



# The structure of clusters in liquid alloys of pseudo-binary PbTe-Bi<sub>2</sub>Te<sub>3</sub> system

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## ABSTRACT

**Purpose:** of this paper is to study the structure of melts of quasi-binary system Bi<sub>2</sub>Te<sub>3</sub>-PbTe by means of X-ray diffraction method. The aim of the research was to investigate the short range order in melts comparing it with the structure in solid state.

**Design/methodology/approach:** Analysis of the structural factors, radial distribution functions of atoms and basic structural parameters showed that the structure of melts at temperatures near the liquidus shows microheterogeneity.

**Findings:** On the basis of the analysis of structural factors, functions of the radial distribution of atoms and basic structural parameters, it is shown that in the given concentration the short range order structure of liquid alloys of pseudo-binary PbTe-Bi<sub>2</sub>Te<sub>3</sub> system is microinhomogeneous and is characterized by the presence of associates, whose atomic arrangement is like to the structure of solid compounds, existing in this concentration range.

**Research limitations/implications:** To complete the understanding of short-range order effect on the formation of the physical properties of Pb-Bi-Te alloys, further studies of the thermoelectric properties of these alloys in the liquid state are needed.

**Practical implications:** The promise of the considered direction requires an experimental and theoretical study of the processes of bulk, thin film and nanostructured material. In this case, it is necessary to develop a technology for the synthesis of compounds of Pb-Bi-Te system, obtaining thin films and nanostructures using the vapour phase methods with studying the mechanisms of thermoelectric properties of the material formation and optimization of technological regimes for obtaining effective thermoelectric materials based on compounds of Pb-Te-Bi system.

**Originality/value:** The processes of structure formation of nanosystems with given characteristics are investigated, because among numerous thermoelectric materials, bismuth telluride (Bi<sub>2</sub>Te<sub>3</sub>) and its alloys are the most important thermoelectric materials used in state-of-the-art devices near room temperature, and lead telluride (PbTe)-based alloys are extensively used in power supplies for space exploration and generators for use at medium to high temperatures.

**Keywords:** Metallic alloys, Short range order, Melts, Thermoelectric materials

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## MATERIALS

## 1. Introduction

The interest in multicomponent melts is due not only to their widespread practical application, but primarily to the fact that representatives of both ordered and disordered systems exhibit a number of interesting physical phenomena and effects. The lack of a complete theory to successfully describe the structure of liquids of different classes makes experimental studies of multicomponent melts systems particularly important. In recent years, the relevance of these studies has been reinforced by the fact that amorphous metal alloys are genetically associated with the liquid state, whose physical features are particularly interesting from both fundamental and applied points of view. Studies of nanosystems of various types are closely related to the study of the features of formation of cluster structural units in melts. Of all the multi-component melts conducted today, double metal melts are the most studied objects. At the same time, the question of the extent to which metal melts are close to atomic solutions remains open. This is especially true of the concentration range corresponding to the diluted solutions. It is very important to study the short-range concentration changes of such melts.

One of the most important and priority areas of research is the development of new energy-saving technologies, autonomous energy sources, the use of thermal waste, and more. In this case, the studies related to the direct conversion of thermal energy into electrical energy are of greatest importance.

The perspective of the considered direction requires experimental and theoretical study of the processes of formation of massive, thin-film and nanostructured material. It is necessary to develop the technology of synthesis of compounds of Pb-Bi-Te systems, production of thin films and nanostructures by vapour-phase methods, to investigate the formation mechanisms and thermoelectric properties of the obtained material; optimize process modes to obtain efficient thermoelectric materials based on Pb-Bi-Te system compounds.

Among numerous thermoelectric materials, bismuth telluride ( $\text{Bi}_2\text{Te}_3$ ) and its alloys are the most important TE materials used in state-of-the-art devices near room temperature, and lead telluride (PbTe)-based alloys and SiGe alloys are extensively used in power supplies for space exploration and generators for use at medium to high temperatures. Significant efforts have been made in recent years to improve their figure of merit by making these materials special-nano-structured, including nanowire arrays and quantum dot superlattice thin films.

Let us ask the question: why do we need to investigate the structures of liquid melts? Any alloys we get from the liquid state. In a liquid state structure and physical properties of the alloys are formed. With specific treatment the alloy in the liquid state, we can influence on the structure of short-range order. For example, special cooling or addition of certain other elements we can control the formation of clusters and their size in the liquid. Respectively we can achieve the formation of nanoclusters in solid state. In connection with this we have investigated the structure of binary Bi-Te, Pb-Te and ternary Bi-Pb-Te melts of compositions within the homogeneity range of  $\text{Bi}_2\text{Te}_3$ , PbTe and cross section phase diagram (Fig. 1) of the quasi binary system of these two compounds. It was also interesting to investigate the structural correlation between the solid state and the melt.

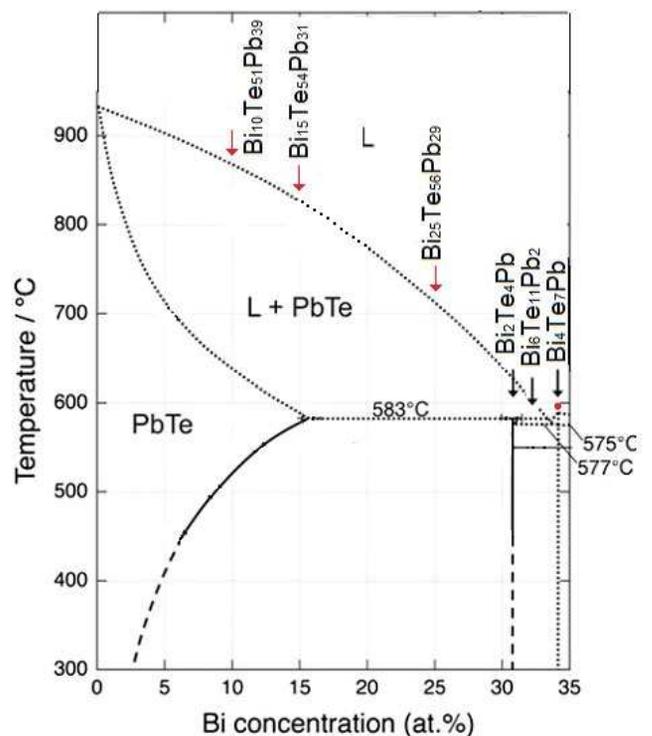


Fig. 1. Cross section phase diagram of the quasi binary system of PbTe- $\text{Bi}_2\text{Te}_3$  [1]

## 2. Experiment

The samples for X-ray diffraction experiment were prepared from Bi and Te with purity of 99.99%. Quartz capsules with the appropriate amounts of the elements were

evacuated, flushed with argon and sealed. After that they were melted and homogenized.

The diffraction measurements were performed in the reflection mode. A primary graphite-monochromator was employed in order to produce monochromatic radiation from X-ray source ( $\lambda_{\text{Cu-K}\alpha}=1,54\text{\AA}$ ). All the measurements were done under 50 mbar helium pressure to reduce tellurium evaporation and to avoid oxidation. The total scattered intensities were recorded within the range  $5^\circ < 2\theta < 90^\circ$  with variable steps. The relative statistical error for the measured points is less than 2%.

X-ray diffraction patterns have been obtained at temperature, which was higher than melting point by 5 K. After calculating the structure factor Fourier analysis was used to obtain the atomic radial distribution functions from which interatomic distances and coordination numbers were obtained [2,3].

### 3. Results

Lead telluride is an effective thermoelectric material for the medium temperature (500-850) K range, and Bi<sub>2</sub>Te<sub>3</sub> at room temperature. The increased interest in them was caused by theoretical prediction and experimental confirmation of the possibility of significant increase of thermoelectric figure of merit in nanoscale structures. In this regard, it is of scientific and practical interest to study the nature of the change in the thickness dependence of the thermoelectric properties of both thin films and nanostructures based on them.

It is known that the increase of thermoelectric figure of merit can be achieved by increasing the value of the current carriers mobility ratio to the thermal conductivity of a substance. This can be done by introducing isovalent substitution atoms to ensure the growth of phonon scattering and a significant decrease in the thermal conductivity. Another important way to increase  $Z$  is to increase the coefficient of thermo emf for strong degeneration due to energy carrier selection by barriers at the crystallite boundaries or at the grain blocks. In addition, recent studies have shown that an increase in  $Z$  can be achieved in the transition from bulk to nanostructured.

The relevance of research is determined by the achievements of recent years in the field of nanotechnology and the need for further in-depth study of nanosystems based on chalcogenides of lead to create thermoelectric devices of the new generation (micro-generators for powering portable devices, integrated

thermal control units, microprocessors power supply). It is known that the formation of the alloy structure occurs in the liquid state, therefore, the study of the short range order of the Pb-Te-Bi ternary system is of objective interest.

At the centre of the phase equilibrium diagram of the Bi-Te system is the chemical compound Bi<sub>2</sub>Te<sub>3</sub>. This system has been studied in sufficient detail in the solid state, since it is of interest as a thermoelectric material. This system is also studied in detail in terms of physical properties and in the liquid state. As shown by the results of such studies, there is a singular point on the concentration dependences of the physicochemical properties, which falls into the homogeneity region of this phase, which makes it possible to attribute it to systems with a maximum on the liquidus curve [4].

Alloys with a content of 52 to 65 at.% Te at temperatures close to melting were investigated by X-ray diffraction. The structural factors of these melts at temperatures close to the liquidus line are shown in Figure 2. As can be seen from the figure, the structural factors have a form that is significantly different from the structural factor characteristics of simple liquids. The first maximum is wide enough and occupies an intermediate position between the principal peaks of pure Te and Bi. On the right branch of the main maximum there are inflows whose positions correspond to the influx of Bi and in the second peak – pure Te. The second maximum position is closer to the corresponding maximum of pure Bi than Te. The tellurium maximum falls on the branch of the second maximum and its contribution to the formation of the second maximum is insignificant. Therefore, the possibility of micro-regions based on pure components is unlikely due to this fact. The comparison of the position of the third maximum with the tellurium peak also denies this. Comparing the height of the fundamental maximum with the peak heights of the pure components, they are almost the same as those of molten bismuth. In the process of adding Te to Bi within the studied concentration range, the height of the main maximum is practically unchanged.

Analysis of the concentration dependence of the position of the first maximum of the structural factor shows that this value is constant up to 60 at.% Te, and then shows a local minimum. Therefore, within the existence of a region of homogeneity by lower tellurium concentrations, the short order within the nearest neighbours is unchanged, and undergoes more fundamental changes only when Te is added to the melt corresponding to the Bi<sub>2</sub>Te<sub>3</sub> compound.

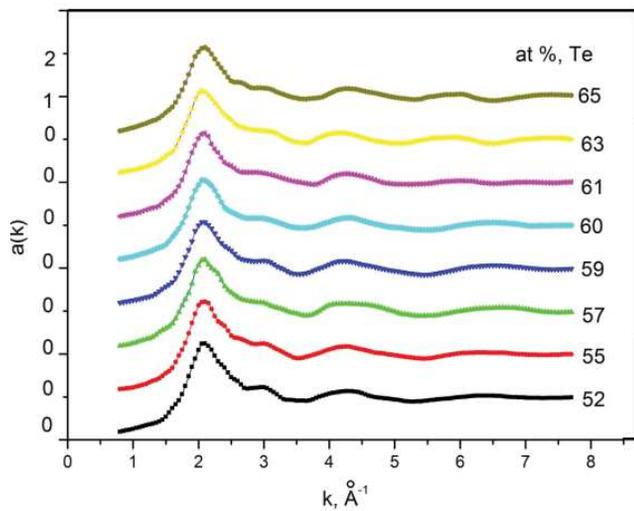


Fig. 2. Structural factors of Bi-Te melts

The pair correlation functions were obtained from the structural factors of the Fourier transform method. The main structural parameter – the distance between the nearest neighbours – is shown in Figure 3. As can be seen, almost identical values of  $r$  are characteristics of melts with a content of 53 to 60 at.% Te. An insignificant minimum exists in the range of 57-60 at.%, which indicates a greater interaction of atoms of different sorts at the stoichiometry of Bi-Te. The addition of bismuth atoms results in a significant increase in the interatomic distance, indicating a change in the short-range structure. Since the radii of the first coordination spheres of pure Bi and Te are respectively 3.38 and 2.81 Å, we see that the experimental values are closer to the most probable interatomic distances of Bi than Te. Despite the fact that with increasing tellurium content  $r$  should decrease, provided that there is a structure of the ideal solution type A-A and B-B, the situation is reversed. This may be when, by analogy to solid intermediate phases, vacancies can be formed in the Bi-Bi concentration spheres, that is, the partial function of the radial Bi-Bi distribution becomes defective, resulting in an increase in interatomic distance. On the other hand, there is an increase in the degree of micro-heterogeneity of the structure due to the fact that the complexes of type Bi-Te will not dissolve the excess tellurium atoms for them, and a short order will exist on the basis of two types of micro-groups: Bi-Te and Te. Self-associated tellurium atoms with a specific structure of coordination spheres will be significantly different from Bi-Te complexes and therefore will not be coherent in the structure and significant defective zones will form, resulting in an increase in interatomic distance.

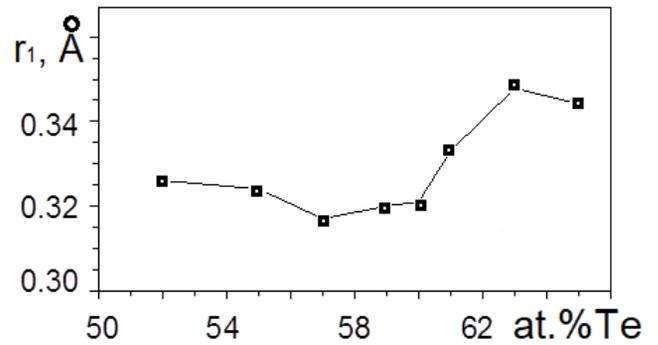


Fig. 3. Concentration dependence of the distance to neighbour atoms of Bi-Te system melts

The dependence of another important parameter – the area under the first maximum – is shown in Figure 4. This distance is commonly considered as effective number of neighbour atoms. We see that the areas estimated by the symmetric method of dividing the first maximum and by calculating it from minimum to minimum have the same nature of change. There is a small minimum in the range of 51-60 at.%, and then a sharp increase in this value to values characteristic of a more dense atomic distribution. Thus, again, we have confirmation that the chemical order is more advanced by the Bi than the Te. The increase in the density of the structure near  $\text{Bi}_2\text{Te}_3$  by addition of the Te atoms may be caused by the dissolution of the latter at vacant sites in the  $\text{Bi}_2\text{Te}_3$  structural fragments.

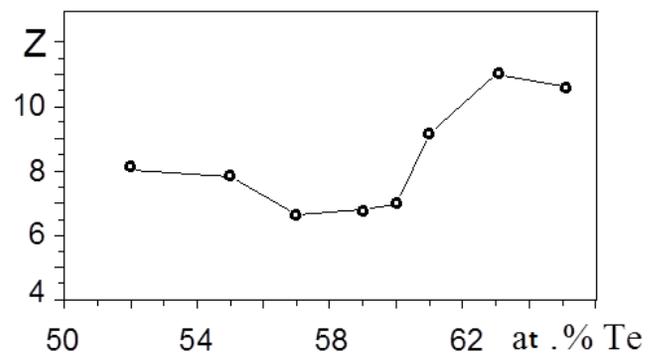


Fig. 4. The concentration dependence of the effective number of neighbour atoms for Bi-Te melts

In a binary Pb-Te system, the physical properties and structure of alloys with different component contents are determined by a chemical compound of  $\text{PbTe}$  of congruent type, which corresponds to the maximum of the hollow form on the line of liquidus the phase equilibrium diagram [4]. From a thermodynamic point of view, this indicates

little stability in Pb-Te melts of structural units based on PbTe-type quasimolecules. In this regard, it can be assumed that near the crystallization temperature, the liquid alloys will have a micro-inhomogeneous structure, which will also be reflected in their physical properties. Thus, according to [5], the maximum isotherm of the concentration dependence of the Pb-Te melts electrical resistance is slightly shifted relative to the equiatomic composition toward tellurium (55 at.% Te). As the temperature rises, it shifts to 60 at.% Te. The analysis of experimental data on electrical resistance revealed that complexes of the type of chemical compound PbTe exist in the liquid state and their share in equiatomic composition is equal to 1/3 of the total number of structural units of the melt [1,7].

In this work, melts of Pb-Te were investigated by X-ray diffractometry in the vicinity of the existence of PbTe at temperatures that are 5 K higher than the liquid curve temperature. We have studied these melts from the point of view of checking the presence of a singular point and the concentration dependences of the structural parameters, as well as the nature of the structure change when deviating from the stoichiometric point.

Figure 5 shows the experimental structural factors (SF) for melts of different concentrations. As can be seen from the figure, the structural factors have a specific appearance and differ from the SF component. Despite the fact that the position of the main peaks does not differ much, the other characteristics are different. In particular, the first peak of lead is symmetrical and high, and tellurium shows a lateral maximum. For a melt with a content of 30 at.% Te, the position of the principal maximum is closer to the corresponding maximum of pure lead. A similar situation is typical for the second and third maxima. The right branch of the SF has inflows due either to the imposition of a side maximum of pure tellurium SF or to its own characteristics. These inflows also exist with higher tellurium content. If we compare the position of the side maximum of pure tellurium, then the experimental structural factor in this interval of wave vectors is a minimum. Therefore, it can be assumed that pure tellurium does not retain its structure in the melt. This is also confirmed by the fact that the second maximum of the tellurium structural factor hits the right slope of the second maximum and does not increase the intensity in this wave vector interval. If there were micro regions based on pure lead, due to the fact that the height of its first peak  $a(k)$  is much larger than tellurium, this feature must also be shown on structural factors for different concentrations. In reality, the main maximum is not high and there is no decrease in its height with increasing tellurium content. On the contrary, even when the lead

content prevails, the height of the first maximum becomes noticeably smaller at 40 and 46 at.% Te. Further, as the amount of this component increases, the peak height increases and becomes relatively larger at 50 and 52 at.% Te. Such an increase in peak height can be attributed to the increase in the degree of melt ordering, since if there is an atomic solution or micro-groups based on pure components, it should decrease. Therefore, on the concentration dependences of structural factors, we observe singularity in the range of 50-52 at.% Te, that is, in the stoichiometry domain of PbTe. In addition to this dependence of the height of the main maxima of structural factors, we also see that near the equiatomic composition the maximum is displaced slightly to the left compared to other curves. But in general, when looking at the features of the main maximum, they are stored in a wide concentration range. It can be assumed from 30 to 40 at.% Te that the influence of short-range order pure lead is significant on the structural state of the melts. But along with the Pb-Pb atomic ones, there are also chemically ordered Pb-Te micro-regions. When the concentration deviates from the composition of the chemical compound PbTe towards tellurium, a more dominant influence of the pure tellurium structure begins to manifest itself. In particular, the position of the principal maximum shifts to the left, and the right branch transforms its inflow so that it becomes closer to the lateral maximum of pure tellurium.

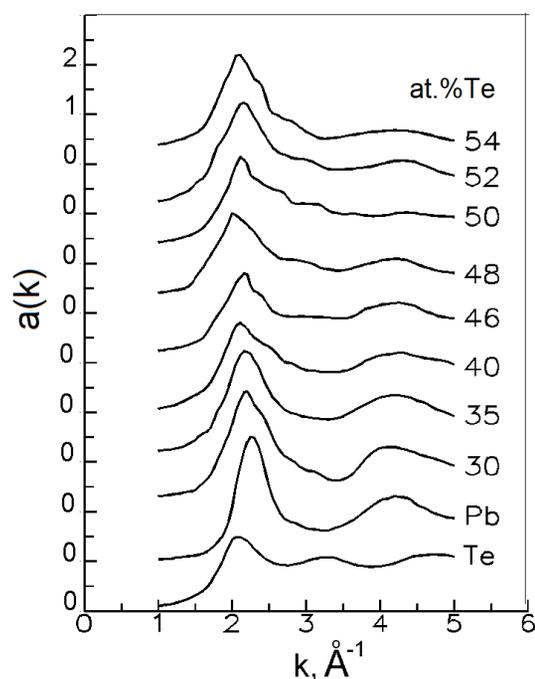


Fig. 5. Structural factors of Pb-Te system melts

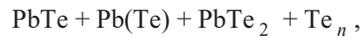
Therefore, the results of X-ray studies indicate that the short-range order of lead-tellurium melts near the PbTe stoichiometry is characterized by an ordered structure similar to that in the crystalline state. Maximum ordering is characteristic of an equiatomic melt. When deviated from this concentration, the melt becomes more micro-heterogeneous due to the appearance of micro areas based on pure lead or tellurium. In the liquid state, there is no significant solubility of lead and tellurium atoms in the micro regions of PbTe, which, thus, makes it possible to assert the absence of a wide range of concentrations of the existence of the chemical compound PbTe in the liquid state and to attribute it to systems with a maximum on the liquidus curve.

The data of structural studies is also confirmed by the results of measurement of physical properties. So, the isotherms of viscosity and electrical conductivity exhibit a distinct singular point, the position of which coincides with the stoichiometry of PbTe. Moreover, even raising the temperature to 1473 K does not lead to its disappearance, but only slightly erodes. Viscosity isotherms also have a minimum which position completely coincides with the eutectic point Te + PbTe. This indicates that the viscosity has a constant deviation from the additive law - negative in the existence interval of Te + PbTe eutectic and positive at other concentrations. Moreover, the temperature dependence of viscosity and electrical conductivity is most noticeable with the stoichiometric ratio of the components.

It should be noted that the PbTe compound belongs to the class of liquid semiconductors. The decrease in conductivity to values specific to this type of material is due to the formation of a large number of ion-covalent bonds. There is a charge transfer from lead to tellurium, which is why we have a largely developed ionic bond.

This agrees with our structural data, which also gives a singular point, slightly offset with respect to the equiatomic composition toward tellurium. Thus, slightly smaller tellurium atoms can dissolve in Pb-Te compound within small limits due to the fact that within the ordered microdomain local structural associations can also be formed, due to the fact that tellurium can exhibit variable valence and give two electrons to the zone Conductivity, complexes of type PbTe can be formed, which in structure can be close to the structure of pure tellurium. As a result, the singular point shifts in the concentration dependence of the structural parameters and physical properties, as well as a significant curvature in the temperature dependence of the viscosity in the melts with excess tellurium relative to the equatorial composition. Therefore, based on our research, the melts with concentration close to the chemical

compound Pb-Te can be structurally represented as a four-component system at the cluster level:



where, PbTe – quasi-chemical complexes of equatorial composition which structure is characterized by ion-covalent bonding and is close to that of the crystalline compound; Pb(Te) – an average solution of lead and tellurium atoms, which structure is denser than PbTe complexes, and the bond is mainly metallic; PbTe – complexes with ion-covalent bond and tellurium topology of atomic distribution; Te – self-associated groups of tellurium atoms.

All of these structural elements are in dynamic equilibrium, and the relationship between them will change with temperature and will determine the physical properties.

The study of short-range order of melts of the ternary Pb-Te-Bi system was carried out in the region of concentrations lying on the line phase diagram of the quasi-binary PbTe-Bi<sub>2</sub>Te<sub>3</sub> system. Objects of the study were samples of three concentrations of PbTe + 10, 15, 25 at% Bi and compounds PbBi<sub>4</sub>Te<sub>7</sub>, Pb<sub>2</sub>Bi<sub>6</sub>Te<sub>11</sub>, PbBi<sub>2</sub>Te<sub>4</sub>.

The study of the structure in the solid state showed that the compounds PbBi<sub>4</sub>Te<sub>7</sub>, PbBi<sub>2</sub>Te<sub>4</sub> exist, even at relatively high temperatures, and in alloys with low concentrations of Bi atoms play a significant role in the formation of their structure [1,8].

The obtained structural factors from the diffraction curves are presented in Figure 6.

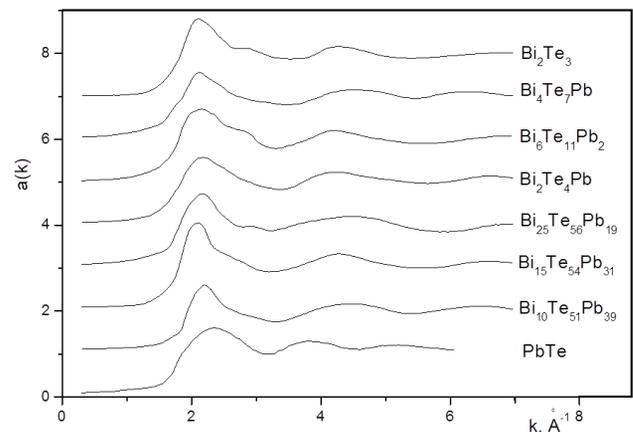


Fig. 6. Structural factors of Bi-Te-Pb melts

Investigations of the structure of the Bi-Te-Pb melts were carried out at temperatures higher than 5 K from the

liquid line (Fig. 6). From the point of view of structural factors, it can be concluded that the structure of short order, even with a slight addition of bismuth atoms, changes significantly. The profile of the principal maximum is more similar to the structure of compounds based on these elements than the maximum of the PbTe compound.

The concentration dependence of the position of the first and second maxima of structural factors does not show large changes, although there is a slight decrease with increasing bismuth content (Fig. 7).

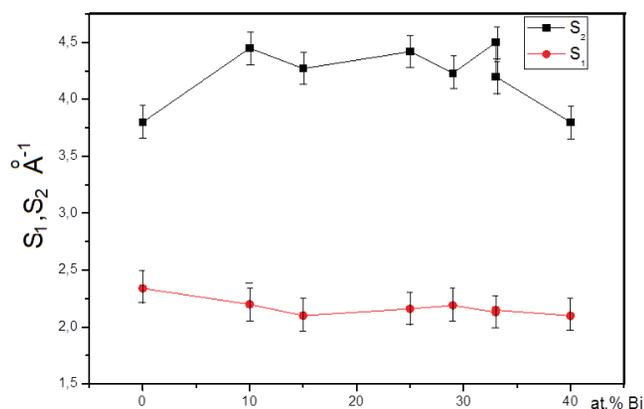


Fig. 7. Concentration dependence of position of the first and second maxima of structural factors of melts of the Bi-Te-Pb system

Functions of the radial distribution for the melts of these alloys were calculated from the experimentally obtained structural factors by the Fourier transform (Fig. 8).

The main structural parameters of the short-range order of the Bi-Te-Pb melts are given in the Table 1.

The positions of the first and second coordination radii of the alloys under investigation are close to those of Bi<sub>2</sub>Te<sub>3</sub> and not PbTe, which is due to the fact that even a slight addition of bismuth leads to the chemical ordering of Bi atoms with Te atoms. This assumption is confirmed by the values of the coordination number.

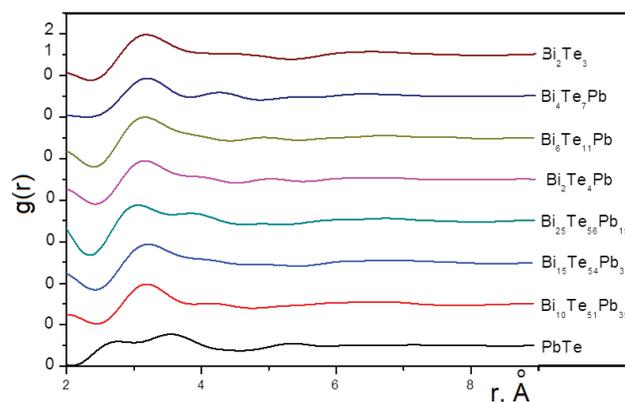


Fig. 8. Pair correlation functions of Bi-Te-Pb melts

Table 1.

The main structural parameters of the short-range order of the Bi-Te-Pb melts

	S <sub>1</sub> , Å <sup>-1</sup>	S <sub>2</sub> , Å <sup>-1</sup>	r <sub>1</sub> , Å	r <sub>2</sub> , Å	N
Bi <sub>2</sub> Te <sub>3</sub>	2.10	4.28	3.17	6.5	6.4
Bi <sub>4</sub> Te <sub>7</sub> Pb	2.13	4.50	3.19	6.45	5.9
Bi <sub>6</sub> Te <sub>11</sub> Pb	2.15	4.20	3.15	6.69	5.7
Bi <sub>2</sub> Te <sub>4</sub> Pb	2.19	4.23	3.15	6.48	5.6
Bi <sub>25</sub> Te <sub>56</sub> Pb <sub>19</sub>	2.16	4.42	3.05	6.34	4.3
Bi <sub>15</sub> Te <sub>54</sub> Pb <sub>31</sub>	2.10	4.27	3.20	6.63	6.2
Bi <sub>10</sub> Te <sub>51</sub> Pb <sub>39</sub>	2.20	4.45	3.19	6.40	6.5
PbTe	2.34	3.80	3.54	5.35	12.6

It should be noted that concentration dependences of most probable interatomic distance  $r_1(x)$  and number of neighbour atoms  $N(x)$  reveal two subregions (Tab. 1). In first of them these parameters decreasing with Bi-atoms content up to 25. at.% , whereas in second one corresponding to

higher content of Bi their increasing occurs. Such behaviour is supposed to be related with existence of solid PbTe binary compound and PbBi<sub>4</sub>Te<sub>7</sub> and PbBi<sub>2</sub>Te<sub>4</sub> ternary compounds. Each of such compounds attempt to promote the formation of short range order similar to own type of structure.

## 4. Conclusions

The melts of the Bi-Te system with concentration close to the  $\text{Bi}_2\text{Te}_3$  compound exhibits singularity on the concentration dependences of the structural parameters at stoichiometric concentration. A chemically ordered short order of this type exists upon dissolution of Bi atoms and becomes microhomogeneous upon addition of Te atoms. It is established that structural complexes of  $\text{PbTe} + \text{Te}$  and  $\text{PbTe} + \text{Pb}$  are formed in Pb-Te melts with concentration in the range of 30-54 at.% Te. The main feature of melts structure of  $\text{Bi}_2\text{Te}_3$ -PbTe quasi-binary system is the presence of associates with the structure like to one of solid compound, existing in this system.

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