



Structural, Optical and Electrical Properties of CuInTe₂ Thin Films Prepared by Spray Pyrolysis

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ABSTRACT

Thin films of CuInTe₂ were grown by spray pyrolysis technique. It is a promising chalcopyrite semiconducting material for solar cell fabrication. We have deposited thin films of CuInTe₂ on glass substrate at 350^oC by this method, using aqueous solutions of Cupric chloride, indium tri-chloride and tellurium tetra-chloride and studied their structural, optical and electrical properties. By varying the proportion of copper or Indium in the films, this material can be made n-type or p- type. From X-ray diffraction patterns the chalcopyrite structure of these films was confirmed. The films were polycrystalline. The value of lattice parameter a and c calculated are 6.182 Å and 12.364 Å respectively. From the analysis of the optical spectra of the films, the two direct allowed transitions is found to be at 0.98 eV and 1.26 eV. The effect of background absorption on the absorption spectrum is found to be negligible. The value of the grain size estimated in the films was of the order of 268 nm from scanning electron micrographs. The resistivity of the film was measured for the temperature ranging from 77 K to 473 K using Four Probe method. The activation energy calculated from Arrhenius plot. The films are not doped intentionally and therefore the defects observed in intrinsic nature. In the low temperature a variable range hopping conduction mechanism is predominates. The Hall mobility and carrier concentration at room temperature were calculated using the Van der Pauw-Hall method.

Keywords- CuInTe₂ thin films, Structural properties, Optical properties and Electrical Properties.

I. INTRODUCTION

CuInTe₂ belongs to the group of I-III-VI₂ ternary chalcopyrite semiconducting compound, which have a direct band gap varying between 0.92 eV and 1.02 eV, which falls in the optimum range for solar energy conversion and a very high absorption coefficient, it requires only a few micron of thickness to make devices, which reduces the cost of materials [1-4]. The highest efficiency with Cu-In-Te base absorber was reported 5.1 % by using conventional MBE system [5]. Various routes to prepare CuInTe₂ thin films exist and many have been atleast partially explored. CuInTe₂ has been successfully deposited using techniques such as flash evaporation, electrochemical deposition method, Vacuum evaporation method, Chemical spray pyrolysis (CSP), sulfurization of Cu/In bilayers, etc. But as reduction in manufacturing cost is an important factor for the ultimate success of photovoltaic device in the market, thin film deposition techniques like CSP has gained importance.



Recently researchers have been consistently trying to understand a defect structure of this materials since a new form of control over fundamental properties of I-III_{2n+1}-VI_{3n+2} compound, where n = 0,1 and 2 has been suggested. Mise and Nakada [6] have also grown the CuIn₃Te₅ ingot at different temperature (200-400 °C). It was reported that the two single crystals with compositions close to CuIn₂Te_{3.5}, CuIn₃Te₅ and CuIn₄Te₆ present a similar chalcopyrite structure. The p- type electrical behavior of the In- annealed CIT samples has been explained consistently by using the covalent models. According to this model, the anion vacancy should behave as an acceptor [7,8], due to the defects levels observed at 58meV and 15 meV in the electrical analysis are attribute [9], using the covalent bonding models to In_{Cu} and V_{Te} respectively. The density of the states effective masses of the electrons and holes are estimated [9] to be m_e* =0.16m_e and m_p* =0.78 m_e respectively.

In this paper we have reported the preparation of CuInTe₂ thin films by spray pyrolysis method and studied their structural, optical, electrical, grain size, Hall mobility and Carrier concentration of the films. This method is simple, inexpensive and suitable for water soluble salts and the deployment of large-scale production facilities with relatively lower capital investment. Thickness of the sample was measured by Michelson interferometer. Absorption edge analysis was carried out using transmittance verses wavelength in the range 350 nm to 1100 nm on UV-1800 Shimadzu Spectrophotometer. The electrical conductivity for temperature ranging from 77 K to 473 K is also studied. Hall mobility and Carrier concentration at room temperature are determined by using Van der Pauw-Hall Method. The conductivity type of the films was determined by the hot- probe method. Grain size of the films was determined by scanning electron microscope (SEM).

II. PREPARATION OF SAMPLE

The films of CuInTe₂ used in this work have been prepared by spray pyrolysis onto clean glass substrate. The chemicals used were of AR grade. Aqueous solution of Cupric Chloride (CuCl₂.2H₂O), Indium tri-chloride and Tellurium tetra-chloride were used for spraying the films on hot glass substrates. The morality of each solution was 0.02 M and mixed together in the ratio of 1:1:3.2 by volume. Excess tellurium is necessary to obtain CuInTe₂ films. The films deposited have a tellurium deficiency if the ratio of solution is taken as 1:1:2. The excess tellurium is used to remove this deficiency. Biological glass slides 1.30 mm thick were used as substrate. Temperature of the substrate was maintained at 350⁰C and was measured by a pre-calibrated thermocouple. The solution was sprayed at a pressure of 12 kg/cm². The glass sprayer was mechanically moved to and fro during spraying to avoid the formation of droplets on the hot glass substrate and to ensure instant evaporation. The rate of flow was maintained at 3.5 ml/min. To prepare low resistivity p-type films a slight excess of selenium, sulfur and tellurium [10-12] was required.

III. RESULTS AND DISCUSSION

3.1 Structural Properties

In order to obtain kinematically interpretable intensities for the different reflections, we have made X-ray powder diffraction pattern. Fig. 1 shows X-ray diffraction spectrum of as deposited CuInTe₂ thin films. The main strongpeaks of CuInTe₂ corresponding to 112, 211, 204, 116/312 are seen in the diffraction pattern, while weak reflections from 200/004, 235, 420 are presents. The lines were indexed by the usual procedure and

confirms the dominantly chalcopyrite structure of the films. The structural parameter 'a' and 'c' calculated from the XRD pattern of polycrystalline films are 6.182 Å and 12.364 Å respectively. These values were in good agreement with literature data (13-15). The high intensity reflection 112 plane indicates the preferred orientation along (112) direction. A similar preferred orientation along the (112) direction has also been reported by Kazmerski et al (13) and Sridevi et al (14) for evaporated method. Loferski et al (16) have reported the presence of an In₂O₃ line at $2\theta = 31^\circ$ in both sputtered and sprayed films in the similar group of compound like CuInS₂, presence of this line indicates that the atmosphere of the chamber in which the films are deposited contains oxygen. However, this particular line is totally absent in our XRD pattern, although the spraying was performed in air.

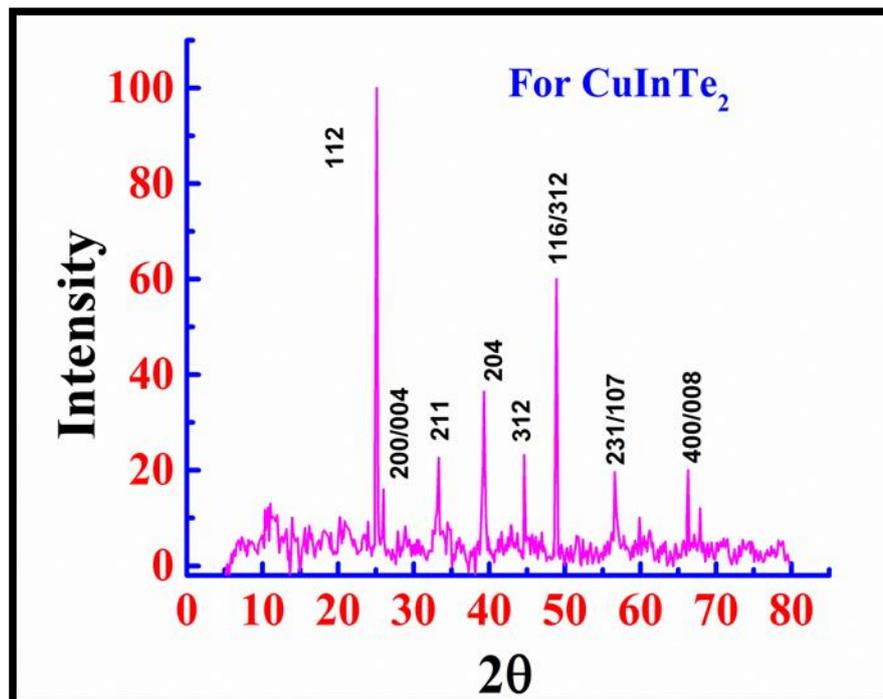


FIG.1 X- Ray diffraction of as deposited CuInTe₂ thin films

3.2 Optical Study

Optical transmission of the sample was measured at room temperature using a UV-1800 Shimadzu spectrophotometer in the range from 350 to 1100 nm. A glass plate identical to the substrate was placed in the path of the references beam and a substrate with deposited thin films was placed in the path of the sample beam for the transmission to be recorded. Fig. 2 shows the transmission (T%) vs Wavelength (λ) variation of the films of thickness 0.2030 μm . The absorption coefficient α at various wavelengths for the sample of thickness t is given by the relation (1),

$$\alpha = 1/t \log(I_0/I) \quad (1)$$

where I_0 and I are the intensities of the incident and the transmitted radiation respectively.

The value of α was calculated at various wavelength from the transmission curve using relation (1). It was observed from the fig.2 that interference effects can be neglected for films deposited on a thick non absorbing

substrate [10]. The variation of the absorption coefficient ‘ α ’ with energy near the fundamental absorption edge is expressed as,

$$\alpha_1 = A_1 / hv(hv - E_g)^p \quad (2)$$

where A_1 is the constant depend on the effective mass and the refractive index and p depends on the nature of transition $p = \frac{1}{2}$ for direct transition and $p = 2$ for indirect transition. The relation (2) is probably responsible for the absorption process. It is well established that copper indium telluride is a direct band gap semiconductor [17, 18]. To calculate exact value of band gap, a graph is plotted between $(\alpha hv)^2$ versus hv (fig.3).

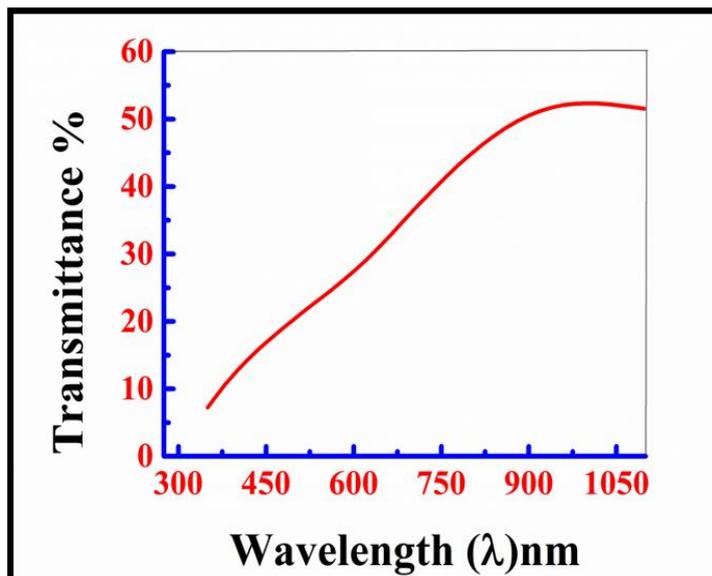


FIG. 2 Transmission (T) as a function of the wavelength (λ) of CuInTe₂ thin films.

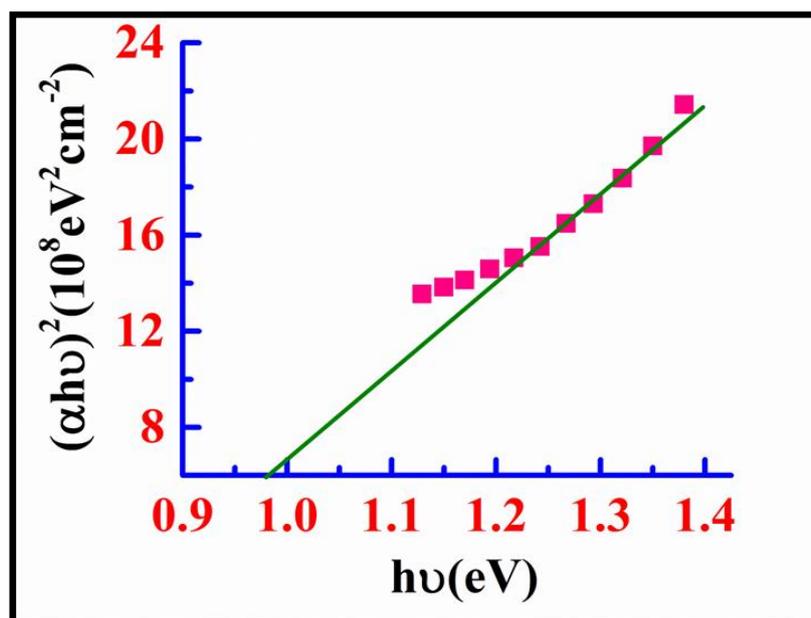


FIG.3. Plot of $(\alpha hv)^2$ against incident radiation ($hv \leq 1.4eV$).

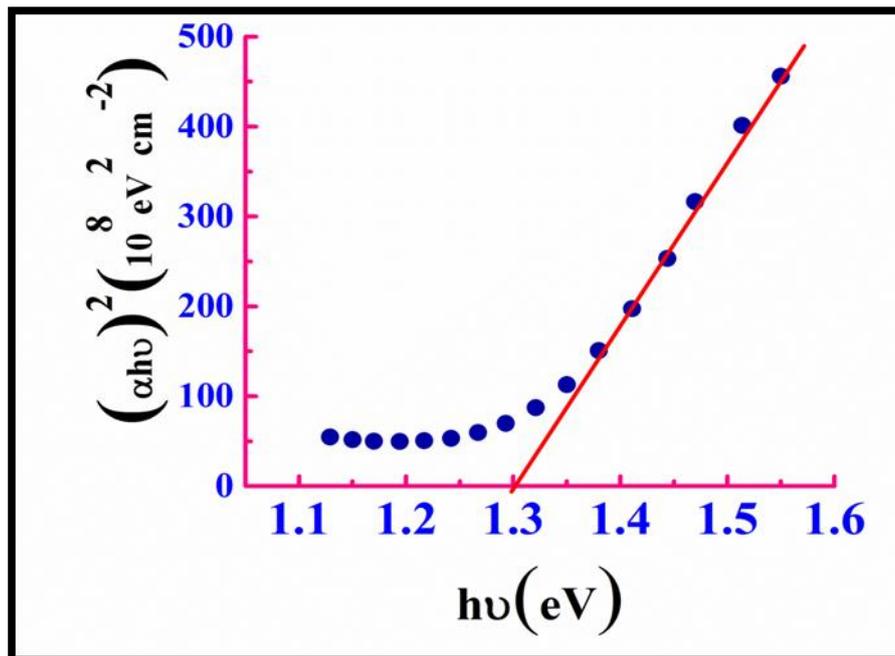


FIG.4. Plot of $(\alpha_2 hv)^2$ against incident radiation ($hv \geq 1.4$ eV).

The value of band gap energy E_{g1} is determined when this linear portion of the curve extrapolated to $(\alpha hv)^2 = 0$, the energy gap is calculated to be 0.98 eV which shows direct allowed transition [11]. This is in good agreement with the result obtained by Boustani et al [15], who have reported the same value of the band gap as 0.97 eV for thermal vacuum evaporation from a single source and for flash evaporation respectively while other workers [18-20] also reported the value of the band gap lies between 0.93 eV to 1.03 eV for different preparation technique. The optical measurement always lower values for the band gap which may be due to the existence of tail states and traps. Using this value E_{g1} and the slope A_1 of the curve, α_1 is calculated for $hv \geq 1.4$ eV. 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 [10]. The absorption due to this additional process is denoted by α_2 , defined by,

$$\alpha_2 = \alpha_{\text{exp}} - \alpha_{1\text{cal}} \quad (3)$$

Fig. 4 shows the graph $(\alpha_2 hv)^2$ versus hv for $hv \geq 1.4$, it is observed that the graph is linear. When it is extrapolated to $(\alpha_2 hv)^2 = 0$, the optical band gap is found to be 1.26 eV. This is due to onset of an additional absorption process. The difference between these two direct allowed transitions at 0.98 eV and 1.26 eV is 0.28 eV. Sridevi and Reddy [22] who have reported this value as 0.31 eV for flash evaporated CuInTe₂ thin films. Our calculated values are smaller than this value. In our method of calculating the absorption coefficient α , the reflection coefficient was assumed to be constant. The value of 0.28 eV obtained by us may be due to the inconstantly cause by the assumption regarding the constancy of the reflection coefficient. Sridevi and Reddy [24] have reported this value as 0.24 eV due to spin-orbit splitting for flash evaporated CuInSe₂ thin films. Similarly Tembhurkar and Hirde [10] have also reported this value about 0.33 eV for spray pyrolytically deposited CuInSe₂ thin films and attributed it to the same cause.

3.3 Electrical Properties

The thin films prepared by this method were invariably found to be p- type as determined by hot probe method. No changes in the type of conductivity were observed when the substrate temperature was changed. This result is in well agree with Sridevi and Reddy [22] who reported that CuInTe₂ is naturally p-type semiconductor. The electrical resistivity of the films was measured by Four Probe technique [25] in the two different ranges of temperatures; range (a) is from 300K to 473 K and range (b) is from 77 K (Liquid nitrogen temperature) to 273K. The resistivity for the range (a) is measured at atmospheric pressure while the resistivity for the range (b) is measured at 10⁻²Torr pressure, for which four probe arrangements together with the sample film was enclosed in a specially prepared stainless steel container, which was immersed in a liquid nitrogen bath. A typical Arrhenius plot for the conductivity of the CuInTe₂ thin films is shown in fig.5. It may be seen that there are three distinct linear regions in the plot. The activation energy (E_a) in p-type film can be calculated using the relation,

$$\sigma \propto \exp(-E_a/kT) \quad (4)$$

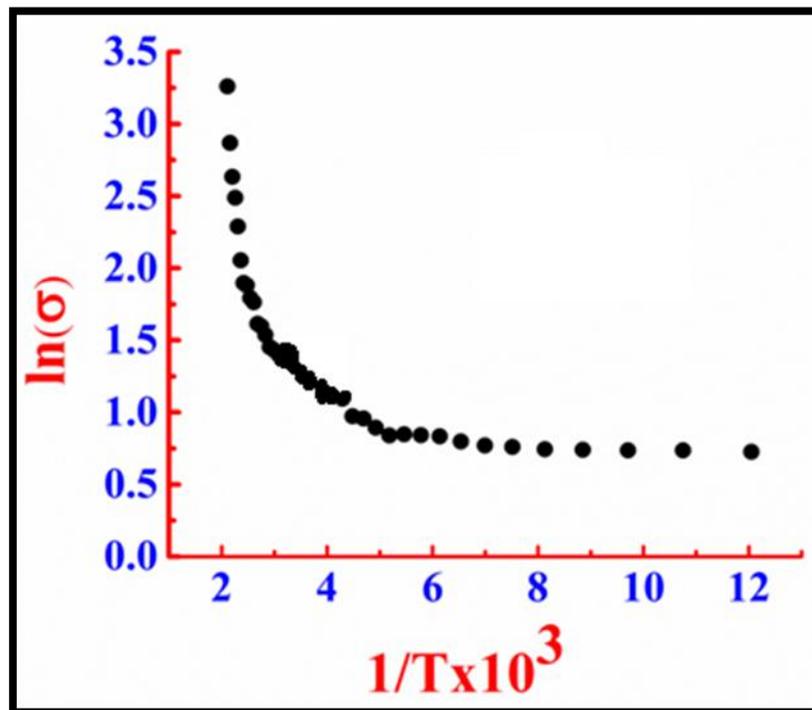


FIG.5. Arrhenius plot of conductivity of CuInTe₂ thin films.

It is seen from the fig.5 that the conductivity increases with increasing temperature. However the increase in conductivity is less in the temperature 77 K -125 K. Activation energies calculated for these three regions using relation (4) are 86 meV, 38 meV and 3 meV for the temperature ranges 300- 473 K, 160 K- 250 K and 90K- 125K respectively. The temperature dependence of the electrical conductivity σ can be represented by the relation,

$$\sigma = 1/T^{1/2} \exp(-E_a/kT) \quad (5)$$

where E_a is the activation energy. Fig.6 shows a plot of $\log(\sigma T^{1/2})$ versus inverse temperature. It is seen that the plot is linear. It can also be seen that plot the graph of $\log \sigma$ varies linearly with 1/T in accordance with equation

(5). Arrhenius plot can yield the different levels which are responsible for different donor or acceptor mechanisms. The change in the carrier mechanism is indicated by the change in the slope of the curve [10]. Films are not doped intentionally and therefore the defects observed in intrinsic nature. As the conductivity is p-type, acceptors like levels are expected to be present [10].

Shallow acceptor levels have been observed in several groups of (Cu-III-VI₂) compounds. Moreover the composition determination using neutron activation analysis indicates that the films are copper deficiency. These levels are two types of defects i.e copper vacancies (V_{Cu}) and antisite defects of Indium on copper sites (In_{Cu}) are more probable than Indium vacancies. In order to identify the origin of activation energy, the acceptor ionization energy of shallow acceptors was calculated by using hydrogenic approximation. The acceptor ionization energy is given by,

$$E_a = 13.6 \frac{(m_p^*/m_0)}{\epsilon_s^2} \quad (6)$$

Where ϵ_s is the static dielectric constant and m_p^* is the hole effective mass [26] ($m_p^* = 0.78m_e$) $\epsilon_s = 11$.

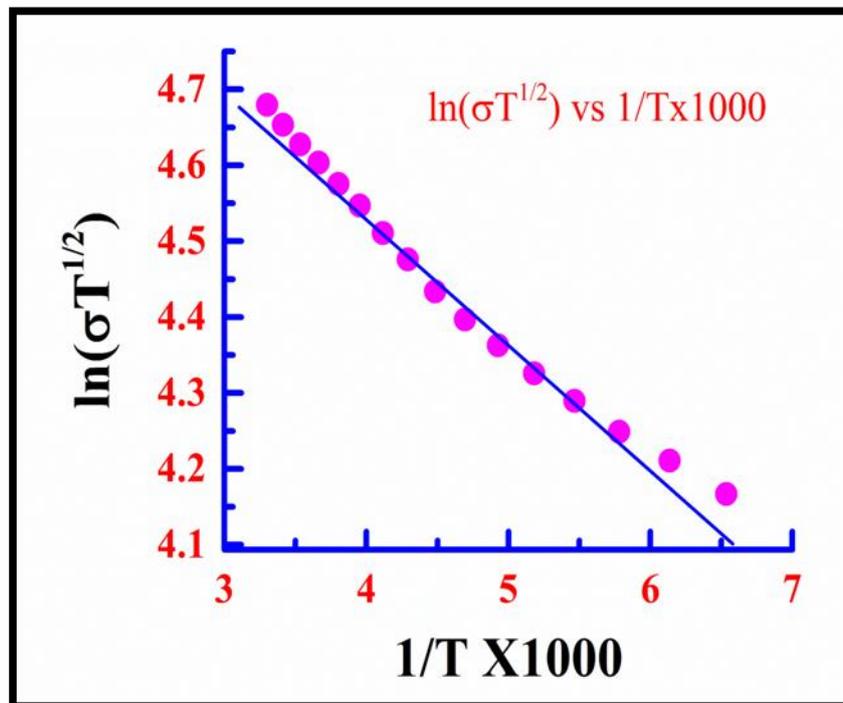


FIG.6. Plot of $\ln(\sigma T^{1/2})$ vs inverse temperature of CuInTe₂ thin films.

Using these values E_a is calculated to be 88 meV, the activation energy of 86 meV obtained in our investigation is agreed well with this value which may be due to the acceptor like levels produced by tellurium interstitials at high temperature region above 250K. Our value are smaller than Sridevi and Reddy [22] who was reported activation energy 100meV for the temperature range 250 K to 300K for flash evaporated CuInTe₂ thin films. In CuInTe₂ thin films, copper and Indium vacancies act as acceptor levels. For these vacancies the activation energy 38 meV were obtained in the temperature range 160 K to 250 K indicating the former to shallow acceptor levels. This value well agreed with Sridevi and Reddy (22) who have reported the activation energy of 39 meV for same temperature range. Dower et al [27] studied the similar works on vacuum deposition CuInTe₂

thin films in the temperature range 77 K to 300 K. They stated that the films would have a large deficiency in copper than indium which consistent with the higher carrier concentration. Since the films are polycrystalline, the activation energy compounds to the potential barrier of the grain boundary. The variable range hopping conduction mechanism [28] predominate in the low temperature region. According to Mott [29] phonon-assisted hopping conduction between localized states at low temperature should follow the relation,

$$\sigma = A \exp(-T_0/T)^{1/4} \quad (7)$$

where, A and T_0 are constants.

Fig.7 shows that plot of $\ln(\sigma T^{1/2})$ vs $T^{-1/4}$ for the temperature range (77 to 125 K) is linear, which indicate the presence of hopping conduction mechanism.

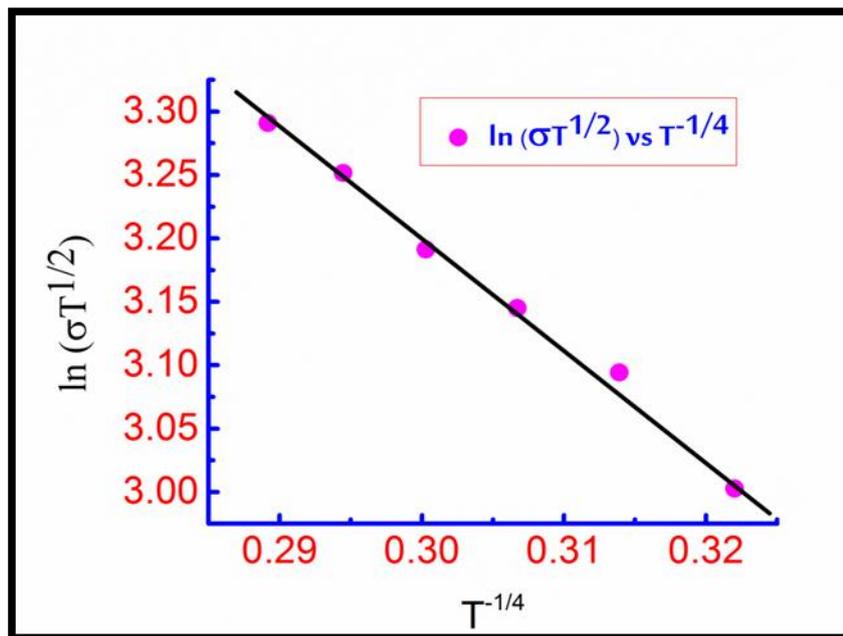


FIG. 7. Plot of $\ln(\sigma T^{1/2})$ vs $T^{-1/4}$ showing hopping conduction in CuInTe_2 thin films.

The existence of the localized states necessary for such a conduction process is a consequence of imperfections associated with polycrystalline films [18]. The activation energy for this temperature range is 3 meV. This value is smaller than Dower et al [27] who has reported activation energy 9.8 meV, for thermally evaporated polycrystalline thin films of CuInTe_2 in the range of Temperature from 77 to 300 K. They observed hopping conduction below 200 K. Similar mechanisms were reported by Sridevi and Reddy [22] for CuInTe_2 thin films and also by other workers [10,24] for CuInSe_2 thin films for sprayed and thermally evaporated method respectively.

IV. HALL MOBILITY AND CARRIER CONCENTRATION

The Hall coefficient at room temperature was determined by using the Van der Pauw Hall technique [30]. The Hall mobility at room temperature was found to be $7.38 \text{ cm}^2 \text{ V}^{-1} \text{ S}^{-1}$. This value well agreed with Kazmerski and coworkers [31, 32] for vacuum evaporation method. They observed Hall mobility in the range $10\text{-}15 \text{ cm}^2 \text{ V}^{-1} \text{ S}^{-1}$. However the value of Hall mobility observed in our film are very low compared with the value of about $107 \text{ cm}^2 \text{ V}^{-1} \text{ S}^{-1}$ reported by Dower et al [27] for bulk specimens at room temperature. The carrier concentration, as

calculated from the relation $P = 1/|eR|$, comes out to be $3.270 \times 10^{18} \text{ cm}^{-3}$. Our value are greater than Newmann et al [32] who have reported the value of hole concentration varied from $2.3 \times 10^{16} \text{ cm}^{-3}$ to $4.2 \times 10^{17} \text{ cm}^{-3}$ for epitaxial films of CuInTe_2 . Dower et al [27] and Newmann et al [32] for thermally evaporated CuInTe_2 thin films both have reported same value of Hall mobility $6 \text{ cm}^2\text{V}^{-1}\text{S}^{-1}$ at room temperature.

V. SEM

Scanning electron microscopy is a convenient method to study the surface morphology of thin films. Surface morphology of material plays an important role in solar energy conversion efficiency of the device. The SEM micrographs of as-deposited CuInTe_2 thin film (grain size $\sim 268 \text{ nm}$) on glass substrate at X15000 magnification is shown in fig.8. The CuInTe_2 thin film has dense, homogeneous and porous growth morphology. The nano-sized grains are uniformly distributed throughout the surface. The grains are quite small with equal size and shape, also their boundaries are well defined, and hence it was easy to calculate the exact average value of grain size from SEM image. The grain size of the films of the order of 268 nm shown in fig. 8. Grain boundary effects are predominant at low temperature.

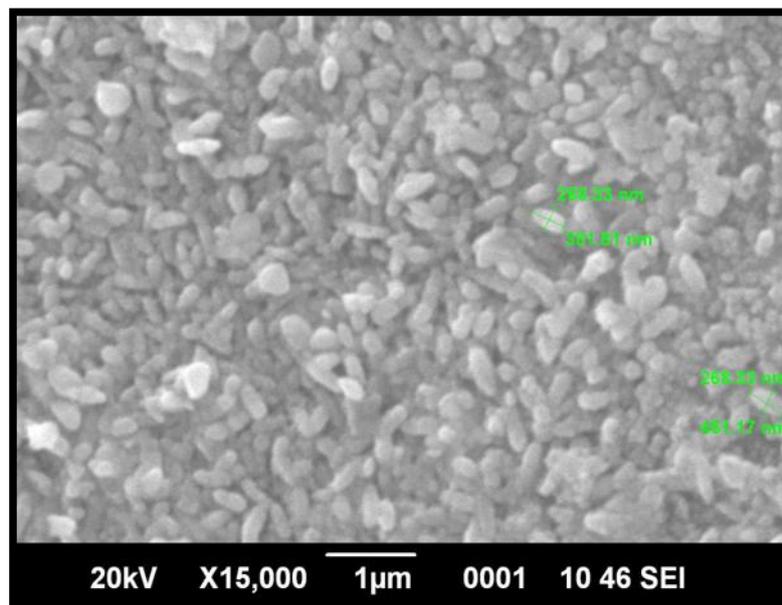


FIG.8. SEM micrograph of CuInTe_2 thin film.

V. CONCLUSION

We conclude that the spray pyrolytically deposited CuInTe_2 polycrystalline films was p-type. The structure is predominantly chalcopyrite. The films have preferred orientation along the 112 direction. The two direct allowed transitions at 0.98 eV and 1.26 eV are present. The activation energy 86 meV due to the acceptor like levels produced by tellurium interstitials and shallow acceptor levels 38 meV above the valence band. The grain size of the films of the order of 268 nm . Grain boundary effects are predominant at low temperature. At very low temperature range variable Hopping conduction mechanism appears to be operative.



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