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Compositional dependence of the optical properties on amorphous $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ thin films

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ABSTRACT

The effect of silver content on the optical properties of films of $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ (with $x \leq 25$ at.%) chalcogenide glasses has been analysed. The films were prepared by pulsed laser deposition from bulk glass target. Morphology, structure and optical properties of the films were characterized by profilometry, X-ray diffraction and UV–visible–NIR transmission spectroscopy (wavelength 400–2500 nm). Refractive index, absorption coefficient, and the optical band gap energy were determined. It was found that the refractive index increases (for every wavelength) whereas the optical band gap energy decreases as the silver content increases.

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1. Introduction

Chalcogenide based systems are easy glass formers which can be synthesized in large composition domains either in bulk or as thin films. These glasses are usually semiconductors with their gap energy depending upon their composition [1] and become ionic conductors when alkali metals, Ag or Cu are added [2]. Due to the absence of long-range order, variations in the glass composition generate a gradual change in their optical properties, making it suitable for a broad range of technological applications. Hence, the study of the dependence on composition of their optical properties is of great importance to improve the understanding of the mechanisms underlying these phenomena as well as to improve their applications in technological devices [3].

$\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ bulk glasses behave as ionic conductors for $8 < x < 25$ at.%, whereas they are semiconductors for $x < 8$ at.% [4]. Electric transport behaviors are related to the glass morphology [5]. Structural investigations of these glasses have been done using X-ray and neutron diffraction [6,7].

In this paper, the optical characterization of $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ films ($0 \leq x \leq 25$) deposited on glass substrate is presented. The refractive index and the optical band gap energy were determined from only the transmission spectra and the results were linked with the properties of the glasses. Usually, the processing of the optical measurements is based on the work of Swanepoel [8] which assumes that the substrate is completely transparent. In our case, the substrate is weakly

absorbing so we incorporate a correction based on Gonzalez Leal et al. [9].

2. Experimental

Thin films of compositions $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ (with $x = 0, 7, 10, 20$ and 25 at.%) were deposited using the pulsed laser deposition (PLD) process (on/axis geometry) using a pulsed Nd:YAG laser (Quanta-Ray Spectra-Physics), operating at a wavelength of 355 nm with a pulse duration of 5 ns. The pulse repetition rate was 10 Hz, the incidence angle was 45° and the laser fluence $1.2\text{--}2 \text{ J/cm}^2$. The laser beam was scanned horizontally by the movement of a mirror in order to get a uniform ablation of the target. Before the irradiation, the deposition chamber was evacuated down to approximately 10^{-6} mbar to avoid ambient contamination. The chalcogenide films were deposited on static substrates (microscope slides) held at room temperature. Bulk chalcogenide glass of the appropriate composition was used as targets, which were synthesized by the usual melt quenching technique [6]. In a previous work, it was found that film's compositions of the Ag–Ge–Se system differ from their target's compositions in about ± 3 at.% [10].

The amorphous nature of thin films was confirmed by X-ray diffraction (XRD) using a D/max Rigaku diffraction system and a monochromatized Cu ($K\alpha$) radiation.

The films thicknesses were measured using a Dektak 3 Veeco profilometer.

The optical transmittance spectra were recorded using a UV–visible–IR spectrophotometer (Cary 5000 from Varian) in the wavelength λ range 400–2500 nm, through a circular mask (diameter $\phi = 1$ mm). Optical absorbance (x), optical band gap energy (E_{opt}) and refractive index (n) of the films were obtained from the transmittance spectra.

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The glass substrates were characterized with a Perkin Elmer spectrophotometer model Lambda 19 with reflectance accessories.

3. Results

The XRD patterns of the films present a notable pre-peak at a diffraction angle $2\theta \approx 14^\circ$ ($q = 4\pi \sin\theta / \lambda \approx 1 \text{ \AA}^{-1}$) that decreases with increasing Ag content. This result was also observed for $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ bulk glasses (with $x=0, 10, 15, 20$ and 25 at.%) [6,11]. The pre-peak is characteristic of amorphous chalcogenide glasses and is associated with medium-range order [12].

3.1. Optical characterization

A typical transmittance spectrum of the stacked structure “film-substrate” for the $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ films on microscope slides are shown in Fig. 1. Typical interference pattern with maxima and minima points is observed in the region of medium and weak absorption. The transmission curve $T(\lambda)$ is delimited by two envelopes $T_+(\lambda)$ and $T_-(\lambda)$ which are first determined using an interpolation function of the maximum and minimum values, T_M and T_m , of the transmission curve and then corrected so that the envelopes are truly tangential to the transmission curve (i.e. they do not intersect it).

3.2. Refractive index and optical absorbance

The refractive index n and the optical absorbance x of the films as a function of λ were calculated from the transmission spectra $T(\lambda)$ of the stacked structure “film-substrate”. The procedure described by Gonzalez-Leal et al. [9] was used. These authors found that this procedure improves the results found with the widely used procedure of Swanepoel [8]. For the case of the refractive index determination, the method was applied in the region of medium and weak absorptions where the interference maxima and minima can be accurately determined.

The method proposed by Gonzalez-Leal et al. is based on the following assumptions [9]:

- An isotropic dielectric thin film covers a weakly absorbing substrate and this optical system is immersed in air.
- The refractive index of the thin film n is larger than the refractive index of the substrate s , i.e. $n > s$.

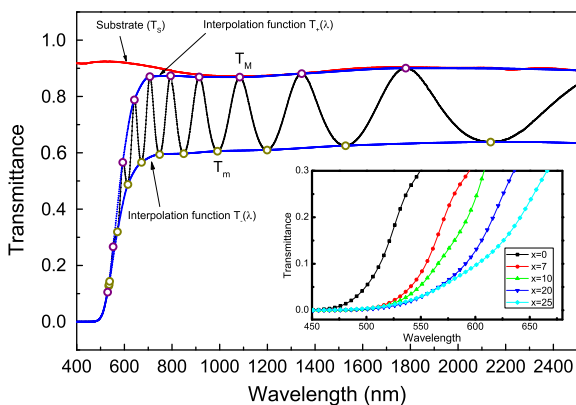


Fig. 1. Typical transmission spectrum $T(\lambda)$ of the stacked structure “film-substrate” (black curve). $T_M(\lambda)$ and $T_m(\lambda)$ functions (blue curves) are determined using an interpolation function of the maximum and minimum values (T_M and T_m). T_s is the transmission of the substrate (red curve). Inset: $T(\lambda)$ in the strong absorption region for the different $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ thin films (with $0 \leq x \leq 25$).

- The film and the substrate are both weakly absorbing, i.e. $n^2 \gg k^2$ and $s^2 \gg k_s^2$ where k and k_s are the extinction coefficients of the film and the substrate respectively.

With the previous conditions, the analytical expression for the transmittance of the structure “films-substrate” is given by Eq. (1).

$$T(\lambda, n, x, t, s, x_s) = \frac{A}{B} \quad (1)$$

$$\begin{aligned} A &= (1-R_1)(1-R_2)(1-R_3)x x_s \\ B &= 1 + R_1 R_2 x^2 - R_1 R_3 x^2 x_s^2 - R_2 R_3 x_s^2 + 2r_1 r_2 (1-R_3 x_s^2) x \cos(\varphi), \end{aligned} \quad (2)$$

with

$$\begin{aligned} R_1 &= r_1^2, \quad R_2 = r_2^2, \quad R_3 = r_3^2, \quad r_1 = \frac{1-n}{1+n}, \quad r_2 = \frac{n-s}{n+s}, \quad r_3 = \frac{s-1}{s+1} \\ \alpha &= \frac{4\pi k}{\lambda}, \quad x = \exp(-\alpha t), \\ \alpha_s &= \frac{4\pi k_s}{\lambda}, \quad x_s = \exp(-\alpha_s t_s), \\ \varphi &= \frac{4\pi n t}{\lambda}. \end{aligned} \quad (3)$$

Where t and t_s are the thicknesses, α and α_s are the absorption coefficients, x and x_s the optical absorbances, of the film and the substrate, respectively.

In Eqs. (1)–(3) the refractive index s and optical absorbance x_s of the substrate were determined experimentally with the method proposed by Gonzalez-Leal et al. [9] using transmittance and reflectance spectra. The results show that s can be considered constant and equal to 1.51 while x_s has a slight dependence on wavelength due to spectral regions of local absorption.

The analytical expression for the upper and lower envelopes $T_+(\lambda)$ and $T_-(\lambda)$ are derived from Eqs. (1)–(3) by substitution of $\cos(\varphi) = \pm 1$:

$$\begin{aligned} T_{\pm}(\lambda; n, x, t, s, x_s) &= \frac{A}{B_{\pm}} \\ B_{\pm} &= 1 + R_1 R_2 x^2 - R_1 R_3 x^2 x_s^2 - R_2 R_3 x_s^2 \pm 2r_1 r_2 (1-R_3 x_s^2) x. \end{aligned} \quad (4)$$

The method is based in comparing Eq. (4) with the experimental data for every wavelength of the studied spectra. The refractive index n and the optical absorbance x of the film are determined by solving the system of Eq. (5).

$$\begin{cases} T_M(\lambda) - T_+(\lambda; n, x) = 0 \\ T_m(\lambda) - T_-(\lambda; n, x) = 0 \end{cases} \quad (5)$$

Where T_M and T_m are the envelopes interpolated from the experimental data and the functions $T_+(\lambda)$ and $T_-(\lambda)$ are given by Eq. (4).

However, if the system of Eq. (5) is solved only for wavelengths where the transmission spectra is tangential to its envelopes λ_{tan} , the accuracy in the determination of n can be improved by means of the graphical method proposed by Swanepoel [8]. The application of this graphic method allows the obtaining of an additional result: the thickness of the film t . The method is based in the basic equation for interference fringes:

$$2nt = m\lambda. \quad (6)$$

Eq. (6) can be written for λ_{tan} as:

$$\frac{l}{2} = 2t \frac{n}{\lambda_{tan}} - m_1 \quad (7)$$

where $l=0, 1, 2$, and 3 , and m_1 is the order number of the extreme near λ_{tan} at the largest wavelength (integer if that extreme is a maximum and half-integer if it is a minimum) [8].

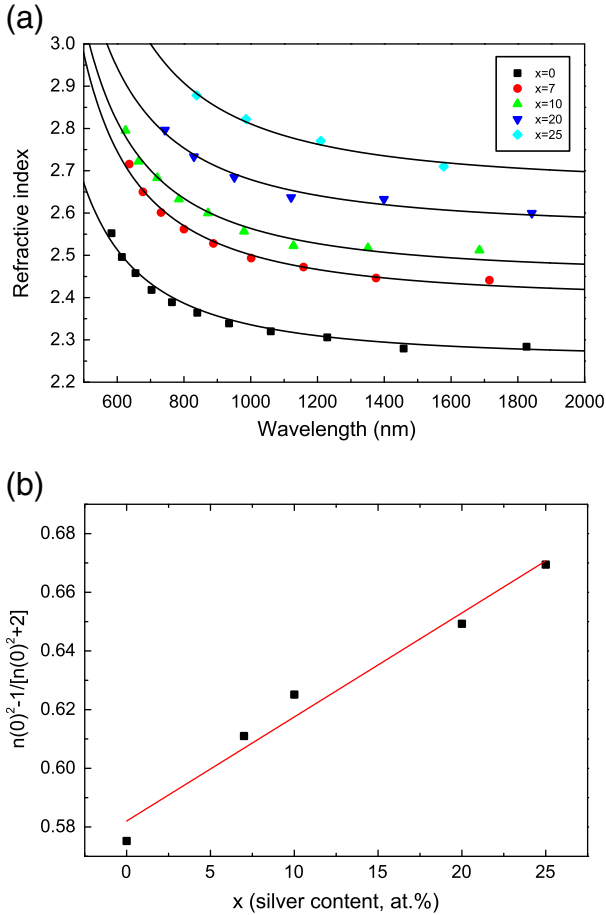


Fig. 2. (a) Dependence of the refractive index n with λ obtained for $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ thin films (with $0 \leq x \leq 25$). The line was computed using the Wemple and Di Domenico model (Eq. (8)). (b): Dependence of $\frac{n(0)^2-1}{n(0)^2+2}$ with the silver content x , $n(0)$ is the refractive index at infinite wavelength (see Table 1).

The calculated values of n vs λ for the amorphous $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ films with different compositions is shown in Fig. 2a. The refraction index increases as the Ag content increases (see n reported for $\lambda = 1.0 \mu\text{m}$ in Table 1).

As expected from Eq. (7), these curves could be fitted by straight lines from which the film thickness was calculated. The agreement between the calculated thicknesses (t_0) and the experimental thicknesses measured by profilometry (t_1) are shown in Table 1.

The behavior of the refractive index with the wavelength was explained using the model of single oscillator proposed by Wemple and Di Domenico [13,14] in Eq. (8).

$$n^2(E(\lambda)) = 1 + \frac{E_0 E_d}{E_0^2 - (E(\lambda))^2} \quad (8)$$

Table 1
Optical properties of $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ thin film glasses: $n(1 \mu\text{m})$ and $n(0)$ are the refractive index at wavelength $\lambda = 1 \mu\text{m}$ and infinity, respectively, E_0 and E_d are the parameters of the Wemple and Di Domenico model (Eq. (8)), E_{opt} and $\beta^{1/2}$ are the parameters of Tauc's law (Eq. (9)), and, t_1 and t_0 are the film thicknesses determined by profilometry and the method proposed by Swanepoel [8] (Eq. (6)), respectively.

x	$n(1 \mu\text{m})$ ± 0.05	$n(0)$ ± 0.05	E_0 (eV)	E_d (eV)	E_{opt} (eV)	$\beta^{1/2}$ ($\text{cm}^{-1/2} \text{eV}^{-1/2}$)	t_1 (nm)	t_0 (nm)
0	2.33	2.25	4.3 ± 0.2	17 ± 1	2.06 ± 0.02	700 ± 40	780 ± 40	799 ± 9
7	2.49	2.39	3.9 ± 0.1	19 ± 1	1.88 ± 0.01	720 ± 20	730 ± 40	703 ± 7
10	2.55	2.45	3.9 ± 0.2	20 ± 1	1.79 ± 0.02	660 ± 20	700 ± 40	670 ± 20
20	2.67	2.56	3.9 ± 0.4	22 ± 3	1.64 ± 0.02	600 ± 20	560 ± 30	530 ± 20
25	2.82	2.66	3.6 ± 0.6	22 ± 5	1.55 ± 0.04	570 ± 30	475 ± 30	440 ± 20

where $E(\lambda)$ is the photon energy, E_0 is the single-oscillator energy and E_d is the dispersion energy. Fig. 2 shows a very good agreement between the Wemple and Di Domenico model (solid lines) and the refractive index data (symbols). The values of $n(0) = n(E = 0)$ (that is, n at infinite wavelength) are shown in Table 1.

3.3. Absorption coefficient and optical band gap

The optical band gap energy E_{opt} was determined from the values of the absorption coefficient α in the strong absorption region ($\alpha > 10^4 \text{ cm}^{-1}$) using Tauc's procedure [15]. In the strong absorption region, when the interference fringes disappear, the curves $T_M(\lambda)$ and $T_m(\lambda)$ converge to a single curve. The absorbance and the absorption coefficient are determined using Eqs. (3) and (5).

According to Wood and Tauc [15], the strong absorption region of the transmission spectra of amorphous semiconductors corresponds to transitions between states in the valence band and states in the conduction band through the optical band gap energy, E_{opt} . These absorption coefficients are governed by Tauc's law:

$$\alpha(\lambda) = \beta \frac{[E(\lambda) - E_{opt}]^M}{E(\lambda)} \quad (9)$$

where β is a constant that depends on the transition probability, $E(\lambda)$ is the photon energy and M is a parameter characteristic of the transition process. In the case of chalcogenide glasses, usually $M = 2$, which is characteristic of indirect transitions [15].

As shown in Fig. 3, the plots of $\sqrt{\alpha(\lambda)E(\lambda)}$ vs $E(\lambda)$ are linear and correspond to $M = 2$. The values of E_{opt} were extracted from the plots as the energy values where the straight lines cross the abscissa axis.

Fig. 3b shows the dependence of the optical band gap energy E_{opt} with the composition of the films. This graph shows that E_{opt} decreases with the increase of Ag content approximately as a linear function.

4. Discussion

Refractive index increases with the increase of the Ag content. The same behavior is observed for thin films of similar compositions: $\text{Ag}_x(\text{Ge}_{0.20}\text{Se}_{0.80})_{100-x}$ (with $x \leq 23$ at.%) [16] and $\text{Ag}_x\text{Ge}_{30-x}\text{Se}_{70}$ (with $x \leq 30$ at.%) [17]. This fact may point out that silver atoms have a higher polarizability than Ge and Se atoms [16,18]. Polarizability ϕ and refractive index are related through the Lorentz-Lorentz equation [18]:

$$\frac{n^2 - 1}{n^2 + 2} = \frac{1}{3\epsilon_0} \sum_j N_j \phi_j \quad (10)$$

with ϵ_0 , the vacuum permittivity and N_j , the number polarized atoms of the type j by mole. Considering the stoichiometry of our alloys, Eq. (10) can be written as:

$$\frac{n^2 - 1}{n^2 + 2} = \frac{1}{3\epsilon_0} \left\{ N \left[\frac{3\phi_{Se} + \phi_{Ge}}{4} + \frac{x}{100} \left(\phi_{Ag} - \frac{3\phi_{Se} + \phi_{Ge}}{4} \right) \right] \right\}. \quad (11)$$

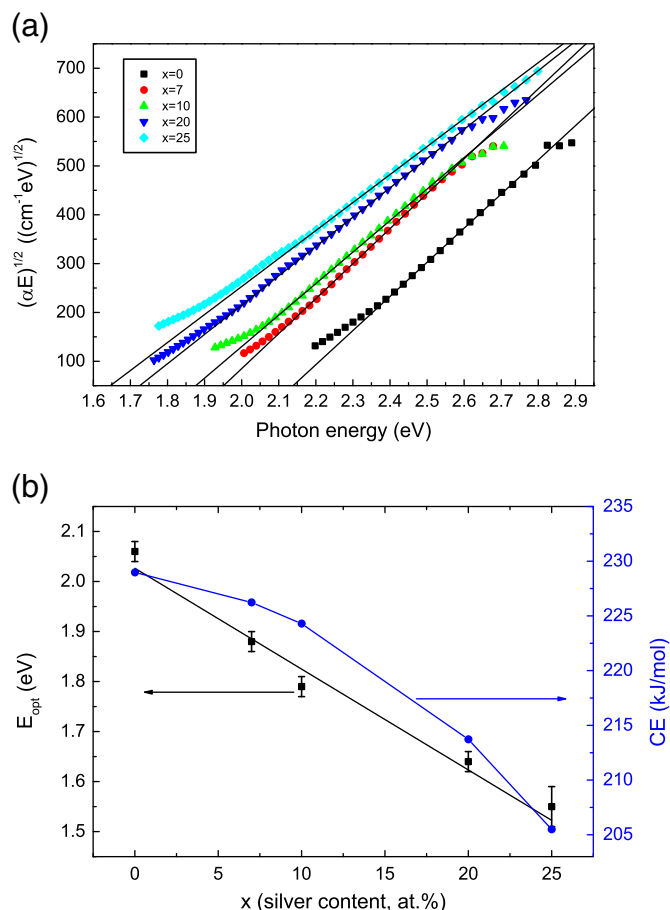


Fig. 3. (a) Plot of $\sqrt{\alpha(\lambda)E(\lambda)}$ versus $E(\lambda)$ obtained using the transmission spectrum of $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ thin films (with $0 \leq x \leq 25$). (b) Dependence of the optical band gap energy E_{opt} and the cohesive energy CE with the Ag content x .

Assuming that ϕ_{Se} , ϕ_{Ge} and ϕ_{Ag} are constant with the change in x (Ag at.%), Eq. (11) shows a lineal dependence with the silver content. Fig. 2b shows the dependence of $\frac{n(0)^2-1}{n(0)^2+2}$ with x , indicating a good agreement between our data and a linear function (see $n(0)$ in Table 1).

The bandgap energy decreases with increasing silver concentration. Other authors found the same behavior for thin films of similar compositions: $\text{Ag}_x(\text{Ge}_{0.20}\text{Se}_{0.80})_{100-x}$ (with $x \leq 23$ at.%) [16] and $\text{Ag}_x\text{Ge}_{30-x}\text{Se}_{70}$ (with $x \leq 30$ at.%) [17]. However, there is controversy in this point in the literature. The inverse trend is detected in other previous works although in one case the samples compositions are not the same: $\text{Ge}_x\text{Se}_y\text{Ag}_{100-x-y}$ with $x = 15$ and $y = 65, 75$, and 85 [19] and in others cases, the optical parameters are obtained from ab initio simulation [20,21]. In our case, the dependence of E_{opt} with the silver content is corroborated experimentally by the transmittance T in the strong absorption region as shown in the inset of Fig. 1. There, it can be observed that the absorption edge is shifted to higher wavelengths as the silver concentration increases. This is consistent with the trend indicated above for the bandgap energy.

The dependence of E_{opt} with x has to be analyzed in terms of the electronic bands structure. In the chalcogenide glasses, the valence band arises from the unshared electron pair (lone pair, LP) of the group-VI atom. The bonding (σ) and antibonding (σ^*) bands are split symmetrically with respect to this LP band energy. The bonding (σ) and LP bands are occupied whereas the antibonding states (σ^*) constitute the conduction band [22]. From a chemical approach, an increase in the bond strengths causes a larger splitting between σ and σ^* bands which results in an increase in E_{opt} [22].

It is known that the optical band gap in the glass is related to the nature of the chemical bond, the change in the bond energy and the distribution of different bond types. Therefore, the behavior of E_{opt} with the silver content can be explained by the different binding energies of Ge–Se and Ag–Se bonds (binding energy of Ge–Se and Ag–Se bonds are 230 kJ/mol [23] and 202 kJ/mol [24], respectively). The lower binding energy of Ag–Se bonds causes a smaller splitting of valence and conduction bands and results in a smaller E_{opt} value [16].

This idea is corroborated by determining the cohesive energy (CE), in other words, variation of E_{opt} may be interpreted in terms of change in CE. The cohesive energies of the investigated compositions were calculated using the method suggested by the chemical bond approach [3]. This model suggests the prevailing formation of heteropolar bonds (Ge–Se, Ag–Se) over homopolar bonds (Se–Se, Ag–Ag). We assumed:

- Ge is bonded to Se in a $\text{GeSe}_{4/2}$ tetrahedral coordination [6,7].
- For the chemical bond of silver, one should consider that $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ alloys are silver ionic conductors for $x \geq 8$ at.%. Consequently, in ionic conductor samples, a fraction of the silver atoms is free and the other fraction is chemically bonded. In a previous work [6], we found that a third of silver atom is bonded to Se in a $\text{AgSe}_{4/2}$ coordination for Ag ionic conductor samples ($x = 10, 20$ and 25 at.%). The remaining Ag atoms are free in the form of Ag^+ cations and we consider that its contribution to the cohesive energy is negligible. We assumed that all silver atom is bonded to Se in a $\text{AgSe}_{4/2}$ coordination in the glass with low conductivity ($x = 7$).
- The remaining Se is in Se_n chains. The binding energy of the Se–Se bond was estimated in 227 kJ/mol.

Fig. 3b shows the dependence of CE with the composition of the thin films. We can observe that E_{opt} and CE show similar dependence with silver content. This decrease of CE with x tends to decrease the energy of the conduction band edge [3], causing a lower splitting between σ and σ^* band and thus resulting in the observed decrease in the band gap energy.

5. Conclusions

The effect of silver content on the optical properties of $\text{Ag}_x(\text{Ge}_{0.25}\text{Se}_{0.75})_{100-x}$ films was analyzed in the wavelength range 400–2500 nm. The refractive index was found to increase with the increase of the Ag content. This may be explained by considering that silver atoms have a higher polarizability. The behavior of the refractive index with the wavelength was explained using the model of single oscillator proposed by Wemple and Di Domenico [13,14]. Band gap energy and cohesive energy decrease with increasing silver concentration. The lower binding energy of Ag–Se bonds causes a smaller splitting of valence and conduction bands and results in a smaller E_{opt} value.

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