MORPHOLOGICAL AND ELECTRICAL PROPERTIES OF SP DEPOSITED CADMIUM SULPHIDE THIN FILMS

Sundus[*] M. A. Al-dujayli, Nathera A. Al- Tememee, Ghuson H. Mohamed, Bahaa T. Chiad, Firas J. Kadhim and Bashair Abdul Rahman

Dept. of Physics College of Science, Baghdad University, Baghdad, Iraq

ABSTRACT

Thin film of CdS has been deposited onto clean glass substrate by using Spray pyrolysis technique. Results of Morphological (AFM) studied; electrical properties and optical conductivity studied are analysis. AFM results show a crystalline nature of the films. From the conductivity measurement at different temperatures, the activation energy of the films was calculated and found to be between 0.188 - 0.124 eV for low temperature regions, and between 1.67-1.19eV for high temperature regions. Hall measurements of electrical properties at room temperature show that the resistivity and mobility of CdS polycrystalline films deposited at 400 C^0, were 3.878×10^3 Ω. cm and $1.302 \times 10^4 cm^2/$ (V.s), respectively. The electrical conductivity of the films was found to be in the range of $10^{-4} - 10^{-6}$ (Ω. cm)$^{-1}$ with n-type of conduction mechanism, which is suitable as optical windows for efficient solar cells.

Keywords: CdS, Thin film, Spray pyrolysis, Electrical property, photoconductivity.

1. INTRODUCTION

In the past few years ago, the II-VI binary semiconducting compounds belonging to cadmium chalcogenide family e.g. CdS, CdSe, CdTe etc. which are considered to be very important materials for wide spectrum, optoelectronic application, particularly CdSe having band gap of 1.75 eV and CdS with band gap of 2.42 eV, which make them appropriate candidates for the conversion of low energy light into electricity [1]. Electrical conductivity is a fundamental parameter of solar energy materials, which is optimized to apply on the materials in the preparation of solar cells. The conductivity depends on the number of charge carrier, in turn is affected by various sources of scattering; when they (carriers) move through the material. Hence, precise knowledge about the mode of transport of carriers in the material is very important for its application, as an optical absorber in the preparation of solar cells

[2]. Polycrystalline CdS thin films is studied extensively because of its good posses of optical transmittance, wide and direct band gap transition (2.42 eV), photoconductivity, high absorption coefficient, reasonable conversion efficiency, good stability, low cost, electrical properties, high electron affinity and n-type conductivity [3,4,5] ,which makes it as one of the ideal material for their application to solar cell fabrication. In general, the pure CdS, either bulk or thin film, shows high resistivity of the order of $10^6 \Omega$ cm. This value is not suitable for graded layers in hetero-junction solar cells. In order to reduce the resistivity of the samples, one can either vary the ratio of Cd to S or add dopants such as Cu, Al and In [6].

CdS is great interest in the applications in electro-optic devices such as Light detector, dis-play panel, LED, optical windows for solar cells [7], photo-conducting cells, photo-sensors, laser materials, optical wave-guides and non-linear integrated optical devices, also used as photo- catalyst [8].

The deposition of CdS films has been explored by different techniques: sputtering, thermal evaporation, chemical bath deposition, pulsed laser deposition successive ionic layer adsorption and reaction (SILAR) and molecular beam epitaxy; in each of these methods polycrystalline, uniform and hard films are obtained, and their electrical properties are very sensitive to the method of preparation [9]. Spray pyrolysis is simple, inexpensive and enables doping and solid solutions to be accomplished [10]. Single phase, either hexagonal or cubic structure, and preferred orientation films can be prepared by controlling the physical parameters of spray pyrolysis such as substrate temperature, spray rate, solution concentration, etc.

In our previous paper [11], we have studied the preparation, structural, and optical properties of CdS thin films. The conductivity and the microstructures of CdS films mainly depend on the ratio of Cd salts (cadmium acetate) and S source (thiourea). Therefore, the aim of this present work is to study the influence of increase the volume ratio of thiourea in the solution (thiourea to the cadmium chloride) on the surface morphology and electrical properties of CdS thin films prepared by the Spray pyrolysis (Sp) deposition.

2. EXPERIMENTAL

The CdS thin films were prepared using spray pyrolsis by taking a 0.1 M aqueous solution of thiourea and 0.1 M aqueous solution of cadmium chloride as starting solutions. Also CdS thin films were prepared from solution having mole ratio of $Cd_X S_{2X}$ (where 25ml of $CdCl_2$ and 50ml of thiourea). The usual cleaning of the glass slides was first carried out and after that prepared the films from different numbers of sprays (6 sprays, 12 sprays, 15 spays). The solutions were mixed thoroughly, and the final solution was sprayed on to heated substrates which were kept at temperature 400^0C. The substrates are microscopic plane glass slides of area 2.5 x 7.6 cm^2. The time of spraying was short about 4 seconds, and the distance from the heater to the end of the capillary tube was 20cm. When the solution was sprayed the following reaction takes place at the surface of the heated substrate.

$$CdCl_2 + (NH_2)_2CS + 2H_2O \rightarrow CdS\downarrow + 2NH_4Cl\uparrow + CO_2\uparrow$$
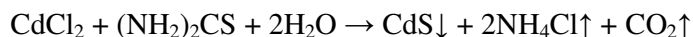
This yield is a yellow-orange deposit layer, and uniform growth of CdS films on substrates. The thickness of the CdS thin films was measured by using Stellar Net Inc spectrometer and Atomic force microscopy (AFM) was used for surface morphological analysis of CdS films. The D.c electrical conductivity of the films was carried out in the

temperature range of 293- 483K using digital electrometer type Keithely 616. Hall Effect measurements can be done using four-probe system to find out carrier density, mobilities and the type of conduction of the CdS thin films. The magnetic field B applied was 0.02 T and the measurement temperature was 293K. The good ohmic constants were obtained by electrodes Aluminum pasted on the surface of CdS thin films.

3. RESULT AND DISCUSSION

As shown in previous paper [11], the increase of the volume ratio of thiourea to the $CdCl_2$ on the CdS thin films is affected on the structural and optical properties of the films. In our work, also this increment is affected on the electrical conductivity properties, the morphology and optical conductivity of the CdS thin films.

3.1 Morphological characterization

In our previous paper [11], we have studied the structure of CdS thin films. Hence, it was confirmed from X-ray diffraction analysis that all the films exhibited a polycrystalline nature and hexagonal structure with (002) as the preferred orientation.

The surface morphology and microstructure of the films could be controlled by varying the processing conditions (e.g. substrate temperature and precursor concentration), reducing the droplet size of the aerosol and ensuring the heterogeneous reactions occurring during deposition. In the polycrystalline thin film, the extinction coefficient of light depends on not only absorption by atoms but also the surface roughness and polycrystalline grain size. Therefore; AFM images were taken in all deposited films in order to study the surface morphology and their relations with other properties, as is shown in fig. 1. Fig. 1 shows an AFM image of a CdS films deposited at 400 ^{0}C using precursor solution with volume ratio (Cd: S. 25:50). We observe that the films have a grain-like surface morphology. The AFM images provide some quantitative data about the surface roughness R_a, root mean square, R_{rms}, and crystallite size of the studied thin forms are listed in table 1. Clearly, the grain size and the surface roughness changed with number of sprays. From the table 1, the grain size increased with increasing number of sprays. This can also be explained by the mobility changes of the particles. The particles with higher mobility easily form larger sized grains. In as deposited thin film of CdS, there is some lattice defects, geometrical and physical imperfections randomly distributed on the surface, and the volume of the film. The roughness of the surface, grain boundaries and inclusions in the volume are the main components of the geometrical imperfection. The film is composed of randomly oriented grains with the appearance of the grain boundaries. The important factor, which is responsible for the physical properties of thin film, is the structure. An increase in the number of sprays of the film affects the structure significantly causing a considerable increase in the main size of the grain and a decrease in the grain boundary area, as show in table 1. As a result of the grain size changing, as can be found in the table, the surface roughness's increased from 2.23 nm to 10.3 nm for the sample C1 and sample C3, respectively. In the case of sample C1 films the surface roughness's are smaller in comparison with those for sample C3. The increase in surface roughness is considered to be due to an increasing the crystalline size and to improved film uniformity.

The small grain size is undesirable for most semiconductor applications because of the barrier effects of grain boundary on the mobility in planar direction as mentioned below. Hence, a large grain size and smooth surface are best suited for solar cell applications [12].

Therefore; there are two possibilities to improve the crystalline of the films: an increase in grain size to decrease the number of grain boundaries and a decrease in barrier height at the grain boundaries [13]. The surface crystallized and porous film to more crystallized dense films as the number of sprays increases. It is observed from the figure that there is a uniform distribution of particles in the sample C3 and increase in coalescence and the crystalline property also increase. As result, it has been found that the increase in volume ratio of thiourea to $CdCl_2$ on the CdS thin films significantly affects the produced films.

The crystalline size distribution for two studied samples is represented in fig.2, the films show an enlargement of the large crystalline and the structure is also strongly densities. This process is accompanied by a reduction of the crystallite high.

Table 1: Roughness parameters of CdS thin films

Sample Code	Number sprays	Thickness (nm)	Grain size(nm)	R_a (nm)	R_{rms} (nm)
C1	6	61.7	114.96	2.23	2.93
C2	12	56.4	192.50	10.8	14.1
C3	15	33.0	268.13	10.3	13.1

(a)   (b)   (c)
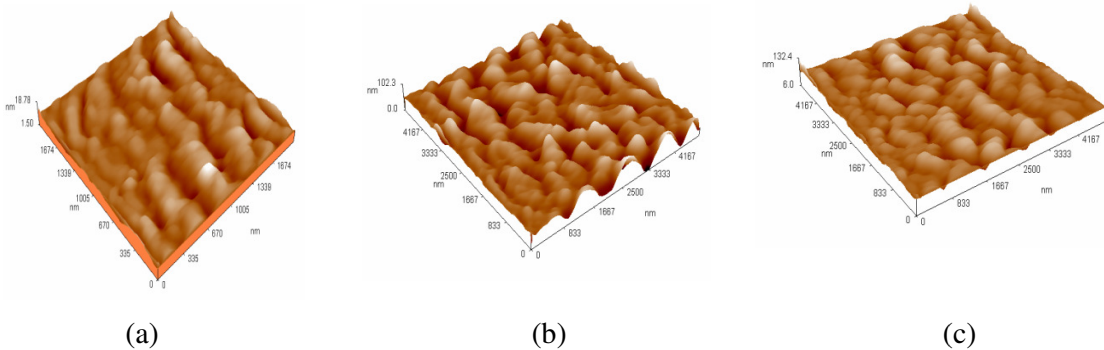
Fig. 1. Three dimensional AFM images of the surface of CdS films: a) sample C1, b) sample C2 and c) sample C3

(a)   (b)
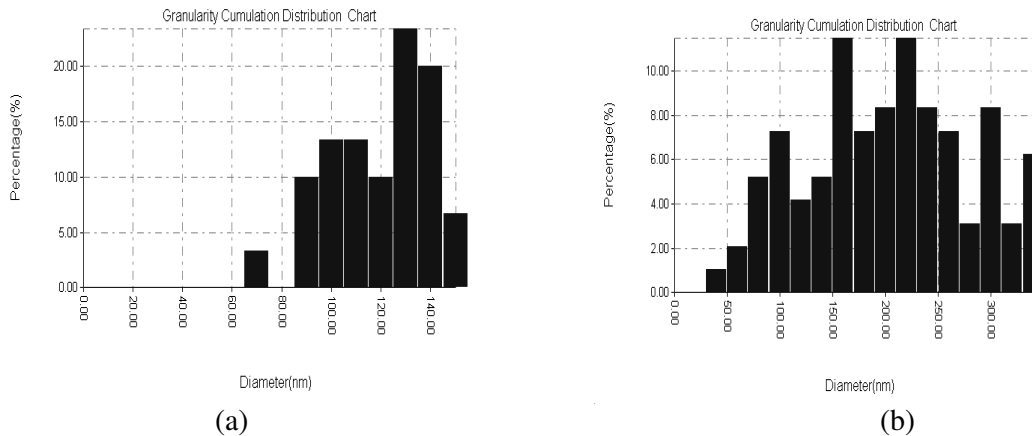
Fig. 2. Grain height distributions of CdS: a) sample C1; b) sample C3

3.2 Electrical properties

It is known [14, 15] that there is a strong correlation between the electronic transport proper- ties of polycrystalline semiconducting films and their structural characteristics. Particularly, both the values and the variation of the electrical conductivity of such films are in connection with their structure and its changes. On this basis, the study of the temperature dependence of the electrical properties of the films may offer useful information about possible changes of the structural characteristics of the films. The conductivity of CdS thin films were measured in the temperature range of 293-483K. The conductivity σ of the films was calculated from the relation:

$$\sigma = L/ RA \qquad \text{(where } \sigma =1/ \rho) \qquad (2)$$

Where R is the resistance of the sample, A is the cross sectional area of the sample and L is the distance between the electrodes. Temperature dependence DC electrical conductivity of samples has been shown in Fig. 3, this figure can observed the conductivity increases non-linearly with temperature for these three samples. This increase in conductivity with increase of temperature is attributed to improvement of charge density. This is explained in terms of structural changes occurring in this thin film with temperature. An increase of temperature of the films affects the structure significantly causing a considerable increase in the size of the grain [16] and a decrease in the grain boundary area. This decrease is due to the migration of the smaller crystallites and joining of those grains, which are similarly oriented, to form bigger crystallites. Because of these structural changes the inter-grain boundary area decreases i.e. there is a decrease in the scattering of the electron. Consequently, the mobility in the inter-grain domains also increases as shown below. This in turn increases the conductivity in order to $2\times10^{-3}(\Omega. \text{cm})^{-1}$ as show from figure 3.
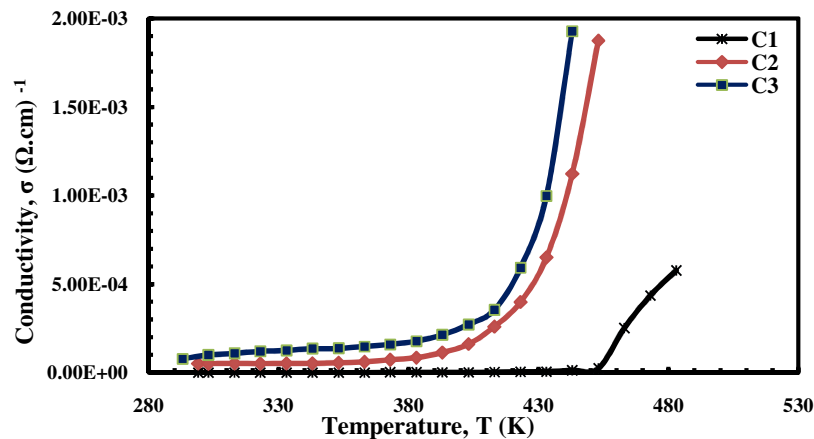

Fig.3. Variation of electrical conductivity with temperature for CdS thin films

The temperature dependence of electrical conductivity (σ) of three samples of CdS thin films follows the Arrhenius law and is given by [17]:

$$\sigma = \sigma_o \exp (-E_a / K_\beta T) \qquad (1)$$

Where, σ_o is the pre-exponential factor or temperature independent conductivity, E_a is the activation energy of the electrical conduction measured from the bottom of the conduction band, K_β is the Boltzmann constant, T is the absolute temperature. Fig.4 shows the graph between ln σ versus 1000/T plots for three representative CdS thin films. The electrical conductivity of all thin films increases with the increasing temperature this indicates to semiconducting nature of the CdS thin films [18] [19]. Conductivity in these films is due to both hopping of holes and charge transport via excited states. On the other hand, the non-linear nature of the plot indicates two regions conduction mechanism one at high temperature (403-483) K and the other at low temperature (303-403) K. From this graph, the slope is less in the low temperature region but increases with further increase of temperature. In such case, the activation energy has been determined for these three samples, and, listed in table 2.

In low temperature regions, the conduction is due to carriers excited into localized states at the band edges. The charge carriers in conduction band may be few to give rise to an appreciable conduction. Hence, the hopping of charge carriers via the impurity band can produce a current in the films at low temperature [20]. Hence, E_{a2} are the activation energy needed to excite the carriers from the corresponding trap levels to the conduction band and are associated with the impurity conduction.

In the high temperature region, the conductivity is strongly dependent on the temperature. Hence, the conduction mechanism of E_{a1} in this case is due to carriers excited beyond the mobility edges into extended states, and, associated with an intrinsic generation process. It is seen from table 2 the activation energies that the films posses the intrinsic conductivity strictly in this range of applied temperature. At these temperatures, the conductivity may be attributed to the thermal excitation of the carriers from the grain boundaries to the neutral region of grains.
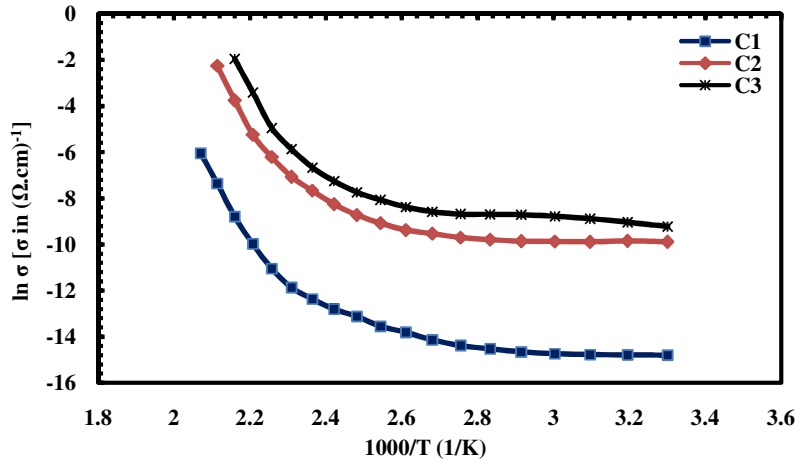

Fig.4. Conductivity (σ) versus 1000/T characteristics for three CdS films

The activation energy of the films in the low temperature region is smaller than the activation energy in the high temperature region. This finding conforms to the conduction mechanism. From plot the activation energy E_{a1} of three CdS thin films were found to be decrease from 1.673 eV to 1.199 eV. The decrease in activation energy suggests that the grain boundary scattering contribution reduces significantly and removal of defect (vacancies) present in the films. Similar characteristics have been reported by other workers [21]. From

table 2, it is observed that the value of activation energy of electrical conduction is lowest for sample C3, which confirms the higher conductivity of sample C3 among all samples. Therefore, it can be observed from figure 4 that the electrical conductivity increases with the increase in the number of sprays. This is probably attributed to better crystalline perfection of the films result from improved film struc-ture and possible due to decrease in band gap and can also be related to increased mobility of charge carrier resulting from the excess thiourea to the cadmium chloride [22]. The lesser conductivity observed at low number of sprays can be explained as due to the lower degree of crystalline and the smaller grain size, the effect of the grain boundaries is dominant when the grin size is small as correlated with XRD results as previously mentioned in our previous paper [11].

In general, the electron transport properties are affected by the presence of a number of defects such as structural disorders, dislocations and surface imperfections. The variation of grain size could also influence the activation energy [23]. Since a polycrystalline film has crystallites joined at their surfaces via grain boundaries, the boundaries between crystallites play an important role in determining the conductivity of polycrystalline films. Hence, an increase in grain size can cause a decrease in grain boundary scattering, which leads to an increase in conductivity. This indicates that the crystallinity of CdS films is improved corresponding to the samples prepared at 15 sprays.

Table 2: Activation energies for different temperature ranges of CdS thin films

Sample Code	Number sprays	Temp. range K	Ea_2 eV	Temp. range K	Ea_1 eV	Band gap eV	Band gap eV
C1	6	303-413	0.188	413-483	1.673	3.34	3. 57
C2	12	303-403	0.103	403-473	1.240	2.48	2. 43
C3	15	303-403	0.124	403-473	1.199	2.39	2. 38

The Hall-effect measurements for the three samples of CdS thin films were performed at room temperature. The resistivity ρ, Hall mobility μ_H, and carrier concentration N of deposited samples were determined using Hall-effect measurements and the values are shown in table 3. From table 3, it is observed that the electrical resistivity was in order of 10^5 Ω cm for CdS film have high amount of sulfide which was decreases to 10^3 Ω cm with increasing of the number of sprays because the sublimation of the sulfide during the sprayed process.

Table 3: Hall parameter of CdS thin films

Sample Code	μ_H cm^2/V. s	σ (Ω. cm)$^{-1}$	R_H cm^2/C	ρ Ω. cm	N (cm)$^{-3}$	υ_d 10^3 cm/s	τ 10^{-6} μ sec	ℓ 10^{-2} cm	Type of conductivity
C1 (6 sprays)	75.3	5.92x10^{-6}	-1.27x10^7	1.68x10^5	-4.91x10^{11}	1.503	0.089	0.0135	n-type
C2 (12 sprays)	127.8	5.13x10^{-6}	-2.49 x10^7	1.94x10^5	-2.50x10^{11}	2.238	0.152	0.0340	n-type
C3 (15 sprays)	13020	2.57x10^{-4}	-5.04x10^7	3.87x10^3	-1.23x10^{11}	6.094	15.53	9.464	n-type

The resistivity is proportional to the reciprocal of product of the free carrier concentration N and the mobility μ_H as is shown the following relation [24]:

$$\rho = 1/ N \, e \, \mu_H \qquad\qquad (3)$$

The resistivity of the CdS films was dominated by the free carrier concentration and mobility.   The low mobility and carrier concentration may result in high resistivity. It is seen in Fig. 5, and table 3, that the resistivity of the CdS films decreased from 1.68 $x10^5$ to 3.7 $x10^3$ Ω.cm with increase in number of sprays, and these values are lower than the literature values [25] in which the CdS thin films were prepared by spray pyrolysis technique. The decrease in electrical resistivity with the increase the number of sprays is due to the improvement in crystallite and increase in the grain size, decrease in defects and grain boundary discontinuities, which leads to an increase in conductivity. The lowest resistivity value calculated as $3.7x10^3$ Ω·cm is very suitable for solar cells. Moreover, it was clearly seen from Fig. 4 that the CdS film at simple C3 (15 sprays) has the best conductivity value.

A higher resistance for CdS film have high amount of sulfide (simple C1) could be attributed to surface scattering. The defects act as scattering centers which results in the formation of trapping states capable of trapping carriers and thereby immobilizing them. This trapping the mobile carriers, the traps become electrically charged and give rise to potential barriers, which impeded the motion of carriers from one crystalline to another, thereby reducing their mobility as shows in table 3. The order of resistivity in the present work is in the range of $10^3 - 10^5$ Ω. cm, which is suitable for efficient solar cells. These results of mobility and resistivity are quite similar to those obtained by Seto [26], Papageorgiou and Evangelakis [27], Duchemin et al. [28] and Ashour et al. [29], on sprayed and vacuum evaporated films by a modified source.

The negative sign in Hall coefficient and carrier concentration indicates that the all CdS thin films have n-type conductivity. The charge mobility value ranges from 75.3 cm^2/Vs to 1.3x 10^4 cm^2/Vs. therefore, the sample C3 film was found to have mobility higher than the other two films, from this can be understood by considering the grain size measurements as shown in table 1; the grain size of sample C3 film is much larger than that of the two films which explains the higher mobility. The larger values of electron mobility make an increase in electrical conductivity, as observed in fig. 5 which illustrated that the mobility of the deposited film at sample C3 (15 sprays) will rapidly increased with increase number of sprays, which attributed to
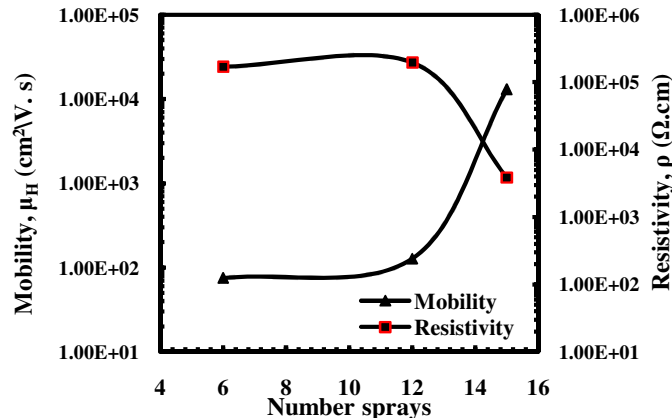

Fig.5. Mobility and resistivity as a function of number of sprays

the reduction of the scattering of the carriers from the surface as well as due to the elimination of the defects in the films and increase in crystalline which consequently decrease the number of grain boundaries. This result conformed by table 2, sample C3 gives maximum conductivity while sample C1 has the lowest conductivity at room temperature among all the samples these changes in the conductivity due to excess thiourea to the cadmium chloride.

From the Hall mobility measurements, we can calculate the drift velocity (υ_d), carrier lifetime (τ) and mean free path (ℓ) by using the following relations:

$$\upsilon_d = \mu_H E \tag{4}$$

$$\tau = \mu_H m^* / e \tag{5}$$

$$\ell = \upsilon_d \tau \tag{6}$$

Where, E is electric field, m^* is the effective mass of free carrier and e is the electron charge (1.6×10^{-19} coulombs). From table 3, it is found that these parameters are increased with increasing of number of sprays. This is explained in terms of increasing μ_H with the number of sprays is due to the improvement in the crystallite and this due to reduce in the scattering of the carrier from the surface and increase in crystallite size leads to decrease in the number of grain boundaries as mentioned previously.

All the results shown above illustrate that the increase of the volume ratio of thiourea to the $CdCl_2$ has a strong influence on the electrical properties of CdS thin films and that it is a good technique to improve the electrical conductivity of the deposited CdS thin films.

3.3 Optical Conductivity (σ_{op})

The optical conductivity, σ_{op} (the frequency response of a material when irradiated by light) of the films was also calculated using the relation [30]:

$$\sigma_{op} = \alpha n c \backslash 4\pi \tag{7}$$

Where α is the absorption coefficient of the deposited thin film, n is the refractive index, c is the velocity of light. The graph of optical conductivity of the specimens against photon energy was plotted as shown in fig. 6. The optical conductivity of the specimens varies with photon energy in approximation similar manner. It is observed that the conductivity increases sharply to various maximum values of about 4.65×10^{15} s^{-1} at 2.53eV ($\lambda = 490$ nm) for Sample C3, 2.71×10^{15} s^{-1} at 2.53eV for Sample C2 but the sample C1 increasing gradually to approach constant values to 2.5×10^{15} s^{-1} at 3.54 eV ($\lambda = 350$ nm) and after that, it is decreased with photon energy. While in sample C3 the fluctuations were observed after decreased. This behavior is caused by the dependency optical conductivity on the nature of refractive index and absorption coefficient because at the UV region α and n are greatest and diminishes as the wavelength increases.
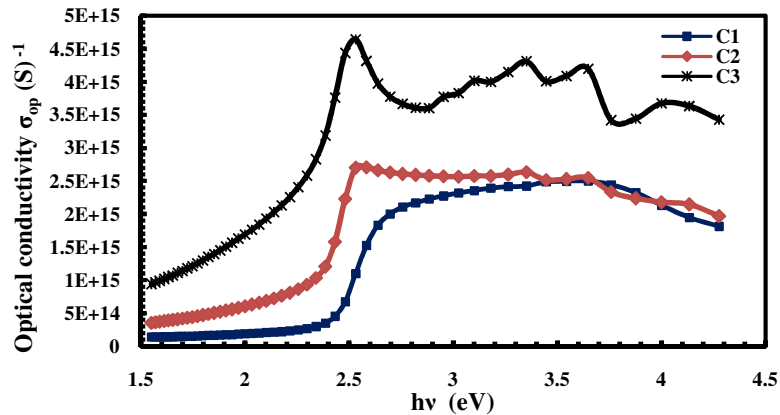
Fig.6. the optical conductivity vs. the photon energy for C1, C2 and C3 of CdS thin films

4. CONCLUSIONS

CdS films were fabricated by a spray pyrolysis technique using a solution with volume ratio of cadmium chloride and thiourea (Cd:S. 25:50). The films were deposited onto glass substrates at the temperatures of 400 ^{0}C. The surface morphology of CdS of thin films has analyzed by AFM. The grain size becomes larger and the morphology becomes denser. The films appeared to have two activation energies, the activation energies are observed to be decreasing with increasing number of sprays. From Hall-effect measurements, it is observed that the electrical resistivity of CdS thin films decreased from 1.68 x10^5 to 3.7 x 10^3 Ω. cm and increases in conductivity, in order of 10^{-3} (Ω. cm)$^-$1, with increasing number of sprays. The films have good electrical properties larger grain size and are well suited for solar cell applications. These results indicate that films obtained at 15 sprays (sample C3) with low resistivity, high mobility and high conductivity with n-type of transport property by SP are good candidates to be applied in different optoelectronic devices.

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