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Calculation of the Optical Constants of Amorphous Semiconducting $As_{40}s_{60}$, $As_{40}s_{35}se_{25}$ and $As_{40}se_{60}$ Thin Films from Transmittance and Reflectance Measurements

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Abstract: Compositional dependencies of the optical properties of as-deposited amorphous $As_{40}S_{60}$, $As_{40}S_{35}Se_{25}$ and $As_{40}Se_{60}$ prepared by thermal evaporation, have been studied. The direct analysis proposed by Swanepoel has been successfully employed and it has allowed us to determine the average thickness $\bar{d}$ and the refractive index, n, of the films, with accuracies approximately 1%. The refractive index, n and film average thickness $\bar{d}$ has been determined from the upper and lower envelopes of the transmission spectra measured at normal incidence, in the spectral range from 400 to 2500 nm. The absorption coefficient α, therefore extinction coefficient k, have been determined from both the transmission spectra and reflection spectra at the strong absorption region. The values of refractive index increase towards $As_{40}Se_{60}$ over the entire spectral range studied increase is related to the increased polarizability of the larger Se atoms atomic radius, 115 pm), in comparison with S atoms (atomic radius, 100 pm). The dispersion of the refractive index is discussed in terms of the single-oscillator Wemple-DiDomenico model and the optical absorption edge is described using the non-direct transition model proposed by Tauc. Likewise, the optical energy gap is derived from Tauc's extrapolation. The optical band gap in three different composition thin films of $As_{40}S_{60}$, $As_{40}S_{35}Se_{25}$ and $As_{40}Se_{60}$ chalcogenide glass are 2.37, 2.06 and 1.81 eV, respectively. The decrease has been obtained in both single-oscillator energy E_o and optical E_g^{opt} gap were interpreted in terms of the bond-strengths of the chemical bonds present in the glass compositions under study.

Key words: Thin films, spectral range, transmission, reflection, refractive index, optical absorption edge

INTRODUCTION

The optical properties of chalcogenide glasses, for example excellent transmittance in the infra-red region, continuous shift of the optical absorption edge and values of refractive index ranging between 2.0 and 3.5, as well as the strong correlation between the former properties and the chemical composition, explain the growing interest in these semiconducting materials for the manufacture of filters, anti-reflection coatings and, in general, a wide range of optical devices[1-4]. This underlines the importance of the characterization of these glassy materials through the determination of their optical constants, refractive index and extinction coefficient, as well as the corresponding optical band gaps. Great efforts have been made to develop the mathematical formulation describing the transmittance and reflectance of different optical systems[5-12].

This study will use the straightforward method proposed by Swanepoel[6], which is based on the use of the extremes of the interference fringes of transmission spectrum for calculation the refractive index and film thickness in both weak absorption region and transparent region and will use transmission spectrum and reflection spectrum for calculation the extinction coefficient in the strong absorption region. As to the method for determining the refractive index of thin films, we must consider that, if the optical thickness of the film is sufficient to generate several interference extremes and also the higher thickness helps us to avoid us the effect of film thickness on the optical constant of thin films.

MATERIALS AND METHODS

Bulk chalcogenide samples were prepared according to the conventional melt-quenched technique. The high-purity elements were weighted and placed together in a precleaned and out gassed silica ampoule, which has been evacuated to a pressure of about 10^{-3} Pa and then, sealed. The synthesis was performed in a rocking furnace at a temperature of approximately 900°C, for about 24 h. After the synthesis, the melt was cooling water-quenched, resulting in a bulk glass of the required chemical composition, $As_{40}S_{60}$, $As_{40}S_{35}Se_{25}$ and $As_{40}Se_{60}$. The glass thin films were deposited by evaporating the bulk chalcogenide samples from a resistance heating quartz glass crucible onto clean glass substrates kept at room temperature, using a conventional coating unit (Denton

Vacuum DV 502 A) and a vacuum of about 2x 10^{-6} Torr. The evaporation rate as well as the film thickness was controlled using a quartz crystal DTM 100 monitor. The mechanical rotation of the substrate holder (≈ 30 rpm) during deposition produced homogeneous film. The temperature rise of the substrate due to radiant heating from crucible was negligible. Small fluctuations in the measured transmittance (≈ 1.0 %) of studied films confirm their homogeneity. The amorphous state of the as-deposited films was checked using Philips X-ray diffractometry (1710).

The measurements of transmittance and reflectance were carried out using a double-beam (Shimadzu UV-2101 combined with PC) computer-controlled spectrophotometer, at normal incidence of light and in a wavelength range between 400 and 2500 nm. Without glass substrate in the reference beam, the measured transmittance spectra were used to calculate the refractive index and the average film thickness of three different glass compositions $As_{40}S_{60}$, $As_{40}S_{35}Se_{25}$ and $As_{40}Se_{60}$ thin films.

Preliminary theoretical considerations: Consider the optical system consists of chalcognide glass thin films evaporated onto thick, finite, transparent substrates. The homogeneous film has thickness d and complex refractive index n = n − ik, where, n is the refractive index and k the extinction coefficient, which can be expressed in terms of the absorption coefficient α by the equation: $k = \alpha\lambda/4\pi$. The thickness of the substrate is several orders of magnitude larger than d and its index of refraction is s. The system is surrounded by air with refractive index $n_0 = 1$. Taking all the multiple reflections at the three interfaces into account, it can be shown that in the case $k^2 << n^2$ the expression for the transmittance T for normal incidence is given by[6,8,13].

$$T(\lambda,s,n,d,k)\big|_{k=0} = \frac{A\chi_\alpha}{B - C\chi_a\cos\varphi + D\chi_a^2} \qquad (1)$$

Where, $A = 16n^2s$, $B = (n+1)^3(n+s^2)$, $C = 2(n^2-1)(n^2-s^2)$, $D = (n-1)^3(n-s^2)$, $\varphi = 4\pi nd/\lambda$ and $\chi_a(\lambda)$, the absorbance, is given by the formula $\chi_a = \exp(-\alpha d)$.

Moreover, the values of the transmission at the extremes of the interference fringes can be obtained from Eq. 1 by setting the interference condition cos φ = +l for maxima and cos φ = −1 for minima. From these two new formulae, many of the equations that provide the basis of the method in use are easily derived[8].

RESULTS AND DISCUSSION

Calculation of the refractive index and film thickness: According to Swanepoel's method, which is based on the idea of Manifacier *et al.*[5] of creating the envelopes of interference maxima and minima (Fig. 1), a first, approximate value of the refractive index of the film n_1, in the spectral region of medium and weak absorption, can be calculated by the expression:

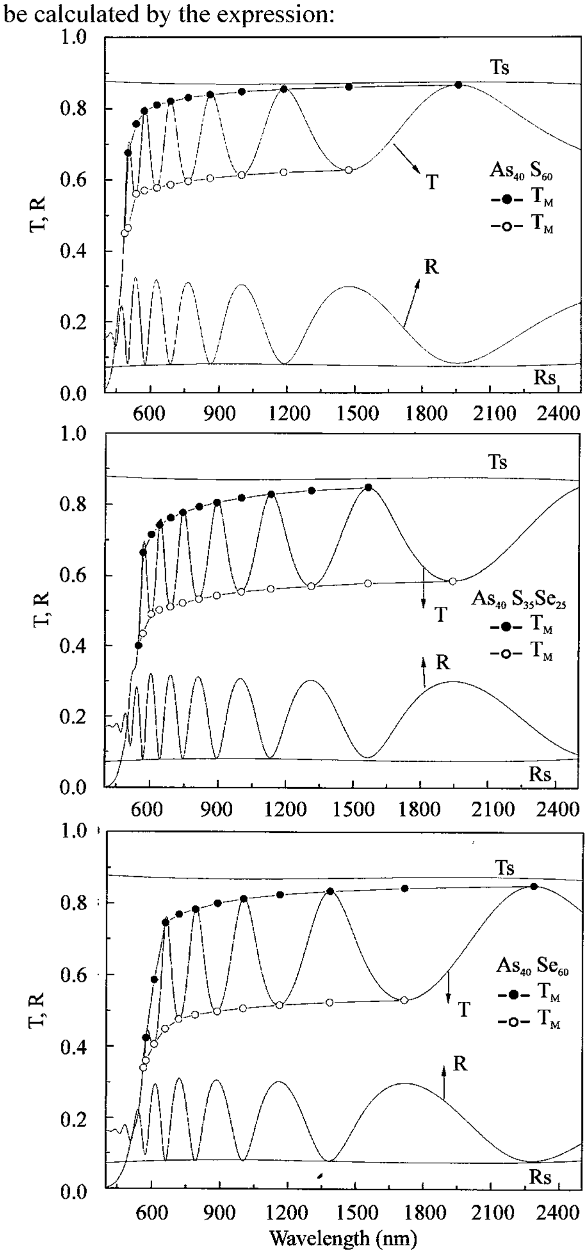

Fig. 1: Three typical transmission T(λ) and reflection R(λ) spectrum for three different composition thin films of $As_{40}S_{60}$, $As_{40}S_{35}Se_{25}$ and $As_{40}Se_{60}$, T_M and T_m according to the text. Ts(λ) and Rs(λ) are the transmission and reflection of uncoated substrate

Table 1: Values of λ, T_M and T_m for the three different composition thin films of amorphous As−S corresponding to transmission spectra of Fig. 1; the underline values of transmittance are the values calculated by orgin program. The calculated values of refractive index and film thickness are based on the envelope method

Sample	λ	T_M	T_m	s	n_1	d_1(nm)	m_0	m	d_2(nm)	n^2
$As_{40}S_{60}$	538	0.7569	0.5608	-	-	-	-	-	-	-
	574	0.793	0.5696	1.527	2.442		5.617	5.5	646.5	2.5
	628	0.8104	0.5767	1.531	2.452	707.9	5.157	5	640.2	2.487
	688	0.8207	0.5857	1.534	2.441	677.6	4.685	4.5	634.3	2.452
	766	0.8308	0.5954	1.537	2.426	667.5	4.183	4	631.5	2.426
	862	0.8392	0.604	1.539	2.412	648.9	3.695	3.5	625.4	2.389
	1000	0.8479	0.6129	1.54	2.397	631.3	3.165	3	625.9	2.376
	1186	0.8553	0.6209	1.538	2.379	628.6	2.649	2.5	623	2.348
	1474	0.8614	0.6273	1.53	2.36		2.115	2	624.5	2.335
	$\bar{d}_1$ = 660.3 nm		σ_1 = 30 nm (4.6 %)		$\bar{d}_2$ = 631.4 nm		σ_2 = 8.4 nm (1.3 %)			
$As_{40}S_{35}Se_{25}$	568	0.6639	0.4347	-	-	-	-	-	-	-
	604	0.7147	0.4903	1.529	2.63		7.092	7	803.8	2.702
	640	0.7417	0.5026	1.531	2.633	954.4	6.702	6.5	789.8	2.658
	688	0.762	0.512	1.534	2.635	855.9	6.239	6	783.2	2.638
	742	0.7766	0.5225	1.536	2.62	791.5	5.751	5.5	778.9	2.608
	814	0.7925	0.5338	1.538	2.604	793.4	5.21	5	781.6	2.601
	892	0.8053	0.5438	1.54	2.587	791.5	4.724	4.5	775.8	2.565
	1000	0.8177	0.5544	1.54	2.567	771.8	4.181	4	779.1	2.556
	1132	0.8284	0.5632	1.539	2.55	785.2	3.669	3.5	776.9	2.532
	1312	0.8391	0.5715	1.535	2.532	771.6	3.144	3	777.1	2.515
	1564	0.8481	0.5796	1.527	2.51		2.614	2.5	779	2.498
	$\bar{d}_1$ = 814.4 nm		σ_1 = 62.5 nm (7.7 %)		$\bar{d}_2$ = 782.5 nm		σ_2 = 8.5 nm (1.1 %)			
$As_{40}Se_{60}$	610	0.5853	0.4056	-	-	-	-	-	-	-
	658	0.7447	0.4488	1.532	2.915		5.513	5.5	620.7	2.853
	718	0.7691	0.4761	1.535	2.824	559.7	4.894	5	635.7	2.831
	790	0.7833	0.4882	1.538	2.794	631	4.401	4.5	636.2	2.803
	886	0.7998	0.4975	1.539	2.782	646.7	3.908	4	636.8	2.794
	1000	0.8132	0.5069	1.54	2.764	639.3	3.439	3.5	633.2	2.76
	1162	0.825	0.5163	1.538	2.74	622.6	2.935	3	636	2.749
	1384	0.8349	0.525	1.533	2.714	633.8	2.44	2.5	637.5	2.728
	1714	0.8436	0.5316	1.523	2.69		1.953	2	637.1	2.703
	$\bar{d}_1$ = 622.2 nm		σ_1 = 31.7 nm (5.1 %)		$\bar{d}_2$ = 634.2 nm		σ_2 = 5.6 nm (0.9 %)			

$$n_1 = \left[N_1 + (N_1^2 - s^2)^{\frac{1}{2}} \right]^{\frac{1}{2}} \quad (2)$$

Where:

$$N_1 = 2s\frac{T_M - T_m}{T_M T_m} + \frac{s^2+1}{2}$$

Here, T_M and T_m, are the transmission maximum and the corresponding minimum at a certain wavelength λ. Alternatively, one of these values is an experimental interference extreme and the other one is derived from the corresponding envelope; both envelopes were computer-generated using the origin version 7 program using more than one procedure. On the other hand, the necessary values of the refractive index of the substrate are obtained from the transmission spectrum of the substrate, T_s using the well-known equation[14]:

$$s = \frac{1}{T_s} + (\frac{1}{T_s} - 1)^{\frac{1}{2}} \quad (3)$$

The values of the refractive index n, as calculated from Eq. 2 are shown in Table 1. The accuracy of this initial estimation of the refractive index is improved after calculating d, as will be explained below. Now, it is necessary to take into account the basic equation for interference fringes

$$2d = m\lambda \quad (4)$$

Where, the order numbers m is integer for maxima and half integer for minima. Moreover, if n_{e1} and n_{e2} are the refractive indices at two adjacent maxima (or minima) at λ_1 and λ_2, it follows that the film thickness is given by the expression:

$$d = \frac{\lambda_1\lambda_2}{2(\lambda_1 n_{e2} - \lambda_2 n_{e1})} \quad (5)$$

The values of thickness d of different composition samples determined by this equation are listed as d_1, in Table 1. The last values usually deviate considerably from the other values and must consequently be rejected. The average value of d_1, corresponding to the three films of different composition are listed in Table 1. This value can now be used, along with n_1, to calculate the order number

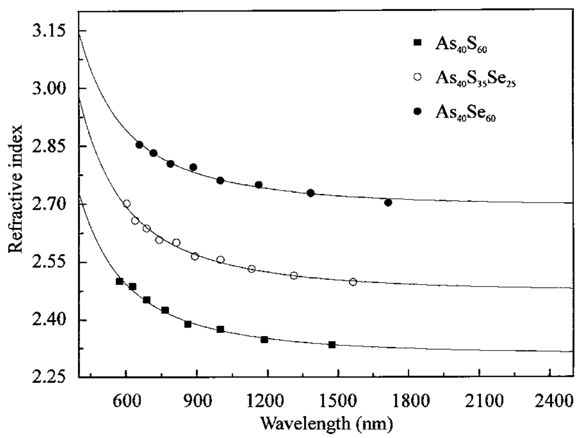

Fig. 2: Refractive index dispersion spectra for the three different composition thin films

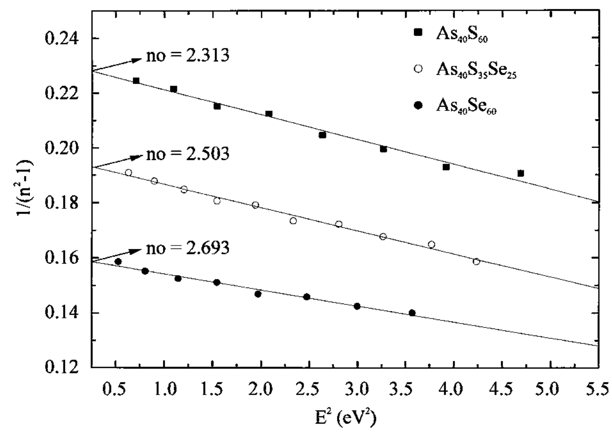

Fig. 3: Plot of refractive index factor $(n^2-1)^{-1}$ versus E^2 for spectra for the three different composition thin films

m_0 for the different extremes using Eq. 4. The accuracy of d can now be significantly increased by taking the corresponding exact integer or half integer values of m associated to each extreme (Fig. 1) and deriving a new thickness, d_2 from Eq. 4, again using the values of n_1. The values of d_2 found in this way have a smaller dispersion ($\sigma_1 > \sigma_2$). It should be emphasized that the accuracy of the final thickness is approximately 1% (Table 1). With the exact value of m and the very accurate value of Eq. 3 can then be solved for n at each λ and thus, the final values of the refractive index n_2 are obtained (Table 1). Figure 2 shows the dependence of n on wavelength for three different composition thin films of $As_{40}S_{60}$, $As_{40}S_{35}Se_{25}$ and $As_{40}Se_{60}$ chalcogenide glass thin films. This figure illustrates that the change in the n values was related to the change in the concentration of S at the expense of Se content, i.e., the values of n increase towards $As_{40}Se_{60}$ over the entire spectral range studied, Ramirez-Mato *et al.*[20] and González-Leal *et al.*[22] have observed similar dependence on As-S, As-S-Se and As-Se cholegoenide glass system. This increase is related to the increased polarizability of the larger Se atoms atomic radius, 115 pm), in comparison with S atoms (atomic radius, 100 pm).

Now, the values of n_2 can be fitted to a reasonable function such as the two-term Cauchy dispersion relationship, $n(\lambda) = a + b/\lambda^2$, which can be used for extrapolation the whole wavelengths[15] (Fig. 2). The least squares fit of the three sets of values of n_2 for the three different composition samples listed in Table 1, yields $n = 2.304 + 68218/\lambda^2$ for $As_{40}S_{60}$ sample, $n = 2.467+81962/\lambda^2$ for $As_{40}S_{35}Se_{25}$ sample and $n = 2.688 + 73372/\lambda^2$ for $As_{40}Se_{60}$ sample.

The energy dependence of n of amorphous materials can be fitted to the Wemple and DiDomenico dispersion relationship, that is single-oscillator model[16]:

$$n^2(E) - 1 = \frac{E_d E_0}{E_0^2 - E^2} \qquad (6)$$

Where, E_0 is the single-oscillator energy and E_d the dispersion energy. By plotting $(n^2-1)^{-1}$ versus E^2 (Fig. 3) and filled the data to a straight line, E_0 and E_d can be determined from the intercept, E_0/E_d and the slope, $-1/E_0E_d$. The values obtained for dispersion parameters Ed, Eo and for static refractive index n (o) (i.e. extrapolated to $h\nu \rightarrow 0$), for three different compositions thin films. E_o is considered as an average energy gap to a good approximation, it varies in proportion to the Tauc gap E_g^{opt} (which will be defined latter, when the optical absorption edge is studied): $E_o \approx 2\,E_g^{opt}$ [18]. On the other hand an important achievement of WDD model is that related to the dispersion energy, E_d, to other physical parameters of material through the following empirical relationship[16,17]:

$$E_d = \beta N_c Z_a N_e \text{ (eV)} \qquad (7)$$

Where, N_c is the effective coordination number of the cation nearest neighbour to the anion, Za is the formal chemical valency of the anion, Ne is the effective number of valence electrons per anion and β is a two-valued constant with the either an ionic or a covalent value ($\beta_i = 0.26\pm0.03$ eV and $\beta_c = 0.37\pm0.04$ eV, respectively). The dispersion energy E_d increases with increasing Se content. Taking into account Eq. (7) and assuming that the parameters $N_c = (40 \times 5 + 60 \times 6)/60 = 28/3$ and $Z_a = 2$ retain the same values along this particular composition, it would seen reasonable to ascribe the trend observed in the values of E_d to an increase in the effective cation coordination number, N_c. On the other hand the possible influence of the parameter β on the increase observed for

Table 2: Values of the single-oscillator energy E_o, dispersion energy E_d, refractive index at ($h\nu \rightarrow 0$) and Tauc optical gap E^{opt}_g

Glass composition	E_d (eV)	E_0 (eV)	E^{opt}_g (eV)	n(o)
$As_{40}S_{60}$	20.85	4.796	2.37	2.313
$As_{40}S_{35}Se_{25}$	22.94	4.359	2.06	2.503
$As_{40}Se_{60}$	25.00	4.000	1.81	2.693

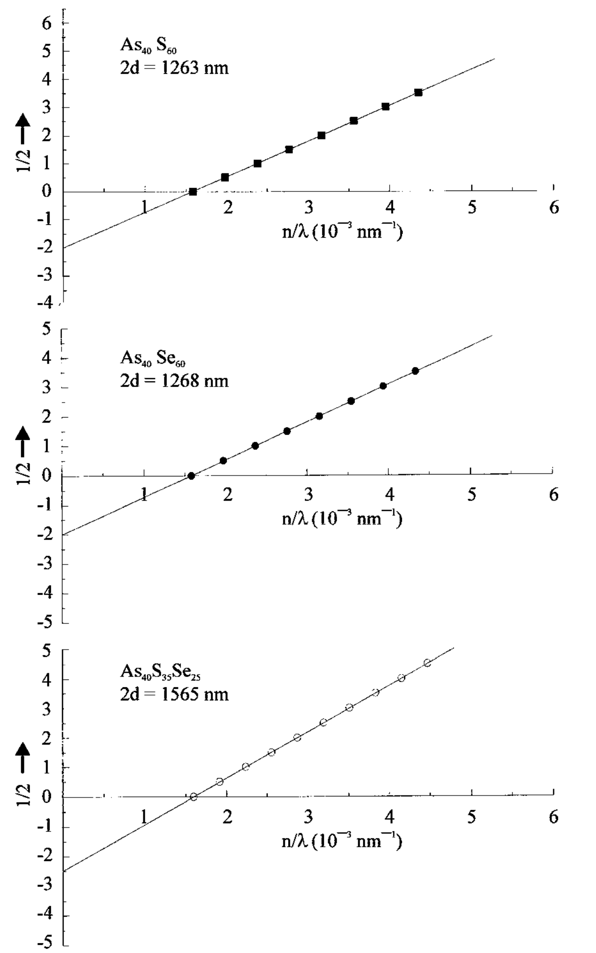

Fig. 4: Plot of $l/2$ against n/λ to calculate the order number and film thickness for spectra for the three different composition thin films

the oscillator strength should be also mentioned. Thus, the nature of the chemical bonding could change towards being less ionic with increasing Se content. Nevertheless, according to Paulings' electronegativities, the ionicity of an As-S bond is ≈ 8% while in the case of an As-Se bond it is ≈ 4%. Therefore β is considered to be a constant, with the covalent value $\beta_c = 0.37\pm0.04$ eV.

The agreement of the different parameters E_d, E_o and n(o) are reported in this work with those of Gonzαlez-Leal *et al.*[22]. The values of single-oscillator energy E_o, dispersion energy E_d and static refractive index n(o) for the three different composition thin films are listed in Table 2.

Furthermore, a simple complementary graphical method for deriving the values of m and d, based on Eq. 4 was also used. This expression can be rewritten for that purpose as:

$$l/2 = 2d(n/\lambda) - m_l \tag{8}$$

Where, l = 0, 1, 2,.....and m, is the first extreme. Therefore, plotting $l/2$ against n/λ yields a straight line with slope 2d and cut-off on the vertical axis of $-m_l$. Figure 4 shows this plot, in which the values obtained for d and m_l are displayed for three different composition thin films.

Determination of the extinction coefficient and optical band gap: The spectral dependence of the optical Transmittance (T) and Reflectance (R) of the investigated sample can be obtained using double beam spectrophotometer. The absorption coefficient α can be obtained from the experimentally measured values of R and T according to the following expression[19]:

$$\alpha = \frac{1}{d}\ln\left[\frac{(1-R)^2+\left[(1-R)^4+4R^2T^2\right]^{1/2}}{2T}\right] \tag{9}$$

Where, d is the sample thickness. In order to complete the calculation of the optical constants, the extinction coefficient is estimated from the values of a and λ using the already mentioned formula $k = \alpha\lambda/4\pi$. Figure 5 shows the dependence of k on wavelength for different samples of thin films.

Finally, the optical band gap will be found from the calculated values of a To that end, it should be pointed out that the absorption coefficient of

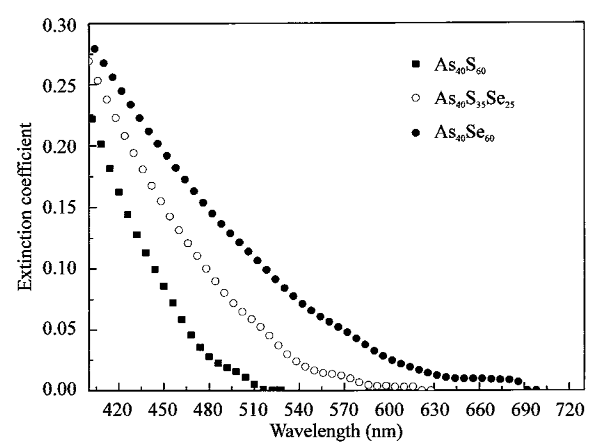

Fig. 5: The extinction coefficient k against wavelength λ for spectra for the three different composition thin films

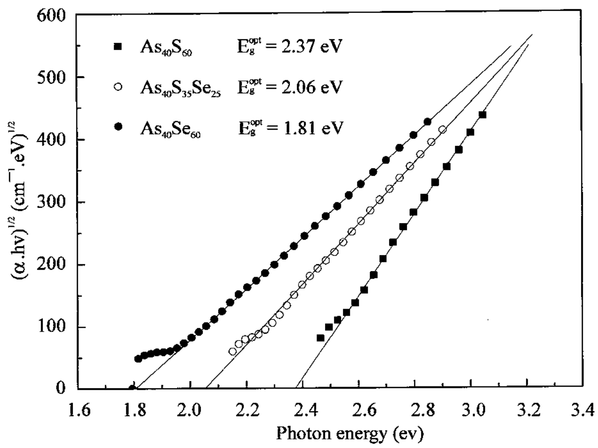

Fig. 6: The dependence of $(\alpha.\ hv)^{1/2}$ on photon energy hv for spectra for the three different composition thin films, from which the optical band gap E_g^{opt} is estimated (Tauc's extrapolation)

amorphous semiconductor. In the high-absorption region ($a = 10^4\ cm^{-1}$), assuming parabolic band edges and energy-independent matrix elements for interband transitions, is given according to Tauc[1] by the following equation:

$$\alpha(h\upsilon) = \frac{K(h\upsilon - E_g^{opt})^2}{h\upsilon} \qquad (10)$$

Where, K is constant which depends on the transition probability and E_g^{opt} is the optical band gap. A good fit between the experimental data for as prepared films and the $(\alpha\ hv)^{1/2}$ versus hv. Figure 6 shows this plot for three different compositions of chalcognide glass thin films and the optical band gap derived for each film is listed in Table 2. The value optical energy gap E_g^{opt} decrease with increasing Se content. (A similar compositional dependence has been observed for the oscillator energy, E_o (Table 2), as expected according to the previously mentioned relationship due to Tanaka[18]. The higher bonding energy of As–S bonds (379.5 kJ mol^{-1}) compared with that of As–Se bonds (96 kJ mol^{-1}), plausibly explains the decrease found in both optical parameters. It is interesting to point out that the molecular vapour species embedded in the as-evaporated amorphous $As_{40}S_{35}Se_{25}$ films ($As_4S(Se)_4$, S(Se) and As_4), introduce homopolar bonds of the types As–As, S–S and Se–Se. Thus, even though the high-bonding energies of such homopolar bonds (382.0, 425.3 and 332.6 kJ mol^{-1}, respectively) could somehow contribute to an increase of the value of both $E^{opt}{}_g$and E_o, according to our results they do not seem to play an important role in the behavior of E_g^{opt} and E_o with adding Se at expense of S. The estimated values of E_g^{opt} of composition $As_{40}S_{60}$ film is in a good agreement with those published earlier[13,15,21,22] and also the estimated values of E_g^{opt} of composition $As_{40}Se_{60}$ film is very close to this published[20,22].

CONCLUSIONS

The optical properties of as-deposited amorphous three different composition thin films of $As_{40}S_{60}$, $As_{40}S_{35}Se_{25}$ and $As_{40}S_{60}$ chalcogenide glass prepared by thermal evaporation, have been determined from their corresponding transmission and reflectance spectrum, measured at normal incidence. The envelope method suggested by Swanepoel has successfully been applied to films with higher thickness, with a reasonable number of interference fringes. This optical method makes it possible to determine the refractive index and average thickness with an accuracy of about 1%. It has been found that the refractive index increases with increasing Se content over the entire spectral range studied. The increase in refractive index is related to the increased polarizability, of the larger Se atoms, in comparison with S atoms. The subsequent fitting of the refractive index to the Wemple-DiDomenico relationship for obtaining the dispersion parameters. The optical band gap is appropriately fitted to the non-direct transition model proposed by Tauc[1], in the strong-absorption region of investigated films. The results indicate that the values of E_o and E_g^{opt} decrease with the increasing Se content, which is plausibly explained by taking into account the higher bonding energy of As–S bonds, in comparison with that of As–Se bonds.

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