

Phase Equilibria in the YbTe-PbTe-Bi₂Te₃ System

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Authors' contributions

This work was carried out in collaboration between all authors. Author KDR managed the literature searches, synthesis of samples and wrote the first draft of the manuscript. Authors ZSA and AYZ carried out DTA XRD and SEM analyses. Authors SZI and KDR managed the DTA results and plot the phase diagrams. Author MBB managed the analyses of the study. All authors read and approved the final manuscript.

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ABSTRACT

The phase equilibria in the YbTe-PbTe-Bi₂Te₃ system were investigated by means of differential thermal analysis (DTA), X-ray diffraction (XRD) and scanning electron microscope with energy dispersive analysis (SEM-EDS) techniques. Isothermal sections at 300 and 800 K, the liquidus surface projection as well as some isopleth sections were experimentally plotted. The already reported ternary YbPbTe₂, YbBi₂Te₄ and YbBi₄Te₇ compounds were not confirmed. Wide solid solubility field of initial binary compounds was revealed and a solubility of YbTe in the ternary tetradymite-like layered compounds in the PbTe-Bi₂Te₃ system are found to be about 2 mol%.

Keywords: Phase diagram; liquidus surface; isothermal sections; ytterbium telluride; lead telluride; bismuth telluride; solid solutions.

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1. INTRODUCTION

Bismuth and lead tellurides and more complex phases based on them are outstanding materials for thermoelectricity and far-infrared (IR) optoelectronics. In particular, the LAST (Lead-Antimony-Silver-Tellurium) compounds exhibit a high ZT values e.g. as $PbBi_2Te_4$ and $PbBi_4Te_7$ [1-4]. In addition, binary and ternary tellurides of p^2 - and p^3 - elements have been attracted considerable attention as 3D topological insulators for realizing quantum topological transport [5-7].

One of the known ways to enhanced of functional properties of such materials is doping matrix phases by different impurities, e.g. RE. Taking this into consideration, we undertook a complete study of the series of quasi-ternary systems containing tellurides of some p^2 and p^3 elements with rare-earth elements [8-10].

The aim of present work is to determine phase equilibria in the YbTe-PbTe-Bi₂Te₃ system denoted (A) from now on.

The isopleths sections YbTe-PbBi₄Te₇ and YbTe-PbBi₂Te₄ of the system (A) are reported in [11]. It is found that the former one is partly quasi-binary (above 1000 K) and has a phase diagram with eutectic type. The latter one is found to be non-quasi-binary with respect to incongruent melting of $PbBi_2Te_4$, whereas is stable in the sub-solidus area. The mutual solid-solubility of the initial components does not exceed 2 mol% at room temperature.

Binary compounds of this system are very well studied. YbTe, PbTe and Bi₂Te₃ melt congruently at 2000 K, 1197 K and 858 K, respectively. YbTe and PbTe compounds crystallize in the NaCl type cubic crystal structure, space group $Fm-3m$ with the unit cell parameters $a=6.366$ Å, $Z=4$ and $a=6.4603$ Å, $Z=4$, respectively), while Bi₂Te₃ crystallizes in the tetradymite type rhombohedral structure (space group $R-3m$ with $a=4.38$ Å, $c=30.4$ Å [12-15].

The boundary sub-systems of the (A) are studied in numerous works. The results of investigations were done by different authors on PbTe-Bi₂Te₃ system till 1990 are summarized in [14] where the numbers and compositions are rather distinct from each other. A more detailed investigation of PbTe-Bi₂Te₃ system was carried by Karpinski and Shelimova [16,17]. According to this diagram, ternary compounds $PbBi_2Te_4$ and

$PbBi_4Te_7$ melt congruently at 856 K and 858K, respectively. In addition, they revealed new ternary compounds $Pb_2Bi_6Te_{11}$, $PbBi_6Te_{10}$ and $PbBi_8Te_{13}$ at first with mixed-layered structure similar to already known $PbBi_4Te_7$. These compounds are reported in the sub-solidus area, where primary crystallization field and melting character is still unknown. A phase relationship between PbTe and YbTe which have same crystal system and very close lattice parameters is studied in [18-20]. According to [18], the maximum solid-solubility of YbTe in PbTe is measured to be about 33 mol% at 770-800K. Aliev and co-authors reported a new ternary compound YbPbTe₂ melts congruently at 1350K and has rhombic structure, $a=4.76$, $b=9.64$, $c=11.28$ Å. The solid-solubility of PbTe in YbTe is measured to be about 35 mol%. A new variant of the phase diagram of PbTe-YbTe system is presented in [20] in which the authors reported the continuous high-temperature solid solution series between YbTe and PbTe. Much more interesting, they have found spinodal-type phase decomposition below ~1050 K. Mutual solubility YbTe and PbTe is found to be about 2 and 35 mol% at room temperature, respectively.

Phase equilibria in the YbTe-Bi₂Te₃ system are studied in [8,21,22]. According to [21], this system contains two ternary compounds, namely YbBi₂Te₄ and YbBi₄Te₇. The former decomposes by a peritectic reaction at 873 K, whereas the latter melts congruently at 918 K. Both compounds are reported to have a Th₃P₄ type cubic structure with the following lattice parameters: $a = 10.48$ Å and $a = 10.62$ Å, respectively. Existence of these compounds is not confirmed in [8], where the phase diagram of the system was reported as a eutectic type with limited solubility based starting compounds.

2. MATERIALS AND METHODS

Bismuth (99.999 mass%), lead (99.999 mass%), ytterbium (99.9 mass%) and tellurium (99.999 mass%) from Alfa Aesar were used as starting materials.

The binary Sb₂Te₃ and Bi₂Te₃ and also ternary $PbBi_2Te_4$, $PbBi_4Te_7$ compounds were synthesized by melting stoichiometric amounts of the pure components in sealed silica ampoules under vacuum ($\sim 10^{-2}$ Pa). In order to achieve complete equilibrium, incongruent-melting $PbBi_2Te_4$ further annealed for about 200 h at 800 K. The synthesis of YbTe was carried out in niobium tubes under the vacuum. First, the

furnace was heated to 800 K for 1h and then the temperature was increased to 1300 K for 10 h following cooled down to 800 K and held at this temperature for about 100 h to achieve a complete homogenization.

Alloys of the system (A) were prepared by melting the stoichiometric quantities of the pre-synthesized binary and ternary compounds in sealed niobium tubes under vacuum which placed in evacuated fused-silica jackets. The niobium tubes were used in order to avoid the interaction of ytterbium with silica. All samples were heated up to 1300 K and were then annealed at 800 K for about 500 h to achieve equilibrium of the samples. In most cases alloys were then quenched in cold water from 800K. Differential thermal analysis (DTA), X-ray powder diffraction (XRD) and SEM-EDS techniques were employed to check the purity of the synthesized starting compounds and analyze the samples.

DTA was performed using a NETZSCH 404 F1 Pegasus differential scanning calorimeter.

The measurement was performed between room temperature and ~1400 K with a heating and cooling rate of 10 K min⁻¹. Temperatures of thermal effects were taken mainly from the heating curves. But, in the some samples thermal effects were taken from cooling curves in order to determine the onset of crystallization. Thermal analysis measurements of the annealed and as-cast alloys were investigated in Al₂O₃ crucibles under a flowing pure Ar atmosphere. The apparatus was calibrated using high pure Sn, Zn, Sb, Ag, Cu as standard samples. The overall uncertainty of the determined phase transformation temperatures was estimated to be ±1 K. Mass of samples were equal 0,1 q.

X-ray powder diffraction (XRD) data were collected at room temperature in reflection mode using a Bruker D8 ADVANCE powder diffractometer equipped with a Cu-target tube and a diffracted beam graphite monochromator. A fixed aperture and divergence slit of 1 mm, a 0.1 mm monochromator slit and 0.6 mm detector slit were used. The specimen was scanned in the 2θ range of 10°–70°, the scan step was 0.02°, and the time of counting in every step was 45 s. Quartz was used as an external standard.

The microstructure and equilibrium compositions of the phases were determined by FEI Quanta™ 250 scanning electron microscope with Oxford

Instruments energy dispersive X-ray spectrometer.

3. RESULTS AND DISCUSSION

The combined analysis of all our experimental data and the results found in the literature on the phase equilibria in the boundary quasi-binary systems [8,17,20] and isopleths sections YbTe-PbBi₄Te₇ (PbBi₂Te₄) [11] enabled us to build a self-consistent phase diagram of the YbTe–PbTe–Bi₂Te₃ quasi-ternary system.

3.1 The Isothermal Sections

By comparing and analyzing the XRD patterns and SEM data of the equilibrated alloys and literature data the isothermal sections of the system (A) were constructed at 300 and 800 K (Fig. 1a and b). Phase composition of some alloys of sections PbTe-0.5[YbBi₂Te₄] and Bi₂Te₃-0.5[YbPbTe₂] at 300 and 800 K, measured by SEM and XRD are shown in Table 1.

The 800 K isothermal section (Fig. 1a) is characterized by a formation of limited solid solution areas based on PbTe(α₁), YbTe (α₂) and Bi₂Te₃ (β). The observed homogeneity field of α₁-phase is ranging from 10 to 35 mol% YbTe at room temperature along PbTe-YbTe system. The homogeneity range of α₁-phase along PbTe-Bi₂Te₃ system is ~10 mol% YbTe and continuously narrows with increasing YbTe content in alloys. In addition, it was found that solubility field based on YbTe also extends continuously from the YbTe-PbTe side (~10 mol%) toward YbTe-Bi₂Te₃ (~2 mol%). Homogeneity range of β-phase slightly extends from PbTe-Bi₂Te₃ side (~7 mol%) toward YbTe-Bi₂Te₃ (~12mol%). α₂-phase based on YbTe [23, 24] forms stable tie lines with all rest phases (α₁, β, PbBi₂Te₄ and PbBi₄Te₇). This leads to the formation in the system three-phase regions α₁+α₂+PbBi₂Te₄, α₂+PbBi₂Te₄+PbBi₄Te₇ and α₂+β+PbBi₄Te₇ (Fig.1a). Two-phase areas α₁+α₂, α₁+PbBi₂Te₄, α₂+β and β+PbBi₄Te₇ are observed in the system.

The isothermal section at 300 K is qualitatively similar to those at 800 K (Fig. 1b). But, it differentiates from those at 300 K only for narrowed homogeneity ranges of α₁, α₂ and β-phases which this fact could be clearly seen from XRD patterns of appropriate alloys (Fig. 2). As

shown in Fig. 2, alloy #1 (90 mol%PbTe+5 mol% YbTe+5 mol%Bi₂Te₃) is monophasic at 800K. The XRD pattern displays only diffraction lines corresponding to the α_1 -phase based on PbTe. Curiously, this alloy is found to be biphasic at 300 K. The XRD pattern displays diffraction lines

corresponding to hexagonal PbBi₂Te₄ together with diffractions lines of cubic α_1 -phase. Further confirmation of the XRD results comes from the SEM data. SEM images of alloys #1 annealed at 800K (Fig. 3a) and slow cooled (Fig. 3b) are shown in Fig. 3.

Table 1. Phase compositions of some alloys of YbTe-PbTe-Bi₂Te₃ system

Section	Composition	Phase composition	
	mol%PbTe	300 K	800 K
PbTe-0.5[YbBi ₂ Te ₄]	95	α_1	α_1
	90	α_1 +S ₁ (trace)	α_1
	85	α_1 +S ₁	α_1
	80	α_1 +S ₁	α_1 +S ₁
	60	α_1 +S ₁	α_1 +S ₁
	50	α_1 + α_2 +S ₁	α_1 + α_2 +S ₁
	33.3	α_2 +S ₁	α_2 +S ₁
	20	α_2 +S ₂	α_2 +S ₂
	10	α_2 +S ₅	α_2 + β +S ₂
	5	α_2 + β	α_2 + β +S ₂
	2	α_2 + β	α_2 + β
Bi ₂ Te ₃ -0.5[YbPbTe ₂]	95	β	β
	90	β + α_2 (trace)	β
	80	α_2 + β +S ₅	α_2 + β +S ₂
	70	α_2 +S ₅	α_2 + β +S ₂
	60	α_2 +S ₄	α_2 + β +S ₂
	50	α_2 +S ₂	α_2 +S ₂
	20	α_1 + α_2 +S ₁	α_1 + α_2 +S ₁
	10	α_1 + α_2 +S ₁	α_1 + α_2 +S ₁
	2	α_1 + α_2	α_1 + α_2

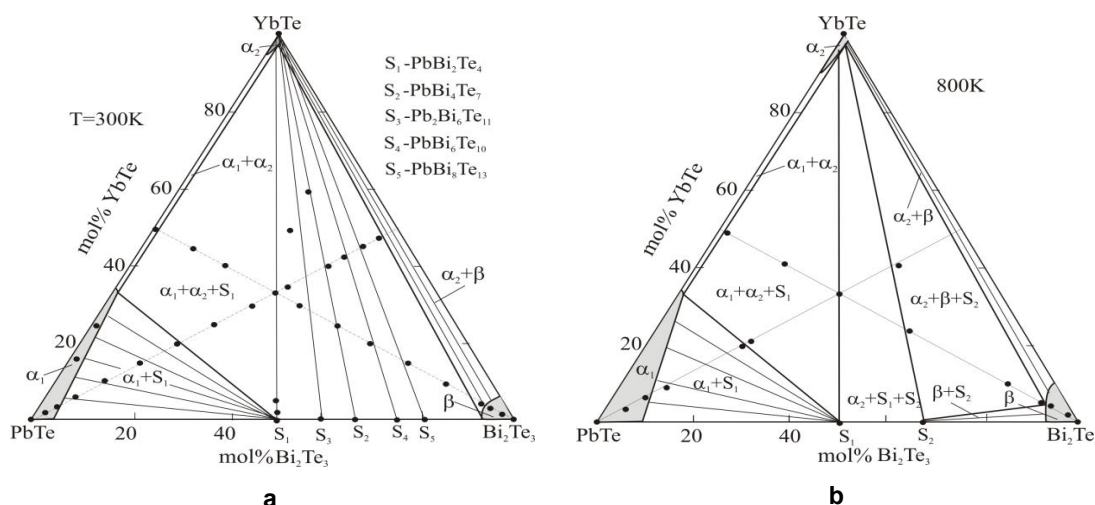


Fig. 1. The isothermal sections of system (A) at 800 K (a) and 300K (b). experimentally examined samples are shown with black circles

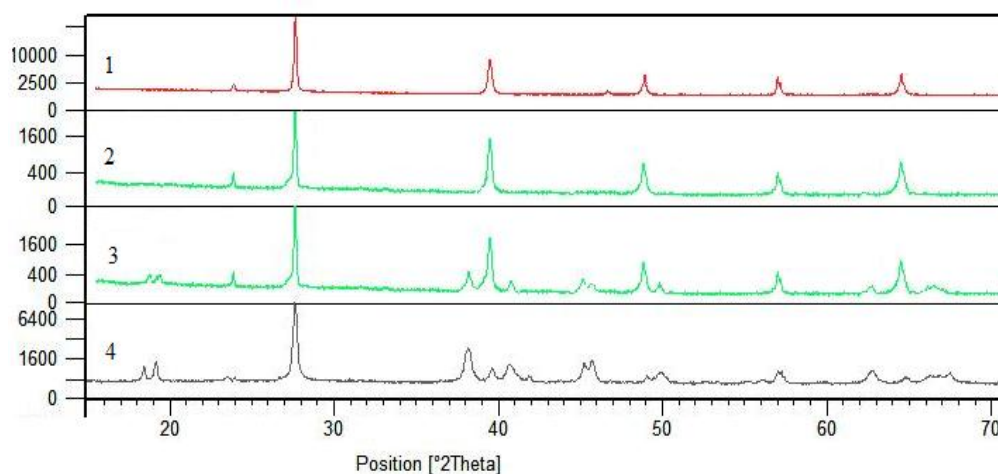


Fig. 2. XRD patterns of different alloys of system (A). 1, PbTe; 2, alloy #1 annealed at 800K; 3, alloy #1 annealed at 300K; 4-PbBi₂Te₄

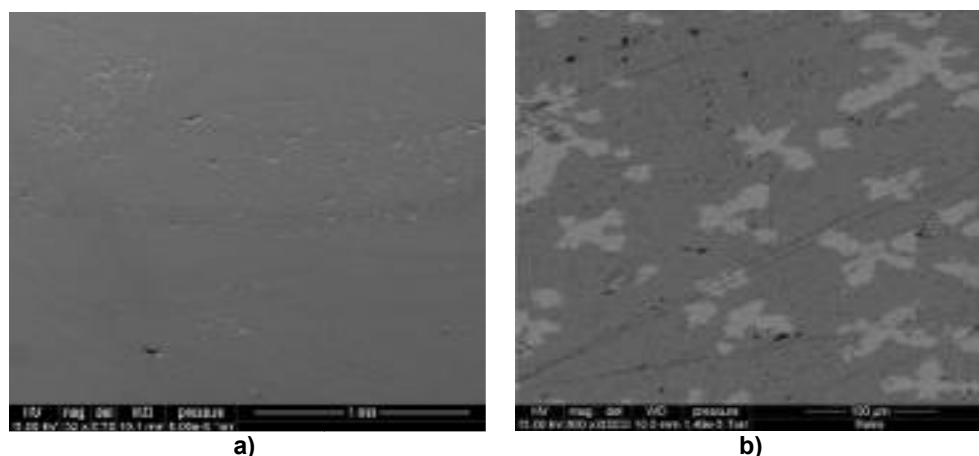


Fig. 3. SEM images of some alloys in the system A: (a) Alloy #1 annealed at 800 K; (b) Alloy #1 annealed at 300 K

In addition, the ternary compounds Pb₂Bi₆Te₁₁, PbBi₆Te₁₀ and PbBi₈Te₁₃ mentioned above [16,17] were also detected in Fig. 1b along with PbBi₂Te₄ and PbBi₄Te₇. These compounds cannot be obtained as a single phase due to the difficulty of achieving the equilibrium [16]. Therefore, the expected tie lines with α_2 -phase are shown with dashed lines.

Thus, obtained experimental data during present investigations were not confirmed the existence of the earlier reported ternary YbPbTe₂, YbBi₂Te₄ and YbBi₄Te₇ compounds [19,21,22].

3.2 The Liquidus Surface

The liquidus surface projection of the system A (Fig. 4) was constructed experimentally up to

1400 K and then by extrapolation. It consists of five fields corresponding to the primary crystallization of the α (below ~ 1050 K- α_1 and α_2), β -solid solutions and ternary compounds PbBi₂Te₄ and PbBi₄Te₇. α -solid solutions (below ~ 1050 K α_2 -phase based on YbTe) has the largest region of the primary crystallization as the most infusible phase of the system.

Table 2 summarizes the types and coordinates of invariant equilibria, including liquid compositions, whereas Table 3 compiles the types and temperature intervals for the monovariant equilibria.

The α -solid solutions along the boundary system YbTe-PbTe primarily crystallize at ~ 1050 K and

more up (Fig. 4). With decreasing temperature takes place the spinodal phase decomposition of α -solid solutions into α_1 -, α_2 - phases based on PbTe and YbTe, respectively [20]. Consequently, the KU curve appears as a result of this event. The critical point K (1050 K) reflects transformation $\alpha \rightarrow \alpha_1 + \alpha_2$ is the starting point for peritectic KU curve with monovariant equilibrium $L + \alpha_2 \leftrightarrow \alpha_1$ reaction which is separate primary crystallization field of α_1 and α_2 - phases.

Liquidus surface of the other phases is separated by the peritectic (pU) and eutectic curves UE₁, e₁e₁, E₁e₄E₂, e₂E₂ and e₃E₂ (Fig. 4, Table 3).

The intersection of the three fields corresponds to invariant four-phase transition (U) and eutectic (E₁ and E₂) equilibrium (Fig. 4, Table 2).

3.3 The Isopleths Sections

To demonstrate the process of crystallization and the character of heterogeneous equilibria two isopleth section of the system (A) were plotted.

3.3.1 The isopleth section PbTe-0.5 [YbBi₂Te₄] (Fig. 5a)

The liquidus surface of this section consists of only one curve corresponding to primary

crystallization of the α -phase. Within the composition area >80 mol% PbTe crystallization process finishes by a formation of monophasic area α (below ~1050 K – α_1). From 1050 K α -phase decomposes into α_1 - and α_2 -phases by a reaction $\alpha \leftrightarrow \alpha_1 + \alpha_2$. This lead for formation two-phase $L + \alpha_1$, $L + \alpha_2$ and three-phase $L + \alpha_1 + \alpha_2$ regions (Fig.5a)

Monovariant equilibrium $L + \alpha_1 \leftrightarrow \text{PbBi}_2\text{Te}_4$ is observed below the liquidus in the range of ~60-80mol% PbTe (Table 2). This peritectic reaction finishes with an excess of α_1 -phase and two-phase region $\alpha_1 + \text{PbBi}_2\text{Te}_4$ is formed in the sub-solidus.

In the composition range ~30-55 mol% PbTe takes place the invariant transition reaction (Table 1, the point U). Depending on the quantitative ratio of the phases involved in this reaction the three-phase $\alpha_1 + \alpha_2 + \text{PbBi}_2\text{Te}_4$ and $L + \alpha_2 + \text{PbBi}_2\text{Te}_4$ regions are forms below 853K. At the 33.3 mol% PbTe (the crossing point of this section with the YbTe-PbBi₂Te₄) transition reaction U completed by a completely disappearance of the two initial phases and formed tie lines $\alpha_2 + \text{PbBi}_2\text{Te}_4$.

Table 2. Invariant equilibria in the YbTe-PbTe-Bi₂Te₃ system

Point on Fig. 4	Equilibrium	Composition, mol%		T, K
		YbTe	Bi ₂ Te ₃	
D	$L \leftrightarrow \text{PbBi}_4\text{Te}_7$	-	66.7	860
p	$L + \alpha_2 \leftrightarrow \text{PbBi}_2\text{Te}_4$	-	61	856
e ₁	$L \leftrightarrow \text{PbBi}_2\text{Te}_4 + \text{PbBi}_4\text{Te}_7$	-	64	850
e ₂	$L \leftrightarrow \beta + \text{PbBi}_4\text{Te}_7$	-	90	848
e ₃	$L \leftrightarrow \alpha_2 + \beta$	15	85	850
e ₄	$L \leftrightarrow \alpha_2 + \text{PbBi}_4\text{Te}_7$	6	63	855
U	$L + \alpha_1 \leftrightarrow \alpha_2 + \text{PbBi}_2\text{Te}_4$	3	60	853
E ₁	$L \leftrightarrow \alpha_2 + \text{PbBi}_2\text{Te}_4 + \text{PbBi}_4\text{Te}_7$	3	63	845
E ₂	$L \leftrightarrow \alpha_2 + \beta + \text{PbBi}_4\text{Te}_7$	5	86	840

Table 3. Monovariant equilibria in the YbTe-PbTe-Bi₂Te₃ system

Curve on Fig. 4	Equilibrium	Temperature ranges, K
KU	$L + \alpha_2 \leftrightarrow \alpha_1$	1050-853
pU	$L + \alpha_1 \leftrightarrow \text{PbBi}_2\text{Te}_4$	856-853
UE ₁	$L \leftrightarrow \alpha_2 + \text{PbBi}_2\text{Te}_4$	853-845
e ₁ e ₁	$L \leftrightarrow \text{PbBi}_2\text{Te}_4 + \text{PbBi}_4\text{Te}_7$	850-845
E ₁ e ₄ E ₂	$L \leftrightarrow \alpha_2 + \text{PbBi}_4\text{Te}_7$	845-855-840
e ₂ E ₂	$L \leftrightarrow \beta + \text{PbBi}_4\text{Te}_7$	848-840
e ₃ E ₂	$L \leftrightarrow \alpha_2 + \beta$	850-840

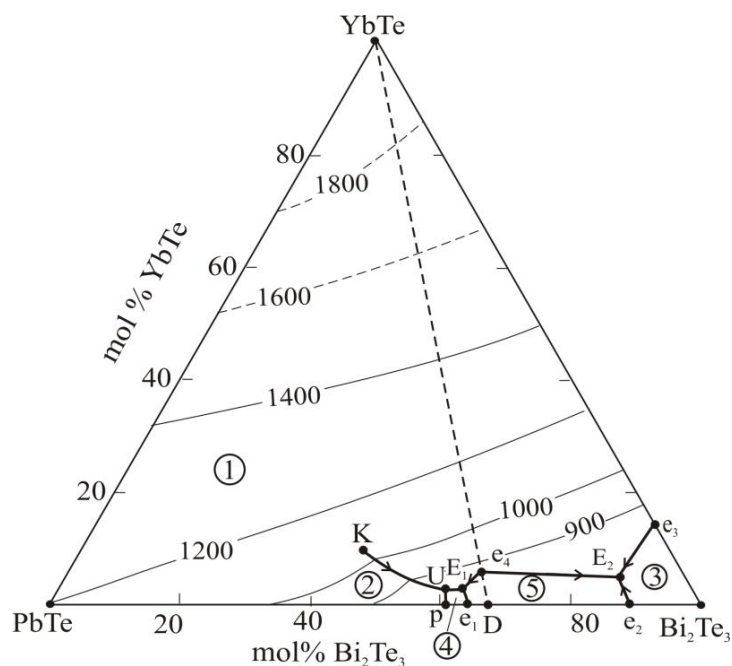


Fig. 4. The liquidus surface projection of the YbTe-PbTe-Bi₂Te₃ system. Primary crystallization fields of phases: 1-α(α₂); 2- α₁; 3-β; 4-PbBi₂Te₄; 5- PbBi₄Te₇

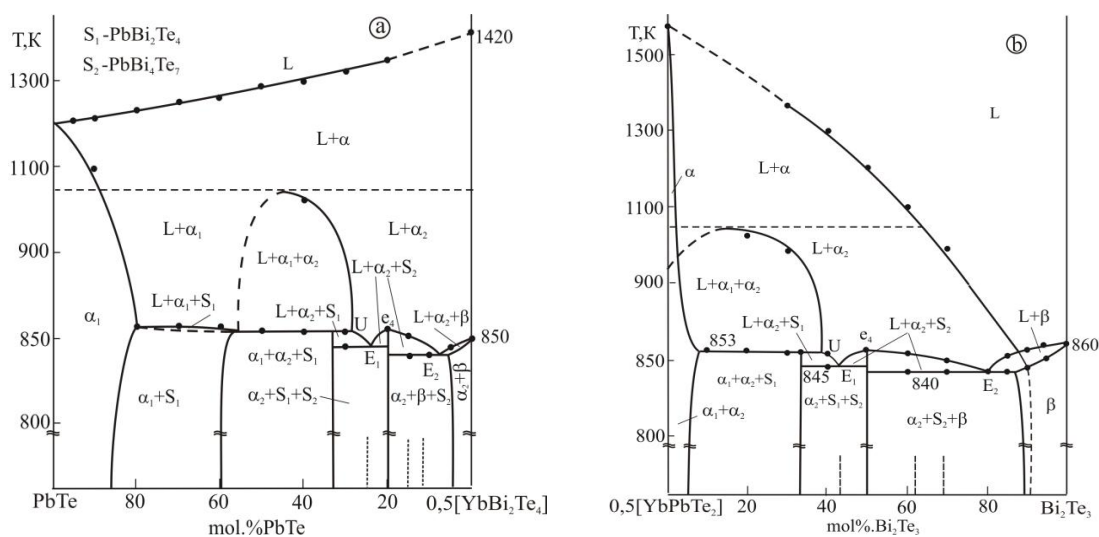


Fig. 5. The isopleths PbTe-0,5[YbBi₂Te₄] (a) and 0,5[YbBi₂Te₄]-Bi₂Te₃ (b) sections

Along the UE₁ curve the monovariant eutectic process occurs (Table 3). Horizontal at 845K (point E₁) reflects the invariant eutectic equilibrium $L \leftrightarrow \alpha_2 + \text{PbBi}_2\text{Te}_4 + \text{PbBi}_4\text{Te}_7$ which leads to the formation of three-phase region $\alpha_2 + \text{PbBi}_2\text{Te}_4 + \text{PbBi}_4\text{Te}_7$.

Curve E₁e₄E₂ corresponds to monovariant eutectic reaction $L \leftrightarrow \alpha + \text{PbBi}_4\text{Te}_7$, while horizontal at 840 K (point E₂) reflects the crystallization of ternary eutectic $\alpha_2 + \beta + \text{PbBi}_4\text{Te}_7$. In the composition range ~0-5 mol% PbTe crystallization is completed by the monovariant eutectic reaction $L \leftrightarrow \alpha_2 + \beta$ (Table 3, the curve

e_3E_2) and in sub-solidus two-phase region $\alpha_2+\beta$ is formed.

3.3.2 The isopleth section 0.5[YbBi₂Te₄]-Bi₂Te₃(Fig.5b)

This section is also crosses almost all phase regions of the YbTe-PbTe-Bi₂Te₃ system at sub-solidus area and primary crystallization fields of α - and β -phases. Below the liquidus surface of α -phase occurs its solid phase decomposition into to α_1 and α_2 which leads to formation of two-phase $\alpha_1+\alpha_2$ phase area. Horizontals at 853, 845 and 840 K reflects U, E₁ and E₂ invariant equilibria (Table. 2) whereas curves corresponds to monovariant UE₁ and E₁e₄E₂ equilibria which are mentioned above in detail. β -phase crystallizes from Bi₂Te₃-rich liquid (>90 mol%) where homogeneity range is achieve 8 mol% on current isopleths.

The expected tie lines of Pb₂Bi₆Te₁₁, PbBi₆Te₁₀ and PbBi₈Te₁₃ compounds with α_2 -phase were showed with dashed lines on Fig. 5.

Thus, considered isopleths sections reflect almost all in- and monovariant equilibria in the YbTe-PbTe-Bi₂Te₃ and allow following their sequence upon temperature. By comparing the Fig. 5a and b with the liquidus surface projection (Fig. 4) and isothermal section (Fig. 1a and b) it can be shown their compatibility and accordance of all parts of the phase diagram.

4. CONCLUSION

We have detailed investigated the phase equilibria in the YbTe-PbTe-Bi₂Te₃ system and shown that it is quasi-ternary plane of the quaternary system Yb-Pb-Bi-Te. Two isopleth and two isothermal sections at 300 and 800 K as well as the liquidus surface projection were constructed. Existence of the ternary compounds YbPbTe₂, YbBi₂Te₄ and YbBi₄Te₇ reported earlier were not confirmed. Wide area of solid solution based on initial binary compounds was revealed in this system. Solubility of YbTe in the ternary tetradymite-like compounds of system PbTe-Bi₂Te₃ is found to be less than 2 mol%. It was also concluded that, the decomposition of the solid solution in this system takes place below 1050 K.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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