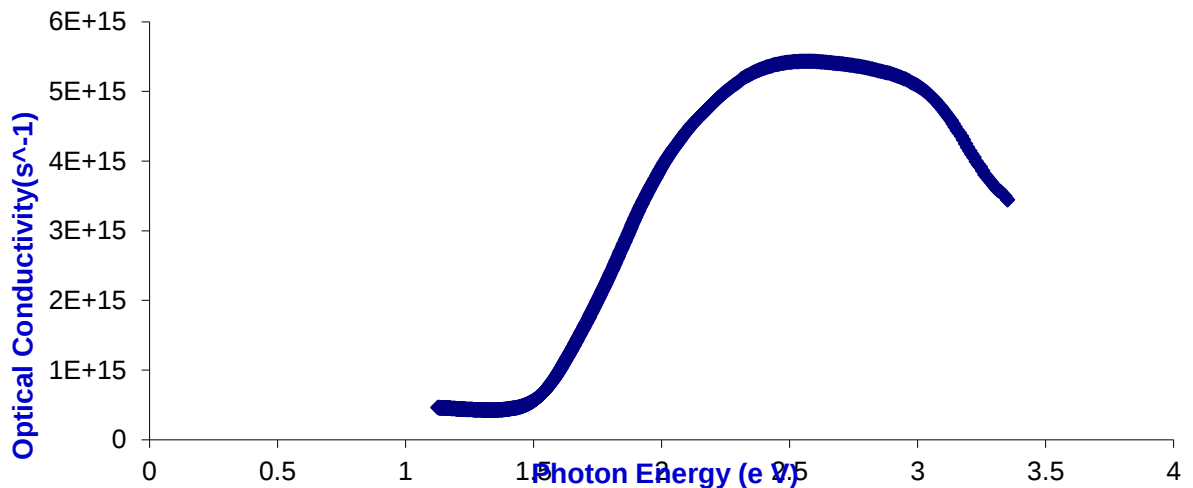


Figure (4-8) represents the change in photoresistance as a function of photon energy.

1. 9-Conclusions:-

After conducting optical examinations of zinc sulfide ZnS films, we can summarize the most important conclusions reached in the following points- :

1. The results showed that the films have high transmittance in the visible region to the near-infrared region. The transmittance value reached 92%.
2. The results showed that the calculated energy gap for the prepared films is of the direct transition type only.
3. The results of the optical examinations showed that the absorption coefficient values of all films are greater than (10^4 cm^{-1}) which indicates a high probability of direct electronic transitions and also indicates that the point of rise in the absorption coefficient is the direct energy gap.
4. The results showed a significant increase in the values of reflectivity (R) and refractive index (n) at photon energies corresponding to the energy gap for all films. From the above properties, it appears to us that this film can be used for some applications such as solar cells, optical coatings and blue light-emitting diodes.

1-11 Recommendations:-

1. Study the electrical properties (constant conductivity, Hall effect, thermoelectric power) of ZnS films
2. Study the structural properties of these films.
3. Prepare pure films by physical methods such as spraying and compare the results of their properties with the results of the current study.

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