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A Quantitative Approach for the Determination of Light Induced Defects in a-$Se_{90}Sb_{10-x}Ag_x$ Thin Films by Using Thermally Stimulated Current Technique

ANJANI KUMAR[a], S.K. TRIPATHI[b] AND A. KUMAR[a,*]

[a]Department of Physics, Harcourt Butler Technological Institute, Kanpur, India

[b]Department of Physics, Panjab University, Chandigarh, India

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Thin films of $Se_{90}Sb_{10-x}Ag_x$ ($x = 0, 2, 4, 6, 8$) glasses have been prepared by vacuum evaporation technique. Present study reports the quantitative estimation of light induced defects in aforesaid thin films by using thermally stimulated current technique. Measurements have been made in a vacuum $\approx 10^{-3}$ Torr before and after exposing amorphous films to white light for different exposure times (0 to 6 h). Results indicate that light induced defects are created due to prolonged exposure of light and this is explained by a microscopic model proposed by Shimakawa and co-workers. It is also found that fractional increase in light induced defect density decreases as Ag concentration increases. A discontinuity has, however, been observed at 4 at.% of Ag which is explained in terms of average coordination number.

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1. Introduction

Various techniques [1–12] have been used in past for the determination of light induced defects in chalcogenide glasses. Bishop et al. [10] have observed that in chalcogenide glasses light exposure at low temperature results in an electron spin density which saturates with exposure time. In contrast, Mollot et al. [11] have observed that spin density does not saturate with exposure time. Biegelsen and Street [1] have also done ESR measurements in some chalcogenide glasses and observed that the electron spin density does not saturate even after 2 h of light exposure and found that spin density depends on the total exposure (intensity × time) independent of the actual intensity.

Abkowitz and Enck [3] have observed light induced defects in chalcogenide glasses using xerographic technique. They found that the defects created by light act as traps for the space charge and hence affect the xerographic properties. Traps are enhanced by one order of magnitude after illumination of light for 60 min.

Photoluminescence (PL) is one of the effective characterization techniques that can provide simultaneous information on both shallow and deep level defects in many semiconductors. Biegelsen and Street [1] have also studied light induced defects in chalcogenide glasses using photoluminescence technique along with ESR measurements.

Ac conductivity technique has also been used to study light induced defects in chalcogenide glasses [5, 6]. It is observed by Shimakawa et al. [5] that due to prolonged exposure of light ac conductivity of chalcogenide glasses increases, suggesting new defect states induced in the band gap. These defects depend on temperature and are induced by illumination.

Shimakawa et al. [13] have observed light induced metastable defects in chalcogenide glasses by photoconductivity measurements. They have observed that prolonged exposure to band gap light decreases the photoconductivity of chalcogenide materials. They have attributed this decrease to the light induced defects which could act as additional trapping and /or recombination centers. They have observed that photocurrent comes back to its original value after annealing. This indicates that light induced metastable defects are removed after annealing. The quantitative estimation of light induced defects could however not be made from this study.

Yannapoulos et al. [9] have reported a detailed study of the photo structural changes in bulk glassy As_2S_3 in vacuum using the Raman scattering. Various excitation energies were employed to achieve resonance and off-resonance conditions. Temperature induced structural changes are also investigated at conditions far from resonance, in order to clarify the role of band gap light on induced structural changes.

Out of the above mentioned techniques, quantitative estimates of light induced defects have been made only by ESR technique. The present work is an attempt to obtain quantitative estimates of light induced defects as a function of exposure time by thermally stimulated current (TSC) technique. This technique has already been applied for amorphous semiconductor to find out defect density [14–19].

It has been observed that Se-based glasses are more useful as compared to pure Se due to their higher photosensitivity, greater hardness, higher crystallization tem-

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*corresponding author; e-mail: dr_ashok_kumar@yahoo.com

perature and lower ageing effect [20]. Bindra et al. [21] have observed that Se–Sb glassy systems are preferred to Se–Te glassy system as addition of small percentage of Sb is sufficient for crystallization of pure a-Se. Therefore the properties of this glassy system have been studied by various workers [22–26].

Silver based chalcogenide glasses exhibit single glass transition and single crystallization temperature and are so useful in rewritable disks. It is also reported that Ag-based thin films of chalcogenide glasses can be used in erasable phase change optical recording devices [27, 28]. Ag-based chalcogenide glasses are preferred because of their ionic nature, too. In the electrical conduction Ag^+ ions play an important role [29, 30]. There have been several reports on the structural and ionic conductivity of these ions conducting chalcogenide glasses [31–34]. However, only a few studies have been done on the photoinduced properties of Ag doped chalcogenide thin films.

In our previous communication [35] we have reported the study of light induced defects in some binary glass system, $\mathrm{Se}_{90}\mathrm{X}_{10}$ (X = Sb, In, Ag). TSC technique was used to determine the density of defects. The present paper reports the effect of Ag addition as third element in binary Se–Sb glassy system. The same TSC technique has been used here to find the defect density on light exposure.

2. Theory of TSC measurements

TSC measurement is a well known non-isothermal technique for the investigation of trap levels in semiconducting materials. In principle, this method consists in filling the traps after cooling down the material to low temperature and then heating the material at a constant rate and observing the TSC as the traps are emptied. Though chalcogenide glasses have distribution of traps, one may use single trap analysis as suggested by Simmons et al. [36, 37]. Slow retrapping and fast retrapping cases have been analysed in the literature. In both the cases one finds that TSC for a material is given by

$$I(T) = A\exp\left(\frac{-E_t}{kT} - \frac{B}{\beta}\int_{T_0}^{T}\exp\left(\frac{-E_t}{kT}\right)\mathrm{d}T\right), \quad (1)$$

where symbol β is the heating rate, k is the Boltzmann constant and E_t is the trap depth. Here initial temperature is T_0 at time t, temperature becomes T [K]. Therefore

$$T = T_0 + \beta t.$$

A and B are parameters that depends on trapping centres and are given below. For slow re-trapping, parameters are

$$A = qn_{t_0}\upsilon\tau\mu EC,\ B = \upsilon.$$

For fast re-trapping, parameters are

$$A = qn_{t_0}N_e\mu EC/N_t,\ B = N_c/\tau N_t,$$

where n_{t_0} is the number of electrons in the traps at $t = 0$, N_t represents the total number of traps, q is the electronic charge, υ is the escape frequency, E represents the electric field, C is the cross-sectional area of the respective sample, N_c is the effective density of states in the conduction band, τ and μ represents the life time of electrons and mobility of electrons in the conduction band, respectively. For maxima it will be necessary that

$$\mathrm{d}I(T)/\mathrm{d}T|_{T=T_m} = 0. \quad (2)$$

From Eqs. (1) and (2)

$$\exp\left(\frac{E_t}{kT_m}\right) = \frac{B}{\beta}\frac{kT_m^2}{E_t}. \quad (3)$$

It is clear from Eq. (3) that on increasing the heating rate β, the TSC maxima should shift towards the higher temperature.

Manfredotti et al. [38] have observed that the trap density can be calculated by using the area of TSC curve according to the following relation:

$$N_\mathrm{t} = \frac{Q}{ALeG}[\mathrm{cm}^{-3}]. \quad (4)$$

Here Q represents the quantity of the charge released during TSC experiment. This can be calculated from the area under TSC peak. L and A are the thickness and area of the sample, respectively, e is the electronic charge and G represents the photoconductivity gain and is estimated using the expression [39]:

$$G = \frac{\tau}{t_\mathrm{tr}}, \quad (5)$$

where τ represents the carrier life time and t_tr represents the carrier transit time between electrodes.

3. Experimental details

Glassy alloys of $\mathrm{Se}_{90}\mathrm{Sb}_{10-x}\mathrm{Ag}_x$ (x = 0, 2, 4, 6, 8) are prepared by quenching technique. High purity 5N materials are sealed in quartz ampoules (length ≈ 5 cm and internal diameter ≈ 8 mm) with a vacuum ≈ 10^{-5} Torr. The ampoules containing the high purity 5N material in vacuum are held at 900 °C for 12 h where the ampoules are constantly rocked to make the melt homogeneous. The melt is cooled rapidly by removing the ampoules from the furnace and dropping into ice-cooled water. The quenched samples of the glassy alloys are taken out by breaking the quartz ampoules. The glassy nature of samples is confirmed by the X-ray diffraction pattern as shown in Fig. 1. No sharp diffraction peak in diffraction pattern indicates the amorphous nature of the prepared materials.

Thin films of glassy alloys are prepared by vacuum evaporation technique keeping glass substrates at room temperature. Vacuum evaporated indium electrodes at bottom are used for the electrical contact. The thickness of the film is ≈500 nm. The coplanar structure (length ≈12 mm and electrode separation ≈0.5 mm) was used for the present measurement. The films are kept in the deposition chamber in dark for 24 h before mounting them in the sample holder. This is done to allow sufficient annealing at room temperature so that a metastable thermodynamic equilibrium may be attained in the samples as suggested by Abkowitz [40].

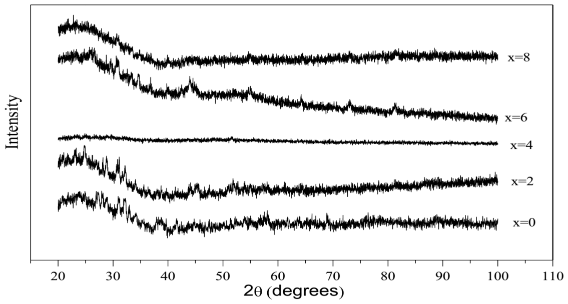

Fig. 1. X-ray diffraction pattern of $Se_{90}Sb_{10-x}Ag_x$ ($x = 0$, 2, 4, 6, 8) glassy alloys.

For TSC measurements, thin films were mounted in a specially designed sample holder containing a transparent window for shining light and a vacuum of 10^{-2} Torr was maintained throughout the measurements. The temperature of the respective film was measured by a calibrated copper constantan thermocouple mounted very near to the film. The films were annealed first at approximately 360 K for one hour in a vacuum of 10^{-2} Torr before the measurements. For exposing the films to white light, 200 W tungsten lamp was used as light source. The films were exposed to white light of intensity 1200 lx by mounting them in the sample holder for different exposure times and measurements were repeated after each exposure. Proper care was taken to avoid the thermal effects of light so that the temperature of the film did not change significantly.

4. Results

For the measurement of TSCs, temperature dependence of current has been measured in two states applying a dc voltage of 100 V in the ohmic region. Kabir [41] studied the transient and steady state current behaviours in a-Se behaviours and considered injection currents from electrode to develop model for the charge carrier transport in these materials. In our earlier communications [42] we have also reported space charge limited conduction in $Se_{90}Sb_{10-x}Ag_x$ in high field region $\approx 10^4$ V/cm. The present measurements are made in ohmic region field less than 10^3 V/cm where injection currents are not important. In state I, the sample is heated at a constant heating rate from room temperature 300 K to 373 K without any light exposure and current is measured during heating. However, in the state II, light is incident on the sample for 2 min at room temperature through the transparent window of the sample holder. The decay of photoconductivity was allowed for 10 min after switching off the light; this was done to give sufficient time for photocurrent to decay. The sample was again heated from 300 K to 373 K at the same heating rate. The difference of conductivity at a particular temperature in these two states is calculated and plotted as a function of temperature to obtain TSC curve.

The experimental TSC curves are studied for $Se_{90}Sb_{10-x}Ag_x$ ($x = 0$, 2, 4, 6, 8) thin films at different heating rates. Figure 2 shows the same in case of a-$Se_{90}Sb_6Ag_4$ and similar results are obtained for other samples. From this graph it is clear that the TSC peaks shifts to the higher temperatures as the heating rate increases. Shift of this peak with heating rate confirms the observation of TSC [36, 37] as also observed by various other workers [15, 16, 43, 44] in chalcogenide glasses. Such peaks may not be related to the relaxation effects at glass transition temperature (T_g) as they are far away from T_g reported in these glasses [45]. To study the effect of prolonged exposure to light, TSC measurements have been made before and after exposing the samples for different exposure times. This was done to see the changes in the density of defect states due to light exposure. The trap density has been calculated using the relation (4) and (5).

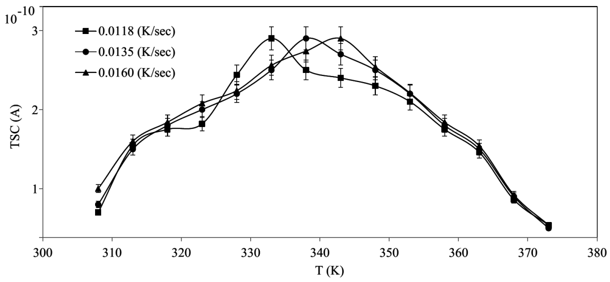

Fig. 2. Experimental TSC curve of a-$Se_{90}Sb_6Ag_4$thin films at various heating rates.

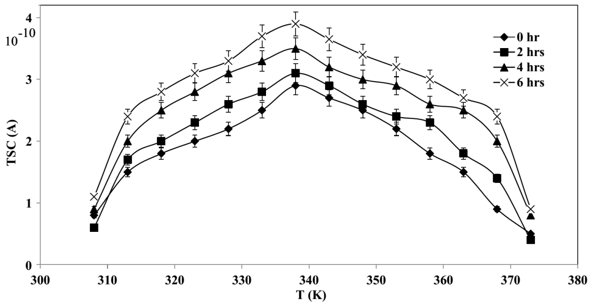

Fig. 3. TSC curve of a-$Se_{90}Sb_6Ag_4$thin films at various exposure times (0, 2, 4, 6 h).

TSC curves in case of $Se_{90}Sb_6Ag_4$ thin films for different exposure times (0, 2, 4, 6 h) are given in Fig. 3, similar results are obtained for other samples $x = 0$, 2, 6, 8 in $Se_{90}Sb_{10-x}Ag_x$. Trap density has been evaluated by using the relation (4) and results are given in Table I for various compositions. Figure 4 shows the concentration of trap density as a function of exposure time in case of $Se_{90}Sb_6Ag_4$. From this figure it is clear that trap density increases with the increase of exposure time of light. Similar results are obtained for $x = 0$, 2, 6, 8 in $Se_{90}Sb_{10-x}Ag_x$ glassy system.

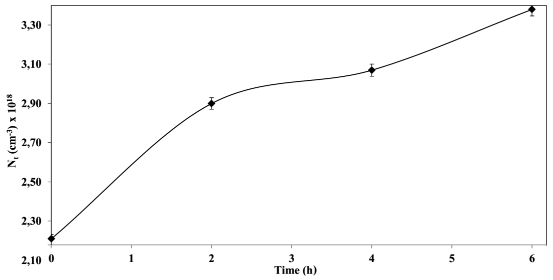

Fig. 4. Variation of trap density as a function of exposure time for a-$Se_{90}Sb_6Ag_4$thin films.

TABLE I

Values of trap density N_t and its fractional increase ΔN_t for various exposure times T in different thin films

T [h]	N_t[cm^{-3} $\times 10^{16}$]	ΔN_t
	$Se_{90}Sb_{10}$	
0	4.2	0
2	4.4	0.05
4	5.4	0.29
6	7.3	0.74
	$Se_{90}Sb_8Ag_2$	
0	2.0	0
2	2.4	0.20
4	2.5	0.25
6	2.9	0.46
	$Se_{90}Sb_6Ag_4$	
0	2.2	0
2	2.9	0.31
4	3.1	0.39
6	3.4	0.53
	$Se_{90}Sb_4Ag_6$	
0	2.5	0
2	2.8	0.14
4	3.2	0.27
6	3.6	0.44
	$Se_{90}Sb_2Ag_8$	
0	1.1	0
2	1.2	0.01
4	1.3	0.21
6	1.4	0.24

5. Discussion

Biegelsen and Street [1] have observed that due to optical irradiation the generation of coordination defect in chalcogenide glasses were opposed to excitation of preexisting defects and suggested light induced electron spin resonance (LESR) experiments in which self trapped excitons (i.e. conjugate pairs of charged defects for example P_2^+, C_1^- or P_4^+, C_1^-) are induced by light exposure. The formation of a self trapped exciton (STE) state is symmetric in the case of a-As_2S_3 (Y_1 and Y_2). However, such STE states would not act as trapping centres for electrons or holes because they are effectively neutral and therefore would not affect the photoconductivity. On the other hand, random pairs (RP) of D^+, D^- defects (e.g. P_4^+, C_1^+ or C_3^+) may result from bond switching reaction at STE centres and therefore act as electron or hole trapping centres and hence would act to decrease the photoconductivity.

Shimakawa et al. have proposed a microscopic model for LIMD creation [13, 46]. This model has explained many kinds of photoinduced phenomenon. The initiation of defect creation was assumed to be the formation of STE in the form of intimate alternation pair (IP) which is stable at lower temperatures. According to them fatigue of main peak accompanying the low energy peak and a part of LESR can be related to these IPs, since these phenomena are removed completely by annealing around 300 K. Following this, defect conserving bond switching which is increased by light exposure leading to defect separation and migration, results in the formation of metastable well separated charged defects known as random pairs (RP) and these RPs are removed only by annealing near the glass transition temperature. These RPs dominate photoconductivity as recombination centres and it has been observed that these IPs and RPs are responsible for increasing AC transport [5, 13].

In charged defect model or valence alternation pair model, bonding defects can have three charged states, which are denoted by D^+, D^-, and D^0 where D denotes defect state or a dangling bond and the superscript denotes the respective charge states. The neutral dangling bond D^0, say a singly coordinated chain-end Se atom, would normally possess spin but it is unstable [47]. Here first the photoinduced creation of D^0 centres from normal bonding (NB) state takes place and it is important that the energy minimum of the photoexcited state is located above the D^0 centers. These D^0 centres are considered to be quasi stable and the decrease in photocurrent with the exposure time observed at the room temperature can be related to the creation of D^- and D^+ from D^0 centers. In this case the existence of D^+ centres is not essential here and the D^- centre can be transferred from D^0 centre at room temperature. According to the equation below

$$D^0 - \mathrm{h} \leftrightarrow D^-,$$

where h denotes hole. These photoinduced charge defects are metastable and can be removed with annealing near the glass transition temperature.

Ovshinsky and Adler [48] have explained the importance of the interaction between the lone pair electrons of the chalcogenide atoms belonging to the different segmental and/or fragmental clusters. The quasi stable state D^0 and D^- can be replaced by the structures D^1 and D^2, respectively. On illumination of light in chalcogenide glasses, the electron hole pair is excited, the hole can diffuse and electron is left behind, which is less mobile.

The hole may be captured by a hole trap which in turn trapped through modifying the intercluster interactions, i.e., the lattice relaxation takes place at room temperature. Thermal energy can excite the trapped electrons leaving the atomic structure unchanged. It is seen that the electrons can diffuse and it can recombine with the trapped hole. In the present case the atomic structure of hole trap will partially be relaxed hence the number of hole trap increases, which is responsible for the decrease in the photocurrent with the exposure time during the illumination.

In our case we have observed increase in density of defects on light shining at room temperature. These defects may act as traps and hence affect the thermally stimulated currents. The defects observed in our case can be random pairs as suggested by Shimakawa et al. [49]. The fractional increase in density of traps due to light exposure has been calculated for each exposure in all these alloys studied and the results are shown in Tables I–V and plotted in Fig. 5. A comparison of these results shows that fractional increase in trap density decreases as Ag concentration increases except at 4 at.% of Ag (see Fig. 6). This type of discontinuity is observed in chalcogenide glasses in many physical properties [50–52] at a particular concentration of the additive element when average coordination number reaches a certain value, e.g., 2.4 in Ge–Se glasses. According to Phillips and Thorpe model [53], structure below a certain average coordination number is floppy type and above this average coordination number it is mechanically optimized structure.

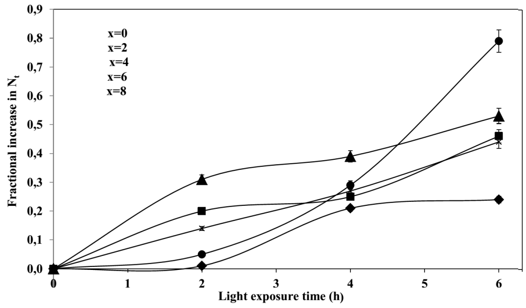

Fig. 5. Fractional increase in N_{t} vs. light exposure time for $\mathrm{Se}_{90}\mathrm{Sb}_{10-x}\mathrm{Ag}_x$ ($x = 0$, 2, 4, 6, 8) thin films.

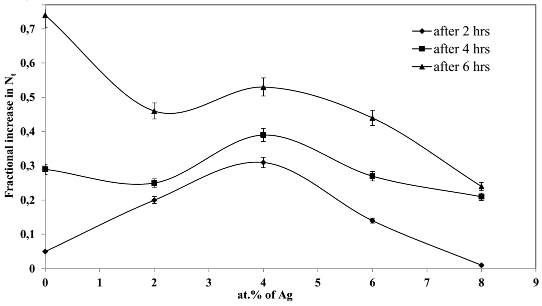

Fig. 6. Fractional increase in N_{t} vs. at.% of Ag concentration for $\mathrm{Se}_{90}\mathrm{Sb}_{10-x}\mathrm{Ag}_x$ ($x = 0$, 2, 4, 6, 8) thin films.

The average coordination number $\langle z\rangle$ of the glasses studied has been evaluated using the standard procedure described by Tanaka [54]. Thus, for the glassy system $\mathrm{Se}_a\mathrm{Sb}_b\mathrm{Ag}_c$ ($a+b+c=1$), the value of $\langle z\rangle$ is given by

$$\langle z\rangle = (aZ_{\mathrm{Se}} + bZ_{\mathrm{Sb}} + cZ_{\mathrm{Ag}})/(a+b+c). \qquad (6)$$

Using the coordination number 2, 3, 4 for Se, Sb and Ag respectively, the value of $\langle z\rangle$ is calculated for each glassy alloy. $\langle z\rangle$ varies from 2.10 to 2.18 in the present case. The $\langle z\rangle$ for the composition at which maxima occur in Fig. 6, comes out to be 2.14. However, Phillips and Thorpe model shows threshold at $\langle z\rangle = 2.4$.

The $\langle z\rangle$ value in the present case is slightly lesser than what is expected from the above model. This may be due to an important limitation of Phillips and Thorpe model. In this model, Phillips considered the interaction between atoms to be purely covalent while arriving at the balance condition. Such an assumption may be valid for Ge–Se glasses, but not for system containing heavier elements like Sb, Ag. The presence of such a heavier element may lead to partial covalent bonding. This can affect the balance condition. The decrease in value of fractional increase in trap density at higher concentration of silver indicates that silver doped alloys may be used for optoelectronic applications as degradation upon light soaking is small.

6. Conclusions

For the determination of light induced defects in chalcogenide glasses, thermally stimulated current technique has been used in amorphous thin films of $\mathrm{Se}_{90}\mathrm{Sb}_{10-x}\mathrm{Ag}_x$ ($x = 0$, 2, 4, 6, 8). Results indicate that trap density increases with the increase in exposure time in all the alloys studied here. The trap density increase with exposure time, indicating that more and more defects are created as exposure time increases. The creation of light induced defects due to prolonged exposure of light is explained by a microscopic model proposed by Shimakawa and co-workers. Results also show that fractional increase in trap density decreases as Ag concentration increases except at 4 at.% of Ag and this is explained on the basis of Phillips and Thorpe model. Fractional increase in light induced defects decreases as Ag concentration increases in silver doped glassy alloys except at 4 at.% of Ag, indicating their usefulness in optoelectronic applications.

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