



## Effects of laser irradiation on optical properties of amorphous and annealed Ga<sub>15</sub>Se<sub>81</sub>In<sub>4</sub> and Ga<sub>15</sub>Se<sub>79</sub>In<sub>6</sub> chalcogenide thin films

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### ARTICLE INFO

#### Article history:

Received 29 April 2010

Received in revised form 8 June 2010

Accepted 10 June 2010

Available online 17 June 2010

#### Keywords:

Annealing

Crystallization

Chalcogenide glasses

Optical band gap

Absorption coefficient

Laser irradiation

### ABSTRACT

Amorphous thin films of Ga<sub>15</sub>Se<sub>81</sub>In<sub>4</sub> and Ga<sub>15</sub>Se<sub>79</sub>In<sub>6</sub> glassy alloys with thickness 3000 Å were prepared by thermal evaporation onto chemically cleaned glass substrates. The changes in optical properties due to the influence of laser radiation on amorphous and thermally annealed thin films of Ga<sub>15</sub>Se<sub>81</sub>In<sub>4</sub> and Ga<sub>15</sub>Se<sub>79</sub>In<sub>6</sub> were calculated from absorbance and reflectance spectra as a function of photon energy in the wave length region 400–1000 nm. Analysis of the optical absorption data shows that the rule of non-direct transitions predominates. The optical band gaps observed to decrease with the increase of annealing temperatures. Furthermore, exposing thin films to laser irradiation leads to a decrease in optical band gap, absorption coefficient, refractive index and extinction coefficient for both as-prepared and annealed films. The decrease in the optical band gap is explained on the basis of change in nature of films, from amorphous to polycrystalline state, with the increase of annealing temperature and by laser irradiation for 10 min exposure time. Outcomes of our study confirm that this system may be used for photovoltaic devices.

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### 1. Introduction

In recent years there has been a great deal of interest in the study of chalcogenide glasses from the point of view of basic physics as well as of device technology. They are suitable material for optical elements, optical memory disks, functional elements in integral-optic systems and IR-fibers that show high flexibility and chemical durability etc. It is well known that thermal relaxation occurs in these glasses, following an instantaneous change in temperature (during the quenching process) as a glassy substance relaxes from a state of higher enthalpy towards an equilibrium state of lower enthalpy. It has been observed that the variations of composition and annealing conditions for glassy chalcogenide semiconductors have a significant influence on the band gap and on the optical properties. Today, research on optical properties of materials draws on not only physicists, who used to be the usual traditional researchers in this field, but also scientists and engineers from widely different disciplines. The optical properties of the materials used in the design and manufacture of infrared interference filters play a vital role in defining the spectral performance achievable from a multilayer filter design. Due to the technological importance, the optical properties and electrical properties of chalcogenide thin

films have been subjected to a lot of investigations [1–8]. Thermal processes are known to be important in inducing crystallization in semiconducting chalcogenide glasses. The optical storage based on the amorphous–crystalline phase transition utilizes the large optical reflectivity and optical absorption changes obtained in some semiconductors–semimetal thin films by heat treatment or laser irradiation [9]. Laser-induced changes in amorphous chalcogenides are an object of systematic investigations with a view to better understanding the mechanism of the phenomena taking place in them as well as their practical applications. So many authors have studied [10–14] the effect of  $\gamma$ -irradiation, annealing, photoinduced changes, gamma, UV, ion beam, deuteron, laser irradiation etc on optical properties of chalcogenide thin films. Huajun et al. [15] have studied structural change of laser irradiated Ge–Sb–Te thin films. Bahishti et al. [16] have studied the effect of laser irradiation on thermal and optical properties of Se–Te alloys. Nemeč and Frumar [17] have investigated the irreversible photoinduced changes in As<sub>48</sub>S<sub>52</sub> amorphous thin films prepared by pulsed laser deposition. The work on electronic and structural changes induced by irradiation or annealing in pulsed laser deposited As–Se films by Kalyva et al. [18], photoinduced effects in chalcogenide thin films under irradiation by synchrotron light by de Moura et al. [19], laser-induced amorphization and crystallization on Se–Te–Pb thin films by Khan et al. [20], the role of thermal treatment on the optical properties of Ge–Se system by Alnajjar [21], laser irradiation of amorphous thin films by Ortiz and Blatter [22] are also worth mentioning.

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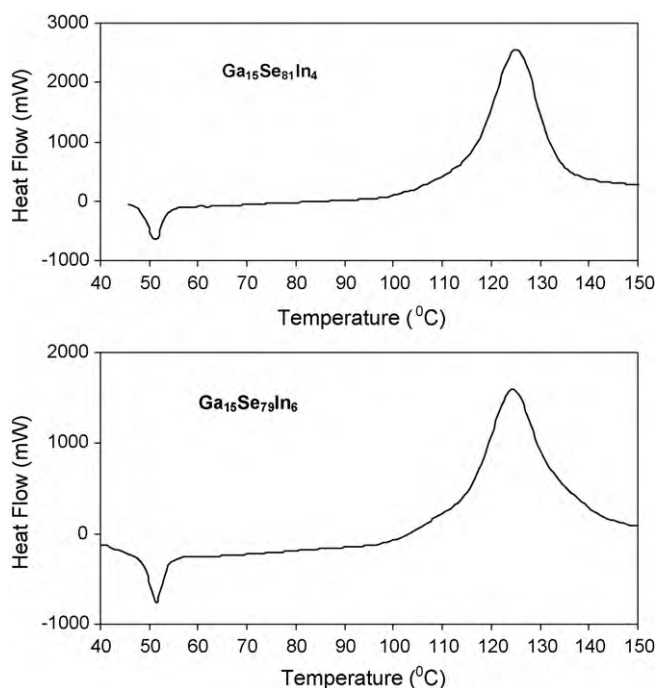


Fig. 1. DSC plots for  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  glasses at heating rate of 15 K/min.

The present communication reports the effect of laser irradiation on optical properties of amorphous and annealed thin films of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  chalcogenide glasses. In the present system, we have used Se as a major content because selenium based chalcogenide glasses have extensive applications as electronic and optoelectronic device materials, due to their transparency in the IR region, good thermal, mechanical and chemical properties. The high refractive index makes them suitable to be used as core materials for optical fibers for light transition in short wave length region. Here we have chosen Ga as one of the additive material with Se. There is a strong tendency for Ga to supercool below its freezing point. So, seeding may be necessary to initiate solidification [23]. In the present system, we have incorporated In in the Ga–Se system. The addition of third element indium (In) has gained a remarkable great interest due to its potential applications in various digital electronic devices such as digital cameras and camcorders, MP3 players, smart phones. Subsequently, its market spreads out swiftly for its non-volatile memory that uses reversible phase transition of chalcogenide resistor [24–26].

## 2. Experimental

Bulk homogeneous  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  chalcogenide glasses were prepared by melt quenching technique with elemental constituents of 5N purity [20,27,28]. Materials were weighed according to their atomic percentages and sealed in quartz ampoules in a vacuum of  $10^{-6}$  Torr. Alloying of the elements was accomplished by putting the sealed ampoules in a microprocessor-controlled programmable muffle furnace with a rocking mechanism. Rocking motion ensures that a complete mixing of the materials takes place. To assure complete chemical reactions between the constituents, the furnace temperature program was adjusted firstly at 473 K for 4 h, secondly at 573 K for 2 h and at last 873 K for 14 h, then rapid quenching in ice-water bath was used to obtain the bulk amorphous material. Differential scanning calorimeter (Model DSC Plus, Rheometric Scientific Company, UK) was used for measuring the glass transition and crystallization temperatures of bulk samples of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$ . DSC scan using non-isothermal measurements was obtained by heating 5 mg of the powdered sample sealed in an aluminum pans at constant heating rate of 15 K/min, shown in Fig. 1. The annealing temperatures were taken in between the glass transition and crystallization temperature of the prepared samples.

Thin films of thickness 3000 Å were prepared using an Edward Coating Unit E-306, onto glass substrates at room temperature on a base pressure of  $10^{-6}$  Torr using a molybdenum boat. The substrates were thoroughly cleaned in a detergent solution and then in a chromic acid and finally, cleaned using trichloroethylene.

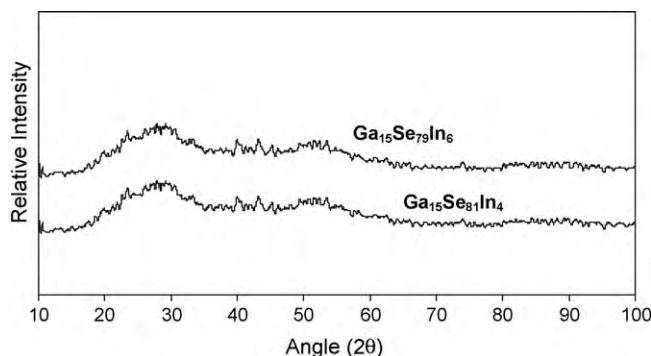


Fig. 2. X-ray diffractogram of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  thin films.

The thickness of the films was measured using a quartz crystal monitor Edward model FTM 7. The X-ray diffraction patterns give valuable information about the nature and structure of the samples. A Regaku X-ray diffractometer Ultima IV was employed for studying the structure of the thin films. The copper target was used as a source of X-rays with  $\lambda = 1.54056 \text{ \AA}$  ( $\text{Cu K}\alpha_1$ ). The scanning angle was in the range of  $10\text{--}100^\circ$ . A scan speed of  $2^\circ/\text{min}$  and a chart speed of  $1 \text{ cm}/\text{min}$  were maintained. The X-ray diffraction traces of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  chalcogenide thin films were taken at room temperature and found to show similar trends, shown in Fig. 2. The absence of sharp structural peaks confirms the amorphous nature of the films. Thin film were crystallized by thermal annealing at different temperatures 338 K, 353 K and 368 K, which is in between the glass transition and crystallization temperature of the samples for 2 h in a vacuum furnace under a vacuum of  $10^{-3}$  Torr. The crystallized thin films at different annealing temperatures and amorphous thin film were irradiated by placing it at a distance of 8 cm from the TEA nitrogen laser (wavelength 337.1 nm, peak power 100 kW) with exposure time 10 min. A JASCO, V-500 ultraviolet–visible–near-infrared computerized spectrophotometer was used for measuring optical absorption and reflectance.

## 3. Results

The optical properties may be closely related to the material's atomic structure, electronic band structure and electrical properties. An accurate measurement of the optical constant can be easily performed on thin film specimens. The optical behavior of materials is important to determine its usage in optoelectronic devices. Optical absorption measurements are used to obtain the band structure and the energy gap of binary and ternary chalcogenide thin films, because the analysis of the optical absorption spectra is one of the most productive tools for understanding and developing the energy band diagram of both crystalline and amorphous materials. The optical behavior of material is generally utilized to determine its optical constants i.e. refractive index ( $n$ ) and extinction coefficient ( $k$ ). Thin films are ideal specimens for absorbance, reflectance and transmittance type of measurement.

The absorption coefficient ( $\alpha$ ) has been obtained directly from the absorbance against wavelength curves using the relation [29,30]:

$$\alpha = \frac{\text{absorbance}}{\text{film thickness}} \quad (1)$$

The calculated values of absorption coefficient ( $\alpha$ ) for  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  thin films (amorphous, annealed and laser irradiated films) are given in Table 1.

Figs. 3 and 4 show the variation of absorption coefficient ( $\alpha$ ) as a function of incident photon energy ( $h\nu$ ) for  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$ : amorphous and annealed films; and laser irradiated thin films.

It has been observed that the value of absorption coefficient ( $\alpha$ ) increases linearly with the increase in photon energy for as-prepared, annealed and laser irradiated thin films of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  glassy alloys.

The knowledge of accurate values of wavelength dependence refractive index and extinction coefficient of chalcogenide thin film is very important for both fundamental and technological point of

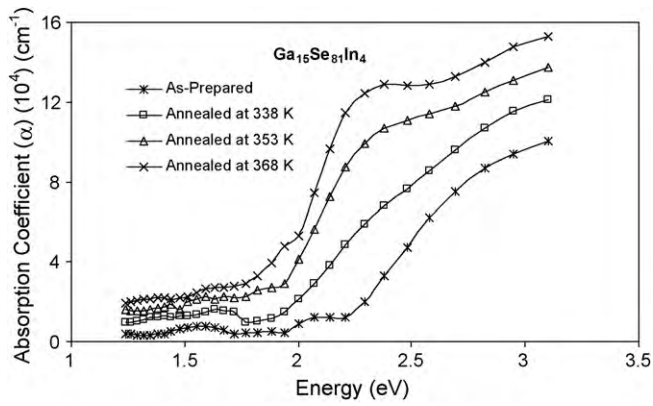


Fig. 3. Absorption coefficient ( $\alpha$ ) against photon energy for as-prepared and annealed thin films of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$ .

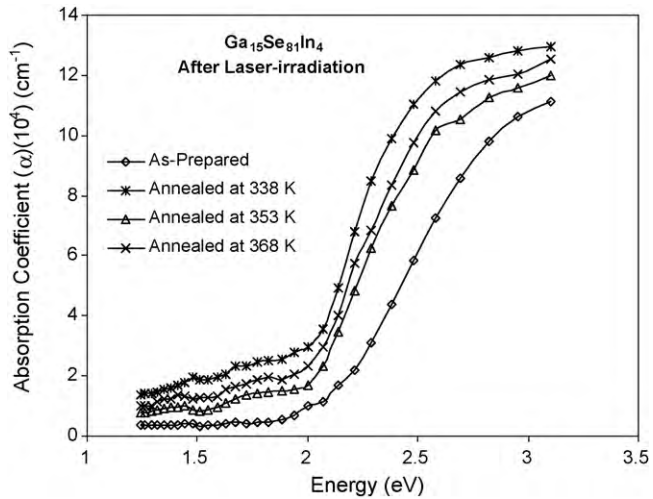


Fig. 4. Absorption coefficient ( $\alpha$ ) against photon energy for as-prepared and annealed thin films of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  (after laser irradiation).

view. Moreover, the refractive index is necessary for the design and modeling of optical components and optical coating such as interference filters. The theory of reflectivity of light has been used to calculate the values of extinction coefficient ( $k$ ) and refractive index ( $n$ ).

The values of extinction coefficient has been calculated using the relation [31–33]:

$$k = \frac{\alpha \lambda}{4\pi} \quad (2)$$

where  $\lambda$  is the wavelength.

The spectral dependence of extinction coefficient ( $k$ ) for  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$ : amorphous and annealed films; and laser irradiated thin films are shown in Figs. 5 and 6.

Table 1

Absorption coefficient  $\alpha$  ( $\text{cm}^{-1}$ ) ( $10^4$ ) in  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  films: as-prepared, annealed and laser irradiated thin films at 620 nm.

Sample	As-prepared	Annealed at 338 K	Annealed at 353 K	Annealed at 368 K
$\text{Ga}_{15}\text{Se}_{81}\text{In}_4$	4.76	7.66	11.07	12.89
$\text{Ga}_{15}\text{Se}_{79}\text{In}_6$	5.61	8.91	12.19	13.24
Laser irradiated as-prepared and thermally annealed thin films				
$\text{Ga}_{15}\text{Se}_{81}\text{In}_4$	3.83	6.23	8.84	9.74
$\text{Ga}_{15}\text{Se}_{79}\text{In}_6$	5.22	7.26	9.83	11.68

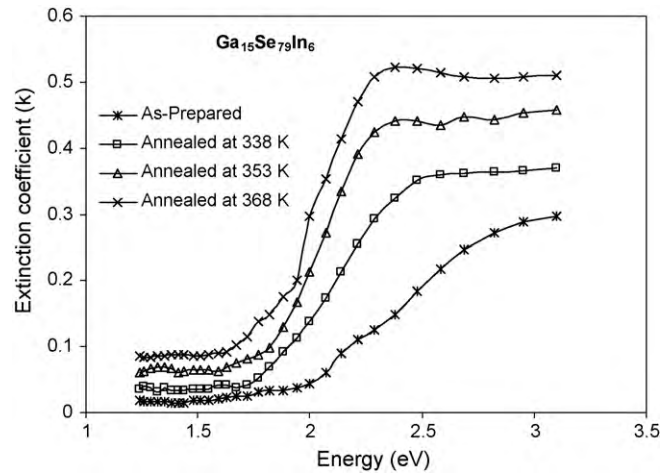


Fig. 5. Variation of extinction coefficient ( $k$ ) with incident photon energy ( $h\nu$ ) for as-prepared and annealed thin films of  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$ .

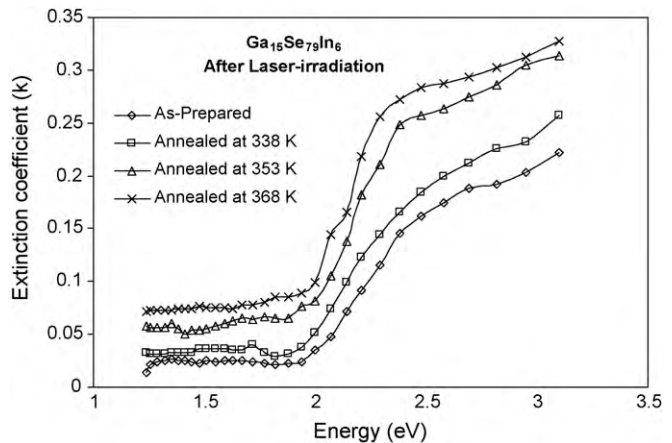


Fig. 6. Variation of extinction coefficient ( $k$ ) with incident photon energy ( $h\nu$ ) for as-prepared and annealed thin films of  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  (after laser irradiation).

The values of refractive index ( $n$ ) have been calculated using the relation: [31–33]:

$$n = \frac{2(1 + R) + [4(1 + R)^2 - 4(1 - R)^2(1 + k^2)]^{1/2}}{2(1 - R)}, \quad (3)$$

where  $R$  is the reflectance or reflectivity.

The variation of refractive indexes ( $n$ ) for  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$ : amorphous and annealed films; and laser irradiated thin films are shown in Figs. 7 and 8.

The calculated values of refractive indexes ( $n$ ) and extinction coefficient ( $k$ ) for  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  thin films (amorphous, annealed and laser irradiated films) are given in Tables 2 and 3.

Table 2

Refractive index ( $n$ ) in  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  films: as-prepared, annealed and laser irradiated thin films at 620 nm.

Sample	As-prepared	Annealed at 338 K	Annealed at 353 K	Annealed at 368 K
$\text{Ga}_{15}\text{Se}_{81}\text{In}_4$	3.44	3.63	3.99	2.81
$\text{Ga}_{15}\text{Se}_{79}\text{In}_6$	3.67	3.38	3.32	3.29
Laser irradiated as-prepared and thermally annealed thin films				
$\text{Ga}_{15}\text{Se}_{81}\text{In}_4$	3.27	2.87	3.66	2.46
$\text{Ga}_{15}\text{Se}_{79}\text{In}_6$	3.48	2.97	3.19	3.07

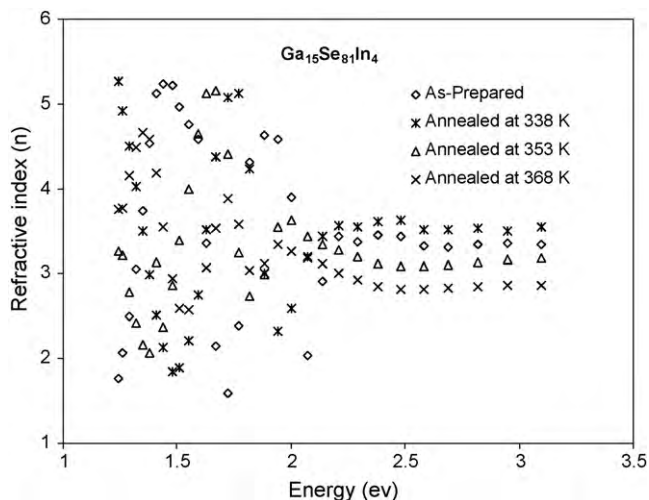


Fig. 7. Variation of refractive index ( $n$ ) with incident photon energy ( $h\nu$ ) for as-prepared and annealed thin films of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$ .

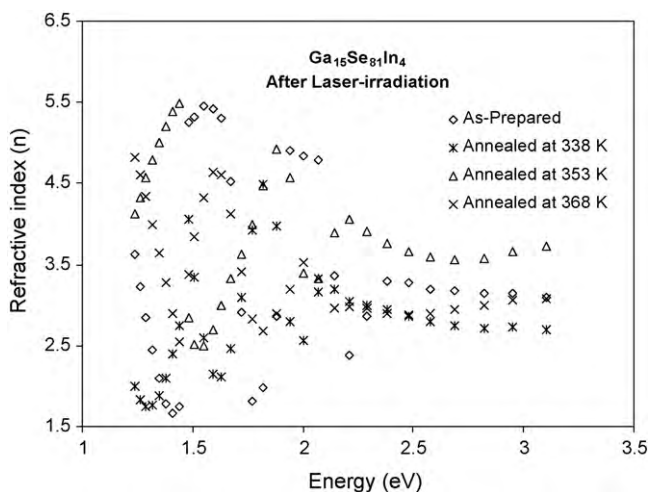


Fig. 8. Variation of refractive index ( $n$ ) with incident photon energy ( $h\nu$ ) for as-prepared and annealed thin films of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  (after laser irradiation).

The variation of  $(\alpha h\nu)^{1/2}$  with photon energy ( $h\nu$ ) for  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  thin films: amorphous and annealed films; laser irradiated films are shown in Figs. 9 and 10. The value of indirect optical band gap ( $E_g$ ) has been calculated by taking the intercept on the X-axis. The calculated values of  $E_g$  for  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  thin films; amorphous, annealed and laser irradiated films are given in Table 4.

Table 3

Extinction coefficient ( $k$ ) in  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  films: as-prepared, annealed and laser irradiated thin films at 620 nm.

Sample	As-prepared	Annealed at 338 K	Annealed at 353 K	Annealed at 368 K
$\text{Ga}_{15}\text{Se}_{81}\text{In}_4$	0.189	0.305	0.457	0.532
$\text{Ga}_{15}\text{Se}_{79}\text{In}_6$	0.184	0.351	0.441	0.521
Laser irradiated as-prepared and thermally annealed thin films				
$\text{Ga}_{15}\text{Se}_{81}\text{In}_4$	0.171	0.283	0.351	0.451
$\text{Ga}_{15}\text{Se}_{79}\text{In}_6$	0.162	0.185	0.257	0.284

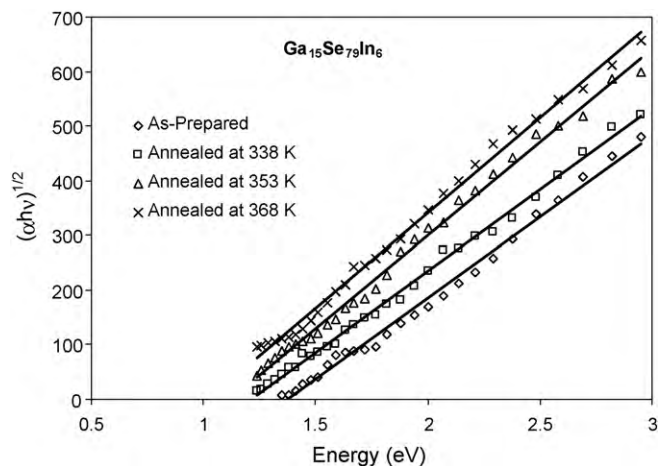


Fig. 9.  $(\alpha h\nu)^{1/2}$  against photon energy ( $h\nu$ ) for as-prepared and annealed thin films of  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$ .

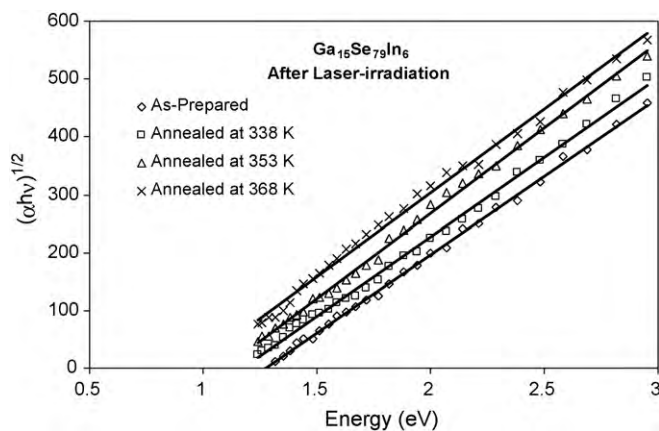


Fig. 10.  $(\alpha h\nu)^{1/2}$  against photon energy ( $h\nu$ ) for as-prepared and annealed thin films of  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  (after laser irradiation).

#### 4. Discussion

In the absorption process, a photon of known energy excites an electron from a lower to a higher energy state, corresponding to an absorption edge. In chalcogenide glasses, a typical absorption edge can be broadly ascribed to one of the three processes, firstly residual below-gap absorption; secondly Urbach tails and thirdly inter-band absorption. Chalcogenide glasses have been found to exhibit highly reproducible optical edges which are relatively insensitive to preparation conditions and only the observable absorption [34] with a gap under equilibrium conditions account for the first process. In the second process the absorption edge depends essentially on the photon energy according to the Urbach relation [35]. In amorphous materials,  $\alpha$  increases exponentially with the photon energy near the energy gap. This type of behavior has also

Table 4

Optical band gap ( $E_g$ ) (eV) in  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  films: as-prepared, annealed and laser irradiated thin films.

Sample	As-prepared	Annealed at 338 K	Annealed at 353 K	Annealed at 368 K
$\text{Ga}_{15}\text{Se}_{81}\text{In}_4$	1.28	1.16	1.02	0.86
$\text{Ga}_{15}\text{Se}_{79}\text{In}_6$	1.39	1.21	1.12	0.99
Laser irradiated as-prepared and thermally annealed thin films				
$\text{Ga}_{15}\text{Se}_{81}\text{In}_4$	1.12	1.04	0.93	0.86
$\text{Ga}_{15}\text{Se}_{79}\text{In}_6$	1.28	1.17	1.09	0.93

been observed in many other chalcogenides [34] and is given by:

$$\alpha \sim \frac{\exp[A(h\nu - h\nu_0)]}{kT}, \quad (4)$$

where  $A$  is a constant of the order of unity and  $\nu_0$  is the constant corresponding to the lowest excitonic frequency.

Absorption of light takes place by the excitation of electrons from the filled state to empty ones. The measurements of transition allow us to determine the absorption coefficient, that is, the number of absorbed photon per incident photon. Clear absorption is observed, showing the dependence of absorption coefficient of amorphous, thermally crystallized and laser-induced films. The variation of  $\alpha$  with photon energy can be explained in terms of: (i) fundamental absorption (ii) exciton absorption and (iii) valance band acceptor absorption. The values of the absorption coefficient ( $\alpha$ ) for  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  for  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  films are in the range  $\sim 10^4 \text{ cm}^{-1}$ , which is consistent with the result of other workers [28–30]. The value of the absorption coefficient ( $\alpha$ ) increases with increasing annealing temperature, while it decreases with laser irradiation. During thermal annealing or laser irradiation, bond breaking and bond arrangement can take place, which result in the change in local structure of the glassy thin films. These include subtle effects such as shifts in the absorption edge, and more substantial atomic and molecular reconfiguration which is associated with changes in optical constants and absorption edge shift.

It is clear from Tables 2 and 3 that the values of refractive index and extinction coefficient both decreases by the exposure of laser irradiation. These decreases in refractive index and extinction coefficient occur due to absorbed heat produced by laser irradiation, as absorbed laser light is instantaneously converted into heat [36,37]. In chalcogenide materials the lone pair orbital forms the valence band, whereas the conduction band is formed by the antibonding orbital [38]. The high-energy radiations excite the electrons from the lone pair and bonding states to higher energy states. Vacancies created in these states are immediately filled by the outer electrons with Auger processes that in turn induce more holes in the lone pair and bonding orbital leading to a vacancy cascade process. In this process, bond breaking or ionization of atoms is easier to occur, which leads to a change in the local structural order of the amorphous network causing a decrease in refractive index and extinction coefficient.

The fundamental absorption edge in most amorphous semiconductors follows an exponential law. Above the exponential tail, the absorption coefficient has been reported to obey the following equation:

$$(\alpha h\nu)^{1/n} = B(h\nu - E_g), \quad (5)$$

where  $\nu$  is the frequency of the incident beam ( $\omega = 2\pi\nu$ ),  $B$  is the constant,  $E_g$  is the optical band gap and  $n$  is an exponent, which can be assumed to have values of 1/2, 3/2, 2 and 3 depending on the nature of electronic transition responsible for the absorption:  $n = 1/2$  for allowed direct transition,  $n = 3/2$  for forbidden direct transition,  $n = 2$  for allowed indirect transition and  $n = 3$  for forbidden indirect transition. The best fit of the experimental results of  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  thin films using Eq. (5), with  $n = 2$  i.e. variation curve of  $(\alpha h\nu)^{1/2}$  with photon energy ( $h\nu$ ) is found to be identical to that of the elemental amorphous semiconductor [28–30]. This indicates that the absorption in  $\text{Ga}_{15}\text{Se}_{81}\text{In}_4$  and  $\text{Ga}_{15}\text{Se}_{79}\text{In}_6$  thin films is due to non-direct transition.

It is evident from Table 4 that the value of optical band gap ( $E_g$ ) decreases with increasing annealing temperature. It also decreases by the influence of laser irradiation on amorphous and annealed thin films. The decrease in optical band gap with increasing annealing temperature and laser irradiation reveals the increase in the crystallinity of compositions [39]. Annealing temperature in between  $T_g$  and  $T_c$  may have enough vibration energy to break some

of the weaker bonds, thus introducing some translational degrees of freedom to the system. Consequently, increase in crystallinity via nucleation and growth become possible [40]. The area occupied by crystallinities increases, where some of them may become interconnected and some may be isolated, this may lead to decrease in optical band gap. The decrease in energy gap and the increase in the width of localized state tails with laser irradiation can be interpreted as a product of surface dangling bonds around the crystallites during the process of crystallization.

Since the optical absorption also depends on short-range order in the amorphous states and defects associated with it, the decrease in optical band gap may be explained on the basis of “density of state model” proposed by Mott and Devis [41]. According to this model, the width of the localized states near the mobility edges depends on the degree of disorder and defects present in the amorphous structure. In particular, it is known that unsaturated bonds together with some saturated bonds are produced as a result of an insufficient number of atoms deposited in the amorphous film. The unsaturated bonds are responsible for the formation of some of the defects in the films, producing localized states in the amorphous solids. The presence of high concentration of localized states in the band structure is responsible for the low values of optical band gap in the case of the amorphous films [42].

## 5. Conclusion

From the above results and discussion, it may be concluded that absorption mechanism is due to indirect transition. The optical band gap decreases with increasing annealing temperatures. The presence of high concentration of localized states in the band structure is responsible for the low values of optical band gap in case of the amorphous films. These low values may be due to the shift in Fermi level whose position is determined by the distribution of electrons over the localized states. The optical constants decrease with the exposure of laser irradiation. The decrease in refractive index and extinction coefficient occur due to absorbed heat produced by laser irradiation, as absorbed laser light is instantaneously converted into heat. Due to the large absorption coefficient and dependence of optical band gap, refractive index and extinction coefficient on annealing temperature and on laser irradiation, these materials may be suitable for optical disk material.

## Acknowledgement

Thanks are due to Deanship of Scientific Research, King Abdul Aziz University, Jeddah, Saudi Arabia (Project No. 3-16/429) for providing financial assistance in the form of research project.

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