Photoinduced Phenomena in Thermally Evaporated a-Ge_x Se_{90-x} Sb_{10} Thin Films

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Abstract: Amorphous Ge_x Se_{90-x} Sb_{10} thin films were prepared by thermal evaporation under vacuum onto glass substrates. Reflectance and transmittance were measured in the wavelength range 190-900nm. The optical properties of the as deposited and UV-irradiated films at different exposure times were reported. The compositional dependence of the optical constants (absorption coefficient, the non-direct optical gap E_g, refractive index (n), and the extinction coefficient (k) were evaluated and discussed in terms of the Ge content and the chemical bond network model.

Keywords: Amorphous thin films, photoinduced phenomena, optical constants.

1. INTRODUCTION

Chalcogenide glasses have received a special attention due to their wide range of applications in various solid state devices such as switching, memory, image converters, biosensors and optical mass memories [1-7]. Chalcogenide glassy films are subjected to many systematic researches because of the changes of their physical and chemical properties, which occur in these materials after illumination or annealing [8-10]. These photo–and thermally induced effects which may be either reversible or irreversible, allow the possibility of using amorphous chalcogenide semiconductors for technological applications such as high-density information storage, high resolution display devices and fabrication of diffractive elements [11-14]. Photo-and thermally induced phenomena in amorphous $Ge_{30}S_{60}Sb_{10}$ films were investigated by Vlćek et al. [15, 16]. They found that, the changes of optical properties in these films are assigned to the change of homopolar bond densities. Group IV, V and VI (chalcogenide) network glasses, which allow the tuning of structural flexibility by composition, display variety of both transient [17] and metastable photo induced phenomena including photo-darkening [18, 19], photo-bleaching [20] and photo-structural change [21]. Recently photo-bleaching was studied in Ge-Sn-Se thin film by Florescu et al. [22]. The present study aimed to study the effect of film illumination with above band gap UV light on the optical properties of $Ge_xSb_{15}Se_{85-x}$ (≤ x ≤) thin films.

2. EXPERIMENTAL

Different compositions of $Ge_xSb_{15}Se_{85-x}$ (≤ x ≤) in bulk and thin films were prepared as well As detailed here [23]. The film thicknesses were controlled to be in the range 250-300nm. The amorphous structure of bulk ingots was confirmed using both X-ray diffraction analysis (XRD) and differential scanning calorimetry (DSC). Optical transmittance and reflectance spectra were measured in the 200-900nm spectral range using Shimadzu 2101 UV-Visible double beam spectrometer. The UV exposure time was taken as 10, 20, 30, 40 and 50min. The incident power was 20 mW/cm_2.

3. RESULTS AND DISCUSSION

3.1. Photo Induced Phenomena in $Ge_xSb_{10}Se_{90-x}$ Thin Films

The photo-darkened effects was found to be accompanied by structural changes, which lead to change in the film transmitivity, reflectivity, refractive index (n) and red shifts in the optical absorption edge. The effect of illumination time (10, 20, 30, 40 and $50_{min.}$) on the optical properties of $Ge_xSb_{10}Se_{90-x}$ thin films where x=30 at% (as an example) have been investigated as shown in Figure **1**.

The observed decrease in the film transmission with the exposure time as shown in Figure **1** indicates the photodarkening (i.e. red shift of E_g^{opt}). A photoinduced change is a structural phenomenon related to the flexible nature of the amorphous network [24]. In the present study, the observed photo darkening in $Ge_xSb_{10}Se_{90-x}$ films. The absorption coefficient (α) was calculated from the measured film transmission (T) and reflection (R) using the following relationship [23].

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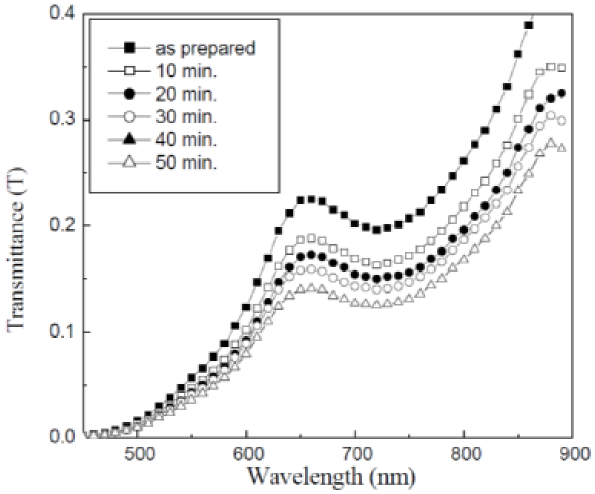

Figure 1: Transmittance spectra for as-prepared and illuminated Ge30Sb10Se60 thin films.

$$\alpha = d^{-1} ln ((1-R)/T) \qquad (1)$$

where d is the film thickness. The variations of the absorption coefficient with photon energy (hv) near the absorption edge obey Tauc's relation in the form of [25]:

$$\alpha(\lambda).hv = B(hv - E_g)^r \qquad (2)$$

where hv is the photon energy, E_g is the optical band gap, B is an energy independent constant (band edge steepness parameter in Tauc's equation) and r is an integer or half integer that determine the nature of optical transition (r =1/2 for direct and r =2 for nondirect transitions). Figure **2** indicate that the plots of $(\alpha hv)^{1/2}$ versus hv for $Ge_{15}Sb_{10}Se_{75}$ thin films according to the power law[126] $\alpha h\nu = B(h\nu - E_g^{opt})^r$ where r = 2 for non-direct allowed optical transition are successfully to describe the optical transition in these films.

It is found that, optical absorption data successfully described by Tauc's relation for indirect optical transitions $(\alpha hv)^{1/2} = \sqrt{B}(hv - E_g)$. The optical band gap E_g^{opt} is obtained from the intercept of $(\alpha hv)^{1/2} = 0$ vs hv straight portion with hv axis at $(\alpha hv)^{1/2} = 0$. Figures **3** and **4** represent the dependence of the optical gap E_g^{opt} and the induced potodarkening $\Delta E(E_g{}^{opt}{}_{virgin} - E_g{}^{opt}{}_{ill})$ on the illumination time for the studied thin films. This two parts of the Figure. Shows that E_g^{opt} decreases whereas ΔE increases with increasing the expuser time. Furthermore, ΔE decrease as Ge content increase up to Ge content 25 at% (Z=2.7), hence it decrease with further increase of Ge content upto 30 at%. The photo-darkening process in $Ge_XSb_{10}Se_{90-X}$ thin films may be attributed to one or more of the following mechanisms:

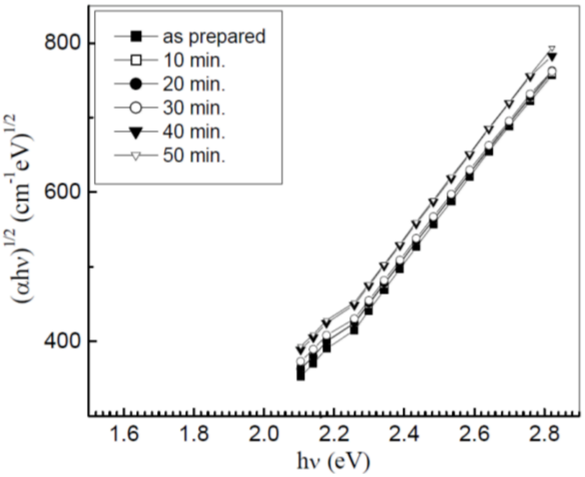

Figure 2: The plots of (αhν)1/2 vs. hv for as-prepared and illuminated Ge15Sb10Se75 thin films.

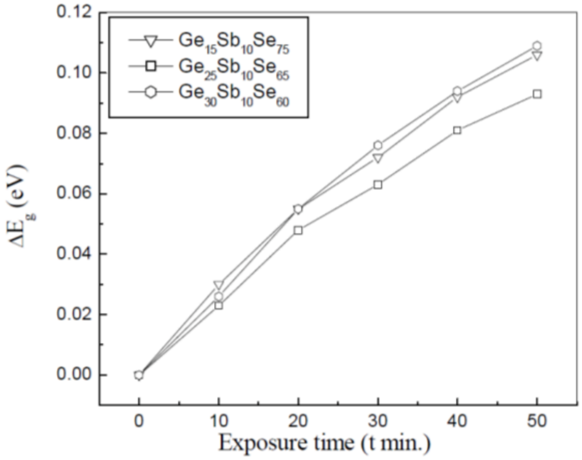

Figure 3: The plots of ΔEg Vs. exposure time for GexSb10Se90-x (x = 15, 25 and 30 at.%) thin films.

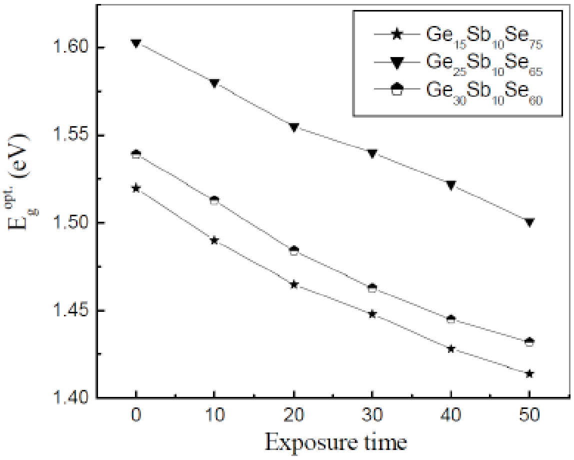

Figure 4: The plots of Eg opt. Vs. exposure time for GexSb10Se90-x (x= 15, 25 and 30 at.%) thin films.

(1) The observed photo darkening in as-evaporated $Ge_xSb_{10}Se_{90-x}$ films illuminated by above band gap light

can be explained according to the simple transformation reaction:

$$2|M - S| \Leftrightarrow M - M + S - S \qquad (3)$$

Proposed by Vlcek *et al*. [16] to explain the photo and thermally induced effects in $Ge_{30}Sb_{10}S_{60}$ films. Where M denotes the atom of metallic element (Ge, Sb) and S refers to the chalcogenide atoms. In this reaction heteropolar bonds Se-Sb, Ge-Sb and Ge-Se can be transformed into homopolar bonds such as Ge-Ge, Se-Se and Sb-Sb and vice versa.

The increase of dangling bonds and homopolar bonds that creates localized states at the edge of the conduction and/or valence band results in the red shift (photo-darkening) of the absorption edge. Then in the present case we assume that the above structural transformation, takes place from left to right to produce the photo-darkening in $Ge_xSb_{10}Se_{90-x}$ thin films. In other way, the observed photo-darkening in $Ge_xSb_{10}Se_{90-x}$ thin films can be attributed to the photo-darkening transformations of heteropolar bonds Se-Sb, Ge-Sb and Ge-Se to homopolar bonds Se-Se, Sb-Sb and Ge- Ge.

(2) According to the macroscopic model suggested by Shimakawa *et al*. [26] the structure of chalcogenide glass is considered as a layer-like one in which electrons and holes become excited when the glass is illuminated by band gap light. Due to the diffusion of holes (with mobility higher than electrons), the space charges accumulate in the material. Thus, the electrostatic repulsive force can be built up due to the existence of space charges between the layers. The latter causes the photo-induced volume expansion (PVE). After PVE process some slip motions between layers may occur. In this case, the value of the interaction between lone-pair electrons of the chalcogen atoms may be changed [26] and this makes fluctuations in the valence band of the material that, results in the photo-darkening process. Because the valence band of chalcogenide glasses consists of lonepair orbital on chalcogen atoms [26].

(3) Tanaka [27] suggested that interlayer widening (photo-induced expansion) can produce additional sites that are responsible for successive photo-darkening process, and the PD process can be induced by some locally excited atomic changes in a small scale [27]. For example, bond-twisting model [26] in which the bond angle is twisted by the movement of the chalcogen atoms.

3.2. Composition Effect of $Ge_xSb_{10}Se_{90-x}$ Thin Films

The measured transmission spectra of $Ge_xSb_{10}Se_{90-x}$ (x=0.10, 15, 25 and 30 at %) thin films with thickness 200nm are shown in Figure **5**. From this Figure we can observed that, the transmittance spectra exhibit a maximum in the wavelength rang (550-680nm) followed by a sharp decrease at wavelength less than 550nm and a reduction to zero at wavelength $\lambda \approx 400$nm. The absorption coefficient (α) was calculated from the transmission T and reflection R using Eq.(1). Figure (**6**) shows the spectral dependence of the absorption coefficient (α) at different values of Ge content.

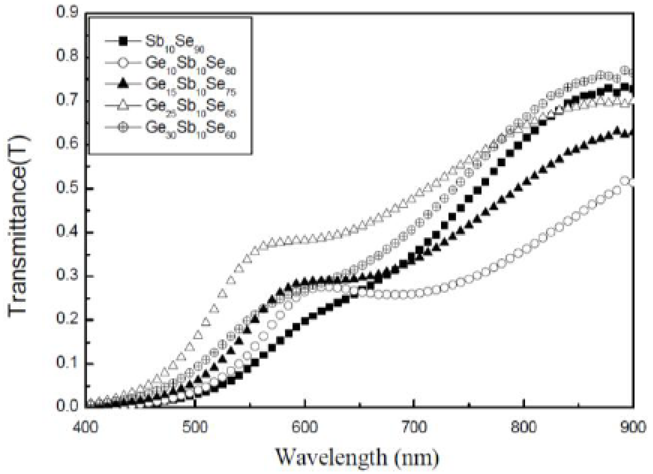

Figure 5: Transmittance spectra for as prepared GexSb10Se90-x thin films.

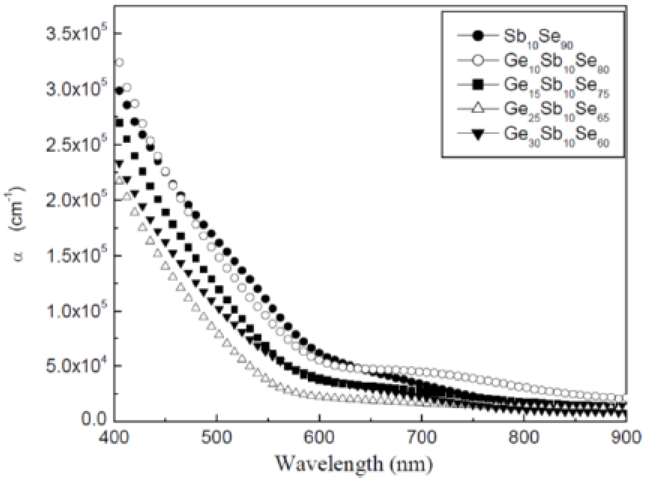

Figure 6: The plots of the absorption coefficient (α) as a function of wavelength (λ) for as-prepared GexSb10Se90-x thin films.

Figure **7** shows the plots of $(\alpha h\nu)^{1/2}$ versus hν plots for a family of $Ge_xSb_{10}Se_{90-x}$ thin films of thickness 200nm. It is found that optical absorption mechanism is successfully described by Tauc's [28] relation for the indirect optical transitions.

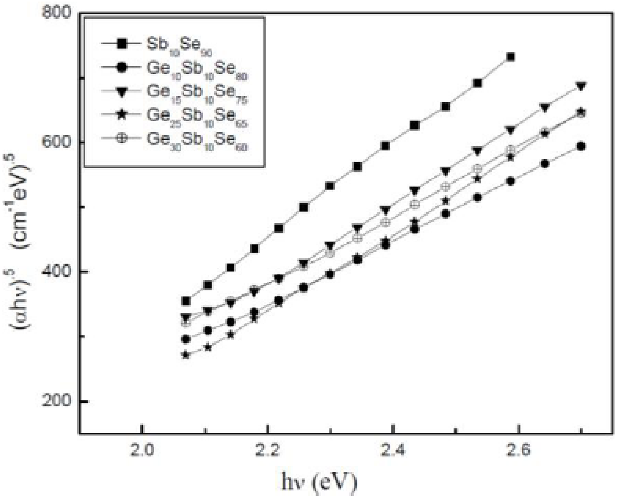

Figure 7: The plots of $(\alpha h\nu)$. 5 as a function of photon energy (hν) for the as-prepared GexSb10Se90-x thin films.

The observed variations in the E_g^{opt} values with composition can be discussed in terms of the average coordination number (Z), the deviation of stoichiometry (R) and the overall mean bond energy [29]. As discussed previously [30] the concept of the threshold at coordination number Z=2.67 for the most of chalcogenides can be used for explanation of compositional dependence of E_g^{opt} Firstly, we can assumed that the threshold at Z=2.6 for $Ge_{25}Sb_{10}Se_{65}$ system represents a chemical threshold of $Ge_xSb_{10}Se_{90-x}$ chalcogenide systems with x = 0, 10, 15, 20, 25 and 30 at%. At Z = 2.6 all homopolar bonds were completely disappeared and the network dimensionality change from 2D layered structure to 3D rigid one.

Secondly, starting form $Sb_{10}Se_{90}$ the nonstoichiometric system, for which Z = 2.2 and E_g^{opt} =1.45 eV. Increasing Ge content on the expense of Se content in this Se-rich chalcogenide glass results in: (i) increasing the coordination number Z in the range $0 \le x \le 30$. This increase of Z is accompanied by the increase of glass transition temperature T_g (Table (**1**) [31] up to Ge content 25 at% and Z=2.6, then T_g decrease with further increase of Ge content. (ii) The increase in T_g where $0 \le x \le 25$ reflects more rigidity or enhanced ordering in the glass structure due to the formation of Sb_2Se_3 trigonal and $GeSe_2$ tetrahedral structural units. (iii) Further increase of Ge content leads to the increase of heteropolar bonds Ge-Sb, Ge-Se and Sb-Se and the decrease of homopolar bonds such as Se-Se, Sb-Sb (IV). At Ge content x = 25 at% and according to the chemical ordering network model [30, 32]. About 65 at% of Se are required to saturate Sb and Ge bonds of $Ge_xSb_{10}Se_{90-x}$ system. Therefore, the glass is composed of completely cross-linked of $GeSe_2$ and Sb_2Se_3 structural units only without any excess of Se and/or Ge atoms. The energies of the different possible bonds in Ge-Sb-Se system [29, 33] are given in Table (**2**).

Table 1: The Average Coordination Number (Z), the Deviation of Stoichiometry (R), the Overall Mean Bond Energy <E> and the Glass Transition Temperature (T_g) for Different Compositions of $Ge_xSb_{10}Se_{90-x}$ thin Films

Composition	Z	R	<E> (eV)	T_g (K)
$Sb_{10}Se_{90}$	2.1	6	1.92	325.79
$Ge_{10}Sb_{10}Se_{80}$	2.3	2.286	2.155	436.5
$Ge_{15}Sb_{10}Se_{75}$	2.4	1.667	2.312	445.12
$Ge_{25}Sb_{10}Se_{65}$	2.6	1	2.686	581.6
$Ge_{30}Sb_{10}Se_{60}$	2.7	0.8	2.653	478.76

Table 2: Bond Energies for the Materials and Elemental Electronegativities. Bond Energies are Estimated form the Formula $E_{AB} = ((E_{AA} + E_{BB})/2 + 23 (X_A–X_B)^2)$ (k cal/mol) [124] Where X_A, X_B are the Elemental Electronegativities

Bond type	Bond energy (k cal/mol)	Bond energy (eV)	Electronegativity
Ge-Se	49.1	2.129	-
Se-Se	44	1.907	2.4
Sb-Se	42.85	1.858	-
Ge-Ge	37.6	1.63	1.8
Ge-Sb	34.418	1.48	-
Sb-Sb	30.2	1.309	1.9

The decrease in both E_g^{opt}, ΔE_{dc} and T_g values at x>25 at% may be ascribed to increase of homopolar bond concentration (such as Se-Se, Ge-Ge and Sb-Sb bonds) invoked by deviation from stoichiometry. Similar behavior of E_g^{opt} dependence of composition parameter x was reported by Shimakawa [34] and street *et al*. [35] in the Ge $_x Se_{1-x}$ system. They found that, the existence of minimum Ge at content x=33 at% and Z=2.67, depending on the bond energy, the relative probability of its formation was calculated by Goyal et al. [36] and its values were tabulated Table (**2**). According to the values of bond energy, the random covalent network model (RCNM) in which Ge atoms can be linked to other Ge atoms were excluded. Because of the low energy of Sb-Sb and Ge-Sb bonds therefore, only Se- Sb, Ge-Se and Se-Se bonds exist in the Ge-Sb-Se chalcogenide system.

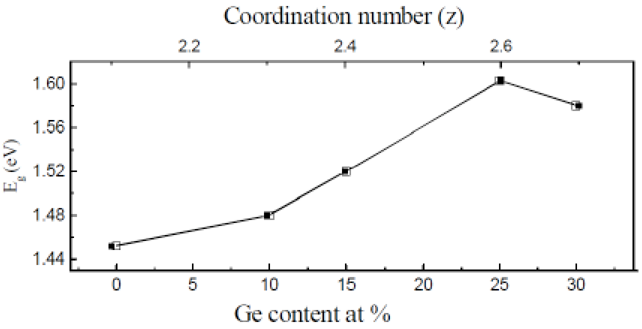

Figure 8: The plots of E_g^{opt} vs. both the coordination number (Z) and Ge content for E_g^{opt} $Ge_xSb_{10}Se_{90-x}$ thin films.

In this system it is worth mentioned that in the IV-VVI system (e.g. Ge-Sb-Se) the chemical threshold occur at Z-value other than Z=2.4 and Z=2.67 [37, 38]. In the present work (Figure **8**) one can assume that the existence of extreme at Z=2.6 for $Ge_{25}Sb_{10}Se_{65}$ represents the chemical threshold of $Ge_xSb_{10}Se_{90-x}$ chalcogenide system as well as E_g^{opt} exhibit a maximum value at Z=2.6. The increase of E_g^{opt} with increasing Ge content from x =0 to 25 at % can be explained according to CONM [30, 39] as follows: (i) The structure of $Ge_{25}Sb_{10}Se_{65}$ system is completely composed of cross-linked of $GeSe_2$ and Sb_2Se_3 structural units without any excess of homopolar bonds and the network dimensionality changed from 2D layered structure to 3D rigid one at Z=2.6, while excess of Se-Se bonds exist in $Sb_{10}Se_{90}$ (ii) Increasing Z with the increase of Ge content is accompanied by an increase in the glass transition temperature (T_g) (see Table **1**) [31] of $Ge_xSb_{10}Se_{90-x}$ system. This increase reflects more rigidity or enhanced ordering in the glass structure due to the formation of $GeSe_2$ and Sb_2Se_3 structural units. Therefore the optical band gap increases from 1.45 to 1.60 eV due to the increase of the overall mean bond energy with increasing Ge content from 0 to 25 at%.

All results above indicate that, the topological model based on the structural phase transition from 2D to 3D cross-linked network is valid in interpreting the changes of optical properties with composition of Ge-Sb-Se chalcogenide thin films.

4. CONCLUSIONS

The above results and discussion leads to the following conclusions:

- The photo-induced changes of the optical constants in $Ge_xSb_{10}Se_{90-x}$ thin films are attributed to the photo-induced redistribution of the chemical bonds that implies increased ordering of the structure with adding of Ge content.
- The optical band gap increases with increasing Ge concentration because of the network transformations from floppy to rigid type.
- The chemical threshold in this system occurs at Z=2.6 where E_g exhibits a maximum value.
- The photo-darkening changes with increasing Ge content are explained by the structure and defect photo-induced changes.

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