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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

INVESTIGATION OF THE Tm-As-S(Se) SYSTEMS IN GLASSY AND CRYSTALLINE STATE AND DETERMINATION OF NEW SUBSTANCES WITH FUNCTIONAL PROPERTIES

Specialty: 2303.01 – inorganic chemistry

Field of science: chemistry

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INTRODUCTION

Relevance and degree of the investigation of the topic. Modern scientific and technological progress, the development of semiconductor technology, the production of new functional materials, especially complex glassy semiconductor materials, and their application are directly related to their application. Chalcogenide glassy substances are highly resistant to aggressive environments and are transparent in the infrared region of the spectrum. The most important thing is to easily control their characteristics by changing their composition. These properties include optical, photoelectric, acousto-optical, magneto-optical properties, etc. The search for new complex functional materials is based on the development of physico-chemical methods of their synthesis, the study of phase equilibria in the corresponding systems, and the physico-chemical, crystal-chemical properties of the initial components

The synthesis of glassy and ideal crystalline substances, the study of their physical, and physico-chemical properties, and the determination of the possibilities of application are among the most important tasks in the field of semiconductors. In this regard, studying the interaction of sulfide and arsenic selenides and sulfide and thulium selenides is one of the most urgent tasks.

Considering the promising properties of chalcogenides of lanthanides and chalcogenides of arsenic, studying the chemical interaction between them allows us to expand the chemistry of chalcogenides of lanthanides and chalcogenides of arsenic. Along with chalcogenides of lanthanides, chalcogenides of arsenic and glassy substances based on them occupied a special place in some areas of semiconductor technology, especially in acoustic optics.

Recently, there has been a lot of literature data about materials based on f-elements and their chalcogenides and arsenic chalcogenides (without oxygen), but there is very little work on thulium with a variable oxidation state and its chalcogenides. In this regard, the study of the physicochemical photo- and acoustic-optical properties of glasses and crystalline substances synthesized in the presence of Tm is one of the actual problems.

A study of the acousto-optical properties of synthesized glassy

alloys based on As_2S_3 showed that they have high acousto-optical properties and photosensitivity. Such glassy substances with such properties occupy a special place in some areas of semiconductor technology, especially in acousto-optics.

Amorphous chalcogenides have long been studied for their important applications in photonics, thermal imaging, phase change memory, integrated fiber optics, biosensors, etc.[1]

Two-dimensional (2D) As_2S_3 materials with high anisotropy and robust stability may be of significant interest in next-generation electronic and optomechanical fields, especially in directed memory devices, and neuromorphic and polarization-sensitive photodetectors.[2]

The study of the electronic and thermoelectric properties of the synthesized 2D As_2S_3 showed that this compound has an indirect band gap of 2.31 eV for the monolayer and 2.08 eV for the bulk.[3]

Object and subject of research. The objects of research are thulium, arsenic, sulfur, selenium, and their binary compounds, and the subject is the study of crystalline and glassy substances in the Tm-As-S and Tm-As-Se ternary systems.

Purpose and objectives of the research. The purpose of the dissertation work is to study the interaction in the Tm-As-S(Se) ternary systems, as well as to study the synthesis and properties of new chalcogenide glassy and crystalline substances with functional properties.

To achieve this goal, the **following specific tasks** were set and solved:

• Development of an optimal regime and method for the synthesis of chalcogenide glassy semiconductors;

• study of ternary systems Tm-As-S, and Tm-As-Se along various sections, determination of the glass formation region in them, and con-

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[1] B.Mortazavi, F. Shojaei, M. Azizi, T. Rabczuk and X. Zhuang. As_2S_3, As_2Se_3 and As_2Te_3 nanosheets: superstretchable semiconductors with anisotropic carrier mobilities and optical properties // J. Mater. Chem. C, P.1-29, 2020, 8, 2400

[2] A.Patel, D. Singh, Y.Sonvane et al. Bulk and monolayer As2S3 as promising thermoelectric material with high conversion performance // Computational Materials Science.2020, 183, 1-5

[3] X.Liu, Z.Zhang, Zh.Ding[b], et al. Highly anisotropic electronic and mechanical properties of monolayer and bilayer As_2S_3 //Applied Surface Science. 2021, 542, 1-13

struction of their phase diagrams;

• determination of physico-chemical properties of glassy alloys and intermediate phases of systems;

• obtaining individual intermediate phases of systems and growing their monocrystals;

• study of the kinetics of bulk crystallization and dissolution of synthesized glassy alloys;

• study of crystallographic properties and the mechanism of formation of new intermediate phases;

• study of the electrophysical, optical, and photoconductive properties of synthesized glasses and intermediate phases;

• identification of new functional materials.

Research methods. Methods of physico-chemical analysis were used to solve the tasks set. The researches were carried out by the methods of differential thermal analysis (DTA), high-temperature differential thermal analysis (HTDTA), X-ray diffraction analysis (XRD), microstructural analysis (MSA), determination of density, measurement of microhardness, chemical analysis for determining the composition of compounds. Measurements of electrophysical, optical, and acoustic-optical properties were carried out using the methods of studying photoelectric properties. DTA of samples up to a temperature of 1300 K was determined with an accuracy of ±5 degrees on the "Thermoskan-2" device. Annealed Al_2O_3 and thermocouple Pt-Pt/Rh were used as standards for DTA. HTDTA was carried out in a helium medium on a BTA-987 device. XRD was carried out on a Bruker XRD D8 Advance with an accuracy of ±5 degrees. MSA was carried out using metallic microscopes МИМ-7 and МИН-8. Measurements of microhardness were carried out on the ПМТ-3 Vickers instrument. Optical properties were measured on a Spectrophotometer SF-5, acoustic-optical and photoelectric properties - on complex devices (monochromator ZMR 3, selective receiver ТТ-1301, selective microvoltmeter).

The main defense propositions.

1. Study of the Tm-As-S(Se) ternary systems along various sections, determination of the boundaries of glass formation areas, and phase diagrams of the investigated sections;

2. Phase equilibria in the studied sections of ternary systems and

their compositions according to the results of research using a complex of physicochemical methods of analysis;

3. Results of studying the physicochemical properties of glassy alloys and intermediate phases found in systems;

4. The mechanism of crystallochemical formation of compounds in the Tm-As-S(Se) ternary systems

5. Study of the kinetics of dissolution and the rate of bulk crystallization of synthesized glasses;

6. Results of studying the physicochemical properties of the studied glassy alloys and intermediate phases;

Scientific novelty. The following ***new scientific results*** were obtained:

• Ternary systems Tm-As-S (Tm-As-Se) were studied along the various sections, non-crystalline regions were identified and phase diagrams were constructed after they crystallized and were brought into an equilibrium state.

• The existence of intermediate phases AsS-TmS, Tm-AsS, As_2S_3-Tm, As_2S_3-TmS, As_2S_3-Tm_2S_3, As-TmSe, AsSe-Tm, As_2Se_3-TmSe, As_2Se_3-Tm_2Se_3 in the sections was established and their composition was determined by a complex of physico-chemical methods of analysis.

• The physicochemical, crystal-chemical, and physical parameters of the compounds obtained were studied. New compounds crystallize in a stibnite-like structure, and the mechanism of their formation has been established.

• The temperature dependence of the electrical conductivity of compounds and glassy alloys was studied and the width of their thermal and optical band gaps was determined. The glass obtained in the systems has high resistance.

• The photoelectric and optical properties of glassy alloys synthesized based on As_2Se_3 and AsSe were studied and it was found that they are sensitive in the infrared part of the spectrum.

• The dissolution rate and kinetics of bulk crystallization of glassy alloys in NaOH alkali were studied and some kinetic parameters were calculated.

Theoretical and practical significance of the research. The theoretical significance of the results obtained in the dissertation

work lies in the fact that data on phase equilibria in the ternary systems Tm-As-S, and Tm-As-Se can be used in the study of corresponding systems involving lanthanides. The constructed phase diagrams can serve to identify new intermediate phases and identify suitable compounds in the presence of other lanthanides. Synthesis of glasses in various rejimes makes it possible to obtain glasses with the required properties. The results of the kinetics of dissolution and crystallization of glasses make it possible to calculate some kinetic parameters and at the same time predict the formation of new structural units in glasses.

The practical significance of the study lies in the fact that, based on the values of the results of physical measurements, new compositions of chalcogenide glasses with promising properties were determined and based on them a patent was obtained from the Republic of Azerbaijan (Patent No. I 2021 0046)).

On the other hand, the constructed phase diagrams, crystal-chemical, physicochemical, etc. properties of the obtained new compounds are fundamental physicochemical parameters and can be included in the corresponding electronic information databases.

Testing and application. 21 scientific works on the topic of the dissertation, including 11 articles in international and local scientific journals, articles and journals of scientific conferences (7 of them in foreign specialized journals), 9 theses, and 1 patent were published.

The main results of the Dissertation work were reported and discussed at the following conferences: "Müasir Təbiət Elmlərinin aktual problemləri" Beynəlxalq elmi konfransları (Gəncə, Azərbaycan, 2017), Ümummilli lider Heydər Əliyevin anadan olmasının 94, 95, 96, 98, 99-cu ildönümünə həsr olunmuş doktorant, magistr və gənc tədqiqatçıların "Kimyanın aktual problemləri" XI, XII, XIII, XV, XVI Beynəlxalq Konfransı, (Bakı, Azərbaycan, 2017, 2018, 2019, 2021, 2022), Doktorantların və gənc təqiqatçıların Elmi konfransının materialları (Bakı, Azərbaycan, 2017, 2018, 2019, 2021, 2022), "Metallurgiya və materialşünaslığın problemləri" mövzusunda 2-ci Beynəlxalq Elmi-Texniki konfrans (Bakı, Azərbaycan, 2017), The XX International Scientific Symposium The Dialogue of Cultures dedicated to the 100th anniversary of A. Jafarzade (Sankt-Petersburg, Russia 2021), Gəncə Dövlət Univer-

siteti Beynəlxalq Elmi Konfrans "Müasir təbiət və iqtisad elmlərinin aktual problemləri" (Gəncə, Azərbaycan, 2022).

The name of the organization in which the dissertation work was carried out. Dissertation work was performed at the "General and Inorganic Chemistry" department of Baku State University.

The total volume of the dissertation with a sign indicating the volume of the structural sections of the dissertation separately. The dissertation consists of an introduction (12683 symbols), five chapters (Chapter I-27968 symbols, II-36825 symbols, III-32531 symbols, IV-33009 symbols, V-16009 symbols), main results (2368 symbols), 190 items of scientific literature used and has a volume of 161 pages. The dissertation contains 59 pictures and 49 tables.

The applicant's contribution to the research conducted. While completing the dissertation, the author directly participated in the analysis of the recent literature data on the topic, conducting experiments, measuring physical and chemical properties, preparing articles, as well as solving other issues.

MAIN CONTENT WORK

In the first chapter of the dissertation, literary data on the topic of the dissertation were presented and analyzed. An overview of literary materials reflecting the interaction in arsenic chalcogen systems, phase diagrams, some physical and chemical properties, the role of physical and chemical factors in glass formation, and features of the interaction of lanthanides forming ternary systems with chalcogens is given. The characteristics of the synthesis of chalcogenides of lanthanoid elements, physical and chemical properties of sulfides and selenides of thulium, arsenic-containing chalcogenide systems, and also the role of lanthanoids in glass formation are given. These data were used in planning experimental studies and processing their results.

In the second chapter, special attention was paid to the selection of synthesis methods and research methods. Complex methods of physico-chemical analysis were used during the research. Investigations include differential thermal analysis (DTA), high temperature differential thermal analysis (HTDTA), X-ray diffraction analysis (XRD), micro-

structure analysis (MSA), density determination, microhardness measurement, chemical analysis to determine the composition of compounds, measurement of electrophysical properties, optical and photoelectric conducted using property survey methods. DTA of alloys up to a temperature of 1300 K was determined with an accuracy of ±5 degrees on the "Termoskan-2" device. Annealed Al_2O_3 and a Pt-Pt/Rh thermocouple were used as standards for DTA. HTDTA was carried out in the helium atmosphere of the brand 51-681-75 in the BTA-987 device. XRD was carried out on a Bruker XRD D8 Advance with an accuracy of ±5 degrees. MSA was performed using МИМ-7 and МИН-8 metal microscopes. The microhardness measurement method was carried out on a ПМТ-3 brand device according to Vickers.

In the third chapter, the properties of glass formation and glassy alloys in Tm-As-S(Se) ternary systems were studied. The results obtained were published in works [3-7, 10, 12, 13, 21].

Glass formations in the ternary system were determined by different sections and syntheses of individual ternary alloys. Different physico-chemical properties of alloys of sections are given depending on the cooling rate. Glass solubility and volume crystallization were determined and certain kinetic parameters were calculated based on their results. Changes in physical and chemical properties of the As_2S_3-Tm, As_2S_3-TmS, AsS-Tm, and As_2S_3-Tm_2S_3, sections depending on the cooling rate were determined.

Section As_2S_3-Tm. The alloys were cooled in three modes at a speed of 1-2 deg/min, 7-10 deg/min, and 450 deg/min (Tables 1-3). The synthesized alloys have a compact shape and cherry color. In the As_2S_3-Tm section, a glass area of 7 at% was observed at a cooling rate of 1-2 deg/min. At a cooling rate of 7-10 deg/min and 450 deg/min, the glass formation areas were 12 and 15 at%, respectively.

As can be seen from Table 1, with an increase in the cooling rate, the glass formation boundaries expand and changes occur in some physical-chemical, macroscopic properties of glasses. After determining the glass formation point, their macroscopic properties were determined according to the corresponding thermograms – glass formation temperature (T_g), crystallization temperature (T_c), and melting temperature (T_m). Microhardness (H_μ) on the ПМТ-3 device and den-

sity (d) were determined pycnometrically.

As can be seen from Tables 1-3, with the increase in the concentration of thulium in the glasses, the values of the macroscopic properties increase.

The macroscopic parameters of glassy alloys obtained in the Tm-As-S and Tm-As-Se ternary systems, depending on the cooling rates are presented in detail in the third chapter of the dissertation.

Table 1

Some physical and chemical properties of the glasses in the As_2S_3-Tm system (cooling rate 1-2 deg/min)

Alloy composition, mol%		Thermal effects T, K			Microhardness, H_{μ},mPa	Density d, q/sm^3	MSA results
As_2S_3	Tm	T_g	T_c	T_m			
100	0	445	-	590	1180	3,58	Dark faza
99	1	450	520	580	1210	3,63	“ ”
97	3	455	525	575	1250	3,65	“ ”
95	5	463	530	565	1280	3,73	“ ”
93	7	470	535	560	1200	3,77	“ ”
90	10	475	545	555	1150	3,90	Glass-crystal

Table 2

Some physical and chemical properties of the glasses in the As_2S_3-Tm system (cooling rate 7-10 deg/min)

Alloy composition, mol%		Thermal effects T, K			Microhardness, H_{μ},mPa	Density d, q/sm^3	MSA results
As_2S_3	Tm	T_g	T_c	T_m			
100	0	445	-	590	1200	4,05	Grey phase
99	1	450	520	580	1250	4,10	“ ”
97	3	455	525	575	1280	4,15	Dark phase
95	5	463	530	565	1300	4,20	“ ”
93	7	470	535	560	1350	4,25	“ ”
90	10	475	545	555	1300	4,35	“ ”
88	12	477	548	565	1350	4,38	“ ”
85	15	480	551	570	1200	4,43	“ ”
80	20	485	555	575	1160	4,48	Glass-crystal

Table 3

Some physical and chemical properties of the glasses in the As_2S_3-Tm system (cooling rate 450 deg/min)

Alloy composition, mol%		Thermal effects T, K			Microhardness, H_{μ}, mPa	Density d, q/sm^3	MSA results
As_2S_3	Tm	T_g	T_c	T_m			
100	0	445	-	590	1450	3,75	Grey phase
97	3	455	525	575	1400	3,79	“ ”
95	5	463	530	565	1370	3,81	Dark phase
93	7	470	535	560	1320	3,85	“ ”
90	10	475	545	555	1280	3,90	“ ”
85	15	480	551	570	1250	3,95	“ ”
80	20	485	555	575	1180	4,05	Glass-crystal

The boundaries of the glass formation area in the ternary systems Tm-As-S and Tm-As-Se are shown in Figure 1, mainly based on the cooling rate in two regimes of certain glass formation areas in binary systems. It was determined that at a cooling rate of v=10 deg/min, the glass formation area takes 33 at.% of the total area of the concentration triangle, and at v=100 deg/min – is 51 at.%.

In the Tm-As-Se ternary system, when the cooling rate is v=10 deg/min, the glass formation area takes about 35 at.% of the total area of the concentration triangle, and when v=100 deg/min, it is 54 at.%.

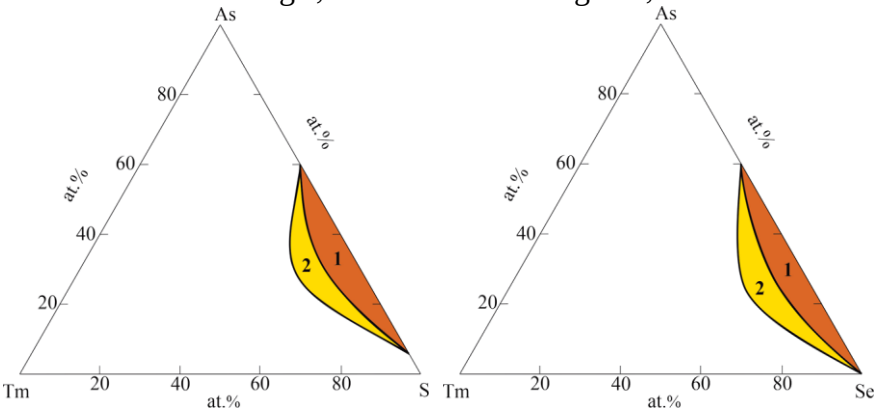

Figure 1. The glass formation areas in the Tm-As-S and Tm-As-Se systems. v=10 deg/min 2. v=100 deg/min

Glass formation in the As_2S_3-TmS section:

After determining the glass formation temperature by the pycnometric method, their macroscopic properties were determined - glass formation temperature (T_g), crystallization temperature (T_c), melting temperature (T_m) according to the corresponding thermograms, while microhardness (H_μ), density (d) were determined pycnometrically: T_g=465 K, T_c=495 K, T_m=580 K (Figure 2).

As_2S_3-Tm_2S_3 section. When studying the alloys, based on the DTA results (Figure 2), it was determined that three thermal effects (T_g, T_c, T_m) are observed in the thermograms of the alloys, it is possible to say that the alloys are glassy form. In the 3-15 mol% Tm_2S_3 compositions range, intense diffraction peaks characteristic of crystalline substances were not detected in the powder diffractograms of the alloys (Figure 3).

*Glasses synthesized based on arsenic selenides***.** Using a complex of physicochemical methods of analysis, the chemical interaction in glassy areas of the Tm-As-Se ternary system was studied and it was established that glass formation occurs in the system. The physicochemical properties (T_g, T_c, T_m, density, microhardness) of the alloys were determined in terms of glass area. It was obtained that T_g=510 K, T_c=530 K, T_m=550 K (Figure 4).

No intense diffraction lines characteristic of crystalline substances were observed on the powder diffractograms of the alloys with the indicated compositions (Figure 5).

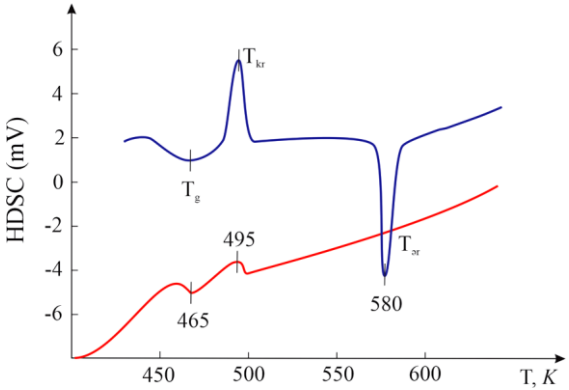

Figure 2. Heating DTA curve of the $[As_2S_3]_{0,90}[TmS]_{0,1}$ alloy

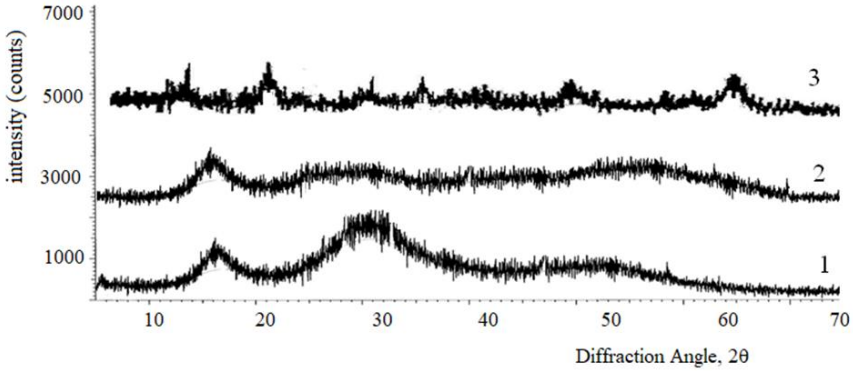

Figure 3. Powder diffractograms of alloys from the glass region of the As_2S_3-Tm_2S_3 system. 1-5 mol %, 2-10 mol %, 3-20 mol % Tm_2S_3.

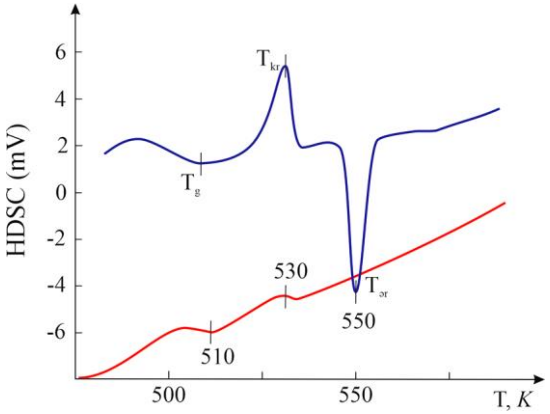

Figure 4. Heating DTA curve of the $[As_2Se_3]_{0.90}[TmSe]_{0.1}$alloy

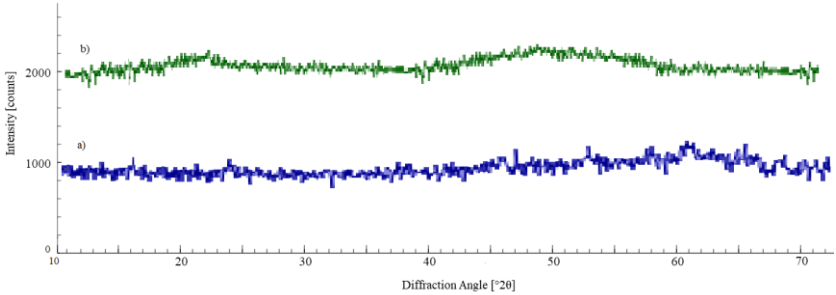

Figure 5. Powder diffraction patterns of alloys with compositions $[As_2Se_3]_{0.90}[TmSe]_{0.1}$ (a) and $[As_2Se_3]_{0.85}[TmSe]_{0.15}$ (b)

Dissolution speed of sulfide glasses in alkalis. Chalcogenide glasses are resistant to practically all aggressive environments, not hygroscopic. They do not react with water and most acids, and dissolve in acids only in nitric acid and alkaline solutions. The dissolution rate of glasses of the $[As_2S_3]_{1-x}[Tm]_x$ system was determined at different concentrations of NaOH alkali (0.25, 0.5, and 1N). The temperature of the solution was maintained at the level of 3-50 °C by using a thermostat. The measurement error is 10%.

The rate of dissolution was determined on three parallel ground and polished samples. The compositions of the glasses are taken according to the formulas $[As_2S_3]_{0.99}[Tm]_{0.01}$, $[As_2S_3]_{0.97}[Tm]_{0.03}$, $[As_2S_3]_{0.95}[Tm]_{0.05}$. Based on the experimentally obtained data, the dissolution rates of the investigated glasses were calculated and the results are given in Tables 4, and 5.

Table 4

Glasses dissolution rates of some investigated alloys

T	1/T	$[As_2S_3]_{0.99}[Tm]_{0.01}$		$[As_2S_3]_{0.97}[Tm]_{0.03}$		$[As_2S_3]_{0.95}[Tm]_{0.05}$	
		$W\cdot10^8$	lnW	$W\cdot10^8$	lnW	$W\cdot10^8$	LnW
298	0,0039	1	-18,42	18	-15,65	35	-11,87
305	0,0032	1,6	-17,98	20	-14,43	38	-14,78
325	0,0030	7,6	-16,42	37	-14,95	40	-14,73

W- dissolution rate, mol/cm^2·sec

Table 5

Some kinetic parameters of glasses with different compositions based on As_2S_3

Composition, mol%	E_0, kcal/mol	C_e,mol/sm^2·sec	C_T, mol/sm^2·sec
As_2S_3	14,6	$1,6\cdot10^{27}$	$3,9\cdot10^{27}$
$[As_2S_3]_{0.99}[Tm]_{0.01}$	4,8	$2,45\cdot10^{20}$	$3,75\cdot10^{27}$
$[As_2S_3]_{0.97}[Tm]_{0.03}$	1,6	$1,6\cdot10^{18}$	$3,69\cdot10^{27}$
$[As_2S_3]_{0.95}[Tm]_{0.05}$	1,4	$4,4\cdot10^{18}$	$3,65\cdot10^{27}$

E_0-activation energy, C_e, C_T- kinetic parameters

As can be seen from Table 5, increasing the amount of thulium element in the glass alloys leads to a sharp decrease in the activation energy.

After studying the crystallization of glasses based on As_2S_3, the volume crystallization rates of glasses based on As_2Se_3 synthesized in the As_2Se_3-Tm_2Se_3 section were studied.

Different compositions were isothermally treated at 525±5K and their properties were studied (Table 6).

Table 6

Effect of isothermal processing on properties as a function of time

Glass composition	Time, hours	Density, g/sm^3	Microhardness H_μ, mPa
$[As_2Se_3]_{99,99}[Tm_2Se_3]_{0,01}$	50	4,55	1350
	100	4,60	1260
	150	4,68	820
$[As_2Se_3]_{0,97}[Tm_2Se_3]_{0,03}$	50	4,61	1260
	100	4,65	1260
	150	4,72	800
$[As_2Se_3]_{0,95}[Tm_2Se_3]_{0,05}$	50	4,63	1300
	100	4,67	1250
	150	4,70	780
$[As_2Se_3]_{99,9}[Tm_2Se_3]_{0,1}$	50	4,67	1210
	100	4,69	1100
	150	4,79	750

The study of the crystallization process of glasses is one of the effective methods of investigating the structure of glasses and determining the glass state. Based on the obtained values, the volume, crystallization time, density, and microhardness of glass alloys with different compositions at a temperature of 525±5 K were determined (Table 7).

Table 7

Volume crystallization of glasses based on As_2S_3 (525±5K)

Glass composition	Time, hours	Density, q/sm^3	Microhardness H_μ, mPa
$[As_2S_3]_{0,99}[Tm]_{0,01}$	50	3,18	1180
	100	3,25	1100
	150	4,18	950
$[As_2S_3]_{0,97}[Tm]_{0,03}$	50	3,20	1100
	100	3,65	980
	150	4,45	900
$[As_2S_3]_{0,95}[Tm]_{0,05}$	50	3,22	980
	100	3,75	900
	150	4,5	850

As can be seen from Table 7, with a decrease in thulium content in glassy alloys, the density of glassy alloys increases, and the value of microhardness decreases. Such a change in prices indicates the crystallization of glassy alloys.

It has been established that after isothermal crystallization and bringing glassy alloys to an equilibrium state, changes occur in the parameters characterizing their physicochemical properties.

In the fourth chapter, based on the results obtained from a complex of physico-chemical methods of analysis in ternary systems Tm-As-S(Se), the phase diagrams of some sections are constructed and phase equilibria in the investigated systems are determined. The results obtained were published in works [1, 2, 8, 9, 11, 14, 15, 20].

AsS-Tm Section. (Figure 6) The alloys of the system were produced by direct synthesis of the initial components in quartz ampoules, followed by stepwise annealing. The synthesis was carried out at temperatures of 720, 900, and 1350 K, respectively, and the alloys were cooled slowly (10 deg/min). Alloys in the range of 0-65 mol% Tm were obtained in compact form, and at higher concentrations - in the powders form.

For the investigations of alloys in crystalline form, they were crushed, pressed into tablets, and subjected to a long-time heat treatment. For homogenization, the alloys were subjected to heat treatment for 500 hours at a temperature of 50-1000°C below the solidus temperature.

The alloys were investigated by a complex of physico-chemical methods of analysis (DTA, MSA, XRD, microhardness measurement, and density determination). The thermal analysis of the investigated alloys up to a temperature of 1350 K was carried out on the Thermoscan-2 device, while at higher temperatures - on the HTDTA BTA-987 device. Detailed descriptions of all research methods are presented in the second chapter of the methodological part of the dissertation. When measuring microhardness, depending on the composition, experimentally determined loads of 10 and 20 grams were used.

Based on the obtained experimental results, a phase diagram of the Tm-AsS system was constructed (Figure 6). The XRD results show that a new phase, TmAsS, is formed in the system, and it is ex-

plained by the fact that it differs from the initial components by specific diffraction lines.

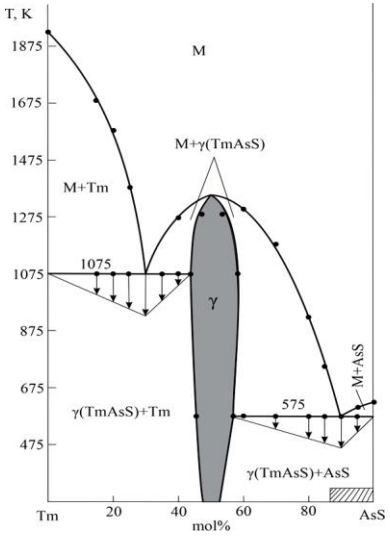

Figure 6. The phase diagram of the Tm-AsS system

As can be seen from the phase diagram of the Tm-AsS system, the liquidus of the system consists of three curves of primary crystallization. The crystallization curve of thulium from liquid and the crystallization curve of the TmAsS compound form a eutectic at the temperature of 1075 K. Between TmAsS and AsS compounds, a second eutectic horizontal line is formed at 575 K. Below the eutectic curves two-phase fields are formed: Tm+TmAsS and TmAsS+AsS. The TmAsS compound has a homogeneity region in the 52-48 mol% Tm concentration range

As_2S_3-TmS section. The alloys of the system were synthesized according to the method specified in the methodological part of the dissertation. For the synthesis, the high-purity arsenic B-5, pure sulfur, and thulium, as well as the initial binary compounds As_2S_3, TmS were used.

The glass area up to 12 mol% was determined by slowly cooling the alloy across the -section (10 deg/min). The alloys were brought into an equilibrium state by isothermal treatment at tempera-

tures below the solidus of 50-100^{0}C and studied using complex methods of physicochemical analysis.

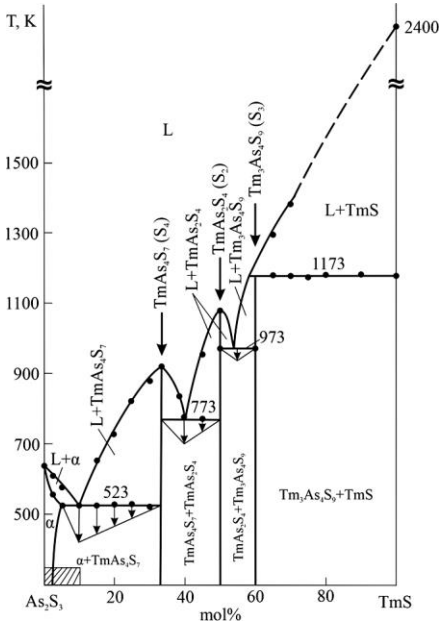

Figure 7. Phase diagram of the As_2S_3-TmS section

The study of alloys by MSA showed that the solubility region of the As_2S_3 in the system is in the range of 0-1.5 mol%. The system is characterized by the formation of the two-phase regions in the 1.5-33.33, 33.33-50, 50-60, and 60-100 mol% TmS composition range. At 33.3 mol% TmS, ($TmAs_4S_7$), at 50 mol% TmS ($TmAs_2S_4$), and at 60 mol% TmS ($Tm_3As_4S_9$) new phases are formed (Figure. 7). Three evectic horizontals are observed in the system: at 523, 773, and 973 K. Coordinates of eutectic points are refined by constructing the Tammann triangle. Below these curves, two-phase fields are formed α+$TmAs_2S_4$, $TmAs_2S_4$+$TmAs_2S_4$, $TmAs_2S_4$+$Tm_3As_4S_9$, and $Tm_3As_4S_9$+TmS.

Based on the results obtained, a phase diagram of the As_2S_3-TmS section was constructed (Figure 7).

As can be seen from the phase diagram, the As_2S_3-TmS section

is a quasi-binary section of the ternary ternary system Tm-As-S. Three new phases are present in the system - $TmAs_4S_7$, $TmAs_2S_4$ and $Tm_3As_4S_9$ compounds. Among them, the compounds $TmAs_4S_7$ and $TmAs_2S_4$ melt congruently, and the compound $Tm_3As_4S_9$ decomposes according to the peritectic reaction $M+TmS \leftrightarrow Tm_3As_4S_9$. The investigated new compounds $TmAs_4S_7$, $TmAs_2S_4$ and $Tm_3As_4S_9$ crystallize in rhombic syngonies, the lattice parameters are as follows: a=1.189, b=1.449, c=0.403Å ($TmAs_4S_7$); a=1.155, b=1.350, c=0.356 Å ($TmAs_2S_4$), a=1.681; b=2.438, c=0.402 Å ($Tm_3As_4S_9$).

As_2S_3-Tm_2S_3 section. Based on the results of DTA, it was established that a new phase compound $TmAsS_3$ is formed in the system with an initial components ratio of 1:1. The results of the X-ray phase analysis confirm the results of the DTA. Microstructure analysis confirms the presence of solid solutions based on As_2S_3 in the range of 0-1.5 mol% Tm_2S_3 and the presence of a $TmAsS_3$ compound. Based on the results of all research methods, a phase diagram was constructed (Figure 8).

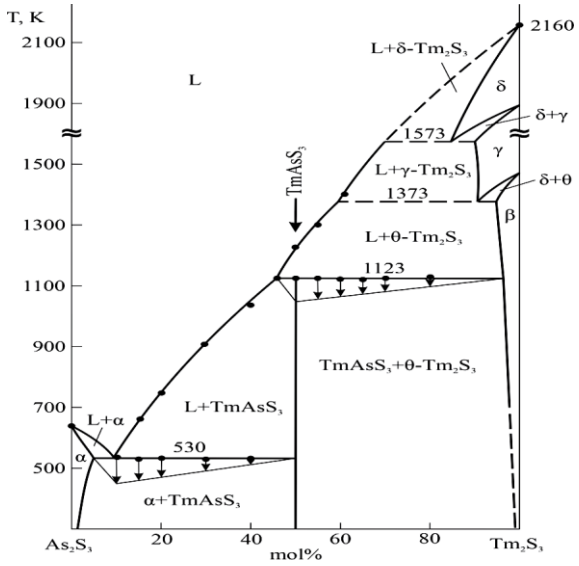

Figure 8. The phase diagram of the As_2S_3-Tm_2S_3 system

As can be seen from the phase diagram, the system has a limited solubility range for the initial components. A region of solid solution of 1.5 mol% based on As_2S_3 and 1 mol% based on Tm_2S_3 was determined (Figure 8). The Tm_2S_3 compound has polymorphism, i.e. it has phase transitions. The polymorphism of the compound also manifests itself in the alloys of the system. As a result, two-phase ($\delta+\gamma$, $\delta+\theta$) and three-phase ($L+\delta+Tm_2S_3$, $L+\gamma+Tm_2S_3$ and $L+\theta+Tm_2S_3$ polymorphic transitions at 1573, 1373, and 1123 K temperatures, acordingly, are formed in the As_2S_3-Tm_2S_3 system. The coordinates of the eutectic and peritectic points are refined by constructing the Tammann triangle. Below the eutectic horizontal at 530 K there is a two-phase region α+TmAsS.

AsSe-Tm section. Alloys based on AsSe were obtained in glass and glass-crystalline form with a solid content of 0-10 mol.%. Alloys of the system were brought to an equilibrium state after heat treatment at a temperature of 200^0C and investigated by complex methods of physico-chemical analysis, and based on the obtained results, a phase diagram of the Tm-AsSe system was constructed (Figure. 9).

As can be seen from Figure 9 and Table 9, when the ratio of components is 1:1, there is a thermal effect at a temperature of 1125 K. Two non-variant processes are fixed in the system. At temperatures of 505 and 800 K, and compositions of 11 and 65 mol% Tm, the corresponding eutectic process and a three-phase reaction occurs

$$M \rightarrow AsSe + \gamma \text{ və } M \rightarrow Tm + \gamma$$

Based on AsSe, an α-solid solution is observed in the range of 0÷15 mol% Tm. The system is characterized by the presence of two two-phase fields: α+TmAsSe and TmAsSe+ γ. The two-phase field $\alpha+\gamma$(TmAsSe) forms in the concentration range of 1.5÷48 mol% Tm. The γ(TmAsSe) phase is observed in the range of 48–52 mol% Tm, and the γ(TmAsSe) and Tm phases in the range of 52–100 mol% Tm. As can be seen from Figure 9, the liquidus of the system consists of three curves. The primary crystallization curve of the boundary phase (α), the primary crystallization curve of the TmAsSe compound, and the primary crystallization curve of the elementary thulium component in the system showed a homogeneity region of the TmAsSe compound in the concentration range of 48÷52 mol. %Tm (Figure 9).

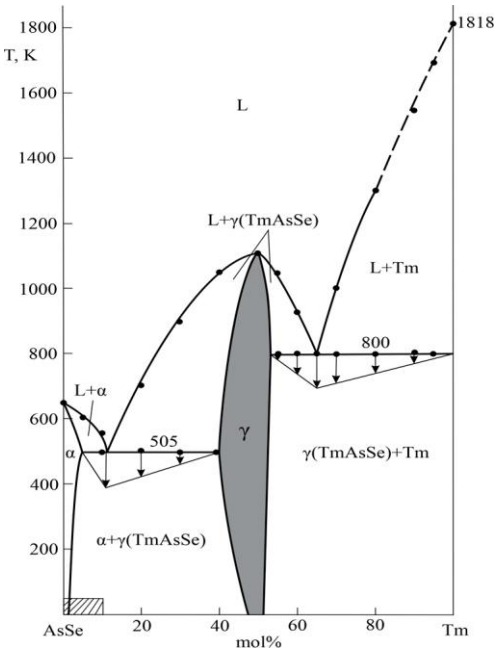

Figure 9. The phase diagram of the Tm-AsSe system

As_2Se_3-TmSe section. The system is characterized by the formation of two ($Tm_3As_4Se_9$ and $TmAs_2Se_4$) incongruent compounds. Complete information about the glass formation region formed in the system is not available. For this reason, we began studying the system again.

Based on the results obtained, a new phase diagram of the As_2Se_3–TmSe system was constructed (Fig. 10).

The phase diagram of the system is a quasi-binary section. Based on As_2Se_3, a narrow region of glass formation was discovered. As can be seen from the diagram, two incongruent compounds ($Tm_3As_4Se_9$ and $TmAs_2Se_4$) and one congruent compound ($TmAs_4Se_7$) are formed in the system. Incongruent compounds are formed at 1100 and 1000 K temperatures, respectively as a result of the following processes.

$$M_{P1} + TmSe \leftrightarrow Tm_3As_4Se_9$$

$$M_{P2} + Tm_3As_4Se_9 \leftrightarrow TmAs_2Se_4$$

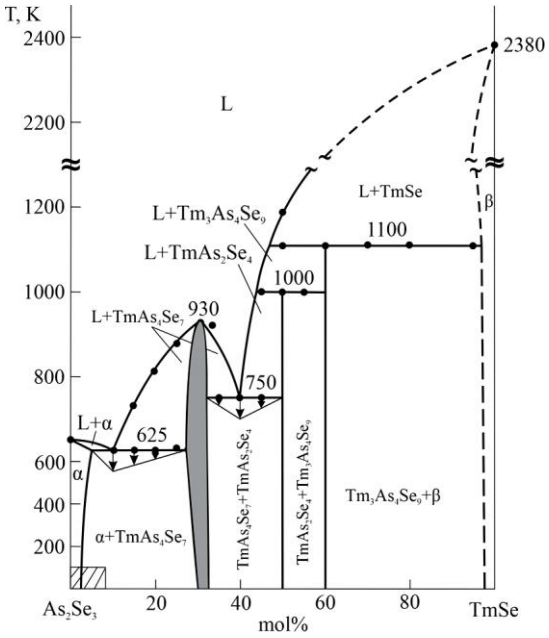

Figure 10. The phase diagram of the As_2Se_3-TmSe system

The congruent compound is formed as a result of the L ↔$TmAs_4Se_7$ process at a temperature of 930 K. The system is characterized by the formation of two eutectic points. A eutectic line is formed between the As_2Se_3 and $TmAs_4Se_7$ compounds (10.5 mol% TmSe, 625 K) and $TmAs_4Se_7$ and $TmAs_2Se_4$ (40 mol% TmSe, 750 K). Their compositions are clarified by constructing the Tammann triangle. Two-phase fields are formed between the initial binary and intermediate ternary compounds: α+$TmAs_4Se_7$, $TmAs_4Se_7$+$TmAs_2Se_4$, $TmAs_2Se_4$+$Tm_3As_4Se_9$, and $Tm_3As_4Se_9$+β. The $TmAs_4Se_7$, $TmAs_2Se_4$, and $Tm_3As_4Se_9$ compounds were obtained individually, their monocrystals were grown, and the lattice parameters were calculated based on the powder XRD results. All three compounds were found to crystallize in orthorhombic syngony: *a*=16,91; *b*=23,58; *c*=4,10 Å ($TmAs_4Se_7$), *a*=11,37; *b*= 13,02; *c*=3,79 Å ($TmAs_2Se_4$), *a*= 6,981; *b*=4,38; *c*=4,02 ($Tm_3As_4Se_9$).

As_2Se_3 – Tm_2Se_3 system. Synthesis of alloys of the system was

carried out in the same way as in the As_2Se_3–TmSe system. The synthesis temperature was stepwise. First, the temperature was raised to 750 K, then to 900-1100 K, and after holding at a higher temperature for 2 hours, alloys containing 15 mol% Tm_2Se_3 were taken out of the furnace and cooled in the air; the glass area was determined for the alloy containing 10 mol% Tm_2Se_3.

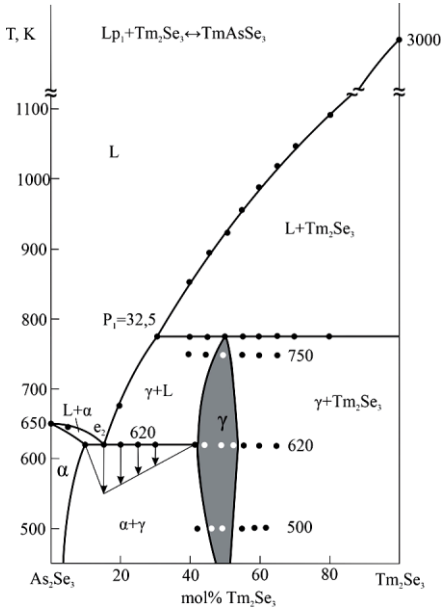

Figure 11. The phase diagram of the As_2Se_3 - Tm_2Se_3 system

After synthesis, alloys were subjected to heat treatment. The annealing temperature for the alloy up to 50 mol.% Tm_2Se_3 was 500 K, and for alloys with a higher Tm_2Se_3 content -700 K. They were kept at these temperatures for 200 hours. Based on the results obtained the phase diagram of the As_2Se_3 – Tm_2Se_3 is constructed (Figure 11).

As can be seen from Figure 11, the system is a quasi-binary section of the Tm-As-Se ternary system. The Liquidus system consists of three primary crystallization curves: As_2Se_3, $TmAsSe_3$, and Tm_2Se_3 compounds. The liquid with the composition of 32.5 mol.%Tm_2Se_3 (point P_1) reacts with Tm_2Se_3 and forms the $TmAsSe_3$ (γ-phase) ternary compound. The system is characterized by the formation of eutectic

point with coordinates of 18 mol% Tm_2Se_3 and 620 K.

At room temperature, the solubility based on As_2Se_3 is 2 mol.% Tm_2Se_3. At the eutectic temperature, it is 5 mol% Tm_2Se_3. In the system, the alloy with a composition of 12 mol.% Tm_2Se_3 was obtained in a glassy form. The microhardness of As_2Se_3 is determined equal to $78 \cdot 10^{-7}$ H/μ^2, which is consistent with literature data.

The fifth chapter presents the results of obtaining monocrystals of ternary phases determined in the studied systems and investigations of their electrical properties. The results obtained were published in works [16-19].

There are several methods for growing monocrystals of semiconductor materials. Taking into account the individual properties of the substances used, the chemical carrier gas was used in this work. The dissertation provides information about the synthesis regime and methods of monocrystals of the $TmAs_4X_7$, $TmAs_2X_4$, $Tm_3As_4X_9$, $TmAsX_3$, TmAsX (X-S, Se) intermediate phases growing.

The optimal regimes of monocrystals growing are given in Table 8.

Based on the analysis of literature data, the most likely reactions are as follows:

1) $2TmAs_2S_4 + 9I_2 \xrightarrow{t} 2TmI_3 + 4AsI_3 + 8S_2$
2) $2TmAs_4S_7 + 15I_2 \xrightarrow{t} 2TmI_3 + 8AsI_3 + 7S_2$
3) $2Tm_3As_4S_9 + 16I_2 \xrightarrow{t} 6TmI_3 + 8AsI_3 + 9S_2$
4) $2TmAsS_3 + 6J_2 \xrightarrow{t} 2TmI_3 + 2AsI_3 + 3S_2$
5) $2TmAsS + 12J_2 \xrightarrow{t} 2TmI_3 + 2AsI_3 + S_2$

Table 8

Optimal regimes of monocrystals growing in the Tm-As-S system

Compositionmol%	Thermal effects, K		The iodine concentration, mg/sm^3	Time hours	Monocrystal sizes, mm^3
	T_1	T_2			
$TmAsS_3$	950	880	4,0	75	1,9x1,2x1
$TmAs_4S_7$	925	845	4,0	72	1,8x1,2x1
$TmAs_2S_4$	1070	980	4,5	48	2x2x1
$Tm_3As_4S_9$	1260	1200	5,0	65	2x1,5x1

The source temperature was determined after heating for 2-3 hours under the conditions necessary for a gas-bearing chemical reaction. A suitable temperature gradient can be achieved within 20-30 minutes. In this case, iodine, chalcogen, and iodide vapors are formed. The temperature gradient is within 60÷90 degrees, and at this time a crystallization center appears.

The stoichiometric composition of single crystals was determined by chemical analysis (Table 9).

Table 9

Crystallochemical and physicochemical properties of compounds formed in the Tm-As-S ternary system

Compound	Sp.gr.	Syngony	Structure type	Lattice parameters				Microhardness H_{μ}, mPa		density d, g/sm^3
				a	*b*	*c*	Z	P_{roent}	P	
$TmAs_4S_7$	Pbnm	Rombik	Sb_2S_3	1,189	1,449	0,403	4	4,19	4,17	1925
$TmAs_2S_4$				1,155	1,350	0,356	4	4,43	4,41	2215
$Tm_3As_4S_9$				1,681	2,438	0,402	4	4,65	4,62	2025
$TmAsS_3$				1,115	1,194	0,403	4	5,09	5,06	1865
TmAsS				1,112	1,187	0,401	4	5,12	5,09	1750

Electrophysical, optical properties and photoconductivity of glassy alloys and compounds.

For a long time, the study of electrophysical properties of glassy alloys was considered one of the main issues in determining the state of glass. One of the important differences between glass and the crystalline state is that glass is less sensitive to additives, and the specific conductivity of glass occurs at lower temperatures.

The electrical conductivity of the As_2S_3 and As_2Se_3 initial compounds and glasses synthesized based on them in the presence of thulium is investigated in the presented dissertation. The synthesis regime of alloys is discussed in detail in the methodical part of the dissertation.

Since arsenic sulfide and selenide are promising semiconductor materials, the study of their electrophysical properties and alloys (glasses) based on them is one of the most urgent tasks. The results of our ex-

periments show that glassy alloys based on As_2S_3 and As_2Se_3 have "p"-type (hole) conductivity, while the obtained new compounds have "n-type" conductivity.

It was determined that the electrical conductivity of As_2S_3 is $1.8 \cdot 10^{-11}$ $Ohm^{-1} \cdot cm^{-1}$, and for As_2Se_3 it is $2 \cdot 10^{-10}$ $Ohm^{-1} \cdot cm^{-1}$. It has been established that the band gap for As_2Se_3 is $\Delta Eg=1.95$ eV, and for As_2S_3 $\Delta Eg=2.2$ eV.

This chapter also presents the results of the temperature dependence of the electrical conductivity of glasses and compounds synthesized based on As_2S_3 and As_2Se_3 compounds.

Figure 12 shows the dependence curves of As_2S_3 and glasses based on it ($10^3/T$). As can be seen from the curves, an increase in the concentration of the thulium in the As_2S_3 increases the electrical conductivity of the samples, and a semiconductor nature of the conductivity is observed. The impurity conductivity field for the $v \sim f(10^3/T)$ curve has not been determined.

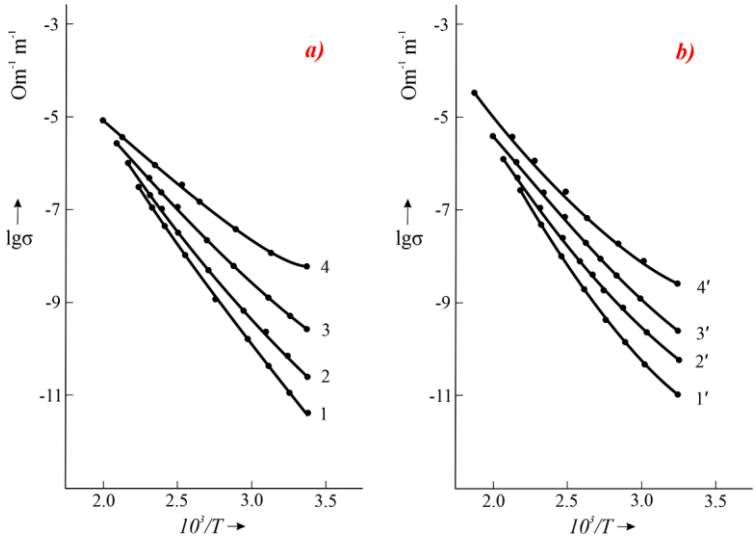

Figure 12. Temperature dependence of the electrical conductivity of glasses of the As_2S_3-Tm_2S_3 system

a) 1- As_2S_3; 2- 99% As_2S_3 +1% Tm; 3- 98% As_2S_3 +2% Tm; 4- 96% As_2S_3 +4% Tm.

b) 1′-99,5 As_2S_3, 2′-98 As_2S_3, 3′-97 As_2S_3, 4′-95 As_2S_3

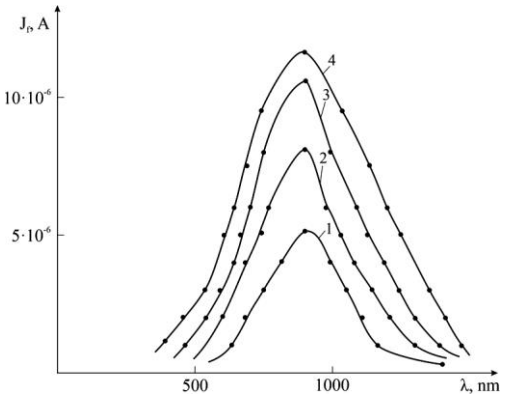

Figure 13. The spectral distribution curve of photoconductivity of glassy alloys based on AsSe. 1-3 mol% TmSe; 2-5 mol% TmSe; 3-7 mol% TmSe; 4-10mol% TmSe

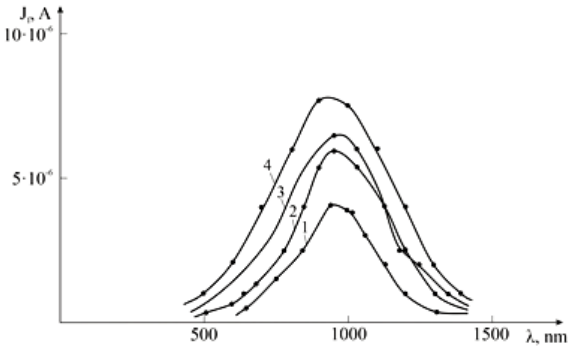

Figure 14. The spectral distribution curve of photoconductivity of glassy alloys based on As_2Se_3. 1-3 mol% TmSe; 2-5 mol% TmSe; 3-7 mol% TmSe; 4-10 mol% TmSe.

The maximum photosensitivity of glasses based on As_2Se_3 compound corresponds to a wavelength of $\lambda = 0.76$ μm, and the maximum photosensitivity of alloys (glasses) based on AsSe corresponds to a wavelength of 0.83 μm.

The band gap is ~1.69 eV for As_2Se_3-based alloys and ~1.494 eV for AsSe-based alloys.

RESULTS

1. Ternary systems Tm-As-S and Tm-As-Se were investigated by a complex of physico-chemical analysis methods in the entire range of concentrations and temperatures along several sections (Tm-AsS, As-TmS, TmS-As_2S_3, Tm_2S_3- As_2S_3, As_2S_3-Tm, AsS-TmS Tm-As_2Se_3, As_2Se_3-TmSe, As_2Se_3-Tm_2Se_3); glass formation fields near arsenic chalcogenides and chalcogenides at different cooling regimes were determined. Compared to sulfur systems, it was found that the area of glass formation in selenium-containing systems is larger. The macroscopic properties (T_g, T_c, T_m, d, H_μ) of the synthesized glassy alloys are studied and the formation of new structural units, except for $AsS_3/2$, is shown as their values increase.
2. The kinetics of dissolution and bulk crystallization of sulfide and selenide glassy alloys were studied and some kinetic parameters (C_T and C_e) were calculated. The crystallization regime of glassy alloys was determined and isothermal crystallization was carried out by heat treatment. The photoelectric properties of glassy alloys synthesized based on arsenic selenides were measured and it was found that they are sensitive in the visible and near-infrared regions of the spectrum.
3. T-x phase diagrams of several sections in the crystalline state were constructed and the formation of the TmAsS, $TmAs_4S_7$, $TmAsS_3$, $TmAs_2S_4$, $Tm_3As_4S_9$, TmAsSe, $TmAsSe_3$, $TmAs_2Se_4$, and $Tm_3As_4Se_9$ ternary compounds in the Tm-As-S and Tm-As-Se ternary systems were determined.
4. The mechanism of crystal-chemical formation of new compounds was studied and the parameters of their elementary lattice were calculated. It is established that the compounds crystallize in rhombic syngonies similar to antimonite.
5. As a result of electrophysical studies, it was established that glassy alloys synthesized based on As_2S_3, As_2Se_3, and arsenic monochalcogenides (AsS, AsSe) are p-type semiconductors with high resistance. The width of the band gaps and optical zones of glasses and compounds is calculated.

6. The acousto-optical properties of glassy alloys synthesized based on As_2S_3 were studied, and it was found that they have high acousto-optical properties and an Az. Resp. the patent was obtained (Chalcogenide glass. Patent I 2021 0446).
7. It has been established that glassy substances synthesized in the Tm-As-S and Tm-As-Se ternary systems are resistant to aggressive environments, resistant to water, organic solvents, hydrochloric and sulfuric acids, and soluble in HNO_3 and alkalis. The kinetics of dissolution of NaOH in alkali was studied and some kinetic parameters were calculated.

The main results of the dissertation work were published in the following scientific works:

1. Ильяслы Т.М., Гахраманова Г.Г., Ф.М. Садыгов [и др.] Фазовые равновесия в системе Tm-S / Актуальные вопросы научных исследований сб. нау. тр по мат. V межд. науч. прокт. конф., – Иваново: 2017, с. 8-10.
2. Ильяслы Т.М., Гахраманова Г.Г., Садыгов Ф.М., Байрамова У.Р., Мамедова Л.М. Квазибинарные разрезы As_2S_3-TmS и As_2S_3-Tm_2S_3 тройной системы Tm-As-S / Международный журнал прикладных и фундаментальных исследований, – Москва: 2017. № 8, ч. 1, с. 40-44.
3. Ильяслы Т.М. Гахраманова Г.Г., Байрамова У.Р., Исмайлов З.И. Стеклообразование в тройной системе Tm-As-S(Se) / Современные тенденции развития науки и технологий. Материалы XXII Международной заочной научно-практической конференции № 1, часть 2, Белгород, – 31 января, 2017, с. 66-70.
4. İlyaslı T.M., Bayramova Ü.R., Qəhramanova G.H. AsS-Tm sistemində şüşələşmə sahəsinin tədqiqi / "Ümummilli Lider Heydər Əliyevin anadan olmasının 94-cü ildönümünə həsr olunmuş doktorant, magistr və gənc tədqiqatçıların "Kimyanın aktual problemləri" XI Respublika elmi konfransı, Bakı, 2017. s.99.
5. Ильяслы Т.М., Гахраманова Г.Г., Г.Наджафоглы. Стеклообразование и свойства стекол на основе As-S в тройной системе Tm-As-S / Metallurgiya və materialşünaslığın problemləri" mövzusunda 2-ci Beynəlxalq Elmi-Texniki konf.materialı. 2017. s. 120.

6. Ильяслы Т.М., Гахраманова Г.Г., Г. Наджафоглы. Физико – химические свойства стекол на основе сульфида мышьяка / Beynəlxalq elmi konfrans «Müasir təbiət elmlərinin aktual promlemləri» I hissə, Gəncə: 4-5 may, 2017. s. 135-138.
7. Ильяслы Т.М., Гахраманова Г.Г., Г.Наджафоглы. Кристаллизация стекла на основе As_2S_3 с участием Tm методом ДТА / East European Scientific Journal, Warsaw: 2018. № 3 (2), p. 44.
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