

Caloric Study of the Bulk Chalcogenide (Bi₁Te₁₅Se_{84-x}Pb_x) Quaternary Alloy System

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Abstract: Bulk glasses of the composition Bi₁Te₁₅Se_{84-x}Pb_x, where $0 \leq x \leq 8$, were synthesized by conventional melt-quenching methods. The crystallization and thermal kinetics of these alloys were investigated through differential thermal analysis (DTA) at various heating rates of 5 K/min, 10K/min, 15 K/min, and 20 K/min. Key kinetic parameters, including the activation energies, were derived from models like Kissinger's and Augis-Bennett's using the functional relationship between heating rate and glass transition temperature (T_g). Interaction of glass transition and crystallization activation energies were tracked as a function of different heating rates and compositions. The amorphous-to-crystalline phase transition, glass-transition behavior, and glass-forming ability of the chalcogenide alloys are described in detail. Important thermal parameters: activation energy for crystallization and glass transition, fragility index, and Hruby's parameter were calculated. The empirical methods were used to estimate E_c and E_g . The average activation energy for crystallization is (122.93 ± 2.95) kJ/mol, whereas for glass transition, it is (203.45 ± 4.12) kJ/mol. The reduced glass transition temperature (T_{rg}) together with the fragility index (F_i) confirmed that the glassy alloy derived from a good glass-forming liquid. The focus of this manuscript is the preparation of glass using the conventional melt-quenching technique and the study of their structural and thermal properties, which is the fundamental basis for this research work.

Keywords: Differential thermal analysis; Glass activation energy; Fragility index; Chalcogenide glasses

1. Introduction:

Thermoelectric materials have garnered significant research attention all over the world due to their possible applications in converting heat into electricity [1]. Earlier studies found that Pb (lead) gave better electrical and optical characteristics of metal-chalcogenide semiconductors, making them suitable for optoelectronic applications [2]. Compounds of Sn or Pb with chalcogen elements, such as Te, S, or Se, have become well-known worldwide for possessing great properties, making them strong candidates for next-generation optoelectronic, thermoelectric, and electronic devices [3].

Among these, amorphous materials, such as chalcogenide glasses based on alloying antimony (Sb), tellurium (Te), and selenium (Se), are highly promising in constructing various solid-state devices. These materials can further improve their properties by doping them with rare earth elements to enhance their performance [4]. Chalcogenide glasses' superior glass-forming capabilities, strong thermal stability, and wide range of possible uses have prompted extensive

research [5-9]. These glasses are transparent across a broad spectrum, from the visible to infrared, and hence are ideal for use in optical lenses, infrared imaging systems, and in various optoelectronic applications. While research on both binary and ternary nanostructured glasses is progressing satisfactorily, work on quaternary nanostructured glasses is still a relatively new endeavor.

This quaternary system that has been chosen for the purpose of this study gains a certain importance from its noteworthy role in a class of materials belonging to the family of chalcogenide glasses. The presence of a fourth element in the system has been shown to promote reversibility while raising both the glass transition temperature, T_g , and the crystallization temperature, T_c . Specifically, the incorporation of bismuth into the base matrix leads to a shift in the electrical conductivity from p-type to n-type behavior [10]. Bi further modifies the system's characteristics by causing structural and compositional chaos [11].

The determination of material properties requires knowledge of other parameters. For instance, the temperature of glass transition (T_g) is quite sensitive to the heating rate and can be used to determine characteristics for example the activation energy of glass transition (E_g) and crystallization energy (E_c). These parameters are obtained using established models, like Kissinger's method [12] and the Augis-Bennett approach [13].

The crystallization kinetics in chalcogenide glasses could be performed using two main methods: isothermal and non-isothermal [14]. By heating the sample quickly to a temperature that is close to the crystallization temperature, the isothermal approach allows for the observation of changes in certain properties over time as the crystallization process progresses. Using non-isothermal methods, the sample is heated steadily while variations in its physical characteristics are noted in relation to the temperature. In this study, the non-isothermal technique was chosen because it is much faster and provides accurate results as compared to the isothermal method.

The incorporation of even a single impurity into the Se-Te binary alloy by substitution of selenium (Se) is currently a subject of great interest on account of the vast majority of benefits it provides. The glass transition temperature (T_g) and thermal stability of the mixed system far exceed those of the original Se-Te alloy due to this alloying once lead (Pb) (or any other element) is introduced. The introduction of Pb or other agents also modifies the basic bonding setup, enabling the formation of a cross-linked network. The qualities such as crystallization peak temperature (T_p), glass transition temperature (T_g), thermal stability, glass-forming ability, and reduction of aging effects are all improved by this structural alteration [15–19]. These materials' glass-forming ability and chemical durability are well known, and more recent research has shown that the intermediate phases for chalcogenide glasses correspond to compositions that have a stable, stress-free network structure and a maximum tendency to form glass. Studies conducted have stated that the relevant composition in question represents a juncture wherein maximal realizability of esteemed properties is realized; such implies that this alloy is a chemically ordered material with heteropolar bonds of elevated energy.

Nanocrystalline chalcogenides, specifically Se, Te, and Pb-based ones, have garnered much attention being possible candidates in applications ranging from solar cells, photodetectors, field-effect transistors, and chemical sensors because of their unique properties [20-22]. The concepts of activation energy and glass transition are strongly interlinked with kinetic investigations, where activation energy serves a decisive role in the nucleation and growth

processes of the substance; these studies have proved helpful in providing insights into the stability and potential applications of particular materials. For example, PbTe remains one of the most widely used materials for thermoelectric power generation, particularly for high-temperature applications, due to already established thermoelectric properties. PbTe is one of the most versatile materials when it comes to thermoelectrics and optoelectronics, making it the backbone of the development of next-generation energy conversion and sensing technologies [23].

This work uses differential scanning calorimetry (DSC) and non-isothermal differential thermal analysis (DTA) techniques to examine the glass transition and crystallization kinetics of the compositions. To examine the material's performance, measurements were made at various heating rates. The kinetics evaluation was based on the application of two established methods, namely, Kissinger's equation and Augis-Bennett approximation. These methods were selected owing to their very much wide exposure and frequent application toward the same by researchers, as judged on the ground of a thorough literature review undertaken. Within this context, this research aims to provide a detailed understanding of the thermal behavior and kinetic parameters of the material.

2. Experimental Techniques:

Synthesis of the crystals was carried out using the traditional melt quenching method. High-purity reagents were mixed and sealed, under vacuum, in quartz ampoules. The sealed ampoules were heated uniformly from room temperature to 1000°C at a rate of approximately 2-3°C/min. The ampoules were shaken manually from time to time to obtain uniformity in the samples. Immediately after the desired melting temperature was reached, the ampoules were plunged into iced water for quenching from the melting temperature to avoid crystallization and achieve the glassy state.

One of the prominent advantages of the melt-quenching method is flexibility, an ability to control the composition and geometry of the material, coupled with access to larger sample sizes than those afforded by other methods. Selenium is the principal $\text{Bi}_1\text{Te}_{15}\text{Se}_{84-x}\text{Pb}_x$ material used in this composition on account of its industrial applications in electronic and optoelectronic devices. $\text{Bi}_1\text{Te}_{15}\text{Se}_{84-x}\text{Pb}_x$ is chosen as the base material for its remarkable optical, electrical, and thermal qualities. The metallic additive lead introduces modifications to the balance between the positively and negatively charged valence alteration pairs, consequently affecting the properties of the material.

Using PerkinElmer Thermal Analysis equipment, Differential Scanning Calorimetry (DSC) has been used for the investigation of the thermal behavior of the synthesized glass at different heating rates ranging from 5 to 20°C/min. This facilitates comprehension of chalcogenide glass's transition characteristics and thermal stability.

3. Results and Discussion:

The DSC curves (Figure 1) indicate that there is one glass transition and crystallization process in the sample. The major temperature regions cover three distinctive temperature ranges displayed in the DSC thermograms. The first region consists of an endothermic peak, which corresponds to the glass transition, where the material changes from a hard to soft state. The second, being exothermic in nature, corresponds to the crystallization, in which the amorphous

material begins to form a crystal structure. Ultimately, the last endothermic peak indicates the melting of the crystalline material, in which the crystalline structure breaks down to give liquid. The initial results show that, with the increase in heating rate, both T_g and T_p appear to increase. By definition, T_g is the temperature at which the structural relaxation time of the material reaches the time of measurement; hence the material must be able to adjust its atomic structure simultaneously with the observation. This essentially indicates the dependence of T_g on the heating rate: increasing the heating rate results in marked changes in relaxation dynamics during the glass transition period, and this tendency becomes less prominent as the heating rate approaches the thermal response and shortens the relaxation time [24]. Thus, the relation between T_g and relaxation time is an inverse function, which states that a heating rate increase will shorten an observation relaxation time and consequently render T_g higher.

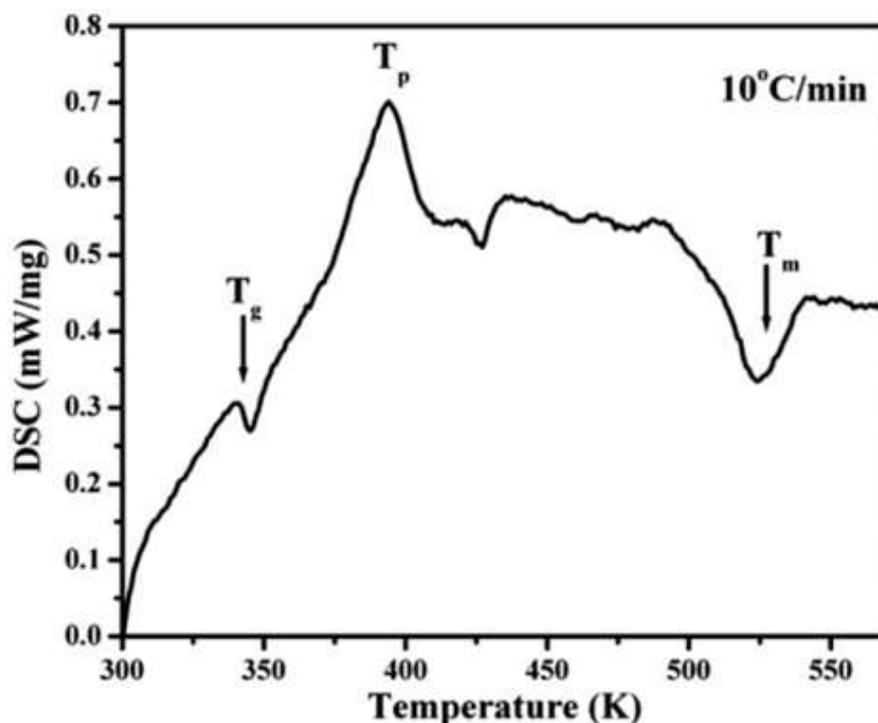


Fig. 1: DSC thermogram of the sample at a heating rate 10°C/min

This phenomenon from the increase of T_p attributed to the increasing heat rate can be explained by the fact that, upon increasing the heating rate, nucleation and crystallization occur over a shorter time interval. Therefore, since less time is available for nuclei formation and crystal growth, crystallization at higher temperatures commences. This shows how heating rates impact the glass transition and crystallization behaviors of the material.

Figure 2 depicts the glass transition temperature, T_g , versus bismuth concentration for various heating rates. The T_g increases with increasing Bi content; this observation has an explanation according to the chemically ordered networks (CON) model in which, on the whole, the material forms more heteropolar bonds than homopolar bonds. Different types of bonds can be present in the examined system: Se-Se, Bi-Se, Te-Te, Bi-Te, and Se-Te. Upon increasing the Bi concentration, Bi appears to form bonds preferentially with selenium than with tellurium because the bond energy between Bi and Se is stronger (170.4 kJ/mol) than that between Bi and Te (125.6 kJ/mol). The stronger the Bi-Se bonds, the more stable the network subsequently

developed, which in turn raises the glass transition temperature. This shows the reason behind the raised glass transition temperature with higher Bi concentration: the material becomes more stable against any structural change during the glass transition state.

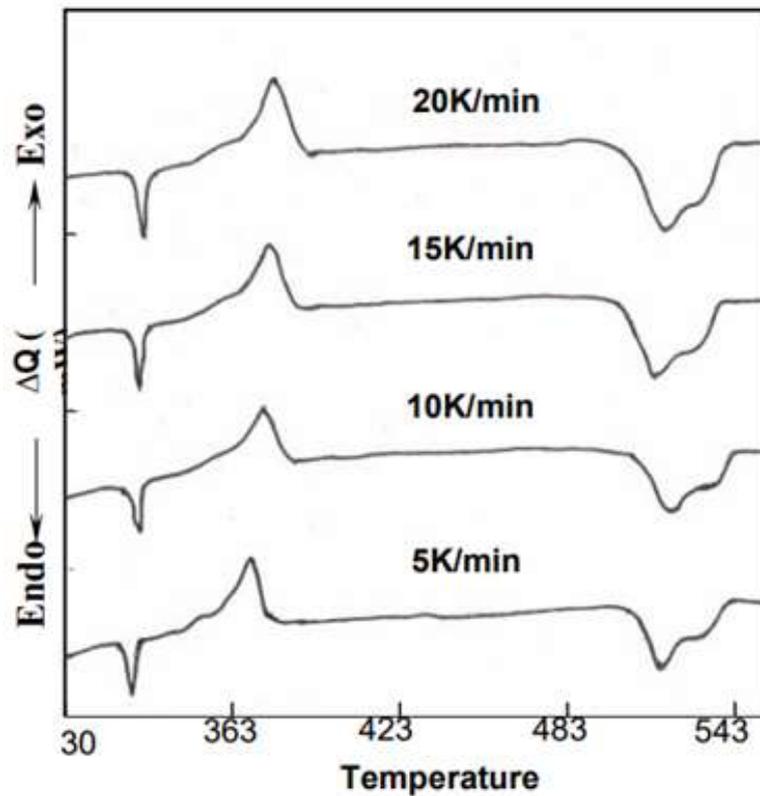


Fig 2: DSC thermograms for the sample

The activation energy for glass transition can correspond to the energy acting on atoms, enabling the transition from one metastable state to another within the glassy state. This energy is important for understanding the dynamics of molecules and the path of atom arrangement that happens in and near T_g . As the sample is heated in a DSC furnace, the atoms in the glass must overcome the energy barriers of various configurations within the configuration space of the material. This space consists of many local potential minima; each of these potentials corresponds to a certain atomic configuration in which the most-stable atomic configuration occurs when the lowest internal energy is present in the glassy structure. Since atoms tend to migrate to a metastable state of lower energy with lower activation energy, these materials are thus more stable. Further addition of bismuth (Bi) modifies the energy landscape of the glass network composition. Bi makes the glass less stable since the glass tends now to undergo structural changes. Addition of bismuth into the glass composition interrupts the glass network and thus reduces the energy barrier for atomic rearrangements, which subsequently increases the glass's susceptibility to transitions between metastable states.

The glass transition temperature and activation energy are the main chemical driving forces in any evaluation of kinetic materials since they connect with nucleation and growth processes. With data of the link between activation energy and these processes, insight is provided into the state of material stability and how suitable it may be as a product material. The Kissinger equation has been one of the most common proposals for the estimation of activation energy, which is expressed as:

$$\ln \frac{\alpha}{T_g^2} = -\frac{E_g}{RT_g} + \text{const.}$$

Here α is the heating rate; R, the universal gas constant; T_g , the glass transition temperature, and E_g , the activation energy associated with thermal relaxation. This equation is used for the calculation of the activation energy during crystallization since the change in T_g with varying heating rates is concomitant to a similar shift with an observed peak crystallization.

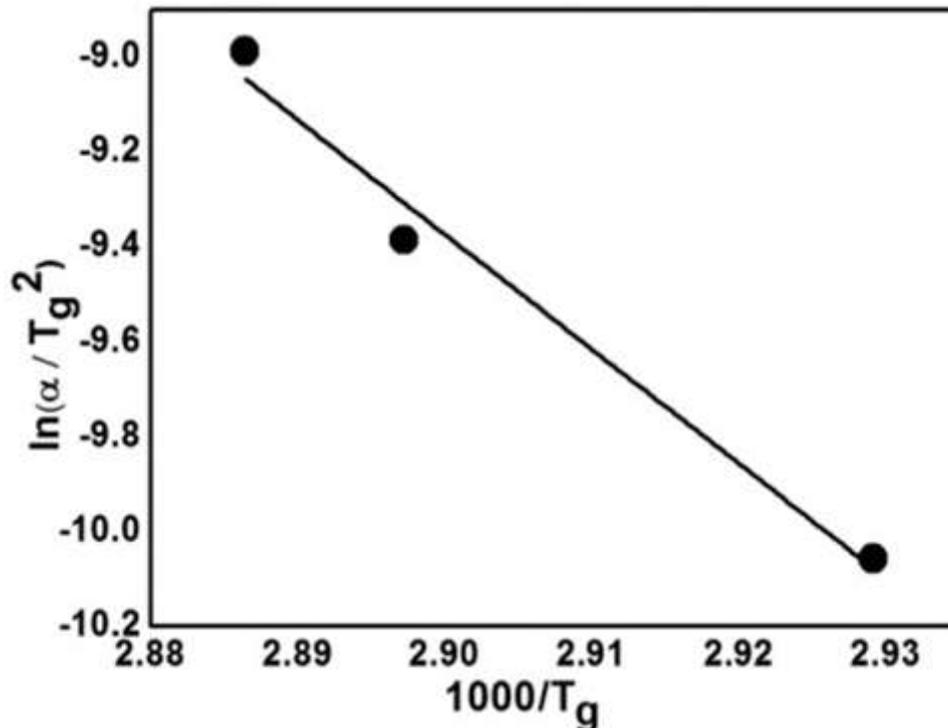


Fig. 3: $\ln(\alpha/T_g^2)$ versus $1000/T_g$ plot

The activation energy can be determined by plotting $\ln(\alpha/T_g^2)$ versus $1/T_g$. This plot will produce a straight line fit, and the apparent activation energy for glass transition can be found by calculating the slope of the line. Based on the results gathered from this experiment, the activation energy determined for crystallization is around (200.53 ± 3.2) kJ/mol, as illustrated in Figure 3. This is the energy that the material will require to shift from an amorphous to a crystalline state during heating.

Besides, the determination of crystallization activation energy can be made by applying the Augis-Bennett method [25]. In that method, E_c represents the effective activation energy for the crystallization process, k_0 is the frequency factor, which is a constant of the crystallization rate, and the equation which is needed to apply in this method as given below:

$$\ln \left[\frac{\alpha}{T_p - T_o} \right] = -\frac{E_c}{RT_p} + \ln(k_o)$$

This method gives an idea of what sort of energy barriers have to be crossed for the crystallization of a given material to take place. The slope of the plot in Figure 4 gives an effective crystallization activation energy. According to Augis and Bennett, the heating rate and degree of fitting of the differential thermal analysis (DTA) or differential scanning calorimetry (DSC) curves have an impact on the crystallization onset temperature (T_o). They recommend a T_o value that is consistent and lower than the minimum observed temperature ($T_o(\text{min})$) of the highest heating rate; in this way, the temperature can be normalized against the different

heating rates. This leads to the activation energy for crystallization being dependent on the T_0 value taken. The shift of the T_0 will give variation to the activation energy, which in turn depends upon the internal physical processes involved during crystallization since it can be changed by altering the T_0 . This indicates somehow a more reliable estimation of the energy required for crystallization varying with conditions.

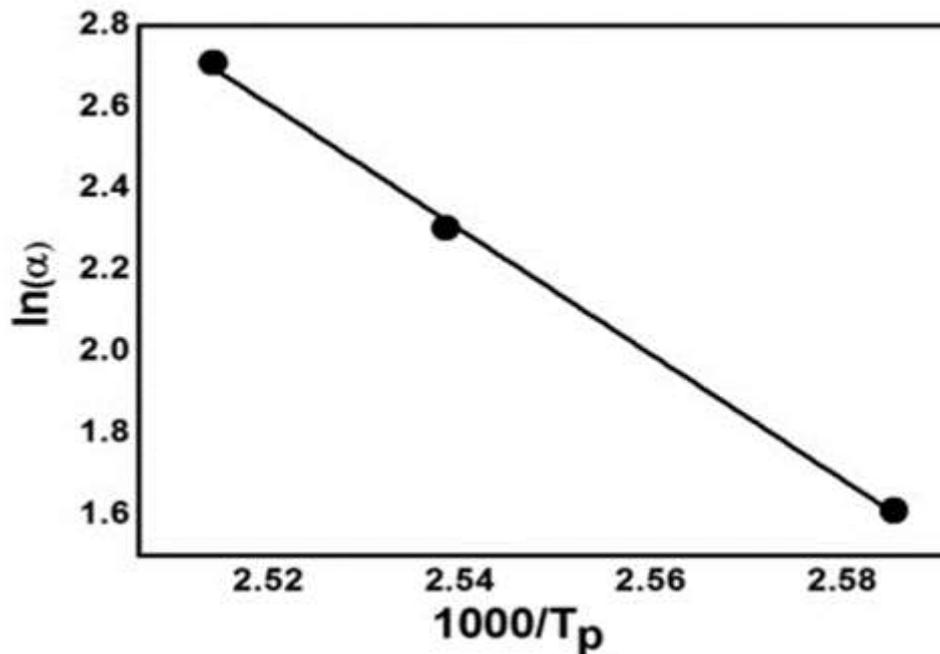


Fig. 4: $\ln(\alpha)$ versus $1000/T_p$ plot

Table 1: Parameters of the studied glass for heating rates, where T_m is the melting temperature, T_{rg} is the reduced glass transition temperature, and K_{gl} is Hruby's parameter. Hruby's parameter is used to evaluate the thermal stability and ease of glass formation of the material.

Heating Rate	T_g (K)	T_p (K)	T_m (K)	T_{rg}	K_{gl}	F_i
5	341.38±2	386.98±2	523.56±2	0.65±0.1	0.33±0.1	44.5±2
10	345.17±3	394.15±3	524.26±3	0.67±0.1	0.38±0.1	30.7±3
15	346.44±4	397.97±4	525.49±4	0.67±0.1	0.40±0.1	26.1±4

3.1 Glass Forming Ability:

Thermal stability and the ability to form glass are critical properties that determine what applications the glass in this study can be used in. The ability of a material to form a glass is determined by calculating the reduced glass transition temperature. This gives one a feel of how easy it is for a material to form a stable glass structure. This value of reduced glass transition temperature is given by the following relation:

$$T_{rg} = T_g/T_m$$

Based on this composition, the average reduced glass transition temperature (T_{rg}) is 0.67; this indicates that such chalcogenide glass has a very strong ability to form glass, suggesting that it is in conditions that favor the formation of stable glass.

The Hruby's parameter, in addition to the reduced glass transition temperature, can be used to assess thermal stability and ease of glass formation. Hruby's parameter combines in a single

parameter the contributions of glass transition and crystallization temperatures, thus providing a kind of measure of the thermal stability of the material.

Hruby's parameter is expressed by the following formula [26]:

$$K_{gl} = \frac{(T_p - T_g)}{(T_m - T_p)}$$

This represents how the material behaves during thermal processes, which helps assess the potential applications of the material, including using it in phase-change memory devices or optical recording materials.

The significance of the difference between the crystallization peak temperature (T_p) and the glass transition temperature (T_g) is tantamount to controlling the nucleation process. A larger temperature difference between these two means that it slows the nucleation process; thus, the material takes longer to start the process of crystal formation. However, the difference between the melting temperature (T_m) and the crystallization peak temperature (T_p) also affects the whole process of crystallization: the smaller the temperature difference between T_m and T_p , the slower the growth of the crystals once nucleation has begun, basically limiting how fast the crystals can grow. The Hruby's parameter serves to relate both the effects of nucleation and crystal growth into one value, thus demonstrating the over-thermal stability of the material within the phase transition. This parameter is crucial for the understanding of material behavior during the transformation from the amorphous to crystalline state. The Hruby's parameter (K_{gl}) ranges between 0.33 and 0.40 for the compositions under study, as shown in the table. Accordingly, this value implies a moderate crystallization and glass formation behavior of this particular polymers' system with the balance between nucleation and crystal growth, indicative of the potential for stable phase transition occurring during processing.

3.2 Fragility Index:

Evaluation of the thermal stability of chalcogenide glasses is necessary for assessing their qualification for use as recording materials. It is the process where these glasses undergo a transition from an amorphous-to-crystal phase, a process that will be vital for the development of advanced-phase change materials for recording technologies. Their thermal stability and glass-forming ability are critical in applications involving memory storage and switching devices in which temperature-dependent stability and responsiveness are paramount.

The fragility index is one of the important parameters used to reflect the glass-forming ability (GFA) of these materials. The fragility index reflects how the relaxation time for the material decreases with temperature increase near the glass transition temperature (T_g); thus, this gives an idea of how easily the material would form a stable glass, which in turn gives an idea about the glass-forming ability. Mathematically, the fragility index is given by [27]:

$$F_i = \frac{E_g}{RT_g} \ln(\alpha)$$

Generally, the fragility index (F_i) decreases with increasing heating rates because of the increase in glass transition temperature (T_g), whereas as it approaches T_g , whilst cooling, the fragility decreases. The observation of decreasing F_i with increasing heating rate indicates that the substances originate from a competent liquid that favors glass formation, as it can readily attain stable glass structure. This trend is consistent with structural relaxation dynamics and heating rates, whereby rapid heating favors the stability of glass formation. Consequently,

materials with lower fragility indices are to be preferred among glass formers since they would show more toughness to changes in temperature throughout the glass transition.

4. Conclusion:

The glass transition, the change from amorphous to crystalline phases, and the alloys' capacity to create glass are the main topics of this study's investigation of significant chalcogenide alloys. Important thermal characteristics were meticulously computed and examined, including the fragility index, Hruby's parameter, reduced glass transition temperature, activation energy for glass transition, and activation energy for crystallization. The activation energies for the crystallization and glass transition processes were determined using a variety of empirical techniques. It was determined that the glass transition activation energy was (203.45 ± 4.12) kJ/mol, and the crystallization activation energy was approximately (122.93 ± 2.95) kJ/mol. The alloy originates from highly effective glass-forming liquid, according to the reduced glass transition temperature (T_{rg}) and fragility index (F_i), indicating good potential for stable glass formation. Thermal stability is a crucial factor for the use of chalcogenide glasses in recording materials, as these materials undergo a significant phase transition from an amorphous to a crystalline state. This transformation is fundamental for developing advanced chalcogenide glasses used in phase-change recording technologies. Moreover, both thermal stability and the ability to form glass are critical properties for these materials' application in memory and switching technologies, where reliability and performance are key.

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