

Study of the Ternary System Tb-Sb-Se Along the Section Sb_2Se_3 -TbSe

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***Corresponding author:** Zakir Ismailov,
Department of General and Inorganic
Chemistry, Baku State University,
Azerbaijan

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Zakir Ismailov*, Zivar Hasanova, Rakhila Mirzaeva and Rahman Fatullzade

Department of General and Inorganic Chemistry, Baku State University, Azerbaijan

Abstract

Chemical interactions in the Sb_2Se_3 -TbSe system were studied using physicochemical analysis methods (differential thermal analysis, X-ray diffraction, microstructural analysis, microhardness measurements, and density determination). Based on the analysis results, a phase diagram of the Sb_2Se_3 -TbSe system was constructed. It was established that the Sb_2Se_3 -TbSe cross section is a quasi-binary cross section of the Tb-Bi-Se ternary system and is eutectic. In the Sb_2Se_3 -based system, a solid solution region containing 8mol.% TbSe forms at room temperature. The solid solution area, determined by microstructural analysis, is 17mol.% at a eutectic temperature of 823K and 5mol.% TbSe at room temperature. The phase diagrams show that a new ternary compound with incongruent melting properties, $TbSb_4Se_7$, is formed in the Sb_2Se_3 -TbSe section as a result of a peritectic reaction at 863K with a component ratio of 1:2. X-ray diffraction analysis revealed that $TbSb_4Se_7$ crystallizes in an orthorhombic system with lattice parameters $a=16.762$; $b=23.860$; $c=4.137\text{\AA}$. The solubility of TbSe in Sb_2Se_3 3.0mol% at 300K. Eutectic point coordinates: 10-15mol.% TbSe, temperature: $\sim 823\text{K}$.

Keywords: System; Analysis; Crystallization; Phase; Diagram; Temperature

Introduction

Modern scientific and technological progress, including the exploration of outer space, is inextricably linked with the development of semiconductor technology [1-4]. The rapid development of semiconductor technology has been the main impetus for the search for complex semiconductor materials [5-8]. However, the growing demand for materials in semiconductor technology is not yet fully met due to the lack of materials possessing different combinations of optical, magnetic, and electrical properties. These requirements for materials open up new challenges for chemical engineers, including the synthesis of new substances with desired properties [9-11].

Chalcogenides of rare earth elements, in particular antimony and bismuth, have valuable optical and electrophysical properties for use in thermoelectric and optical devices [12-15]. Therefore, the study of the nature of the chemical interaction of the Sb_2Se_3 -TbSe system is relevant.

Experimental Part

The system was synthesized in a sealed quartz glass ampoule with air removed to 0.133Pa. The furnace was heated very slowly. Particularly after 200 °C, the furnace temperature increased by 100 °C above the melting point of the substance and maintained at this temperature for 1 hour. Cooling of the alloy was accomplished by turning off the furnace. After synthesis, the alloys were heat treated for 500 hours at the solidus temperature in a quartz ampoule under vacuum conditions.

For the synthesis of samples, terbium metal ingots TbM-1 purity 99.9%, stibium grade GOST 1089-82 and selenium GOST ГОСТ 3778-98. were used. The synthesis mode was

selected based on the physicochemical properties of the elementary components, binary compounds. The alloys were obtained by direct fusion of the components in evacuated quartz ampoules at 1500K, followed by slow cooling with the furnace turned off.

The study was conducted using differential thermal analysis, high-temperature differential thermal analysis, X-ray diffraction (XRD), microstructural analysis (MSA), and microhardness measurement. An etchant of the composition $0.1 \cdot K_2Cr_2O_7 \cdot H_2SO_4 + H_2O$ (1:1) was used to study the microstructure of the alloys. Thermal analysis was performed on an NTP-73 pyrometer and a Thermoscope-2. Accuracy ± 5.0 (Pt-Pt/Rh thermocouples were used).

High-temperature thermal analysis (VTTA) was performed using a VTA-987 pyrometer (using W-W/Re thermocouples). X-ray phase analysis of the alloys was performed using a D2 Phaser

and Bruker D8 diffractometer (CuK α -radiation). Microstructural analysis of alloys (MSA) was performed on a MIM-7 microscope.

Results and Discussion

The Tb-Sb-Se (Terbium-Antimony-Selenium) system is a ternary semiconductor system being studied for the creation of new functional materials. The focus is on the study of phase equilibria, particularly along the Sb_2Se_3 -TbSe cross-section, where a ternary compound and a eutectic mixture are formed. These equilibria are studied using differential thermal analysis and X-ray diffraction. Selenium was added in excess to maintain the stoichiometric composition. Thermograms of the alloys show that the observed heating effects are endothermic and reversible. The results of DTA, microhardness and density measurements of the alloys of the Sb_2Se_3 -TbSe system are presented in Table 1.

Table 1: Results of DTA, microhardness and density of Sb_2Se_3 -TbSe system alloys.

N	Composition, mol		Thermal effects T, K	Microhardness, MPa			Density g/cm
	Sb_2Se_3	TbSe		Sb_2Se_3	$TbSb_4Se_7$	TbSe	
1							7,85
2	100	0	848	800-1500		1500-2200	7,92
3	99	1	847	800-1500		1500-2200	8,55
4	97	3	849,805	800-1500		1500-2200	8,67
5	95	5	823,806	800-1500		1500-2200	8,72
6	90	10	823,807				8,91
7	80	20	823		Eutectic		8,96
8	70	30	823,960				8,99
9	60	40	823,985,1030				9,12
10	50	50	823,1000,1175		1000-1800		9,23
11	40	60	1000,1200		1000-1800		9,35
12	30	70	1000,1320		1000-1800		9,42
13	20	80	1000,1480		1000-1800		9,45
14	10	90	1000,1510			2000	9,55
15	5	95	1000,1550			2000	9,61
16	0	100	1620			2000	9,62

The x-ray density of the compound is $9.76g/cm^3$, and the pycnometric density is $9.45g/cm^3$. A solubility range of 3mol% TbSe based on Sb_2Se_3 was found in the system. A solubility range of 3mol% TbSe based on Sb_2Se_3 was found in the system. To determine the solubility limit, samples of 1mol% TbSe based on Sb_2Se_3 were synthesized and placed in ice water after heat treatment for 250 hours. As a result of microstructural analysis, it was mainly determined that the solubility was 5mol% TbSe based on Sb_2Se_3 at 823K, and when the temperature decreased, the solubility decreased to 3mol% TbSe.

Based on the powder method, the structure type and lattice constants of the triple compound were determined, and it was determined that the compound crystallizes in rhombic syngonia. Lattice parameters are $a=16.762$; $b=23.860$; $c=4.137\text{\AA}$.

As can be seen from Figure 1, the system is quasi-binary and

eutectic. Based on Sb_2Se_3 a solubility field of 3mol.% TbSe is formed in the system at 300K. In the system between Sb_2Se_3 and TbSe, the eutectic crystallizes at a temperature of 823K with a content of 20mol.% TbSe. To determine the boundary of the solid solution based on Sb_2Se_3 , 4 samples with a content of 0.5mol.% were synthesized, which were subjected to heat treatment at temperatures of 400, 500, 600 and 700K for 300 hours. Each sample is subjected to microscopic quality analysis. According to the results of microscopic analysis, it was established that at the eutectic temperature (823K), the solubility is 5mol.% in terms of Sb_2Se_3 .

Eutectic point coordinates:

Composition: 10-15mol.% TbSe

Temperature: $\sim 823K$

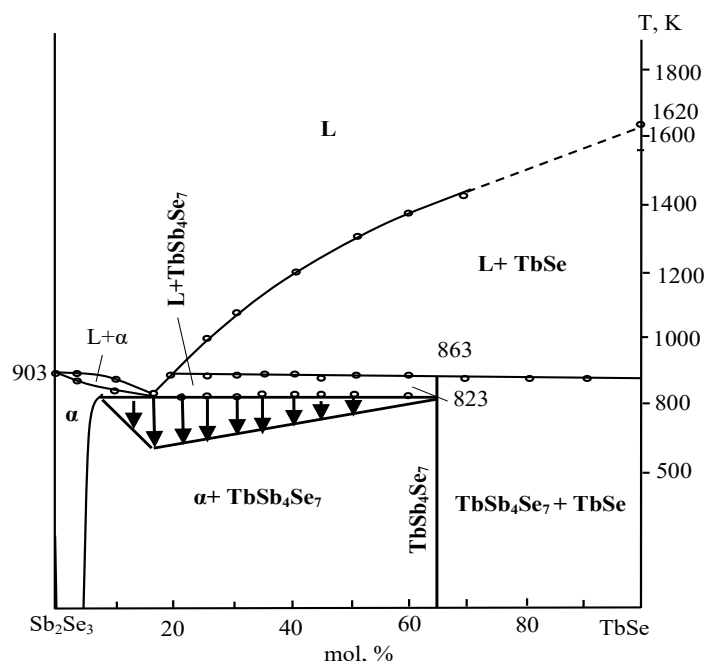


Figure 1: Phase diagram of the Sb_2Se_3 -TbSe system.

Conclusion

1. The Sb_2Se_3 -TbSe system was studied by complex methods of physicochemical analysis in the entire solidity interval, and the phase diagram of the system was constructed.
2. An incongruent melting compound containing TbSb_4Se_7 is formed in the system in a 1:2 ratio of components. It was determined that the compound crystallizes in rhombic syngonia by setting the crystal lattice parameters

$$a=16.762; b=23.860; c=4.137 \text{ \AA}.$$
3. It was determined that Sb_2Se_3 based on the eutectic temperature, the solubility was 5mol%, and as the temperature decreased, the solubility decreased to 3mol% TbSe. The electrophysical properties of some samples based on Sb_2Se_3 were measured at room temperature and it was determined that they are "p" type semiconductors and have thermoelectric properties.

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