

Glass transition activation energy, glass-forming ability and thermal stability of $\text{Se}_{90}\text{In}_{10-x}\text{Sn}_x$ ($x = 2, 4, 6$ and 8) chalcogenide glasses

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Abstract

Differential scanning calorimeter (DSC) has been employed to investigate the glass transition activation energy E_t , thermal stability and the glass-forming ability (GFA) of $\text{Se}_{90}\text{In}_{10-x}\text{Sn}_x$ ($x = 2, 4, 6$ and 8) chalcogenide glasses. From the dependence of the glass transition temperature T_g on the heating rate β , the E_t has been calculated on the basis of Moynihan and Kissinger models. Results indicate that T_g and E_t attain their minimum values at 6 at% of Sn. Thermal stability has been monitored through the calculation of the temperature difference $T_c - T_g$, the stability parameter S , the enthalpy released during the crystallization process H_c and the crystallization rate factor K_p . The GFA has been investigated on the basis of Hurby parameter H_r , which is a strong indicator of GFA, and the relaxation time. Results of GFA are in good agreement with the fragility index F_i calculation and indicate that $\text{Se}_{90}\text{In}_4\text{Sn}_6$ is the best glass former. The compositional dependence of the above-mentioned parameters was discussed on basis of the topological model of Thorpe and Phillips and the critical composition found to occur at an average coordination number $\langle z \rangle \approx 2.36$, which is thermally most stable. © 2007 Elsevier B.V. All rights reserved.

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1. Introduction

Semiconducting chalcogenide glasses remain an interesting object in the field of disordered solids due to their wide range of application in optoelectronics as optical waveguide [1], infrared optical fibers [2], fabrication of inexpensive solar cells [3] and in optical recording systems [4]. Since the discovery [5] of switching and optical memory effects, amorphous Se has become a material of considerable commercial importance in device technology. The rapidly increasing use of amorphous Se motivated several authors to improve its physical properties, of low sensitivity and thermal instability, by alloying with other elements.

The structure of amorphous Se [6–9] and the effect of alloying In into Se have been carried out by workers and reported [10,11] in the literature. These studies indicate that when In is incorporated to amorphous Se it is dissolved in the Se chains to satisfy its coordination requirements and to form a cross-link structure, which retarded the crystallization probability and enhanced thermal stability. Besides, it is also found that the optical band gap of Se–In is of the order of 1.3 eV at 300 K, which is close to the theoretical optimum for solar energy conversion. In spite of that, these alloys are still found to have low glass transition and crystallization temperatures and hence their physical properties may deteriorate with temperature and time during use. The addition of a third element, like Sn, to Se–In alloys will expand the glass-forming region [12] and create compositional and configurational disorder, which offer ample possibilities for controlling the desired thermal properties by means of changing the chemical composition.

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Understanding the glass transition kinetics of chalcogenide glasses is therefore of great importance to establish their thermal stability and glass-forming ability (GFA) and ultimately to determine the useful range of operating temperatures for a specific technological application before the eventual crystallization takes place.

The GFA of a glassy alloy is related to ease by which melt can be cooled with the avoidance of crystal formation. Meanwhile, the thermal stability indicates the resistance of crystallization of glassy alloy through the nucleation and growth process. To evaluate the GFA and thermal stability of chalcogenide glasses, different simple quantitative methods are usually used. The most commonly used methods are those suggested by Kissinger [13] and Moynihan [14], which are based on some characteristic temperatures such as the glass transition T_g , crystallization T_c and melting T_m temperatures and the activation energy of thermal relaxation E_t as monitored by the differential scanning calorimeter (DSC).

The aim of the present paper is to report the effect of variation of Sn on the GFA and thermal stability of $\text{Se}_{90}\text{In}_{10-x}\text{Sn}_x$ ($x = 2, 4, 6$ and 8) semiconducting glasses using DSC. In this regard, the dependence of T_g on the heating rate with varying composition has been studied and the activation energy of thermal relaxation has been deduced on the basis of Kissinger and Moynihan models. The requirements of thermal stability have been investigated through the calculations of the temperature difference $T_c - T_g$, thermal stability parameter S , the enthalpy released during the crystallization process H_c and the crystallization rate factor K_p . Meanwhile, the GFA has been investigated through the calculation of Hruby parameter H_r and the total relaxation time τ_{T_g} . Besides, to ascertain that the prepared glasses were obtained from strong glass-forming liquids, the fragility index F_i has also been obtained.

2. Experimental details

High-purity (99.999%) selenium, indium and tin in appropriate atomic percent proportions were weighed in a quartz glass ampoule (length 5 cm and internal diameter 8 mm). The content of the ampoule was sealed in vacuum at 10^{-6} Torr and heated in a furnace where the temperature was raised at a rate of 3–4 K/min up to 1100 K and kept around that temperature for 10 h. The ampoule was frequently rocked to ensure the homogeneity of the sample. The molten sample was then rapidly quenched in ice-cold water. About 10 mg of the powder samples were taken in aluminum pan and subjected to the DSC (Perkin Elmer DSC-7) at five different heating rates (10, 20, 30, 40 and 50 K/min). The temperature precision of this equipment is ± 0.1 K with an average standard error of about 1 K in the measured values. The temperature range covered in DSC is from room temperature to 400 K. 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 the literature [15]. The instrument constant of this DSC, as measured, is 1.

3. Results and discussion

3.1. Dependence of T_g on the heating rate

Fig. 1 shows the DSC thermograms obtained for $\text{Se}_{90}\text{In}_6\text{Sn}_4$ chalcogenide glasses at five different heating rates 10, 20, 30, 40 and 50 K/min. All the DSC traces exhibit double endothermic peaks, which are characteristic of glass transition region followed by a single exothermic peak at higher temperatures and is the characteristic of crystallization region. As suggested by other workers [2,8–11,16,17], both T_g and T_c have been defined as the temperatures that correspond to the intersection of two linear portions adjoining the transition elbow of the DSC traces in the endothermic and exothermic directions, respectively. The appearance of a double glass transition at T_{g1} and T_{g2} indicates unusual phase separation occurring in these glasses. This manifestation of phase separation by double T_g arises after the glass to super cooling transition and has been observed in many chalcogenide glasses [22,23]. It is also mentioned [18–23] that, for compositions with single crystallization temperature, glasses may still be diaphasic but the phases crystallize at nearly the same temperature. It is pointed out [18–23] that the glass transition temperature, which in the present study taken as T_{g1} , can be considered as T_{g1} or T_{g2} . The obtained values of T_{g1} for $\text{Se}_{90}\text{In}_{10-x}\text{Sn}_x$ ($x = 2, 4, 6$ and 8) at five different heating rates are given in Table 1. It is interesting to note that T_{g1} decreases with the addition of Sn and attains minimum value at 6 at% of Sn and again increases at 8 at%. Meanwhile, T_c shows a behavior

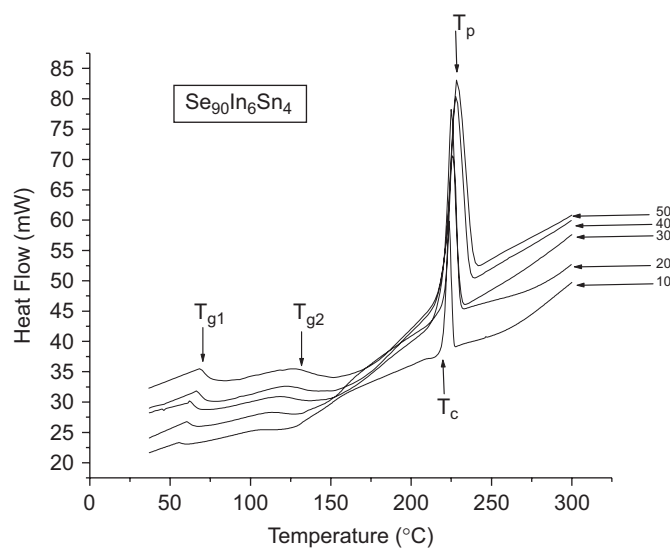


Fig. 1. Non-isothermal DSC curves of $\text{Se}_{90}\text{In}_6\text{Sn}_4$ glass at five different heating rates (10, 20, 30, 40 and 50 K/min).

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