

ELECTRICAL AND OPTICAL PROPERTIES OF A- $\text{Se}_{90}\text{In}_{9.3}\text{Cu}_{0.7}$ THIN FILMS

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ABSTRACT

The dc electrical conductivity of metal chalcogenide glassy alloys films $\text{Se}_{90}\text{In}_{9.3}\text{Cu}_{0.7}$ and its variation with temperature have been studied. The electrical conductivity, plotted versus inverse temperature, obeys an Arrhenius type dependence for as-deposited and annealed films. The optical properties of the above films have also been studied for as deposited films. Various optical constants have been calculated for the present system of $\text{Se}_{90}\text{In}_{9.3}\text{Cu}_{0.7}$. The values of the refractive index (n) and the extinction coefficient (k) decrease with wavelength.

It is found that, the types of optical transition responsible for optical absorption is indirect allowed transitions with energy gap 1.96 eV. The Wemple-Didmenico single-oscillator model parameterize the refractive index.

KEYWORDS: *Se-In-Cu alloy film system; Electrical conductivity; Optical constants; Optical band gap; Single-oscillator model.*

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1. Introduction:

The electrical properties of Se-based glasses are very interesting, since these materials exhibit effects such as optical memory and photodarkening [1,2], for which the study of their electrical behavior could shed some light. The addition of transition metals into these amorphous materials, even at very low concentrations, leads to a significant increase in their electrical conductivities [3].

Cu In Se₂ is a semiconducting compound which belongs to the I-III-VI chalcopyrite family. It is convenient to regard the chalcopyrite structure in terms of two interpenetrating sublattices in which I-III atomic positions which act as cations rest on one sublattice and the group VI atoms which act as anions, rest on the other sublattice [4]. Cu In Se₂ is a direct band gap [5] material with band gap ~ 1.0 eV at room temperature. Further, its band structure and the fact that it can be doped to make both n and p-type within a wide range of resistivity [6], makes it a very strong candidate for photovoltaic devices. Cu In Se is a ternary semiconductor that crystallizes in the chalcopyrite crystal structure. It has an absorption coefficient $\sim 10^5$ cm⁻¹ in thin film form the highest reported to date for any semiconductor [7]. Accurate knowledge of the optical functions of these materials is very important for many of these applications. In spite of the considerable amount of research devoted to these materials, this knowledge is still incomplete.

Various workers [8,9] have recently reported the use of a-semiconducting films for reversible optical recording by the amorphous-to-crystalline phase change chalcogenide glasses have been found to exhibit a change in the refractive index under the influence of light, which makes it possible to use these materials to record not only the magnitude but also the phase of illumination. The latter is especially important in the holographic optical data storage, in the fabrication of various integrated optics components, and in devices such as selective optical filters, mixers, couplers and modulators [10]. Attempts have been made to investigate Se-based chalcogenide thin films. The lattice perfection and energy band gap of the material play a major role in the fabrication of a device for a particular wavelength; these can be modified by the addition of dopants. Pure amorphous selenium films exhibit optical absorption and photosensitivity in the visible range of wavelength, while the alloys exhibit the same properties over a large wavelength range [11].

The aim of the present work is to measure electrical conductivity as well as the structure of a-Se₉₀In_{9.3}Cu_{0.7} thin films by using X-ray diffraction techniques. Also, we present careful transmittance and reflectance measurements over the wavelength range 400-2500 nm. The optical properties such as refractive index (n), extinction coefficient (k), provide the Wemple-DiDomenico single oscillator model parameterizes are determined.

2. Experimental

Polycrystalline ingots of $\text{Se}_{90}\text{In}_{9.3}\text{Cu}_{0.7}$ were prepared by fusion of the constituent elements in the stoichiometric ratio in sealed evacuated silica tubes (10^{-3}Pa), were left at 1200°C for 10 h, and then quenched in iced water at 1000°C . X-ray powder diffraction revealed some crystalline phase in these ingots because there is Cu atoms is more. Thin films were prepared with material from these ingots by a single source thermal evaporation method using (Edwards E 306A) unit with resistively heated molybdenum boat. The working vacuum was kept at about $\sim 6 \times 10^{-4}\text{Pa}$ during deposition. The rate of the film deposition was nearly 3 nm/sec. The film thickness was determined using quartz crystal thickness monitor (FTM4) and also interferometrically.

The X-ray diffraction patterns of both bulk and thin films were recording using an X-ray diffractometer (Shimadzu X D-3). The patterns were run with Cu as target and Ni as filter. The amorphous $\text{Se}_{90}\text{In}_{9.3}\text{Cu}_{0.7}$ thin films were annealed at 375 and 425 K for 1 h in vacuum $\sim 10^{-2}\text{Pa}$.

Electrical conductivity was recorded for virgin sample as a function of temperature in the temperature range 300-425 K, and after each annealing step. The contact between the terminals of the resistance measurements and the film was silver paste. Accordingly, the contact effects and space charge are minimized.

The transmittance (T) and reflectance (R) of a- $\text{Se}_{90}\text{In}_{9.3}\text{Cu}_{0.7}$ thin films deposited on a quartz substrates, at normal incidence of light in the wavelength 400-2500 nm were measured using a double beam spectrophotometer (Type JASCO Corp., Model V-570).

If I_{ft} and I_q are the intensities of light passing through the film-quartz system and that through the reference quartz, respectively, then [12]:

$$T_{\text{exp}} = (I_{ft} / I_q) (1-R_q) \quad \dots\dots\dots (1)$$

where R_q is the reflectance of quartz. In addition, if the intensity of light reflected from the sample reaching the detector is I_{fr} and that reflected from reference Aluminum mirror is I_{Al} then [13]:

$$R_{\text{exp}} = (I_{fr}/I_{Al}) R_{Al} [1+ (1 - R_q)^2] - T^2 R_q \quad \dots\dots\dots (2)$$

From the measured T_{exp} , R_{exp} and film thickness ($d = 940\text{nm}$) the values of refractive index, n , and the extinction coefficient, k , were computed by a special computer program [14] based on minimizing $(\Delta T)^2$ and $(\Delta R)^2$ simultaneously, where

$$(\Delta T)^2 = | T_{(n,k)} - T_{\text{exp}} |^2 \quad (3)$$

$$(\Delta R)^2 = |R_{(n,k)} - R_{\text{exp}}|^2 \quad (4)$$

where T_{exp} and R_{exp} are the experimentally determined values of T and R , respectively, and $T_{(n,k)}$ and $R_{(n,k)}$ are calculated values of T and R , using the Murmann's exact equations [15, 16].

The experimental errors were taken into considerations as follows: $\pm 2.2\%$ for film thickness measurements $\pm 0.1\%$ for T and R calculations, $\pm 3\%$ for refractive index and ± 2.5 for extinction coefficient. The crystallized materials at different stages of crystallization reactions were used for structure identifications using X-ray diffractometer as shown in Fig. (1).

3. Results and Discussion

3.1. Electrical conductivity

The d.c. conductivity of $a\text{-S}_{90}\text{In}_{9.3}\text{Cu}_{0.7}$ thin films was measured as a function of temperature in the range 300-425 K. The d.c. conductivity dependence on temperature was measured after each annealing, this fit well to the relation:

$$\sigma = \sigma_0 \exp(-E/KT)$$

where σ_0 is the pre-exponential factor ($\Omega^{-1}\text{Cm}^{-1}$), E is the electrical activation energy (eV) and K is the Boltzmann constant. The virgin films were pretreated by heating to 380K for 10 min in vacuum $\approx 10^{-3}$ Torr. This steps was repeated before starting film characterization in order to clean the samples from defects [17]. Therefore, the conductivity during the cooling cycle closely coincides with that in the heating cycle in vacuum $\approx 10^{-3}$ Torr. Hence for all our calculations we have used the data of the films during the heating cycle. The thermal activation energy of the films was determined from the slopes of $\ln \sigma$ versus $1000/T$ (K^{-1}) plots as shown in Fig. 2. It can be seen that the conductivity increases with increasing temperature but that the increase is slow in the low temperature region up to 330 K. Above 340 K, the conductivity increases linearly with increasing temperature. Fig. 2, plots of $\ln \sigma$ ($\Omega^{-1}\text{Cm}^{-1}$) versus $(1000/T)$ (K^{-1}) in two domains of temperature, the data is fitted by two straight lines for all steps of measurement. Such results can be interpreted as a change from a hopping conduction in the band tail [$E_{a2} = 0.4 - 0.23$ eV] to a free carriers conduction [$E_{a1} = 0.53 - 0.38$ eV] for virgin and annealed films at 375 and 425 K for 1 h. It is noticed that the conductivity is found to increase very slowly with temperature in the range 300-330 K, while above 340K, the conductivity is found to increase very sharply with temperature. The low activation energy is either due to first phase Cu In_6 or due to the excitation of the charge carriers from a shallow acceptor level or due to the predominance of the hopping conduction mechanisms [4]. Table 1, illustrates the pre-exponential factor of conductivities at (0K) (σ_0 's) of virgin, annealing at 375 K

and 425 K and the two energies of each case. These conductivities at (0K) increase with increasing annealing of temperature, while, the energy of each regime decreases as shown in table 1. Due to increase localized state in the band gap, which leads to increase the conductivity.

The presence of a hopping regime strongly suggests important localized states as band tails [18]. The highest activation energy (E_{a1}) is an indication of the position of the Fermi level, (E_F) in the band

Table (1):

Parameters	Temperature		
	300 K	375 K	425 K
σ_o ($\Omega^{-1} \text{ Cm}^{-1}$)	2.7×10^3	3.53×10^3	7.33×10^3
E_{a2}, E_{a1} (eV)	0.40, 0.53	0.27, 0.42	0.23, 0.38

gap, due to the original phase Cu In Se₂. The hopping mechanism in amorphous materials is either due to transfer charge carriers from valence band to localized state or due to transfer charge carriers from localized state to the conduction band, a process which requires thermal activation. On the other hand, the high activation energy observed at high temperature corresponds to the excitation of the charge carriers from the valence band to the conduction band. Both activation energy (E_{a1}) and hopping energy (E_{a2}) decrease with increasing temperature of annealing as shown in Fig. 2. Accordingly, the Fermi level (E_F) moves towards the valence band edge. We can observe the increasing of the conductivity of each region due to crystallization as shown in Fig. (2). At the grain boundary of each of the crystallites, the incomplete atomic bonding can act as trap centers [19]. These trap centers can trap the charge carriers at the grain boundaries, and hence, a space charge can be built up locally. Therefore, the conduction pass becomes easier and increasing the conductivity [20,21].

3.2. Crystallization products

In this study, the X-ray diffraction technique is used to determine the phases present and the orientation of polycrystalline a-Se₉₀ In_{9.3} Cu_{0.7} thin films deposited by thermal evaporation. The annealing temperatures 375 and 425 K were selected to be between the glass transition (T_g) and the crystallization temperature (T_c) as drawn out from DTA traces. After each step of annealing, the X-ray diffraction was recorded with the diffraction angle range 10-60° as shown in Fig. (1). It shows there are different phases, the identification of the observed crystalline peaks confirmed that phases of Se, In₆ Se₇, Cu₅ Se₄ and Cu₂Se in the films under investigation as shown in Table 2. The peaks a,b,c,d,e and f were selected to investigate the

crystalline phases and the crystalline structure of each phase. The peaks a, b and c grow and continue to grow as the temperature of annealing increases at the same time of annealing, may be attributed to the growing up of the crystalline phases on the expense of the old amorphous matrix. This in turn leads to the decrease of the activation hopping energy and the decrease of the energy barrier between the neighboring crystalline phases [22]. However, the peaks d, e and f are phases formation at higher temperature of annealing. In a random network model of a glass, the preservation of nearly optimal bond lengths cause fluctuations in bond angles and Van der waals distances. These local fluctuations may be large enough to give rise to electronic or vibrational properties quite different from those of the typical bonding configurations in the random-network model. Such fluctuations, then contribute to tails of localized states on the valence and conduction bands [23].

Table (2): X-ray crystallographic data of annealed a-Se₉₀ In_{9.3} Cu_{0.7} thin film at two different temperatures in vacuum for 1h.

Temperature of annealing T(K)	Experimental			ASTM cards			
		2θ	d(Å)	Crystal phase	d Å	(hkl)	
375	a	23.6	3.765	Se	3.779	310	Monoclinic
	b	29.5	3.024	In ₆ Se ₇	3.026	114	-----
	c	41	2.199	Cu ₅ Se ₄	2.200	042	Orthorhombic
425	a	23.2	3.89	Se	3.89	-112	Monoclinic
	b	29.5	3.024	In ₆ Se ₇	3.026	114	-----
	c	41.0	2.199	Cu ₅ Se ₄	2.20	042	Orthorhombic
	d	43.4	2.08	Cu ₂ Se	2.06	404	Tetragonal
	e	51.5	1.772	Cu ₂ Se	1.76	533	Tetragonal
	f	65.2	1.429	Cu ₂ Se	1.440	800	Tetragonal

3.3. Optical properties

The optical transmission and reflection spectra of a-Se₉₀ In_{9.3} Cu_{0.7} thin films were recorded at room temperature in the spectral range 400-2500 nm as shown in Fig. (3). At longer wavelengths with respect to the absorption edge, T+R=1 indicating that Se₉₀ In_{9.3} Cu_{0.7} thin films transparent and no light is scattered [24]. The variation of the optical absorption near the fundamental

absorption edge has allowed use to determine the optical energy gap. The theory of interband absorption shows that at the optical absorption edge, the absorption coefficient (α) varies with the photon energy ($h\nu$) according to the expression:

$$(\alpha h\nu) = B (h\nu - E_g)^2$$

where B is a constant and E_g is the optical energy gap situated between the localized states near the mobility edges according to the model density of states proposed by Mott and Davis [25, 26]. As shown, $(\alpha h\nu)^{1/2}$ varies linearly with $(h\nu)$, which is in good agreement with the classical theory of the band transition. Extrapolating the straight parts of these relation to the $(h\nu)$ axis yields values of the forbidden gap as shown in Fig. (4). It is found that the optical energy gap (E_g) is 1.96 eV.

Fig. 5 shows the variation of the refractive index (n) and the absorption index (k) with the wavelength. The values of n and k decreased with increasing wavelength in the wavelength range 500-1000 nm. Above wavelength 1000 nm, k (wavenumber) = 0 and n was nearly saturated with increasing wavelength. The values of refractive index in the range 2.9 – 3.4, these values of the refractive index are in good agreement with those determined optically by Alonso et al., [27]. The change of the refractive index, n , and absorption index, k , associated with the introduction of Cu in the usual manner has a direct relation to the amount and strength of the possible different bonds formed in the network structure of the investigated composition.

The relation between the refractive index, n , and the oscillator strength below the band gap is given by the expression [28].

$$n^2(h\nu) = 1 + \frac{E_d E_o}{E_o^2 - (h\nu)^2}$$

where $h\nu$ is the photon energy, E_o is the oscillator energy and E_d is the oscillator strength or dispersion energy which measures the average strength of interband optical transitions. Fig. 6 shows the relation between $(n^2-1)^{-1}$ and the photon energy $(h\nu)^2$ for the investigated composition. The values of E_d , and E_o have been determined from the slope and the intersection of the straight line with $(n^2-1)^{-1}$ axis. The values of both dispersion energy and oscillator strength are $E_d = 29.5$ eV and $E_o = 3.9$ eV respectively. The oscillator energy is an "average" double energy gap ($E_o \sim 2E_g$) and to reasonably good approximation, it agree well as was first found by Tanaka [29].

4. Conclusions

The characteristic conductivity and structure of a-Se₉₀In_{9.3}Cu_{0.7} thin films were investigated before and after annealing at 375 and 425 K in vacuum

for 1 h. The results showed that the conductivity of annealed films was increased with increasing operating temperature, due to transformation of the amorphous film to crystalline state. X-ray diffraction have shown that the chalcopyrite structure of CIS was easy to form with the exception of the presence of traces of some binaries for large deviations in composition towards the In_6Se_7 , Cu_5Se_4 and Cu_2Se directions on the ternary compound diagram.

The band gap energy calculated using the optical measurement data 1.96 eV, which is in good agreement with the literature. This composition resemble indirect carrier transition involve the absorption of an electromagnetic wave by an electron in the valence band that raise above the forbidden energy gap to the conduction band. The value dispersion energy, E_d , of amorphous films was 29.5 eV and the oscillator energy, E_o , was 3.9 eV respectively. The spectral dependence of the absorption coefficient indicated indirect transition. The oscillator energy is an average double optical energy gap ($E_o \approx 2E_g$) and to reasonably good approximation, it agree well as was first found by Tanaka

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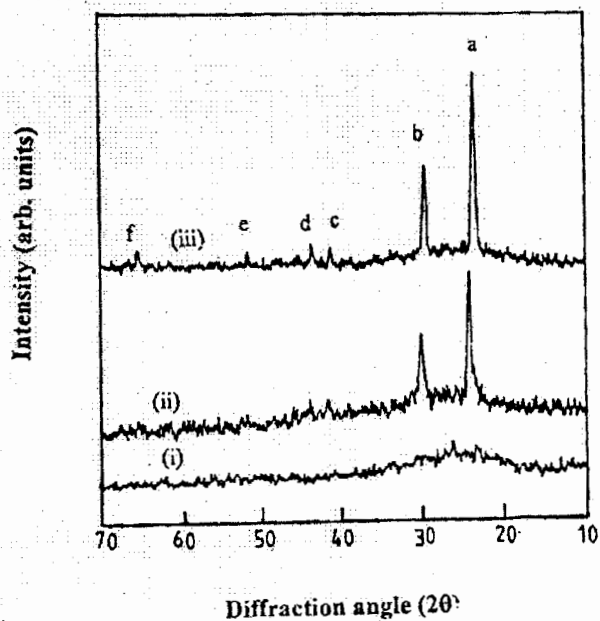


Fig. 1. X-ray diffractogram of a-Se₉₀In_{9.3}Cu_{0.7} thin film (i) as-deposited (300 K) (ii) annealed at 375 K (iii) annealed at 425 K in vacuum for 1h.

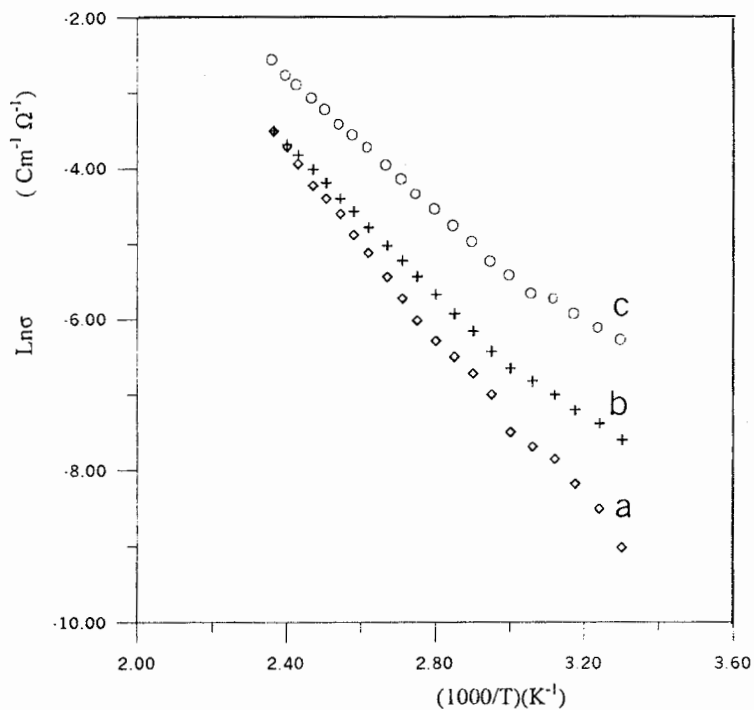


Fig. 2. Electrical conductivity ($\ln \sigma$) versus reciprocal temperature ($1000/T$) for a-Se₉₀In_{9.3}Cu_{0.7} thin film (a) virgin (b) annealed at 375 K (c) annealed at 425 K.

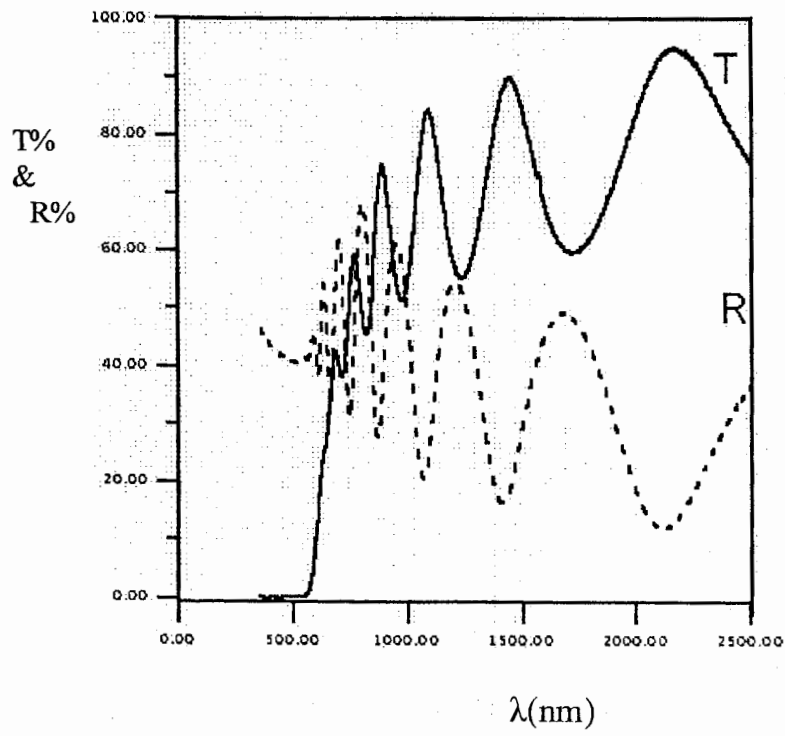


Fig. 3. Transmission and reflection versus wavelength of a-Se₉₀In_{9.3}Cu_{0.7} thin film

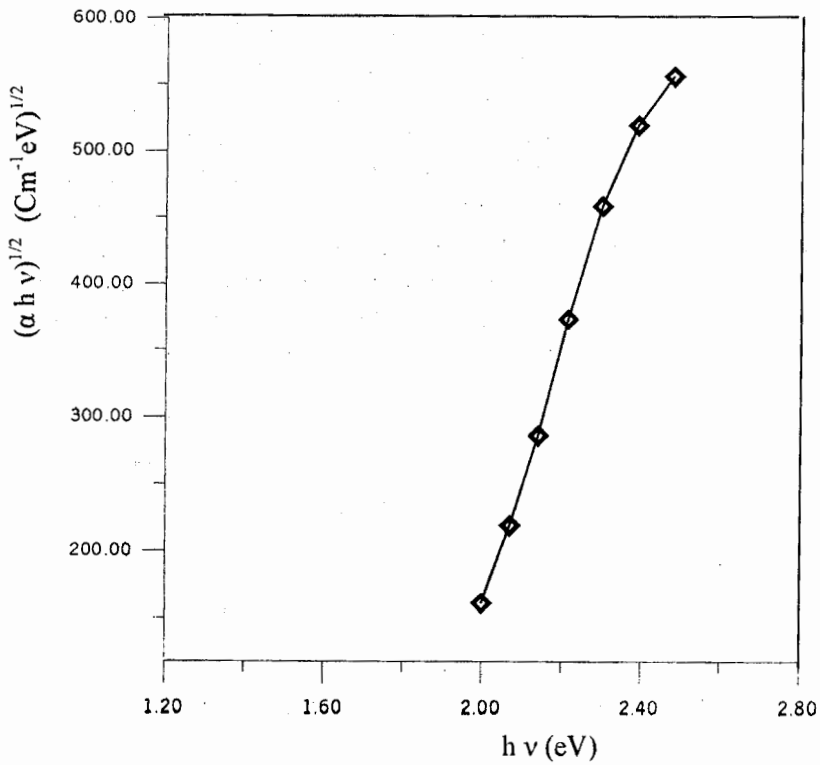


Fig. 4. $(\alpha \cdot hv)^{1/2}$ versus $h\nu$ (eV) for a-Se₉₀In_{9.3}Cu_{0.7} thin film

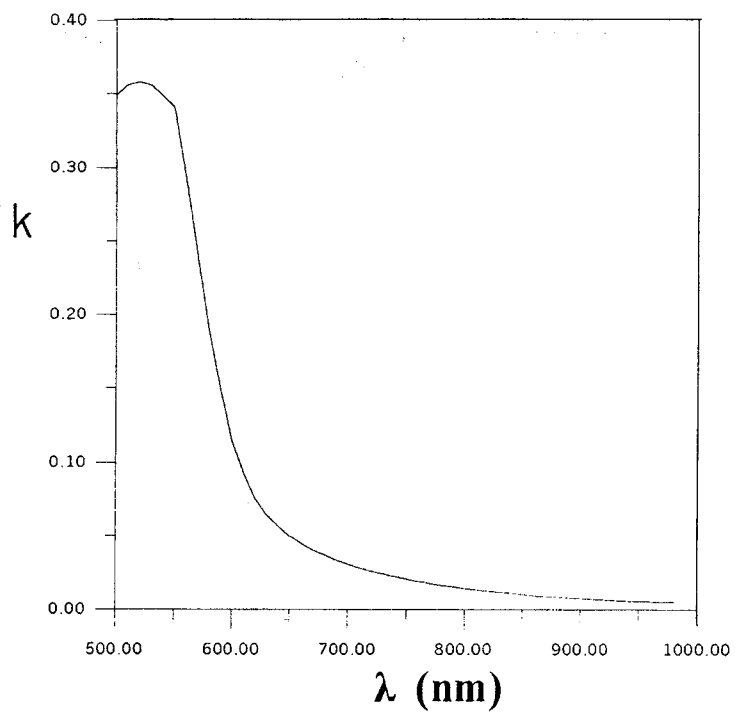
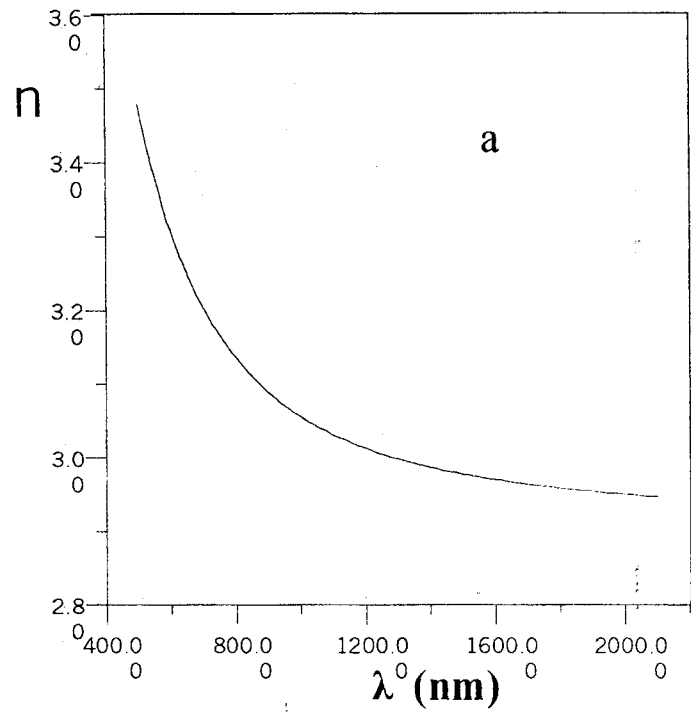


Fig. 5. (a) The relation between the refractive index (n) and the wavelength (nm) of a-Se₉₀In_{9.3}Cu_{0.7} thin film.

(b) The relation between the extinction coefficient (k) and the wavelength (nm) of a-Se₉₀In_{9.3}Cu_{0.7} thin film

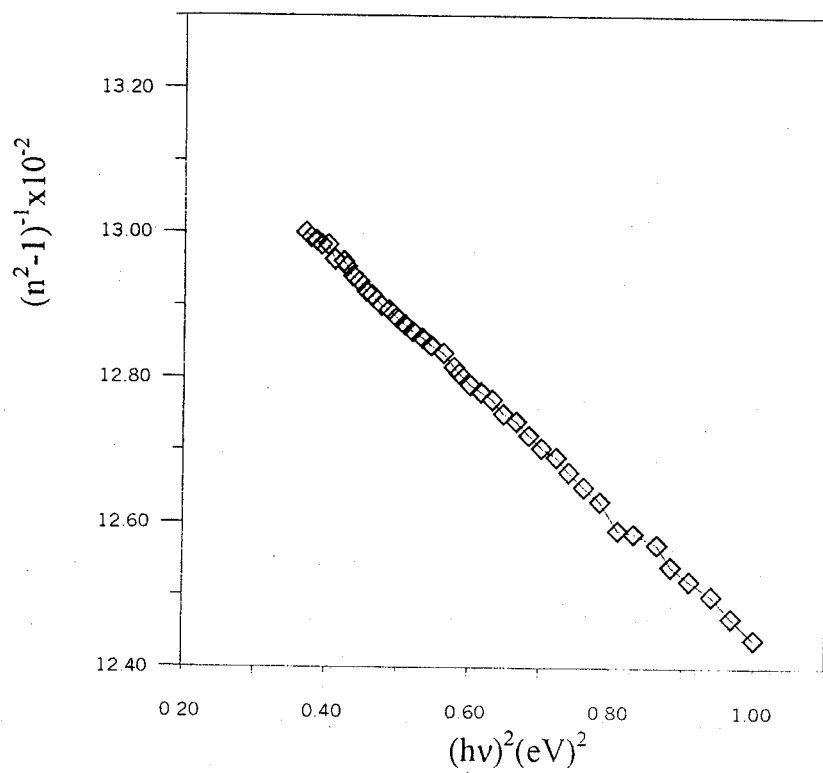


Fig. 6. The relation between $(n^2-1)^{-1}$ and $(hv)^2 (eV)^2$ for $a\text{-Se}_{90}\text{In}_{9.3}\text{Cu}_{0.7}$ thin film

الخواص الكهربائية والضوئية لأفلام $Se_{90} In_{9.3} Cu_{0.7}$ الغير بللورية

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المخلص:

تم تحضير سبيكة من النظام $Se_{90} In_{9.3} Cu_{0.7}$ وذلك بأخذ الأوزان المناسبة من العناصر النقية المكونة للنظام ووضعها فى أنابيب من السليكا وصهرها تحت تفريغ عال (10^{-2} باسكال) لمدة عشر ساعات ثم تبريدها فجائى وقد اختيرت درجات الحرارة اللازمة لذلك بما يتلاءم مع درجة حرارة إنصهار كل عنصر وكذلك درجة حرارة إنصهار الخليط المتوقع. ثم تم تحضير شرائح رقيقة من هذه المواد بطريقة التبخير عند تفريغ 10^{-4} باسكال وكان معدل ترسيب الفيلم ٣ نانومتر لكل ثانية. تم اختبار هذه الأفلام بالأشعة السينية (X-ray diffraction) ووجد أنها مادة غير بللورية (أمورفية). ثم تم عمل تحمية لهذه الأفلام عند درجتى الحرارة ٣٧٥ كلفن، ٤٢٥ كلفن لمدة ساعة تحت تفريغ 10^{-2} باسكال.

تم قياس التوصيل الكهربى عند الثلاث حالات للمادة وجد أن التوصيل الكهربى يزداد مع زيادة درجة التحمية عن الحالة الأصلية (قبل التحمية) وتوجد منطقتين للتوصيل الكهربى. المنطقة الأولى وتكون عند درجات الحرارة المنخفضة وتخضع لحالة (hopping conduction) أما المنطقة الثانية تكون عند درجات الحرارة العالية وتخضع (Free carrier conduction) وتم حساب طاقة التنشيط لكل منطقة ووجد أنها تقل مع زيادة درجة التحمية وهذا يرجع إلى وجود مستويات محلية (Localized state) داخل فجوة الطاقة وتحرك مستوى فيرمى (E_F) فى اتجاه حزمة التكافؤ (Valence band).

وعلى التوازي تم قياس الخواص الضوئية ممثلة فى قياس معاملى الإنتقال والانعكاس للأفلام عند الثلاث حالات باستخدام جهاز الاسبكتروفوتومتر خلال المدى ٣٢٥ - ٢٥٠٠ نانومتر. وجد أن فرق النتائج قبل التحمية وبعد التحمية لا تتعدى نسبة الخطأ فتم حساب النتائج للحالة الابتدائية فقط (قبل التحمية).

ومن خلال القياسات تم حساب فجوة الطاقة الضوئية (E_0) وطاقة التبيد (E_g) ومعامل الإنكسار (n) وكذلك معامل الإمتصاص (K) كدالة فى الطول الموجى ووجد أنه يوجد تغير فى قيمة (n) و (K) مع الطول الموجى وذلك لوجود مادة النحاس (Cu) التى أدت إلى تغير فى التركيب البلورى للمركب.