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Research Article

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INVESTIGATION OF SOME PHYSICAL AND STRUCTURAL PROPERTIES OF MELTS BY ULTRASOUNDS

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Abstract. *A modern effective way of influencing molten metal is ultrasonic treatment - a type of dynamic influence on liquid and crystallizing metal. At certain parameters of the intensity of ultrasonic treatment, which causes acoustic cavitation of the liquid metal, the refining process actively occurs. Moreover, powerful ultrasound allows, during the modification process, to introduce various composite elements and refractory alloys into the metal, acting directly on the crystal lattice. The calculations carried out on the properties of elastic waves make it possible to identify such features in the behavior of the sound absorption coefficient polytherms, which with sufficient certainty indicate the absence or presence of structural changes when the melt is heated, and establish the possibility of implementing various mechanisms of structural changes in the same melt in different temperature ranges. The conducted studies of elastic wave energy absorption as well as ultrasound velocity allow us to identify such features in the behavior of sound absorption coefficient polyterms that indicate the absence or presence of structural changes during melt heating. The comparative analyses make it possible to conclude that there is not a single mechanism of structural changes The possibility of implementing various mechanisms of structural changes in the same melt in different temperature ranges is shown.*

Keywords: viscous, liquid metals, ultrasound propagation speed, structural changes, melt.

1. Introduction

Ultrasonic melt processing attracts considerable interest from both academic and industrial communities as a promising route improving melt quality. The significance of this problem is predetermined by the matter liquid state problem. The application of power ultrasound during liquid to solid transformation is believed to be an effective way to improve the solidification microstructures and mechanical properties [1]. In fact, the entire arsenal of modern experimental and theoretical physics is connected to the research of the physicochemical behavior of melts [2]. Acoustic methods are the most promising among experimental methods for research of the matter liquid state. They are simple, reliable, highly sensitive to changes of matter structure and the interatomic interaction. The results of this research make it possible to predict the melts elastic properties of simple substances and extend it to complex substances.

At present, the electrophysical, thermophysical, thermodynamic and viscous properties of liquid semimetals and semiconductors based on the electronics industry have been widely studied. However, the ongoing research in the field of studying these properties is not sufficient to solve the problem of the liquid semimetals and semiconductor's structure. It is also impossible to obtain an unambiguous result by only structural research. In this aspect, it is known that «modern acoustic research methods are a powerful tool for obtaining information about the structure of melts and semiconductors» [3]. Melts and semiconductors were not widely also studied, since the high-temperature acoustic experiments technology with aggressive melts of semimetals and semiconductors complicated the research process [1–5]. Our research includes: development liquid semimetals and semiconductor's structure model; experimental and theoretical studies of the propagation speed and absorption coefficient of ultrasound temperature dependences in liquid semimetals and semiconductors; regularities generalization of liquid metals, semimetals and semiconductors structural properties.

2. Theory of the Method

The conducted studies of elastic wave energy absorption as well as ultrasound velocity allow us to indicate the absence or presence of structural changes during melt heating.

This work demonstrated the practical importance of acoustic measurements in metal melts. We performed calculations of ultrasonic treatment of molten systems, investigated the physical and chemical effects of ultrasound on the structural properties of liquid molten systems. We found that the chemical effect is an irreversible and permanent change in the atomic weight and atomic-weight distribution due to ultrasound. Calculations showed that with an increase in the ultrasound intensity, the atomic weight of molten metals decreases, while the orientation of atoms along the flow direction decreases. Ultrasonic vibration increases the motion of atoms, a change in the structure of atoms occurs, which makes them more disordered. Ultrasonic vibration affects the relaxation process of molten metals, resulting in a weakening of the elastic effect.

2.1 The practical value of direct acoustic measurements in liquid metals

A hypothesis about the micro-inhomogeneous structure of liquids arose in connection with Stuart's research using X-rays in the 1920. Micro-inhomogeneity extends to any melts from alkali metals to semiconductors. But the hypothesis remained a hypothesis, since there were no interpreted experimental data. The experiments were performed with a solidification apparatus incorporated with ultrasonic generator. The propagation of elastic waves is associated with the fundamental properties of material media, including the mass of particles, their space, the bonds between particles of matter.

Let's consider the Stokes-Kirchhoff formula (1) [7], and transform (1) to (2), (3):

$$\beta = \frac{2\pi^2 f^2}{\rho \mathrm{v}_S^3}\left[\frac{4}{3}\eta + \frac{\chi}{C_P}(\gamma - 1)\right] \tag{1}$$

$$\frac{\beta \mathrm{v}_S}{f^2} = 2\pi^2 \alpha_S \left[\frac{4}{3}\rho\upsilon + \frac{\chi}{\left(\frac{dQ}{dT}\right)_P} m(\gamma - 1)\right]. \tag{2}$$

$$\frac{\beta \mathrm{v}_S}{f^2} = \sigma M_A. \tag{3}$$

$$\gamma = \frac{C_P}{C_V}, \qquad \sigma = 2\pi^2 \alpha_S \left[\frac{4}{3}\frac{\upsilon}{V_A} + \frac{\chi}{\left(\frac{dQ}{dT}\right)_P} N_A(\gamma - 1)\right].$$

χ - is the thermal conductivity coefficient, C_V is the heat capacity at constant volume, C_P - is the heat capacity at constant pressure, υ is the kinematic viscosity, $\frac{1}{\rho \mathrm{v}_S^2} = \alpha_S$ is the adiabatic compressibility, $C_P = \frac{dQ}{dT}\frac{1}{m}$ is the melt heat capacity, N_A is the Avogadro number, M_A is the atomic mass, V_A is the atomic volume,.

These indicators, namely, the inertial factor (mass of particles), the spatial factor (volume per particle), the stiffness factor between particles (compressibility), are sufficient fora general description of elastic waves absorption and propagation speed [6].

2.2 Algorithm of the method

The obtained equation (3) makes acoustic parameters monitoring in simple substances melts more accessible. Experimental measurements monitoring by value using reference data [8-12] is shown in Fig.1.

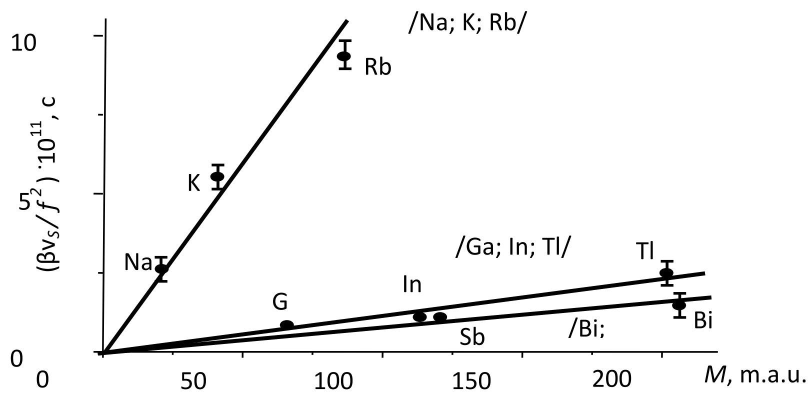

Fig.1. $\frac{\beta v_S}{f^2}$ – M simple substances dependence at crystallization temperatures

We have established the correlation dependence between measurement results $\frac{\beta v_S}{f^2}$ and the parameter values M_A, where σ is a constant value for each group. The bonding factors intragroup similarity not only observed in condensed bodies, but also diatomic molecules it. Highest value of the dissociation energy is observed for five electrons in the outer shell, the most rigid bonds exist in diatomic molecules [13].

In the Fig. 1 that the parameter $\frac{\beta v_S}{f^2}$ increases from *Na* to *Rb* in the alkali metal series is shown. This is confirmed by the fact that these groups of metals tend to loosen the structure with increasing atomic mass M_A. The $\frac{\beta v_S}{f^2}$ temperature dependence is quite complex. Immediately after melting, there is a decrease in the absorption coefficient of ultrasonic waves, which increases monotonically with increasing temperature. This includes the alkali metals and further all other simple metals that are densely packed in the solid state. Such behavior is natural for all other simple metals and for two-component liquid metal solutions. Metallic melts in which the polytherms of absorption of ultrasonic waves do not increase with increasing temperature belong to the second class, containing semimetals and semiconductors, in which significant changes in their structural properties occur during melting. For example, bismuth, antimony, tellurium are characterized by anomalous behavior of the polytherms of absorption and propagation velocities, therefore structural changes in them continue in a certain temperature range after melting. The experimental results are shown in Fig. 2 - 19 for *Ga, Bi, Sb, Ge, Se, Te, GaSb*, *InSb*, $Bi_{0,25}Sb_{0,75}$, $Bi_{0,5}Sb_{0,5}$, $Bi_{0,75}Sb_{0,25}$, *Bi-Sb*, $Sn_{0,30}Te_{0,70}$, $Sn_{0,5}Te_{0,5}$, $Sn_{0,70}Te_{0,30}$,, *Sn-Te* compounds. The solid lines are the result of the experimental data approximation by the equation

$$v_S(T) = v_{SL} - \beta(T - T_L),$$

$\beta = \frac{dv_S}{dT}$ is the ultrasound propagation speed temperature coefficient, T_L is melting point, v_{SL} is ultrasound propagation speed at T_L.

3. Practical approbation and results

Thus, the acoustic analysis in electron melts experimental measurements shows that the absorption polytherms and the propagation velocity behavior of ultrasonic waves depends on the semimetals and semiconductors acoustic properties. This was the reason for separating these melts into an electron melts separate class. A straight-line relationship between $\frac{\beta v_S}{f^2}$ and M_A inertial factor has been established.

Ultrasonic vibrations were introduced into the melt continuously during the entire time of exposure perpendicular to the surface, which corresponded to the longitudinal scheme of their introduction in relation to the beam direction and the predominant heat removal from the melt zone. The frequency and intensity of ultrasonic vibrations are selected depending on the specific melt. At an ultrasound intensity of more than $105\frac{W}{m^2}$, the kinetic energy of collapsing bubbles, concentrated in an extremely small volume, is transformed partly into a force impulse and partly into thermal energy. In liquid, when exposed to ultrasonic vibrations, electrokinetic phenomena also arise, caused by the directed movement of charged particles, which affect the diffusion processes and structural properties, in particular the orientation of atoms.

Experimental measurements of the velocity and absorption of ultrasound in liquid were carried out using ultrasonic Doppler velocimetry. To obtain sufficient Doppler signals, the problems of ultrasonic beam passage through a channel wall made of stainless steel, acoustic coupling between the transducer and the channel wall, and wetting of the inner wall surface with liquid metal were studied, respectively. Measuring the sound speed The DOP3000 allows to measure the sound speed in a liquid by measuring with precision the time that is taken by an ultrasonic burst to propagate over a define distance.

The DOP3000 Velocimeter

The propagation of elastic waves is associated with the fundamental properties of material media, including the mass of particles, their space, the bonds between particles of matter. These indicators, namely, the inertial factor (mass of particles), the spatial factor (volume per particle), the stiffness factor between particles (compressibility), are sufficient fora general description of elastic waves absorption and propagation speed [13]. First of all, this is the relationship of the selected factors with the absorption coefficient of ultrasonic waves. For the occurrence of such observations in the behavior of the melts elastic properties, one can use the system analysis methods based on the D.I. Mendeleev Periodic phenomenon.

Gallium has a low melting point, is rather easily supercooled and is a convenient object for studying the structural correspondence of liquids at temperatures above and below the melting point. Fig. 2 show the results of investigations of the absorption coefficient temperature dependence, ultrasound propagation velocity and β_S theoretical values in liquid gallium at 30, 50, 70 MHz frequencies. For comparison, the data on the ultrasound velocity in liquid gallium obtained in [14] are given. The results obtained correlate well with those of the authors [14-16] in Fig. 2.

The polyterm of the ultrasound absorption coefficient in liquid gallium at temperatures 370 K and 545 K has extrema. This indicates structural changes occurring in liquid gallium and noted in [14]. It should be noted that such behavior of the $\frac{\beta}{f^2}$ and v_S polyterms was not observed in metal melts. The totality of these experimental facts indicates the complexity of the structure of liquid gallium. The $\frac{\beta}{f^2}$ and v_S polyterms behavior for liquid gallium is explained by the two-structure liquid model proposed in [14]. The research results of ultrasound absorption and velocity temperature dependence as well as theoretical values obtained using the density functional, in bismuth melt from five different melts are presented in Fig. 3 in comparison with the authors data [17]. It can be seen that even in this case the results of $\frac{\beta}{f^2}$ and v_S measurements correlate well with the data of other authors. On the polyterm $\frac{\beta}{f^2}$ at temperature T=650K, the extremum of the ultrasonic absorption coefficient is observed. Fig. 4 shows the results of temperature dependence of ultrasound absorption and velocity in antimony melt of six different melts in comparison with the authors' data [18]. We used the functional in calculating β_S by $\rho_n = N_n \frac{\lambda^{\chi_n+1}}{\chi_n!} r^{\chi_n} e^{-\lambda r}$.

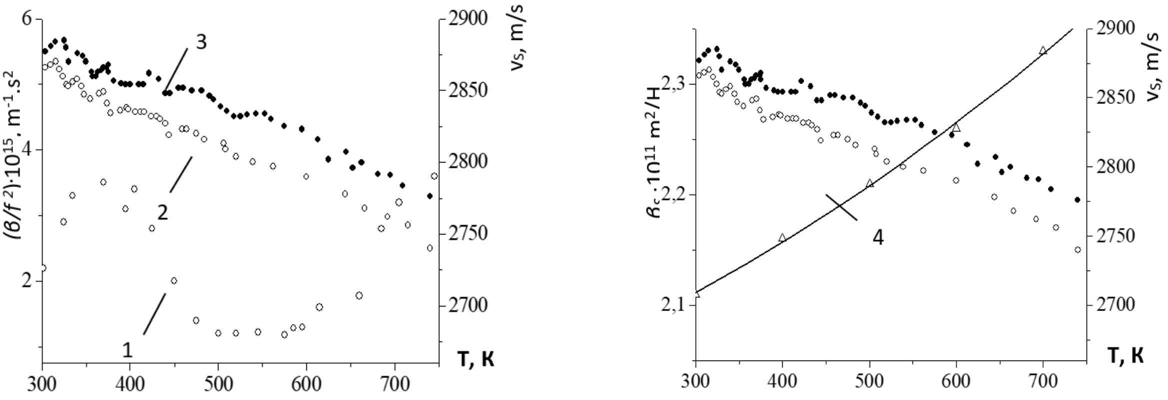

Fig. 2. Absorption, ultrasound propagation velocity and compressibility in liquid gallium: 1 - $\frac{\beta}{f^2}$ data of present work, 2 - v_S data of present work, 3 - v_S data of [14-16], 4 - β_S

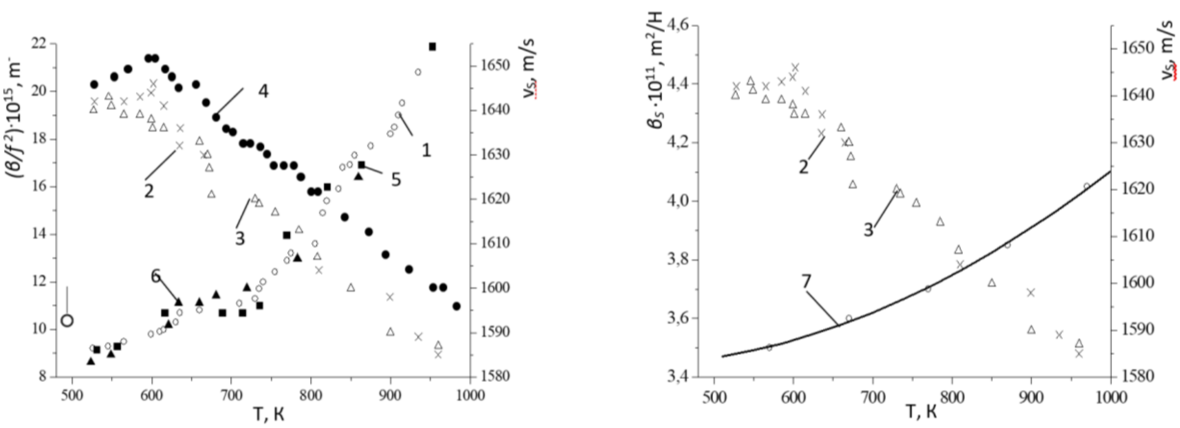

Fig.3. Absorption coefficient, ultrasound velocity and compressibility in bismuth melt: 1 - $\frac{\beta}{f^2}$, 2 - v_S at f = 53.1 MHz and 3 - v_S at f = 31.9 MHz data of present work, 4 - v_S, 5 - $\frac{\beta}{f^2}$ and 6 - $\frac{\beta}{f^2}$ data of [17-18], 7 – β_S

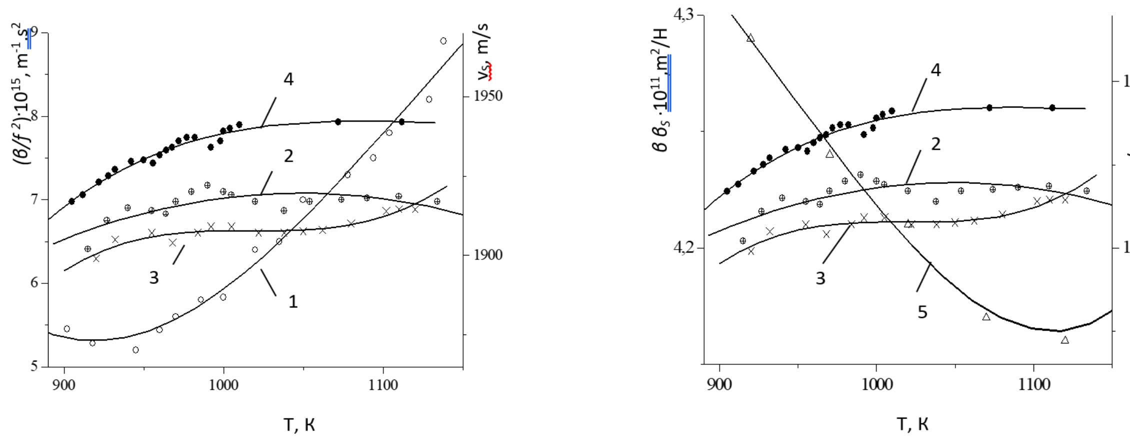

Fig. 4. Absorption coefficient, ultrasonic velocity and compressibility in antimony melt:

1- $\frac{\beta}{f^2}$ data of present work, 2- v_S at f = 52.0 MHz, 3 - v_S at f = 31.0 MHz and 4- v_S data of [18], 5 - β_S

Despite the fact that germanium belongs to the most studied semiconductor substances, interest to it does not weaken, as germanium serves as a key «type-setting» object, with which the properties and behavior of complex semiconductor materials are compared [19]. The research results are presented in Fig. 5. In the interval T_{ml} - 1000K there is a pronounced extremum of the absorption value, and at further heating the ultrasound absorption in liquid germanium noticeably increases as in most liquid metals [19]. The noted anomalies of absorption polytherms and ultrasound velocity in this case in the period after melting directly indicate the process of structural rearrangement in the direction of the melt described two-structure cluster model formation in this paper.

In accordance with this model, the melt near the melting point is a «solution» of two components: atomic matrix with metallic chemical bonds and larger associations of atoms - clusters, in which the chemical bond between atoms is realized mainly by covalent type. Obviously, during heating the clusters are disaggregated and at the same time their content in the total volume of the melt decreases. Both of these processes can lead to a decrease in the absorption of ultrasonic waves. Further heating of the germanium melts leads to the usual thermal loosening of its structure, due to which the absorption of ultrasound increases. The study of viscosity, density and electrical conductivity of selenium and tellurium melts shows features in the behavior of these properties as heating [14,16,20,21].

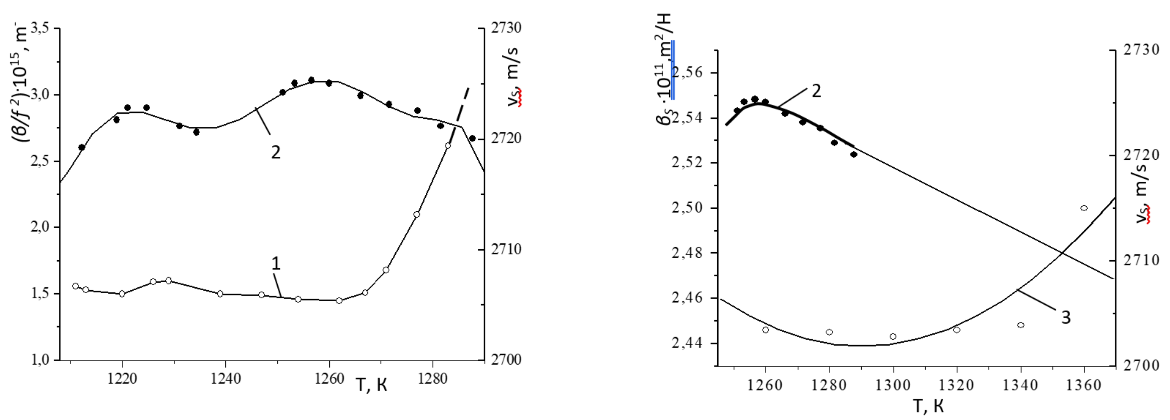

Fig.5. Absorption coefficient, ultrasound velocity and compressibility in liquid germanium:

1 - $\frac{\beta}{f^2}$ data of present work, 2 - v_S data of [19], 3 - β_S

Fig. 6 shows the results of studies of absorption coefficient, ultrasonic velocity and theoretical values of β_S in liquid selenium at temperatures from the melting point to 700 K. Data from [14,16,20,21] are plotted in the same figure (curves 3,2,6,5, respectively). The ultrasound velocity data presented in [20,21] correspond to a frequency of 5 MHz. Nevertheless, the results up to 680 K agree quite satisfactorily. At higher temperatures, our data show that the v_S polyterm decreases steeper with temperature. According to the character of the change in the steepness of the v_S polytherm, we divided the whole temperature interval of the study into three sections. At the first site $\frac{d\text{v}_s}{dT} = 0.975 m/s\cdot K$, at the second site $\frac{d\text{v}_s}{dT} = 0.675 m/s\cdot K$,at the third site $\frac{d\text{v}_s}{dT} = 1.125 m/s\cdot K$. It is interesting to note that at the temperature T = 580 K in Fig. 6 the steep change in the temperature dependence of the viscosity coefficient is completed [16] and it is at this temperature that there is the first kink in the ultrasonic velocity polyterm and a minimum in the $\frac{\beta}{f^2}$ curve.

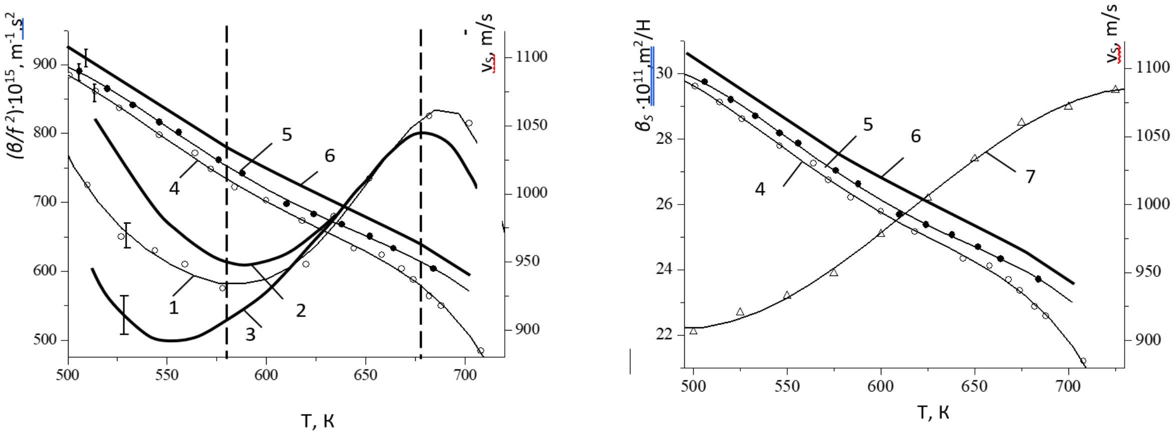

Fig.6. Absorption coefficient, ultrasound velocity and compressibility in liquid selenium:
1- $\frac{\beta}{f^2}$ and 4 - v_S data of present work, 2 - $\frac{\beta}{f^2}$, 3 - $\frac{\beta}{f^2}$, 5 - v_S , 6 - v_S data of [14, 20], 7 - β_S

Fig.7 shows our results on the elastic properties of the tellurium melt together with the data of [21] on the absorption (curve 3) and ultrasound velocity (curve 5).

According to the character of the $\frac{\beta}{f^2}$ polytherm, the whole temperature range can be divided into three sections. In section 1 from the melting point up to about 790 K, the ultrasound absorption decreases with increasing temperature. A decreasing $\frac{\beta}{f^2}$ polyterm was not found by the authors [20]. It should be noted that they did not start their measurements from the melting point, but at 45 degrees above the latter. At site II, a very slight increase in absorption with temperature is observed and, finally, at site III, a substantial increase in absorption commensurate with that in liquid metals [21].

Gallium and indium antimonide compounds are the closest electronic analogues of germanium [14-16,19]. The melting of GaSb and InSb compounds results in a dramatic change in the near-order structure and the nature of chemical bonding. Fig. 8 and 9 show the results of ultrasound absorption $\frac{\beta}{f^2}$ in GaSb and InSb melt together with the data [22] on the ultrasound propagation velocity v_S and the value of β_S. It can be seen that the polyterm of the absorption coefficient normalized by the square of the carrier frequency $\frac{\beta}{f^2}$, near the melting temperature, decreases with temperature and, only after passing through a pronounced minimum (for GaSb), increases with further heating.

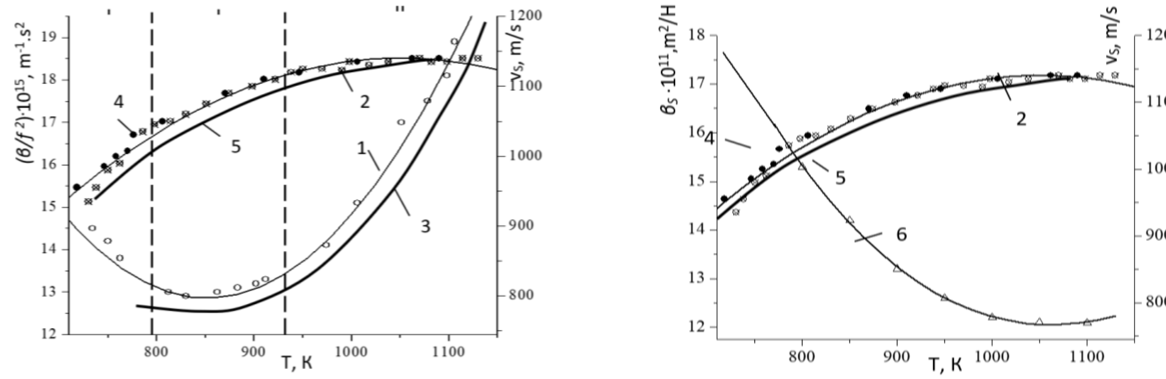

Fig.7. Absorption coefficient, ultrasound propagation velocity and compressibility in tellurium melt: 1- $\frac{\beta}{f^2}$ and 2- v_S data of present work, 3 - $\frac{\beta}{f^2}$, 4- v_S and 5- v_S data of [21], 6-β_S

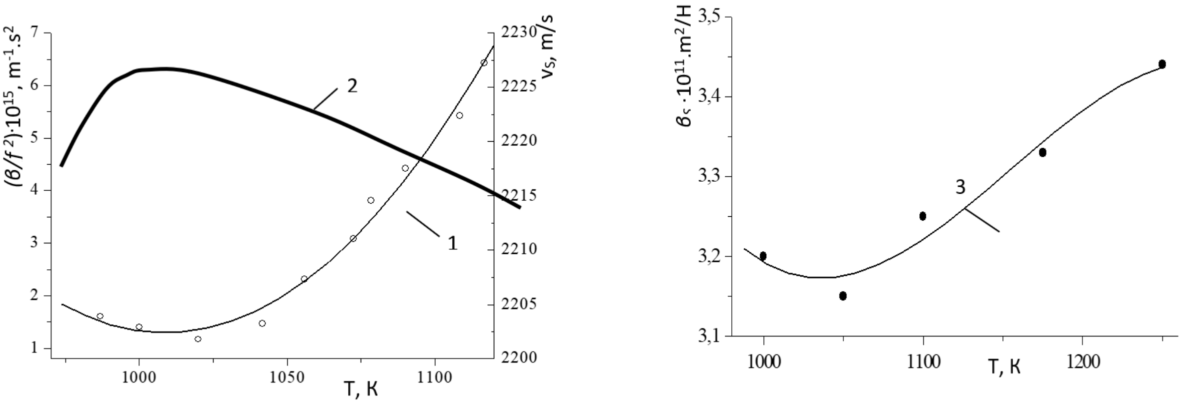

Fig.8. Absorption coefficient, ultrasonic velocity and compressibility in gallium antimonide melt [14-16]: 1 - $\frac{\beta}{f^2}$ and 2 - v_S data of present work, 3 - β_S

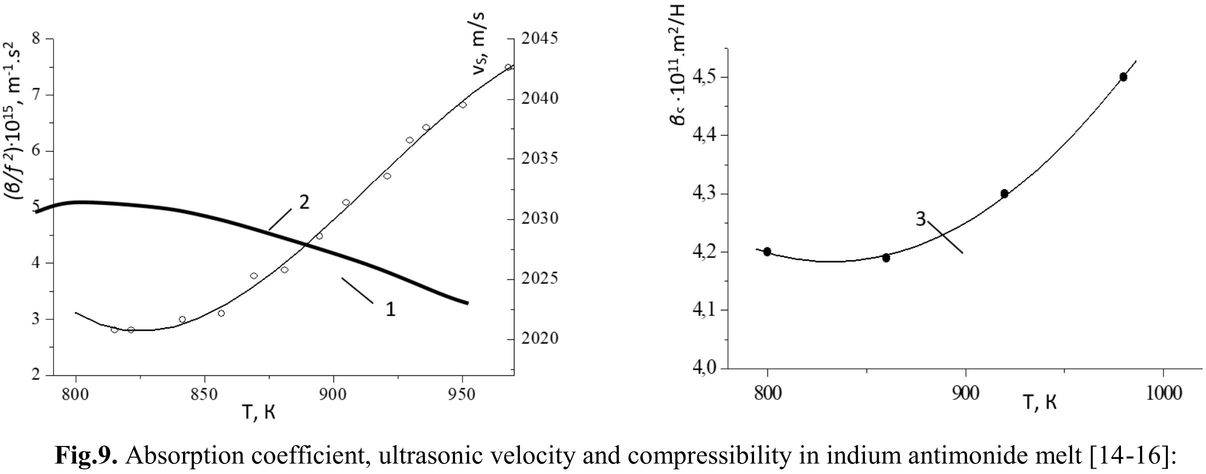

Fig.9. Absorption coefficient, ultrasonic velocity and compressibility in indium antimonide melt [14-16]: 1 - $\frac{\beta}{f^2}$ and 2 - v_S data of present work, 3 - β_S

4. Conclusion

Thus, the conducted studies of elastic wave energy absorption as well as ultrasound velocity allow us to identify such features in the behavior of sound absorption coefficient polyterms that:

1. With sufficient certainty indicate the absence or presence of structural changes during melt heating.

2. In comparative analyses make it possible to conclude that there is not a single mechanism of structural changes.

3. Show the possibility of different mechanisms realization of structural changes in the same melt in different temperature ranges.

This work demonstrated the practical importance of acoustic measurements in metal melts. We performed calculations of ultrasonic treatment of molten systems, investigated the physical and chemical effects of ultrasound on the structural properties of liquid molten systems. We found that the chemical effect is an irreversible and permanent change in the atomic weight and atomic-weight distribution due to ultrasound. Calculations showed that with an increase in the ultrasound intensity, the atomic weight of molten metals decreases, while the orientation of atoms along the flow direction decreases. Atomic orientation is an effect caused by the excitation of atoms by ultrasound. Ultrasonic waves propagate in a straight line. The wavelength and intensity of the ultrasonic vibrations determine the resolution and attenuation of the signal. High frequency of ultrasonic vibrations (short wavelength) is characterized by an improvement in spatial resolution, while the penetration depth decreases. Low frequency (long wavelength) is characterized by an increase in the wave depth, but the resolution decreases. Ultrasonic vibration increases the motion of atoms, a change in the structure of atoms occurs, which makes them more disordered. Ultrasonic vibration affects the relaxation process of molten metals, resulting in a weakening of the elastic effect. The relaxation process is attributed to the slow establishment of equilibrium between the atoms of the melts. The $\frac{\beta}{f^2}$ polytherms behavior in the «post-melting» temperature range is associated with relaxation mechanisms in the high-temperature region, where viscosity has a maximum value and the ultrasound absorption coefficient is minimal. At higher temperatures, despite the fact that viscosity has a minimum value, the sound absorption coefficient remains virtually unchanged. Relaxation occurring in alloys under the action of periodic stresses is a structural relaxation associated with a change in the average number of nearest neighbors surrounding a given atom. Relaxation is characterized by diffusion rates.

Conflict of interest statement.
The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

CRediT author statement
Kazhikenova S.Sh.: conceptualization, methodology; writing – review and editing; **Shaikhova G.S.:** formal analysis, investigation; **Shaltakov S.N.:** resources, supervision. All authors have read and agreed to the published version of the manuscript.

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