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STUDY OF CRYSTAL CHARACTERIZATION AND CHEMICAL INTERACTION IN THE TERNARY SYSTEM Ho-Sb-Te

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Complex methods of physical and chemical analysis: differential thermal analysis (DTA), X-ray phase analysis (XRD), microstructural analysis (MSA), as well as density and microhardness measurements, were used to study the nature of chemical interaction in the Ho-Sb-Te ternary system over the entire concentration range along the following sections:1. Sb_2Te_3-Ho_2Te_3, 2. Sb_2Te_3-HoTe, 3. HoTe-Sb, 4. HoSb-HoTe, 5. Sb_2Te_3-Ho, 6. HoSb-Te. The projection of the liquidus surface of the ternary system and the diagrams of the state of internal sections were constructed according to experimental data and literature data on binary systems: Sb-Te, Sb-Ho, Ho-Te. It is established that sections (1), (2), (3), (4) are quasi-binary, and (5), (6) are non-quasi-binary sections of the ternary system. The incongruently melting compound $HoSbTe_3$ is formed in the Sb_2Te_3-Ho_2Te_3 system and the area of solid solutions based on Sb_2Te_3 ~ 3-5 mol% at 300K was found.
Keywords: *System; Cut; Liquidus; Quasi binary; Non-quasi binary*
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1. INTRODUCTION

Chalcogenide semiconductors are materials with a wide range of applications. Therefore, these materials' physical and chemical properties have been studied for a long time. The results obtained during the research are also important for the purchase of new semiconductors. In particular, the results obtained during structural studies are important for the explanation of the physical properties of these materials. Therefore, it is important to study the structure and structural phase transitions in chalcogenides [1-5].

Compounds containing $B_2^V X_3$ (B^V-Sb, Bi; X-Se, Te) occupy an important place among chalcogenide semiconductors. It was determined that a number of properties of these compounds are resistant to temperature and radiation [6-8]. The ternary phases (solid solutions, compounds) and composite materials based on them have unique properties such as valuable semiconductors, thermoelectrics and topological insulators. Some of them (materials with thermoelectric properties) are used as the positive and negative arms of the thermocouple in the development of energy converters for the conversion of environmentally friendly, economically efficient solar energy into electricity as an alternative energy source, and topological insulators are promising materials in spintronics and quantum computing [9-13].

When obtaining new chalcogenide semiconductors with cation-cation substitutions, conducting structural studies and studying phase analyzes are very important. During the experiments carried out by the X-ray diffraction method, it was determined that a single-phase system does not form during some substitutions. A two-phase and even three-phase system is formed [14,15]. It is important to study the processes of phase formation in these compositions.

Although phase formation processes have been studied in many systems in chalcogenide semiconductors, the Ho-Sb-Te system has not been sufficiently studied. In this study, the Ho-Sb-Te ternary system was studied in a wide temperature and solidity interval, and the process of phase formation in the system, chemical interaction, crystallization areas of the ternary system, and equilibrium processes occurring in the system were studied.

2. RESEARCH METHODS

During the study of the ternary system, the synthesis of its alloys was carried out using elements of a special pure brand - Holmium metallicity-Holm-0, antimony-B-4, tellurium-TA-2. When performing the experimental part of the research work, the following methods were used: high-temperature differential-thermal analysis (DTA), X-ray phase analysis, microstructure analysis, determination of microhardness, and density measurement. DTA experiments were carried out in an argon atmosphere in a BTA-987 calorimeter. The DTA method is a unique method for studying the processes occurring due to thermal energy in solid bodies [16,17]. Structural studies were performed using D8 ADVANCE and D2 Phaser devices, microstructure analysis was performed using metallographic microscope MIM-7, and microhardness using PMT-3 brand devices. The X-ray diffraction method is one of the modern research methods for studying the structure of crystals and structural phase transitions. This method allows us to determine lattice parameters, symmetry, and space groups of solids and to determine structural phases [18-20].

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The alloys of the studied system were synthesized in a high-temperature furnace at a temperature of 800-1250 K in vacuumed quartz ampoules. To create an equilibrium state in the alloys, they were thermally treated in vacuum ampoules at a temperature of 800 K for 900 hours. The starting component crystallizes in rhombohedral syngonia of Sb_2Te_3 tetradymite type in the R3m space group, lattice parameters: a=4.383, c=30.487 Å, and melting temperature: 895 K. The compound Ho_2Te_3 crystallizes in orthorhombic syngonia in space group Pbb with lattice parameters: a=12.17, c=8.606 Å, and melting temperature: 1643 K.

3. RESEARCH RESULTS AND DISCUSSION

HoSb-HoTe system. The thermal properties of the HoSb-HoTe system were studied using a W-W/Re thermocouple in an argon atmosphere in a BTA-987 calorimeter in graphite crucibles. 2 endothermic effects were observed in all thermograms. The structure of these compositions was studied by the X-ray diffraction method. The obtained X-ray spectra are given in Figure 1.

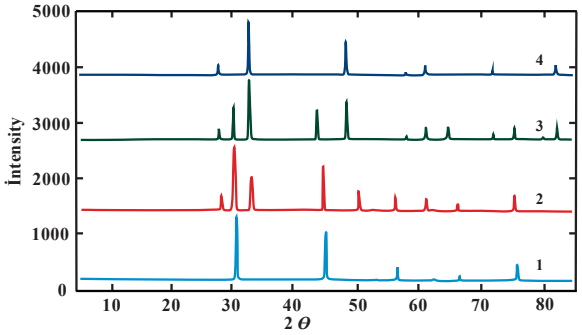


Figure 1. X-ray diffraction spectra of the HoSb-HoTe system: 1- HoSb, 2-65 mol%, 3-75 mol%, 4-100 mol% HoTe.

During the analysis of the X-ray diffraction spectra, it was determined that the X-ray spectra of the compositions correspond to the mixture of the starting components. The microstructure analyzes of these compositions were studied under a MIM-7microscope. The results of the microstructure analysis of HoSb-HoTe alloys are given in Figure 2.

Figure 2. Microstructure of alloys of the HoSb-HoTe system: a) 20 mol%, b) 60 mol%, c) 80 mol% HoTe

The microhardness of the obtained compositions was also studied. It was determined that 2615 MPa corresponds to the HoSb phase, and 2575 MPa corresponds to the HoTe phase. Thus, based on the obtained results, a phase diagram of the HoSb-HoTe system was constructed (Figure 3).

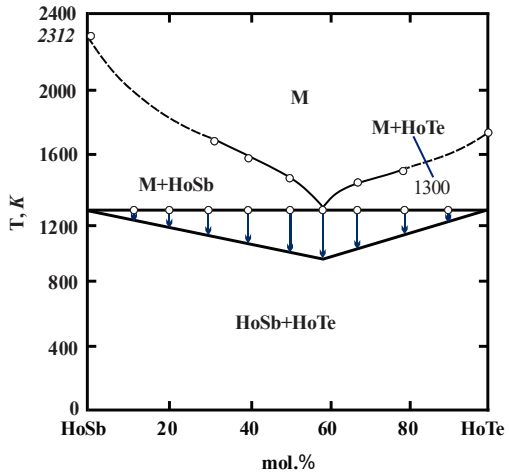


Figure 3. Phase diagram of the HoSb - HoTe system.

As can be seen from Figure 3, the HoSb-HoTe system is a quasi-binary part of the Ho-Sb-Te ternary system and is a simple eutectic type. The coordinates of the eutectic are 60 mol% HoTe and 1300 K. The existence of a homogeneous field based on the primary components is practically not determined. A three-phase nonvariant equilibrium process (M↔HoSb+HoTe) occurred in the eutectic.

HoTe-Sb system. Two endothermic effects were revealed during the thermal analysis of the HoTe-Sb system. This indicates that a new phase has not formed in the system. To confirm this process, the structure of the ingredients was studied by the X-ray diffraction method. The obtained spectra are shown in Figure 4. X-ray diffraction spectra show that this composition consists of a mixture of starting materials.

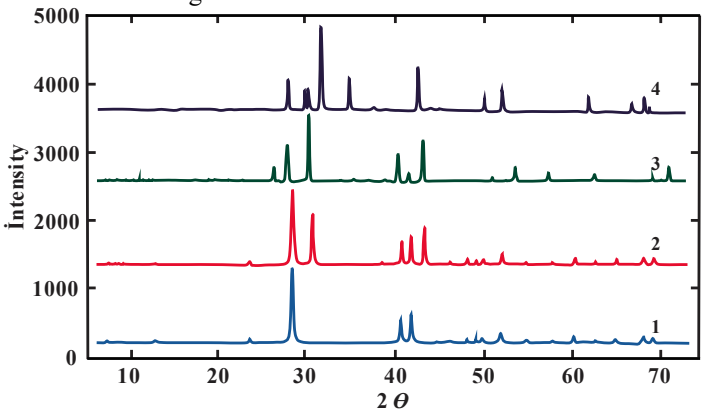
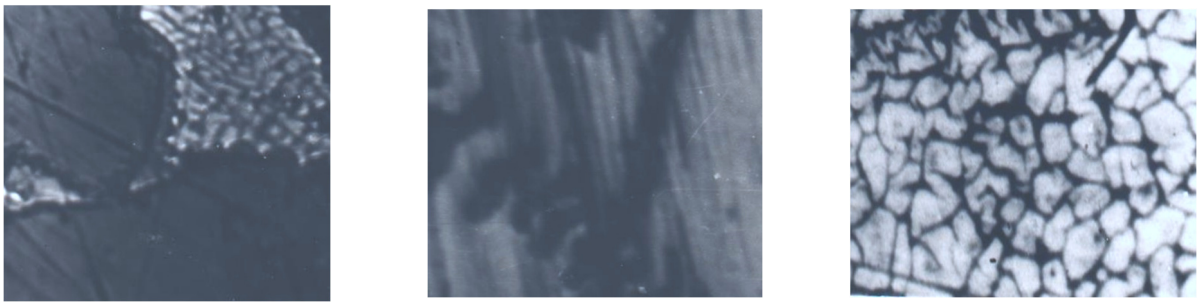


Figure 4. X-ray diffraction spectra of the HoTe-Sb system: 1- Sb, 2-40 mol%, 3-75 mol%, 4-100 mol% HoTe.

The microstructure analyses of these compositions were studied under a MIM-7microscope. The results of the microstructure analysis of HoTe-Sb alloys are given in Figure 5.



a) b) c)

Figure 5. Microstructure of alloys of the HoTe-Sb system: a) 20 mol%, b) 50 mol%, c) 90 mol% Sb

The microhardness of the HoTe-Sb system was also investigated. When analyzing the obtained results, it was determined that 750 MPa corresponds to Sb metal, and 2575 MPa corresponds to the HoTe combination.

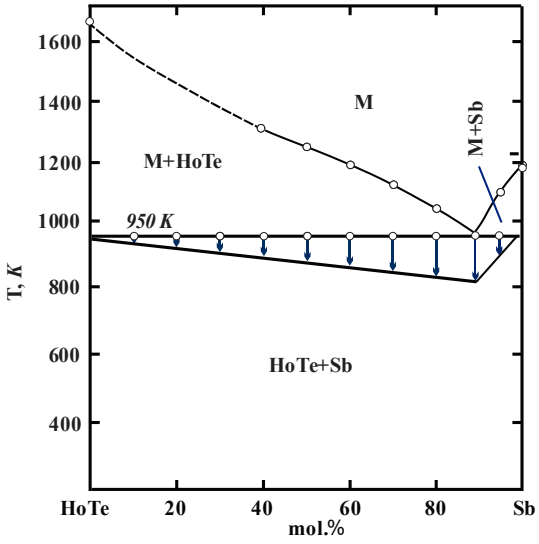


Figure 6. Phase diagram of the HoTe - Sb system

Based on the obtained results, a phase diagram of the HoTe-Sb system was constructed (Figure 6). It was determined that the HoTe-Sb system is a quasi-binary section of the Ho-Sb-Te ternary system and is of a simple eutectic type. In the

system, the eutectic is formed by the equilibrium reaction M↔HoTe+Sb. Eutectic coordinates: 90 mol% Sb and 850 K are set. No solubility domain was detected in the system.

HoSb-Te system. The thermal properties of the HoSb-Te system were also studied. Based on the obtained DTA spectra, it was determined that three endothermic effects are mainly observed in these compositions. To confirm this process, the structure of the ingredients was studied by the X-ray diffraction method. The obtained spectra are shown in Figure 7. X-ray diffraction spectra show that this composition consists of a mixture of starting materials.

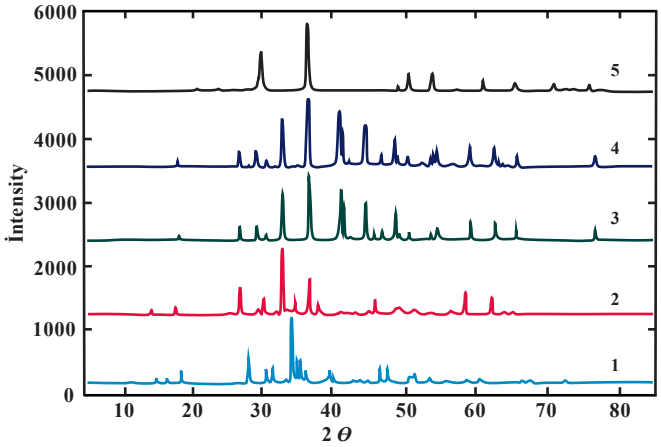


Figure 7. X-ray diffraction spectra of the HoSb-Te system: 1-HoSb, 2-30 mol%, 3-50 mol%, 4- 80 mol%, 5- 100 mol% Te

To study the surface structure of the HoSb-Te system, microstructure analyzes were carried out under the MIM-7 microscope. The results of microstructure analysis of HoSb-Te alloys are given in Figure 8.

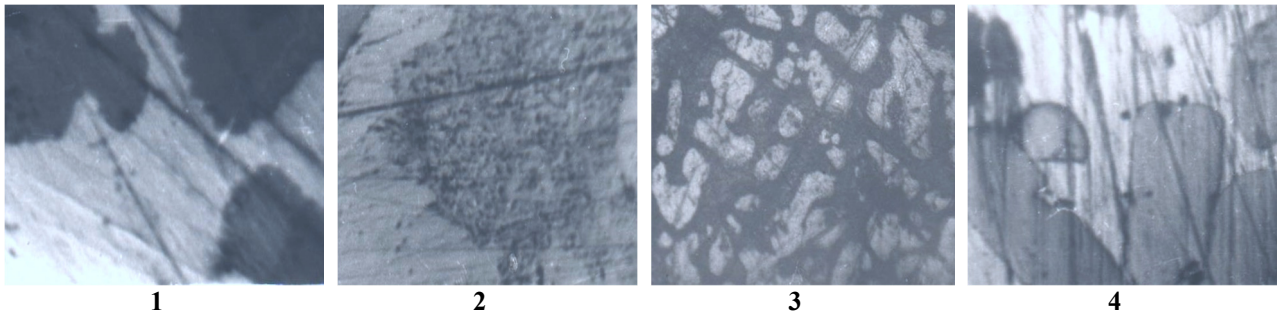


Figure 8. Microstructure of alloys of the HoSb-Te system: a) 20 mol%, b) 50 mol%, c) 90 mol% Sb

The microhardness of the HoSb-Te system was also investigated. Analyzing the obtained results, it was determined that the microhardness of 650 MPa corresponds to Te chalcogenide, and the microhardness of 2615 MPa corresponds to the HoSb compound.

Based on the results of the complex analysis, a phase diagram of the HoSb-Te system was constructed (Figure 9).

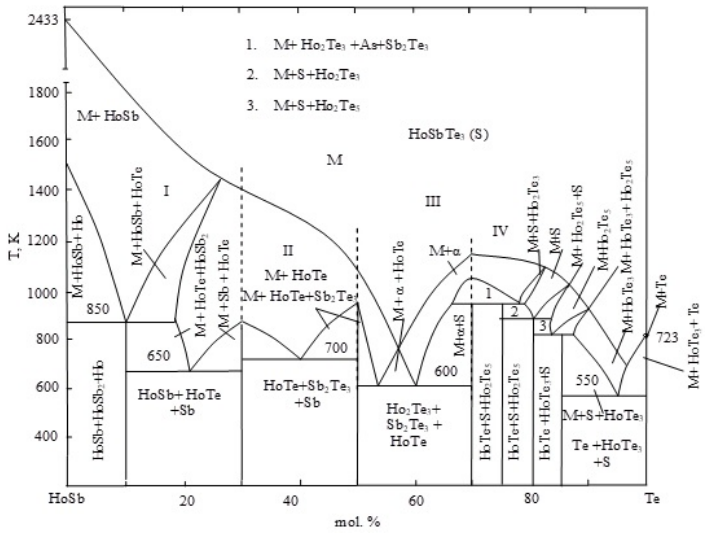


Figure 9. Phase diagram of the HoSb - Te system

As can be seen from Figure 9, the chemical interaction occurring in the system has a complex nature, and the system is a non-quasi binary section of the Ho-Sb-Te ternary system and passes through the area of four subordinate triangles.

In the first part of the system (0-30 mol% Te) a nonvariant triple peritectic and triple eutectic equilibrium processes were reflected:

$$M+HoSb \overset{850\,K}{\longleftrightarrow} HoSb_2 + HoTe$$
$$M \overset{650\,K}{\longleftrightarrow} HoSb_2 + HoTe + Sb$$

In the second part of the system (30-50 mol % Te), only one ternary nonvariant eutectic equilibrium process occurs.

$$M \overset{700\,K}{\longleftrightarrow} HoTe + Sb + Sb_2Te_3$$

The following 4-phase equilibrium processes take place in the third part (50-70 mol% Te) and the fourth part (75-100 mol% Te) of the system, respectively.

$$M + Ho_2Te_3 \overset{900\,K}{\longleftrightarrow} HoSbTe_3\,(S) + \alpha$$
$$M \overset{600\,K}{\longleftrightarrow} Sb_2Te_3 + Ho_2Te_3 + HoTe$$
$$M+Ho_2Te_3 \overset{900\,K}{\longleftrightarrow} HoSbTe_3\,(S) + \alpha$$
$$M+S \overset{850\,K}{\longleftrightarrow} Ho_2Te_5 + Sb_2Te_3$$
$$M + Ho_2Te_5 \overset{650\,K}{\longleftrightarrow} HoTe_3 + Sb_2Te_3$$
$$M \overset{725\,K}{\longleftrightarrow} Sb_2Te_3 + HoTe_3 + Te$$

Projection of the liquidus surface of the Ho-Sb-Te ternary system. The tranquillity of this triple system was carried out. It was determined that the ternary system is divided into five subordinate systems: 1. HoSb-Ho-HoTe, 2. HoTe-Sb-HoSb, 3.HoTe-Sb-Sb_2Te_3, 4.Sb_2Te_3-HoTe-Ho_2Te_3, 5.Sb_2Te_3-Te-Ho_2Te_3. The studied internal cross-sections - Sb_2Te_3-Ho_2Te_3, Sb_2Te_3-HoTe, HoTe-Sb, HoSb-HoTe, Sb_2Te_3-Ho, HoSb-Te and Sb-Te, Ho-Te, Ho-Sb binary systems with the sides of the triangle, based on literature data and the theory of physico-chemical analysis, the projection of the liquidus surface of the mentioned ternary system in the entire solidity range was established (Figure 10).

The nature of the interaction in the Ln-B^V-X, B^V-Sb, Bi, X-Se, and Te ternary systems studied in the order of lanthanoids is of the same type. In all ternary systems, an incongruent melting compound containing LnB^VX^3 and a B^2X^3-based solid solution area of ~5-7 mol % is formed.

The liquidus surface of the system consists of crystallization areas of 13 phases bounded by 26 monovariant curves. The temperature conditions of monovariant and nonvariant equilibrium processes occurring in the system are given in Table 1. 14 nonvariant equilibrium processes occurred in the system, of which five are E_1-E_5 triple nonvariant equilibrium eutectic points, and nine are nonvariant P_1-P_9 peritectic equilibrium points.

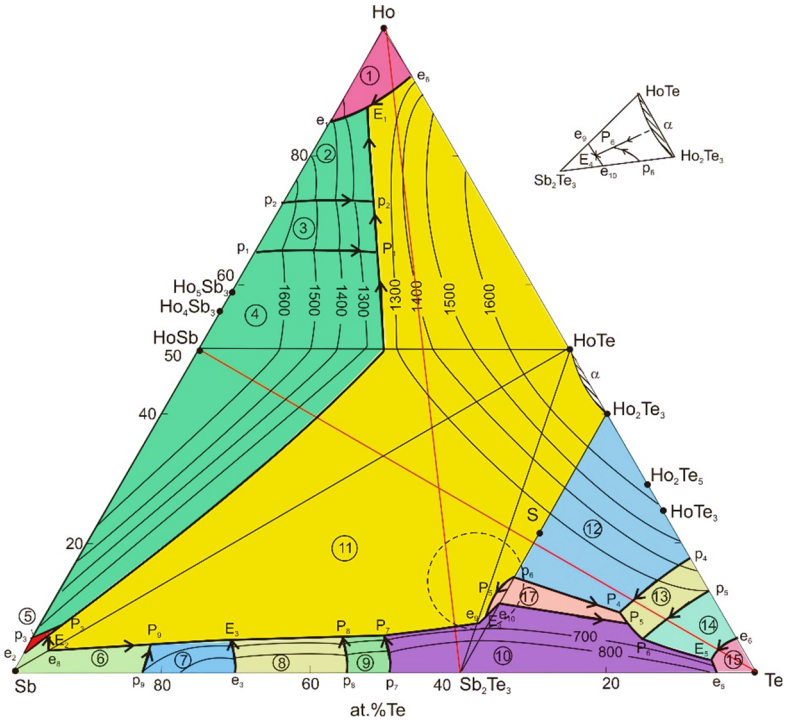


Figure 10. Projection of the liquidus surface of the Ho-Sb-Te ternary system
1-Ho; 2- Ho_5Sb_3; 3- Ho_4Sb_3 ; 4-HoSb; 5-$HoSb_2$; 6-Sb; 7-β; 8-β'; 9-ɲ; 10-Sb_2Te_3; 11-HoTe; 12-α(HoTe+Ho_2Te_3); 13-Ho_2Te_5; 14-$HoTe_3$; 15-Te; 16-α; 17- $HoSbTe_3$(S).

Table 1. Temperature conditions of monovariant and nonvariant processes occurring on the liquidus surface of the Ho-Sb-Te ternary system

№	Equilibrium points and curves	Reactions	Temperature, K
1	E1	M↔Ho+α-Ho4Sb3+HoTe	650
2	E_2	M↔$HoSb_2$ +Sb+HoTe	750
3	E_3	M↔Sb+Sb_2Te_3 +HoTe	600
4	E_4	M ↔ S + Sb_2Te_3 +α	600
5	E_5	M↔Te+$HoTe_3$ + Sb_2Te_3	625
6	P_1	M+ α - Ho_4Sb_3↔ Ho_5Sb_3+HoTe	950
7	P_2	M+ β - Ho_4Sb_3↔α - Ho_4Sb_3+HoTe	900
8	P_3	M+HoSb↔$HoSb_2$ +HoTe	850
9	P_4	M+ Ho_2Te_3 ↔$HoSbTe_3$ +α	900
10	P_5	M+$HoSbTe_3$ ↔Ho_2Te_5+ Sb_2Te_3	675
11	P_6	M+ Ho_2Te_5↔$HoTe_3$+ Sb_2Te_3	725
12	P_7	M+ Sb_2Te_3↔ɲ+HoTe	775
13	P_8	M+ɲ↔ β'+HoTe	720
14	P_9	M+Sb↔β+HoTe	800

Isothermal lines were drawn every 100 K to monitor the course of monovariant curves in the system. (Figure 11). It was determined that the subsolidus of the Ho-Sb-Te ternary system consists of 24 parts. 12 of them are three-phase, and 12 are two-phase.

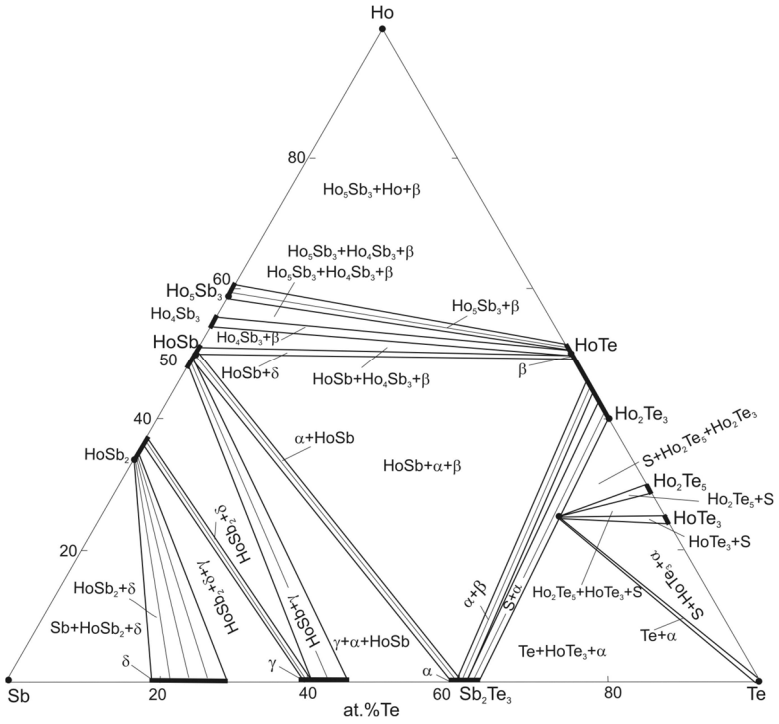


Figure 11. Isothermal section of the Ho-Sb-Te system at 300 K

CONCLUSIONS

1. HoSb-HoTe, HoTe-Sb, and HoSb-Te systems were studied with complex methods of physico-chemical analysis (DTA, RFA, MQA, microhardness and density determination methods) and phase diagrams were constructed.

2. According to the research results, it was determined that the HoSb-HoTe and HoTe-Sb systems are a quasi-binary section of the Ho-Sb-Te ternary system, and the HoSb-Te system is a non-quasibinary section of the Ho-Sb-Te ternary system.

3. The HoSb-Te section passes through the area of four subordinate triangles of the ternary system 1.HoSb-Sb-HoTe, 2.HoTe-Sb- Sb_2Te_3, 3.HoTe- Sb_2Te_3- Ho_2Te_3, 4. Sb_2Te_3-Te- Ho_2Te_3

4. Based on the research results of HoSb-HoTe, HoTe-Sb, and HoSb-Te sections, the liquidus surface of the Ho-Sb-Te ternary system was established, and the coordinates and temperatures of the nonvariant equilibrium points were determined.

5. A diagram of the isothermal section at 300K was constructed to monitor the processes in the solid phase of the ternary system.

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ДОСЛІДЖЕННЯ ХАРАКТЕРИСТИКИ КРИСТАЛІВ ТА ХІМІЧНОЇ ВЗАЄМОДІЇ В ПОТРІЙНІЙ СИСТЕМІ Ho-Sb-Te

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Для вивчення характеру хімічної взаємодії Ho використовували комплексні методи фізико-хімічного аналізу: диференційний термічний аналіз (ДТА), рентгенофазовий аналіз (РФА), мікроструктурний аналіз (МСА), а також вимірювання густини та мікротвердості. -Sb-Te потрійна система в усьому діапазоні концентрацій по ділянках: 1. Sb_2Te_3-Ho_2Te_3, 2. Sb_2Te_3-HoTe, 3. HoTe-Sb, 4. HoSb-HoTe, 5. Sb_2Te_3-Ho, 6. HoSb-Te. Проекцію поверхні ліквідусу потрійної системи та діаграми стану внутрішніх перерізів побудовано за експериментальними та літературними даними для подвійних систем: Sb-Te, Sb-Ho, Ho-Te. Встановлено, що ділянки (1), (2), (3), (4) є квазібінарними, а (5), (6) – неквазібінарними ділянками трійкової системи. У системі Sb_2Te_3-Ho_2Te_3 утворюється інконгруентноплавка сполука $HoSbTe_3$ і знайдено площу твердих розчинів на основі Sb_2Te_3 ~ 3-5 мол.% при 300 K.

Ключові слова: *система; розріз; ліквідус; квазібінарний; неквазібінарний*