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Effect of temperature on optical properties of $CuInS_2$ thin films

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Abstract-Polycrystalline $CuInS_2$ thin films were grown by Spray pyrolysis method. This films have been deposited onto the glass substrate by varying substrate temperature from 275^0C, at the interval of 25 to 350^0C by using aqueous solution of Cupric chloride, Indium tri-chloride and thiourea of 0.02 M and Seems to be more important parameters affecting physical properties of the semiconductor. On account for the temperature dependent optical properties of polycrystalline $CuInS_2$ thin films, grain boundary states were operative in nature. The values of absorption coefficient, extinction coefficient, refractive index, and dielectric constant have been calculated from the optical measurements. The prepared films at different substrate temperatures were found to have high transmittance (76 %) with the lowest thickness approximately t = 0.126 μm and at low transmittance (50 %) of the films with highest thickness nearly equal to t = 0.2163 μm. Due to temperature variation, the atoms are arrange regular in manner compound of $CuInS_2$ films are investigates. As the films are not doped intentionally, defect observed in intrinsic nature by Sulphur interstitials. The most significant results in present study, the temperature varied with thickness of the film can be used to modify the optical band gap, extinction coefficient, refractive index and real-imaginary part of dielectric constants of $CuInS_2$ thin films.

Keywords- Spray pyrolysis, $CuInS_2$ Ternary Semiconductor, Optical Constants

.I. INTRODUCTION

In recent years, the development of nano-structured I-III-VI_2 ternary compound semiconducting materials in the form of thin films occupy a prominent place in basic research and solid state technology due to their expanding range of potential applications [1] in the diverse field such as photovoltaic cells, electronic components, fabrication of large area photodiode arrays, photoconductors, sensors, antireflection coatings, optical filters, surface acoustic wave devices, solar selective coatings and solar cells etc [2-4]. The conversion efficiency of this area devices based on other chalcopyrites like $CuInTe_2$ [5] $CuInS_2$ [6-8] is almost found higher values. $CuInS_2$ is one of the I-III-VI_2 chalcopyrite-type mixed crystal semiconductor [2]. So many researcher [8] were prepared $CuInS_2$ thin films by spray pyrolysis method and studied some physical properties. $CuInS_2$ as an absorber layer is very promising for thin film photovoltaic applications [4], which matches well with the solar spectrum, its high absorption coefficient of approximately 10^4 cm^{-1} and its direct band gap energy obtained from 1.32 – 2.45 eV [3-5] at near the red edge of the visible spectrum and high absorption coefficient [5-6] and its controllable conduction type. $CuInS_2$ is less toxic than selenide based compounds like $CuInS_{2x}Se_{2(1-x)}$ $CdZnSe_2$ and $CuInTe_{2(1-x)}Se_{2x}$. As the temperature of the glass substrate increases, band gap (Eg_1 and Eg_2) values, extinction coefficient (k), imaginary part of dielectric constant (ε_1) increased whereas real part of dielectric constant (ε_2), refractive index (n) decreases with wavelength (λ) from 350 – 1100 nm respectively. Refractive index and thickness of $CuInS_2$ and CdS films was obtained using an Ellipsometer by other researcher [9]. Different method have been used to produce $CuInS_2$ thin films, with including electrodeposition [10], evaporation [4, 6 and 11-12], vapor deposition [2] spray pyrolysis [9] and Sputtering technique [13]. In sulfurization process, an elemental sulfur vapor avoid highly toxic H_2S [14], developed Cu-rich $CuInS_2$ films showed an energy band gap of 1.38 eV at absorption coefficient of about 10^5 cm^{-1} [9] [12].

II. RELATED WORK

In this paper we focused on the effects of temperature on preparing thin films at different temperatures in air atmosphere and studied optical properties of $CuInS_2$ films by spray pyrolysis method. This method is one of the best technique to producing polycrystalline films from all chemical compound of solid solution which are form aqueous solution and this system not only inexpensive, easy deposition in moderate temperature but also developed films in large area. UV- 1800 Shimadzu spectrophotometer used to find optical absorption edge from the evaluated spectra from transmittance curve by varied substrate temperature for the preparation of films. We investigates, temperature dependence of optical properties and other related optical constants characterization of $CuInS_2$ thin films by varied temperatures (250 to 375^0C at the interval of temperature 25^0C).

III. EXPERIMENTAL DETAILS

The deposition was carried out onto commercially available glass substrates of the size (7.5 ×2.5×0.1) cm^3. The analytical reagent grade chemicals used were Cupric chloride ($CuCl_2$), Indium tri-chloride ($InCl_3$) and thiourea $(NH_2)_2CS$) were prepared of 0.02 M. Solution of $CuCl_2$, $InCl_3$ and $(NH_2)_2CS$ in appropriate volumes in order to attain Cu:In:Te in the ratio 1:1:3.2. Stoichiometric amounts of the elements of 98-99% purity $CuCl_2$, $InCl_3$, and $(NH_2)_2CS)S$ were used to prepare the initial chemical compound in aqueous solution mixed for the fomration of $CuInS_2$ thin films. Excess sulphur required to reduce the deficiency of sulphur [15-17]. If ratio of Cu:In:Te used in the ratio 1:1:2, shows the tellurium deficiency [18]. The aqueous solution was then sprayed as a fine mist containing the reactant molecules on the preheated glass substrate of kept at various temperature. By Bernoulli's action solution spray will take place through this nozzle of glass sprayer when air flows through the carrier gas nozzle and a fine mist is produced at the barrier which is allowed to deposit on the pre heated glass substrate [19]. In order to find optimized condition for deposition of $CuInS_2$ thin films, the depositions were carried out by varying one of the parameters as substrate temperature and keeping the others at fixed value. The sprayed droplets on reaching the hot substrate undergo pyrolytic decomposition and form a single crystal, cluster, or crystallites of the product [20]. Films of thicknesses ranging from 0.14-0.22 µm were deposited at various temperature for optical measurement at a room temperature. The films thickness was measured by Michelson-interferometer. Samples of various thicknesses (0.14-0.22 µm) were obtained. Electrical resistivity calculated by Four-probe technique. The experimental set-up we used for our sprayed process is diagrammed in Figure 1, and has been details described in references [16-17 and 20].

IV. OPTICAL PROPERTIES

Transmittance (T) and Reflectance (R) spectra of the $CuInS_2$ films was recorded at room temperature using UV-1800 Shimadzu Spectrophotometer for the wavelength range of 350 to 1100 nm. The absorption coefficient (α), Energy band gap (Eg) refractive index (n), extinction coefficient (k) and Dielectric constant value in Real and Imaginary Parts (ε_1 and ε_2) were obtained by utilizing these spectrum [22]. In our experiment we used this spectrophotometer for measuring the transmittance and reflectance spectra of the $Ge_{30}Se_{70}$ films [23] also deposited on glass substrate at various temperatures. The high transmittance is probably due to the existence of an interfacial layer with low refractive index between $CuInS_2$ solid material on films and by glass. The absorption coefficient of all of solution sprayed films is close to 10^4 cm^{-1}. This change in absorption edge can be related to the film thickness and temperature of the substrate, this behavior indicates an increase in the disorder with decreasing thickness and may be attributed to the introduction of some defects which create localized states in the band-gap and therefore increase the band-gap. Similar results were also observed by other researchers [5, 24]. It is also known that the thickness becomes important in the thin films. If the semiconductor thickness is larger than the absorption length, most light will be absorbed; and if the diffusion length is larger than the film thickness, most photo generated carriers can be collected. Their efficiencies have reached higher than ~15% [11, 25]. The dominant feature of the energy dependence of the absorption coefficient (α) is the onsets of absorption near the region of inter-band transition from valence to conduction bands. Figure (2) shows the variation of transmittance versus wavelength plots of the $CuInS_2$ thin films deposited using different chemical compound for temperature 250 to 375^0C at the interval of 25^0C (spectra of reflectance not taken here). As expected transmittance of spray deposited films increases with changing value of 'Temperature' in above mention interval. It was observed that transmittance (above 70 %) was constant for higher wavelength and then suddenly decreased [9]. The onset of decrease of transmittance depends upon the concentration and represents the fundamental absorption edge [15, 16, 22 and 27]**,** whereas thickness values increases upto optimum temperature. In the absorption measurements, we are concerned with the light intensity 'I' after traversal of a thickness (t) of material as compared with the incident intensity 'I_0', thereby defining the absorption coefficient (α),

$\alpha = 1/t \log (I_0/I)$ ……. (1)

The absorption coefficient (α) which is the decrement ratio of incident radiation relative to unit length in the direction of wave propagation inside the medium is relating with the absorbance (A).

It can be seen from Fig.2 that the absorption coefficient decreases with wavelength (increasing the film thickness) exponentially, then becomes constant and reaches the value 10^4 cm^{-1} in the visible region. This result is very important because we know that the spectral dependence of the absorption coefficient affects the solar conversion efficiency [28]. It is observed from this graph that absorption coefficient decreases with varies the temperature and it is also found that the film thickness increase [29]. The absorption coefficient (α), is related to the extinction coefficient (k), by the following formula,

$\alpha = 4\pi k/\lambda$ ….. (2)

Where, λ is the wavelength.

In the present works absorption coefficient (α), calculates optical band gap values and with help of many researchers [5, 28 and 30] put the empirical equation (Tauc relation) between the optical energy gap (Eg) and energy of the incident photon which is,

$$\alpha h\nu = A(h\nu - Eg)^n \qquad \ldots\ldots\ (3)$$

Here, α is the absorption coefficient, h is the plank constant, ν is the frequency of light, A is the characteristics constant as well as a function of density of states near the conduction and valance band edges, Eg is the optical band gap energy (eV), n is the integer and has a values 1/2, 3/2, 2 and 3 depending on the material and the type of the optical transition corresponding to the allowed direct, allowed indirect, forbidden direct and forbidden indirect transition respectively [5,30 and 31].

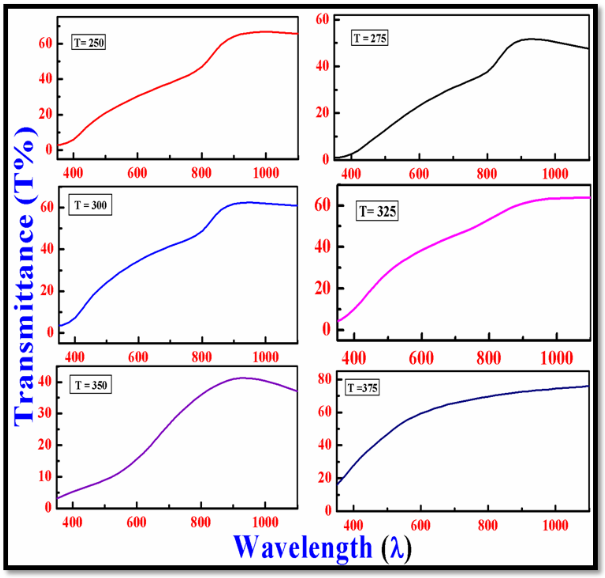

Fig. 2 Variation of Transmittance versus wavelength plots of $CuInS_2$ thin films deposited at 250^0C to 375^0C.

The optical band gap energy values for experimental thin films have been determined by using Tauc equation (3) which is used to find the type of the optical transition by plotting the square of the absorption coefficient (α) versus photon energy (hν) and extrapolating the straight line portion of the graph to the hν axis (fig 3) [33]. This relation (3) is between absorption coefficient and optical band gap energy is expressed to calculate the band gap of the compounds by the following relationship. Since the plots are linear, the direct nature of the optical transition is confirmed [5, 33]**.** It is found that the relation for n =1/2 yields linear dependence.

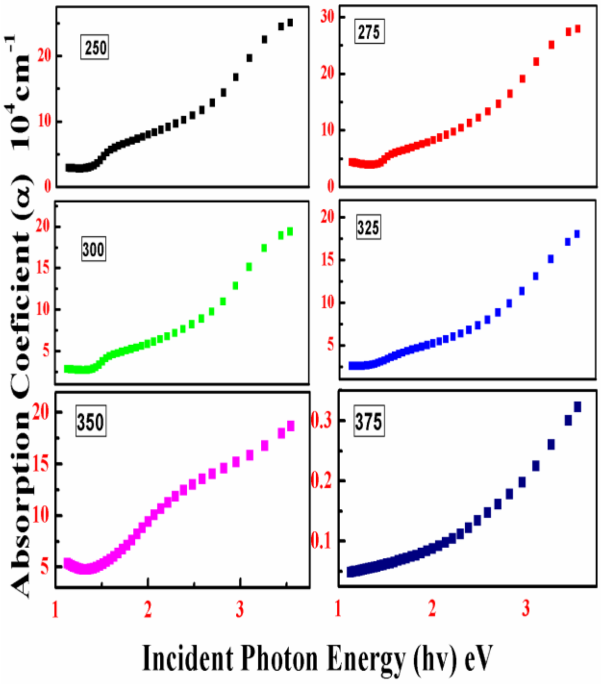

Fig. 3 Absorption coefficient versus incident photon energy hν (eV) for various temperatures (250 to 375^0C)

The nature of optical band gap can be determined with the help of the relation $\alpha h\nu = A\ (h\nu - Eg)^n$,where absorption coefficient, A is a constant and n depends on the nature of the transitions. It is well known that the fundamental absorption for $CuInS_2$ was due to allowed direct transitions. Typical Fig. (4) Illustrates $(\alpha h\upsilon)^2$ as a function of photon energy (hυ) for all thin films of as-deposited and $CuInS_2$ film with temperature 350^0C. The linear portion of each was extrapolated to $(\alpha h\upsilon)^2 = 0$ and meet energy (hν) axis given the values of band gap Eg_1 [34]. The absorption coefficient is calculated by using these values of band gap (Eg_1) and slope A of the linear portion of the curves, for hν values corresponding to the non-linear portion of the curve. It is found that the films with higher thickness and optimum temperature have the smallest band gap $Eg_1 = 1.42$ eV. This can only be explained, if we assume the presence of an additional absorption process. Using this value Eg_1 and the slope A_1 of the curve, α_1 is calculated for hν. It can be seen from fig (6, 7) and tabulated in table. 1, that there is a shift in the band gap energy towards lower energies with the increase in the films thickness [12] and corresponds with temperature.

It was observed that calculated the value of α_1 is always less than observed absorption coefficient α for this range of wavelength. This can be only explained if we assume an additional absorption process [16, 35]. The absorption due to this additional process is denoted by α_2, defined by relation [5],

$$\alpha_2 = \alpha_{exp} - \alpha_{1cal} \qquad (4)$$

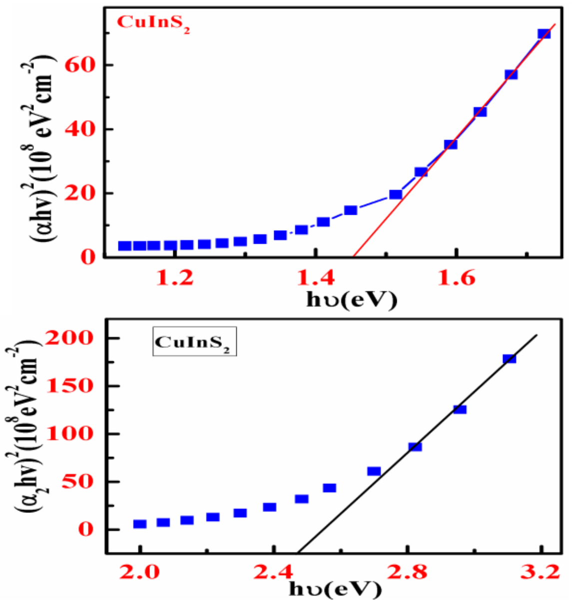

Fig. 4 Variation of $(\alpha h\upsilon)^2$ and $(\alpha_2 h\upsilon)^2$ with incident photon energy (hv) for typical $CuInS_2$ thin films deposited at 350^0C.

Fig. 4 shows the graph $(\alpha_2 h\nu)^2$ versus hν for the composition of typical $CuInS_2$ thin films as deposited at 350^0C temperature. It observed that the graph was linear. When it is extrapolated to $(\alpha_2 h\nu)^2 = 0$, the optical band gap (Eg_2) is found to be 2.43 eV for $CuInS_2$ thin film prepared at 350^0C temperature. This also indicates the presence of second direct allowed transition.

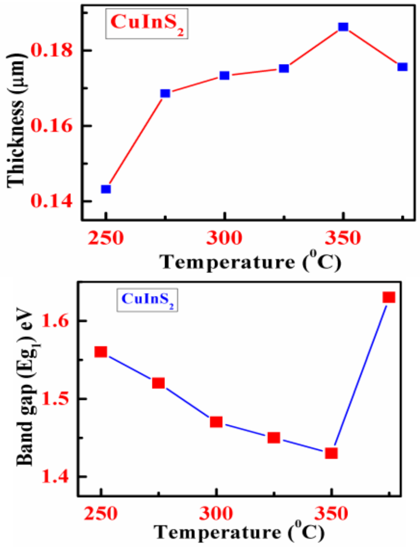

Fig. 5 Variation of thickness and optical band gap energy versus Temperatures

V. OPTICAL CONSTANTS

In general, optical constant [34] of any of the material is that any number of quantities characteristic of the optical behavior of a substance such as the refractive index, absorption coefficient, or reflectivity for a specified wavelength, it is combination referred as called optical constant and have wide applications in designing different optical components and modeling optical coatings [36]. These values are evaluated from the transmittance and / or reflectance spectra.

Optical measurements can also be used to study lattice vibrations (phonons). This can be conveniently taken into account by defining a complex refractive index, (n* = n + ik). Here, the real part denoted by 'n' is the refractive index and it is also indicates the phase velocity of any materials, while the imaginary part k is called the extinction coefficient although k can also referred as mass attenuation coefficient and indicates the amount of attenuation when the electromagnetic wave propagates through the material. The real part of the refractive index n determines the propagation velocity (ν and wavelength (λ) in the medium,

$$n = c / v \qquad \text{.....} \quad (6)$$

The refractive index of materials varies with the wavelength (and frequency) of light. As the refractive index varies with wavelength, so will the refraction angle as light goes from one material to another.

For non-magnetic materials, we can take μ = 1, the real and imaginary part of dielectric constant can be calculated by using the following equations,

$$\varepsilon = \varepsilon_1 + i\varepsilon_2 = (n + ik)^2 \qquad \text{.......} \quad (7)$$

$$(n - ik)^2 = \varepsilon - i\varepsilon \qquad \text{.....} \quad (8)$$

Where, $\varepsilon_1 = n^2 - k^2$ .... (9) And

$\varepsilon_2 = 2nk$ ...... (10)

Where, n is the refractive index and k is the extinction coefficient and where we note that they are all frequency dependent.

The refractive index value can be calculated from the formula,

$$n = \left(\frac{4R}{(R-1)^2} - k^2\right)^{1/2} - \frac{(R+1)}{(R-1)} \qquad \text{.......} \quad (11)$$

$$\text{And, } n = \frac{1+\sqrt{R}}{1-\sqrt{R}} \qquad \text{.......} \ (12)$$

Where, R- is the reflectance, the extinction coefficient, which is related to the exponential decay of the wave as it passes through the medium.

Next, in order to complete the calculation of the optical constants, the extinction coefficient *k* is estimated from the values of absorption coefficient (k) and wavelength (λ),

$$k = \frac{\alpha\lambda}{4\pi} \qquad \text{.......} \ (13)$$

Where λ is the wavelength of the incident radiation and absorption coefficient (α) is calculated by equation (1 and 2).

Plotting the graph in between k and incident photon energy (hv), therefore it is illustrates the dependence of extinction coefficient (k) versus hv for the studied thin film. The film thickness, energy band gap, extinction coefficient dielectric constant and refractive index of the films were calculated by the using the values of transmittance and reflectance data UV spectral graph, these values are listed in table 1.

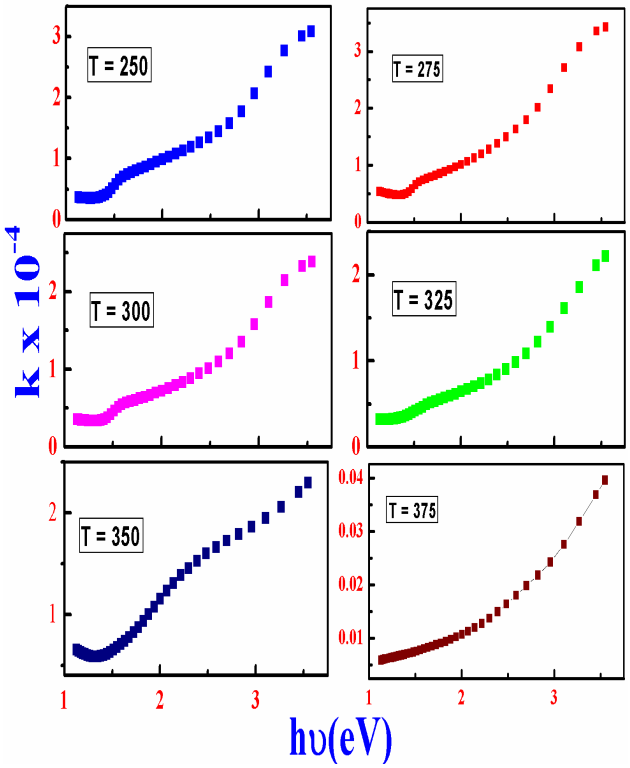

Fig. 6 Extinction coefficient versus incident photon energy (eV)

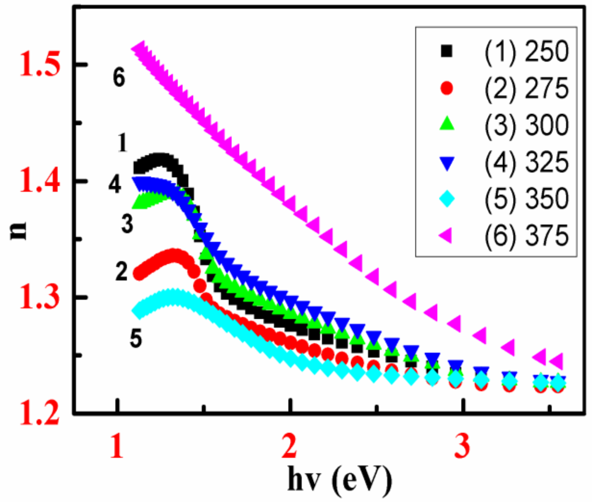

Fig. 7 Refractive index (n) versus incident photon energy (hv) (eV)

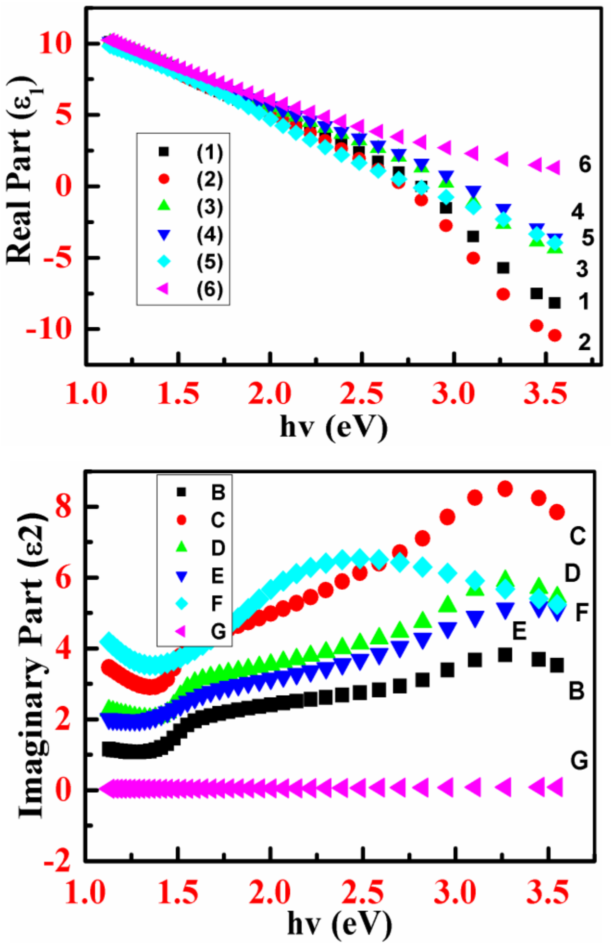

Fig. 8 Real (ε_1) and maginary (ε_2) Part of dielectric constant constant versus incident photon energy (hv) (eV)

Table (1) Tabulated values of Extinction Coefficient (k) and Refractive Index (n) real and imaginary part of Dielectric constant and other parameters with various temperatures of $CuInS_2$ thin films

T (^{0}C)	Thickness	Eg (eV)	Extinction Coefficient (k)	Refractive Index	Dielectric constant	
					ε_1	ε_2
250	0.143	1.56	0.40-1.92	1.41-1.22	10.11-0.99	1.15-3.52
275	0.168	1.52	0.40 -1.94	1.32-1.22	9.95-0.25	7.84-3.46
300	0.173	1.45	0.35-2.381	1.38-1.22	10.11-0.22	5.45-2.25
325	0.175	1.37	0.31-2.21	1.39-1.22	10.14-0.77	5.07-2.01

350	0.186	1.43	0.65- 2.29	1.28- 1.22	9.81- 0.50	5.24- 4.18
375	0.175	1.65	0.005- 0.03	1.51- 1.24	10.24- 1.30	0.03- 0.09

VI. RESULTS AND DISCUSSION

The band gap values calculated from the Tauc plot for $CuInS_2$ thin films deposited at different substrate temperatures is shown in Fig. 3. The thickness was varied from 0.1432 to 0.1862 µm. Many researchers [12, 29 and 37] showed, as the thickness increases with band gap values are also changes and optimize in favorable parameters. The data shows that typical optical energy band gap and thickness values varies for all these thin films decreases with increasing substrate temperature (Fig. 6 and 7), indeed Eg for $CuInS_2$ increases from 1.37 to 1.65 eV and shows direct allowed transition [32]. The extrapolation of the straight line to $(\alpha h\nu)^2 = 0$ axis gives the value of the energy band gap [24, 38]. From figure (3) it is known that optical absorption coefficient and the transmittance of the thin films affects the Eg_1 value, these are 1.56, 1.52, 1.45, 1.37, 1.43 and 1.65 eV for the temperatures 250 to 375^0C respectively. The energy band values of $CuInS_2$ for these samples decrease with increase in substrate temperature upto 350^0C and further increase in substrate temperature [37], the band gap energy at 350^0C of the crystalline material confirms the stability of materials with nearly stoichiometry the variation of band gap energy with substrate temperature is shown in fig. 7. The increasing of optical energy band gap values with can be related with decreasing of defect states [23], no excess Cu_2S phase [31] and enhancement of crystalline structure [35] declared from XRD pattern [17]. On the other hand the increasing of band gap with the increasing of deposition temperature may be ascribed to increasing of transmittance hence which mean that the materials become more transparent (less absorbance). We noted a decrease of band gap value with the annealing temperature due the improvement of the crystallinity of the films.

The band gap for $CuInS_2$ was fairly in good agreement with result obtained produced by spray pyrolytically deposited films; the energy gap was 1.44 eV and 1.38- 1.5 eV respectively [31, 32 and 33]. This lower value of optical band gap may be due to the existence of tail states and traps. This is in agreement with the results published by other authors [39-40]. (Eg_1 = 1.43 – 1.5 eV). The calculation of the optical parameters of the structure of $CuInS_2$ thin films on glass substrate were obtained by mean transmittance values only using a method previously reported by Lontchi et al [4], was noted a decrease of band gap value with the annealing temperature due the improvement of the crystallinity of the films. Values was between 1.3 eV - 1.6 eV around the optimal band gap value for the un-doped and the Na-doped $CuInS_2$. The refractive index values was well agreed [4] values are reported results 2.6 and 2.7 for $CuInS_2$ and CdS films [9]. This low refractive index can be attributed to the singular ear structure, which could grow from first nucleation layer.

From Figure (4), it is observed that graph is a straight line, with extrapolated intercept on hν axis gives the values of band gap. In case of $CuInS_2$ the difference between two direct allowed transitions namely 1.43 and 2.48 eV is 1.06. Sridevi and Reddy [5] have reported thins value as 0.24 and have stated that this is spin-orbit splitting for flash evaporated $CuInSe_2$ thin films.

Using these equations (11, 12), easy to calculate the values of n and k respectively. The variations of refractive index n and extinction coefficient k with incident photon energy for $CuInS_2$ thin films of as-deposited and variation temperatures (275 to 375^0C) are shown in fig (7, 8). The extinction coefficient (k) values show a decreasing dependence on the photon energy and refractive index (n) values shows increases with incident photon energy as shown in figure (8). This coefficient characterizes absorption of the electromagnetic wave energy in the process of propagation of a wave through a material. It was also taken into account the scattering and luminescence effects. The values of refractive index n and the extinction coefficient (k) have been calculated at as deposited (350^0C), various temperatures (275 to 375^0C), and these values listed in Table (1). It is observed that the extinction coefficient decreases with varied temperature of the substrate of different films and it also corresponds thickness values varies [29] with temperature.

The real and imaginary parts of ε (ε_1 and ε_2) are related to the refraction index (n) and the extinction coefficient (k). Therefore, the complex dielectric function of a material and its variation with wavelength is used to interpret the transmission features and/or reflection spectra of semiconductor films. Real and imaginary parts of dielectric constant (ε_1 and ε_2) of $CuInS_2$ thin films deposited at various temperatures with different thickness were calculated.

Mahir N. Al-Jabery et al [39] were grown thin films. They are also extinction coefficient (k), refractive index (n) and the real and imaginary dielectric constants (ε_1, ε_2) have been investigated. It may be also suggested that there small defects/ imperfect or non-stoichiometric results were observed at lower temperature [41]. So that temperature is important parameter for better results of films.

V. CONCLUSION AND FUTURE SCOPE

In Summary, we have prepared $CuInS_2$ thin films successfully by a simple and inexpensive spray pyrolysis deposition method. The optical properties of $CuInS_2$ films were investigated in present works. We show that due to the increased temperature, the band gap and structure of the polycrystalline films get rearranged. The characterization of the $CuInS_2$ layer on glass substrates gave thin film with band gap value between 1.43 and .2.48 eV and absorption coefficient in the range of 10^4 cm^{-1} with a direct optical transition and it has been suggested that the reported results of thin films could be a suitable candidate for using as an absorber in photovoltaic applications. The other properties appear to be new to the improved material. The values of absorption coefficient, extinction coefficient, refractive index and dielectric constant were found to be in conformity with previous works.

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