Characterization of CuInSTe/CdS Heterojunctions

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Abstract. In this work, the role of substrate temperature in the range (293-423)K on the structure and microstructure of CuInSTe thin films was investigated. The results showed that the structure was amorphous for films prepared at ambient temperature Ts=293K while it was polycrystalline for films prepared at high temperature. The grain size and roughness increased with increasing substrate temperature. The frequency and temperature dependencies upon AC conductivity were investigated in the frequency range 100Hz-10MHz and temperature of heat treatment in the range of 303-453K. The AC activation energy was found to increase from 0.1132 to 0.1258 eV with increasing substrate temperature from 293 to 423K and decrease to 0.0962 eV with increasing frequency from 100 to 107 Hz. The exponents show a nonsystematic sequence with the increment of substrate temperature. The obtained results gave indication that there is a relation between the structure and preparation temperature as well as heat treatment. According to the current-voltage characteristic, the CuInSTe/CdS heterojunction prepared at substrate temperature of 423K showed the best electrical characteristics under illumination conditions.

Keywords: quaternary alloy CuInSTe; Thin films; Heterojunction; Activation energy, dielectric properties.

1. Introduction

The quaternary compounds (CuInSeTe, CuInSeS and CuInSTe) are considered as absorbing materials for solar cell applications. There are few data available about their bulk material properties [1-3] as well as thin films [4-7]. The available data refer that there is a strong dependence between their structure and preparation conditions, which may be due to the amorphous nature as well as the dependencies of their properties on the ambient conditions. Solaiman has investigated the electrical properties [8] and optical properties [9] for CuInSTe, CuInSeTe and CuInSeS thin films. Landery et al [10] prepared Chalcopyrite semiconductor compounds, MMIIIE₂ (MI = Cu, Ag; MII = Al, Ga, In; E = S, Se, Te) using microwave irradiation of appropriate mixtures of the pure elements in a domestic microwave oven and examined by X-ray diffraction (XRD) and energy-dispersive X-ray (EDX) analysis. William et al [11] presented the results of preparing photovoltaic CuInSSe cell using solution processed at law temperature ~350 °C. They indicated that band gap in the range (1.0-1.14) eV can be tuned by changing the amount of sulphur in the precursor solution. The obtained power conversion of 7.43% at 1-sun intensity.

The present paper is devoted to perform quaternary alloy of CuInSTe and then to prepare thin films at various substrate temperatures (298, 373, and 423) K using thermal evaporation method and point out the dependencies of dielectric and structural properties on deposition condition using relation between the preparation temperature and these properties.
2. Experimental Work
The bulk samples of CuInSTe were prepared by quenching technique. The exact amount of high purity elements copper, indium, sulphur and tellurium (99.99) supplied from Certified Chemicals, UK according with their atomic percentages are weighed using an electronic balance with the least count of (10⁻⁴ g). The material was then sealed in evacuated (~10⁻⁵ Torr) quartz ampoule (length ~ 25 cm and internal diameter ~ 8 mm). The ampoules containing material are heated to 1000 °C using controlled oven type Heresies electronic for 5 hours according to the phase diagram. The temperature of the furnace was raised at a rate of 10 °C/min. During heating the ampoules are constantly rocked. This is done to obtain homogeneous glassy alloys. After that the obtained melt was rapidly quenched into water.

The vacuum unit system, which is used to prepare thermally evaporated CuInSTe films at different substrate temperatures (293, 373 and 423K), was Edward Coating unit model 306A. High vacuum inside the enclosure (chamber) obtained using rotary pump to about 10⁻² mbar and diffusion pump where the pressure decreases to about 4x10⁻⁵ mbar. The deposition rate of thin films is about (10⁻¹⁵ nm/min).

The structural properties of the prepared CuInSTe films were studied using Shimadzu XRD-6000 X-ray diffractometer. The microstructure of the deposited films was also examined using AA3000 atomic force microscope and structural parameters such as crystallite size and roughness were obtained. Aluminum electrodes with thickness of 200nm were deposited on each adjacent surfaces of specimen by thermal evaporation technique under pressure of 10⁻³ Torr using an Edward coating unit. The specimen was fixed in a holder and placed into temperature-controlled oven (type Heresies electronic). High and low holder terminals are connected to dielectric analyzer (type Hewlett-Packard HP4274A & HP4275A), the third holder terminal was connected to the earth. The dielectric parameters like total resistance (RT), total capacitance (CT) and dissipation factor (tanδ) were measured (in parallel mode) under certain frequency range 10⁻²-10⁶ Hz. The device structure fabricated in this work consists of (from the bottom) an Al layer as the back contact (≈ 200 nm), thin film layer of CuInSTe thickness of ~700nm and CdS layer (t~300nm), all these layers are prepared by thermal evaporation. The wires were connected by silver baste. The sunlight incident upon the device from the top. The current-voltage measurements were carried out in dark for the prepared CuInSTe/CdS heterojunctions using a Keithley-616 digital electrometer and DC power supply. The bias voltage was varied from -1 to 1 volt in the forward and reverse bias conditions.

3. Results and Discussion
Figure 1 showed the X-ray diffraction patterns for bulk CuInSTe compound. Table 1 showed a comparison between the experimental and the standard peaks from Landry et al [10] and their miller indices since there is no available data for ternary compound to compare our results with them. It shows that the main peaks located at 2θ=32.91° and 43.99° which are corresponding to the reflection planes (212) and (110) are identically with the CuInS2,Te phase standard peaks of hexagonal structure, another traces of other minor phases like Cu1.52S and CuInS2 were found. Our results are similar to the finding of Robbins et al[12].
Figure 1. The XRD pattern for CuInSTe powder

Table 1. The X-ray diffraction data for CuInSTe powder.

| Samples   | 2θ (deg) | d_{exp} (Å) | (I/I_{o})_{exp} | FWHM (deg) | D (Å) | d \times 10^{-5} (Å)² |
|-----------|----------|-------------|------------------|-------------|-------|-----------------------|
| powder    | 27.381   | 3.254       | 23%              | 0.1956      | 417.58| 0.573                 |
|           | 27.858   | 3.199       | 43%              | 0.1655      | 494.79| 0.408                 |
|           | 32.916   | 2.718       | 9%               | 0.2346      | 353.26| 0.801                 |
|           | 43.993   | 2.056       | 100%             | 0.1620      | 529.11| 0.357                 |
|           | 46.180   | 1.964       | 13%              | 0.1774      | 487.02| 0.422                 |

The XRD patterns of CuInSTe thin films prepared at different substrate temperatures (293, 373, and 423 K) are plotted in Figure 2. The pattern of the thin films deposited at room temperature i.e. 293 K give indication of amorphous structure since it absent from any diffraction peaks, while there were many diffraction peaks belonged to the X-ray patterns of thin films deposited at elevated temperatures i.e. 373 and 423 K, this give indication of polycrystalline structure. The main diffraction peak for thin films prepared as 373 K belonged to the CuInS_{2-x}Te with hexagonal structure, there was some other traces of another phase like Cu_{1.96}S and In Te, while the main diffraction peaks for thin films prepared as 423 K belonged to the CuInS_{2-x}Te with tetragonal structure and there was no any other traces of any phase which identical with the data of Landry et al. and Robbins et al. [10 and 12]. It was pointed out that reaction of a stoichiometric mixture of the S, In, Cu and Te elements required for the chalcopyrite, CuInS_{2-x}Te_x does not yield the alloy phase, as determined by XRD, but a mixture of CuInS_x and CuInTe_x. This proposed that the alloy, CuInS_{2-x}Te_x, represents the limit of sulfide solubility in CuInTe_x. In addition, the formation of CuInS_{2-x}Te_x requires the relative concentration of sulfur to be higher than the solubility limit. Thus the obtained structure type at different substrates temperatures can be related with variation of solubility limit of sulfur in the prepared samples which showed
strongly dependence upon substrate temperatures. Debye–Scherrer’s formula [13] is used to estimate the crystal size of the film:

\[ D = \frac{0.94 \lambda}{\text{FWHM} \cos \theta} \]  

(1)

The estimated parameters are listed in table 2 the inverse relation between the crystal size (D) and dislocation density (δ) is clearly remarked.

Figure 2. The XRD patterns for CuInSTe thin films thickness of 700nm deposited at different substrate temperatures.

Table 2. The X-ray diffraction data of CuInSTe thin films thickness of 700nm deposited at different substrate temperatures (293, 373 and 423K)

| Samples | 2Θ (deg) | d_{exp} (Å) | (I/I_{0})_{exp} | FWHM (deg) | D (Å) | δ x10^5 (Å)^2 |
|---------|----------|-------------|-----------------|------------|-------|---------------|
| Films T_s=293K | 32.457  | 2.756 | 100% | 0.1310 | 631.89 | 0.25 |
|          | 43.542  | 2.103 | 9%  | 0.1700 | 503.41 | 0.394 |
|          | 66.750  | 1.400 | .99 | 0.2873 | 331.22 | 0.911 |
| Films T_s=373K | 27.758  | 3.211 | 100% | 0.24675 | 33.180 | 0.910 |

The grain size (grain diameter) and average roughness of CuInSTe thin films samples deposited on glass substrates with different substrate temperature have been measured using AFM, the obtained images is presented in Figure 3 and the results were listed in table 3.
Figure 3. AFM images of CuInSTe thin films deposited at different substrate temperatures

Table 3. The average grain size and average roughness of the prepared CuInSTe thin films

| Average roughness (nm) | Grain size (nm) | T_s (K) |
|------------------------|-----------------|---------|
| 3.42                   | 66.29           | 293     |
| 4.73                   | 80.53           | 373     |
| 5.25                   | 77.92           | 423     |

It is obvious from Table 3 that the average grain size (but not in systematic manner) and roughness get to increase with the increasing of T_s, moreover they increases from 66.29 to 77.92 and from 3.42 to 5.25 nm respectively when T_s increases from 293 to 473K due to structure enhancement by the action of heat. As seen from the x-ray diffraction pattern the crystallization takes place at elevated substrate temperatures (i.e., T_s=373 and 423K). The well-pronounced single phase of CuInSTe at high substrate.
temperature (423K) has produced much more crystallites arranged vertically with a size of 77.92 nm of the film. The total conductivity \( \sigma_{\text{tot}} \) at a certain frequency and temperature is defined as:

\[
\sigma_{\text{tot}}(\omega) = A_t \omega^s + \sigma_{\text{d.c}}
\]  

(2)

Where \( \sigma_{\text{d.c}} \) is the D.C conductivity which depends strongly on temperature and dominate at low frequencies, while \( \sigma_{\text{a.c}} \) is the A.C. conductivity which is weaker temperature dependence than \( \sigma_{\text{d.c}} \) and dominate at high frequency and nondependent temperature and \( (s) \) is a function of frequency and is determined from the slope of a plot \( \ln \sigma_{\text{a.c}}(\omega) \) versus \( \ln(\omega) \). \( \sigma_{\text{tot}}(\omega) \) obtained from the equation

\[
\sigma_{\text{tot}}(\omega) = \frac{t}{R.A}
\]  

(3)

where \( R \) is the sample resistance , \( A \) is the effective area for capacitance, \( t \) is the thickness. Figure (4) shows the angular frequency dependence of total conductivity \( \sigma_{\text{tot}}(\omega) \) for CuInSTe films deposited at different substrate temperatures(293, 373 and 423)K and thermally treated at (303, 333, 363, 393, 423 , and 453) K. It is clear that there is proceeding increase of \( \sigma_{\text{tot}}(\omega) \) that increased in the whole frequency range, i.e., the conductivity is frequency dependent or pure A.C and \( \sigma_{\text{tot}}(\omega) \) increases exponentially with frequency in the hole frequency range which give indication of domination of the second term of equation (2)upon the first term (D.C conductivity) ,and the polarization is electronic. The exponent \( s \), which is estimated from the slope of plot \( \ln \sigma_{\text{tot}}(\omega) \) against \( \ln(\omega) \) are calculated and listed in table 4. It is found that \( s \) increases with the increase of thermal treatment fro thin films samples deposited at low temperatures i.e. \( T_s=293 \) and 373K , indeed \( s \) increased from 0.58 to 0.79 and from 0.534 to 0.79 for when thermal treatment temperature increases from 303 to 453 K for thin films deposited at \( T_s=293 \) and 373K respectively , while \( s \) get to vary in contrast manner for thin films samples deposited at elevated substrate temperature i.e. 423K , indeed \( s \) decreases from 0.95 to 0.81 when thermal treatment temperature increases from 303 to 453 K. The reduction of \( s \) values fro thin films deposited at low \( T_s \) reflect the hoping mechanism. Thus, the suitable models for these samples are SP and CBH, respectively, as seen from Table 4.
The AC activation energy, EAC in the studied for CuInStTe films are estimated from the drawing of Ln σtot (ω) against the reciprocal absolute temperature by fitting different regions with the equation

$$\sigma = \sigma_0 e^{E_A / k_B T}$$

as indicated in Figure.5. are calculated. Table.3 illustrates the EAC values at selected frequencies (200Hz, 1kHz, 4 kHz, 10 kHz and 100 kHz). The results show that each sample declared one conductivity mechanism and hence, one activation energy EAC. On the other side, there is a direct relation between EAC and frequency, while there is an inverse relation between activation energy value EAC and TS. The EAC decreases from 0.1132 to 0.0962eV with the increase of TS from 293 to 423K, while EAC increases from 0.1132 to 0.1258eV with increasing frequency from 1kHz to 100kHz. The increase in frequency results in an increase of vibrating energy and hence the EAC values will be decreased. This indicated that conductivity is purely AC, while the elimination of the localized states and vacancies explained the increase of EAC with increasing TS. Seto’s [14] proposed the presence of large amount of trapping states at the grain boundary that able to capture free charge carries. These charged states at grain boundary create potential barriers, which oppose the passage of carriers from grain to neighboring ones, where these sites increase with increasing TS, which resulted in decreasing conductivity or increasing EAC values.
Figure 5. ln(σ_{tot}) with 1000/T for CuInSTe thin films deposited at different substrate temperatures.

Table 4. Values of $E_{AC}$ for CuInSTe thin films

| $E_{AC}$(eV) | $T_s$(K) | Frequency(Hz) | Thin Films Samples |
|-------------|---------|---------------|--------------------|
|             | 423     | 373           | 293                | CuInSTe            |
| 0.1258      | 0.1210  | 0.1132        | 200                |
| 0.0517      | 0.1126  | 0.1115        | 1000               |
| 0.0336      | 0.1089  | 0.1022        | 4000               |
| 0.021       | 0.0449  | 0.0973        | 10000              |
| 0.0114      | 0.0103  | 0.0962        | 100000             |

The real dielectric constant determined using the following relation: $\varepsilon_r = C / \varepsilon_0 . A$ (5) where $C$ is the capacitance, $\varepsilon_0$ is the permittivity of free space, $\omega$ is the angular frequency.

The real dielectric constant ($\varepsilon_r$) of CuInSTe films prepared at different $T_s$(293, 373, and 423)K and thermally treated at different temperatures(303, 333, 363, 393, 423, and 453), are measured within the employed frequency range (10^2-10^7 Hz). It is clearly from the $\varepsilon_r$ pattern versus Log($\omega$), in Figure.6 that $\varepsilon_r$ exhibits to increase with the increasing of heat treatment temperature, while it decreases with increasing frequency. This is ascribed to the fact that the electrode blocking layer is dominated, thus, the dielectric behaviour is affected by the electrode polarization, while $\varepsilon_r$ attained minimum values at high frequencies, which indicates that the dielectric signal is not affected by electrode polarization [8]. On the other hand, it is noticeable remarked that the values of $\varepsilon_r$ at frequency of 10^2 Hz, increase with increasing heat treatment and substrate temperatures. Indeed, the $\varepsilon_r$ increases from 615 to 1064 and from 615 to 802 when heat treatment temperature increases from 303 to 453K and substrate temperature increases from 293 to 423K, respectively.
The variation $\varepsilon_i$ with $\ln \omega$ for as deposited CuInSTe thin films and annealed at different temperatures. The imaginary dielectric constant $\varepsilon_i$ calculated from the relation

$$\varepsilon_i = \frac{\sigma_{AC}}{\varepsilon_\infty \omega}$$

(6)

The variation $\varepsilon_i$ with $\ln(\omega)$ are drawn in Figure 7 for CuInSTe thin films annealed prepared at different $T_s$ (293, 373, and 423)K and thermally treated at different temperatures (303, 333, 363, 393, 423, and 453). It can be seen that the dielectric loss absorption bands appear for CuInSTe films treated and deposited at high temperatures. While the loss bands disappear from most of the deposited samples. The absorption band observed at characteristic frequency proposed the foundation of relaxation dipoles for samples deposited and thermally treated at high temperatures. It is remarked. The relaxation peak shift to high frequency with the increase of temperature of deposition and treatment, but return to shift to lower frequency at high treatment temperature i.e. 453K. With the increase of temperature of deposition it is considered that there is tendency to crystallization as seen from x-ray diffraction of thin films, figure 2. the increase of crystalline content speed up the segmental motion throughout reducing the free volume. It is illustrated by the loss ban shifting toward high frequency range, resulting in reducing of relaxation time.
Figure 7. Variation of $\varepsilon_i$ with $\ln \omega$ for as deposited CuInSTe thin films and annealed at different temperatures.

It is well known that dielectric data is a contribution of two components: the contribution of conductivity that results an increase of dielectric constant $\varepsilon_r$ and dielectric loss $\varepsilon_i$ with the decreasing of frequency and relaxation process which show at $\varepsilon_i$ which tend to shift to low frequency side with the increase of temperature. Also the dielectric loss composed from the contribution of two components diffusion of charge carrier and dipolar orientation.

Using the equation $\omega_D = 1/\tau$ the relaxation time were estimated from frequencies corresponding to the loss maxima, where $\omega_{\text{max}}$ is ~ macroscopic relaxation time $\tau_D$, the values of $\tau$ were listed in Table 4. It is clearly remarked that rising of thermal treatment temperature lead to foundation of more than one absorption loss peak in ($\varepsilon_i$) spectrums.

The appearing of more than one loss peak is related with presence of amorphous and crystalline phase in the thin films samples of CuInSTe; the vanishing of absorption bands in law frequency range of the $\varepsilon_i$ diagram versus frequency give rise to the dominating of one phase. Thus rising of temperature of deposition or treatment caused the overall increase of $\varepsilon_r$ and $\varepsilon_i$ which ascribed to simultaneous contribution of dipolar and free charge. The increase of charge mobility resulted in increase of free charge carriers and the shift of loss peak toward higher frequency side.

Cole-Cole diagrams are shown in Figure 8. The polarizability ($\alpha$) was determined and listed in Table (2). It can be noticed that the values of $\alpha$ also show a nonsystematic variation with the increasing of TS, while $\alpha$ decreases with the increasing of thermal treatment, moreover $\alpha$ increases from 0.122 to 0.2016, when TS increases from 293 to 373K, while $\alpha$ decreases from 0.3920 to 0.1680, when TS increase from 303 to 453K for CuInSTe films that prepared at 373K, see Table (4). The decreasing and
increasing of $\alpha$ with the increasing of heat treatment resulted from the rise and weaken of the intermolecular forces respectively [16-18].

Figure 8. Cole-Cole diagrams for CuInSTe thin films deposited at different substrate
Table 5. Values of s, τ and α for CuInSTe thin films

| Ts=423K | Ts=373K | As deposited | Ta(K) |
|---------|---------|-------------|-------|
| α       | τx10^{-4} (sec) | S          | α       | τx10^{-4} (sec) | s          |
|---------|---------|-------------|---------|---------|-------------|
| 0.2016  | 0.2800  | 0.0398      | 0.956   | 0.3920  | 0.1792      | 0.534   | 0.1120  | 0.585  | 303     |
| 0.2240  | 0.2016  | 0.0398      | 0.929   | 0.1904  | 0.1008      | 13.281  | 0.544   | 0.1456  | 0.639  | 333     |
| 0.1456  | 0.2800  | 13.281      | 0989    | 0.903   | 0.1008      | 0.1680  | 13.281  | 0.508   | 0.1008  | 0.713  | 363     |
| --------|---------|-------------|---------|---------|-------------|
| --------|---------|-------------|---------|---------|-------------|
| 0.2240  | 0.1680  | 0.0398      | 0.845   | 0.0672  | 0.0336      | 0.796   | 0.621   | 0.0560  | 0.1120  | 0.159  | 0.834  | 423     |
| 0.2240  | 0.2016  | 0.0796      | 0.815   | 0.1680  | 0.1680      | 0.159   | 0.791   | 0.1232  | 0.1120  | 0.159  | 0.791  | 453     |

Variation of current under illumination at different power density with biasing voltage of p-CuInSTe/n-CdS thickness of 700nm deposited at various temperatures (293, 373 and 423K) is plotted in Figure.9. The measurements were carried out under illumination with different power intensity. From this figure it is clear that the photocurrent increases with increasing of the bias voltage. At forward biasing voltage the current rises exponentially with forward voltage until the slope becomes more gradual. This can be due to high level injection of carriers such that the applied voltage is no longer totally developed across the depletion region. At the high reverse biasing breakdown can occur due to impact ionization or Zener tunneling. These mechanisms can be separated by temperature dependence. The I-V parameters V_{oc}, I_{sc}, and η were calculated and listed in table 5. It is clear that increasing deposition temperature had significant effect on the illumination current, i.e., there is valuable increment in the illumination current at elevated substrate temperature. This arises from the phase transformation from amorphous to crystalline structure. The illumination current increases with the increase of Ts as a result of increasing grain size. This is in agreement with previous results [19].

It is evident from table .5 that V_{oc} tends to vary in non systematic sequence with Ts, i.e., V_{oc} increases and decreases with increasing of TS. The increase in V_{oc} is resulted from decreasing electron affinity (χ) between the CuInSTe system and CdS layer, while the decreasing of V_{oc} ascribes to increasing electron affinity between the two layers of the junction. The high values of V_{oc} and I_{sc} at TS=423K for CuInSTe/CdS indicate that this junction is very suitable for solar cell fabrication. For photocurrent consideration, which is equal to short circuit current I_{sc}, the smaller band gap is required because more photons are collected. It was found that maximum efficiency can be obtained when the band gap lies in the range 0.8-1.4eV and the ideal factor increases primarily due to the increase of V_{oc} while the photocurrent is increased linearly with the light intensity [20].

4. Conclusions
   Depending on the results from this work, one can put the following points:
   It can be concluded that the single phase with chalcopyrite structure of CuInSTe becomes more pronounced at elevated temperatures. The quaternary alloy CuInSTe was successfully prepared using quenching from the melt method. The exponent s value of CuInSTe films show progressive increase with the increase of temperature of deposition and thermal treatment giving rise that the conductivity is
pure A.C. The increase of deposition temperature result in increasing of polarizability \( \alpha \) values and hence reducing of molecular forces while increasing of thermal treatment temperature result in reducing of polarizability \( \alpha \) values of CuInSTe films consequently increases the intermolecular force. The continuous variation of \( \alpha \) value gives indication that the structure may be more enhanced with further increase of temperature.

Figure 9. I-V characteristics for p-CuInSTe/n-CdS heterojunction with \( t=700\text{nm} \) at different substrate temperatures and power densities

Table 6. Illustrated the values of \( I_{sc} \), \( V_{oc} \) for for p-CuInSTe/n-CdS heterojunction thickness of 700nm at different substrate temperatures (293, 373 and 423 K).

| \( T_s \) (K) | \( V_{oc} \) (V) | \( I_{sc} \) (mA) | \( P \) (mW/cm\(^2\)) | \( \eta \) (%) |
|-----|-----|-----|-------|-----|
| 293 | 0.09 | 0.80 | 100   | 0.072 |
|     | 0.10 | 0.77 | 160   | 0.048 |
|     | 0.10 | 0.80 | 230   | 0.040 |
| 373 | 0.07 | 0.09 | 100   | 0.004 |
|     | 0.07 | 0.90 | 160   | 0.032 |
|     | 0.06 | 1.00 | 230   | 0.06  |
| 423 | 0.18 | 46.3 | 100   | 4.2   |
|     | 0.19 | 48.4 | 160   | 5.7   |
|     | 0.20 | 46.2 | 230   | 9.2   |
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