

The effect of Pb concentration and annealing temperatures on optical properties of Pb_xS_{1-x} films

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Abstract

A polycrystalline lead sulfide Pb_xS_{1-x} alloys with various Pb concentrations ($x=0.50, 0.51, 0.52, 0.54$ and 0.55) has been prepared successfully. Pb_xS_{1-x} films of thickness $1.5\mu\text{m}$ have been deposited on n-Si substrates by flash thermal evaporation method at room temperature (RT) under vacuum 10^{-6} torr and deposition rate $0.83 \times 10^{-3} \mu\text{m}/\text{sec}$. These films have been annealed at different annealing temperatures ($T_a = \text{RT}, 323, 373, 423, 473$ and 523) K.

The optical measurement showed that Pb_xS_{1-x} films have a direct energy band gap, which increases with increasing T_a and decreases with increasing Pb concentration. The absorption coefficient, refractive index, extinction coefficient, dielectric constant, and the width of the tails of localized states in the gap decrease with increasing T_a and increase with increasing Pb concentration.

Keywords: Pb_xS_{1-x} Films; Optical constants; Energy gap; Urbach energy.

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1. Introduction

Semiconductors have found widespread industrial and scientific applications as remote temperature sensors, spectrometers, and in infrared imaging system [1]. A wide range of binary compounds and ternary alloys have been used to fabricate IR detectors. PbS is a direct narrow gap semiconductor very suitable for infrared detection application. This material has also been used as a photoresistance, diode lasers, humidity and temperature sensors, decorative coatings and solar control coatings, among others application. For these reasons, many research groups have an increasing interest in the development and study of this material. Preparation of semiconductor thin films are made by two main procedures [2], however lead sulfide (PbS) detectors usually take one of two forms, both of which used chemically or evaporated deposited thin layers of the sensitive material [3]. The PbS films, which have been evaporated in vacuum, seem always to be n-type semiconductors and can be reversibly changed from n-type to p-type by more exposure to oxygen at room temperature or by more exposure to vapor of sulfur S. The maximum photosensitivity occurred when the resistance is greatest [4]. Lead sulfide is a polar semiconductor which has

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a nature of chemical bending of mixed ionic-covalent bonds where in a purely ionic compound the lead ion Pb^{+2} has three vacant p states, while the S^{-2} ion has one pair of electron in (s-state) and three pairs of electrons in the p-state, each sulfur ion is surrounded octahedrally by six lead ions and similarly, a lead ion is surrounded by six sulfur ions [5].

Lead sulfide occurs naturally as the mineral galena, in which form it is a grey metallic-looking opaque crystal usually having electrical properties lying somewhere between those of a metal and of a semiconductor with small energy gap (E_g) of 0.41eV[6]. Lead sulfide has many special characteristics in comparison with other semiconductors [7]:

- The band gaps are smaller at lower temperatures, i.e, the temperature coefficients of E_g are positive while they are negative for all other elemental or compound semiconductors.
- The lattice structure may be somewhat unstoichiometric. The vacancies and interstitial control the conductivity type, an excess of Pb causes n-type conductivity and excess of chalcogene causes p-type conductivity.
- The band gap of PbSe is smaller than the band gap of the PbTe and PbTe is smaller than PbS, although commonly the band gap in the metal chalcogenide semiconductors diminishes as the atomic number of the chalcogenide increases.
- The static dielectric constant of lead chalcogenide is much larger than those of the other semiconductors.

The optical properties of a semiconductor are related to intrinsic details of the electronic energy band structure. Based on the intrinsic location of the top of the valence band (VB) and bottom of the conduction band (CB) in the band structure, the electron –hole pair generation occurs directly or indirectly [8].

When a semiconductor absorbs light below a threshold wavelength (λ_c) i.e[$h\nu \geq E_g$] or:

$$\lambda (\mu m) = hc / E_g = 1.24 / E_g \quad (1)$$

where λ is the wavelength, h is Planck's constant and c is the speed of light in vacuum, the transitions between the edges of the two bands (VB and CB) occur and these transitions are described by Tauc formula [8]:

$$\alpha h\nu = B (h\nu - E_g)^r \quad (2)$$

where B is inversely proportional to amorphousity, α is the absorption coefficient, $h\nu$ is the incident photon energy, and r is constant and may take values (1, 2/3, 1/2, 3/2) depending on the material and the type of the optical transitions whether it is direct or indirect. There are two types of transitions which are:

- The direct transition occurs between the top of VB and bottom of CB at the same wave vector [9] ($\Delta K=0$). The allowed direct transition refers to that transition which occur between top of valence band and the bottom of the conduction band when the change in the wave vector is zero ($\Delta K=0$) and $r=1/2$. In some semiconductors materials when the quantum selection rules does not allow the direct transition between the extreme of the VB and the minimum of CB but

allowable at different positions of VB and CB. (since $\Delta K = 0$), this transition is called forbidden direct transition[10]and $r=3/2$.

- The indirect transition occurs when the valence band maximum and the conduction band minimum lie at different wave vector [9-11] .Therefore these transitions are limited by the low of momentum conservation in crystalline semiconductors while it is not limited to an amorphous semiconductor [11].

Indirect transition requires absorption or emission of phonons to balance the crystal momentum. In this case the electron cannot make a direct (perpendicular) transition from the valence band to the conduction band, because of the change in their crystal momentum, such transition may take place by two step process: the electron either absorbs both a photon and a phonon or absorbs a photon and emits a phonon simultaneously. The photon supplies the needed energy while the phonon supplies the required momentum conservation [9]. This transition is described by the equation:

$$\alpha h\nu = B^* (h\nu - E_g \pm h\nu_{ph})^r \quad (3)$$

where $h\nu_{ph}$ is phonon energy, B^* is a constant, r may take different value e.g. $r=2$ or 3 for allowed and forbidden indirect transition respectively[12].

The optical constants are very important because they describe the optical behavior of the materials. The absorption coefficient (α) of the material is very strong function of photon energy and band gap energy. The absorption coefficient represents attenuation occurs in incident photon energy on the material for unit thickness, and the main reason for this attenuation is attributed to the absorption processes. The Lambert relation between the absorption coefficient and the incident power intensity on thin films with thickness ($t-t_0$)[10,12] is:

$$I_{(t)} = I_{(t_0)} \exp[-\alpha(t-t_0)] \quad (4)$$

Where $I_{(t)}$ is incident photon energy at thickness (t) inside the material, and $I_{(t_0)}$ is incident photon energy at the surface of the material, the negative signal refer to the decreases in photon energy.

The absorption coefficient is a function of the wavelength of incident radiation and it is very important because it gives absorption range to the radiation. The following relation is referred to the absorption [12]:

$$A+R+T=1 \quad (5)$$

The refraction (R) can be expressed by the relation [12]:

$$R = \frac{[(n-1)^2 + k^2]}{[(n+1)^2 + k^2]} \quad (6)$$

where n is the refractive index^[10] and it can be calculated from the equation:

$$n = \left(\frac{4R}{(R-1)^2} - k^2 \right)^{1/2} - \frac{(R+1)}{(R-1)} \quad (7)$$

and k represents the extinction coefficient (imaginary part of refractive index) which is then, calculated by the relation [12]:

$$k = \alpha \lambda / 4\pi \quad (8)$$

the real and imaginary parts of the dielectric constant (ϵ_1 and ϵ_2) can be calculated by using equations [10,12]:

$$\epsilon = \epsilon_1 - i\epsilon_2 \quad (9)$$

and from the relation [$n = (\epsilon)^{1/2}$], we get

$$(n - ik)^2 = \epsilon_1 - i\epsilon_2 \quad (10)$$

so that

$$\epsilon_1 = n^2 - k^2 \text{ (real part)} \quad (11)$$

$$\epsilon_2 = 2nk \text{ (imaginary part)} \quad (12)$$

Monakhov et al (1986) [13] have determined the density of states in allowed band InSb and PbS. For PbS the CB and VB are similar and the effective masses are similar ($m_e = 0.088m_o$, $m_h = 0.084m_o$), $E_g = 0.41\text{eV}$, $\epsilon_o = 175$. Therefore they found the density of states effective masses for electron and hole are $m_{de} = (0.091 \pm 0.015) m_o$, $m_{dh} = (0.089 \pm 0.015) m_o$ respectively and that the density of states does not conflict with the published data. George et al (1987) [14] have studied the electrical and optical properties of amorphous of PbS films prepared by reactive evaporation. The optical measurements indicate a band gap of 1.66eV at RT and the absorption coefficient $1 \times 10^5 \text{cm}^{-1}$ and $\epsilon_o = 174.4$. For the electrical properties the resistivity at RT is about $1.7 \times 10^2 (\Omega \cdot \text{cm})$. Mohamed (2002) [15] has studied the optical properties of $\text{Pb}_x\text{S}_{1-x}$ thin films and showed that $\text{Pb}_x\text{S}_{1-x}$ films have direct and indirect energy gap. It is found that the energy gap increases when the annealing temperature is increased within the range (RT-673) K while it decreases when the amount of Pb concentration is increased. The refractive index, extinction coefficient and the dielectric constant were also studied. The width of the tails of localized states (E_u) was observed to decrease with increasing annealing temperature but increase with x . It is found that the values of n is within the range (4.083-3.899 at $x = 0.50$ and (4.214-3.985) at $x = 0.53$, with $T_a =$ (RT, 373, 473, 573 and 673) K. Al- Miyali [16] has also studied the optical properties of PbS thin films on Si substrate. He found that the value of energy gap for direct transition was 0.45 eV at RT and for indirect transition was 0.38eV. The optical properties of these films are very important for designing the energy band diagram of PbS heterojunction detectors and have a high demand for many applications in various industrial fields especially in photonic technology.

The aim of this work, thus, is to prepare Pb_xS_{1-x} as a bulk and films by flash thermal evaporation technique and study the effect of x concentration and annealing temperature on the optical properties of Pb_xS_{1-x} films.

2. Experimental Procedure

The Pb_xS_{1-x} alloys at different Pb concentration ($x=0.50, 0.51, 0.52, 0.54$ and 0.55) have been prepared. The Pb_xS_{1-x} films have been prepared by vacuum flash evaporation technique using Edward E306A under lower pressure of about 10^{-6} mbar with $1.5\mu\text{m}$ thickness. By the flash evaporation techniques the weights of the required amount of a material placed into molybdenum boat. To have high sensitive layers, various heat treatments have been used in vacuum by electric furnace in the range (RT, 373, 423, 473 and 523)K. Then the samples are ready for testing using the FTIR Spectrophotometer to carry out the transmittance (T) and absorbance (A) spectra in the wavelength (λ) region (1-5) μm for Pb_xS_{1-x} films deposited on Si substrate at different values of Pb concentration and annealing temperatures (T_a).

The values of λ , T and A are placed in a computer program to deduce the optical energy gap, the fundamental optical edge and other optical constants such as α , k , n , ϵ_1 and ϵ_2 which can be calculated from equations (4),(8), (7),(11) and (12) respectively.

3. Results and Discussion

The optical properties of Pb_xS_{1-x} films of thickness ($1.5\mu\text{m}$) deposited on silicon substrates with different x concentration (0.50, 0.51, 0.52, 0.54 and 0.55) and annealing temperature ($T_a=RT, 373, 423, 473$ and 523)K have been determined from FT-IR transmittance spectra in the region (1-5) μm . The absorption, transmission, and reflection spectra have been studied as well as the energy gap and the optical constants have been determined. In general, we can observe that increasing T_a shifts the peak of transmittance spectrum to a shorter wavelength compared to the un annealed films for all annealed films. The shift in the film's peak position may be attributed to the crystallite of the film structure by increasing the grain size and this is in agreement with our results of XRD study [17].

Also it can be observed from fig.(1) that the transmittance decreases with increasing Pb content except at $x=0.54$ value which has increased and shifted to a higher wavelength and then decreased for $x=0.55$. This may be attributed to create levels at the energy band by adding more Pb content which leads to smaller energies shift. This also indicates that the alloying leads to increase new states in the energy gap similar to impurity states and this can be visualized as an increase in the width of valence or conduction tails leading to a shift of the Fermi level towards the valence or conduction bands [18]. The absorbance and reflectance spectra are presented as shown in figures (2) and (3) respectively. It is obvious that the behavior is opposite to the transmittance spectra.

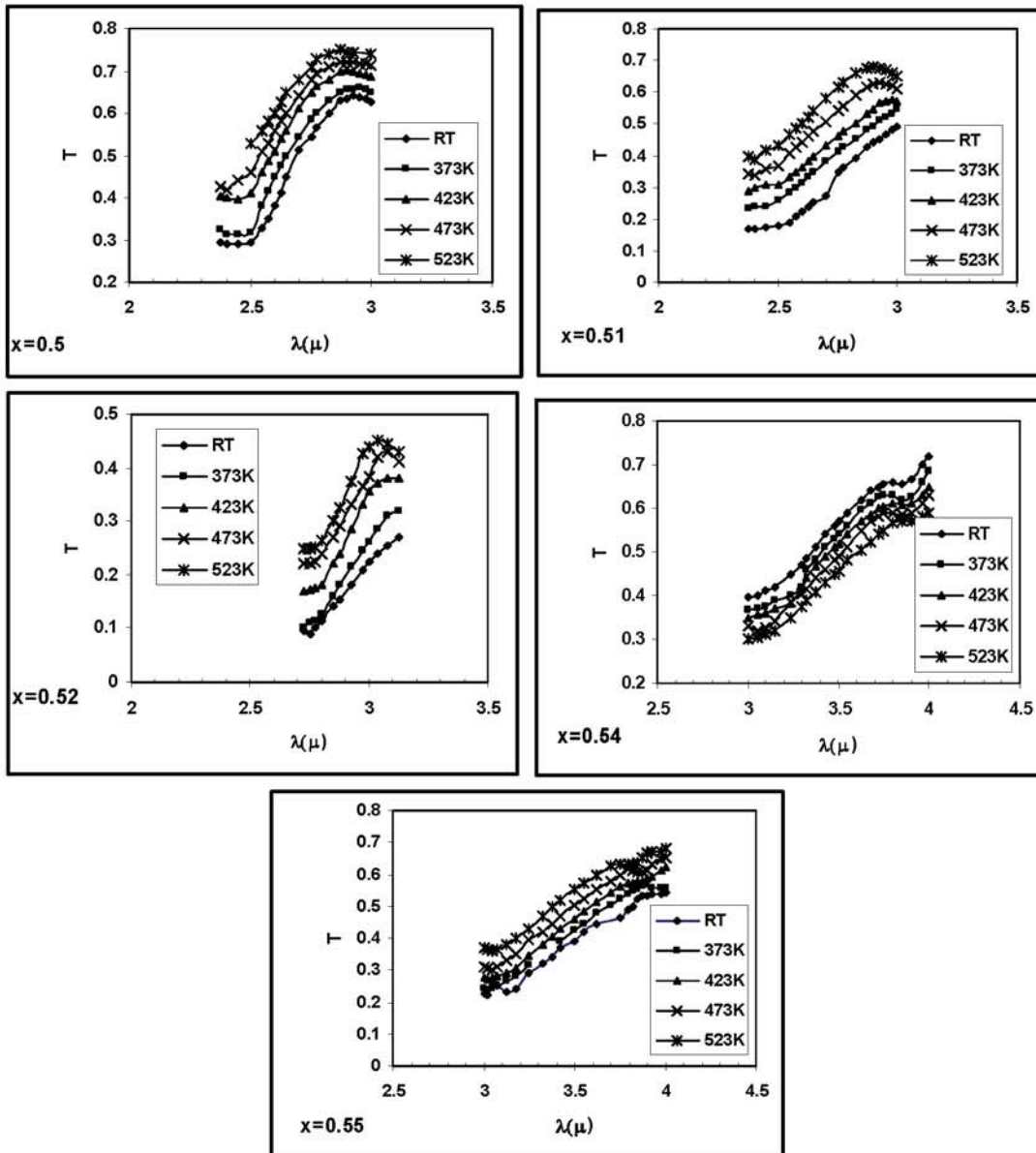


Fig. 1: The transmittance spectra of Pb_xS_{1-x} films with different x concentration and T_a .

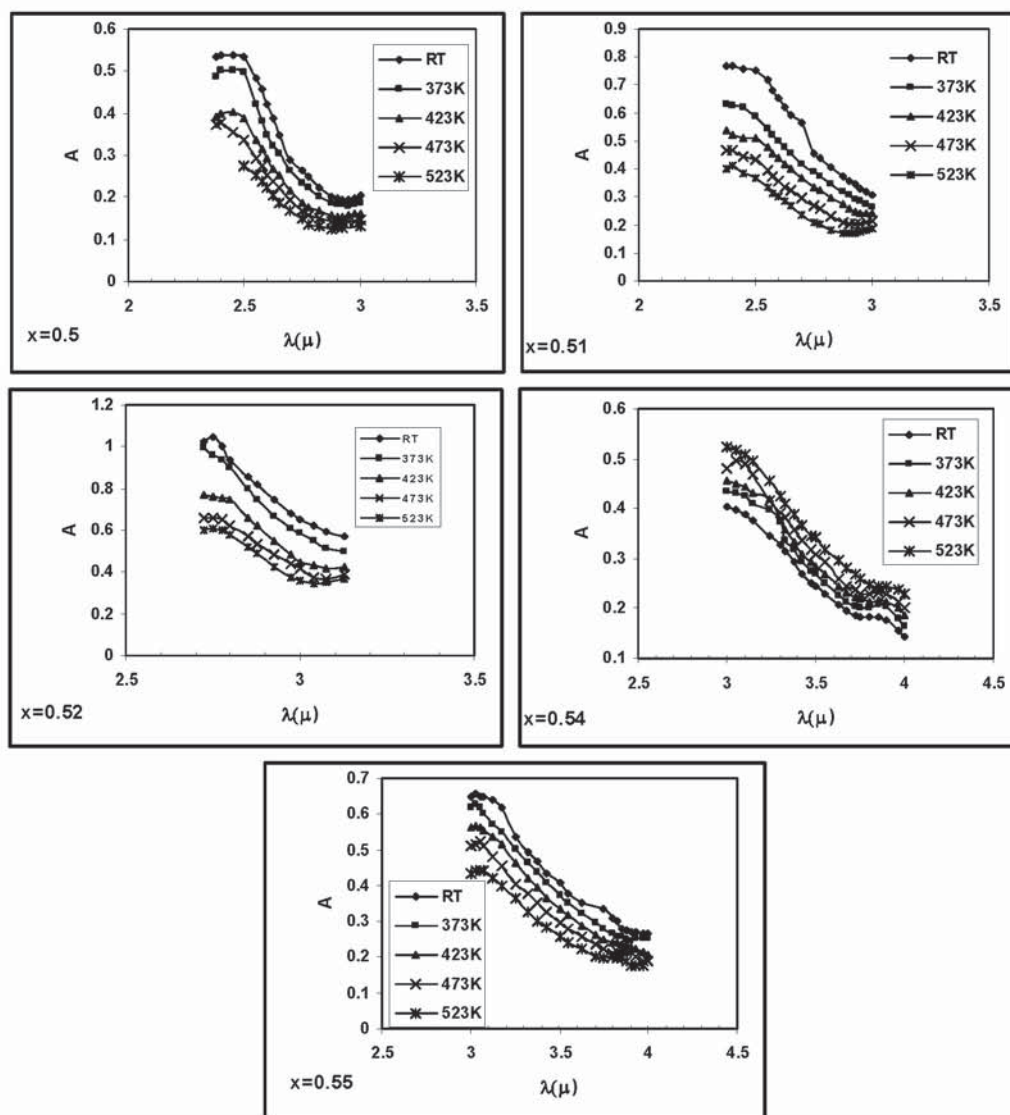


Fig. 2: The absorbance spectra of PbS_{1-x} films with different x content and T_a .

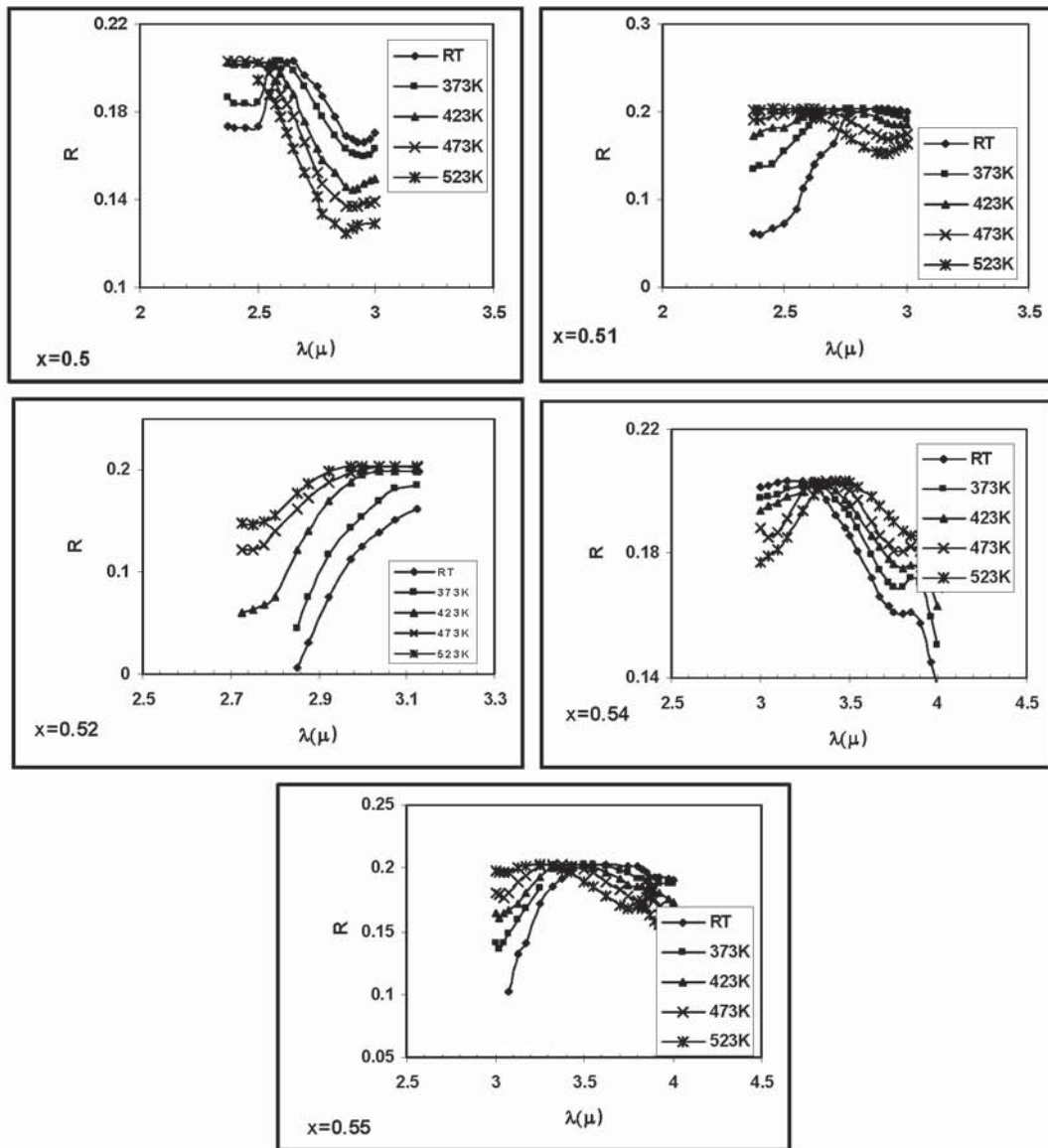


Fig.3: The reflectance spectra of Pb_xS_{1-x} films with different x content and T_a .

3.1 The Absorption Coefficient

The behaviour of α with wavelength is similar to that of absorptance spectra (see Fig.(2)). Table (1) shows the absorption coefficient of Pb_xS_{1-x} films of thickness $1.5\mu m$ with different x concentration (0.50, 0.51, 0.52, 0.54, and 0.55) and annealing temperatures

(RT, 373, 423, 473, and 523)K. The values of α at wavelength corresponding to E_g decreases with increasing annealing temperatures for all samples as shown in Table(1) and this is due to increasing the value of energy gap with annealing temperatures. Our value of α which varies between 0.30×10^4 and $0.99 \times 10^4 \text{ cm}^{-1}$ for x content (0.50-0.55) are in agreement with other researchers [14,15,19] who found that the value of α equals to $4.29 \times 10^4 \text{ cm}^{-1}$, $1 \times 10^4 \text{ cm}^{-1}$ and $8.6 \times 10^3 \text{ cm}^{-1}$ respectively. The difference in the value of α is due to the preparation methods. It can also be seen from Table (1) that the value of α increases three times with increasing x content for all samples except at $x=0.54$ (and this is attributed to the large shifting in the absorption edge from 2-3 μm to 3-4 μm). This is related to the increase in the metal concentration of Pb that causes a change in the type of the charge from p-type to n-type (as it is seen from the electrical properties) [20]. This non stoichiometric in the films leads to increase the dangling bonds or the density of localized states which increases the absorption coefficient. This is also in agreement with Mohammed [15] who found that α decreased with increasing annealing temperature and increased with increasing Pb concentration.

3.2 The Optical Energy Gap

To determine the type of optical transitions we have examined $(\alpha h\nu)^{1/2}$, $(\alpha h\nu)^{1/3}$, $(\alpha h\nu)^{3/2}$, $(\alpha h\nu)^{2/3}$, $(\alpha h\nu)^2$, versus $h\nu$ and found that the last relation yielded a linear dependence which describes allowed direct transitions. From fig.(4) the energy gap is determined by plotting the Tauc equation (2) and taking the extrapolation of the linear portion where $(\alpha \geq 10^4 \text{ cm}^{-1})$ of the function $(\alpha h\nu)^2$ vs. $h\nu$ curve to $\alpha=0$. The direct energy gap value was extrapolated and listed in Table(1), which is in agreement with other literatures [21,22]. The value of the optical energy gap increases with increasing annealing temperature for all samples as shown in Table(1) and this is due to the growth of the grain size and the decrease in the defect states near the bands which in turn increased the value of E_g . The value of E_g is nearly constant at higher Pb content and this is due to eliminate the defects and decreasing the defect states inside E_g . The value of E_g is in a qualitative agreement with other researchers. Al- Miyali [16] found that the value of E_g was 0.45eV for PbS which prepared by electrical deposition while Schoolar and Dixon [19] found that the value of E_g was equal to 0.4eV for PbS crystals.

The constant (B) (Tauc slope) in the Tauc equation has been obtained from the square root slope of the straight line in the fig. (4). Following the work of Al-Ani [22], the coefficient B in equation(2) may be obtained from the (slope)^r of the linear region of the plot $(\alpha h\nu)^{1/r}$ vs. $h\nu$ and its units can be calculated according to $\text{cm}^{-1} \text{eV}^{1-r}$. Thus, from this figure the value of B is calculated to be of the order of $10^8 \text{ cm}^{-1} \text{eV}^{1/2}$ (Table1) and decreased with increasing T_a . But it is constant for Pb concentration ($x=0.50$ and 0.51) then increased for other samples except at $x=0.54$ value. We know that B is inversely proportional to amorphous nature [21]. The E_g decreases with increasing x concentration as shown in Table (1). This is due to increase the density of localized states in the E_g which cause a shift in E_g to lower energy gap with increasing x content while E_g increases slightly with increasing T_a for all x concentration except at $x=0.54$ value.

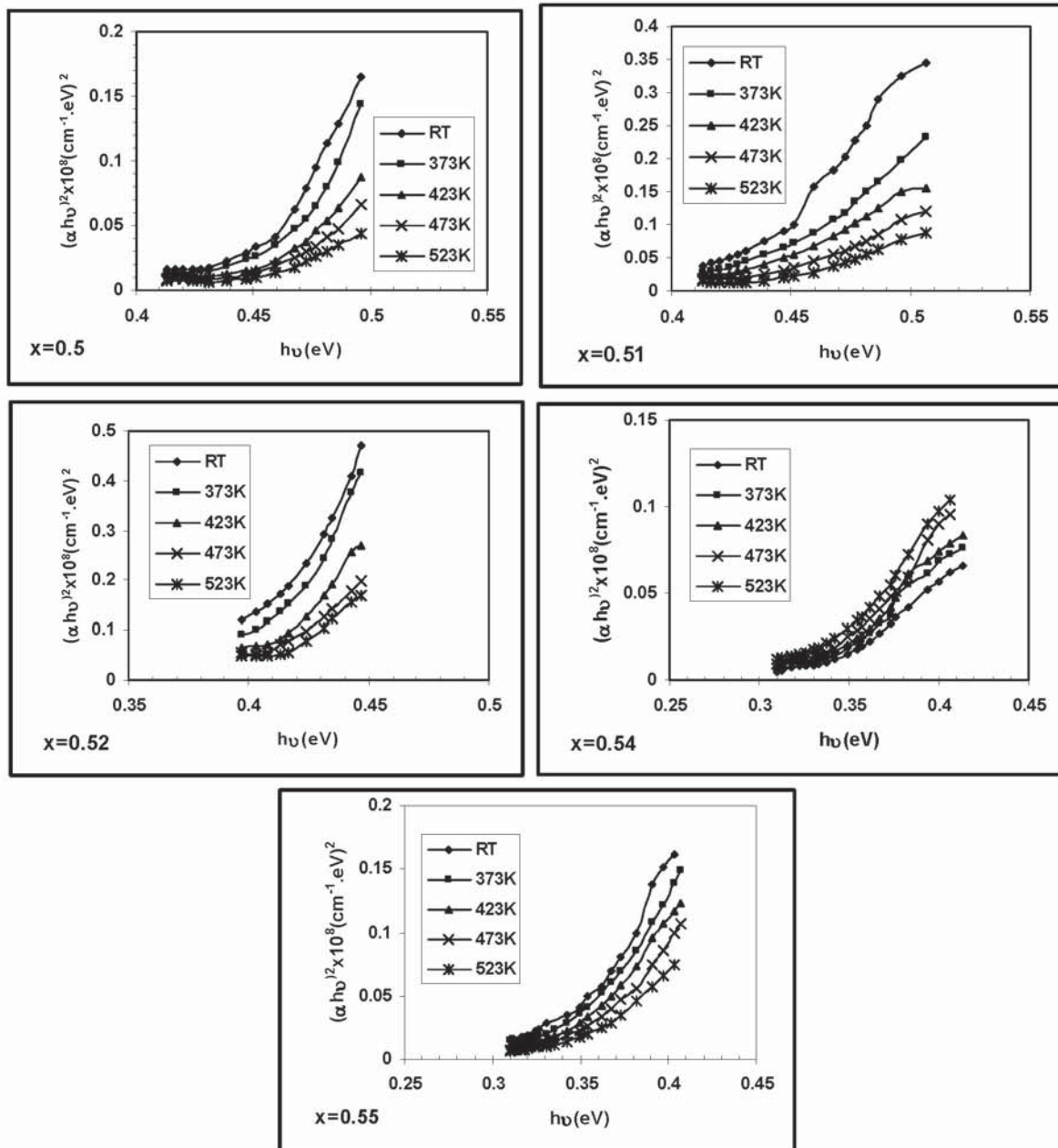


Fig. 4.: The function $(\alpha h\nu)^2$ vs. $h\nu$ for $\text{Pb}_x\text{S}_{1-x}$ films with different x content and T_a .

3.3 The Urbach Energy

The density of localized states in the band gap can be evaluated from the Urbach[23,24] energy (E_u) at $\alpha < 10^4 \text{cm}^{-1}$ which is referred to absorption tails at energies smaller than the optical energy gap using the relation:

$$\alpha(\nu) = \alpha_0 \exp(h\nu/E_u) \quad (13)$$

by plotting $\ln \alpha$ as a function of $h\nu$, the reciprocal of the slope of the linear part give the value of E_u . We can see from Table (1) that E_u decreases from (0.0621, 0.105, 0.079, 0.094, and 0.099)eV to (0.059, 0.076, 0.057, 0.075 and 0.083)eV for 0.50, 0.51, 0.52, 0.54 and 0.55 respectively with increasing annealing temperature (RT, 373, 423, 473 and 523)K for all samples. This may be attributed to the improvement in the structure by annealing and decreasing the amorphous degree of the films leading to a decrease in the localized states. It is found that the value of E_u increases with increasing Pb concentration except at $x = 0.52$ value and this increase is due to the creation of localized states in the E_g gives the value of E_u . It can be seen from Table (1) that E_u decreases from (0.0621, 0.105, 0.079, 0.094, and 0.099)eV to (0.059, 0.076, 0.057, 0.075 and 0.083)eV for $x = 0.50, 0.51, 0.52, 0.54$ and 0.55 respectively with increasing annealing temperature (RT, 373, 423, 473 and 523)K for all samples. This may be attributed to improvement in the structure by annealing and decreasing the amorphous degree of the films leading to decrease the localized states in agreement with the increase of E_g vs T_a . It is found that the value of E_u increases with increasing Pb concentration except at $x = 0.52$ value and this increasing is due to create localized states in the energy gap E_g .

3.4 The Refractive Index

The optical behavior of materials is generally utilized to determine its optical constants such as the refractive index. Table (1) shows the variation of the refractive index at different T_a and x concentration at wavelength corresponding to E_g . It is interesting to see that n decreases slightly with increasing annealing temperatures. This behavior is due to increase in the energy gap which causes to expand the lattice and grow the grain size and decreases the defects. That means increasing the absorption and decreasing the reflection which the refractive index depends on it and this is in agreement with early results of Zemel et al [24]. It can also be seen from Table (1) that the value of n increases with increasing x content and this is due to increase in the packing density and decrease in the degree of amorphousity. The increase in the reflectivity is due to the fact that the value of n for Pb is greater than that for S and moreover the addition of Pb causes an increase in the density and changes the distribution of dangling bonds and thus leads to a higher refraction and consequently increasing in the refractive index. Our value of n are in agreement with other researchers [25-29] who found that the value of n are equal to 4.8 and 4.22 respectively for PbS films grown by hot wall method. Mohammed [15] found that the value of n for $\text{Pb}_x\text{S}_{1-x}$ films which prepared by vacuum evaporation within the range (4.083-3.89) at $x = 0.50$ and (4.214-3.985) at $x = 0.53$, when annealed at (RT-673) K.

3.5 The Extinction Coefficient

The behavior of extinction coefficient (k) is nearly similar to the corresponding absorption Coefficient at different T_a and x content. From Table(1), it is seen that k decreases with increasing T_a from RT to 523K by two times for all alloys and it is increased with increasing x content. This is attributed to the same reason which mentioned above on the absorption coefficient.

3.6 The Dielectric Constants

Table (1) shows the variation of real dielectric constant (ϵ_1) and imaginary dielectric constant (ϵ_2) with different annealing temperatures and x content. The behavior of ϵ_1 is similar to refractive index because the smaller value of k^2 comparison of n^2 while ϵ_1 is mainly depends on the k values which are related to the variation of absorption coefficient. It is found that ϵ_1 and ϵ_2 decrease with increasing T_a and this agree with Henry [4] results, whereas it increases with increasing Pb content. Also Mohammed[15] found that ϵ_1 equal to 17.4 at 300K and decreases by 1.05 with the temperature increasing to 373K while the value of ϵ_2 equal to 1.690 and this decreases by 1.096 when T_a is increased to 373K.

Table (1): The optical constants and related parameters of Pb_xS_{1-x} films.

X	0.50							
T_a	$E_g(eV)$	$BX10^9$ ($cm^{-1}eV^{1/2}$)	N	k	ϵ_1	ϵ_2	α (cm^{-1}) $\times 10^4$	E_u eV
RT	0.450	0.70	4.080	0.070	16.641	0.571	0.30	0.0621
373	0.460	0.63	4.050	0.068	16.397	0.550	0.29	0.062
423	0.467	0.54	4.030	0.059	16.237	0.475	0.25	0.060
473	0.468	0.44	4.029	0.053	16.230	0.427	0.22	0.060
523	0.469	0.31	4.026	0.046	16.206	0.370	0.20	0.059
				0.51				
RT	0.430	0.70	4.15	0.110	17.210	0.913	0.47	0.105
373	0.433	0.54	4.14	0.086	17.132	0.712	0.41	0.102
423	0.410	0.44	4.22	0.088	17.800	0.742	0.37	0.102
473	0.425	0.44	4.17	0.078	17.382	0.650	0.33	0.090
523	0.448	0.31	4.09	0.065	16.723	0.531	0.27	0.076
				0.52				
RT	0.38	0.83	4.336	0.210	18.756	1.821	0.87	0.079
373	0.39	0.77	4.290	0.187	18.369	1.604	0.75	0.067
423	0.39	0.70	4.290	0.160	18.378	1.372	0.65	0.062
473	0.40	0.63	4.250	0.150	18.062	1.127	0.59	0.06
523	0.41	0.54	4.220	0.130	17.808	1.109	0.54	0.057
				0.54				
RT	0.33	0.31	4.549	0.192	20.656	1.746	0.80	0.094
373	0.32	0.31	4.596	0.227	21.071	2.086	0.77	0.086
423	0.33	0.31	4.549	0.167	20.665	1.519	0.70	0.084
473	0.34	0.31	4.510	0.159	20.314	1.434	0.66	0.078
523	0.33	0.28	4.549	0.148	20.671	1.346	0.60	0.075
				0.55				

RT	0.330	0.447	4.549	0.230	20.640	2.092	0.99	0.099
373	0.344	0.447	4.490	0.227	20.108	2.038	0.95	0.091
423	0.340	0.441	4.510	0.206	20.297	1.858	0.86	0.085
473	0.345	0.428	4.480	0.187	20.035	1.675	0.78	0.084
523	0.340	0.356	4.510	0.150	20.317	1.353	0.66	0.083

4. Conclusion

The optical properties of these films are very important for fabrication photoconductive and photovoltaic detectors and designing the model of energy band diagram [27,28].

From the data of the present work it can concluded that:

- The Pb_xS_{1-x} alloys for ($x = 0.50, 0.51, 0.52, 0.54$ and 0.55) have successfully prepared, which were bulks and films prepared at different conditions.
- The increasing in T_a shifts the peak of transmittance spectrum to shorter wavelength compared to the un-annealed films for all alloys. Also it is found that the transmittance is decreasing with increasing Pb concentration except at 0.54 value which increases and shifts to a higher wavelength.
- The absorption coefficient decreases to half, and one and half times with increasing annealing temperature for all samples and it increases three times with increasing x concentration for all samples.
- The value of the optical energy gap is due to direct transitions. It is also found that the value of E_g increases with the increasing T_a but decreases with increasing Pb concentration.
- The width of the tails of localized states in the gap was found to decrease with increasing T_a while it increases with increasing Pb concentration in a qualitative change of E_g .
- The refractive index, extinction coefficient, and dielectric constant were decreasing with increasing T_a but they increase with increasing the amount of x .

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