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On the vacancy nature of the high-temperature background of internal friction in solids

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High-temperature internal friction in an amorphous CuTi alloy is investigated. Exponential regions with different activation energies are observed on the dependence of internal friction on temperature on both sides of the glass transition temperature. An exponential increase in the background of internal friction with temperature in both sites is associated with the migration of vacancy-like defects in the amorphous structure under the influence of mechanical stresses, while frozen defects of constant concentration migrate to the glass transition temperature. After the transition to a state of thermodynamic equilibrium, the concentration the number of migrating defects increases exponentially. Based on the experimental results of measuring the high-temperature background, estimates of the activation energy of migration and the formation of vacancies of similar defects in the amorphous structure of the alloy under study are made.

Keywords: Internal friction, relaxation time, high temperature background, amorphous alloy.

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Introduction

Many experimental results of the study of temperature dependence of internal friction (IF), IF increases almost linearly in crystals up to the temperatures of $(0.7-0.8)T_m$ (T_m is melting temperature). The exponential dependence $Q^{-1}(T)$, called the high-temperature background of internal friction, is often observed above these temperatures [1,2]. The high-temperature background of IF is generally rather well described by the following equation

$$Q_I^{-1} = A\exp(-H/RT), \tag{1}$$

where T — temperature, [K]; R — universal gas constant; A and H — constants [1,3].

At present, much experimental material has been accumulated and various mechanisms have been suggested to explain the increasing IF of crystalline solid bodies in this temperature region [1–5]. Activation heat H, calculated using expression (1), was often several times lower than the activation energy for self-diffusion of point defects, due to which complex decay mechanisms were suggested to explain the process.

The high-temperature background of IF in crystals was studied in many theoretical papers (e.g., [6–9]). A theoretical model of IF background, related to vacancy migration, was published by Escayg in 1962 [6], but the vacancy mechanism of IF background in crystals was not confirmed experimentally for a long time since this mechanism acts in a narrow range at temperatures close to the melting temperature of the material under study, when measurements are hindered [10]. The authors of [8] made a conclusion that the IF background is formed not by one vacancy mechanism but by a whole set of subsystems: vacancies, dislocations, crystallites, interstitial impurity atoms and substitutional impurity atoms; and the IF background can be represented by several straight-line segments on the plot of $\ln Q^{-1}$ vs. $1/T$, which have significantly different slope angles. However, a sequence of change of mechanisms accountable for IF increase has not been yet determined either theoretically or experimentally. The experimental results of the study of high-temperature IF in various solid bodies with an amorphous structure have show that an exponential increase of IF long before the glass-transition temperature (the region of the so-called α-relaxation) is observed in inorganic glasses with covalent bonds [11], in metallic glasses [12–14], in bulk metallic glasses [15–17], chalcogenide glasses [18,19], polymers [20–22], which, the same as in crystalline solid bodies, can be referred to the high-temperature background of IF in this material class, since the amorphous state of such systems at higher temperatures changes either to the crystalline state or to the state of supercooled liquid. The exponential increase of IF in solid bodies with an amorphous structure is explained by scientists by various decay mechanisms, but consensus on this problem has yet to be reached. All classes of solid bodies with an amorphous structure (amorphous organic polymers, inorganic glasses, metal amorphous alloys, chalcogenides etc.) are characterized by an excessive free volume (low-density regions characterized by a certain size distribution) [23–24], due to which the exponential increase of internal friction was associated with migration of vacancy-like defects of an amorphous structure [10,14].

New experimental results have been obtained in this paper by the example of study of the IF background in CuTi metallic glass; the results confirm the vacancy mechanism of the IF high-temperature background, which is associated with migration of vacancy-like defects of an amorphous structure (holes or excessive space) in a system with a non-ordered atom arrangement.

1. Experimental procedure

Specimens of $Cu_{66}Ti_{34}$ amorphous alloy were obtained by tempering from the liquid state using rotating rolls [25]. For this purpose, the alloy of the given composition (several tens of grams) was melted in a quartz glass ampoule in vacuum, and the drop, pressed out by gaseous helium, came between two steel rolls pressed to each other by springs and rotating at the speed of ~ 8000 rpm (the roll diameter was 50 mm). As it passed between the rolls, the drop was flattened to a film and hardened. Tempering rate dT/dt was $\sim 10^6$ K/s. Thin homogeneous amorphous films approximately $30\,\mu$m thick formed. Structure amorphism was checked by the X-ray method in filtered CoK_α-radiation using the „Dron-2“diffraction meter. The glass transition temperature, determined by differential thermal analysis, was 633 K.

Internal friction of cantilevered specimens $2-3$ mm wide was measured in the frequency interval of 10^2-10^4 Hz with the maximum relative deformation of 10^{-5} in the temperature region of $300-900$ K by the method of freely decaying flexural vibrations as per [26]. The internal friction measurement error did not exceed 3%.

2. Experimental results and discussion

Fig. 1 shows the temperature plot of IF for the $Cu_{66}Ti_{34}$ amorphous metal alloy, where the high-temperature background of IF is overlaid by an asymmetric maximum at $T = 690$ K, the height of which decreases as the mechanical vibration frequency increases under the law $Q^{-1} \sim 1/f^{1/2}$ (the insert in Fig. 1), where f — specimen vibration frequency. Intensive increase of the amorphous alloy IF begins at $T \geq 520$ K, while in the temperature interval of $620-650$ K curves *1* and *2* have a mild bend which becomes a maximum at higher frequencies (curve *3*). A similar maximum was also observed for other amorphous alloys [27–29]. After heating to 780 K, i.e. after the amorphous structure crystallizes, the temperature plot of IT has no peculiarities (curve *4*). Since the nature of the IF maximum at the temperatures of $620-650$ K is outside the scope of the present work, but should be discussed separately, let us give a more detailed analysis of the IF background in solid bodies with an amorphous structure.

Analysis of the data on high-temperature IF of the $Cu_{66}Ti_{34}$ amorphous metal alloy, presented in Fig. 1, and other amorphous alloys [29], as well as bulk metallic glasses [30] shows that the IF background reaches large values at low frequencies and decreases as the specimen vibration frequency increases, while decay of elastic vibrations in the high-temperature background range for kilohertz range frequencies range decreases under the law $Q^{-1} \sim 1/f^{1/2}$ (the insert in Fig. 1).

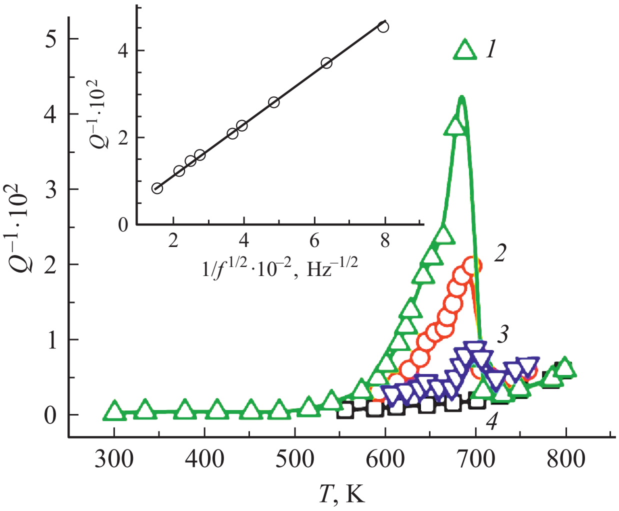

Figure 1. Temperature plots of IF of amorphous (*1–3*) and crystalline (*4*) $Cu_{66}Ti_{34}$ alloys at the mechanical vibration frequency of: 151 (*1*), 2175 (*2*), 2786 (*3*), 200 Hz (*4*). The insert shows the plot of height of the IF maximum vs. $1/f^{-1/2}$.

Another characteristic feature of IF spectra for amorphous metal alloys is the fact that the high-temperature part of the background can be represented as two straight-line segments on the plot of $\ln Q^{-1}$ vs. $1/T$, which have significantly different slope angles (Fig. 2). Let us consider this regularity in more detail. When studying the inner body in solid bodies with an amorphous structure, IF in the high-temperature background region (or in the α-relaxation region of inorganic, chalcogenide glasses and amorphous polymers) is analyzed using temperature-frequency plots in $\ln \omega = f(1/T)$ coordinates; the plots are curvilinear, which means that these processes depend on temperature [31,32]. Under such consideration, the main condition of the maximum $\omega\tau = 1$ is replaced by the Deborah correlation [33]:

$$\omega\tau = c, \qquad (2)$$

where c — non-dimensional coefficient, has certain ambiguity [32]. Certain provisions of the relaxation spectrometry theory associate the value of this coefficient with relaxation process scale: the value of this coefficient for large-scale processes can be equal to 10, while for small-scale processes it can be equal to one [20]. With such an approach, the activation energy of the glass transition process consists of two components due to the dual nature of glass transition:

$$U_\alpha = U_1 + U_2(T), \qquad (3)$$

where U_1 and $U_2(T)$ — activation energies for the first and second stages of mechanical relaxation respectively. The first component (U_1) is associated with the potential barrier of transition to the activated state. The second component

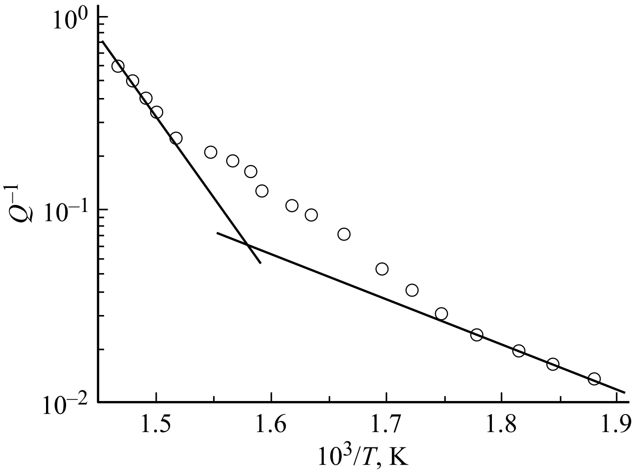

Figure 2. Temperature plot of IF of $Cu_{66}Ti_{34}$ amorphous alloy in $\ln Q^{-1}$ coordinates vs. $1/T$ at the mechanical vibration frequency of 151 Hz. The solid curves show the sums of two exponential dependences with the activation energies of 0.47 and 1.4 eV.

$(U_2(T))$ is associated with the energy required for formation of the free volume and for transition of a kinetic unit to the neighboring position [34].

We think that this approach is not entirely correct, because the observed maximum usually does not meet the condition $\omega\tau = 1$, but is caused by superposition of crystallization-related losses (in metallic glasses) onto the high-temperature background of IF, or by transition of glass into the supercooled liquid state (in inorganic, chalcogenide glasses and polymers). This manifests itself in the presence of an asymmetric IF peak both for metallic glasses (Fig. 1) and for other amorphous solid bodies — polymers [35] and inorganic glasses [36].

In terms of the phenomenological theory of inelasticity, any relaxation process that manifests itself as a peak of dissipation losses on the IF spectrum can be described by the following equation of a standard linear body [1]:

$$\mathrm{tg}\,\varphi = Q^{-1} = \Delta \frac{\omega\tau}{1+\omega^2\tau^2}, \tag{4}$$

where $\Delta = (M_n - M_r)/M_r$ — relaxation degree or modulus defect, ω — circular frequency, M_n and M_r — non-relaxed and relaxed elasticity moduli respectively, τ — relaxation time changing under the Arrhenius law

$$\tau = \tau_0 \exp(H/RT), \tag{5}$$

where H — activation heat of a certain relaxation process, τ_0 — time parameter weakly dependent on temperature, T — temperature, [K], R — universal gas constant.

It follows from correlation (4) that, due to a change of relaxation time τ with temperature, the maximum value of internal friction is reached when the condition $\omega\tau = 1$ is met. Since the maximum condition is not achieved in the amorphous alloy under study, the relaxation process activation energy can be found according to the low-temperature slope of IF in the α-relaxation region. Thus, the following can be written from formula (5) and expression $Q^{-1}_{\max} = \frac{1}{2}\Delta$

$$Q^{-1} = \frac{2Q^{-1}_{\max}\omega\tau}{1+\omega^2\tau^2}. \tag{6}$$

Then it results from formulas (5) and (6) for the low-temperature leg of the peak $(\omega\tau \gg 1)$ that

$$Q^{-1} \sim \exp\left(-\frac{H}{RT}\right). \tag{7}$$

As seen in Fig. 2, the plot of $\ln Q^{-1}$ vs. $1/T$ has two straight-line segments with the activation energy of high-temperature background $U_{b1} = 0.47 \pm 0.02$ eV and $U_{b2} = 1.4 \pm 0.2$ eV for the $Cu_{66}Ti_{34}$ amorphous alloy. Two straight-line segments on the plot of $\ln Q^{-1}$ vs. $1/T$ were also observed for other amorphous solid bodies: bulk metallic glasses $Zr_{52.5}Ti_5Cu_{17.9}Ni_{14.6}Al_{10}$ with the activation energies of high-temperature background equal to $U_{b1} = 0.70 \pm 0.02$ eV and $U_{b2} = 2.1 \pm 0.2$ eV on the low-temperature and high-temperature segments respectively [30], in chalcogenide semiconductors of the Ge–As–Se system in the region of α-relaxation process [18], for glass fiber plastic in the T-107 [21] host and other systems. Such an analysis was not carried out for many polymers and inorganic glasses, but peak asymmetry is undoubtedly observed in the region of the α-relaxation process in such systems [37–40]. As seen in Fig. 2, the activation energy of the relaxation process in the region of high-temperature background also consists of two components, but, as distinct from equation (3), both components of activation energy in the given approach do not depend on temperature.

The fact that IF in the region of high-temperature background is proportional to $f^{-1/2}$ may mean a diffusion nature of decay of mechanical vibrations in the considered temperature range. Various variants of the free volume theory [41,42] became widespread in the physics of liquids, amorphous polymers and metallic glasses; these variants are based, in fact, on the main provisions of the Fraenkel hole theory [43]. Ya.I. Fraenkel considered the free liquid volume as an excessive volume compared to the volume of a crystalline solid body. According to him, the fraction of free liquid volume is about 3% [43].

The traditional free volume of amorphous substances (or the void space between atoms) is a part of atomic structural complexes. It is called a structurally conditioned, geometrical free volume distributed in the form of fluctuations which can be considered as vacancy-like defects of the amorphous structure [44].

At the glass-transition temperature, fluctuation atomic rearrangements generate a local fluctuation volume which exceeds the atom delocalization volume; this is a condition for transition of a kinetic unit to a new position with formation of new structural objects. The fluctuation space of an amorphous substance is conditioned by thermal displacements of atoms, while its volumetric fraction is weakly dependent on the nature of amorphous substances and is $f_g \approx 0.02{-}0.03$ [44,45]. The fluctuation displacement of a kinetic unit is considered as the atom delocalization process [46]. In silicate glasses it is the displacement of bridge oxygen in the Si−O−Si oxygen, in metallic glasses — the formation of vacancy-like defects, and in polymeric hydrocarbons — the limit displacement of a coupling link in the macromolecule main chain [46].

As temperature increases, mobility of such units increases, and superimposition of mechanical stresses causes their directed migration from tensile regions to compression regions. From the physical viewpoint, it means that IF is proportional to the concentration of defects frozen during tempering n and their average migration length L, i.e.

$$Q^{-1} \sim nL \sim n\sqrt{Dt} \sim \frac{n}{\omega^{1/2}} \exp\left(-\frac{E_m}{2kT}\right), \qquad (8)$$

where D — diffusion coefficient, t — time, k — Boltzmann's constant, E_m — migration activation energy.

Assuming that exponential increase of IF in the case of amorphous metal alloys is also due to migration of vacancy-like defects, formula (8) can be used to estimate the energy characteristics of vacancy-like defects of the amorphous structure. We will consider that the concentration of amorphous structure defects n is determined, according to [13], as

$$n = \begin{cases} n_0, & T < T_g \\ A\exp(-E_v kT), & T > T_g \end{cases}. \qquad (9)$$

Here n_0 and A are constants, E_v is the energy of formation of a vacancy-like defect in the amorphous structure at temperatures above the glass-transition temperature, T_g is the glass-transition temperature. When applying the above-mentioned approach to amorphous metallic alloys, the migration energy of vacancy-like defects must be twice as high as the activation energy of the high-temperature background on a low-temperature area, i.e. $E_m = 0.94 \pm 0.04$ eV, while the formation energy, determined as the difference $E_v = U_{b2} - U_{b1} = 0.93 \pm 0.04$ eV for the $Cu_{66}Ti_{34}$ alloy. When applying the given approach to bulk metallic glass $Zr_{52.5}Ti_5Cu_{17.9}Ni_{14.6}Al_{10}$, the migration activation energy is $E_m = 1.40 \pm 0.04$ eV, and the formation energy is $E_v = 1.40 \pm 0.04$ eV [30], i.e. the values of defect formation energy match (within the error) their migration energies. Based on the Fraenkel liquid theory [43] (assuming that amorphous alloys are frozen liquids), where the defects are „holes"formed by opening of the chemical bond between neighboring atoms, values of E_m and E_v can be assumed to be equal.

Thus, despite the fact that the authors of experimental papers in various amorphous structures differently interpret the reasons of an exponential increase in IF, the high-temperature of IF for all solid bodies with an amorphous structure can be represented as two straight-line segments in $\ln Q^{-1}$ coordinates vs. $1/T$ with an intersection point near the glass-transition temperature and can be associated with migration of vacancy-like defects. Thereat, if a composite specimen is used (e.g., the specimen under study is mated with a material that does not absorb vibrations in the given temperature region [47]), then the glass-transition temperature can be determined according to the temperature at the intersection of two straight-line segments of the high-temperature background in $\ln Q^{-1}$ coordinates vs. $1/T$. For example, paper [21] deals with measurements of temperature dependence of internal friction in glass carbon fiber-reinforced plastics in a polymer host T-107 in the region of the main relaxation process, i.e. α-relaxation [48]. The glass fabric having a higher glass-transition temperature made it possible to reduce the overall background of glass fiber plastic IF and to measure the IF background in the polymer host before and after the polymer glass-transition temperature. The study results have shown that IF in the region of the host material glass-transition temperature does not depend on filler concentration but is determined by the polymer IF background, the results of study of which made it possible to estimate the glass-transition temperature of the polymer.

The above-mentioned studies have been carried out on relatively large specimens, when vacancy-like defects do not have enough time to diffuse from the stretched areas of the specimen to compressed ones within the vibration period. When IF in nanostructured specimens is studied, a case is possible when $Q^{-1} \sim \omega^{-1}$ in the region of the high-temperature background. In particular, the studies of the high-temperature background of IF in nanocomposites $(Co_{45}Fe_{45}Zr_{10})_x(Al_2O_3)_{100-x}$ have shown that the activation energy of the high-temperature background in the $Co_{45}Fe_{45}Zr_{10}$ amorphous metal alloy in this case is equal to the migration activation energy $H_b = E_m = 1.4 \pm 0.1$ eV [49].

Thus, the high-temperature background of IF in amorphous solid bodies can be associated both with the migration of vacancy-like defects of the amorphous structure with a frozen (constant) concentration up to the glass-transition temperature, and with the concentration changing under the Arrhenius law, after the glass-transition temperature.

Let us summarize the experimental data on the background of internal friction using a thermodynamic scheme (Fig. 3), which shows a plot of free energy F vs. temperature T and volume V, acting as a structural parameter of the condensed state of matter [50]. The plot of free energy vs. the structural parameter V has the form of a potential hole both for the solid crystalline state F_S and for the liquid stage F_L. This pattern of the plot of free energy vs. the structural parameter is due to the fact that the system has the energy minimum in the equilibrium state. For instance, the elastic part of free energy of a solid body U can be represented as a quadratic function of change in

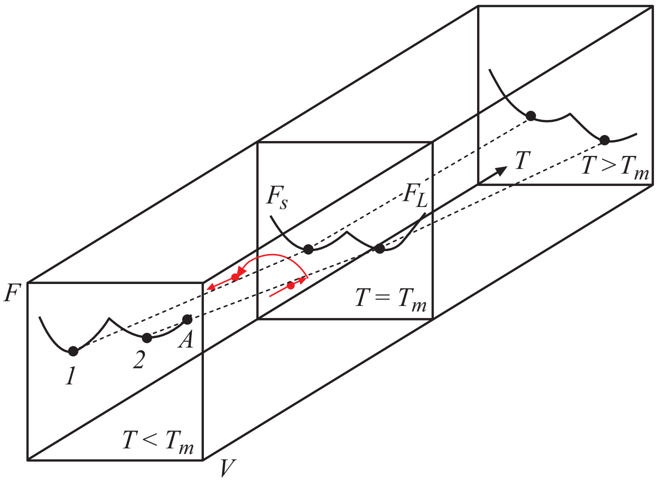

Figure 3. Change of free energy of the condensed state versus temperature and volume.

volume V [43]:

$$U(V) - U(V_o) = 1/2K(V - V_o)^2/V_o. \qquad (10)$$

Here V_o is the equilibrium value of volume, K is the modulus of all-around compression.

The given diagram shows a change in free energy of the condensed state vs. temperature and volume in the form of two „troughs": for the crystalline state (with a smaller volume value) and the liquid state (with a greater volume value). The change of free energy vs. temperature along the „trough" bottom characterizes the system's thermodynamic equilibrium state (or the metastable equilibrium state). Deviations from the thermodynamic (or metastable) equilibrium state characterize non-equilibrium (labile) states.

Quenching from the liquid state for systems prone to glass transition (inorganic glasses, chalcogenides and other systems) can ensure the following: at temperatures below the melting temperature, the system remains in the metastable state typical for a supercooled liquid (point *2* at $T < T_m$ in Fig. 3). The structure freezes at temperatures below the glass-transition temperature (T_g) and the system is already characterized by point A (Fig. 3 at $T < T_m$). This state is non-equilibrium not only in relation to the stable crystalline state, but also the metastable equilibrium typical for a supercooled liquid. The process of relaxation of an amorphous structure from the non-equilibrium state (point A at $T < T_m$ in Fig. 3) to the state of metastable equilibrium (point *2* at $T < T_m$ in Fig. 3) is called structural relaxation, as distinct from the crystallization process which is related to a transition to the state with the absolute minimum of free energy (point *1* in Fig. 3). According to the outlined representation, a change of free energy vs. temperature and volume is described by the correlation

$$dF = \left(\frac{\partial F}{\partial T}\right)_V \cdot dT + \left(\frac{\partial F}{\partial V}\right)_T \cdot dV, \qquad (11)$$

from here

$$\frac{dF}{dT} = \left(\frac{\partial F}{\partial T}\right)_V + \left(\frac{\partial F}{\partial V}\right)_T \cdot \frac{dV}{dT}. \qquad (12)$$

It follows from this equation that the temperature coefficient of free energy F is generally determined by the temperature coefficient $(dF/dT)_V$ and a certain quantity equal to the temperature coefficient of volume change dV/dT, multiplied by the structure-dependent coefficient $(dF/dV)_T$. Consequently, a change in physical properties of amorphous solid bodies, conditioned by a structure change in the structural relaxation process, can be unequivocally associated with various defects present in the amorphous structure. A deviation of the alloy structure from metastable equilibrium upon cooling from the liquid state is due to freezing of a certain concentration of defective configurations. When considered in this way, glass-transition temperature is a temperature at which the concentration of frozen defects becomes equal to the equilibrium temperature, i.e. glass-transition temperature is identical to temperature of vacancy freezing in a crystal upon cooling from pre-melting temperatures.

Heating of a frozen amorphous structure of metallic glasses to the glass-transition temperature (Fig. 1) causes migration of frozen vacancy-like defects of a constant concentration which starts changing under the Arrhenius law after the glass-transition temperature. The plot of the high-temperature background of IF for amorphous solid bodies, observed in our experiment, has the form of two straight-line segments in $\ln Q^{-1}$ coordinates vs. $1/T$ with an intersection point near the glass-transition temperature (Fig. 2) in the amorphous state. Thereat, the activation energy for migration of vacancy-like defects can be estimated according to the low-temperature area of the high-temperature background of IF, and their formation energy can be estimated according to the high-temperature area. Upon crystallization of the amorphous structure, the system goes from the local minimum of free energy into a crystalline state characterized by the absolute minimum of free energy, without achieving it (the arrow in Fig. 3). When the crystalline structure is cooled, there are no defects typical for the amorphous state, and the plot of IF vs. temperature shows no anomalies (curve *4* in Fig. 1).

We would like to conclude by saying that the nature of glass transition of the liquid is being discussed again in recent years: whether this transition is purely kinetic or it is a second-order phase transition [24,51]. The above-mentioned results answer the question posed in the review paper [51]: „is glass transition a purely kinetic phenomenon or is it caused by a transition to the state of „perfect glass". The experimental results show that the glass transition process is a purely kinetic phenomenon and is not caused by a phase transition to the state of „perfect" glass. The state of „perfect" glass (similarly to a crystal, this is defect-free glass) must be presented in the diagram shown in Fig. 3 by point *2* in the local minimum at the absolute zero, while the state of a perfect crystal must be presented by point *1* in the absolute minimum at 0 K.

Conclusion

The experimental results of study of IF in the $Cu_{66}Ti_{34}$ amorphous alloy show that $Q^{-1} \sim f^{-1/2}$ in the high-temperature background region, which is due to migration of vacancy-like defects of the amorphous structure under variable-sign mechanical stresses. It has been found that the high-temperature part of the background consists of two straight-line segments which have significantly different slope angles on the plot of $\ln Q^{-1}$ vs. $1/T$ with an intersection point near the glass-transition temperature.

Thereat, the low-temperature area of the IF background is associated with migration of non-equilibrium (frozen) structural defects, when the IF background activation energy comprises only the defect migration activation energy. The high-temperature area of the IF background is due to migration of equilibrium point defects of the real structure, while the effective energy of background activation is determined by two components: migration activation energy and defect formation energy. The estimates of these components for the $Cu_{66}Ti_{34}$ alloy have showed that the migration activation energy is $E_m = 0.94 \pm 0.04$ eV, while the formation energy is $E_v = 0.93 \pm 0.04$ eV.

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Conflict of interest

The authors declare that they have no conflict of interest.

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