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PHASE EQUILIBRIA OF THE PbBi_2Te_4 –" PbSb_2Te_4 " SECTION OF THE PbTe – Bi_2Te_3 – Sb_2Te_3 SYSTEM AND SOME PROPERTIES OF THE SOLID SOLUTIONS

A.I.Aghazade

M.Nagiyev Institute of Catalysis and Inorganic Chemistry, NAS of Azerbaijan

aytenagazade94@gmail.com

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PbTe – Bi_2Te_3 – Sb_2Te_3 system has been studied by DTA and X-ray diffraction methods along the PbBi_2Te_4 – " PbSb_2Te_4 " section. It was found that the compound of the composition PbSb_2Te_4 , specified in the literature does not exist. On the basis of the latter a wide (more than 50 mol%) area of solid solution (γ) was detected. By using obtained experimental results, a fragment of the solid-phase equilibrium diagram of the PbTe – Bi_2Te_3 – Sb_2Te_3 system was constructed and boundaries of the heterogeneous areas $\alpha+\gamma$, $\alpha+\beta$, $\alpha+\beta+\gamma$ (α - and β - are solid solutions based on PbTe and Sb_2Te_3 , respectively) have been determined.

Keywords: PbTe – Bi_2Te_3 – Sb_2Te_3 system, phase diagram, solid solutions, tetradymite-like structure, topological insulators.

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Introduction

Ternary tetradymite-like $\text{A}^{\text{IV}}\text{B}^{\text{V}}_2\text{Te}_4$, $\text{A}^{\text{IV}}\text{B}^{\text{V}}_4\text{Te}_7$, $\text{A}^{\text{IV}}\text{B}^{\text{V}}_6\text{Te}_{10}$ and other compounds formed in A^{IV} – B^{V} –Te systems (A^{IV} – Ge, Sn, Pb; B^{V} – Sb, Bi) are thermoelectric materials with low thermal conductivity [1–4]. Recent studies have shown that these compounds also have three-dimensional topological insulator properties [5–9] and are perspective for use in spintronics, quantum computing, medicine, and security systems [10–15].

Creating new multicomponent functional materials is based on the phase equilibria data of the appropriate systems [15–18]. Among the functional materials, solid solutions are of particular interest, due possibility to optimize their properties by changing their composition. The investigations of the systems consist of compounds with structural or formula analogs are expedient for obtaining solid solutions [19–23].

The PbTe – Bi_2Te_3 – Sb_2Te_3 system is interesting in terms of searching for solid solutions with $\text{Bi} \leftrightarrow \text{Sb}$ substitutional based on binary and ternary compounds with a tetradymite-like layered structure. The boundary systems of such concentration triangle have been investigated in several works [24–34].

The PbBi_2Te_4 formed in the PbTe – Bi_2Te_3 quasi-binary system melts incongruently at 864 K

[24]. This compound crystallizes in a hexagonal structure (*Sp.Gr.* $R\bar{3}m$) and has the following lattice parameters: $a=4.438(1)$ Å, $c=41.773(7)$ Å. Cristal lattice consists of seven-layered blocks (Te – Bi – Te – Pb – Te – Bi – Te) which are repeated along c axis and has Van der Waal's bonds between themselves [25].

A literature data about the PbTe – Sb_2Te_3 quasi-binary system is contradictory. According to [26] $\text{Pb}_2\text{Sb}_6\text{Te}_{11}$ compound with incongruently melting (855 K) is formed in this system. Authors [27, 28] found the layered structure PbSb_2Te_4 compound in the PbTe – Sb_2Te_3 system. But the latest studies [29–32] showed the absence of compound PbSb_2Te_4 , and the existence of $\text{Pb}_2\text{Sb}_6\text{Te}_{11}$ indicated in the [26]. Using high-temperature microstructural analysis method authors [29–32] obtained that, a $\text{Pb}_2\text{Sb}_6\text{Te}_{11}$ sample is a stable in a small temperature range (849–851 K) and is decomposed into $\text{PbTe} + \text{Sb}_2\text{Te}_3$ two-phased mixture in less than 849 K.

According to Roseboom's classification a phase diagram of the Bi_2Te_3 – Sb_2Te_3 system regards to the I type and is characterized by the formation of continuous solid solutions with layered tetradymite-like structure [33, 34].

Considering the above mentioned, we have undertaken the study a phase equilibria in the PbTe – Bi_2Te_3 – Sb_2Te_3 quasi-ternary system.

Here, the results of the investigations of the pointed system along the PbBi_2Te_4 – PbSb_2Te_4 section are presented.

Experimental part

As a rule, tetradymite-like ternary compounds melt incongruently [15, 24], therefore, for to prepare the alloys of PbTe – Bi_2Te_3 – Sb_2Te_3 system we used starting binary compounds which melt congruently. For this purpose, firstly initial compounds PbTe , Bi_2Te_3 , Sb_2Te_3 were synthesized from elementary components with a high degree of purity (99.999 % purity from Alfa Aesar) taken in stoichiometric ratio. The synthesis was carried out by co-melting of elemental components in a vacuum (10^{-2} Pa) in sealed quartz ampoules at the temperature by 30–35° higher than the melting point of the synthesized compound [35].

The individuality of the synthesized compounds was controlled by differential thermal analysis (DTA) and powder X-ray diffraction technique (XRD).

Identified compounds were used for the preparation of samples along the PbBi_2Te_4 – PbSb_2Te_4 section. Samples were heated at 1000 K in the furnace and then quenched in ice water. For achieving maximum equilibrium

state the samples were annealed at 730–750 K for 800 hours.

The obtained alloys were studied by DTA and XRD. DTA was carried out on a Linseis DSC400 differential scanning calorimeter using the Linseis TA V 2.3.1 software. XRD was performed on a Bruker D2 PHASER diffractometer, with $\text{CuK}_{\alpha 1}$ -radiation. PXRD patterns were indexed by using TOPAS 4.2 software.

Results and discussion

Annealed alloys with compositions 20, 30, 40, 50, 60, 80 mol% PbBi_2Te_4 of the PbBi_2Te_4 – PbSb_2Te_4 section were studied by the PXRD technique. X-ray diffraction patterns of some alloys are presented in Figure 1. As can be seen, alloys with compositions 50 and 80 mol% PbBi_2Te_4 have qualitatively similar diffraction patterns. Diffraction peaks of these alloys are slightly shifted relative to the diffraction peaks of the PbBi_2Te_4 compound that is associated with a change in the crystal lattice parameters during the formation of solid solutions.

Diffraction patterns of the samples containing 20 and 40 mol % PbBi_2Te_4 (Figures 2b, 2a) show their non-homogeneity. They were two- and three-phase mixtures, respectively.

Fig. 1. Powder XRD patterns of some alloys of the PbBi_2Te_4 – PbSb_2Te_4 section.

Fig. 2. Powder XRD patterns of the alloys containing 40 (a) and 20 (b) mol% PbBi_2Te_4 .

The diffraction peaks of α -phase (solid solution based on PbTe) coincide with diffraction lines for pure PbTe , while the peaks of the β -phase (solid solution based on Sb_2Te_3) slightly shifted relative to pure Sb_2Te_3 towards small angles. This is due to the formation of a continuous solid solution in the Bi_2Te_3 – Sb_2Te_3 system [33, 34].

Comparative analysis of diffraction patterns (Figures 1 and 2) with the literature data on PbTe – Bi_2Te_3 [24, 25], PbTe – Sb_2Te_3 [26, 29–32], Bi_2Te_3 – Sb_2Te_3 [33, 34] systems allowed us to construct a certain fragment the

solid-phase equilibria diagram of the PbTe – Bi_2Te_3 – Sb_2Te_3 quasi-ternary system. According to the phase diagram (Figure 3), a wide area (more than 50 mol%) of solid solutions (γ -phase) is formed in this system along the PbBi_2Te_4 – PbSb_2Te_4 section. At lower concentrations of PbBi_2Te_4 , the alloys are three-phase ($\alpha+\beta+\gamma$) and two-phase ($\alpha+\beta$).

In order to construct a complete scheme of phase equilibria in PbTe – Bi_2Te_3 – Sb_2Te_3 system, additional experimental researches are required.

Fig. 3. Fragment of the solid phase equilibria diagram of PbTe–Bi₂Te₃–Sb₂Te₃ system. The dots indicate the composition of the samples studied by X-ray.

Crystal lattice parameters of the solid solutions were determined based on the powder X-ray patterns using TOPAS 4.2 software (Table 1). Based on these data, of the lattice parameters dependence on composition was constructed (Figure 4). As seen from Figure 4, both lattice parameters are linear depend vs composition, and a breakpoint was observed at a limit concentration of the solid solution (~48 mol% PbBi₂Te₄).

According to the DTA and XRD results (Table 1, Figure 5), the phase diagram of the PbBi₂Te₄–"PbSb₂Te₄" polythermal section of the phase diagram of the PbTe–Bi₂Te₃–Sb₂Te₃ system was constructed (Figure 6).

As is seen in Figure 6 in the PbBi₂Te₄–"PbSb₂Te₄" section of the PbTe–Bi₂Te₃–Sb₂Te₃ system, the α-phase based on PbTe is primarily

crystallized from the liquid in the entire range of compositions. This means that this section is located entirely in the primary crystallization field of the α-phase. Crystallization below liquidus over a wide range of compositions (60–100 mol% PbBi₂Te₄) most likely occurs by the L + α ↔ γ peritectic reaction and finishes with the formation of a homogeneous γ-phase in the 55–100 mol% PbBi₂Te₄ composition. It is not possible to accurately determine the characters of the phase equilibria in the 0–45 mol% PbBi₂Te₄ composition area. Taking into account the decomposition of Pb₂Sb₆Te₁₁ compound into PbTe and Sb₂Te₃ lower 849 K [29–32] and as well as the obtained XRD results, we determined the phase regions in the subsolidus in the indicated composition region.

Table 1. DTA results and determined lattice parameters of PbSb_{2-x}Bi_xTe₄ alloys

Alloys' composition, mol. % from "PbSb ₂ Te ₄ "	Thermal effects, K	Phase composition	Crystal lattice parameters, Å	
			<i>a</i>	<i>c</i>
PbBi ₂ Te ₄	864, 940		4.4382(5)	41.775(6)
80	858, 943	γ	4.4183(4)	41.735(3)
60	852–857, 950	γ	4.3971(5)	41.6974(7)
50	850, 950	γ	4.3847(5)	41.6731(8)
40	848, 947	α+β+γ	*4.3843(5)	*41.6693(9)
30	848, 945	α+β	—	—
20	853, 937	α+β	—	—
"PbSb ₂ Te ₄ "	854–860, 927	α+β	—	—

Note: The * – indicated parameters belong to the γ-phase.

Fig. 4. Concentration dependence of lattice parameters for some alloys of the PbBi_2Te_4 – PbSb_2Te_4 section.

Fig. 5. DTA results for some alloys of the PbBi_2Te_4 – PbSb_2Te_4 section.

Fig. 6. T–x diagram of the PbBi_2Te_4 – PbSb_2Te_4 section of the PbTe – Bi_2Te_3 – Sb_2Te_3 system.

Conclusion

PbTe–Bi₂Te₃–Sb₂Te₃ system was studied along the PbBi₂Te₄–"PbSb₂Te₄" section. It has been shown that a large (~55 mol%) solid solution area with a layered tetradymite-like structure based on PbBi₂Te₄ is formed in this system. It is not possible to accurately determine the phase equilibria in the 0–45 mol% PbBi₂Te₄ composition area of the *T*–*x* diagram. However, it was established that in subsolidus, the samples from this composition field consist of heterogeneous mixtures of α + β (0–20 mol% PbBi₂Te₄) and α + β + γ (20–45 mol% PbBi₂Te₄). The obtained experimental results allowed us to construct a fragment of the solid-phase equilibria diagram of the PbTe–Bi₂Te₃–Sb₂Te₃ system at room temperature, and to determine the areas of homogeneity of the α -, β - and γ -phases, as well as to define the boundaries of the heterogeneous areas α + β , α + γ , and α + β + γ . The obtained γ - and β -solid solutions are of interest as topological insulator materials.

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PbTe – Bi_2Te_3 – Sb_2Te_3 SİSTEMİNİN PbBi_2Te_4 – PbSb_2Te_4 KƏSİYİ ÜZRƏ FAZA TARAZLIQLARI VƏ BƏRK MƏHLULLARIN BƏZİ XASSƏLƏRİ

A.İ. Ağazadə

DTA və RFA üsulları ilə PbTe – Bi_2Te_3 – Sb_2Te_3 sistemi PbBi_2Te_4 – PbSb_2Te_4 kəsiyi üzrə öyrənilmişdir. Müəyyən edilmişdir ki, ədəbiyyatda göstərilən PbSb_2Te_4 tərkibli birləşmə mövcud deyil, PbBi_2Te_4 birləşməsi əsasında geniş (50 mol%-dən artıq) bərk məhlul (γ) sahəsi mövcuddur. Alınmış təcrübə nəticələri əsasında PbTe – Bi_2Te_3 – Sb_2Te_3 sisteminin bərkfaza tarazlıqları diaqramının fragmenti qurulmuş, sistemdə $\alpha+\gamma$, $\alpha+\beta$, $\alpha+\beta+\gamma$ (α və β müvafiq olaraq PbTe və Sb_2Te_3 əsasında bərk məhlullardır) heterogen sahələrinin sərhədləri təyin edilmişdir.

Açar sözlər: PbTe – Bi_2Te_3 – Sb_2Te_3 sistemi, faza diaqramı, bərk məhlullar, tetradimitəbənzər quruluş, topooloji izolyatorlar.

ФАЗОВЫЕ РАВНОВЕСИЯ В СИСТЕМЕ PbTe – Bi_2Te_3 – Sb_2Te_3 ПО РАЗРЕЗУ PbBi_2Te_4 – PbSb_2Te_4 И НЕКОТОРЫЕ СВОЙСТВА ТВЕРДЫХ РАСТВОРОВ

А.И. Агазаде

Методами ДТА и РФА изучена система PbTe – Bi_2Te_3 – Sb_2Te_3 по разрезу PbBi_2Te_4 – PbSb_2Te_4 . Выявлено, что соединение с составом PbSb_2Te_4 , указанное в литературе не существует. На основе соединения PbBi_2Te_4 выявлена широкая область (более 50 мол %) твердых растворов (γ). На основе полученных результатов построен фрагмент диаграммы твердофазных равновесий системы PbTe – Bi_2Te_3 – Sb_2Te_3 , определены границы гетерогенных областей $\alpha+\gamma$, $\alpha+\beta$, $\alpha+\beta+\gamma$ (α - и β -твердые растворы на основе PbTe и Sb_2Te_3) соответственно.

Ключевые слова: система PbTe – Bi_2Te_3 – Sb_2Te_3 , фазовая диаграмма, твердые растворы, тетрадимитоподобная структура, топологические изоляторы.