

## Crystallization Mechanism and Thermal Stability of $Se_{98}In_{2-x}Sn_x$ ( $x = 0, 0.5, 1$ and $1.5$ ) Semiconducting Glasses

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### **Abstract**

Results of Differential Scanning Calorimeter (DSC) under non-isothermal condition on  $Se_{98}In_{2-x}Sn_x$  ( $x= 0, 0.5, 1$  and  $1.5$ ) chalcogenide glasses have been reported and discussed. In the glassy region, the dependence of the glass transition temperature  $T_g$  on the heating rate  $\alpha$  obey a power law,  $T_g = T_0[\alpha]^y$ , and the glass transition activation energy decreases with the addition of Sn. The crystal growth kinetics has been investigated using Kissinger, Gao et.al, and Ozawa equations. Results indicate that the crystallization activation energy decreases and the crystallization ability is retarded, due to the formation of cross-linked structure, with the addition of Sn. Besides to that the crystal growth is found to occur in 2-dimensions. Investigation of thermal stability through the calculations of the temperature difference  $T_c - T_g$ , S-parameter, Hrubby number, crystallization rate factor and the enthalpy released during the crystallization process, indicates that  $Se_{98}In_{0.5}Sn_{1.5}$  glass is thermally most stable in the composition range of study.

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## آلية التبلور والاستقرار الحراري لأشباه الموصلات الزجاجية

$$\text{Se}_{98}\text{In}_{2-x}\text{Sn}_x \quad (x = 0, 0.5, 1, 1.5)$$

### ملخص

تناقش هذه الورقة نتائج دراسة على أشباه الموصلات الزجاجية  $\text{Se}_{98}$  حالة عدم ثبات درجة الحرارة. تخلص الدراسة إلى أن درجة التحول الزجاجي  $T_g$  تعتمد على معدل التسخين وذلك وفقاً للعلاقة  $T_g = T_o [\alpha]^y$  وأن طاقة التحول تتناقص بزيادة كمية القصدير في العينة. أما في ما يتعلق بآلية التبلور، والتي تمت دراستها باستعمال معادلات Kissinger, Gao et al, Ozawa. فقد وجد أن النمو البلوري يحدث في بعدين وأن طاقة التبلور تتناقص بزيادة كمية القصدير. يعتبر الاستقرار الحراري مؤشراً مهماً لتحديد المجال الذي تستخدم فيه هذه العينات، وقد تمت دراسته من خلال الفرق في درجة الحرارة الذي تستخدم فيه هذه العينات، رقم Hruby، معامل معدل التبلور و المحتوى الحراري. تشير النتائج إلى أن  $\text{Se}_{98}\text{In}_{0.5}\text{Sn}_{1.5}$  هو الأكثر استقراراً من بين العينات التي تناولتها هذه الدراسة.

## **1. Introduction**

Great attention has been devoted to chalcogenide glasses during the last two decades due to their wide range of utilization in high-tech fields. Their applications extended from well established field of xerography to the very exciting field of threshold and memory switching and the current interest of these materials centers on x-ray imaging and photonics [1,2]. Se-In glass has got several advantages over pure and amorphous Se [3] and several attempts have already been made to utilize it in solar cells [4,5]. From a technological point of view, these glasses should be thermally stable with time and temperature during use. However, thermal instability leading to crystallization is found to be one of the drawbacks of these alloys, which could be improved through the addition of a third element [6,7]. Two different approaches have been adopted to investigate chalcogenide glasses: first is the analysis of their structure and physical properties [8,9, 10], while the second is concerned with their stability, that is the study of phase transformation and crystallization processes. Since the glassy state is essentially a quasi-stable state, it inherently possesses the possibility of transforming into a more stable crystalline state. The most promising properties of chalcogenide glasses have been found to deteriorate drastically during crystallization. Understanding the mechanism of crystallization to impede or control crystallization is therefore a prerequisite for most of the applications, as stability against crystallization determines their effective working limits. Since thermo-analytical techniques, such as differential scanning calorimeter DSC, are simple and informative, they are increasingly being employed to investigate glass-crystalline transformation. Successful application of thermo-analytical data, using DSC, required suitable methods for analyzing the experimental data, and a large number of approaches [11,12,13,14] have been formulated based on the formal theory of transformation kinetics.

The purpose of the present paper is to investigate the crystallization kinetic and thermal stability of  $\text{Se}_{98}\text{In}_{2-x}\text{Sn}_x$  ( $x = 0, 0.5, 1, 1.5$ ) chalcogenide glasses. In this context the glass transition temperature  $T_g$ , the onset temperature  $T_c$  as a function of heating rate and the dependence of  $T_g$  on the coordination number with varying composition have been studied. The activation energy in both phases has been obtained using theoretical models [11,14] reported in literature. Besides to that the dimensionality of crystal growth i.e Avrami exponent ( $n$ ) has been obtained. The demands of thermal stability have been ensured through the calculations of the temperature difference  $T_c - T_g$ , S-parameter, Hruby number  $H_R$ , crystallization rate factor  $k_p$ , and enthalpy  $H_c$  released during the crystallization process.

### **3. Experimental**

High purity (99.999%) of Selenium, Indium and Tin in appropriate atomic percent proportions was weighed in a quartz glass ampoule (length 5cm and internal diameter 8mm). The content of the ampoule was sealed in a vacuum of  $10^{-6}$  torr and heated in a furnace where temperature was raised at a rate of 3-4K per minute up to 1120K and kept around that temperature for 10h. The ampoule was frequently rocked to ensure the homogeneity of the sample. The molten sample was then rapidly quenched in ice cooled water. Differential Scanning Calorimeter (DSC) is used to measure the caloric manifestation of the crystallization and to study the crystallization kinetics under non-isothermal condition. The temperature precision of this equipment is  $\pm 0.1\text{K}$  with an average standard error of about 1K in the measured values. The DSC scans are taken at five different heating rates i.e, 5, 7, 10, 15 and 20K/min on accurately weighed sample taken in aluminum pan. The masses of the samples varied between 10 and 15mg and were measured by weighing with an accuracy of about  $100\mu\text{m}$ . The temperature range covered in DSC is from room temperature to 493K. The DSC equipment is calibrated prior to

measurements, using high purity standards Pb, Sn and In with well-known melting points. The results of temperature and enthalpy calibrations obtained for the standard materials were within 3% of the values given in literature [15]. The instrument constant of this DSC, as measured is 1.5.

#### **4. Results and Discussion**

Differential scanning calorimeter (DSC) has been employed to obtain the thermograms of  $Se_{98}In_{2-x}Sn_x$  ( $x=0, 0.5, 1$  and  $1.5$ ) chalcogenide glasses at five different heating rates. Typical DSC thermograms of  $Se_{98}In_{1.5}Sn_{0.5}$  at five different heating rates is shown in figure 1. Similar thermograms were also obtained for other compositions. Two characteristic phenomena are evident in the DSC thermograms. The first one corresponding to the glass transition region (endothermic reaction) and the other to the crystallization process (exothermic reaction). The glass transition temperature  $T_g$  and the onset crystallization temperature  $T_c$  have been defined as the temperatures, which correspond to the intersection of the two linear portions adjoining the transition elbow of the DSC trace in the endothermic and exothermic directions, respectively.

It is interesting to note that the  $T_g$  varies with the heating rate  $\alpha$  as shown in figure 2. This behavior may be represented by a power law of the form  $T_g=T_0[\alpha]^y$ , where  $\alpha$  is the normalized heating rate and  $T_0$  is  $T_g$  of the glass at a heating rate of 1K/min, i.e  $T_0=(T_g)_1$ . Normalization has been done with respect to a heating rate of 1K/min. The exponent  $y=\log_{10}[(T_g)_{10}/(T_g)_1]$ , where  $(T_g)_{10}$  stands for the  $T_g$  value at a heating rate of 10K/min. This dependence of the glass transition temperature on the heating rate is an experimentally observed [16,17] fact. In the present study  $T_g$  increases with the increase of the heating rate. This is due to the fact that  $T_g$  is the temperature at which the relaxation time becomes equal to the experimental time of observation. At the same time,  $T_g$  varies

inversely [18] as the relaxation time. With increasing heating rate, the experimental time of observation decreases and hence the glass transition temperature increases. Furthermore, it is observed that  $T_g$  increases as Sn content increases for the five different heating rates. It is reported [19] that  $T_g$  represents the strength or the rigidity of the glass structure. The observed increase in  $T_g$  with increasing Sn impurity in  $Se_{98}In_{2-x}Sn_x$  glasses may be explained by considering the structural changes occurring due to the further addition of Sn atoms. The addition of Sn is at the cost of In and since Sn has a coordination number of 4 so it has to satisfy its coordination requirements by making bonds with Se. The replacement of some of the original Se-Se and Se-In structural units by Sn-Se units, which have a higher bond energy, results in a slight increase in the cohesive energy and accounts for the observed increase in the  $T_g$  [20].

The glass transition activation energy  $E_t$  is that amount of energy, which is absorbed by a group of atoms in the glassy region so that a jump from one metastable state to another is possible. This activation energy is involved in the molecular motions and rearrangements of atoms around the glass transition temperature [21]. When the sample is reheated, in DSC furnace, the atoms undergo infrequent transitions between the local potential minima separated by different energy barriers in the configuration space where each local minimum represents a different structure. The most stable local minimum in the glassy region has lower internal energy [21,22]. Accordingly, the atoms in a glass having minimum activation energy have higher probability to jump to the local minimum state of lower internal energy and hence the most stable. The evaluation of  $E_t$  has been widely [19,20,23,24] carried out using the relation,

$$d\ln\alpha/d(1/T_g)=-E_t/R \quad (1)$$

where  $R$  is the universal gas constant. It is clear that the plot of  $\ln\alpha$  vs  $1/T_g$  gives a straight line the slope of which gives the glass transition activation energy. Figure 3 shows the plot of  $\ln\alpha$  vs  $1000/T_g$  for  $Se_{98}In_{1.5}Sn_{0.5}$  glass. The values of  $E_t$ , along with their uncertainties, for all compositions are listed in table 1. From this table it is observed that addition of Sn decreases the glass transition activation energy which is an indication that  $Se_{98}In_{0.5}Sn_{1.5}$  is the most stable glass in the composition range of investigation.

The crystallization process under non-isothermal condition has also been analyzed in terms of composition dependence of onset crystallization temperature  $T_c$ , peak crystallization temperature  $T_p$ , and crystallization activation energy  $E_c$ . Besides regular features, the dimensionality of crystal growth has also been computed for all the compositions. It has been pointed out [14,19,25], in the crystallization process three types of activation energy have to be considered: the activation energy of nucleation, the activation energy of growth, and the activation energy of the whole crystallization process. However, it has been shown by various studies [25,26] that the activation energy for growth may be taken equal to the activation energy of the whole crystallization provided it is evaluated using the thermal analysis of the samples.

One of the most popular methods used in analyzing the crystallization process using DSC is the method developed by Kissinger [11]. In this model the activation energy for crystallization  $E_c$  is obtained from the variation of the peak crystallization temperature  $T_p$  with the heating rate  $\alpha$  as suggested by Kissinger equation:

$$\ln(T_p^2/\alpha) = E_c/RT_p + \text{const.} \quad (2)$$

Figure 4 shows the plot of  $\ln(T_p^2/\alpha)$  against  $1000/T_p$  for  $Se_{98}In_{0.5}Sn_{1.5}$  glass as an example. The slope of the plot gives the activation energy of crystallization  $E_c$  and it comes out to be  $55 \text{ kJmol}^{-1}$ . The values of

the activation energy for all other compositions have been obtained in the same way and are listed in table 1.

The activation energy of crystallization  $E_c$  can also be evaluated from the variation of the maximum reaction rate  $(dx/dt)_p$  with the peak temperature  $T_p$  at different heating rates using Gao et al [14] equation

$$d[\ln(dx/dt)_p]/d(1/T_p) = -E_c/R \quad (3)$$

The plot of  $\ln(dx/dt)_p$  against  $1000/T_p$  yields a straight line the slope of which gives the activation energy of crystallization  $E_c$ . Figure 5 shows such plot for the  $Se_{98}In_{0.5}Sn_{1.5}$  glass in which the activation energy comes out to be  $54 \text{ kJmol}^{-1}$ . The activation energy for other compositions have also been calculated and are listed in table 1. The difference in the activation energy obtained by the two formulations is within the experimental error. This is due to the fact that the two equations have been derived based on the Johnson-Mehl-Avrami [27,28] equation and have also considered the same assumptions. The values of the activation energy, obtained with either method, decrease with the addition of Sn atoms to binary Se-In glass. When In is added to amorphous Se it is dissolved in the Se chains to satisfy its coordination requirements and to form a cross-linked structure as a result of which the crystallization ability is retarded. The decrease in the activation energy with the addition of Sn may be understood in terms of the decrease in the rate of crystallization due to the cross-link structure of the Se-In-Sn glass. Furthermore, according to Tanaka [29] in the glassy compounds containing heavy elements, the physical properties of such glasses are influenced substantially by metallic characters. Indeed x-ray k-absorption edge studies as carried out by Kumar et al.[30] indicate that the type of bonding exist in Se rich glasses containing heavy elements, like In and Sn, is not covalent but metallic or ionic-covalent.



In addition to the evaluation of the activation energy, the dimensionality of growth has also been evaluated from the Ozawa [12] equation,

$$d\{\log_{10}[\ln(1-x)]\}/d\log_{10}(\alpha) = -n \quad (4)$$

where  $x$  is the fraction crystallized and is obtained at the same temperature at different heating rates,  $\alpha$  is the heating rate, and  $n$  is the Avrami exponent or the dimensionality of crystal growth. The plot of  $\log_{10}[\ln(1-x)]$  against  $\log_{10}(\alpha)$  gives a straight line the slope of which is the dimensionality of crystal growth. Figure 6 shows such plot for  $Se_{98}In_{1.5}Sn_{0.5}$  glass in which  $n$  comes out to be 2.3. This is an indication of the fact that the crystal growth of ternary Se-In-Sn glasses occurs in two dimensions. Earlier investigations of binary Se-In glasses as carried out by the author [21] indicate that crystal growth or Avrami exponent is one-dimensional. It has been reported [31] that the Avrami exponent  $n$  assumes the values from  $n=1$  for 1-dimensional growth (or growth from surface nuclei), to  $n=3$  for 3-dimensional growth. Therefore, it is reasonable to state that the addition of Sn to Se-In glasses will change the mechanism and morphology of the crystal growth. In fact, the change in the Avrami exponent from  $n=1$  to  $n=2.3$  suggests that the devitrification mechanism is controlled by the glass-crystal interface reaction [31]. The values of Avrami exponent for other compositions of Se-In-Sn glasses are listed in table 1.

Thermal stability of amorphous glasses is a term often used while ascertaining the potential of a glass for practical applications. The stability of glass forming system against crystallization is often reported [19,21,24,32,33,34,35] in terms of the temperature interval between the glass transition temperature  $T_g$  and the onset temperature of crystallization  $T_c$ , detected during heating a glassy sample at a steady rate. The temperature difference  $T_c-T_g$  for the glasses under investigation has been calculated at all heating rates. This difference,

for all compositions, has been tabulated in table 2 at a heating rate of 20K/min. It is clear from the table that  $T_c-T_g$  increases with the addition of Sn, which is an indication of the enhancement of the stability. For further confirmation of this result various characteristic parameters of thermal stability reported [19,21,24] in literature have been considered. It is known that some empirical criteria of glass stability [24] allow the prediction of the tendency to devitrification from values of S- parameter, Hruby number  $H_R$  [24,32], the crystallization rate constant  $k_p$  [33], and the enthalpy released  $H_c$  during the crystallization process [34,35]. The S-parameter is defined as,

$$S=(T_c-T_g)(T_p-T_c)/T_g \quad (5)$$

and the Hruby number  $H_R$  is given by

$$H_R= (T_c-T_g)/(T_m-T_c) \quad (6)$$

where  $T_m$  is the melting temperature. The S-parameter reflects the resistance to devitrification after formation of the glass. Higher value of  $T_c-T_g$  delay nucleation and the small value of  $T_m-T_c$  retard the growth process of the nucleated crystals. The Hruby number thus combines the nucleation and growth aspects of the crystallization process.  $T_p-T_c$  is related to the rate of devitrification of the glassy phase. Values of S-parameter and Hruby number for different compositions of  $Se_{98}In_{2-x}Sn_x$  ( $x=0, 0.5, 1, 1.5$ ) glasses have been calculated for all heating rates and are listed in table 2. It is clear that the highest values of S-parameter and  $H_R$  occurs for  $Se_{98}In_{0.5}Sn_{1.5}$  glass and may be taken as the most stable one in the composition range of investigation.

The crystallization rate constant  $k_p$  corresponding to the temperature at which the crystallization rate is maximum, has been calculated from

the analysis of the DSC thermograms and through the use of the following condition [33]:

$$\alpha E_c / R k_p T_p^2 = 1 \quad (7)$$

where the symbols carry their usual meaning used throughout the text. The values of  $k_p$  for the glasses under study have been calculated and their average values  $\langle k_p \rangle$  are listed in table 2. The obtained values indicate that  $\langle k_p \rangle$  decreases with the addition of Sn and hence we can argue that the crystallization ability decreases when Sn is incorporated to Se-In glass. This is in agreement with the conclusions drawn by others [33] while investigating thermal stability of chalcogenide glasses.

For further confirmation of the glass stability the enthalpy released during the crystallization process  $H_c$  has been determined by measuring the area under the crystallization peak and converted into Joule through the use of the instrument constant. Using the formula  $H_c = kA/M$  [19,24,34,35], where  $k$  is the instrument constant which is found to be 1.5,  $A$  is the area under the crystallization peak and  $M$  is the mass of the sample.  $H_c$  has been calculated for various compositions and are listed in table 2 for heating rate of 5K/min as an example. The enthalpy released during the crystallization is associated with the stability of glasses and the least stable glass with minimum  $T_c - T_g$  values are supposed to have maximum  $H_c$  [19,24,34,35]. It is observed that the  $Se_{98}In_{0.5}Sn_{1.5}$  glass has the lowest enthalpy released and since  $T_c - T_g$  is maximum for this particular glass so it is the most stable glass in the composition range of investigation. A similar behavior has also been observed [19] in other chalcogenide glasses.

## **5. Conclusion**

Systematic investigation of the crystallization kinetics and thermal stability of  $\text{Se}_{98}\text{In}_{2-x}\text{Sn}_x$  ( $x=0, 0.5, 1$  and  $1.5$ ) chalcogenide glasses have been carried out and the following conclusions may be drawn:

1. The glass transition temperatures for Se-In-Sn glasses depend on both heating rate and composition. This heating rate dependence of  $T_g$  has been utilized to calculate the glass transition activation energy  $E_t$ .
2. The crystallization activation energy decreases with the addition of Sn and the crystallization mechanism in Se-In and Se-In-Sn occurs in one- and two-dimensional growth, respectively, according to the obtained values of Avrami exponent ( $n$ ).

No correlation has been found between the activation energy and thermal stability. The parameters that determine thermal stability indicate that the nucleation and growth process occur sluggishly with the addition of Sn. It is known [31] for crystallization process to proceed the crystallization rate factor  $k_p$  should be large enough that the crystal growth kinetic barrier can be overcome, but the calculated values of  $k_p$  decrease with the addition of Sn. Therefore, it is reasonable to state that greater activation energies obtained from DSC data should not necessarily be indicative of greater thermal stability.

## **Acknowledgement**

One of the authors (Dr. Mousa Imran) thanks Al-Balqa Applied University for financial support provided in form of project.

Thanks are also due to Prof. N.S.Saxena, Department of Physics, University of Rajasthan, India and Prof. M.Hussain, Department of Physics, Jamia Millia Islamia, India for co-operation in materials preparation and DSC Data.

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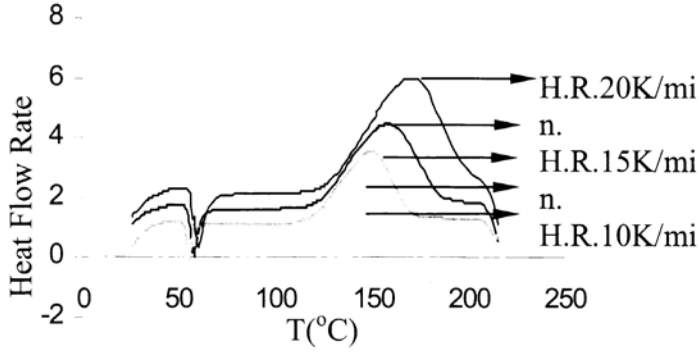


Figure 1: DSC Thermograms of  $Se_{98}In_{1.5}Sn_{0.5}$  at Five Different Heating Rates

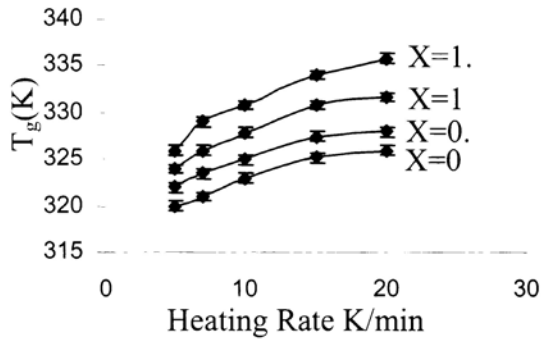
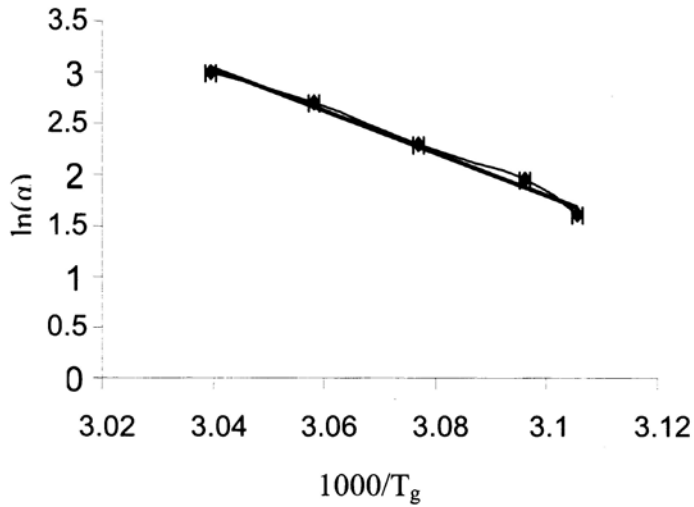
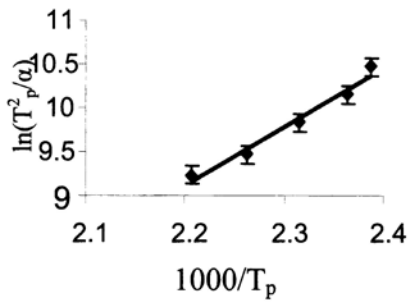


Figure 2: Glass Transition Temperature for  $Se_{98}In_{2-x}Sn_x$  ( $x=0, 0.5, 1, 1.5$ ) at Five Different Heating Rates.



**Figure 3: Plot of  $\ln\alpha$  Vs.  $1000/T_g$  for  $Se_{98}In_{1.5}Sn_{0.5}$  Semiconducting Glass.**



**Figure 4: Plot of  $\ln(T_p^2/\alpha)$  Vs.  $1000/T_p$  for  $Se_{98}In_{0.5}Sn_{1.5}$  Semiconducting Glass.**



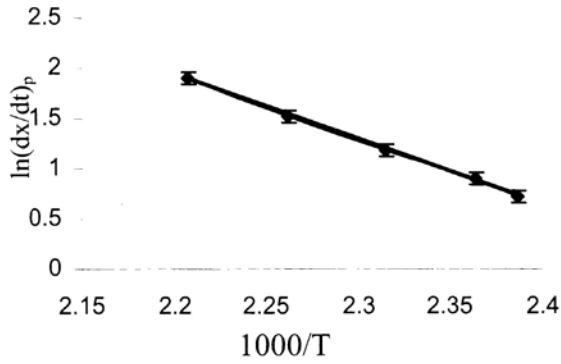


Figure 5: Plot of  $\ln(dx/dt)_p$  Vs.  $1000/T_p$  for  $Se_{98}In_{0.5}Sn_{1.5}$  Semiconducting Glass.

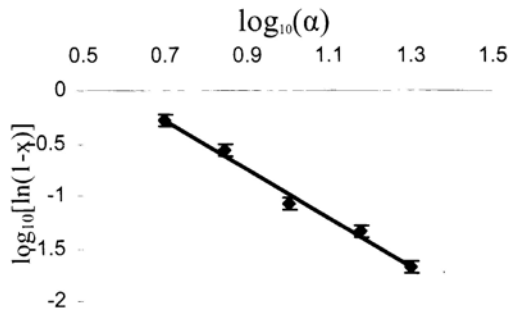


Figure 6: Plot  $\log_{10}[\ln(1-x)]$  Against  $\log_{10}(\alpha)$  for  $Se_{98}In_{1.5}Sn_{0.5}$  Semiconducting Glass.

**Table 1: glass transition activation energy, crystallization activation energy and the dimensionality of crystal growth**

Composition $Se_{98}In_{2-x}Sn_x$	$E_t$ (kJ/mol)	$E_c$ (kJ/mol) Kissinger Eq.	$E_c$ (kJ/mol) Gao et.al Eq.	n
X=0	199±3	94±5	92±3	0.8 [21]
X=0.5	166±2	63±3	64±4	2.3
X=1	138±4	59±3	58±3	2.3
X=1.5	116±2	55±2	54±3	2.4

**Table 2: Parameters that determine thermal stability of semiconducting glasses.**

Composition $Se_{98}In_{2-x}Sn_x$	$T_c-T_g$ (20K/min)	S-parameter (20K/min)	Hruby Number $H_R(20K/min)$	Crys. Rate Factor $\langle k_p \rangle / (min)$	$H_c$ (4.18J/g) (5K/min)
X=0	55	4.72	0.514	$6.57 \times 10^{-4}$	604
X=0.5	72	9.21	0.827	$4.30 \times 10^{-4}$	382
X=1	73	9.41	0.890	$4.05 \times 10^{-4}$	354
X=1.5	74	9.72	0.960	$3.72 \times 10^{-4}$	326