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THE Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 ISOPLETH SECTION OF THE Tl-Pb-Bi-Gd-Te SYSTEM

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The purpose of this study was to investigate the phase relations in the Tl-Pb-Gd-Te quaternary system on the Tl_9GdTe_6-Tl_9BiTe_6-Tl_4PbTe_3 composition area. Based on the results of the differential thermal analysis, the powder X-ray diffraction technique and the microhardness measurements, it revealed that the system is characterized by unlimited solid solutions crystallized in Tl_5Te_3 structure type.
Keywords: *thallium-lead telluride; thallium-gadolinium tellurides; thallium-bismuth tellurides; phase equilibria; liquidus and solidus surfaces; solid solutions.*

1. INTRODUCTION

Heavy metal chalcogenides, multicomponent phases and composites based thereon are valuable functional materials [1-3]. In recent years, interest in complex thallium chalcogenides has increased due to topological insulators [4-6], Weyl semimetals [6] and thermoelectric materials with anomalously low thermal conductivity [8-10]. Some of these compounds have photoconductivity and are promising for use as detectors of γ and X-ray radiation [11, 12].

Among thallium chalcogenides, Tl_5Te_3 is one of the most suitable matrix compounds for the preparation of new ternary compounds - structural analogues and multicomponent phases. According to the phase diagram of the Tl-Te system [13], this compound melts congruently at 725 K and has a wide homogeneity region (34.5-38 at.% Te). Due to the crystal structure features [14], Tl_5Te_3 has a number of the ternary cation-substituted structural analogues [15-18]. This compound and its ternary analogues attract the attention of researchers, primarily as potential thermoelectric materials [19-22]. Tl_9BiTe_6 compound shows the best thermoelectric figure among these compounds (ZT = 0.65 at 300 K and ZT = 1.2 at 500 K) [22].

The development of physicochemical bases for goal-oriented synthesis of new multicomponent inorganic phases and materials is based on fundamental studies on phase equilibria and thermodynamic properties of the corresponding systems [23-25]. In this respect, the most interesting are systems where it is possible to expect the formation of structural analogues of known binary and ternary compounds or solid solutions based on them.

In recent years, thallium- REEs tellurides with the common formula of Tl_9LnTe_6 have attracted the attention of researchers as promising materials that combine thermoelectric and magnetic properties [26-30]. It is assumed that these compounds can have good thermoelectric

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properties, such as Tl_9BiTe_6 since the introduction of lanthanide atoms lighter than thallium and bismuth into the Tl_5Te_3 crystal lattice should lead to significant mass fluctuations and as a result to a decrease in thermal conductivity and an improvement in thermoelectric properties.

Earlier we presented the results of the research of some Tl_9LnTe_6 compounds as well as the phase relations for a number of systems, including the Tl_5Te_3or Tl_9LnTe_6 compounds [31-35]. The formation of unlimited solid solutions was found for these systems.

In this study, we continue to investigations of the similar systems and present the experimental results on phase relations in the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 section of the Tl-Pb-Bi-Gd-Te system.

The initial compounds of the above-mentioned system were studied in a number of papers. Tl_4PbTe_3 and Tl_9BiTe_6 melt congruently at 893 K [18], and 830 K [17], respectively, while Tl_9GdTe_6 is formed by the peritectic reaction at 800 K [32].These compounds have the following lattice parameters: *a*=8.841, *c*=13.056Å, z=4 (Tl_4PbTe_3)[36]; *a* = 8.855, *c* = 13.048 Å, z=2(Tl_9BiTe_6) [37];*a*=8.870; *c*=13.027 Å, z=2 (Tl_9GdTe_6) [32].

The Tl_4PbTe_3-Tl_9BiTe_6 boundary system is quasi-binary and characterized by the formation of unlimited solid solutions (δ) with Tl_5Te_3-structure [38]. The Tl_9GdTe_6-Tl_9BiTe_6 boundary system is not quasi-binary due to the peritectic character of Tl_9GdTe_6 melting [39].

2. EXPERIMENTAL

2.1. Materials and syntheses

For the experiments we used the following chemical reagents: thallium (granules, 99.999 %), lead (ingot, 99.99 %), gadolinium (powder, 99.9%), bismuth (granules, 99.999 %), and tellurium (broken ingots 99.999 %). Taking into account that thallium and its compounds are highly toxic, the protective gloves were constantly used when working with thallium.

Stoichiometric amounts of the starting components were put into silica tubes of about 20 cm in length and a diameter of about 1.5 cm, sealed under a vacuum of 10^{-2} Pa. Tl_4PbTe_3 and Tl_9BiTe_6 were synthesized by heating in a resistance furnace at 920 K followed by cooling in the switched-off furnace.

In order to avoid the reaction of gadolinium with quartz, the ampoule was graphitized using pyrolysis of toluene. After synthesis, the intermediate ingot of Tl_9GdTe_6 was powdered in an agate mortar, carefully mixed, pressed into a pellet and annealed at 750 K within~700 h.

The purity of resulting compounds was established by the differential thermal analysis (DTA) and X-ray diffraction (XRD).

The alloys of the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 system were prepared by melting of the previously synthesized and identified ternary compounds. After synthesis, the samples containing >60% Tl_9GdTe_6 were powdered, mixed, pressed into pellets and annealed at 750 K during ~ 800 h in order to complete the homogenization.

2.2. Methods

DTA and XRD analyses, as well as micro-hardness measurements, were used to analyze the samples of the investigated system.

DTA heating curves were obtained in the range within room temperature ~1400 K at a heating rate of 10 K· min^{-1} using a Netzsch 404 F1 Pegasus differential scanning calorimeter system. The crystal structure of the initial compounds and intermediate alloys was studied by XRD method on a Bruker D8 powder diffractometer (Cu*K*α radiation) in the 2θ range of 10–75°. The tetragonal lattice parameters were indexed using the Topas V3.0 software. Micro-hardness measurements were performed on a PMT-3 micro-hardness tester and an indenter load of 0.2 N.

3. RESULTS AND DISCUSSION

The combined analysis of experimental results and literature data on boundary system Tl_4PbTe_3-Tl_9BiTe_6 [38] and Tl_9GdTe_6-Tl_9BiTe_6 [39] allowed us to establish the scheme of phase relations in the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 (Fig.1-5).

The Table presents the results of DTA, micro-hardness measurements, and parameters of the tetragonal lattice for some intermediate alloys of the Tl_4PbTe_3-Tl_9GdTe_6 system.

Tl_4PbTe_3-Tl_9GdTe_6 section (Fig.1) is non-quasi-ternary due to the peritectic character of melting of Tl_9GdTe_6 but stable below the solidus and characterized by the formation of a continuous series of solid solutions (δ-phase with Tl_5Te_3-structure). Within the composition range 0–60 mol % Tl_9GdTe_6, the δ-solid solutions primarily crystallize, and within a range richer in Tl_9GdTe_6, the $TlGdTe_2$ does. According to the Gibbs phase rule, L+$TlGdTe_2$+δ three-phase area should form within the range 60–100 mol % Tl_9GdTe_6 below 800 K in the phase diagram because of the mono variant peritectic reaction L+$TlGdTe_2$↔δ. However, this area was not experimentally fixed because of the narrow temperature range and was shown by a dashed line.

Table. Experimental data of the DTA, micro-hardness measurements and parameters of tetragonal lattice for the alloys of the Tl_4PbTe_3-Tl_9GdTe_6 section of the Tl-Pb-Bi-Gd-Te system

Solid phase compositions	Thermal effects, K	Micro-hardness, MPa	Tetragonal lattice parameters, Å	
			a	*c*
Tl_4PbTe_3	893	1120	8.8409	13.0556
$Tl_{8.2}Pb_{1.6}Gd_{0.2}Te_6$	865-882	1170	8.8467	13.0498
$Tl_{8.4}Pb_{1.2}Gd_{0.4}Te_6$	842-865	1200	8.8525	13.0441
$Tl_{8.5}Pb_{1.0}Gd_{0.5}Te_6$	837-856	-	-	-
$Tl_{8.6}Pb_{0.8}Gd_{0.6}Te_6$	824-845	1190	8.8583	13.0384
$Tl_{8.8}Pb_{0.4}Gd_{0.8}Te_6$	809-824;1110	1150	8.8641	13.0327
$Tl_{8.9}Pb_{0.2}Gd_{0.9}Te_6$	804-812;1150	-	-	-
Tl_9GdTe_6	800; 1190	1100	8.8701	13.0270

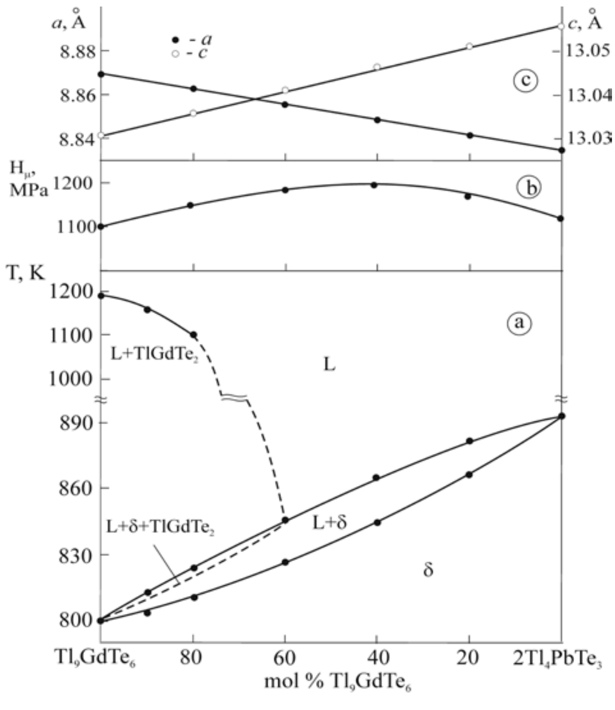


Fig.1. Phase diagram (a), concentration dependencies of micro-hardness (b), and lattice parameters (c) of the alloys of the Tl_4PbTe_3-Tl_9GdTe_6 section of the Tl-Pb-Bi-Gd-Te system.

In order to determine the phase constituents, polished surfaces of the intermediate samples were visually observed under the microscope of micro-hardness meter. The micro-hardness curves have a flat maximum which is characteristic for systems with a continuous series of substitutional solid solutions (Fig.1b) [40].

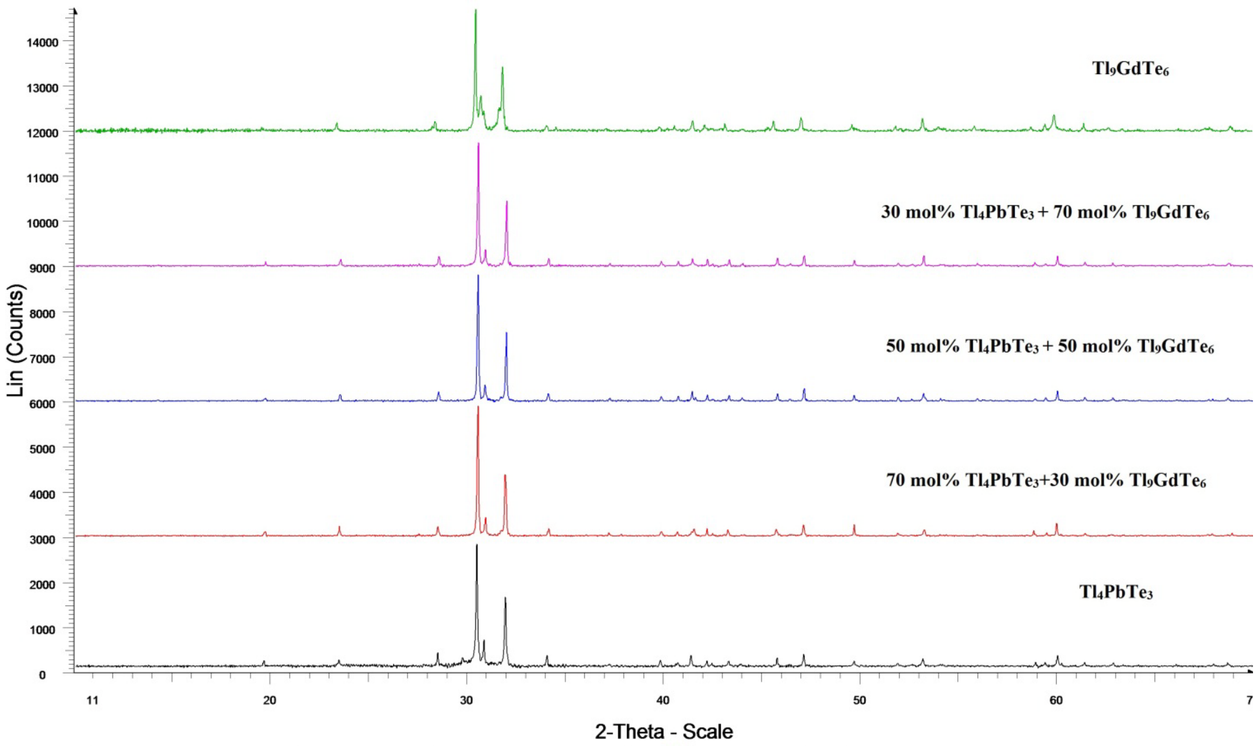


Fig. 2. XRD powder patterns for starting compounds and some alloys of the Tl_4PbTe_3-Tl_9GdTe_6 section.

The XRD data also confirm the phase diagram of theTl_4PbTe_3-Tl_9GdTe_6 systems and indicate the formation of a continuous series of solid solutions. As seen in Fig. 2, the XRD patterns of the initial tellurides and intermediate alloys in this system are similar to that of Tl_5Te_3, with a slight displacement of their reflections.The composition dependences of the lattice parameters of the solid solutions are subject to the Vegard's rule within the experimental uncertainty [41].

The liquidus and solidus surfaces projections (Fig.3).

The projection of the T–x–y phase diagram onto the Gibbs composition triangle (Fig. 3) showed that the liquidus surface consists of two-phase areas of the primary crystallization of the δ-solid solutions (area 1) and $TlGdTe_2$ (area 2). These surfaces are separated by curve *ab* which represents the $L+TlGdTe_2 \leftrightarrow \delta$ peritectic equilibrium. The solidus projection is formed by a single surface (dashed isotherms), corresponding to the onset of the melting of the δ-phase.

Isopleth sections of the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 system (Fig.4).

In order to construct a complete T-x-y diagram and refine the boundaries of primary crystallization areas of δ-phase and $TlGdTe_2$, we constructed some isopleth sections. Fig.3 shows the vertical sections ~~of~~ Tl_9GdTe_6-[A], Tl_9BiTe_6-[B] and Tl_4PbTe_3-[C] of the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 system (where A, B, and C represent the 1:1 alloys in the boundary systems as shown in Fig.3).

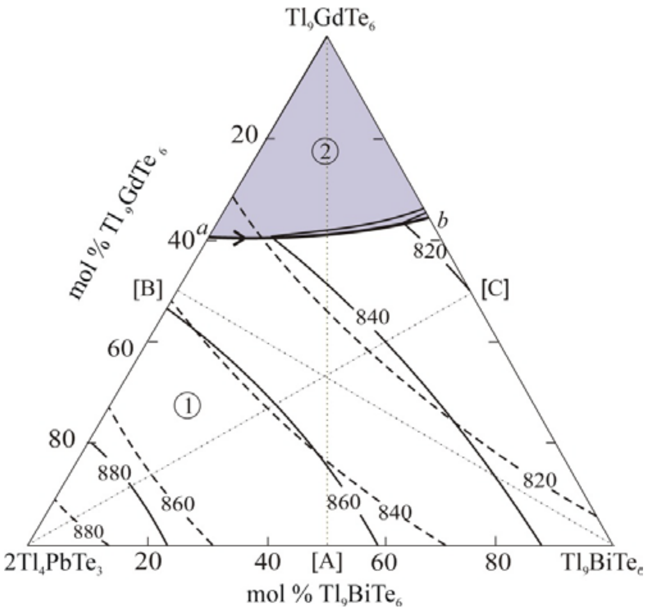
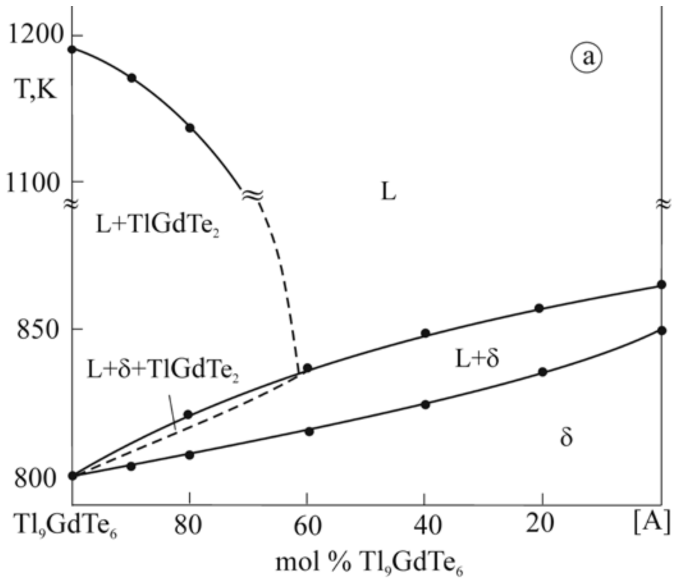


Fig.3. Liquidus and solidus surfaces projections of the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 section of the Tl-Pb-Bi-Gd-Te system. The investigated sections are shown by dash-dot lines. A,B and C are equimolar compositions of the boundary systems. Primary crystallization phases: 1-δ; 2-$TlGdTe_2$

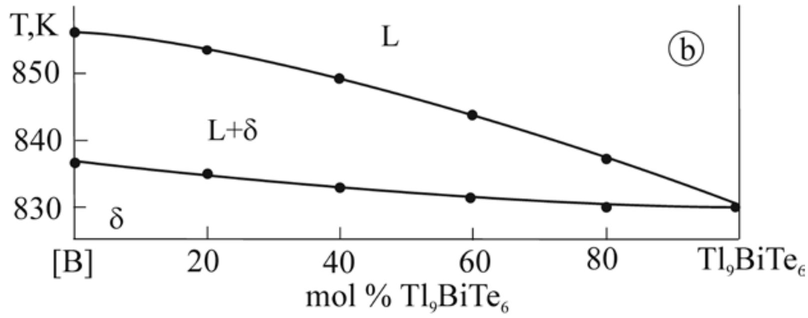
It is seen in Figs. 4a and 4c that only the δ-phase crystallizes from the melt over the entire composition range on the Tl_4PbTe_3-[C] and Tl_9BiTe_6-[B] systems.

On the Tl_9GdTe_6-[A] section, the δ-phase crystallizes from the melt in the composition area <60 mol% Tl_9GdTe_6. At higher Tl_9GdTe_6 content, the $TlGdTe_2$ first crystallizes. This is followed by the monovariant peritectic reaction $L+TlGdTe_2 \leftrightarrow \delta$. As a result, the $TlGdTe_2$ disappears and the excess melt crystallizes to give the δ-phase(Fig.4a).

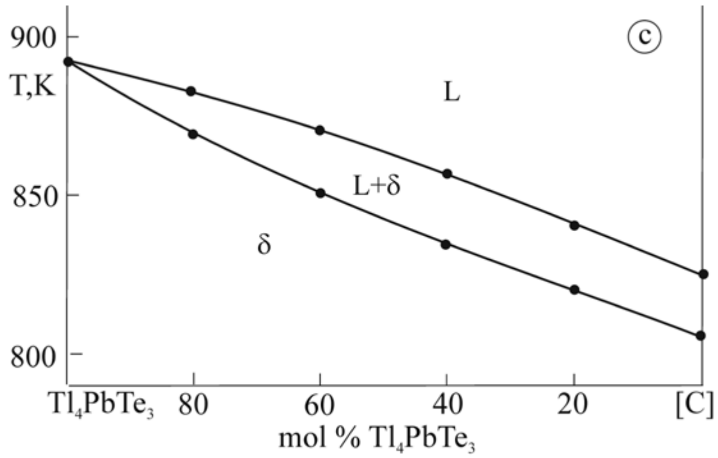
The XRD powder patterns for selective alloys on polythermal sections reaffirmed continuous solid solutions with the Tl_5Te_3-structure.



a)



b)



c)

Fig.4. Polythermal sections Tl_9GdTe_6-[A], Tl_9BiTe_6-[B] and Tl_4PbTe_3-[C] of the phase diagram of the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 section of the Tl-Pb-Bi-Gd-Te system. A, B, and C represent the 1 : 1 alloys in the boundary systems as shown in Fig.3.

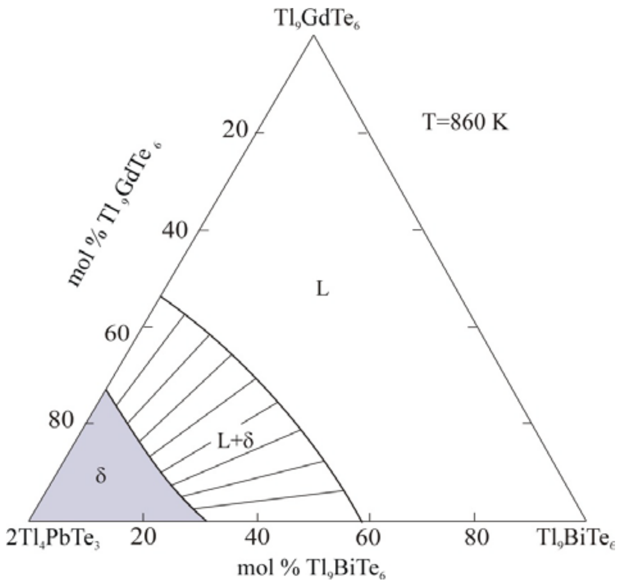


Fig.5. Isothermal sections at 860 K in the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 section of the Tl-Pb-Bi-Gd-Te system.

Isothermal section at the 860 K of the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiT_6 section (Fig.5)

It should be noted that in the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 section the directions of connodes in the *L*+δ two-phase regions do not coincide with the *T–x* planes of inner sections and vary with temperature. The directions of the connodes at 860 K are clearly indicated the corresponding isothermal section of the phase diagram (Fig. 5).

4. CONCLUSION

T-x-y phase diagram of the Tl-Pb-Bi-Gd-Te system in the Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 composition range is constructed. A continuous series of solid solutions is established in the studied section. Received experimental data can be used for choosing the composition of solution-melt for the growth of the high-quality crystals of the δ- phase which is of interest as a thermoelectric material.

5. ACKNOWLEDGMENT

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ПОЛИТЕРМИЧЕСКИЙ РАЗРЕЗ Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 СИСТЕМЫ Tl-Pb-Bi-Gd-Te

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В работе приведены новые результаты по фазовым равновесиям в системе Tl-Pb-Bi-Gd-Te в концентрационной области Tl_9GdTe_6-Tl_9BiTe_6-Tl_4PbTe_3. На основании данных, полученных методами ДТА, РФА и измерений микротвердости, установлено, что система характеризуется образованием непрерывных рядов твердых растворов со структурой Tl_5Te_3.
Ключевые слова: *теллуриды таллия-свинца, теллуриды таллия-гадолиния, теллуридыталлия-висмута, фазовые равновесия, твердые растворы, кристаллическая структура.*

Tl-Pb-Bi-Gd-Te SİSTEMİNİN Tl_4PbTe_3-Tl_9GdTe_6-Tl_9BiTe_6 POLİTERMİK KƏSİYİ

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İşdə Tl-Pb-Bi-Gd-Te sisteminin Tl_9GdTe_6-Tl_9BiTe_6-Tl_4PbTe_3 politermik kəsiyi üzrə faza tarazlıqlarına aid yeni nəticələr təqdim olunur. DTA, RFA və mikrobərkliyin ölçülməsilə müəyyən edilib ki, sistemdə Tl_5Te_3tipli tetraqonal quruluşa malik fasiləsiz bərk məhlullar əmələ gəlir.
Açar sözlər: *tallium–qurğuşun telluridləri, tallium–qadolinium telluridləri,tallium–bismut telluridləri, faza tarazlıqları, bərk məhlullar, kristal quruluş.*