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Structural and wavelength dependent optical study of thermally evaporated Cu₂Se thin films

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Abstract: The present work encloses structural and optical characterization of copper (I) selenide (Cu₂Se) thin films. The films having thickness 85 nm have been deposited using thermal evaporation technique in initial step of work. The structural and morphological studies of deposited thin films are then done by X-ray diffraction (XRD), scanning electron microscope (SEM), and surface profilometer measurements. Later on, ultraviolet-visible-near-infrared (UV-VIS-NIR) spectrophotometer and Raman spectroscopic measurements are performed for optical characterization of films. The structure and morphology measurements reveal that deposited material of films is crystalline. The optical band gap estimated from the optical transmission spectra of the film has been found 1.90 eV. The mean values of refractive index, extinction coefficient, real and imaginary dielectric constant are received 3.035, 0.594, 9.623, and 3.598, respectively. The obtained results are compared and analyzed for justification and application of Cu₂Se thin films.

Keywords: copper (I) selenide; Raman spectroscopy; thin films; transmittance; XRD.

1 Introduction

The copper chalcogenide thin films have generated a wide interest among the researchers due to their various applications in optoelectronic devices such as solar cells, photo detectors, photo conductors, photo thermal conversion,

electro conductive electrodes, sensor, laser diodes, thin film transistor etc. [1–4]. It is a semiconducting material, which has electrical and optical properties suitable for optoelectronic application [5]. The copper selenide compositions exist in different structural forms such as stoichiometric (CuSe, CuSe₂, Cu₂Se, and Cu₃Se) and non-stoichiometric compositions (Cu_{2-x}Se) [6, 7].

The structural and optical properties of the Cu₂Se thin films have been investigated in detail by different researchers. The current and voltage equation for Cu₂Se nanowires are reported to be non-linear and rectifying in nature while conduction in it is followed by different mechanisms in different voltage regions [7]. Mane and co-workers studied the p-type copper (I) selenide (Cu₂Se) thin films and found its potential application in p-n heterojunction solar cells [8]. The synthesis Cu₂Se nanoparticles through electrochemical procedure and its optical/electrical characterization are reported by Rong et al. [9]. It is described in literature that Cu₂Se composite having P-Cu₂Se heterojunction comprises gas-sensing properties towards acetone gas [10]. Yue et al. synthesized Cu₂Se electrode on copper grid substrate and his study indicates that Cu₂Se is suitable cathode material for sodium ion batteries [11].

The semiconducting material TiO₂ of energy band gap ~3.4 eV and refractive index ~2.5 is used in removal of organic impurity under ultra-violet photo catalysis by oxidation [12, 13]. When the ultraviolet (UV)-light incidents on TiO₂, electron-hole pairs are generated, that oxidizes organic pollutants in CO₂ and H₂O. The material TiO₂ anatase having high refractive index (>3.0) has been reported to be suitable for visible light photo catalysts as due to its low band gap (<2.0 eV) [14]. The surface area enhances and band gap reduces at nanoscale. Due to increase in surface area, more pollutants come across the surface of material and hence, the oxidation rate enhances. The reduction in band gap makes the material to be active in visible region. The nano material of Cu₂Se having particle sizes (75 nm) are reported to have 1.94 eV band gap [9]. Therefore, nano material of Cu₂Se and its thin film can be used as visible light photo catalysts with improved efficiency for removal of organic pollutants of water.

Even though, seldom works have been done on structural, electrical and optical properties of Cu₂Se thin films but

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the studies on wavelength dependent variation of refractive index, extinction coefficient and dielectric constant of nano Cu₂Se thin films are not much reported in literature. The present work is, therefore, concentrated on synthesis and characterization of Cu₂Se nano thin films. In initial step, thin films of Cu₂Se having thickness 85 nm are prepared using thermal evaporation technique. The deposited films are characterized by X-ray diffraction (XRD), scanning electron microscope (SEM) and surface profilometer measurements for its structural and morphological properties. Later on, UV-visible-near-infrared (VIS-NIR) and Raman spectroscopic measurements are performed for the optical characterization of the films. The obtained results of deposited films are discussed and analyzed to explore its inherent structural, optical and dielectric properties.

2 Synthesis and measurements

For the preparation of films, Cu₂Se powder of 99.9% purity was purchased from Sigma-Aldrich. After cleaning glass substrate with acetone and isopropyl alcohol, the deposition of Cu₂Se on glass substrate was carried out by thermal evaporation technique using vacuum coating unit model 12A4D-T “HIND HIVAC” under vacuum 5×10^{-5} mbar. In this process, roughing and backing vacuum were maintained at 6×10^{-2} mbar while primary and secondary current were settled at 6.0–6.5 A and 110–120 A, respectively. Furthermore, the deposition on substrate was taken only for 30 s to form the films. After the formation of films, surface profilometer (Veeco dektak 150) apparatus was used to measure the thickness of thin films. Later on, XRD measurements of deposited film was carried out with X’ Pert Powder PANalytical using Cu-K α radiation ($\lambda = 1.5405 \text{ \AA}$). The diffracted intensity was measured for angular variation of 10° – 80° . The SEM (Carl Zeiss EVO 40 Cambridge UK) was used to record the surface morphology of films. The UV-VIS-NIR spectrometer lambda 750 was used to measure the transmission of thin film in the spectral range 300–1100 nm. A Raman spectrum was monitored by using Renishaw in Via Raman microscope in the range of 100–1500 cm⁻¹.

3 Results and discussion

3.1 Structural and morphological study

3.1.1 Thickness and XRD measurements

The measurements made on deposited thin films with surface profilometer reveal that the Cu₂Se films (having

deposition time 30 s) have thickness 85 nm. This confirms that the thickness of prepared films is of the order of nanometer. The XRD is an important investigative technique for phase identification and structure analysis of grown film. The XRD pattern of Cu₂Se thin film is shown in Figure 1. A comparison with JCPDS file no. 65-2982 confirms that the obtained diffraction peaks of the film at $2\theta = 27.11^\circ$, 44.72° , 52.92° , 64.90° , and 71.52° can be assigned as (111), (220), (311), (400), and (331), respectively. It also confirms that the phase of the film is cubic with lattice parameters $a = 5.7600 \text{ \AA}$, and space group Fm $\bar{3}$ m {225}. The crystallite size (D) of grown film is determined using Debye-Scherrer’s formula from the full width at half maximum (β) of peaks expressed in radians [15].

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where, constant K is equal to 0.94 and λ is the X-ray wavelength while θ is diffraction angle. The calculation with Equation (1) provides that the crystallite size of the film which is found equal to 41 nm. From Figure 1, it also confirms that Cu₂Se thin film is polycrystalline in nature. Sudha et al. have reported 84 nm crystallite size of Cu₂Se thin film having thickness 134 nm [1]. The synthesis and non-destructive characterization of zinc selenide thin films crystallite size increases with increasing thickness/deposition time [16]. Yet, the crystallite size of film is dependent of various parameter during deposition of films such as temperature of substrate deposited on glass, deposition current, deposition time, concentration of structural defect, increasing thickness of the film etc. However, the integrated study of present and Sudha et al. works reveals that crystallite size of Cu₂Se thin films increases with increasing thickness.

3.1.2 SEM and EDS measurements

The SEM has been used to explore the surface morphology of the deposited nano thin film. Figure 2 represents the SEM micrograph of the film under study which predicts that deposited particles on glass substrate are randomly distributed over the surface with an average size ~ 70 nm having approximately spherical shape. The deposited film surfaces are also confirmed by SEM micrograph. Xue et al. have also found the spherical shape of particles in Cu₂Se thin films [17]. Figure 3 shows the energy-dispersive spectroscopy (EDS) spectrum of the prepared thin film sample by the emergence of their respective peaks which confirms the presence of Cu and Se elements in the thin film. The spectrum also shows that the prepared thin film does not contain any additional impurity elements.

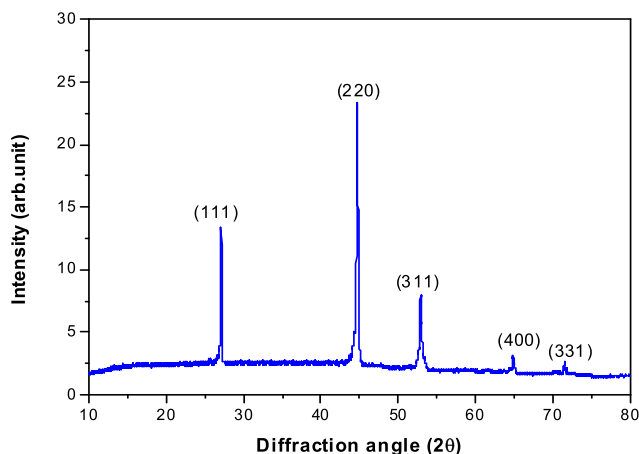


Figure 1: X-ray diffraction (XRD) patterns of the Cu₂Se thin film.

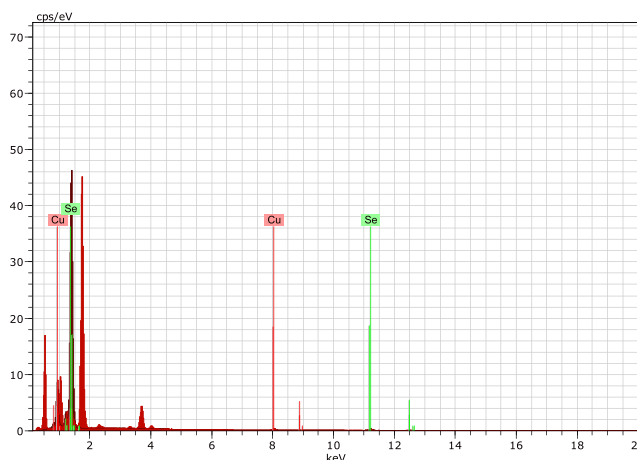


Figure 3: The EDS spectrum of the deposited Cu₂Se thin film.

3.2 Optical characterization

3.2.1 Optical transmission measurements

The optical transmittance spectra of the Cu₂Se thin film is recorded with UV-VIS-NIR spectrophotometer in transmittance mode for the range of 300–1100 nm as shown in Figure 4. The intensity of transmitted light for mid-VIS to IR region is received to vary 20–60% (Figure 4). The optical transmittance is found to increase with wavelength. The optical transmission of the light in the IR region is obtained as 40–60%. The Fresnel's theory of electromagnetic wave deduced that the refractive index of concern medium enhances with decay in amplitude ratio of transmitted and incident electromagnetic waves or transmittance of the concern medium [18]. Thus, the gradual increase of transmittance from visible to infra-red regions for Cu₂Se film reveals the decay in refractive index of film.

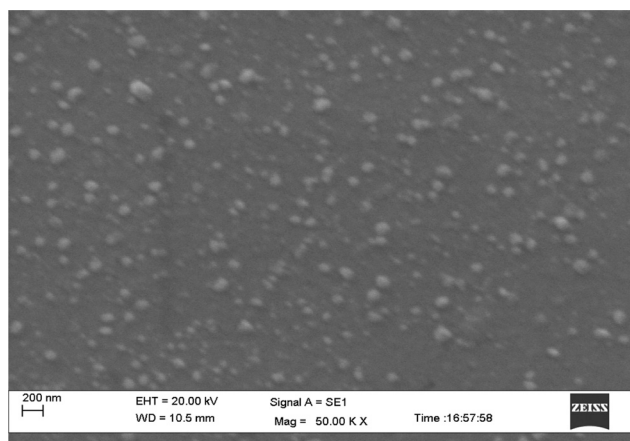


Figure 2: Scanning electron microscope (SEM) micrograph of the deposited Cu₂Se thin film.

We have also calculated the optical band gap for the deposited thin film using Wood and Tauc formula [19]:

$$\alpha = \frac{B(h\nu - E_g)^m}{h\nu} \quad (2)$$

where, $h\nu$, E_g and B are energy of radiation, optical band gap and tailoring band constant, respectively. The constant m has value 1/2 for the direct band gap of allowed transition. The obtained curves between $(\alpha h\nu)^2 \sim h\nu$ for the deposited thin film is shown in Figure 5. The estimated value of optical band gap with optical transmission spectra (Figure 5) for the present deposited Cu₂Se film (85 nm thickness) is found 1.90 eV. This value of band gap is quite close to the earlier reported values for the Cu₂Se films [8, 9]. Sudha et al. have reported 1.75 eV band gap for Cu₂Se thin film having thickness 134 nm [1]. The band gap of thin films have been reported to decrease with increase in film thickness/crystallite size [16]. The present finding along with Sudha et al. work supports the same characteristics of band gap for Cu₂Se thin film. Since, the wavelength corresponding to 1.9 eV is 653.3 nm which belongs to visible region therefore; the present thin film can be used in production of laser light. The reported values of energy band gap for CdSe, CuSe, ZnSe, ZnO, and ZnS, nano thin films are 1.7 eV, 2.03 eV, 2.6 eV, 3.3 eV, and 3.5 eV, respectively [6, 16, 20–22]. Hence, the present nano thin film has low band gap in comparison to other oxide/sulphide semiconducting nano thin films while possesses approximately same band gap as for CdSe and CuSe.

3.2.2 Refractive index, extinction coefficient and dielectric constant calculations

The refractive index is an important property of the thin film that signifies the amount of light transmitting through

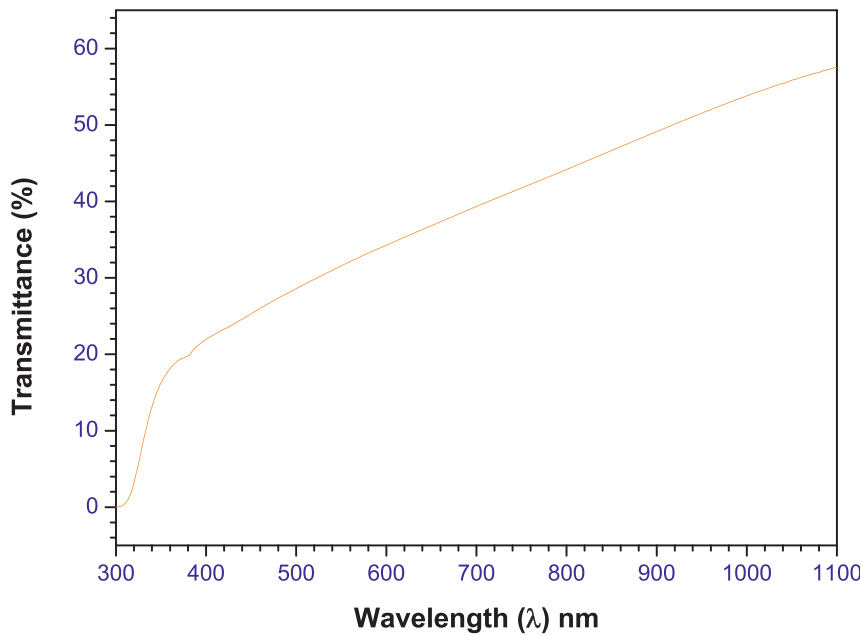


Figure 4: Optical transmission spectra of the Cu₂Se thin film.

it. During the transmission of electromagnetic wave through thin film having negligible absorbance, the light propagates through several layers (1st, 2nd, 3rd to t th layer) of a transparent medium. The transmittance light of t th layer can be written as [23].

$$T_n = (1 - R)^t \quad (3)$$

For normal incidence, the reflectance 'R' is function of refractive index 'n' and extinction coefficient 'k' [23].

$$R = \frac{(n - 1)^2 + k^2}{(n + 1)^2 + k^2} \quad (4)$$

On solving Equation (4) using componendo and dividendo method, the following expression of refractive index n is obtained.

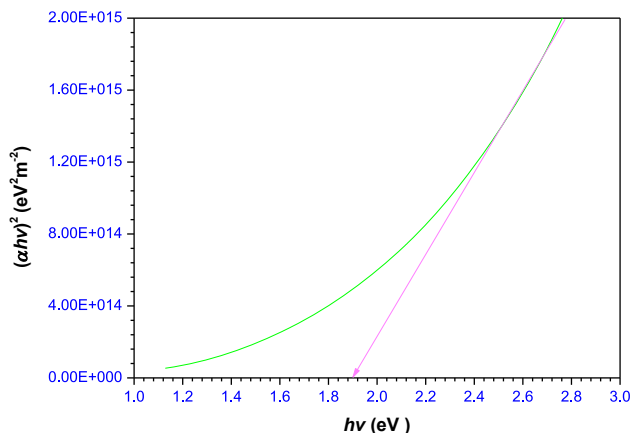


Figure 5: Plot $(\alpha h\nu)^2$ vs. $h\nu$ for Cu₂Se thin film.

$$n = \frac{(1 + R) \pm \sqrt{4R - k^2(1 - R)^2}}{(1 - R)} \quad (5)$$

The extinction coefficient (k) measures the absorption of light by the medium. Numerically, it is equal to $\alpha\lambda/4\pi$ [24]. Here, α is absorption coefficient and can be obtained with the help of transmittance as $\alpha = d^{-1}\ln T^{-1}$. Here, d is thickness of the film. The quantities k and n for the deposited Cu₂Se film are estimated using the measured transmittance under wavelength variation. The estimated extinction coefficient and refractive index with wavelength variation are shown in Figure 6. The mean value of extinction coefficient and refractive index of present Cu₂Se thin film are found 0.594 and 3.035 for wavelength range 300–1000 nm, respectively. The refractive index of ZnO, ZnS, CuSe, ZnSe, and CdSe nano thin films have been reported to be in between 2.0 and 2.8 in visible region [25–29]. Thus, the present nano thin film encompasses large refractive index in comparison to other semiconducting sulphide/selenide/oxide nano thin films. The polynomial curve fit analysis of n and k indicates that these physical quantities are eighth order polynomial function of wavelength (n or $k = \sum_{i=0}^8 B_i \lambda^i$). Here, B_i and λ^i are the polynomial coefficients and wavelength, respectively. The value of polynomial coefficients and its error are mentioned in Table 1.

The dielectric constant is a well-known fundamental intrinsic property of material. Since, the wave propagation vector is a complex number ($n + ik$) for anisotropic medium or thin film. Therefore, the dielectric constant of the

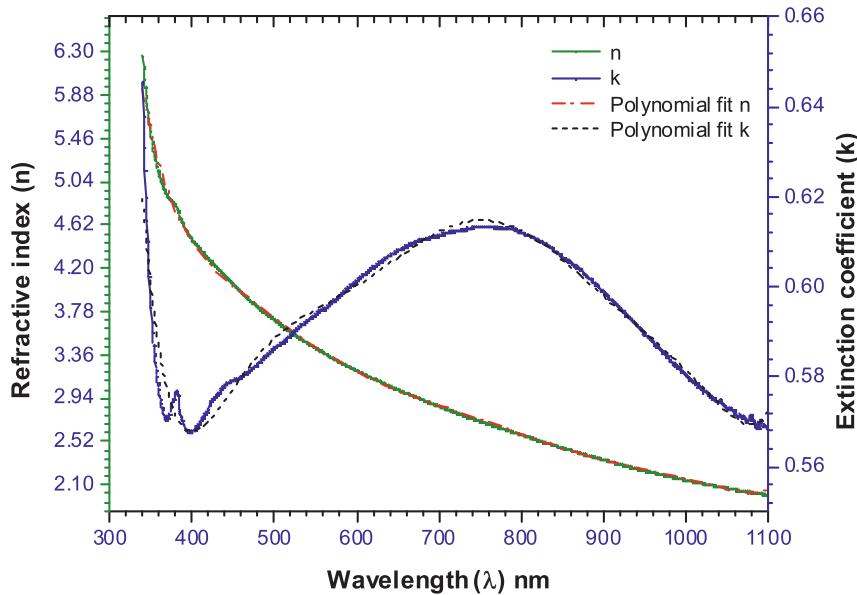


Figure 6: Refractive index and extinction coefficient of the Cu₂Se thin film.

medium/thin film reduces to $(n + ik)^2$ with a real component $\varepsilon_1 = n^2 - k^2$ and imaginary component $\varepsilon_2 = 2nk$. Both of these quantities inflict the property of slowing down the speed of light in thin film with respect to free space. The dielectric constant ε_1 and ε_2 for the deposited Cu₂Se thin film are estimated using the measured quantities n and k under wavelength variation. The estimated real dielectric constant ε_1 and imaginary dielectric constant ε_2 with wavelength variation are shown in Figure 7.

Both the component of dielectric constants has found to decay with wavelength of incident light. Besides this, the real part of ε has also found to be larger than its imaginary part. The similar characteristic of dielectric constant for thin film has been reported in literature [30]. The decay in dielectric constant/refractive index reveals the enhancements of phase velocity/transmittance with respect to increase in wavelength of incident light for the present thin film. The mean value of ε_1 and ε_2 of present Cu₂Se thin film

are found 9.623 and 3.598 for wavelength range 300–1100 nm, respectively. The polynomial curve fit analysis of ε_1 and ε_2 indicates that these physical quantities are eighth order polynomial function of wavelength (ε_1 or $\varepsilon_2 = \sum_{i=0}^8 B_i \lambda^i$). The value of polynomial coefficients and its error are mentioned in Table 2.

3.2.3 Raman measurements

Figure 8, shows Raman spectra of the Cu₂Se thin film. The sample gives various bands centered at 259, 928 and 1115 cm⁻¹. In these spectra, the band at 259 cm⁻¹ is more intense compared to the other bands, which corresponds to Cu-Se vibration. The two other modes of longitudinal optical phonon frequency have been observed at 928 and 1115 cm⁻¹ with relatively weak intensity in the Raman spectra. The other reported works related to this study of Cu₂Se thin films having different thickness have also found

Table 1: Polynomial fit of refractive index and extinction coefficient of the Cu₂Se thin film.

Fit equation →	$n = B_0 + B_1\lambda^1 + B_2\lambda^2 + B_3\lambda^3 + B_4\lambda^4 + B_5\lambda^5 + B_6\lambda^6 + B_7\lambda^7 + B_8\lambda^8$		$k = B_0 + B_1\lambda^1 + B_2\lambda^2 + B_3\lambda^3 + B_4\lambda^4 + B_5\lambda^5 + B_6\lambda^6 + B_7\lambda^7 + B_8\lambda^8$	
Constants ↓	Value	Error	Value	Error
B ₀	6.259×10^{-2}	0.212×10^{-2}	5.179×10^{-1}	0.178×10^{-1}
B ₁	-7.439×10^{-0}	0.271×10^{-0}	-6.220×10^{-1}	0.229×10^{-1}
B ₂	3.842×10^{-2}	0.149×10^{-2}	3.240×10^{-3}	0.125×10^{-3}
B ₃	-1.116×10^{-4}	0.046×10^{-4}	-9.427×10^{-6}	0.383×10^{-6}
B ₄	1.990×10^{-7}	0.085×10^{-7}	1.683×10^{-8}	0.071×10^{-8}
B ₅	-2.233×10^{-10}	0.100×10^{-10}	-1.888×10^{-11}	0.084×10^{-11}
B ₆	1.539×10^{-13}	0.072×10^{-13}	1.300×10^{-14}	0.060×10^{-14}
B ₇	-5.968×10^{-17}	0.290×10^{-17}	-5.029×10^{-18}	0.244×10^{-18}
B ₈	9.964×10^{-21}	0.504×10^{-21}	8.374×10^{-22}	0.425×10^{-22}

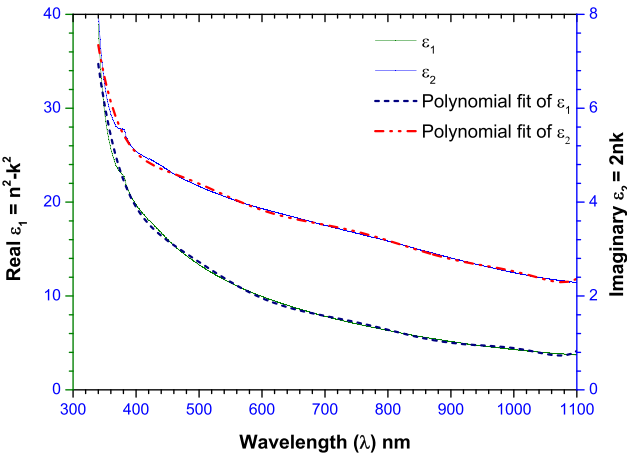


Figure 7: Real and imaginary dielectric constants of the Cu₂Se thin film.

main peak in Raman spectra either at 259 cm⁻¹ or 260 cm⁻¹ or 262 cm⁻¹ [31–33]. Since, the main peak in Raman spectra of Cu₂Se thin film are found approximately same wave-number therefore, Cu-Se bond length and force constant for Cu₂Se will be independent of film thickness.

4 Conclusions

The Cu₂Se thin films were deposited on a glass substrate using thermal evaporation techniques. The XRD pattern of the film verifies the nano-crystalline nature having cubic structure, while SEM micrographs of film confirm the formation of spherical particles in its surface. The EDS measurement shows the presence of Cu and Se elements in the thin film. The optical transmittance is found to increase

Table 2: Polynomial fit of real and imaginary dielectric constants of the Cu₂Se thin film.

Fit equation	$\epsilon_1 = B_0 + B_1\lambda^1 + B_2\lambda^2 + B_3\lambda^3 + B_4\lambda^4 + B_5\lambda^5 + B_6\lambda^6 + B_7\lambda^7 + B_8\lambda^8$		$\epsilon_2 = B_0 + B_1\lambda^1 + B_2\lambda^2 + B_3\lambda^3 + B_4\lambda^4 + B_5\lambda^5 + B_6\lambda^6 + B_7\lambda^7 + B_8\lambda^8$	
→				
Constants ↓	Value	Error	Value	Error
B ₀	7.164×10^{-3}	0.250×10^{-3}	1.325×10^{-3}	0.046×10^{-3}
B ₁	-8.559×10^{-1}	0.320×10^{-1}	-1.593×10^{-1}	0.060×10^{-1}
B ₂	4.419×10^{-1}	0.175×10^{-1}	8.270×10^{-2}	0.329×10^{-2}
B ₃	-1.280×10^{-3}	0.053×10^{-3}	-2.408×10^{-4}	0.100×10^{-4}
B ₄	2.287×10^{-6}	0.100×10^{-6}	4.304×10^{-7}	0.188×10^{-7}
B ₅	2.565×10^{-9}	0.118×10^{-9}	-4.835×10^{-10}	0.221×10^{-10}
B ₆	1.768×10^{-12}	0.085×10^{-12}	3.336×10^{-13}	0.159×10^{-13}
B ₇	-6.856×10^{-16}	0.343×10^{-16}	-1.293×10^{-16}	0.064×10^{-16}
B ₈	1.144×10^{-19}	0.059×10^{-19}	2.161×10^{-20}	0.111×10^{-20}

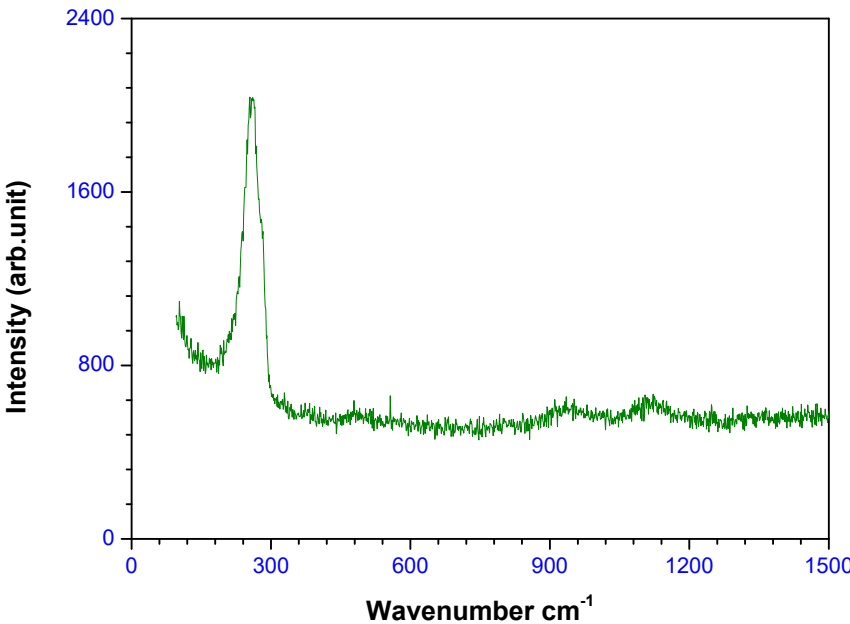


Figure 8: Raman spectra of the Cu₂Se thin film.

with wavelength. The estimated value of optical band gap with optical transmission spectra for the present deposited Cu₂Se film (85 nm thickness) is found 1.90 eV. Since, the wavelength corresponding to 1.9 eV is 653.3 nm which belongs to visible region therefore the present thin film can be used in production of laser light. The mean value of refractive index, extinction coefficient, real and imaginary dielectric constant of present Cu₂Se thin film are found 3.035, 0.594, 9.623, and 3.598, respectively for wavelength range 300–1100 nm. The band at 259 cm⁻¹ in Raman spectra is more intense compared to the other bands, which corresponds to Cu-Se vibration. The present study opens a new base for further study and application of Cu₂Se thin films.

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