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Phase diagram of the Sb-Se-I system and thermodynamic properties of SbSeI

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1. Introduction

Chalcogen-halides of arsenic, antimony and bismuth are regarded as potential functional materials. Quite a number of their representatives display promising properties as low-gap semiconductors, ferroelectrics, piezoelectric, thermoelectrics, photoconductors, and piezoelastics. In particular, SbSI is known as a compound combining properties of a ferroelectric and semiconductor. Several other compounds with the same structure type, though having less pronounces functional properties, are useful as doping agents in various attempts to enhance the functional properties in terms of the Curie temperature and dielectric permittivity [1,3,6].

Most of the chalcogen-halides of arsenic, antimony and bismuth have the 1:1:1 stoichiometry and belong to just three structure types, one of which is known as the SbSI type [1,2]. SbSeI belongs to the 1:1:1 compounds and crystallizes in the SbSI structure type (Fig. 1), space group *Pnma*, with the unit cell parameters a = 8.6862(9), b = 10.3927(9), c = 4.1452(3) Å [4]. In its crystal structure, antimony and selenium form double chains spreading along the "*c*" direction of the unit cell. Within these chains, the Sb–Se bonds range from 2.60 to 2.79 Å. Iodine atoms separate the chains, with the Sb–I distances being 3.15 Å. Taking into account the structural features and the values of the interatomic distances, the semi-ionic description (SbSe)⁺I⁻ is frequently applied to this compound, similar to other phases of the SbSI structure type [1].

ABSTRACT

The Sb–Se–I system was investigated by using the DTA and XRD analyses and EMF measurements with an antimony electrode. The *T*–*x* diagram of the binary Sb–I system was accurately redefined. A number of polythermal sections and the projection of the liquidus surface were constructed. The fields of the primary crystallization, as well as the types and coordinates of non- and monovariant equilibria were determined. It is shown that the quasi-binary sections Sb₂Se₃–Sbl₃, Sb–SbSeI, Sbl₃–Se, and SbSeI–Se triangulate the Sb–Se–I system, leading to five independent subsystems. A broad area of immiscibility, that overlaps a certain part of the antimony primary crystallization field, was found. From the EMF measurements, the partial molar functions of antimony ($\Delta \tilde{G}$, $\Delta \tilde{H}$, $\Delta \tilde{S}$) as well as standard integral thermodynamic functions of SbSeI were calculated. The latter were found to have the following values: $\Delta G_{f,298}^0 = -80.12 \pm 1.81 \text{ kJ/mol}$; $\Delta H_{f,298}^0 = -77.3 \pm 1.8 \text{ kJ/mol}$; $S_{298}^0 = 155.2 \pm 9.5 \text{ J/(mol K)}$.

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Selenium atoms can be partially replaced by sulfur atoms in the SbSe double chains thus modifying the properties [5].

The ternary Sb–Se–I system was investigated on the basis of various polythermal sections, among which the quasi-binary Sb₂Se₃–SbI₃ section was the most studied [6–10]. According to Ref. [3] this section includes the only compound SbSeI that melts congruently at 721 K, whereas the eutectic compositions were found to be 69 and 3 mol% Sb₂S₃ at temperatures 703 and 428 K, respectively. Other authors [7,8] report slightly different data on the melting points and eutectic compositions. The melting points vary from one study to another, covering the range of 675–725 K; similar disagreement can be found for the eutectic (443 K) is reported for the SbI₃ side [9]. The SbI₃–Se and SbSeI–Se quasi-binary sections are shown to be of eutectic type. The eutectic compositions have melting point of 433 and 428, respectively, and have 62 and 74 mol% Se [10].

The data presented above already show that the data on the phase equilibria in the Sb–Se–I system are far from being complete and self-consistent. In this work, we report the results of the complex investigation of phase equilibria in the Sb–Se–I system and of thermodynamic properties of the only ternary compound in this system, SbSeI. Similar reports on the investigation of the Sb(Bi)–Te–I systems have been already presented in the literature [11–14].

2. Experimental

 Sb_2Se_3,Sbl_3 and SbSel were synthesized from the elements of high purity grade in evacuated ($\sim\!10^{-2}$ Pa) silica ampoules according to the following schemes. Sb_2S_3 was prepared by a one-step annealing of the stoichiometric mixture of the elements

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Fig. 1. Projections of the crystal structure of SbSel on [001] (top) and [100] (bottom).

at 950 K, which is above the melting points of both Sb (900 K) and Sb₂S₃ (863 K), followed by cooling with the furnace. For the preparation of Sbl₃ and SbSel, the specially designed method was used taking into account high volatility of iodine. The synthesis was performed in the inclined three-zone furnace, with two hot zones kept at 470 and 750 K, whereas the temperature of the cold zone was about 400 K. After the main portion of iodine reacted at 470 K, the ampoule was relocated such that the product melted at 750 K. The melt was stirred at this temperature and then cooled with the furnace. The quality of the products was checked using differential thermal analysis (DTA) using the NTR-72 pyrometer equipped with two chromel-alumel thermocouples and X-ray powder diffraction (XRD) analysis (DRON-2, Cu-K α).

The alloys in the Sb–Se–I system for the investigation of the properties were synthesized from the pre-prepared compounds Sb_2Se_3 , Sbl_3 and SbSeI. A total load for each sample was 0.5 g. After determining the solidus temperatures, the sintering temperatures were adjusted to be 20–30K below the solidus. Thus, the samples were annealed for 800–1000 h at 670 K within the Sb–Sb_2Se_3–SbSeI field and at 420 K within the Sb–Sbl₃–SbSeI and Sbl₃–Sb₂Se₃–Se field.

For the electro-motive force (EMF) measurements, the following concentration chains were used:

$$(-) Sb (solid)/glycerin + KI + SbI_3/(Sb-Se-I) (solid) (+)$$
(1)

In the chains of type (1), metallic antimony was the left (negative) electrode, while equilibrium alloys of the Sb–Se–I system were exploited as right (positive) electrodes. The saturated glycerin solution of KI with the addition of 0.1 mass% of Sb₃ was used as the electrolyte. The details of the methods for the preparation of electrolytes and electrodes were previously described in our papers devoted to the thermodynamic study of the Sb(Bi)–Te–I systems [11–13]. EMF was measured by the compensation method in the temperature range of 305–400 K, with the accuracy of the temperature control being 0.2 K. In each experiment the first reading was performed after approximately 30 h after the start of the experiment, and then the data were read 4–5 h after reaching the desired temperature, which ensures the achievement of equilibrium.

3. Results and discussion

The inspection of the DTA data for the Sb–SbI₃–SbSeI compositional field showed that our results disagree with those reported in Ref. [15]. Consequently, we undertook the complete study of this



Fig. 2. Phase diagram of the Sb-I system.

part of the Sb–Se–I system. That enabled us to build its T–x diagram (Fig. 2), which shows substantial differences with that reported previously, especially in the Sb–SbI₃ binary subsystem. According to our data the monotectic temperature is 893 K, and the immiscibility area ranges from 5 to 70 at.% of I, which is very far from the data reported in Ref. [15] (442 K and 28–74 at.%, respectively).

The combined analysis of all our experimental data and the results found in the literature on the equilibria in the Sb–Se and Se–I system enabled us to construct the self-consistent diagram of the phase equilibria in the Sb–Se–I system.

3.1. The section Sb₂Se₃-SbI₃

The DTA results for the selected alloys in this system showed the coincidence with the results reported in the literature [7]. SbSeI melts congruently at 725 K, its eutectic crystallizes at 58 mol.% Sb₂Se₃ at 716 K, and the eutetic with SbI₃ is degenerate (Fig. 3a). The concentration dependence of EMF of the chains (1) is expressed by two horizontal lines (Fig. 3b) such that the neglectible homogeniety range is confirmed for SbSeI.

3.2. The quasi-binary section Sb_{0.25}I_{0.75}-Se

The quasi-binary section $Sb_{0.25}I_{0.75}$ -Se is of the simple eutectic type. The eutectic composition lies at 20 at.% Se and 435 K, which slightly disagrees from the data reported in Ref. [10].

3.3. The polythermal sections Sb-[Se_{0.5}I_{0.5}] and [Sb_{0.5}I_{0.5}]-Se

The polythermal sections $Sb-[Se_{0.5}I_{0.5}]$ and $[Sb_{0.5}I_{0.5}]-Se$ (Figs. 4 and 5) that pass the stoichiometric composition of SbSeI are the partially quasi-binary sections, in which one of the components (expressed in square brackets) which is in fact of heterogeneous compositions.

Sb–SbSeI is the quasi-binary part of the Sb– $[Se_{0.5}I_{0.5}]$ section. It is characterized by the monotectic ($m_3m'_3$, 865 K) and eutectic (e_8 , 705 K) equilibria. In the compositional range SbSeI– $[Se_{0.5}I_{0.5}]$ this section intersects the SbI₃–SbSeI–Se and SbI₃–Se–I triangles, which are the subsystems of the Sb–Se–I system. The comparative analy-



Fig. 3. T-x [7] (a) and E-x (b) diagrams of the SbI₃-Sb₂Se₃ system. Our data are shown with black dots.



Fig. 4. Polythermal section Sb–[Se_{0.5}I_{0.5}] of the Sb–Se–I phase diagram.



Fig. 5. Polythermal section [Sb_{0.5}I_{0.5}]-Se of the Sb-Se-I phase diagram.

sis of Fig. 4 and Table 1 leads to the conclusion that the liquidus of this area in composed of the primary crystallization curves of SbSeI and Se. The horizontal line at 465 K corresponds to the join crystallization of these two phases, while those at 432 and 325 K are the temperatures of the ternary eutectics in the respective subsystems.

The SbSel–Se part of the Sb_{0.5}I_{0.5}–Se section exhibits the eutectic type of the *T*–*x* diagram. The eutectic contains approximately 98 at.% Sb and crystallizes at 492 K, which is far from the data published in Ref. [10]. In the Sb_{0.5}I_{0.5}–SbSel part this section crosses the Sb–SbI₃–SbSel subsystem. The phase diagram explicitly shows the existence of the immiscibility area between 0 and 25 at.% Sb.

Table 1

Nonvariant equilibria in the Sb-Se-I system.

Point on Fig. 7	Equilibrium	Composition, at.%		Т, К
		Se	I	
D_1	$L \leftrightarrow Sb_2Se_3$	60	-	865
D_2	$L \leftrightarrow SbI_3$	-	75	445
D ₃	L ↔ SbSeI	33.3	33.3	725
<i>e</i> ₁	$L \leftrightarrow Sb + Sb_2Se_3$	50	-	814
e ₂ *	$L \leftrightarrow Se + Sb_2Se_3$	>99	-	493
$e_3^{\tilde{*}}$	$L \leftrightarrow SbI_3 + Sb$	-	~ 74	443
e4	$L \leftrightarrow I_2 + SbI_3$	-	88	353
e ₅	$L \leftrightarrow Se + I_2$	50	50	330
<i>e</i> ₆	$L \leftrightarrow Sb_2Se_3 + SbSeI$	38	28	716
e [*] ₇	$L \leftrightarrow SbSeI + SbI_3$		73	443
e ₈	L ↔ Sb + SbSeI	30	30	705
e_{q}^{*}	L ↔ SbSeI + Se	>99		492
e ₁₀	$L \leftrightarrow SbI_3 + Se$	20	60	435
E_1	$L \leftrightarrow Sb + Sb_2Se_3 + SbSeI$	32	28	698
E_2^*	$L \leftrightarrow Sb_2Se_3 + SbSeI + Se$	>98		488
$E_3^{\tilde{*}}$	$L \leftrightarrow Sb + SbSeI + SbI_3$		~73	440
E_4	$L \leftrightarrow SbSeI + SbI_3 + Se$	19	59	432
E ₅	$L \leftrightarrow SbI_3 + Se + I_2$	46	52	325
$m_1(m'_1)$	$L_1 \leftrightarrow L_2 + Sb$	16(43)	-	853
$m_2(m'_2)$	$L_1 \leftrightarrow L_2 + Sb$	-	5(70)	895
$m_3(m_3^{7})$	$L_1 \leftrightarrow L_2 + Sb$	5(28)	5(28)	865

3.4. The polythermal section Sb_{0.4}Se_{0.6}-I

The polythermal section $Sb_{0,4}Se_{0,6}$ –I that crosses three subsystems is shown in Fig. 6. It enables distinguishing primary crystallization fields of Sb_2Se_3 , SbSeI, SbI_3 , Se and I_2 . Also it helps to precise the positions of the eutectic curves.

3.5. The liquidus surface of the Sb-Se-I system

The liquidus surface of the Sb–Se–I system (Fig. 7) consists of six fields corresponding to primary crystallization of the elemental components and compounds Sb₂Se₃, Sbl₃ and SbSeI. The system exhibits the broad immiscibility region $(m_1m_3m_2m'_2m'_3m'_1)$, which lies within the field of primary crystallization of antimony and overlaps some 90% of its area.

The quasi-binary sections (dashed lines in Fig. 7) triangulate the Sb–Se–I triangle forming five independent subsystems. They

Table 2
Monovariant equilibria in the Sb-Se-I system.

Curve on Fig. 7	Equilibrium	Temperature range, K
e_1E_1	$L \leftrightarrow Sb + Sb_2Se_3$	814-698
e_6E_1	$L \leftrightarrow Sb_2Se_3 + SbSeI$	716-698
e_6E_2	$L \leftrightarrow Sb_2Se_3 + SbSeI$	716-488
e_8E_1	$L \leftrightarrow Sb + SbSeI$	705–698
e ₈ E ₃	$L \leftrightarrow Sb + SbSeI$	705–440
e_4E_5	$L \leftrightarrow SbI_3 + I_2$	353-325
e_5E_5	$L \leftrightarrow Se + I_2$	330-325
$e_{10}E_5$	$L \leftrightarrow SbI_3 + Se$	435-325
$e_7 E_4$	$L \leftrightarrow SbSeI + SbI_3$	443-432
e_9E_4	$L \leftrightarrow SbSeI + Se$	492-432
$*e_{10}E_4$	$L \leftrightarrow SbI_3 + Se$	435-432
$*e_{2}E_{2}$	$L \leftrightarrow Sb_2Se_3 + Se$	493-488
$*e_9E_2$	$L \leftrightarrow SbSeI + Se$	492-488
*e ₃ E ₃	$L \leftrightarrow Sb + SbI_3$	443-440
*e7E3	$L \leftrightarrow SbSeI + SbI_3$	443-440
$m_3 m_1 (m'_3 m'_1)$	$L_1 \leftrightarrow L_2 + Sb$	865-853
$m_2 m_3 (m_2 m_3)$	$L_1 \leftrightarrow L_2 + Sb$	895-865



Fig. 6. Polythermal section Sb_{0.4}Se_{0.6}-I of the Sb-Se-I phase diagram.

are Sb–Sb₂Se₃–SbSeI (I), Sb–SbI₃–SbSeI (II), SbSeI–SbI₃–Se (III), Sb₂Se₃–SbSeI–Se (IV), and SbI₃–Se–I₂ (V). The subsystems (I) and (II) are characterized by monotectic and eutectic equilibria. The other subsystems belong to the ternary eutectic type. The E_2 and E_3 eutectics are degenerate at the SbI₃ side.

Table 1 summarizes the types and coordinates of nonvariant equilibria, including binary border systems, whereas Table 2 compiles types and temperature intervals for monovariant equilibria, with the asterisk marking the degenerate equilibria.



Fig. 7. Projection of the liquidus surface of the Sb–Se–I system. Primary crystallization fields are shown: 1, Sb; 2, Sb₂Se₃; 3, SbSeI; 4, Se; 5, SbI₃; 6, I₂. Quasi-binary sections are shown with dashed lines.

Table 3

Temperature dependencies of the EMF for the chains of type (1).

Phase area	$E, mV = a + bT \pm 2S_E()$
SbI ₃ + SbSeI + Se	$E = 227, 15 + 0,056 \pm 2 \left[\frac{0.54}{26} + 3,7 \times 10^{-5} (T - 350,96)^2 \right]^{1/2}$
Sb ₂ Se ₃ + SbSeI +	$SE = 236, 70 - 0,036 \pm 2 \left[\frac{1.81}{26} + 1, 4 \times 10^{-4} (T - 342, 68)^2 \right]^{1/2}$

Table 4

Relative partial thermodynamic functions of antimony in the alloys of the Sb–Se–I system at 298 K.

Phase area	$-\Delta \bar{G}_{\rm Sb}$	$-\Delta \bar{H}_{\rm Sb}$	$\Delta \bar{S}_{Sb}$
	kJ/mol		J/(mol K)
SbI ₃ + SbSeI + Se Sb ₂ Se ₃ + SbSeI + Se	$\begin{array}{c} 70.58 \pm 0.21 \\ 65.41 \pm 0.33 \end{array}$	$\begin{array}{c} 65.75 \pm 1.23 \\ 68.52 \pm 2.34 \end{array}$	$\begin{array}{c} 16.2 \pm 3.5 \\ -10.4 \pm 6.9 \end{array}$

Table 5

Standard integral thermodynamic functions of compounds in the Sb-Se-I system.

Compounds	$-\Delta G_{298}^{0}$	$-\Delta H_{298}^{0}$	S ⁰ ₂₉₈
	kJ/mol		J/mol K
SbI ₃ [19]	99.2 ± 5.0	100.4 ± 2.8	215.5 ± 1.7
Sb ₂ Se ₃	$\begin{array}{c} 130.8 \pm 0.7 \\ 132.4 \pm 8.0 \ [14] \end{array}$	$\begin{array}{c} 137.0 \pm 4.7 \\ 134.0 \pm 6.0 \ [14] \\ 127.7 \pm 1.0 \ [20] \end{array}$	$\begin{array}{c} 208 \pm 14 \\ 212.1 \pm 4.2 \ [19] \end{array}$
SbSeI	80.12 ± 1.81 -	$\begin{array}{c} 77.3 \pm 1.8 \\ 93 \pm 12 \ \textbf{[20]} \end{array}$	$\begin{array}{c} 155.2 \pm 9.5 \\ 130 \pm 7 \ [20] \end{array}$

3.6. Thermodynamic functions

The EMF measurement results for the chains of type (1) not only allowed confirming the correctness of all drawn solid-state equilibria but also served as the basis for the calculation of the thermodynamic functions for SbSeI.

The analysis showed the linearity of the EMF dependencies upon temperature for various alloys belonging to the heterogeneous subsystems SbI₃–SbSeI–Se and Sb₂Se₃–SbSeI–Se. Accordingly, the linear least-square treatment of the data was performed [16] and the results were expressed according to the literature recommendations [17] as

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

where *n* is the number of pairs of *E* and *T* values; *S*_{*E*} and *S*_{*b*} are the error variances of the EMF readings and *b* coefficient, respectively; \overline{T} is the mean absolute temperature; *t* is the Student's test. At the confidence level of 95% and $n \ge 20$, the Student's test is $t \le 2$ [16]. Using this model (Table 3) and common thermodynamic functions the partial molar functions of antimony at 298 K were calculated, and the values are shown in Table 4.

According to the phase diagram of the Sb–Se–I system, the partial molar functions of antimony in the SbI₃–SbSeI–Se subsystem are the thermodynamic functions of the following potentialforming reaction [14]:

$$Sb(solid) + 0.5SbI_3(solid) + 1.5Se(solid) = 1.5SbSeI(solid)$$
 (3)

Using this equation, the integral thermodynamic functions of formation of SbSel can be calculated as:

$$\Delta Z_{\rm SbSel}^{0} = 0.667 \Delta \bar{Z}_{\rm Sb} + 0.333 \Delta Z_{\rm SbI_3}^{0} \tag{4}$$

where $\Delta \bar{Z}_{Sb}$ is the partial thermodynamic function of antimony; $\Delta Z_{Sbl_3}^0$ is the standard thermodynamic function of formation of Sbl₃. For the calculations according to (4) thermodynamic parameters of Sbl₃ were taken from the literature [19].

Standard entropy of the ternary compound was calculated using the following equation:

$$S_{\rm SbSel}^{0} = 0.667(\Delta \overline{S_{\rm Sb}} + S_{\rm Sb}^{0}) + 0.333S_{\rm Sbl_3}^{0} + S_{\rm Se}^{0}$$
(5)

For the calculations, the standard entropies (S_{298}^0) of antimony and selenium were taken from the database [20] as $45,689 \pm 0.627$ and $42,132 \pm 2092$ J/mol K, respectively. The results of the calculations are presented in Table 5. In all cases the estimated standard deviations were calculated by accumulation of errors. For comparison, Table 5 also contains thermodynamic data obtained from the tensimetric measurements [7,20]. Also, our EMF measurements for the alloys in the Sb₂Se₃–SbSeI–Se subsystem allowed us calculating the standard integral thermodynamic functions of Sb₂Se₃ (Table 5), which were found to be in a good agreement with the literature data [14,18,19]. Such an agreement additionally confirms that the data obtained in this research are self-consistent.

4. Conclusions

As the principal conclusion, the application of various experimental methods enabled us constructing the self-consistent phase diagram of the Sb–Se–I system, which can be the base for growing single crystals of SbSeI. The partial molar functions of antimony and the standard integral thermodynamic functions of SbSeI have been calculated form the reliable experimental data.

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