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Optical and Structural Studies of Binary Compounds by Explosive Laser Irradiation and Heat Treatment

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1. Introduction

The chalcogens are the name for the periodic table group 16 (old-style: VIB or VIA) in the periodic table. It is sometimes known as the oxygen family. It consists of the elements oxygen (O), sulfur (S), selenium (Se), tellurium (Te), the radioactive polonium (Po), and the synthetic ununhexium (Uuh). Their compounds, particularly the sulfides, selenides and tellurides are collectively known as chalcogenides. Binary compounds of the chalcogens are called chalcogenides (rather than chalcides, which breaks the pattern of halogen / halide and pnictogen / pnictide).

Chalcogenide compounds have the structure mostly composed of segments, chains and molecules. The primary covalent type structure prevailing within the chains of segments in particular a weak one. The materials of low covalent bond strength shows optical transparency in the mid to far IR. While the chalcogenide glasses (Boer & Ovshinsky, 1970; Collins, 1970; Zope M & Zope J, 1984; Afifi et al., 1988; Fadel & El-Shair , 1992 & Fadel et al. ,1992) are regarded as weakly bonded materials in contact to oxide glasses containing both types of heteropolar and homopolar covalent bond viz. Ge-Se, Ge-S etc. and Se-Se, S-S etc. Substantial number of the works on chalcogenide glasses were pointed towards device making games in memory thin film electronic switches (von Allmen, 1987 & Fadel, 1993). Although the applications are manifold yet the necessary structures are still to be understood in the sense of transition between amorphous to crystalline states (Brice,1986). A tendency towards attaining glassy states for ternary chalcogenide glasses increases in the following order



This principle adopted is an indicator that the thin films of Ge-As-S system are comparatively easier than Ge-P-S system. The softening temperature decreases with the increase of atomic number and with the addition of any other element to the host of Ge- Se

and Ge-Te (Katsuyama & Matsumura, 1992) may increase the temperature of softening thereby shifting of the crystalline temperature on to much higher order.

Chalcogenides have wide range of applications in materials science domain. Several structural or photochemical changes have been observed in amorphous elements and compounds viz. through the route of laser treatment for photo-crystallization (Katsuyama & Matsumura, 1992; Mott & Davis, 1979; Andonov, 1982) and heat treatment. Selenium is one such characteristic element, which undergoes unstable morphological transitions from amorphous to crystalline phases. Generally Se thin films grown by evaporation technique, crystallize to pattern resembles like a spherulitic one in hexagonal form compounds of helical chains (Kawarada & Nishina, 1975). During transitions, the different phases of the a-Se films reveal that the structure, growth processes and other properties depend on the method of preparation, deposition conditions (vapor temperature, rate and angle of deposition), physical condition of the substrates and vacuum environment (Kim & Turnbull, 1973 & Gross et al., 1977).

The present work deals with the synthesis of thin films of binary chalcogenide compounds of Ge, Se & Sn elements under explosive laser shots and heat treatments. A number of experimental results were used to explain a new phase generation of the composite materials of chalcogenide. The present attempt is mainly confined with the formation of chalcogenide films and their characterization through Optical Microscopy, SEM, XRD, UV-VIS-NIR etc. In general the chalcogens are viewed as two fold coordinated atoms and can easily be obtained in the form of thin films. Melting, quenching and vapour deposition process are the main tools to fabricate the chalcogenide glasses and alloys. The analysis of the micrographs accumulated by means of SEM and optical Microscopy from the experiment, which are carefully monitored and analyzed.

2. Experimental details

2.1 Importance of vacuum in film preparation

For the preparation of thin film of a material, it is required (a) suitable source to heat the material and convert it from solid to vapour phase (b) suitable chamber at a reduced pressure (c) a supporting material on which the vapour will condense to form a film. If the pressure inside the chamber is not in the order of 10^{-5} to 10^{-6} torr, the deposition is not good. As a result the film often peels out after deposition.

Substrate is the material on which the film is to be deposited. Different materials have been used as a substrates. The common substrate materials are used like glass, quartz, plastic etc. To prepare a good type of cleanliness of substrate is more essential. Temperature of the substrate during deposition is also another factor.

2.2 Cleaning of substrates

To prepare a high quality thin film made of CIS cleaning procedure of substrates glass is more essential (Chopra, 1969). The step by step cleaning procedure is given below.

- i. clean with dilute HCl (1-5%) and rinse the water,
- ii. rub the substrate with brush and a mild detergent,
- iii. wash in double distilled deionnized water several times,
- iv. place in ultrasonic cleaner with the glass slide immersed in water,
- v. boiled them in an electric heater,
- vi. degrease in vapour of isopropyl alcohol in a degreasing chamber fitted to a condenser system.

2.3 Film preparation

Thin films of different specimens have been prepared by adopting thermal evaporation technique through a vacuum coating unit (modified Edward 12EA - 784) as shown in (Fig.1.) under a stable vacuum of the order of 5×10^{-6} torr. on a good quality

E-glass substrates. The vacuum system consists of a rotary pump followed by silicone oil (704 grade oil) diffusion pump. The vacuum measurement is conducted through a Pirani gauge followed by a Penning gauge. The circulation of the chilled water is maintained all throughout the course of the experiment. There is a provision in the evaporator that takes care of the substrates thermal state. The distance between the source and the substrate is optimized to the order of 10 cm in order to prepare good quality uniform thin films.

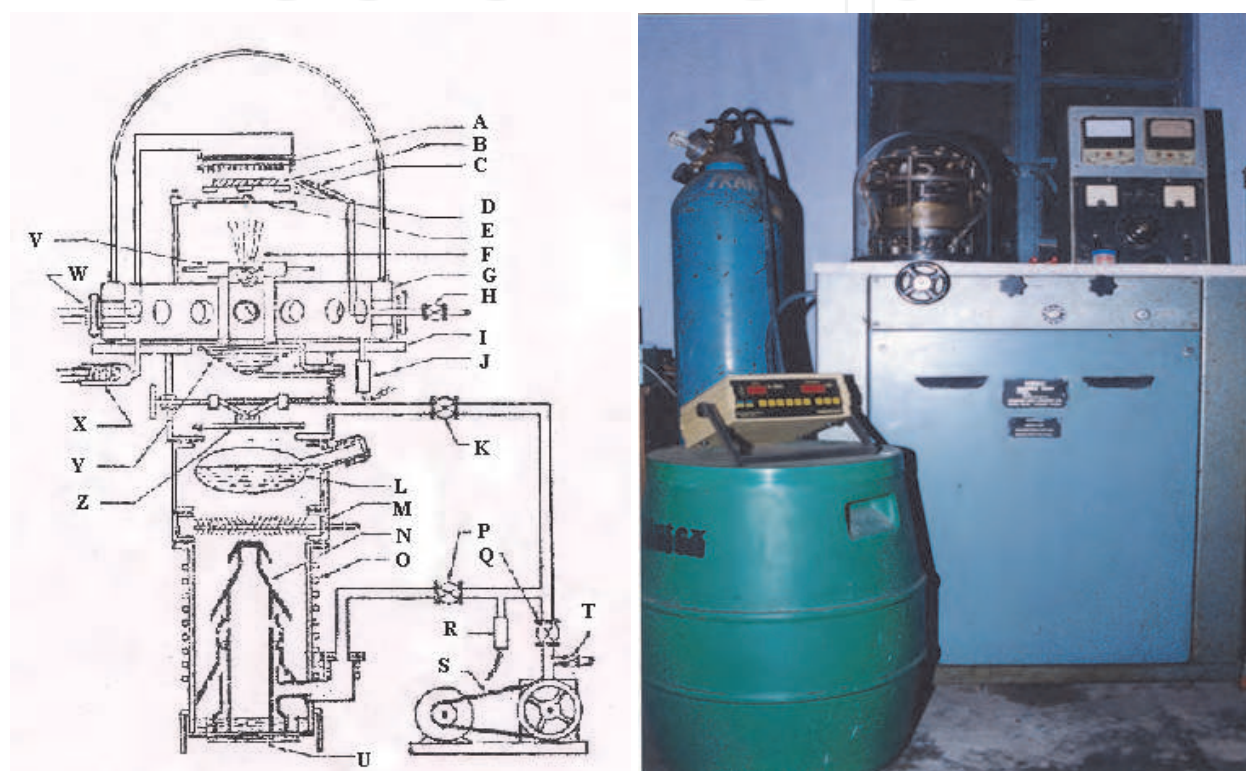


Fig. 1. A typical oil-diffusion pump evaporation station used in the laboratory. The notation stand for A, quartz iodine lamp heater; B, substrate; C, quartz crystal rate controller and deposition monitor; D, substrate mask; E, shutter (mechanical); F, vapour from evaporation source; G, adapter collar between the bell jar and the pump base plate flange; H, air inlet valve; I, base plate flange; J, Pirani or thermocouple gauge; K, roughing valve; L, liquid air trap; M, cooled chevron baffles; N, diffusion pump; O, cooling coils; P and Q, backing valves; R, Pirani gauge; S, fore-pump with air inlet valve T; U, diffusion pump heater; V, filament holders; W, multiple feedthrough; X, ionization gauge; Y, Miessner trap; Z, baffle valve.

The substrates were initially washed in warm soap solution followed by chemical cleaning with anhydrous grade tri-chloro-ethylene, acetone and alcohol followed by CIS ultrasound cleaning. These initial treatments render the occluded materials to be wiped away before the specimen is put in the evaporation chamber for deposition. Different specimens from the chalcogenide groups of various thickness have been deposited on the substrates for both individual and stacked bi-layer samples. The adhesion of the film and its quality is highly

depending on the cleanliness, temperature of the substrate and order of the vacuum during deposition. If the substrate is not properly cleaned or the residual gas inside the chamber is not being sufficiently low during deposition, it has been found that the film often peels out after deposition. After cleaning, the substrate has been baked inside the vacuum chamber to remove the absorbed water vapor and further cleaned with ion bombardment in presence of argon atmosphere. Argon ion cleaning was carried out before the evaporation process started and also the initial sputtering of the source materials are ignored with the help of a movable umbrella placed over the tungsten boat.

Materials used for thermal evaporation of purity grade 5N (99.999 %) from Johnson & Matthey Co. Ltd. The shutter so used is important by which the unwanted atomic depositions were filtered and the heating has been raised with immediate removal of the shutter. The thickness of the film, the rate of deposition and the pre-factors associated with the elements used were monitored with the help of Edward FTM5 quartz crystal monitor unit. Deposition rate of the material on the substrate was in the order of 5 to 8 nm sec⁻¹. After the completion of the deposition, the films were exposed to high purity dry argon atmosphere for quite sometime and then taken out from the chamber, for the photo-inducement and heat treatment.

• Coating Unit	: Edward 12EA-784
• Thickness monitor Unit	: Edward FTM5 Quartz Crystal
• Materials	: Se, Ge, Sn
• Purity grade	: 5N (99.999 %)(Johnson and Mathay Co. Ltd.)
• Substrates	: Glass slides
• Substrates are cleaned with tri - chloro - ethylene, acetone and alcohol.	
• Order of Vacuum maintained	: ~10 ⁻³ Pa.
• Substrate to film material gap	: ~ 10 cm
Shutter arrangement is being interposed between source and the substrate to avoid unwanted material deposition.	
• Ar ion cleaning was initially carried on achieving the requisite vacuum prior to deposition, in order to improve the ultimate vacuum.	
• Deposition rate	: 5 ~ 8 nm sec ⁻¹

Table 1. Summary of deposition Conditions

Films of amorphous Ge, Sn & Se on E-glass substrates have been prepared singularly by vacuum evaporation technique under a vacuum of the order of 5x10⁻⁶ torr. Out of such individual films the two as deposited front surfaces (one combination of Ge & Se and other Sn & Se) are sandwiched by applying small mechanical pressure in order to prevent air pockets at the film interface. The composite films are then placed on a micro-positioner facing Se-substrate interface towards the laser beam. The sandwiched films are then irradiated under pulsed shot through Nd : YAG laser beam for fundamental and 3rd harmonics to form compounds of Ge-Se and Sn-Se.

Several specimens of different films (Se, Ge, Sn, Ge-Se, Sn-Se etc.) are grouped in various ways following their depositing thickness. The deposition rate and thickness were recorded by Edward FTM5 thickness monitoring system housed in the vacuum chamber. All the films were heat-treated and laser treated according to their needs in the experimental process.

2.4 Laser treatment

For laser treatment individual films were sandwiched by applying mechanical pressure to make the interface free from air pockets and then irradiated by pulsed laser output from a third harmonics generator (KDP) of 355 nm UV wavelength pumped by Nd:YAG laser of Spectra Physics Model from one side. Depending upon the requirement one to hundred shots of 20-40 mJ cm⁻² were applied on the composite films.

The full power of the laser pulse is around 50W and the reason for using this amount of power during the irradiation process is to make approximately equivalent heat treatments, otherwise the quality of equivalence under the two different processes may be confused.

In laser-assisted reactions generally, the problems encountered during laser shots on multilayer thin films are the following:

- a. Precise knowledge of absorbance of the optical energy by the films remains unknown. This is because the film absorbance and refractivity are time-dependent phenomenon during the phase transition period.
- b. Conduction of heat depends on time during reaction.
- c. Sticking co-efficient (Bhadra et al., 1994), roughly the diffusion of reactant in the substrate is unknown. The reaction kinetics due to pulsed laser irradiation generally synthesize thin chalcogenide films (Antoniadis & Joliet, 1984). In this way laser pulse shots may have the advantage of making tailored one-dimensional multilayer systems.

2.5 Heat treatment

Individuals and composite bi-layer films were heated under constant temperature for particular time with the help of a temperature controlled furnace and they were quenched to room temperature. For heat treatment, bi-layer of Ge-Se and Sn-Se films and individuals of Ge, Sn, Se films were heated under constant temperature of 100 °C, 150 °C etc. for 30 minutes, 45 minutes with the help of a temperature controlled furnace and they were returned to room temperature by decreasing the temperature in steps of about 1/ 2 °C per minute.

2.6 Measurements

Microstructures of treated and untreated films have been studied through polarizing optical microscope [OLYMPUS – KH (MLX – TR) model]. Some representative SEM micrographs of treated samples have been carried out with the help of Jeol SEM, Model JSM 5200. X-ray diffraction pattern of different specimens were carried out with model BRUKER-aXS-D8 Advance of Cu K- α irradiation of monochromatic wavelength 1.5406 Å. UV-VIS-NIR transmission spectra were yielded by a spectrophotometer (Shimadzu UV – 3101PC).

3. Results and discussions

The present attempt is mainly confined with the formation of chalcogenide films and their characterization through Optical Microscopy, SEM, XRD UV-VIS-NIR etc. In general the chalcogens are viewed as two fold coordinated atoms and can easily be obtained in the form of thin films. Melting, quenching and vapour deposition process are the main tools to fabricate the chalcogenide glasses and alloys. The analysis of the micrographs accumulated by means of SEM and optical Microscopy from the experiment, which are carefully monitored and analyzed. It was known in the 80's and authenticated by Mott & Davis, 1979 that under rapid cooling of chalcogenides on melting by Laser bombardment can yield material, which can be achieved in amorphous form.

3.1 Morphological study

Microstructures (**Fig. 2 & 3**) of treated and untreated films have been studied through polarizing optical microscope [OLYMPUS – KH (MLX – TR) model].

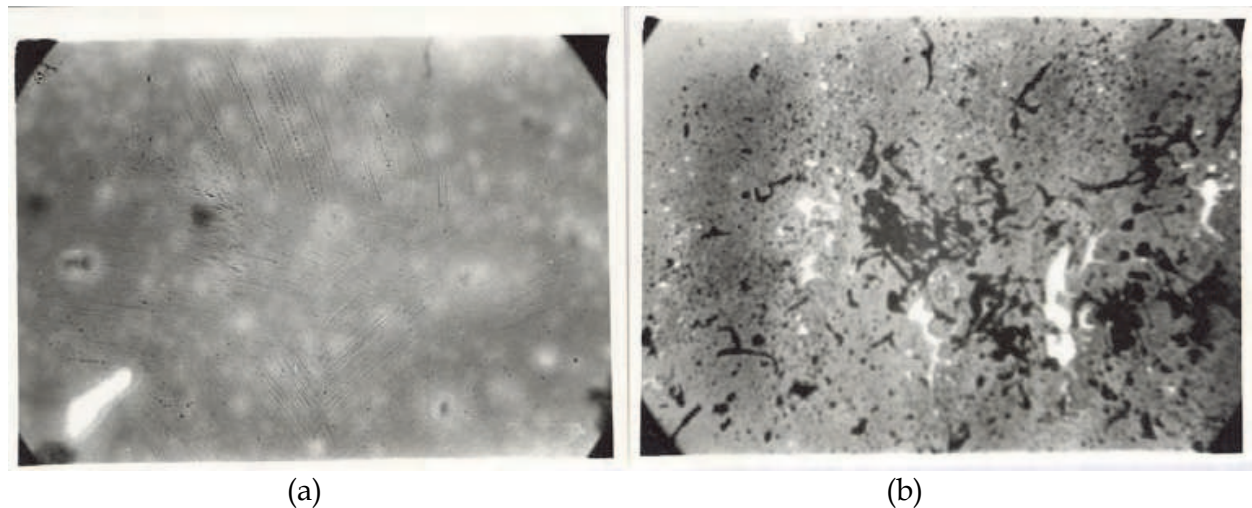


Fig. 2. Optical Micrographs (a) GeSe Film (stacked), (b) GeSe Film (sandwiched).

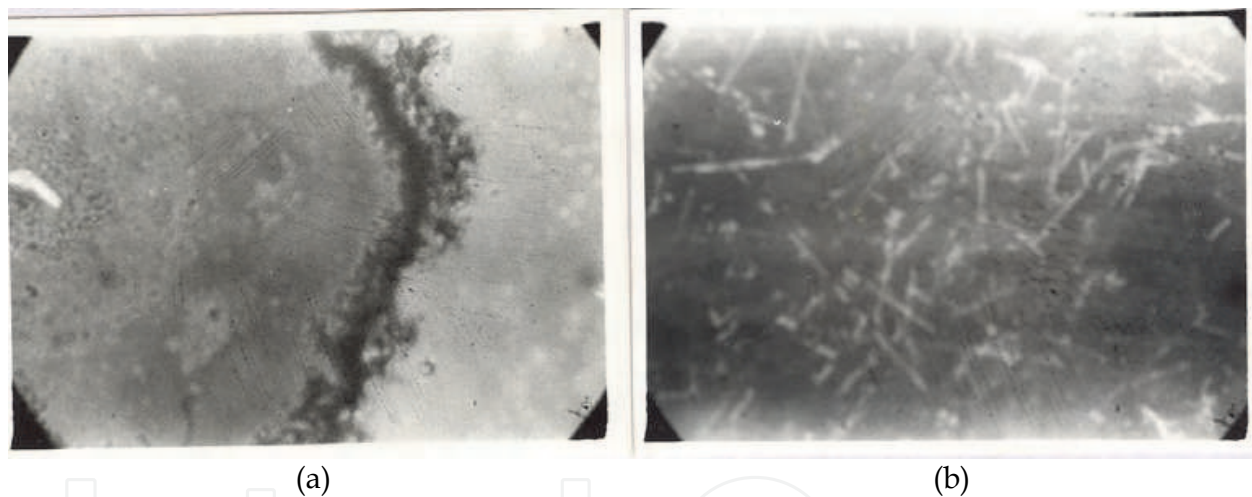


Fig. 3. Optical Micrographs (a) SnSe Film (stacked) and (b) SnSe Film (sandwiched)

The dendritic feature observed in **Figs. 4 - 8** arises from the differences in the solute content at a faster rate than the movement of the solidification isotherms (Bhadra et al., 1994; Griffiths et al., 1969 & Bhadra et al., 1998). The flow initially dispersed in many paths since the flowing warm material starts to dissolve the already solidified materials. The face growth rate lags and the corner growth increases to maintain the growth rate more or less steady. In order to attain the forced growth rate, the corner penetrates further while the faces grow slowly in depleted part of the material in the film (Brice, 1986; Antioniadis & Joliet, 1984; Griffiths et al., 1969; Bhadra et al., 1998 & Pamplin, 1980) when Se film heat-treated at 150 °C and returned to room temperature, the warm Se cools on a highly polished substrate, the stem of the dendrite and the branches grow from its sides which are directed along the fast growing crystallographic axes. The symmetry of each flake may be due to hexagonal symmetry of Se and the substrate topology. Morphological instability occurs when the film subsequently heat-treated at higher temperature.

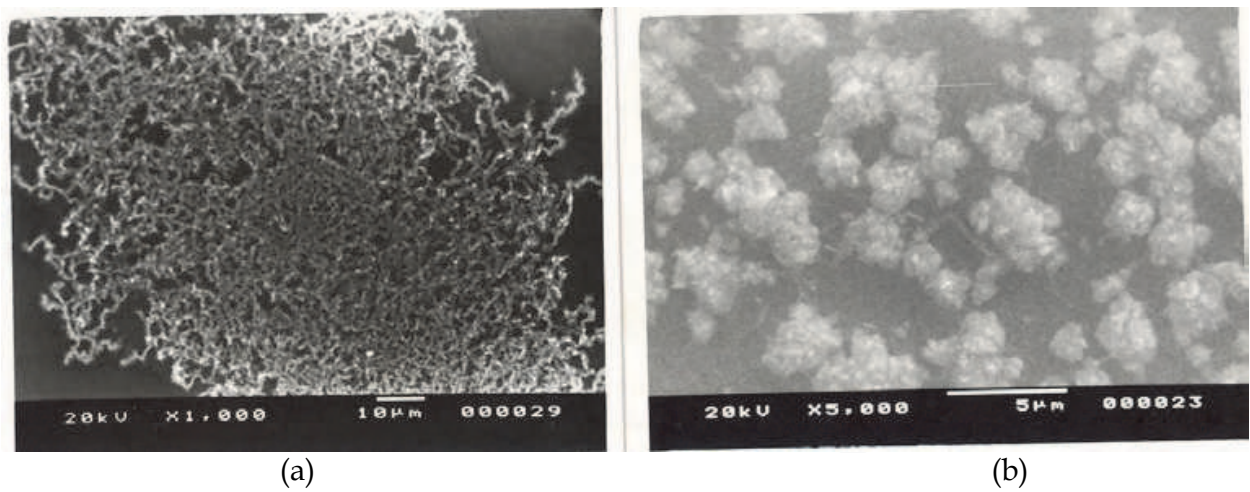


Fig. 4. Scanning Electron Micrographs (SEM) (a) Se Film (Heat treated) and(b) Se Film (as deposited).

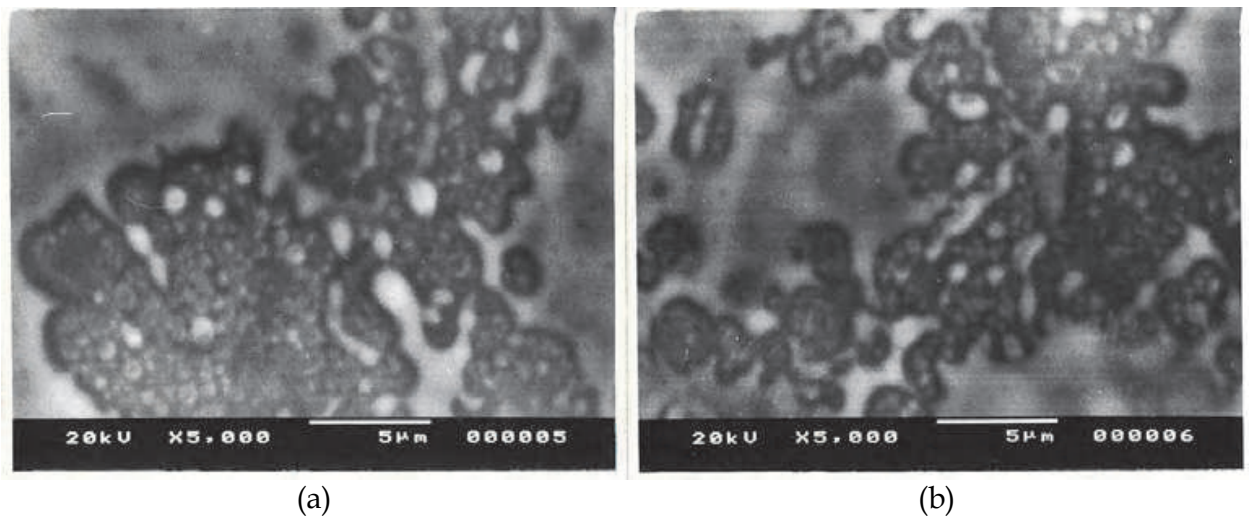


Fig. 5. Scanning Electron Micrographs (SEM) (a) & (b) GeSe Film (Heat treated)

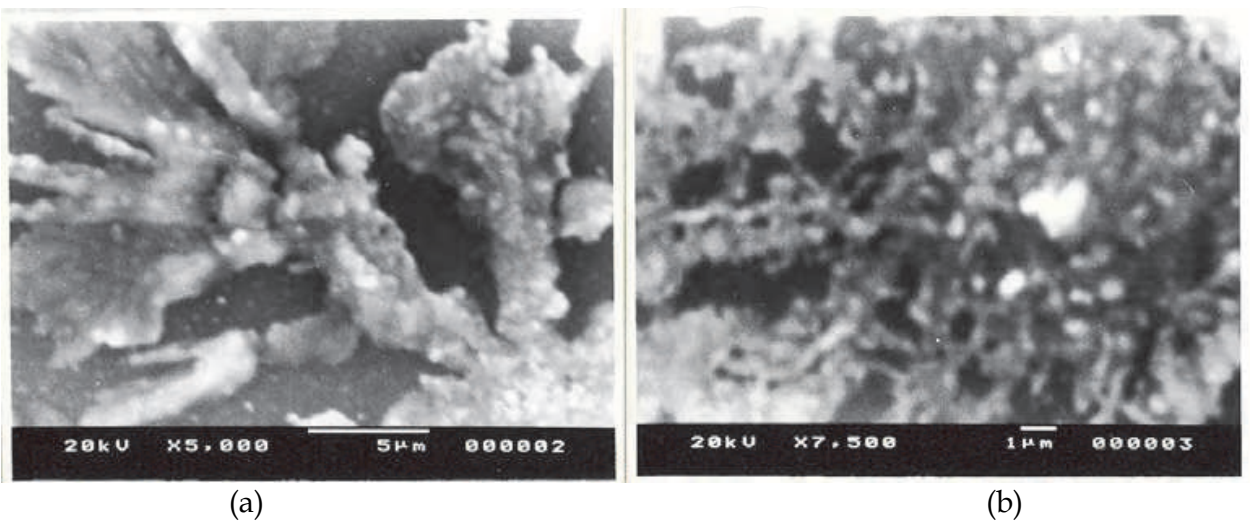


Fig. 6. Scanning Electron Micrographs (SEM) (a) & (b) SnSe Film (Heat treated).

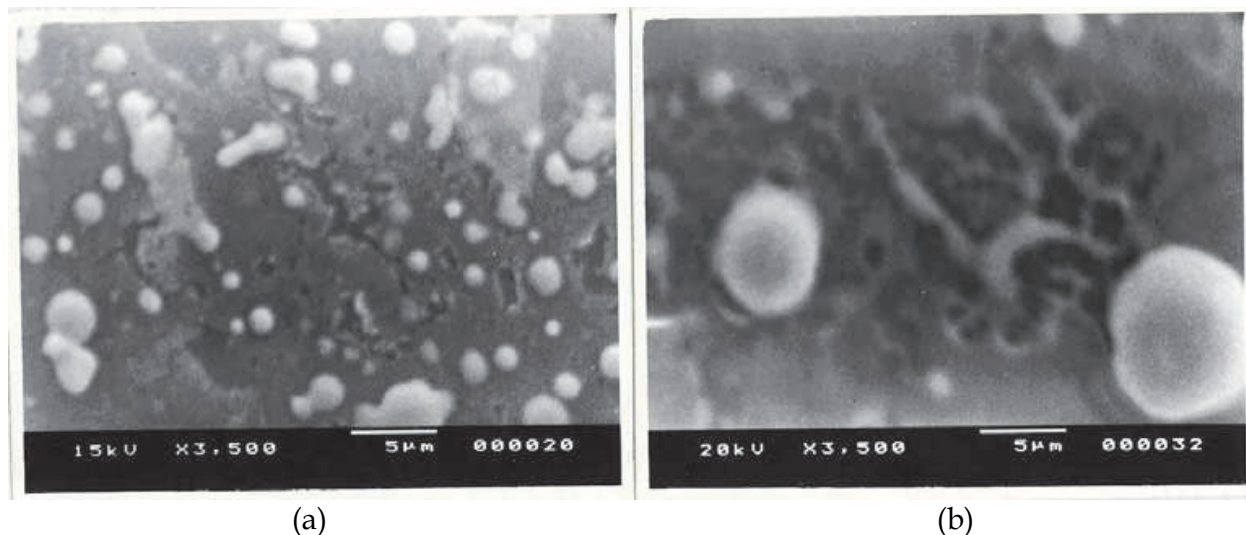


Fig. 7. Scanning Electron Micrographs (SEM) (a) & (b) GeSe Film (Laser treated)

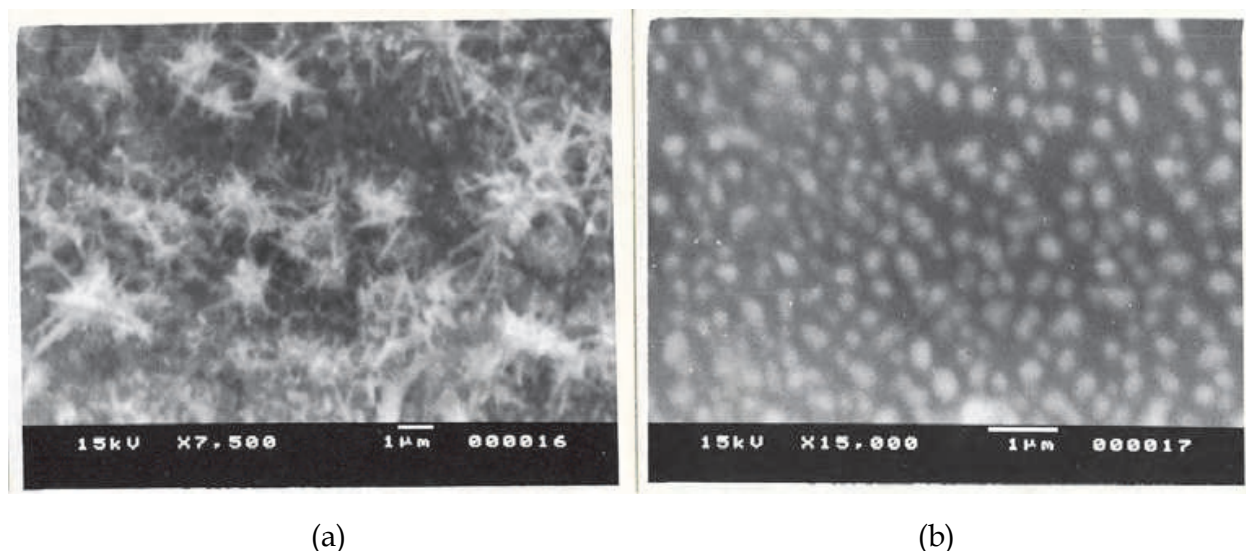


Fig. 8. Scanning Electron Micrographs (SEM) (a) & (b) SnSe Film (Laser treated).

It has been observed that if the materials of Ge be deposited over Se film or Sn be deposited over Se the photo micrographs show practically a little change in structure. In the event of singular deposition of Sn & Se and sandwiched and then laser shots be made through Se in front some changes are found even the shots are not optically compatible that is to say fundamental beam. The difference in the explosive system is clearly observed through SEM studies and it is concluded that third harmonics generated a picture, which is optically compatible and the rest is equivalent to the simple heating process. The work on such explosive laser irradiation is found to be very important towards the appropriate formation of compounds.

3.2 Crystallographic study

The structural changes on various types of vapour grown amorphous films during both laser and heat treatments have been analyzed by XRD. Two significant peaks have been noticed at 2θ values 23.43° and 41.37° for both the films under treatments as mentioned above in the XRD

(Fig. 9) peaks corresponds to (100) and (110) diffraction planes and are resembling to that of hexagonal patterns of Se. In case of heat treated binary films the diffraction peaks corresponds to Ge-Se (201), Sn-Se {(042) & (212)} were clearly observed. Experimental results from XRD pattern were given in **table - 2**. The corresponding XRD data i.e. the inter-planar distance d and (hkl) plane are verified to the standard JCPDS data [JCPDS data. ID nos. 42-1425 (Se), 18-0549 (Ge), 19-1365 (Sn), 15-0404 (GeSe) and 35-1042 (SnSe)].

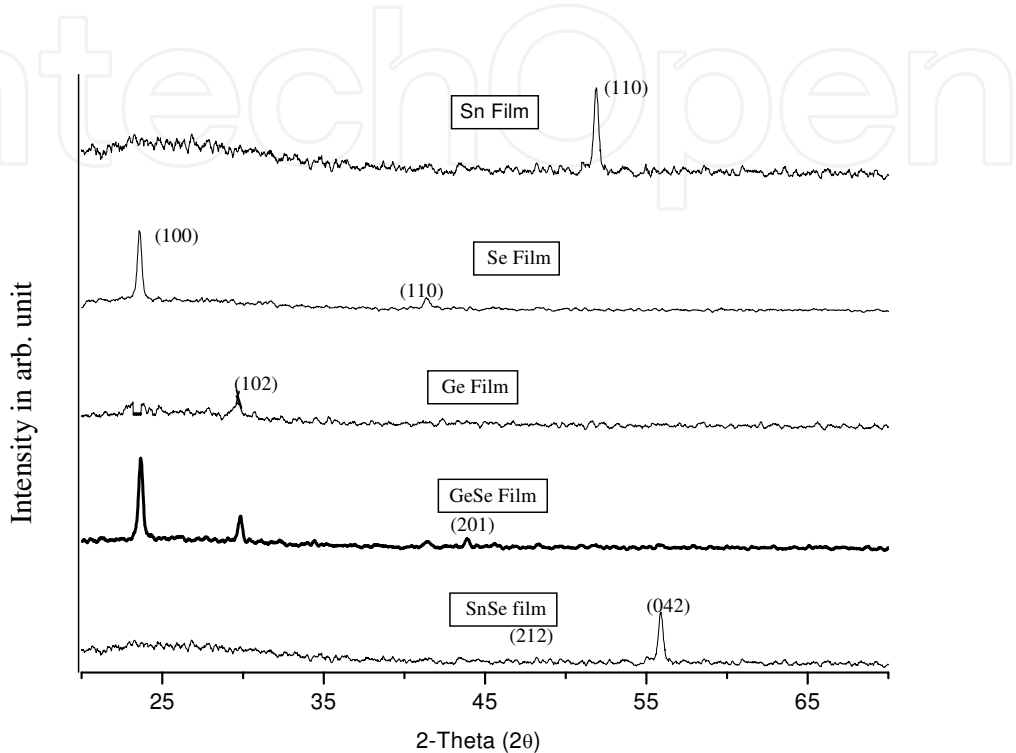


Fig. 9. XRD pattern of different films

Specimen	d-values from JCPDS (Å ^o)	2θ in degree	d-values observed (Å ^o)	(hkl)
Se	3.77	23.43	3.79	(100)
	2.18	41.37	2.17	(110)
Ge	3.01	29.84	3.01	(102)
Sn	2.61	51.96	2.62	(110)
GeSe	2.14	43.96	2.13	(201)
SnSe	1.85	49.17	1.81	(212)
	1.73	55.61	1.70	(042)

Table 2. Experimental results from XRD pattern

The changes in the thermally treated specimens are much faster than those observed in case of laser treatment (Kar et al.,2007). This is due to the fact that the optical matching of these materials are tantamount with the third harmonics of the Nd : YAG pulsed laser in that zone when the energy is mostly utilized under lasing action. The behaviors of the laser treated

with third harmonics irradiation in Ge-Se are the example of yielding faster results. Thus, it is imperative to activate the specimens as per the optical fundamental peaking or else the process will not generate the expected results.

3.3 Optical study

The optical transmission spectra before and after laser irradiation of Ge-Se films and Sn-Se films are shown Fig. 10-12. The transmission plot has been grouped into transparent, weak and medium absorption zones to find the optical constants. Using Manificier et al.,1976 and Swanepoel, 1983, the transmission T is a complex function

$$T = f(\lambda, s, n, d, \alpha)$$
 (1)

Where *s* is the refractive index (RI) of the substrate, *n* is the RI of the film, *d* is the film thickness and *α* the absorption coefficient.

The refractive index value *n* is given by the expression

$$n = [N + (N - s)^{0.5}]^{0.5}$$
 (2)

where

$$N = 2s \frac{(T_M - T_m)}{T_m T_M} + \frac{(s^2 + 1)}{2}$$
 (3)

and *T_m*(λ) and *T_M*(λ) are the envelopes of the minima and maxima in the transmission spectra (Fig.10).

By using Equation 2 the refractive index as well as the thickness were calculated and verified with the experimental data.

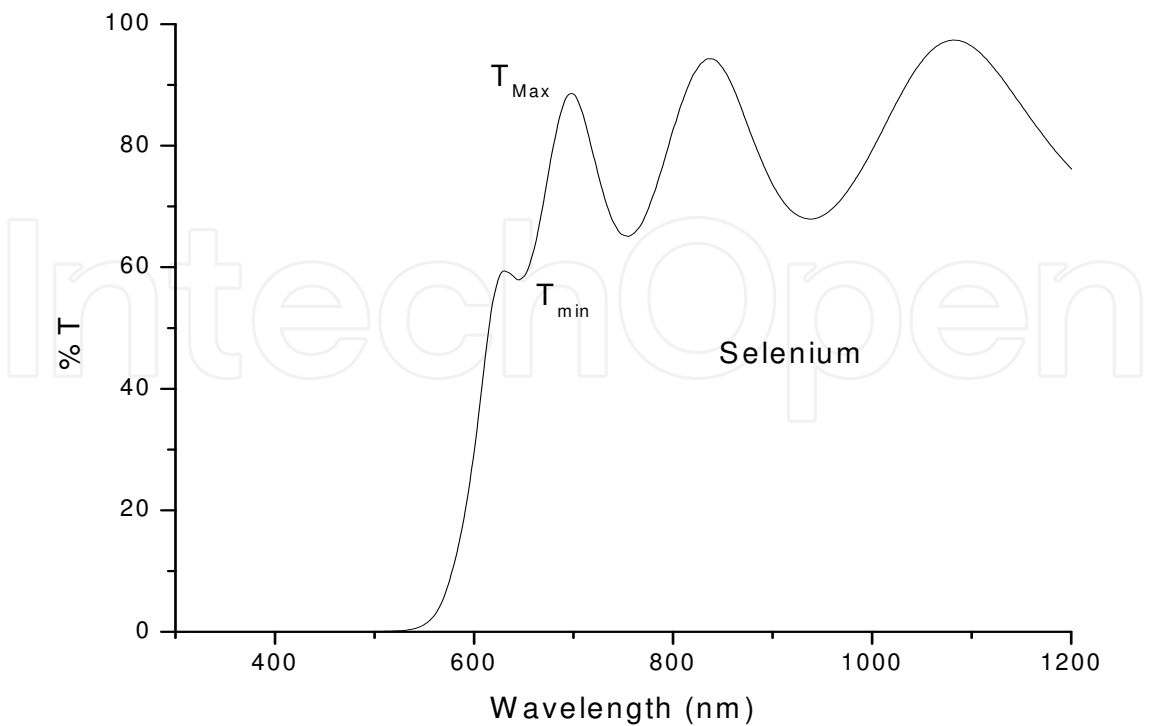


Fig. 10. UV-VIS-NIR transmittance (% T) versus wavelength plot of typical Se film

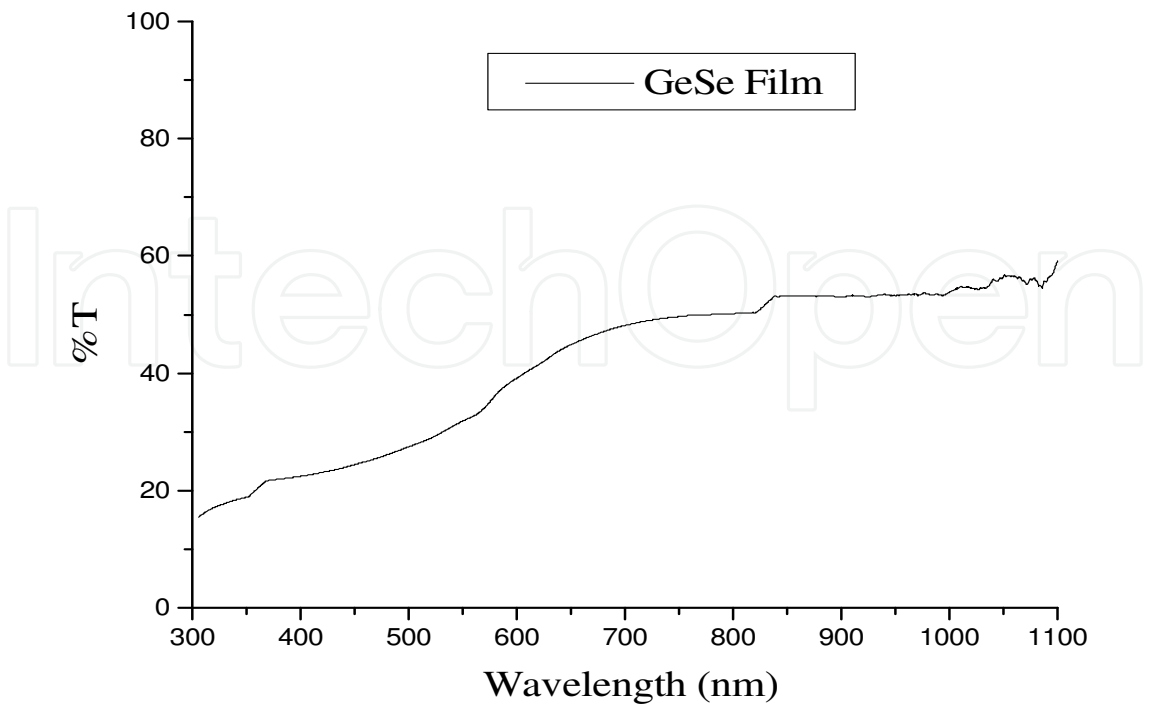


Fig. 11. UV-VIS-NIR transmittance (% T) versus wavelength plot of typical GeSe film

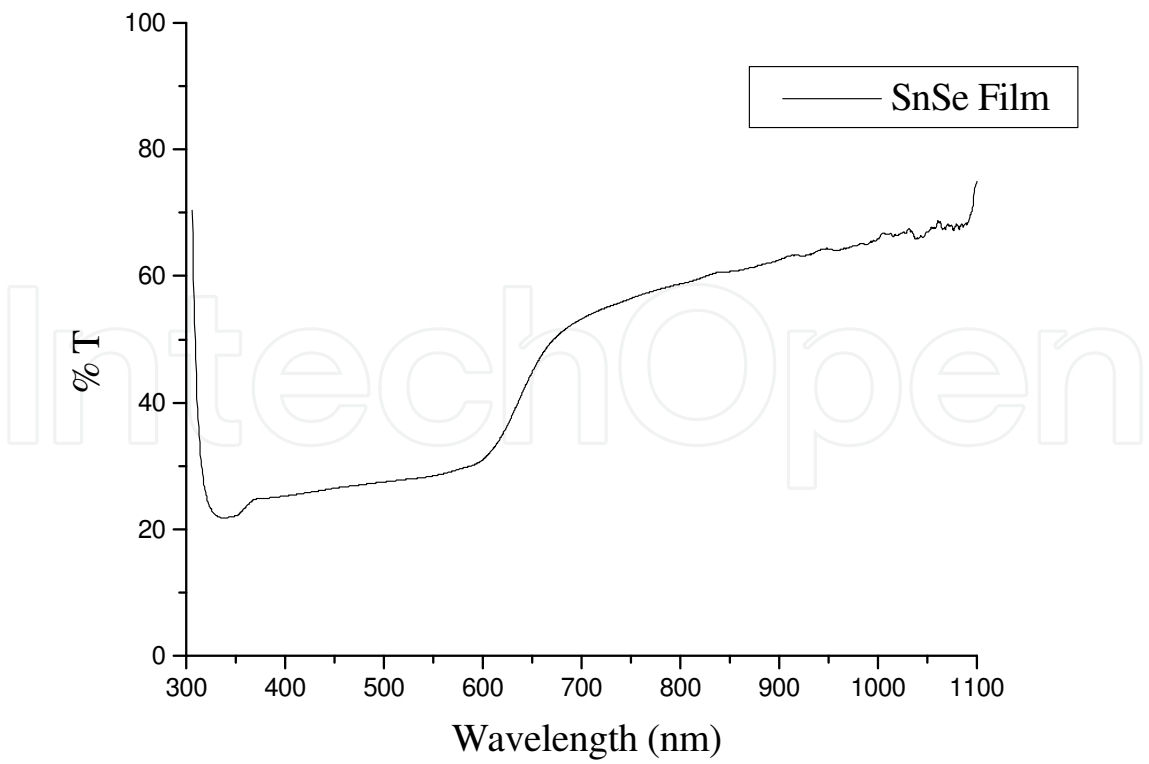


Fig. 12. UV-VIS-NIR transmittance (% T) versus wavelength plot of typical SnSe film

The optical absorption coefficient (α) of the films was calculated using the formula

$$\alpha = d^{-1} \ln \left(\frac{I_0}{I} \right) \tag{4}$$

Where d and I are the sample thickness and transmitted intensity, respectively, and I_0 is the effective incident photon intensity (Bhadra et al., 1999).
The UV-VIS-NIR transmission spectra were carried out through a spectrophotometer (Shimadzu UV-3101PC) for the films deposited on glass substrates. The optical band gap was calculated from the Tauc, 1974 relation;

$$(\alpha h\nu)^{1/2} = A (h\nu - E_g) \tag{5}$$

where A is a constant and $h\nu$ is the photon energy, and α the absorption coefficient. The graph showing $(\alpha h\nu)^{1/2}$ versus $h\nu$ is shown in Fig. 13. From the intercept on the $h\nu$ axis $(\alpha h\nu)^{1/2} = 0$ gives the band gap energy E_g which comes out for Ge-Se to be **1.45 eV** and for Sn-Se **1.62 eV**. The variation of absorption coefficient (α) versus $h\nu$ plot is shown in Fig. 14. The optical absorption edge (Tauc, 1974 & Connell, 1979) is usually described as having three different regions. At high energies, and, for $\alpha > 10^4 \text{ cm}^{-1}$ Eq. (4) is valid for evaluating the optical band gap. In Eq. (4), A contains an average matrix element, constant with energy, and joint density of states for the conduction and valence bands. The equation is formulated on the basis of a square root dependence of the density of states on energy in both bands. The selection rule for the crystal momentum is broken by disorder and so the value of A is much greater than in the crystal. The energy gap in an amorphous material is empirically defined as E_0 of the above-mentioned state.

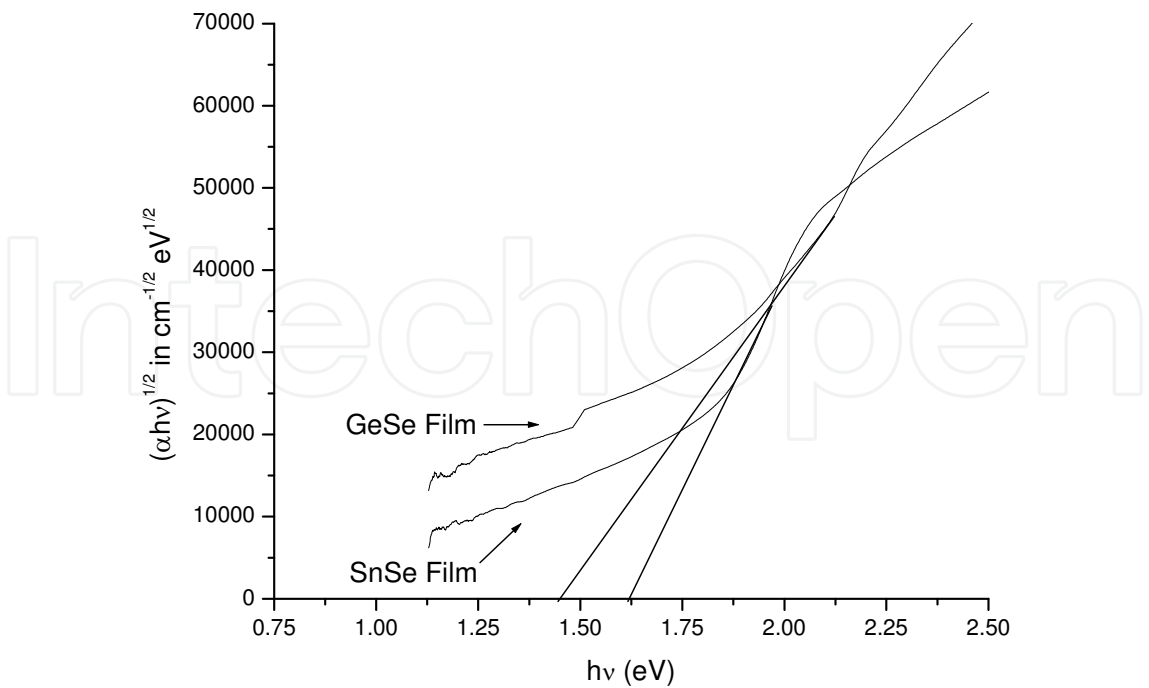


Fig. 13. Variation of $(\alpha h\nu)^{1/2}$ versus $h\nu$ plot

Experimental data on heat-treated films were recorded in the higher absorption region, and then the best fit least squares method were made to fit in the Tauc plot. The transition from the amorphous to crystalline state was realized on heat treatment. Thus the extrapolation is made on a very small region of the absorption energy without incorporating any possible error.

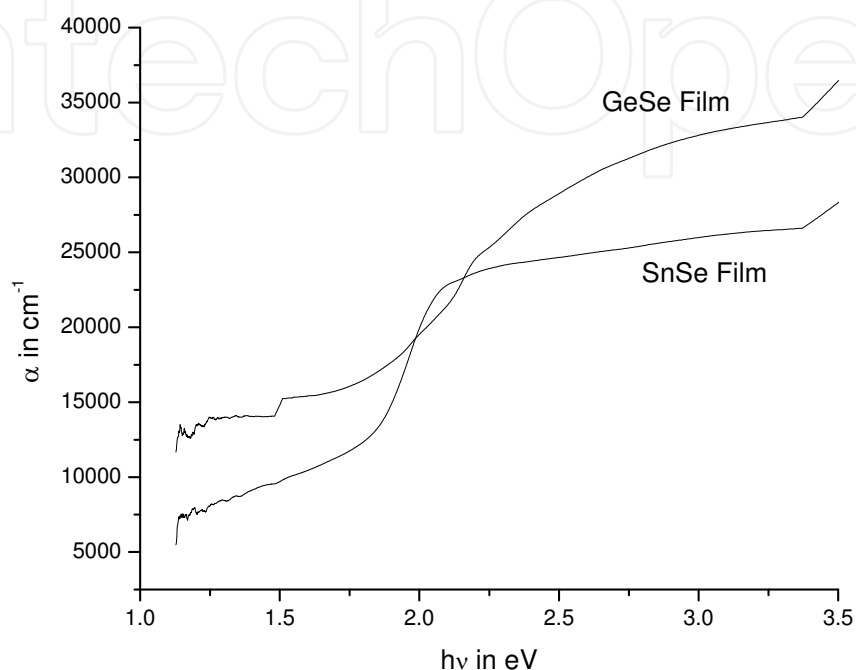


Fig. 14. Variation of absorption coefficient (α) versus $h\nu$ plot

The changes in the thermally treated specimens are much faster than those observed in case of laser treatment. This is due to the fact that the optical matching of these materials is related to the third harmonic of the Nd:YAG pulsed laser in that zone where the energy is mostly utilized under lasing action. The behavior of the laser treated with third-harmonic irradiation in Ge-Se is an example of yielding more rapid transitions. Thus, it is imperative to activate the specimens as per the optical fundamental peaking or the process will not generate the expected results.

4. Conclusions

The process of preparation of thin films of mono-system and also binary chalcogenide alloys and there various structural and morphologies have thoroughly been studied and lots of interesting information was gathered to explain a new phase generation of the composite materials. The typical molecular nature of chalcogenide alloys give rise to a subtle changes in optical properties by photo induction. As the semiconductor Se is photo - sensitive with a lower melting temperature than Ge and Sn; so it is possible to diffuse Se into these elements by an adiabatic impulsive energy. The short range ordering structure of chalcogens when mixed up with four fold-coordinated atoms like Ge, Sn etc. produce alloy or compound with certain specific signature. The optical band gaps calculated from the Tauc plot were found to

be 1.45 eV for Ge-Se and 1.62 eV for Sn-Se and they behave like semiconductors. The laser impulse on those materials proves an advantage over the other adopted methods because of the fact that the instant quenching under laser irradiation pave the path for the materials to be alloyed properly. The disadvantage in laser induction is that the large-scale compound preparation may be a different proposition.

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Nanocomposites with Unique Properties and Applications in Medicine and Industry

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This book contains chapters on nanocomposites for engineering hard materials for high performance aircraft, rocket and automobile use, using laser pulses to form metal coatings on glass and quartz, and also tungsten carbide-cobalt nanoparticles using high voltage discharges. A major section of this book is largely devoted to chapters outlining and applying analytic methods needed for studies of nanocomposites. As such, this book will serve as good resource for such analytic methods.

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