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$^\circ$C, at the interval of 25 to 350$^\circ$C 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 $t = 0.126 \, \mu m$ and at low transmittance (50%) of the films with highest thickness nearly equal to $t = 0.2163 \, \mu m$. Due to temperature variation, the atoms are arrange regular in manner compound of CuInS$_2$ films are investiogates. 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 chalcopryites like CuInTe$_2$ [5] CuInS$_2$ [6-8] is almost found higher values. CuInS$_2$ is one of the I-III-VI$_2$ chalcopryite-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 $10^4 \, \text{cm}^{-1}$ 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$_2$, CdZnSe$_2$ and CuInTe$_2$[1- 8]. As the temperature of the glass substrate increases, band gap (E$_g$ and E$_g$) values, extinction coefficient (k), imaginary part of dielectric constant (e$_i$) increased whereas real part of dielectric constant (e$_r$), refractive index (n) decreases with wavelength (l) 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$_2$S [14], developed Cu-rich CuInS$_2$ films showed an energy band gap of 1.38 eV at absorption coefficient of about $10^5 \, \text{cm}^{-1}$ [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°C) at the interval of temperature 25°C).

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$_2$)$_2$CS) were prepared of 0.02 M. Solution of CuCl$_2$, InCl$_3$ and (NH$_2$)$_2$CS 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$_2$)$_2$CS)S were used to prepare the initial chemical compound in aqueous solution mixed for the formation 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 spray 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 ($\alpha$), Energy band gap (E$_g$) refractive index (n), extinction coefficient (k) and Dielectric constant value in Real and Imaginary Parts ($\varepsilon_r$ and $\varepsilon_i$) 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 ($\alpha$) 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°C at the interval of 25°C (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 up to 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 ($\alpha$), $\alpha = 1/t \log (I_0/I)$ …… (1)

The absorption coefficient ($\alpha$) 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 ($\alpha$), is related to the extinction coefficient (k), by the following formula, $\alpha = 4\pi k/\lambda$. …… (2)

Where, $\lambda$ is the wavelength.
In the present works, absorption coefficient ($\alpha$), 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 ($E_g$) and energy of the incident photon which is,

$$\alpha h\nu = A(h\nu - E_g)^n$$

…… (3)

Here, $\alpha$ is the absorption coefficient, $h$ is the plank constant, $\nu$ 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, $E_g$ 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].

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 ($\alpha$) versus photon energy ($h\nu$) and extrapolating the straight line portion of the graph to the $h\nu$ axis given the values of band gap $E_{g1}$ [34]. The absorption coefficient is calculated by using these values of band gap ($E_{g1}$) and slope $A$ of the linear portion of the curves, for $h\nu$ 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 $E_{g1} = 1.42$ eV. This can only be explained, if we assume the presence of an additional absorption process. Using this value $E_{g1}$ and the slope $A_1$ of the curve, $\alpha_1$ is calculated for $h\nu$. 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 $\alpha_1$ is always less than observed absorption coefficient $\alpha$ 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 $\alpha_2$, defined by relation [5],

$$\alpha_2 = \alpha_{exp} - \alpha_{cal}$$

Fig. 3 Absorption coefficient versus incident photon energy $h\nu$ (eV) for various temperatures (250 to 375°C)

The nature of optical band gap can be determined with the help of the relation $\alpha h\nu = A (h\nu - E_g)^n$, 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 $(\alpha h\nu)^2$ as a function of photon energy ($h\nu$) for all thin films of as-deposited and CuInS$_2$ film with temperature 350°C. The linear portion of each was extrapolated to $(\alpha h\nu)^2 = 0$ and meet energy ($h\nu$) axis given the values of band gap $E_{g1}$ [34]. The absorption coefficient is calculated by using these values of band gap ($E_{g1}$) and slope $A$ of the curve, for $h\nu$ 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 $E_{g1} = 1.42$ eV. This can only be explained, if we assume the presence of an additional absorption process. Using this value $E_{g1}$ and the slope $A_1$ of the curve, $\alpha_1$ is calculated for $h\nu$. 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.
Fig. 4 shows the graph \((\alpha hv)^2\) versus \(hv\) for the composition of typical CuInS\(_2\) thin films as deposited at 350\(^\circ\)C. It observed that the graph was linear. When it is extrapolated to \((\alpha hv)^2 = 0\), the optical band gap \((E_g)\) is found to be 2.43 eV for CuInS\(_2\) thin film prepared at 350\(^\circ\)C temperature. This also indicates the presence of second direct allowed transition.

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 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 \((v\) and wavelength \((\lambda)\) in the medium, 
\[n = c / v\] …… (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 \(\mu = 1\), the real and imaginary part of dielectric constant can be calculated by using the following equations,
\[\varepsilon = \varepsilon_1 + \varepsilon_2 = (n + ik)^2 \] \(1\) …… (7)
\[(n - ik)^2 = \varepsilon - i\varepsilon \] \(2\) …… (8)
Where, \(\varepsilon_1 = n^2 - k^2 \) \(3\) …… (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,
\[n = \left(\frac{4R}{(R-1)^2} - k^2\right)^{1/2} - \frac{(R+1)}{(R-1)} \] \(4\) …… (11)
And, \(n = \frac{1+\sqrt{R}}{1-\sqrt{R}} \) …… (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 \((\lambda)\),
\[k = \frac{\alpha \lambda}{4 \pi} \] …… (13)

Where \(\lambda\) is the wavelength of the incident radiation and absorption coefficient \((\alpha)\) is calculated by equation (1 and 2).
Plotting the graph in between k and incident photon energy (hv), therefore it 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.

![Graph showing extinction coefficient versus incident photon energy](image1)

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

![Graph showing refractive index versus incident photon energy](image2)

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

![Graph showing real and imaginary part of dielectric constant](image3)

**Fig. 8** Real ($\varepsilon_1$) and imaginary ($\varepsilon_2$) Part of dielectric constant constant versus incident photon energy (hv) (eV)

| T (°C) | Thickness | Eg (eV) | Extinction Coefficient (k) | Refractive Index | Dielectric Constant |
|--------|-----------|---------|----------------------------|------------------|--------------------|
| 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         |
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 1.862 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 (αhv)$^2 = 0$ 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°C respectively. The energy band values of CuInS$_2$ for these samples decrease with increase in substrate temperature upto 350°C and further increase in substrate temperature [37], the band gap energy at 350°C 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$_x$S 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 Lontchitch 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 hv 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 CuInS$_2$ 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°C) 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 as at deposited (350°C), various temperatures (275 to 375°C), 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 $\varepsilon$ ($\varepsilon_1$ and $\varepsilon_2$) 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 ($\varepsilon_1$ and $\varepsilon_2$) 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 ($\varepsilon_1$, $\varepsilon_2$) 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 10^5 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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