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## مجلة علمية محكمة تصدر عن كلية التربية

# جامعة المرقب

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## Effects of composition and substrate temperature on the optical properties of CuInSe<sub>2</sub> thin-film

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**Abstract:** In this paper, the optical properties of CuInSe<sub>2</sub> thin films prepared by Stacked Elemental Layers (SEL) technique have been studied. The thin films were deposited in an evacuated chamber ( $<10^{-4}$  torr) by evaporating Cu, In and Se from electrically controlled heating appropriate crucible, then they were annealed under argon atmosphere at different temperatures and time. The band gap of CuInSe<sub>2</sub> was measured from the absorption spectrum. It was observed that the values obtained were strongly dependent on the Cu/In ratio and the annealing time and temperature. The optical properties of the films were characterized by transmission spectrophotometer measurements. The band gap of the samples was estimated using optical transmittance and found to be  $\sim 1.03$  eV.

Keywords: *CuInSe<sub>2</sub> thin film, photovoltaic energy, absorption coefficient of CuInSe<sub>2</sub>*

### 1- Introduction

CuInSe<sub>2</sub> (CIS) thin film is a direct band gap material with band gap  $\sim 1.03$  eV, high optical absorption coefficient ( $>10^4 \text{cm}^{-1}$ ), reasonable work function, good stability and large efficiency (it achieved an efficiency of 17%) [1,2]. Therefore, these properties make CuInSe<sub>2</sub> a promising material for photovoltaic applications [3, 4].

It is known that the electrical, optical, morphological and structural properties of this material are strongly influenced by the elaboration technique used, and by other several experimental parameters.

Several techniques are used for elaborating this material, among them: Close spaced vapour transport (CSVVT) [5], RF sputtering [6, 7], Spray pyrolysis [8], Atomic layer deposition [9], Electrodeposition [10, 11], Stacked Elemental Layer (SEL) [12] etc.



## 2 - Experimental procedure

A microscopic slide glass with dimension  $25 \times 75$  mm, was used as a substrate. It has been mounted inside a holder fixed to the upper side of a rotating wheel, installed inside an evacuation chamber ( $< 10^{-4}$  torr). The substrate was mechanically placed above the elemental sources at about 17 cm, from each of them successively by rotating the wheel. The temperature of the substrate was monitored by control nickel - nickel chrome thermocouples connected to the substrate to observe any increasing in the substrate temperature. Each of these sources was connected to a suitable power supply. Because of the very high temperature needed to evaporate *Cu* and *In* (2336 °C, 2000 °C respectively) a cooling system has been used to cool down the holders of these sources to keep a reasonable temperature inside the evacuation chamber.

Deposition rate of the elemental materials and the thicknesses of deposited layers have been monitored by a quartz crystal monitor, which was mounted near the upper side of the wheel, at the level of the substrate, and in the middle of the evacuated system. To prevent quartz monitor heating, it was connected to the cooling system also.

Two mechanical controlled shutters have been used to screen/unscreen both *Cu* and *In* sources simultaneously and the *Se* source the other time, while the second one used as an additional shutter to screen the *Se* source, to ensure shielding the *Se* source until it cools down the *Se* evaporation temperature (688 °C), which takes time up to five minutes. Each source has been shielded from other sources by fixed shields installed between the sources.

## 3 - Optical properties of *CuInSe<sub>2</sub>*:

Major sources of information about the band structure and energy levels are the optical properties. Most of the data reported about *CuInSe<sub>2</sub>* are for the energy range near the fundamental absorption edge. The results published are contradictory, and even the fundamental gap energies scatter widely. The main reason for this discrepancies is the tendency of *CuInSe<sub>2</sub>* to deviate from stoichiometry, which gives rise to high intrinsic defect concentrations that influence not only the electrical but also the optical properties of *CuInSe<sub>2</sub>*. The optical properties are highly dependent on films composition, defect chemistry, and growth parameters and are dominated by the degree and manner of polycrystallinity, including features such as rough surfaces and



secondary phases at free surfaces and grain boundaries. The absorption edge is observed to be quite abrupt for both the thin films and single crystals indicative of a direct band gap semiconductor. Values for the direct valence to conduction band transition energies and corresponding valence band splitting due to spin orbit interaction can be extrapolated from the optical data [14]. It is well established that  $CuInSe_2$  is a direct-gap semiconductor with the band extreme located at the center of the Brillouin zone. It has been shown that the dependence of the absorption coefficient  $\alpha$  on the photon energy  $h\nu$  at the band gap edge is always follows the relation:

$$\alpha(h\nu) = \frac{A}{h\nu} (h\nu - E_g)^{1/2} \quad (1)$$

where  $A$  is a constant.

The fundamental transition  $E_g$  is derived by extrapolating  $(\alpha h\nu)^2$  vs  $(h\nu)$  to the photon energy axis intercept [14]. Figure 1, represents the typical  $(\alpha h\nu)^2$  vs photon energy.

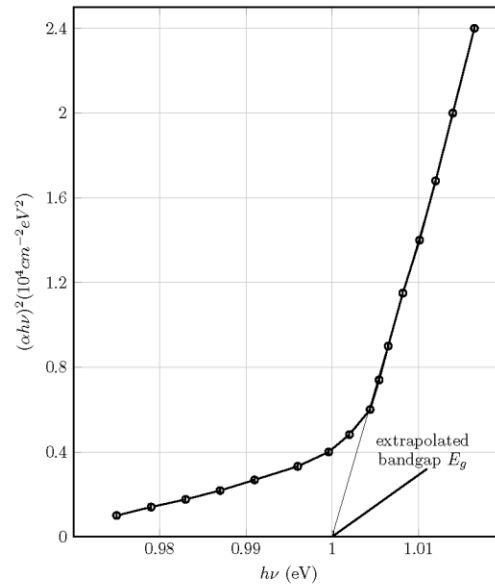


Figure 1: The typical  $(\alpha h\nu)^2$  as a function of energy [16].



This result indicates that the fundamental edge is due to direct allowed transitions between parabolic bands. From the theoretical point of view it is evident that the most reliable  $E_g$  value must be expected from an analysis of the optical absorption spectra of thin films. Only in this case the absorption spectrum can be measured up to photon energies well above  $E_g$  where all other factors affect the shape of the absorption become negligible and Eqn. 1 is valid. The room temperature gap energy is found to be  $(1.02 \pm 0.01) eV$  [14].

For energies lower than the band gap edge, the curve is not linear as expected from Eqn.1. This behavior is expected from the transitions associated with phonon absorption in this energy range. The absorption coefficient related the transition with phonon absorption is given by

$$\alpha = \frac{B(h\nu - E_{gi} + E_p)^2}{(\exp E_p/kT) - 1} \quad (2)$$

where  $B$  is a constant nearly independent on the photon energy,  $E_p$  is the phonon energy,  $E_{gi}$  is the indirect energy gap,  $T$  is the absolute temperature, and  $k$  is the Boltzmann constant .

As a function of composition, the absorption coefficient increases as the [Cu]/[In] ratio approaches unity, with band gap energy ( $\sim 1$ ) eV and then drops off sharply for copper rich material [15]. Near stoichiometry and Cu-rich films exhibit an onset of absorption at higher photon wavelength than do Cu-poor which will decrease the effective band gap. Copper rich films exhibited tail absorption (above  $10^3 cm^{-1}$ ) below the band gap edge [15]. This tailing is believed to be related with the secondary phase  $Cu_{2-\delta}Se$  at grain boundaries and free surfaces. It was found that the decrease in the measured gap energy was always connected with an increase of the tail absorption well below the gap energy. To account for this result it may be suggested that band gap narrowing effects due to high free carrier and impurity concentrations which are responsible for the observed variations in the gap energies of  $CuInSe_2$  in single crystal [13].

NaCN - based chemical treatment of  $CuInSe_2$  to remove the  $Cu_{2-x}Se$  phase results in decreasing of the tail absorption, increasing the band gap and change the composition of the film. The band gap energies varied from maximum values for the samples with the lowest electron and impurity concentration to the minimum values for the highest electron and impurity concentration [15].



The gap energies determined from optical spectra of thin films are practically same and close to the gap energies derived from un-doped single crystal, which indicate the small effect of the band gap narrowing in thin films [13].

The fundamental transition usually near  $1.02\text{ eV}$  for most thin films and near  $0.95 - 0.96\text{ eV}$  for single crystals for several causes:

1. Grain boundaries are sources of electric fields in polycrystalline materials. Dow-Red field model, predicts changes in the absorption characteristics as a result of these fields.
2. A more likely reason is differences (although slight) in the stoichiometry and compositional uniformity between the single crystals and the thin films. In connect with the last point, the heat treatment affects the films homogeneity and increases the crystal size which lead to gaps energies approximately same as those of single crystal.

#### 4 - Methodology

The absorption coefficient was determined at room temperature from transmission measurements made in the spectral range  $11200-14000\text{ nm}$ , using *MDR-3* photomonochromator. The transmitted photons were detected by a *PbS* detector and fed to Lock-in amplifier referenced to light chopper. The transmitted spectrum as a function of wavelength is recorded by PC computer, Figure. 2.

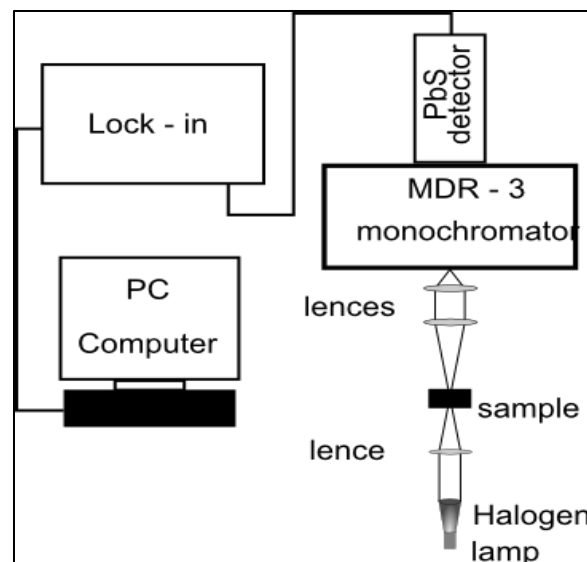


Figure 2: Scheme of the absorption apparatus.



The absorption coefficient was measured from the relation:

$$\alpha = -\frac{1}{d} \ln \left( \frac{I/I_0}{(1-R^2)} \right) \quad (3)$$

where  $d$  is the thickness of the thin films,  $I_0$  and  $I$  are the incident and transmitted light, and  $R$  is the reflectivity, its accepted value is 0.3.

The absorption coefficient was found to fit the form  $\alpha hv \sim (hv - E_g)^{1/2}$  in the high energy side of the curve indicating a direct energy gap, which is in agreement with the most of articles published about this material [13]. The energy gaps were calculated by extrapolating of the straight line section of the  $(\alpha hv)^2$  vs  $hv$  to the intersection point with energy axis.

## 5 - Results and discussions:

A typical absorption spectrum for  $CuInSe_2$  with near stoichiometric composition ( 25.44 Cu at. %, 24.97 In at. %, 49.59 Se at. %), as seen in Figure 3, is characterized by the abruptly increasing of the absorption coefficient at the band gap edge value ( $\sim 1eV$ ) and reaches very high values ( $> 10^4 cm^{-1}$ ) indicating a direct band gap.

Absorption coefficient values are affected by many factors, including initial composition, annealing environmental condition and annealing time.

The principal annealing at  $400^\circ C$  in vacuum or argon for 40 minutes is not enough to obtain a good quality of  $CuInSe_2$  films [12]. Additional annealing in air improves the transmittance of samples in the low energy region and affects the photo response as well.

### 5.1 - Absorption coefficient dependence on the annealing time and temperature:

$CuInSe_2$  thin films with the same composition have been annealed in different temperatures in argon ambient for 40 minutes. Figure 3, shows the absorption coefficient,  $\alpha$  for these samples as a function of photon energy. Films have been annealed at  $250^\circ C$  and  $300^\circ C$  show low transmittance in energies ranges less than the band gap. The films have been annealed at  $400^\circ C$ ,  $450^\circ C$  and  $500^\circ C$  exhibit high transmittance and the absorption coefficient increases abruptly at band gap value with steeper absorption edges. The morphology improvement and the dominating of the chalcopyrite phase are the main reasons for these changes [12]. The band edge becomes nearly insensitive to the annealing temperature in the range  $400^\circ C$ - $450^\circ C$ . Annealing at higher





temperatures causes samples' damage. Film's color became light brown and transparent, and seems to be completely out of chalcopyrite phase.

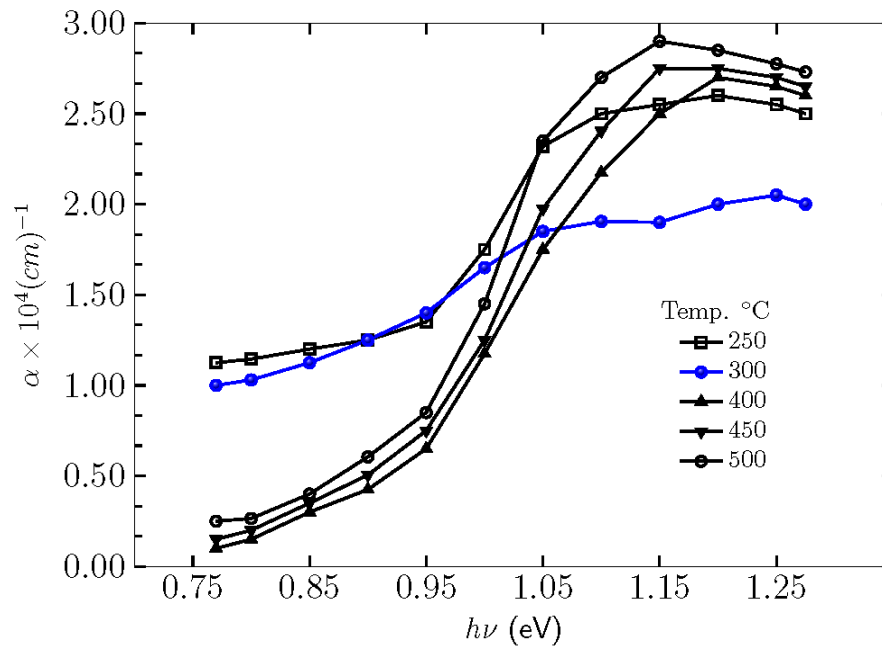


Figure 3: The absorption spectrum of four samples with near stoichiometry composition annealed in different temperature for 40 minutes.

The chalcopyrite phase structure is formed in the first few minutes of annealing, two to five minutes. The time of annealing has a considerable effect on the chalcopyrite phase which determines the quality of the  $CuInSe_2$  thin film. In Figure 4, six parts from one sample with near stoichiometry composition have been annealed at  $400\text{ }^\circ\text{C}$  for different times, 25, 30, 35, 40, 45 and 50 minutes. Samples annealed for time less than 35 minutes were with very poor absorption spectrum and exhibit very low transmittance indicating the not good crystallization and the dominating of not chalcopyrite phase. As the time of annealing become more than 30 minutes the band gap edge become steeper and the transmittance in low energy side increases. Increasing the time of annealing to 40, 45 and 50 minutes improve the transmittance of the thin films. The



growth of the crystal at boundaries and the dominating of the chalcopyrite phase are the main reasons for the improvement of the absorption spectrum.

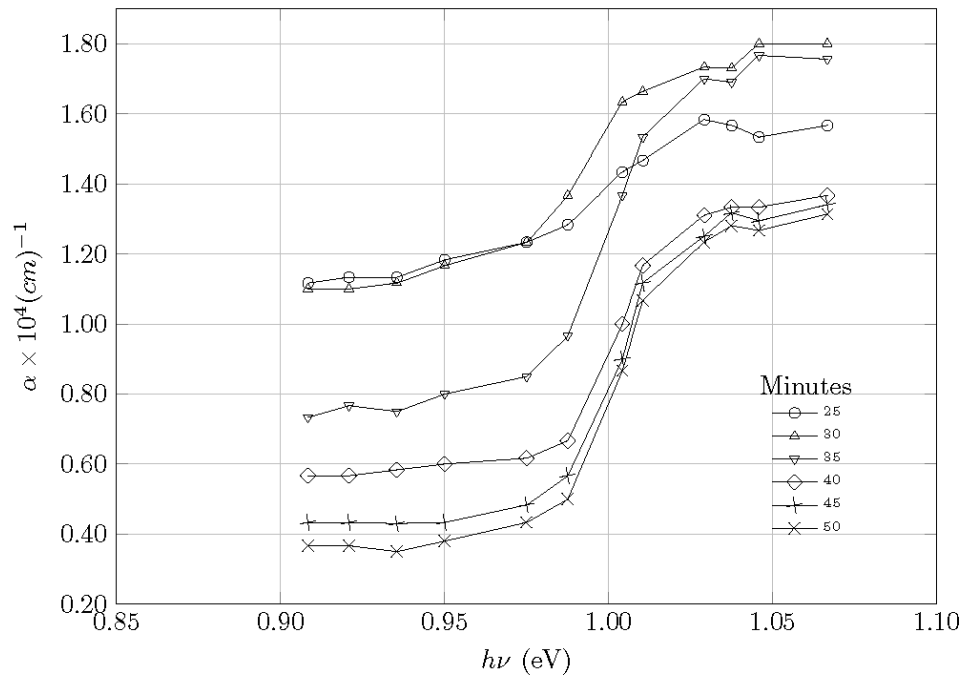


Figure 4 Six parts from the same samples were annealed for different times at 400°C.

Additional annealing in air is important to improve the absorption spectrum of the thin films. In order to demonstrate the effect of the annealing in air at low temperature (not > 150°C) on the absorption coefficient, the absorption spectrums for two samples with different compositions have been measured before and after annealing in air for two hours at 130 °C. Figure 5, represents the absorption coefficients as function of photon energy for these samples. It is obvious from these spectra that a principal change in the thin film structure has taken place after annealing in air.

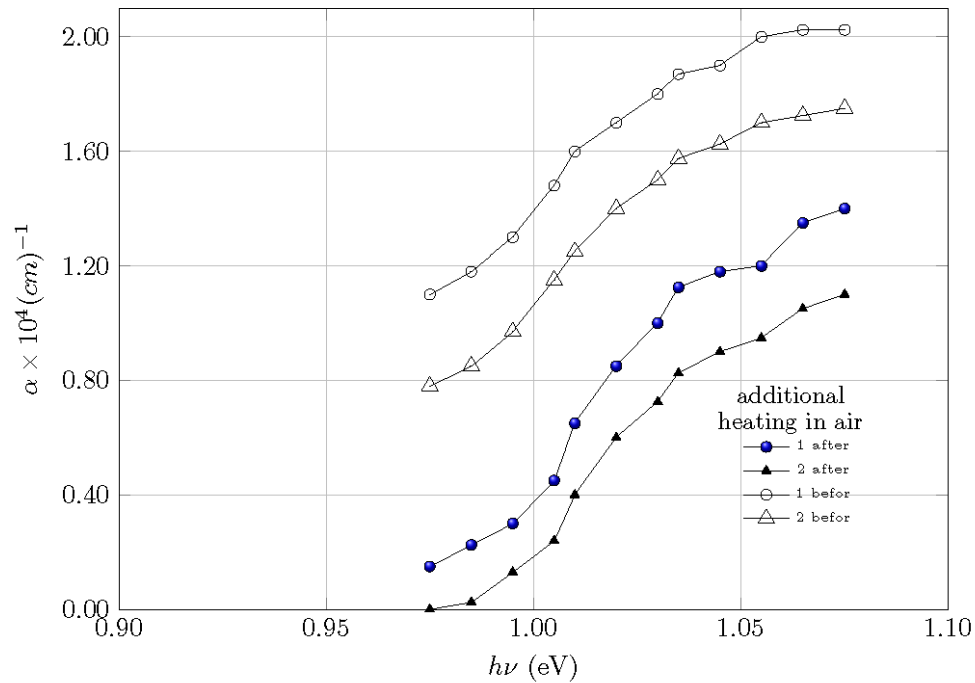


Figure 5: Absorption coefficients of two samples as a function of photon energy before and after additional annealing in air.

The most important result from the additional annealing is that the films become more homogeneous and the degree of compensation decreases. This effect can be observed in the low energy region of the absorption spectrum. The next important result is the growth of the crystal which improves the grain boundaries and affects the gap energy value. Traces of oxygen were detected by electron microprobe analysis in films have been annealed in air, and it is believed that the oxygen incorporation to the compound improves its optical and electrical properties.

The annealing for longer time or the annealing at higher temperatures causes the damage of the films which became pinhole.



## 5.2- Absorption coefficient and the transmittance dependence on the [Cu]/[In] ratio:

$CuInSe_2$  thin films with different  $Cu/In$  ratios from 0.82 to 1.27 have been annealed in argon for 40 minute at  $400^\circ C$ . In figure 6, the absorption coefficients at the band edge ( $\sim 1 eV$ ) for these films as a function of the  $Cu/In$  ratio are shown. The absorption coefficient increases as the  $[Cu]/[In]$  ratio approaches unity and then drops off for  $Cu$  rich films.

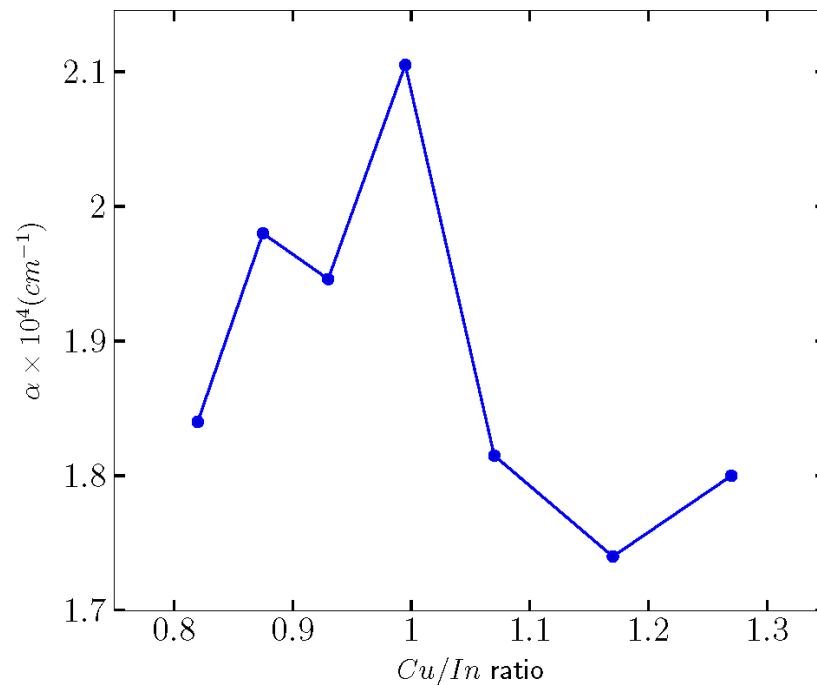


Figure 6: The absorption coefficient at band energy edge as a function of Cu/In ratio.

The transmittance of the thin films increases as the  $In$  increases, while the increasing of  $Cu$  content to the ideal stoichiometry value (25 at. %) decreases the transmittance. Samples exhibit good transmittance if they are little  $Cu$  poor ( $Cu$  not  $> 23.5$  at. %). In Figure 7, three samples with different  $Cu/In$  ratios have been annealed under same condition as functions of wavelength are represented.

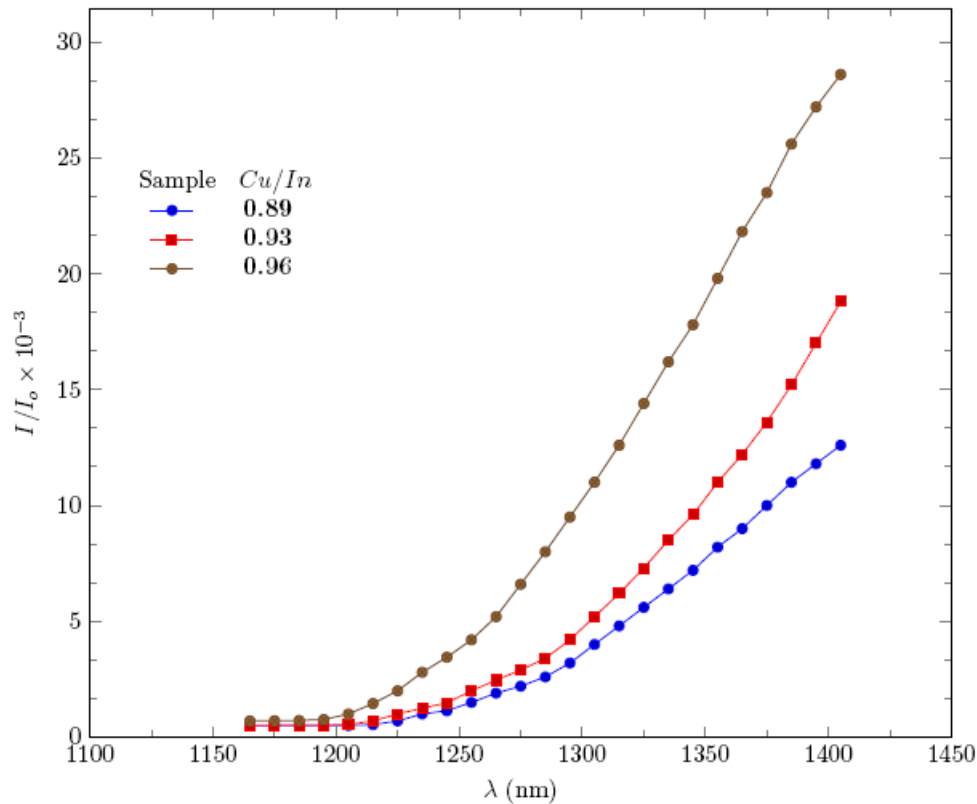


Figure 7: The transmittance for samples with different Cu/In ratio as a function of wavelength.

### 5.3 - $CuInSe_2$ band gap

The band gap for different composition  $CuInSe_2$  films have been extrapolated from the  $(\alpha hv)^2$  intersection with photon energy axis 'at  $\alpha = 0$ '. In Figure 8, the  $(\alpha hv)^2$  as a function of photon energy for five samples with nearly the same  $Se$  content ( $49.5 < Se < 50$  at. %) are shown. Two of them are little  $Cu$  poor ( $Cu$  23.8 at. %) the other three are near stoichiometry and  $Cu$ -rich ( $Cu$  24.5 - 26.7 at. %). Films with little  $Cu$  poor compositions exhibit the higher band gap values and less absorption tailing.

The band gap values were significantly affected by the additional annealing in the air at temperatures not more than  $150^\circ C$ . The band gaps for the samples shown in Figure 3 are extrapolated from the  $(\alpha hv)^2$  vs photon energy and are shown in Figure 8. It is very clear that



there is a gap shifting towards higher values with reducing of the tailing absorption. The highest band gaps' values obtained from samples with little *Cu* (~2.3 at. %) and are bit more than 1.0 eV.

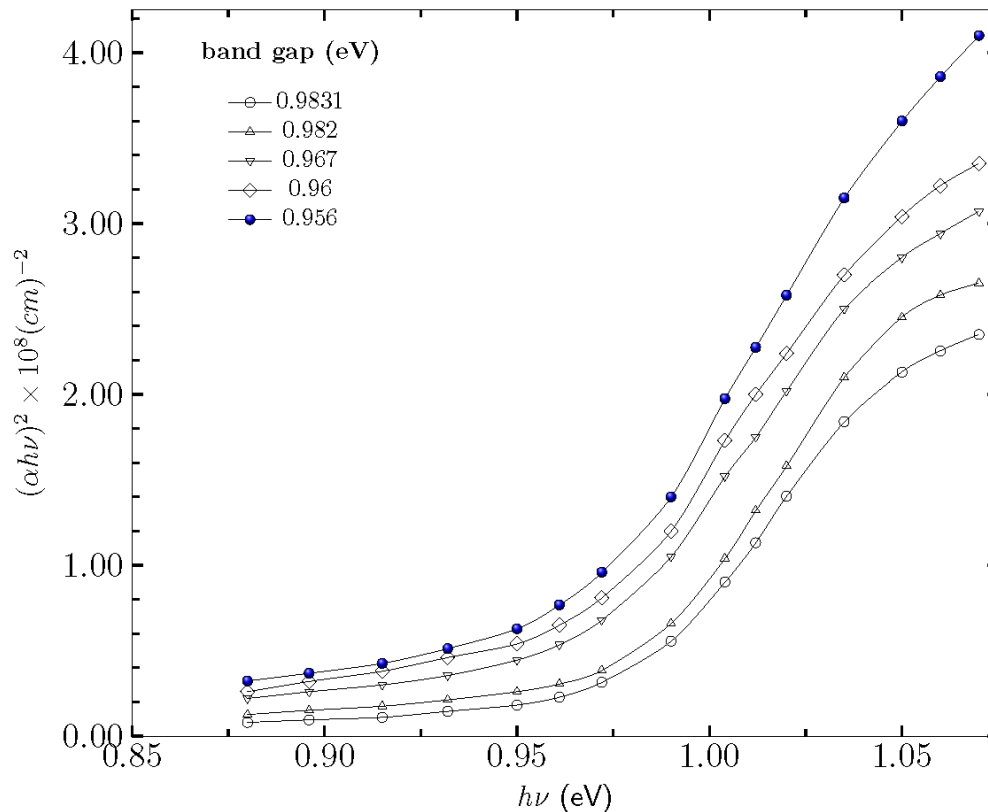


Figure 8:

The  $(\alpha h\nu)^2$  as a function of photon energy for five samples

## 6- Conclusion

The band gap of  $\text{CuInSe}_2$  was measured from the absorption spectrum. The values obtained were strongly dependent on the  $\text{Cu/In}$  ratio and the annealing time and temperature. However, the absorption coefficient increases as the  $\text{Cu/In}$  increases to reach unity then drops down for higher values. The lower energy side of the gap edge exhibited tail absorption especially for  $\text{Cu}$  rich films. The  $\text{Cu}_{2-\delta}\text{Se}$  binary phase which forms at surfaces grain boundaries is expected to be reason for this effect. The  $\text{Cu}_{2-\delta}\text{Se}$  has high absorption coefficient ( $>10^3 \text{ cm}^{-1}$ ) at these energies, which supports this explanation. The absorption coefficient for  $\text{CuInSe}_2$  with copper poor



composition ( $\text{Cu} \approx 23.5 \text{ at. } \%$ ) was ( $> 2 \times 10^4 \text{ cm}^{-1}$ ). Band gaps have been extrapolated from the absorption coefficient were in agreement with those measured from the photoconductivity and with those published.

Annealing at higher temperatures destroyed films by re-evaporation of Se and films exhibited pinholes.

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