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THERMODYNAMIC INVESTIGATION OF THE PbTe–AgSbTe₂ SYSTEM BY MEANS OF EMF METHOD

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2PbTe–AgSbTe₂ system was investigated by measuring the electromotive force of the concentration with respect to PbTe chains in the temperature range of 300–450 K. A wide region of PbTe-based solid solutions (20–100 mol% PbTe) was found in the title system. The partial thermodynamic functions of PbTe and lead in the alloys are calculated from the equations of the temperature dependences of the EMF. Standard thermodynamic functions of formation and standard entropies of solid (2PbTe)_x(AgSbTe₂)_{1-x} ($x=0.2, 0.4, 0.6, 0.8$) solids are calculated by integrating Gibbs-Duhem equation along the 2PbTe–AgSbTe₂ section using literature data on the relevant thermodynamic data for Ag₂Te, PbTe and Sb₂Te₃ compounds.

Keywords: PbTe–AgSbTe₂ system, thermodynamic properties, EMF method, solid solutions, silver-lead-antimony tellurides.

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Introduction

Tellurides of heavy metals, as well as complex phases and composites based on them, are promising functional materials for electronic engineering [1–4]. Recent studies have shown that many binary and complex tellurides with tetradimite-like layered structure are topological insulators and can be used in spintronics and quantum computing [5–7]. Some complex silver tellurides possess ionic conductivity over the Ag⁺ cation and can be used as electrochemical sensors, electrodes or electrolyte materials in electrochemical energy conversion devices – in solid-state batteries, displays etc. [8–10].

Among complex telluride phases the alloys of the LAST (Lead–Antimony–Silver–Tellurium) family with the general formula AgPb_mSbTe_{2+m} are of particular interest. These phases with the NaCl structure, which are formed over the PbTe–AgSbTe₂ section of the quasiternary Ag₂Te–PbTe–Sb₂Te₃ system, have a high thermoelectric figure of merit [3, 4]. In [11], it was shown that the compound of the AgSbTe₂ composition previously mentioned in the literature [12, 13] does not exist, and the cubic phase in the Ag₂Te–Sb₂Te₃ system has a slightly different composition (Ag₁₉Sb₂₉Te₅₂). According to [14], Ag₁₉Sb₂₉Te₅₂ decomposes by solid-phase reaction upon cooling and does not exist below 250 K. Thus, the results of [11, 14] cast doubt on the existence of continuous solid solutions in the PbTe–AgSbTe₂ system.

This work is devoted to the thermodynamic study of solid solutions in the PbTe–AgSbTe₂ system using the electromotive forces method (EMF). Previously, the phase equilibrium and thermodynamic properties of a number of similar systems using the EMF method were studied [15–18].

Experimental part

A thermodynamic study of the 2PbSe–AgSbTe₂ system was carried out using the EMF method with glycerol electrolyte. This variant of the EMF method is successfully used for the thermodynamic study of binary and more complex metallic semiconductor and other systems [19–26].

Concentration chains of the following type were constructed and their EMF was measured in the temperature range of 300–450 K:

Earlier in [25, 26], it was shown that at studying such systems, the chains with respect to the lead electrode are irreversible, i.e. the EMF values continuously decrease during the experiments.

Equilibrium alloys (2PbTe)_x(AgSbTe₂)_{1-x} ($x = 0.1, 0.15, 0.2, 0.4, 0.6, 0.8$) served as the right electrodes in the circuits of type (1), which were synthesized by fusing the elementary components in the required ratios into evacuated to $\sim 10^{-2}$ Pa and sealed quartz ampoules. In

the synthesis, simple substances from the EVOCHEM ADVANCED MATERIALS GMBH company (Germany) of high purity were used: silver in granules (Ag-00047; 99.999%), antimony in granules (Sb-00002; 99.999%), lead in granules (Pb-00005; 99.9995%), tellurium pieces (Te-00005; 99.9999%). In order to maximally approximate the alloys to the equilibrium state, the cast non-homogenized samples obtained by quenching the melts from 1100 K were ground into powder, thoroughly mixed, pressed into tablets weighing 0.3–0.5 g and annealed first at 750 K (500 hours), and then at 450 K (200 h). Synthesized alloys were identified by XRD, which showed that samples with compositions $x = 0.1$ and 0.15 are three-phase

and consist of a mixture of $\text{Ag}_2\text{Te} + \text{Sb}_2\text{Te}_3 + \beta$ (where β is solid solutions based on PbTe), and alloys with $x = 0.2$ – 0.8 are single-phase (β -phase). As an example, Figure 1 shows the powder X-ray diffraction patterns of two alloys from the homogeneity region of the β -phase.

To prepare electrodes the PbTe (left electrode) and annealed alloys $(2\text{PbTe})_x(\text{AgSbTe}_2)_{1-x}$ (right electrodes) were powdered and pressed onto molybdenum current leads in the form of tablets with a diameter of ~ 0.6 cm and a thickness of ~ 0.3 cm.

A solution of KCl in glycerol with the addition of PbCl_2 was used as the electrolyte. In order to prevent the presence of moisture and oxygen in the electrolyte anhydrous, chemically pure salts

Fig. 1. X-ray diffraction patterns for alloys $(2\text{PbTe})_{0.3}(\text{AgSbTe}_2)_{0.7}$ and $(2\text{PbTe})_{0.5}(\text{AgSbTe}_2)_{0.5}$.

were used, as well as glycerin was previously de-

The electrochemical cell described in [20] was assembled. EMF measurements were carried out in an inert atmosphere using a high-voltage digital voltmeter V7-91. Before starting the measurements, the electrochemical cell was kept at ~350 K for 40–60 h, after which the first equilibrium EMF values were obtained. Subsequent measurements were carried out every 3–4 hours after the establishment of a certain temperature. The EMF values, which, regardless of the direction of the temperature change did not differ from each other at a given temperature by more than 0.2 mV, were considered to be equilibrium.

Results and discussion

The EMF measurements of the chains of type (1) showed that the EMF values for samples $(2\text{PbTe})_x(\text{AgSbTe}_2)_{1-x}$ with compositions $x = 0.1, 0.15$ and 0.2 are the same, and with further increase of the concentration of PbTe the values of EMF continuously decrease (Figure 2). This indicates that in the system on the basis of

hydrated and outgassed by pumping at ~400 K.

PbTe there are produced up to 80 mol % solid solutions. Analysis of the temperature dependences of the EMF showed that for all samples they are linear. Therefore, the experimental data were processed by the least squares method in the approximation of the linear temperature dependence of the EMF. For this purpose, the "Microsoft Office Excel 2010" software was used.

The obtained linear equations are presented in Table 1 have the following appearance recommended in [19, 20]:

$$E = a + bT \pm t \left[(S_E^2 / n) + S_b^2 \cdot (T - \bar{T})^2 \right]^{1/2} \quad (2)$$

Where n is the number of pairs of values of E and T ; S_E and S_b are the dispersions of individual measurements of EMF and coefficient b , respectively; $\bar{T}$ is average absolute temperature, t is Student's test. With a confidence level of 95% and the number of experimental points $n \geq 20$ the Student's test is $t \leq 2$.

Fig. 2. Concentration dependence of the EMF of chains of type (1) at 298 K.

Table 1. Temperature dependences of the EMF of cells of type (1) for the $(2\text{PbTe})_x(\text{AgSbTe}_2)_{1-x}$ alloys in the 300÷450 K temperature range

Composition	$E, \text{ mV} = a + bT \pm t \cdot S_E(T)$
$(2\text{PbTe})_{0.2}(\text{AgSbTe}_2)_{0.8}$	$38.2 + 0.048T \pm 2 \left[\frac{1.5}{24} + 4.8 \cdot 10^{-5} (T - 363.3)^2 \right]^{1/2}$
$(2\text{PbTe})_{0.4}(\text{AgSbTe}_2)_{0.6}$	$23.6 + 0.027T \pm 2 \left[\frac{1.8}{24} + 5.4 \cdot 10^{-5} (T - 362.6)^2 \right]^{1/2}$
$(2\text{PbTe})_{0.6}(\text{AgSbTe}_2)_{0.4}$	$12.2 + 0.016T \pm 2 \left[\frac{1.1}{24} + 3.5 \cdot 10^{-5} (T - 363.6)^2 \right]^{1/2}$
$(2\text{PbTe})_{0.8}(\text{AgSbTe}_2)_{0.2}$	$5.8 + 0.009T \pm 2 \left[\frac{1.2}{24} + 3.7 \cdot 10^{-5} (T - 361.7)^2 \right]^{1/2}$

The partial molar functions of PbTe ($\Delta\bar{Z}_{\text{PbTe}}$) in alloys at 298.15 K were calculated from the data of Table 1 according to the following relations [19]:

$$\Delta\bar{G}_{\text{PbTe}} = -zFE \quad (3)$$

$$\Delta\bar{S}_{\text{PbTe}} = zF \left(\frac{\partial E}{\partial T} \right)_p = zFb \quad (4)$$

$$\Delta\bar{H}_{\text{PbTe}} = -zF \left[E + T \left(\frac{\partial E}{\partial T} \right)_p \right] = -zFa \quad (5)$$

and listed in Table 2. As can be seen from Figure 2, all these functions are continuous functions of the composition in the field $x \geq 0.2$.

The partial molar functions of PbTe are the difference between the partial molar values of lead in (2PbTe)_x(AgSbTe₂)_{1-x} solid solutions

($\Delta\bar{Z}_{\text{Pb}}$) and in pure PbTe:

$$\Delta\bar{Z}_{\text{PbTe}} = \Delta\bar{Z}_{\text{Pb}} - \Delta\bar{Z}'_{\text{Pb}} \quad (6)$$

где $Z \equiv G$ (or H).

PbTe is the only compound of the Pb–Te system and has an almost constant stoichiometric composition [27]. According to [26] in such cases:

$$\Delta\bar{Z}'_{\text{Pb}} = \Delta_f Z^0(\text{PbTe}) \quad (7)$$

Considering relations (6) and (7), the partial molar functions of lead in solid solutions (2PbTe)_x(AgSbTe₂)_{1-x} can be calculated from the relation:

$$\Delta\bar{Z}_{\text{Pb}} = \Delta\bar{Z}_{\text{PbTe}} + \Delta_f Z^0(\text{PbTe}) \quad (8)$$

The values obtained by the relation (8) are presented in Table 3.

Table 2. Relative partial thermodynamic functions of PbTe in the 2PbTe–AgSbTe₂ alloys at 298 K

Composition	$-\Delta\overline{G}_{\text{PbTe}}$	$-\Delta\overline{H}_{\text{PbTe}}$	$\Delta\overline{S}_{\text{PbTe}}$,
	kJ·mole ⁻¹		J·K ⁻¹ ·mole
(2PbTe) _{0.2} (AgSbTe ₂) _{0.8}	10.13±0.20	7.37±0.97	9.26±2.67
(2PbTe) _{0.4} (AgSbTe ₂) _{0.6}	6.11±0.21	4.56±1.03	5.21±2.84
(2PbTe) _{0.6} (AgSbTe ₂) _{0.4}	3.27±0.17	2.35±0.83	3.09±2.28
(2PbTe) _{0.8} (AgSbTe ₂) _{0.2}	1.64±0.18	1.12±0.85	1.74±2.34

Fig. 3. Concentration dependences of partial thermodynamic functions of PbTe in the 2PbTe–AgSbTe₂ solid solutions at 298 K.

Table 3. Relative partial thermodynamic functions of lead in the 2PbTe–AgSbTe₂ alloys at 298 K

Composition	$-\Delta\bar{G}_{\text{Pb}}$	$-\Delta\bar{H}_{\text{Pb}}$	$\Delta\bar{S}_{\text{Pb}},$
	kJ·mole ⁻¹		J·K ⁻¹ ·mole
(2PbTe) _{0.2} (AgSbTe ₂) _{0.8}	77.43±1.70	75.97±1.57	4.90±4.77
(2PbTe) _{0.4} (AgSbTe ₂) _{0.6}	73.41±1.71	73.16±1.63	0.84±4.94
(2PbTe) _{0.6} (AgSbTe ₂) _{0.4}	70.57±1.67	70.95±1.43	-1.74±4.38
(2PbTe) _{0.8} (AgSbTe ₂) _{0.2}	68.94±1.68	69.72±1.45	-2.62±4.44

The calculation of standard thermodynamic functions of the formation of a solid solution of the limiting composition (2PbTe)_{0.2}(AgSbTe₂)_{0.8}, which is in equilibrium with Ag₂Te and Sb₂Te₃, was performed using the following potential-forming reaction:

According to this reaction, the standard Gibbs free energy of formation and the enthalpy of formation (2PbTe)_{0.2}(AgSbTe₂)_{0.8} can be calculated by the relation:

$$\Delta_f Z^0[(2\text{PbTe})_{0.2}(\text{AgSbTe}_2)_{0.8}] = 0.4\Delta\bar{Z}_{\text{PbTe}} + \\ 0.4\Delta_f Z^0(\text{PbTe}) + 0.4\Delta_f Z^0(\text{Ag}_2\text{Te}) + \\ + 0.4\Delta_f Z^0(\text{Sb}_2\text{Te}_3) \quad (9),$$

and entropy can be calculated from the relation:

$$S^0[(2\text{PbTe})_{0.2}(\text{AgSbTe}_2)_{0.8}] = 0.4S_{\text{PbTe}} + \\ 0.4S^0(\text{PbTe}) + 0.4S^0(\text{Ag}_2\text{Te}) + \\ + 0.4S^0(\text{Sb}_2\text{Te}_3) \quad (10).$$

Standard integral thermodynamic functions of the formation of solid solutions with compositions x = 0.4; 0.6 and 0.8 were calculated by integrating the Gibbs-Duhem equation:

$$\Delta_f Z^0[(2\text{PbTe})_x(\text{AgSbTe}_2)_{1-x}] = \\ = 2(1-x) \int_{0.2}^x \frac{\Delta\bar{Z}_{\text{PbTe}}}{(1-x)^2} dx + \\ + x\Delta_f Z^0[(2\text{PbTe})_{0.2}(\text{AgSbTe}_2)_{0.8}] \quad (11)$$

Errors were found by the method of errors accumulation. The first term on the right side of equation (11) was determined by integrating by means of trapezoid method using the "Microsoft Office Excel 2010" software.

Literature data on corresponding standard integral thermodynamic functions of the Ag₂Te, PbTe and Sb₂Te₃ compounds in addition to own experimental results (Table 2), were used at calculations of equations (9) and (10) (Table 4).

The values of standard enthalpies of formation and entropies for PbTe and Sb₂Te₃ given in reference books [28, 29] practically coincide.

Table 4. Standard integral thermodynamic functions of the (2PbTe)_{1-x}(AgSbTe₂)_x solid solutions

Phase	$-\Delta_f G^0$ (298K)	$-\Delta_f H^0$ (298K)	S^0 (298K) ,
	kJ·mole ⁻¹		J·K ⁻¹ ·mole
PbTe [28, 29]	67.3±1.5	68.6±0.6	110.0±2.1
Sb ₂ Te ₃ [28, 29]	56.9±1.0	56.5±0.4	246.4±2.1
Ag ₂ Te [30]	40.2±0.3	35.0±0.5	152.0±2.0
(2PbTe) _{0.9} (AgSbTe ₂) _{0.1}	128.5±2.8	130.0±1.2	217.6±4.1
(2PbTe) _{0.8} (AgSbTe ₂) _{0.2}	119.0±2.5	121.9±1.1	218.4±3.9
	122.2±2.9*	122.3±2.5*	220.0±3.5*
(2PbTe) _{0.6} (AgSbTe ₂) _{0.4}	104.3±2.0	104.2±0.9	213.7±3.4
	106.4±2.3*	105.2±2.0*	216.1±3.5*
(2PbTe) _{0.4} (AgSbTe ₂) _{0.6}	86.3±1.6	85.4±0.9	208.2±3.5
	89.1±1.8*	86.8±1.5*	211.5±3.8*
(2PbTe) _{0.2} (AgSbTe ₂) _{0.8}	67.3±1.1	66.0±0.7	202.2±3.0
	69.8±1.2*	67.0±0.9*	204.9±3.7*

* these values are obtained by the EMF method with solid electrolyte [31]

The standard Gibbs energy of formation $\Delta_f G$ of these compounds was calculated using the data [28, 29]. For the Ag₂Te compound, the data of [30] obtained by the EMF method were used. The data for investigated solid solutions previously obtained by the EMF method with solid Ag⁺ conducting electrolyte in [31] are also listed in Table. As can be seen, the results obtained by two modifications of the EMF method are in good agreement.

Conclusion

The formation of a wide (up to 80 mol%) region of solid solutions based on PbTe, of great practical interest as thermoelectric materials with anomalously low thermal conductivity in the 2PbTe–"AgSbTe₂" system was confirmed by measuring of EMF of the concentration with respect to the PbTe electrode chains. A new mutually agreed complex of data on standard partial and integral thermodynamic functions of solid solutions (2PbTe)_x(AgSbTe₂)_{1-x} ($x=0.2, 0.4, 0.6, 0.8$) was obtained, which is in good agreement with the results obtained earlier by EMF method with solid Ag⁺ conductive electrolyte.

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PbTe–AgSbTe₂ SİSTEMİNİN EHQ ÜSULU İLƏ TERMODİNAMİK TƏDQIQI

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$2PbTe-AgSbTe_2$ sistemi PbTe-də nəzərən qatılıq dövrələrinin EHQ-nin ölçülməsilə 300–450 K temperatur intervalında tədqiq edilmişdir. Sistemdə PbTe əsasında geniş (20–100 mol% PbTe) bərk məhlul sahəsi aşkar olunmuşdur. EHQ-ni temperaturdan asılılıq tənlikləri əsasında xəlilələrdə qurğuşun telluridin parsial termodinamik funksiyaları hesablanmışdır. $(2PbTe)_x(AgSbTe_2)_{1-x}$ ($x=0.2, 0.4, 0.6, 0.8$) tərkibli bərk məhlulların standart integral termodinamik funksiyaları Gibbs-Dühem tənliyinin $2PbTe-AgSbTe_2$ kəsiyi üzrə inteqrallanması ilə və Ag_2Te , PbTe və Sb_2Te_3 birləşmələri üçün müvafiq ədəbiyyat məlumatları istifadə etməklə hesablanmışdır.

Acar sözlər: $2PbTe-AgSbTe_2$ sistemi, termodinamik xassələr, EHQ üsulu, bərk məhlullar, gümüş-qurğuşun-stibium telluridləri.

ТЕРМОДИНАМИЧЕСКОЕ ИССЛЕДОВАНИЕ СИСТЕМЫ PbTe–AgSbTe₂ МЕТОДОМ ЭДС

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В работе представлены результаты исследования системы $2PbTe-AgSbTe_2$ измерением ЭДС концентрационных относительно PbTe цепей в интервале температур 300–450 K. Показано образование в системе широкой (20–100 мол% PbTe) области твердых растворов на основе PbTe. Из уравнений температурных зависимостей ЭДС вычислены парциальные термодинамические функции PbTe и свинца в сплавах. Стандартные термодинамические функции образования и стандартные энтропии твердых растворов $(2PbTe)_x(AgSbTe_2)_{1-x}$ ($x=0.2, 0.4, 0.6, 0.8$) рассчитаны интегрированием уравнения Гиббса-Дюгема по разрезу $2PbTe-AgSbTe_2$ с использованием литературных сведений по соответствующим термодинамическим данным для соединений Ag_2Te , PbTe и Sb_2Te_3 .

Ключевые слова: система $PbTe-AgSbTe_2$, термодинамические свойства, метод ЭДС, твердые растворы, теллуриды серебра-свинца-сурьмы.