

Physicochemical and X-ray diffraction study of phase formation in the InSe-SrSe system and study of electrophysical properties of solid solution alloys

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Using the methods of physicochemical analysis (DTA, X-ray diffraction, MSA, as well as density and microhardness measurements), phase formation in the InSe-SrSe system has been studied and its phase diagram constructed. It is found that the InSe-SrSe system is a quasi-binary section of the ternary system Sr-In-Se and is eutectic. A ternary compound of the formula SrInSe₂ is formed in the system with a component ratio of 1:1. The SrInSe₂ compound melts congruently at 1180°C. According to the results of X-ray structural analysis, it is established that the SrInSe₂ compound crystallizes in the orthorhombic syngony, the lattice parameters are: $a = 10.95 \text{ \AA}$; $b = 8.65 \text{ \AA}$; $c = 8.42 \text{ \AA}$. The coordinates and temperatures of the two eutectics formed in the system are 15 and 65 mol % SrSe and $t = 847 \text{ K}$ and $t = 1273 \text{ K}$, respectively. In the system at room temperature based on the InSe compound, solid solutions reach up to 5 mol % SrSe, and on it is SrSe up to 3 mol % InSe. As a result of studying the electrical conductivity (σ), thermal conductivity (α), current (I) and specific resistance (ρ) of solid solution alloys (InSe)_{1-x}(SrSe)_x ($x=0.01$; 0.02; 0.03; 0.05), it has been established that solid solution alloys are semiconductors with medium resistance.

Keywords: System, Quasi-Binary, Eutectic, Phase, Incongruent

It is known that indium chalcogenides, ternary and quaternary phases and solid solutions based on them are widely used in various fields of the electronics industry as photosensitive semiconductor materials¹⁻⁶. If sulfide and selenide compounds of antimony are also photosensitive materials, then its telluride compounds are thermoelectric materials and are successfully used for energy conversion⁷⁻⁹. Alkaline earth element chalcogenides are very active substances. They form compounds and solid solutions with chalcogenides of various metals. Ternary systems with the presence of chalcogenides of calcium subgroup elements have been studied¹⁰⁻¹³. Systems involving strontium chalcogenides have not been sufficiently studied. Alkaline earth element chalcogenides and ternary compounds based on them exhibit strong luminescent properties due to the action of activators¹⁴⁻²⁰.

From this point of view, it could be expected that new phases and solid solutions obtained by chemical interaction of indium and strontium chalcogenides could also represent semiconductor materials with complex properties.

We have previously investigated a number of ternary systems based on alkaline earth metal chalcogenides²¹⁻²⁴. The InSe-SrSe system has not yet been investigated.

The aim of this research work is to study phase formation in the InSe-SrSe system using physical and chemical analysis methods, construct its phase diagram, and study the electrophysical properties of solid solution alloys.

The InSe compound is a stable compound, melts at 933 K, crystallizes in hexagonal syngony, lattice parameters: $a=4.05$; $c=16.93 \text{ \AA}$, sp. gr. P6₃/mmc-D⁴_{6h}, density $\rho=5.56 \text{ g/cm}^3$ (Ref. 25). The SrSe compound melts congruently at 11873 K, crystallizes in cubic syngony, lattice parameter: $a = 6.243 \text{ \AA}$, spatial group Fm3m, density $\rho = 4.50 \text{ g/cm}^3$, microhardness $H\mu = 1250 \text{ MPa}$ (Ref. 26).

Experimental Section

In order to clarify the chemical processes occurring in the InSe-SrSe system, alloys with 5 and 10% content of each were synthesized. To synthesize

ternary compounds, the initial components were synthesized first. The synthesis of the SrSe compound is described in Chapter 3 on page 54. After obtaining the initial components, the alloys of the InSe-SrSe system were synthesized from the SrSe and InSe components at a temperature of 1073-11473 K in a quartz ampoule with air suction to a pressure of 0.1333 Pa. The alloys were subjected to heat treatment for 240 hours at a temperature of 823 K to bring them to equilibrium.

Then the alloys of the InSe-SrSe system were studied by methods of physicochemical analysis (DTA, X-ray diffraction, MSA, as well as density and microhardness measurements).

X-ray phase analysis of the alloys was performed on a D2 PHASER X-ray diffractometer. A $\text{CuK}\alpha$ electrode was used as an irradiator.

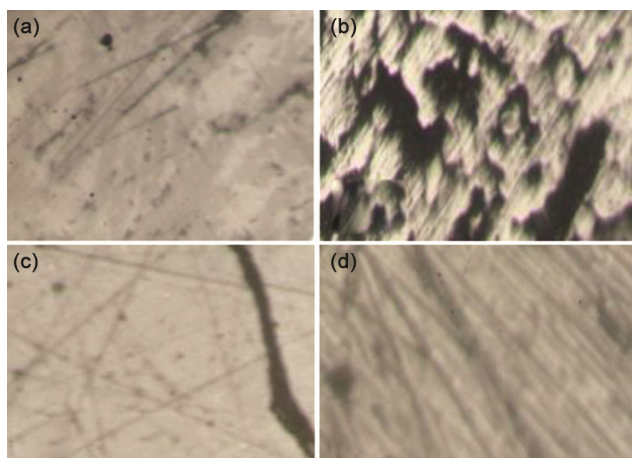
Microhardness was measured on a PMT-3 metallographic microscope. During the measurements, the dependence of microhardness on weight was studied.

Microstructure analysis was performed on an MIM-8 microscope. When determining the densities of the alloys of the system, the pycnometric method was used and toluene was used as a filling solution. Electrical conductivity and thermoelectric potential, which are the main properties of semiconductors, are coefficients. Physical measurements were carried out by the potentiometric method²⁷⁻³⁰.

Results and Discussion

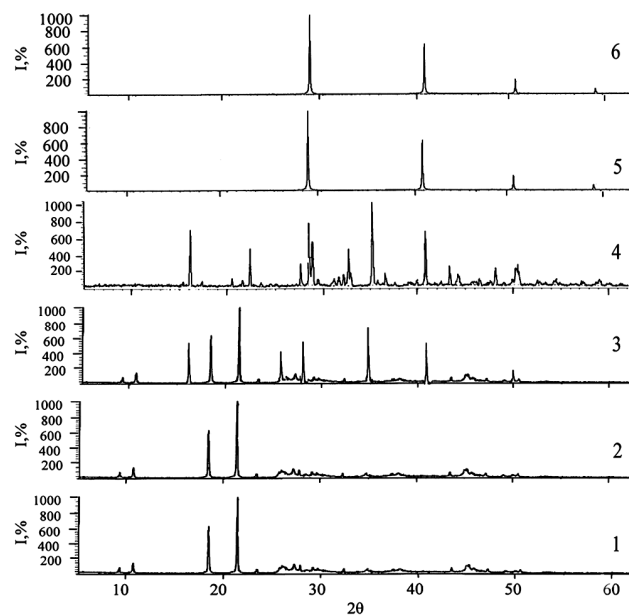
Alloys rich in SrSe compounds undergo hydrolysis due to absorption of moisture from the air during prolonged exposure to open air. Alloys in the range of 0–60 mol. % SrSe are resistant to environmental influences. Alloys of the SrSe-InSe system are highly soluble in strong acids (HCl , HNO_3 , H_2SO_4). Based on the results of the differential thermal analysis of the alloys, it was established that two endothermic effects are observed on the thermograms of the alloys.

This shows that alloys are two-phase. To confirm this, a microstructural analysis of alloys was carried out. It is determined that there are single-phase alloys based on primary components, and the rest of the alloys are two-phase. At RT, the area of the solid solution based on the InSe compound is 5 mol % SrSe, and the solubility is 3 mol % InSe based on SrSe in Fig. 1 presents the microstructure of alloys with the content of 5, 40, 50 and 97 mol % SrSe. As can be seen from the figure, single-phase samples of SrSe with a content of 5 and 97 mol % SrSe are solid-solution alloys based on InSe and SrSe



1-5 mol %, 2-40 mol %, 3-50 mol % (SrInSe_2), 4-97 mol % SrSe

Fig. 1 — Microstructure of alloys of the InSe-SrSe system



1-InSe, 2-5, 3-40, 4-50 (SrInSe_2), 5-97, 6-100 mol. % SrSe

Fig. 2 — Diffraction patterns of alloys of the InSe-SrSe system

compounds, respectively. The sample with 50 mol % SrSe was also single-phase and is a chemical compound with the formula SrInSe_2 . The sample containing 40 mol % SrSe is a two-phase alloy.

An X-ray phase analysis of the InSe-SrSe alloys containing 5, 40, 50 and 97 mol % SrSe was performed (Fig. 2). The diffraction lines in the diffraction patterns of single-phase samples containing 5 and 97 mol % SrSe correspond to the diffraction patterns of InSe and SrSe compounds, *i.e.* the samples are solid solution alloys based on these compounds. The diffraction pattern of the 40 mol % SrSe sample contains diffraction lines of the InSe and SrInSe_2 components. This sample is two-phase.

The diffraction lines in the diffraction patterns of the 50 mol % SrSe sample differ from the diffraction lines of the primary components in their intensity and interplanar distance, which means the formation of a new phase in the system. This composition corresponds to the formula SrInSe_2 .

Thus, the alloys of the InSe-SrSe system were studied by the methods of physicochemical analysis and their phase diagram was constructed (Fig. 3). The InSe-SrSe system is quasi-binary and is characterized by the formation of a congruent compound SrInSe_2 . The melting point of the ternary compound containing SrInSe_2 at a component ratio in the system of 1:1, the temperature is 1453 K. In the system at RT, a solid solution of 5 mol % SrSe is formed based on the InSe compound, the solubility of which is 3 mol % InSe in terms of the SrSe compound. The composition of the eutectic formed between the InSe and SrInSe_2 compounds is 15 mol. % SrSe, the temperature is 560°C. The composition of the eutectic point was determined by constructing the Tamman triangle. The composition of the other eutectic formed in the system is 65 mol % SrSe, temperature -1273 K.

The liquidus of the InSe-SrSe system is surrounded by monovariant equilibrium curves of the α -solid solution based on the InSe compound, the SrInSe_2 compound, and the β -solid solution based on SrSe, which are in equilibrium with the liquid. Based on the InSe compound, the initial crystallization of the α -solid solution from the liquid ends at the eutectic point of 15 mol % SrSe. In the system, the two-phase alloys consisting of $(\alpha + \text{SrInSe}_2)$ crystallize below the solidus line in the region of 5-50 mol % SrSe. The

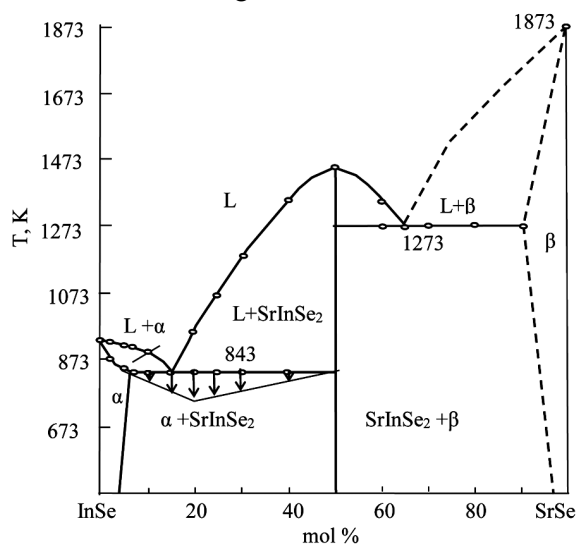


Fig. 3 — Phase diagram of the InSe-SrSe system.

two-phase alloys consisting of $(\text{SrInSe}_2 + \beta)$ crystallize in the SrSe concentration range of 50-97 mol % and below the solidus line. Some physicochemical properties of the InSe-SrSe system alloys are given in Table 1. When measuring the microhardness of the alloys in the system, three types of microhardness values were obtained.

The microhardness value (600-690) MPa corresponds to the hardness of the α -solid solution based on InSe, the value (1100-1160) MPa corresponds to the microhardness of alloys containing SrInSe_2 , and the microhardness value (1250-1280) MPa corresponds to the microhardness of β -solid solutions based on SrSe.

An X-ray structural analysis of the SrInSe_2 compound formed in the InSe-SrSe system was carried out, and it was established that the SrInSe_2 compound crystallizes in rhombic syngony with lattice parameters: $a=10.95$; $b=8.65$; $c=8.42$ Å. The results of the X-ray phase analysis of the compound SrInSe_2 are shown in Table 2.

Electrophysical properties of the alloys of solid solutions $(\text{InSe})_{1-x}(\text{SrSe})_x$

To study the electrophysical properties of the alloys of solid solutions $(\text{InSe})_{1-x}(\text{SrSe})_x$ ($x=0.01$; 0.02; 0.03, 0.05), samples were synthesized and subjected to heat treatment at a temperature of 770 K for 150 hours. To study the electrophysical properties, samples were prepared in the form of parallelepipeds measuring $1.5 \times 1.0 \times 0.6$ cm³.

In the study, the condition of excitation of the crystal along the c axis of the light beam was assumed. Electrical conductivity and thermoelectric properties of InSe-based solid solution alloys were studied in the temperature range of 300-660 K. The temperature dependence of the electrical conductivity of the $(\text{InSe})_{1-x}(\text{SrSe})_x$ ($x=0.01$; 0.02; 0.03, 0.05) solid solution alloys is shown in Fig. 4. When adding high-resistance samples of 1; 2; 3 and 5 mol. % SrSe to the InSe composition, the electrical conductivity decreases depending on the increase in the amount. On the other hand, when adding 1; 2; 3 and 5 mol. % SrSe to the InSe compound, they fill the space between the InSe compound layers, that is, defects are filled, which leads to a decrease in electrical conductivity depending on the composition. For the samples, the electrical conductivity increases with temperature, which means that they have semiconductor properties. The electrical conductivity value for n-type semiconductors is calculated using the following formula.

Table 1 — Composition of InSe-SrSe system alloys, results of DTA, density and microhardness measurements

Composition (mol %)		Thermal Effects (K)	Density (q/sm^3)	Microhardness, MPa		
InSe	SrSe			α P=0,10 N	SrInSe ₂ P=0.15 N	β
100	0,0	933	5,56	600	—	—
97	2,0	873,933	5,58	630	—	—
95	5,0	848,927	5,57	660	—	—
93	7,0	843,913	5,49	690	—	—
90	10	843,898	5,45	690	—	—
85	15	843	5,42	Eutec.	Eutec.	—
80	20	843,953	5,35	—	—	—
75	25	843,1063	5,30	—	1100	—
70	30	843,1183	5,25	—	1100	—
60	40	843,1343	5,17	—	1100	—
50	50	1453	5,05	—	1150	—
40	60	1273,1343	4,90	—	1160	—
35	65	1273	4,87	—	Eutec.	Eutec.
30	70	1273	4,82	—	—	1280
20	80	1273	4,71	—	—	1280
10	90	1273	4,61	—	—	1270
0,0	100	1873	4,50	—	—	1250

Table 2 — Intensity of the diffraction lines (I), interference indices (hkl) and interplane distances (d) of the compound SrInSe₂

N _o	I, %	2 θ	$d_{\text{exp}}, \text{\AA}$	h	k	l
1	75	16,178	5,4770	4	0	0
2	9	20,537	4,3229	0	2	0
3	8	21,099	4,2104	0	0	2
4	47	22,410	3,9654	1	0	2
5	4	23,563	3,7760	0	1	2
6	31	27,699	3,3386	3	0	1
7	86	28,627	3,1146	3	1	1
8	69	28,928	3,0832	2	1	2
9	8	29,523	3,0258	0	2	2
10	13	31,208	2,8666	0	3	0
11	19	32,186	2,7815	0	0	3
12	78	32,712	2,7368	6	0	0
13	100	35,205	2,5495	1	1	3
14	19	36,604	2,4551	2	3	1
15	8	39,601	2,2758	3	3	0
16	66	40,827	2,2105	3	0	3
17	38	43,409	2,0849	5	1	1
18	16	44,267	2,0473	1	4	1
19	11	46,540	1,9513	3	1	1
20	40	48,193	1,8879	0	2	4
21	6	49,076	1,8561	3	4	0
22	48	50,550	1,8052	2	4	2
23	8	54,471	1,6842	0	0	5
24	6	57,377	1,6058	7	0	0
25	9	59,100	1,5631	6	2	2

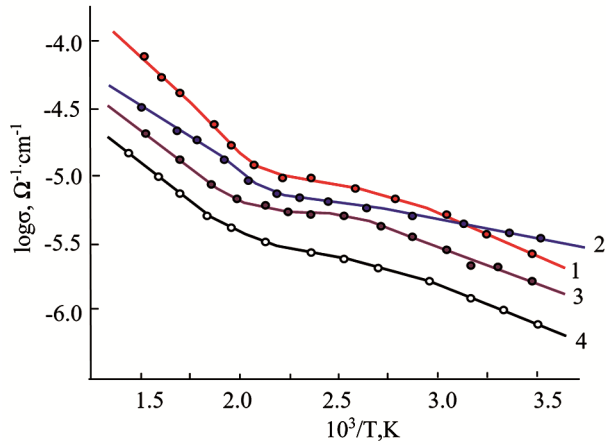


Fig. 4 — Temperature dependence of electrical conductivity of solid solution alloys (InSe)_{1-x}(SrSe)_x, 1-1, 2-2,3-3, 4-5 mol % SrSe

$$\sigma = \sigma_0 \cdot e^{-\frac{E_g - F_n}{K_T}}$$

Here F_n is the chemical potential, and for $\sigma_0 = \text{const}$ the dependence $\sigma \sim 1/T$ will be completely exponential.

For solid solution alloys containing 1; 2; 3 and 5 mol. % SrSe, impurity conductivity occurs in the temperature range of 300–500 K. Above 500 K, the electrical conductivity of the samples increases. At this time, electrons and holes participate jointly in the conductivity.

At RT, the logarithmic values of the electrical conductivity of solid solution alloys with 1; 2; 3 and 5 mol. % SrSe were $\log \sigma = -5.60 \Omega^{-1} \cdot \text{cm}^{-1}$, $\log \sigma = -5.50 \Omega^{-1} \cdot \text{cm}^{-1}$, $\log \sigma = -5.80 \Omega^{-1} \cdot \text{cm}^{-1}$ and $\log \sigma = -6.20 \Omega^{-1} \cdot \text{cm}^{-1}$, respectively. At 660 K; the logarithmic values of electrical conductivity for samples containing 1; 2; 3 and 5 mol. % SrSe, amounted to $\log \sigma = -4.15 \Omega^{-1} \cdot \text{cm}^{-1}$, $\log \sigma = -4.50 \Omega^{-1} \cdot \text{cm}^{-1}$, $\log \sigma = -4.65 \Omega^{-1} \cdot \text{cm}^{-1}$ and $\log \sigma = -4.80 \Omega^{-1} \cdot \text{cm}^{-1}$, respectively.

Temperature dependence of the thermo-EMF of the solid solution alloys (InSe)_{1-x}(SrSe)_x

The temperature dependence of the thermo-EMF of the solid solution alloys (InSe)_{1-x}(SrSe)_x ($x=0.01; 0.02; 0.03, 0.05$) is shown in Fig. 5.

When introducing 1, 2, 3 and 5 mol. % SrSe alloys into the InSe compound, the thermoelectric power coefficient decreases depending on the composition. As can be seen from Fig. 5, the thermoelectromotive force (thermo-EMF) reaches its maximum value upon transition from RT to the region of intrinsic conductivity and then decreases. The temperature dependence of the thermo-EMF corresponds well to

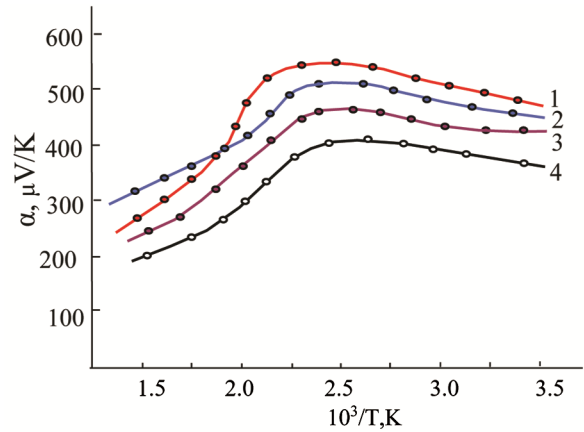


Fig. 5 — Temperature dependence of thermo-EMF of solid solution alloys (InSe)_{1-x}(SrSe)_x, 1-1, 2-2,3-3, 4-5 mol % SrSe.

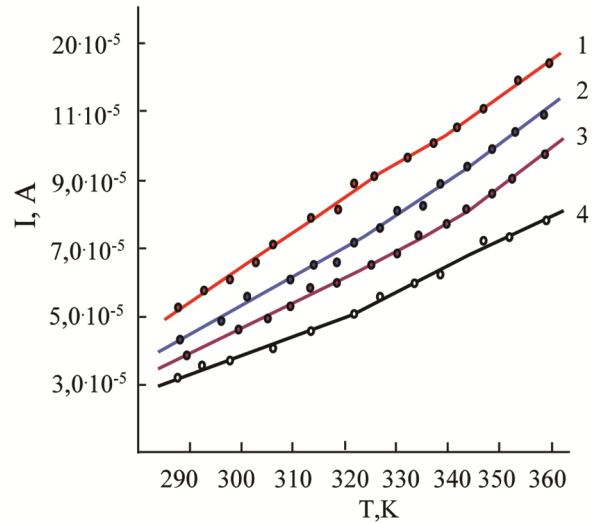


Fig. 6 — Temperature dependence of current strength (I) in solid solution alloys (InSe)_{1-x}(SrSe)_x ($x=0.01; 0.02; 0.03; 0.05$). 1-1; 2-2; 3-3; 4-5 mol. % SrSe.

the electrical conductivity of the alloys. The maximum values of the thermo-EMF of 1, 2, 3 and 5 mol. % SrSe alloys at 400 K are $\alpha = 550 \mu\text{V/K}$, $\alpha = 510 \mu\text{V/K}$, $\alpha = 465 \mu\text{V/K}$ and $\alpha = 400 \mu\text{V/day}$. The values of the thermoelectric coefficient of these alloys at a temperature of 660 K are: $\alpha = 380 \mu\text{V/K}$, $\alpha = 310 \mu\text{V/K}$, $\alpha = 250 \mu\text{V/K}$ and $\alpha = 200 \mu\text{V/K}$, respectively.

Fig. 6 shows the temperature dependence of the current of the solid solution alloys of 1, 2, 3 and 5 mol. % SrSe. As can be seen from Fig. 6, as the temperature increases, as is typical for semiconductors, the current also increases. As the temperature increases, it becomes easier for electrons to move from the valence band to the conduction band, which leads to an increase in the current (I).

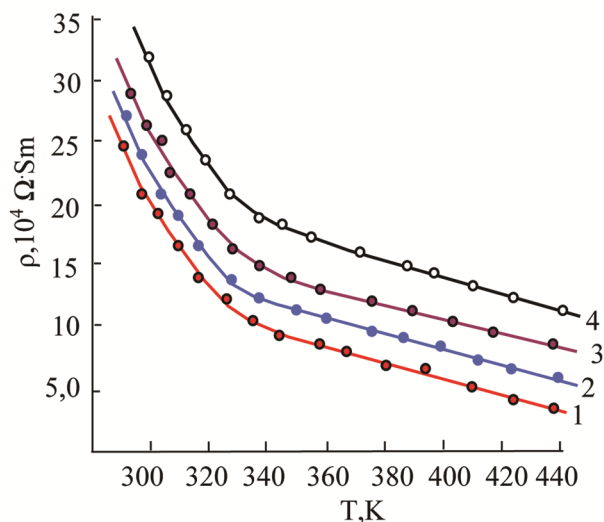


Fig. 7 — Temperature dependence of the specific resistance (ρ) in solid solution alloys $(\text{InSe})_{1-x}(\text{SrSe})_x$ ($x=0.01; 0.02; 0.03; 0.05$). 1-1; 2-2; 3-3; 4-5 mol % SrSe.

However, in metals, conductivity decreases with increasing temperature, since metals do not have a band gap, and electrons freely moving inside the crystal cannot enhance the vibrations of atoms at the crystal sites as the temperature increases.

Fig. 7 shows the composition and temperature dependence of solid solution alloys containing 1, 2, 3 and 5 mol. % SrSe. As can be seen from Fig. 7, the addition of high-resistance alloys with a concentration of 1, 2, 3 and 5 mol. % SrSe to the InSe compound leads to an increase in resistance depending on the composition. From this graph it can be seen that all four alloys show a decrease in resistivity with increasing temperature.

Conclusion

Thus, the InSe-SrSe system has been studied and its phase diagram has been constructed. The phase diagram of the system is a quasi-binary section of the ternary Sr-In-Se system and is of the eutectic type. It has been found that in the InSe-SrSe system, a compound consisting of SrInSe_2 is formed in a component ratio of 1:1. The SrInSe_2 compound melts congruently at a temperature of 1453 K. According to the results of microstructural analysis, it has been established that 5 mol % SrSe dissolves in solid solutions based on InSe at RT, and up to 3.0 mol % InSe dissolves in the SrSe compound. X-ray phase analysis showed that the SrInSe_2 compound crystallizes in a cubic syngony with the lattice parameters: $a = 10.95$; $b = 8.65$; $c = 8.42$ Å. The

coordinates of the two eutectics formed in the InSe-SrSe system are 15 mol % SrSe, $t=843$ and 65 mol % SrSe, $t=1273$ K. The electrophysical properties of solid solution alloys $(\text{InSe})_{1-x}(\text{SrSe})_x$ ($x=0.01; 0.02; 0.03; 0.05$) were studied. As a result of the study of electrical conductivity (σ), thermo-EMF (α), current strength (I) and specific resistance (ρ) of solid solution alloys $(\text{InSe})_{1-x}(\text{SrSe})_x$ ($x=0.01; 0.02; 0.03; 0.05$) it was established that solid solution alloys are semiconductors with medium resistance.

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