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A thermo-physical study of In additive Se–Te chalcogenide glasses

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Abstract. The study discusses the impact of indium on the thermo-physical properties of $Se_{85-x}Te_{15}In_x$ $(0 \leq x \leq 15)$ glassy alloys. Theoretical predictions of some physical parameters are taken and an effort is made to describe the various trends of the examined physical properties with the addition of indium content. It is observed that the structural parameters, namely coordination number, constraints, heat of atomization and mean bond energy, increase with increasing indium concentration. Experimentally observed and theoretically calculated glass transition temperature (T_g), using Gibbs–Di Marzio relation and an empirical equation formulated by Tichy and Ticha, is increased with the addition of indium. The results indicate that the incorporation of In to the Se–Te system increases the rigidity of this system. However, the theoretically predicted T_g (Tichy and Ticha approach) is not consistent with experimental values.

Keywords. Chalcogenide glasses; DSC; thermo-physical study; glass transition temperature.

1. Introduction

Chalcogen-based alloys have made manifestations in the last two decades owing to their exciting characteristics that can be meticulously controlled by changing their chemical compositions. Selenium-based semiconducting glasses are of specific interest because of their current utilization as photoconductors in high-definition TV pickup tubes, and mainly in digital X-ray imaging. Recently, Se–Te-based glassy semiconducting alloys have been the focus of scientific and technological applications. Although they are considered capable compositions for phase-change memory materials, they present significant issues when employed as a recording layer in optical phase-change devices [1,2]. Mainly two issues are of concern: lesser reversibility [3] and lower glass transition as well as crystallization temperatures. The mentioned disadvantages may be overwhelmed by doping another suitable element to the Se–Te system as a chemical modifier. The insertion of a third element, such as indium, creates compositional and configurational disorder in the Se–Te system and gives stability in thus formed glassy alloys [4,5]. The addition of indium to the Se-rich Se–Te system forms a cross-linked structure that increases the glass transition and crystallization temperatures, resulting in a harder Se–Te alloy matrix [4,5].

In this research work, indium is chosen as a doping element to the Se–Te system to realise the impact of the addition of this element on physical properties. The use of indium as a doping element for the Se–Te system is due to its more attractive and important advantages in chalcogen-based glassy alloys [6–9]. It is found in other research contributions that the addition of indium alters the transport properties [10,11]. The ternary system under study may be a probable entrant for reversible PC optical recording material as adding indium in chalcogen-based binary Se–Te glassy samples will impact minimum eraser time [12]. The negligible study of physical properties of indium additive Se–Te ternary glasses is reported in the literature and motivates us to study these materials in detail.

The purpose of the research contribution is concerned with examining the compositional dependence of physical properties: average coordination number $< r >$, average number of constraints f, lone-pair electrons, bond energy, cohesive energy, the heat of atomization, deviation of stoichiometry, mean bond energy, electronegativity and glass transition temperature (T_g) for $Se_{85-x}Te_{15}In_x$ $(0 \leq x \leq 15)$ glassy alloys. T_g is important to describe that glass could exist in this phase with operating temperature, and here it is estimated theoretically using the relation given by Tichy *et al.* [13] and the modified Gibbs–Di Marzio equation [14]. The estimated theoretical values were compared to experimental results to verify the accuracy of the deduction of T_g.

2. Experimental

Bulk samples of $Se_{85-x}Te_{15}In_x$ $(0 \leq x \leq 15)$ are synthesized via melt-quench procedure. Each material of high purity (99.999%) is weighed based on atomic percentage and sealed in quartz ampoules under vacuum ($\sim 2 \times 10^{-5}$ mbar). Thereafter, these ampoules are placed in the furnace and the temperature is raised as per the usual procedure. The final maximum temperature of 900°C is maintained for 10 h, and the ampoule is also rocked often to maintain the homogeneity of the melt. The melted substance is rapidly cooled by submerging it in ice-cold water.

The synthesized bulk samples are thus ground into a fine powder for further study. X-ray diffraction of the prepared bulk powder samples was carried out at room temperature in the range $10° < 2\theta < 90°$. The X-ray diffraction pattern of the parent sample $Se_{85}Te_{15}$ is given in another publication [15]. Figure 1 shows the patterns of the bulk samples ($x = 2$, 6, 10 and 15) with indium additives that are currently under investigation. The absence of prominent or sharp peaks ensures the amorphous or glassy nature of the synthesized samples. The thermal behaviour of synthesized bulk samples is obtained using a differential scanning calorimetry (DSC) experiment. About 5.0 mg of powdered sample is encapsulated in a typical pan and reheated at heating rates: 5, 10, 15 and 20°C min^{-1} between 30 and 500°C. Table 1 lists characteristic temperatures: glass transition temperature T_g, onset temperature of crystallization T_o, peak temperature of crystallization T_p and melting temperature T_m data derived from the DSC curves. The characteristic temperatures shift to higher temperatures with increasing heating rate (α). Figure 2a–c displays DSC curves of $Se_{83}Te_{15}In_2$, $Se_{79}Te_{15}In_6$ and $Se_{75}Te_{15}In_{10}$ at four different heating rates of 5, 10, 15 and 20°C min^{-1} and the similar DSC curves are also observed for parent sample $Se_{85}Te_{15}$ and $Se_{70}Te_{15}In_{15}$ [5,15]. It is found that both endothermic and exothermic temperatures, i.e. T_g and crystallization temperature (T_p), respectively increase with increasing indium content (table 1). The peak values of T_g and T_p are considered in all calculations and conclusions.

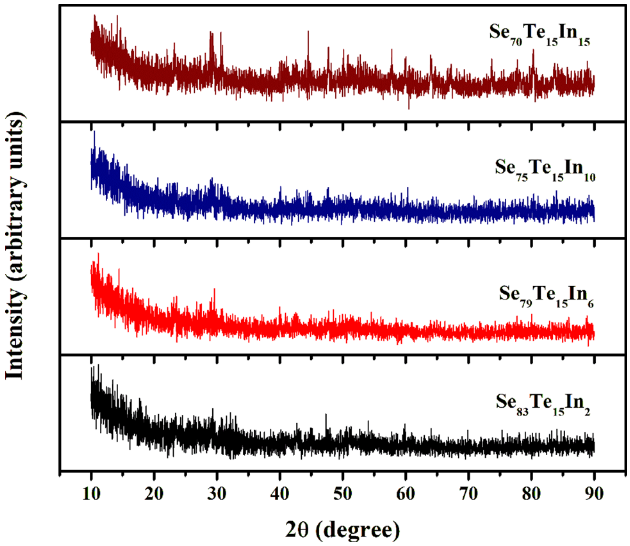


Figure 1. X-ray diffraction patterns of In additive chalcogenide $Se_{85-x}Te_{15}In_x$ (x = 2, 6, 10 and 15 at.%) glasses.

3. Results and discussion

3.1 *Determination of coordination number, constraints and fraction of floppy modes*

The average coordination number is vital as it tells about the cross-linking in the glassy structure and may be formulated as the atom-averaged covalent coordination of the constituent elements. The average coordination number $< r >$ in a ternary compound $Se_aTe_bIn_c$ (a, b and c are the atomic percentages) is calculated using the standard procedure [16]:

$$< r > = \frac{aZ_{Se} + bZ_{Te} + cZ_{In}}{a + b + c}, \tag{1}$$

where Z_{Se}, Z_{Te} and Z_{In} are coordination number of elements in subscript (table 2). The value of $< r >$ for $Se_{85-x}Te_{15}In_x$ system has been evaluated (table 3), indicating that it enhances with increasing indium content (figure 3). The average coordination number pertaining to covalent bonds is the measure representing the character of atomic units. It is found in the range between 2.00 and 2.15 for the considered system.

In any chalcogenide glass system, covalent coordinating units may be forced to mechanical constraints by interatomic valence forces, viz., bond-stretching and bond-bending. The optimum composition of the glass is formed when the constituting network attains the mechanical critical point. The critical point is attained when constraints per atom (N_{con}) become equivalent to the degrees of freedom per atom. N_{con} arises from bond-bending ($N_\beta = 2 < r > - 3$) and bond-stretching ($N_\alpha = < r >/2$) [17]. Therefore, the average number of constraints N_{con} is given as ($N_\alpha + N_\beta$) and calculated values for each investigated sample are listed in table 3. Thus, it can be seen that N_{con} increases in this composition; therefore, the coordination number becomes a prominent factor as the degree of cross-linking is enhanced with the increase in N_{con}. The variation of N_{con} with indium content is described in figure 3.

According to Thorpe [18], the uncoordinated networks having a finite fraction of zero frequency normal vibration modes are termed as floppy modes in the absence of weak long-range forces. The fraction f of zero frequency modes is given by:

$$f = 2 - \frac{5}{6} < r > . \tag{2}$$

f is calculated for varying composition of In at the cost of Se and is listed in table 3. It has been observed that with increasing In content in the system, the value of the fraction of floppy modes (f) decreases (figure 4), indicating that the rigidity of the system increases.

3.2 *Lone-pair electrons and ease of glass formation*

Chalcogen-based glassy alloys are often called lone-pair semiconductors. The presence of lone-pair electrons in

Table 1. Characteristic temperatures: T_g, T_o, T_p and T_m data derived from the DSC curves.

		Heating rate			
Composition	Characteristic temperature	5°C min^{-1}	10°C min^{-1}	15°C min^{-1}	20°C min^{-1}
$Se_{85}Te_{15}$	T_g	335.23	337.77	339.11	340.08
	T_o	357.98	361.71	365.51	365.10
	T_p	372.02	375.87	380.99	380.57
	T_m	517.30	518.23	519.51	518.89
$Se_{83}Te_{15}In_2$	T_g	338.04	341.27	343.57	345.34
	T_o	367.32	373.59	376.10	378.70
	T_p	388.60	397.90	398.29	405.31
	T_m	520.05	521.68	523.38	524.02
$Se_{79}Te_{15}In_6$	T_g	339.21	343.39	345.57	347.41
	T_o	373.88	383.24	381.18	390.10
	T_p	392.63	401.44	403.51	410.35
	T_m	522.77	525.38	525.25	526.90
$Se_{75}Te_{15}In_{10}$	T_g	341.00	344.52	346.85	348.69
	T_o	374.62	379.01	382.18	385.31
	T_p	392.66	399.81	403.80	407.27
	T_m	526.65	528.20	529.34	530.53
$Se_{70}Te_{15}In_{15}$	T_g	344.79	348.95	351.65	353.78
	T_o	382.82	386.92	390.51	395.91
	T_p	392.38	399.09	405.10	408.37
	T_m	537.55	539.98	541.29	543.18

chemical bonds leads to flexibility, and these types of bonds can be easily distorted compared to the bond without unshared electrons. Thus, larger the lone-pair electrons lesser will be the strain energy in the system, which in turn favours glass formation [19]. Therefore, lone-pair electrons have vital importance in the formation of chalcogen-based glassy materials. The significance of lone-pair electrons can be understood in the context of valence shell electron pair repulsion theory and adequate number of these electrons leads to the existence of stable state in the system.

Zhenhua [20] proposed a criterion to determine the tendency of chalcogen-based systems to form a glassy state. This criterion states that a certain number of lone-pair electrons (L) are required to form a glassy state in the system. According to said criterion, (i) $L > 4$ for individual elemental system, (ii) $L > 2.62$ for any binary system and (iii) $L > 1$ for any ternary system. Many materials that can be solidified in the glassy-like state are found to possess structural 'bridges'. In glassy materials, bridge formation is observed with periodic table elements from groups VI and VII. The presence of bridges of atoms along with lone-pair electrons leads to flexibility [21]. Selenium exists with two lone-pair electrons. The flexibility of chemical bonds pertaining to chalcogens leads the atoms to form amorphous networks instantly. The number of lone-pair electrons for the present chalcogen-based system is estimated using the following equation [22]:

$$L = V - <r>, \qquad (3)$$

where V is the valence electrons. The value of L calculated from equation (3) is given in table 3 and it can be observed that L decreases as indium is incorporated into the parent binary system. The minimum value of lone-pair electrons is 3.25, which directs that the composition considered for study may lead to a good glass-forming system. The plot of L *vs*. indium at.% is shown in figure 4.

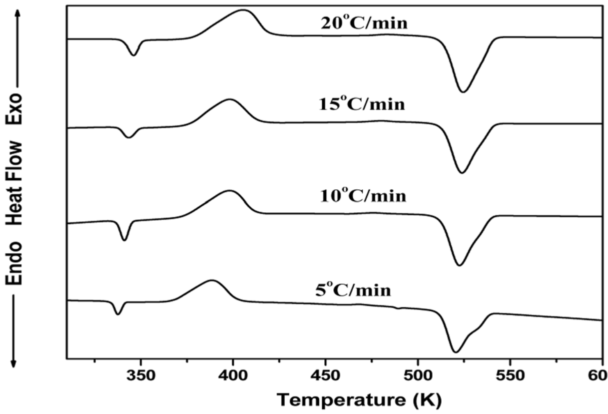
3.3 *Chemical-bonds and electronegativity*

The probable formation of bonds in the present system under analysis are Se–Se, Se–Te, Se–In, etc. CONM [23] recommends that hetero-polar bonds are favoured compared to homo-polar bonds. Also, these bonds are created in such a way that bonds with lower bond energy are formed later. Hetero-polar bond energy (D_{A-B}) can be estimated via homo-polar bond energy and electronegativity of the constituent elements [24] using the expression given below:

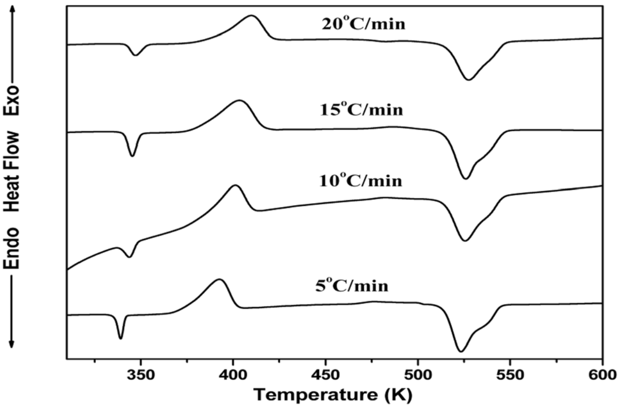
$$D_{A-B} = [D_{A-A}.D_{B-B}]^{1/2} + 30(X_A - X_B)^2, \qquad (4)$$

where D_{A-A} and D_{B-B} are homo-polar bond energies and X_A and X_B are the electronegativity values of constituent elements. Both electronegativity values and homo-polar bond energies for Se, Te and In are given in table 2. Further, the formation of bonds happens in such a way that bonds with the highest possible energy are expected to occur first, followed by bonds with decreasing energy until all the

(a) $Se_{83}Te_{15}In_2$



(b) $Se_{79}Te_{15}In_6$



(c) $Se_{75}Te_{15}In_{10}$

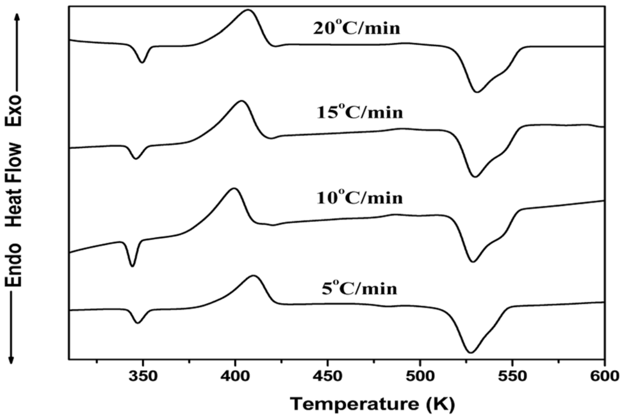


Figure 2. DSC curves of (**a**) $Se_{83}Te_{15}In_2$, (**b**) $Se_{79}Te_{15}In_6$ and (**c**) $Se_{75}Te_{15}In_{10}$ at four different heating rates of 5, 10, 15 and 20°C min^{-1}.

available valence of atoms is satisfied. In the examined composition, In–Se bonds (D_{In-Se} = 227.28 kJ mol^{-1}) are expected to occur first for having the highest possible bond energy, lagged by Se–Te bonds (D_{Se-Te} = 184.92 kJ mol^{-1}) to fill all available valence of selenium. Some of the unfilled vacancies may be filled with homo-polar Se–Se defect bonds.

Table 2. Physical parameters of the constituent elements.

Physical parameter	Se	Te	In
Coordination number	2	2	3
Electronegativity	2.55	2.1	1.7
Bond energy (kcal mol^{-1})	44.04	33.0	24.2
Heat of atomization (kcal per g-atom)	58	46	49.4

Cohesive energy (CE) can be deduced knowing these bond energies. The chemical bond approach [25] can be used to calculate the cohesive energy of the investigated samples by considering the additive nature of bond energies. It is defined as the stabilization energy of an infinitely large cluster of material per atom. Therefore, CE of primed glasses is estimated by taking the sum of bond energy of all the bonds assumed in the system as below:

$$\mathrm{CE} = \sum \frac{C_\mathrm{i} D_\mathrm{i}}{100}, \tag{5}$$

where C_i is the expected chemical bond and D_i is the corresponding bond energy. The evaluated CE and distribution of chemical bonds in each investigated glass are reported in table 4. It is observed that CE increases with increasing indium concentration. Values of electronegativity (X) for $Se_{85-x}Te_{15}In_x$ ($0 \leq x \leq 15$) glasses are also deduced by Sanderson's method [26], according to which it is the geometric mean of electronegativity values of the atoms that constitute the glass. It can be found from table 4 and figure 4 that the electronegativity declines with the enhancement in indium concentration.

3.4 *Average heat of atomization, deviation of stoichiometry and mean bond energy*

3.4a *Average heat of atomization:* The heat of atomization is the amount of heat required to change one mole of an atom in its standard state (25°C) to gaseous atoms. The heat of atomization $H_\mathrm{s}^{\mathrm{AB}}$ at normal temperature and pressure of binary semiconductor made of elements A and B is the sum of the heat of atomization ΔH and the average of the heats of atomization, $H_\mathrm{s}^{\mathrm{A}}$ and $H_\mathrm{s}^{\mathrm{B}}$, corresponding to the average non-polar bond energy of the two elements [27]:

$$H_\mathrm{s}^{\mathrm{AB}} = \Delta H + \frac{1}{2}\left[H_\mathrm{s}^{\mathrm{A}} + H_\mathrm{s}^{\mathrm{B}}\right] \tag{6}$$

with

$$\Delta H \propto (X_\mathrm{A} - X_\mathrm{B})^2. \tag{7}$$

Sadagopan and Gatos [28] extended this to higher-order semiconductor alloys and using this, the average heat of

Table 3. Values of $< r >$, N_α, N_β, N_{con}, f, number of lone pair electrons L, H_s, average single bond energy ($H_s/< r >$), R and mean bond energy ($< E >$) for $Se_{85-x}Te_{15}In_x$ alloys.

Composition	$< r >$	N_α	N_β	N_{con}	f	L	H_s	$H_s/< r >$	R	$< E >$
$Se_{85}Te_{15}$	2.00	1.00	1.00	2.00	0.333	4.00	48.99	24.45	∞	1.91
$Se_{83}Te_{15}In_2$	2.02	1.01	1.04	2.05	0.300	3.90	49.06	24.05	32.7	1.95
$Se_{79}Te_{15}In_6$	2.06	1.03	1.12	2.15	0.233	3.70	49.41	23.30	10.4	2.04
$Se_{75}Te_{15}In_{10}$	2.10	1.05	1.20	2.25	0.167	3.50	49.75	22.61	6.0	2.15
$Se_{70}Te_{15}In_{15}$	2.15	1.08	1.30	2.38	0.083	3.25	50.18	21.82	3.8	2.30

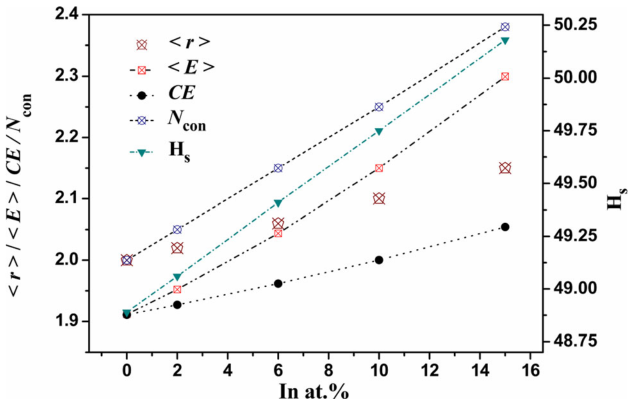


Figure 3. Variation of $< r >$, $< E >$, CE, N_{con} and H_s with In at.%.

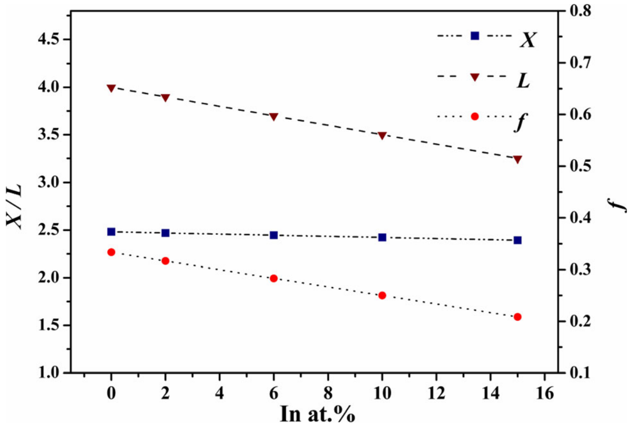


Figure 4. Plot of X, L and f *vs.* In at.%.

atomization (H_s) for a compound $Se_aTe_bIn_c$ may be defined as:

$$H_s = \frac{aH_s^{Se} + bH_s^{Te} + cH_s^{In}}{a+b+c} \tag{8}$$

H_s^{Se}, H_s^{Te} and H_s^{In} of the superscript elements are given in table 2. The calculated values for the investigated $Se_{85-x}Te_{15}In_x$ ($0 \leq x \leq 15$) glassy alloys are given in table 3 and it can be seen that H_s increases with the increase in indium concentration. The graphical presentation of H_s for different studied compositions is shown in figure 3. Table 3 includes the average single bond energy $H_s/< r >$.

3.4b *Deviation of stoichiometry R:* R is defined as the ratio of the covalent bonding possibilities of chalcogen elements to that of non-chalcogen elements in a system. For any ternary $Se_aTe_bIn_c$ system, R may be deduced using the expression [29,30]:

$$R = \frac{aZ_{Se} + bZ_{Te}}{cZ_{In}}. \tag{9}$$

The value of R for $Se_{85-x}Te_{15}In_x$ is determined and given in table 3. It is said that if R is greater than unity ($R > 1$), it indicates chalcogen-rich materials, while $R < 1$ indicates chalcogen-poor materials. Therefore, $R = 1$ is the critical point where hetero-polar bonds exist. From table 3, it is obvious that $R > 1$ for all investigated glasses indicates that these examined glasses are rich in chalcogens.

3.4c *Mean bond energy:* The characteristics of chalcogen additive glassy alloys may be linked with the overall mean bond energy $< E >$ because it relates to average coordination number, bond energy and types of bonds. Using the Tichy *et al.* [13] expression, $< E >$ for a chalcogen Se-rich $Se_aTe_bIn_c$ system is given by:

$$< E > = E_{cl} + E_{rm}, \tag{10}$$

where E_{cl} is due to strong hetero-polar bonds and may be written as:

$$E_{cl} = P_r D_{hb}, \tag{11}$$

where P_r represents the tendency for cross-linking and can be deduced by:

$$P_r = \frac{bZ_{Te} + cZ_{In}}{a+b+c}, \tag{12}$$

whereas D_{hb} represents average hetero-polar bond energy and is proposed as:

$$D_{hb} = \frac{bZ_{Te}D_{Se-Te} + cZ_{In}D_{In-Se}}{bZ_{Te} + cZ_{In}}. \tag{13}$$

Table 4. Values of electronegativity X, CE (eV per atom), distribution of their respective bonds and excess Se–Se bonds for $Se_{85-x}Te_{15}In_x$ alloys.

		Distribution of chemical bonds				
Composition	X	Se–Se	Se–Te	In–Se	CE	Excess Se–Se bonds
$Se_{85}Te_{15}$	2.48	0.823529	0.176471	—	1.91	140
$Se_{83}Te_{15}In_2$	2.47	0.783133	0.180723	0.036145	1.93	130
$Se_{79}Te_{15}In_6$	2.45	0.696203	0.189873	0.113924	1.96	110
$Se_{75}Te_{15}In_{10}$	2.42	0.600000	0.200000	0.200000	2.00	90
$Se_{70}Te_{15}In_{15}$	2.39	0.464286	0.214286	0.321429	2.05	65

Table 5. Glass transition temperature using the formulations of Tichy and Ticha [29,30]. Modified GDM law [14] and experimental values at three different heating rates for $Se_{85-x}Te_{15}In_x$ alloys.

	Heating rate						
	10°C min^{-1}		15°C min^{-1}		20°C min^{-1}		
Composition	Modified GDM law	Exp.	Modified GDM law	Exp.	Modified GDM law	Exp.	Tichy and Ticha
$Se_{85}Te_{15}$	—	337.77	—	339.11	—	340.08	314.42
$Se_{83}Te_{15}In_2$	340.08	341.27	341.95	343.57	342.93	345.34	319.44
$Se_{79}Te_{15}In_6$	341.78	343.39	343.14	345.57	344.12	347.41	330.24
$Se_{75}Te_{15}In_{10}$	343.58	344.52	344.95	346.85	345.93	348.69	342.19
$Se_{70}Te_{15}In_{15}$	347.40	348.95	348.78	351.65	349.78	353.78	359.06

Further, the augmentation arising from the weaker bonds that persist even after all strong bonds are maximized is deduced by the relation:

$$E_{\mathrm{rm}} = \frac{2D_{\mathrm{Se-Se}}[0.5(<r>) - P_{\mathrm{r}}]}{<r>}. \quad (14)$$

The calculated value of $< E >$ for all compositions of investigated $Se_{85-x}Te_{15}In_x$ $(0 \leq x \leq 15)$ glassy alloy are listed in table 3. It can also be observed that $< E >$ enhances with the enhancement in indium concentration. Figure 3 shows the enhancement of $< E >$ as the at.% of indium increases.

3.5 *Glass transition temperature*

The temperature associated with the glass transition region (here denoted as T_g) is vital for describing the vitreous state. It may be predicted by making use of different theoretical methods. T_g can be calculated using an expression suggested by Tichy and Ticha [29,30]:

$$T_g = 311[<E> - 0.9]. \quad (15)$$

The deduced values of T_g are mentioned in table 5 and enhancement with indium concentration is observed. It can also be determined by another theoretical approach known as Gibbs–Di Marzio's law. An empirical relation is developed between T_g and cross-linking density in the system of molecular chains. Sreeram *et al* [14] modified Gibbs–Di Marzio equation and suggested the following form:

$$T_g = \frac{T_o}{1 - \beta(<r> - 2)}. \quad (16)$$

Here T_o is taken as T_g of base glass (binary $Se_{85}Te_{15}$ alloy) and the denominator of the above expression defines the significance of cross-linking. β is a system-dependent parameter and is evaluated for each ternary alloy by substituting the values of T_g and T_o at a known heating rate (5°C min^{-1}) and the value of $< r >$ in equation (16). The values of T_o for the non-crosslinked $Se_{85}Te_{15}$ alloy at the other three heating rates used in the present work are given in table 5. T_g values for investigated In additive alloys obtained experimentally and estimated theoretically are tabulated in table 5 at the other three heating rates. The good harmony is seen between experimentally observed and theoretically estimated T_g for indium additive ternary alloys at the three heating rates. However, applying the Tichy–Ticha approach [29,30] to $Se_{85-x}Te_{15}In_x$ system, the estimated T_g is not consistent with experimentally observed T_g. However, there is an increasing trend in T_g with increasing

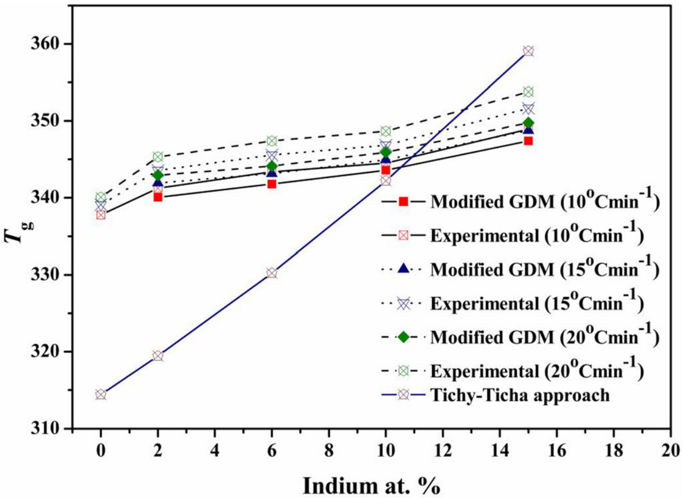


Figure 5. Variation of experimental and theoretical values of T_g with indium.

indium concentration (table 5 and figure 5). The increasing trends of the theoretical as well as the experimental values (table 5) indicate that the addition of In to binary $Se_{85}Te_{15}$ alloy by compromising Se increases the rigidity of the system. The inclination of T_g with the enhancement of indium concentration can be described by considering the composition and configuration modifications due to indium doping. The more general known approach of amorphous selenium considers its two molecular species [31]: (i) meandering chains comprising helical chains of trigonal selenium and (ii) Se_8 ring molecules of monoclinic selenium. The network of any Se–Te unit synthesized by the melt-quench procedure is taken [31–34] as the combination of Se_8 rings, Se_6Te_2 rings and Se–Te copolymer chains. As indium doping occurs by compromising selenium in any binary Se–Te unit, the number of Se–Se long chains likely increases relative to the number of Se_8 rings, resulting in a reduction [35]. It is recognized that [36] T_g enhances with the increase in chain length and declines with the increase in ring concentration.

4. Conclusions

The present study of thermo-physical properties of $Se_{85-x}Te_{15}In_x$ ($0 \leq x \leq 15$) glasses shows that the insertion of indium at the cost of Se leads to changes in physical parameters. Results indicate that adding indium in the system $Se_{85-x}Te_{15}In_x$ leads to an increase in coordination number, the heat of atomization, average bond strength and the number of constraints. But L, f and electronegativity values decrease in the same trend. CE of examined glasses is deduced using CBA and is found to increase with the increase in In at.%. T_g is theoretically estimated using Tichy and Ticha methods and a modified Gibbs–Di Marzio's law. The theoretically predicted and experimental values of T_g at different heating rates show an increasing trend with an increase in indium content. However, theoretically deduced T_g by considering Tichy and Ticha relation is not in harmony with experimentally observed values.

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