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GLASS FORMATION AND PHASE EQUILIBRIA IN THE Tm-As-S AND Tm-As-Se SYSTEMS AND THEIR PROPERTIES

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Abstract: *The nature of the physicochemical interaction in the Tm-As-S and Tm-As-Se systems was studied along various cross sections by methods of Differential Thermal (DTA), X-Ray Diffraction (XRD) and Microstructural (MSA) Analyzes, measuring their micro-hardness and determining their electrophysical properties. The investigation of the alloys of the system started by studying the properties of the initial components in cooling modes of 7–10deg/min. After establishing the boundary of the glass formation region in the systems, the physicochemical properties of glasses and intermediate phases were studied. Analysis showed that all effects on the thermograms were reversible. The compounds of $TmAsS_3$, $TmAs_4S_7$, $TmAs_2S_4$, $Tm_3As_4S_9$ compositions were formed in the As_2S_3-Tm_2S_3, As_2S_3-TmS systems. The compounds of $TmAs_4S_7$, TmAsS and $TmAs_2S_4$ compositions melted congruently, while the compounds of $Tm_3As_4S_9$ and $TmAsS_3$ melted incongruently. TmAsSe, $TmAsSe_3$, $TmAs_4Se_7$, $TmAs_2Se_4$ and $Tm_3As_4Se_9$ phases were formed in the Tm-As-Se system while $Tm_3As_4Se_9$ was formed by the peritectic reaction $TmSe + L \leftrightarrow Tm_3As_4Se_9$. Microstructural analysis showed that all alloys of the Tm-As-S, Tm-As-Se cross sections were two-phase, except for the composition of intermediate phases. Proceeding from the results of XRD, the lattice parameters of TmAsS, $TmAsS_3$, $TmAs_4S_7$, $TmAs_2S_4$, as well as parameters of selenium-containing compound were calculated; it found that it crystallized in the rhombic syngony. Based on the results of physicochemical analysis, it was established that the studied cross sections were quasi-binary sections of the Tm-As-S and Tm-As-Se ternary systems. The phase diagram of cross sections with the participation of sulfur and selenium was plotted using the results of physicochemical analysis; it established that the studied cross sections were quasi-binary sections of the Tm-As-S and Tm-As-Se ternary systems.*

Keywords: *analysis, cross section, microhardness, alloy, temperature, crystallization*
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Introduction

Interest in rare earth elements (REE) and their compounds, as well as in arsenic chalcogenide, is related to the use of their compounds in various fields of technology, as well as in obtaining materials with pre-targeted properties. Lanthanide compounds are used as catalysts, HTSC (high-temperature superconducting) ceramics of conductive materials, additives to various alloys for increasing their mechanical resistance, thermal resistance, for obtaining special types of glass used in nuclear technology, for the manufacture of luminous compositions and luminescent materials, in radio technology and optoelectronics, as well as in special probes to study the structure of solutions [1-4]. There are sufficient data on the study of the Ln-As-X system [5-7].

There are excerpted data on the interaction of the Tm-containing-systems and its chalcogenides with arsenic chalcogenides and on the intermediate phases [9-10] formed in them.

Purpose of the study

The purpose of this work is to study the nature of the physicochemical interaction in the Tm-As-S(Se) system along various cross sections: Tm-AsS, Tm-As_2S_3, As_2S_3-TmS, As_2S_3-Tm_2S_3, Tm-AsSe, As_2Se_3-Tm, As_2Se_3-TmSe, As_2Se_3-Tm_2Se_3 and the study of the electrophysical properties of compounds in the wide temperature range.

Material and research methods

TmS, AsS, As_2S_3, AsSe, As_2Se_3, Tm_2Se_3 were synthesized from the elements to study abovementioned cross sections; arsenic of grades A-5, Tm–A-1 (99.97), sulfur of the high purity grade for analysis, selenium of grade B-4 were for this synthesis. The synthesis regime was selected on the basis of physicochemical properties of the initial components and the thermogram –the record of alloys synthesis.

After vacuum pumping the ampoules, the initial samples (3 g) were placed in the oven which was heated to 650-750K for 6-8 hours, and then the temperature was raised to 1200K. The ampoules were kept at this temperature for 5 hours and the ampoules were slowly cooled to room temperature together with the furnace.

The alloys were subjected to homogenizing annealing for 650 hours depending on the concentration of components at appropriate temperatures, 50-70 degrees below the solidus.

The nature of the physicochemical interaction in the Tm-As-S and Tm-As-Se systems was studied by the methods of differential thermal (DTA), X-ray phase (XRD) and microstructural (MSA) analyzes, measuring the microhardness and determining the electrophysical properties.

For the study of the high-temperature part, HTSC 987 equipment was used. DTA was carried out by PDS-021 (two-coordinate self-recorder potentiometer). Microscope MIM-7, MIN-8 and microhardness tester PMT-3).

Results of the study and their discussion

The equilibrium state of the alloys was controlled by the MSA and XRD methods. Alloys with a high content of arsenic sulfide were cherry-red and the color of selenide-based alloys was black. Analysis of thermograms showed that all effects on thermograms are reversible. Mainly, two endothermic effects were obtained on the heating curves, in addition to the compositions corresponding to distectic compounds.

AsS-Tm cross section

Alloys of the system were synthesized in rotary tube furnaces; cooling the alloys was carried out at the rate of v=7-10 deg/min (Table 1).

As is seen from Table 1, with a cooling rate of v=7-10 deg/min, the glass formation area reaches to 10 mol%. Some physicochemical properties of glasses are given in Table 1.

Table 1. Some macroscopic properties of alloys (glasses) of the AsS-Tm system (the rate of cooling v=7-10 deg/min)

№	Composition of the alloys, mol%		Thermal effects T,K			Density d,g/см^3	Microhardness H_{μ}, g/mm^2	Results of MSA
	AsS	Tm	T_g	T_{crys}	T_{melt}			
1	100	0	430	-	580	3.75	118	Single-turbid phase

2	99	1	436	470	575	3.81	120	Single-turbid phase
3	97	3	450	475	568	3.85	125	Single-turbid phase
4	93	7	465	485	545	3.94	133	Single-turbid phase
5	90	10	468	497	535	3.98	135	Single-turbid phase
6	85	15	475	900	530	4.15	140	Crystalline glass

The equilibrium state of the alloys was provided to establish phase equilibrium in the studied systems.

They were annealed for 500 hours below the solidus by 50-100°C to homogenize the alloys. After the equilibrium state, the alloys were studied again and, the phase diagram of Tm-AsS was plotted on the basis of the results of the integrated research methods of physicochemical analysis (Fig. 1).

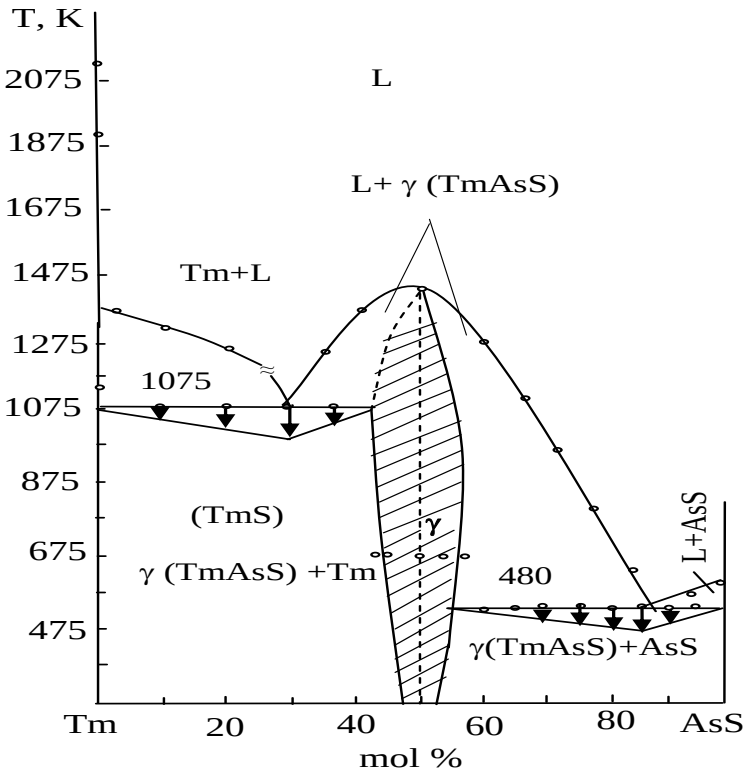


Fig. 1. Phase diagram of the Tm-AsS system

As is seen from Fig., the TmAsS (γ) intermediate phase melting at 1350K was obtained.

As_2S_3 –TmS cross section

These alloys were synthesized similarly to the alloys of the As_2S_3–Tm system. After synthesis, the alloy was cooled at the rate of v=7-10 deg/min. After cooling the alloys, about 13 mol% TmS was obtained in the form of glass. Glassy alloys were investigated physically and chemically. The research results are presented in Table 2.

Table 2. The values of some macroscopic properties of alloys of the As_2S_3 –TmS system (glasses)

№	Composition of alloys, mol	Thermal effects T,K	Micro-hardness	Density d, g/cm^3	Results of MSA

	%					H_{μ}, g/mm^2		
	As_2S_3	TmS	T_g	T_{crys}	T_{melt}			
1	100	0	440	470	580	118	3.75	Single-turbid phase
2	97	3	445	478	550	125	3.79	Single-turbid phase
3	95	5	448	480	595	130	3.81	Single-turbid phase
4	93	7	453	485	555	135	3.85	Single-turbid phase
5	88	10	455-465	475-495	525-580	140-145	3.90-3.95	Single-turbid phase
6	87	13	475	495	565	135	4.01	Single-turbid phase

Three thermal effects appeared on thermograms of the alloys (Fig. 2).

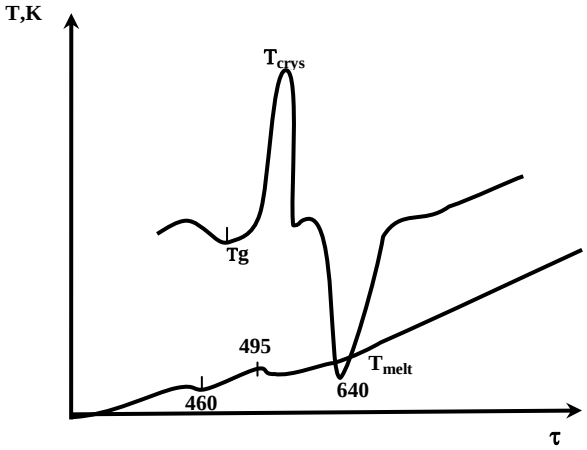


Fig. 2. Thermograms of the alloys of $[As_2S_3]_{0,90}[TmS]_{0,1}$ composition
T_g – vitrification temperature, T_{crys} – temperature of crystallization, T_{melt} – melting temperature

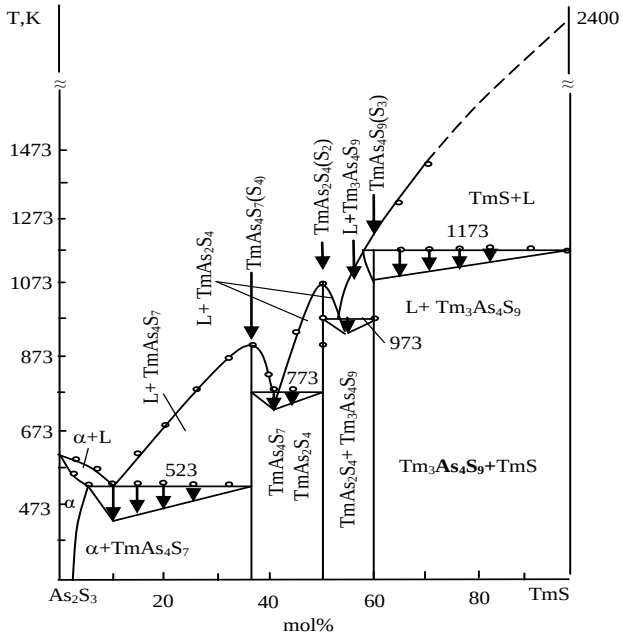


Fig. 3. Phase diagram of the As_2S_3 –TmS system

The phase diagram of the As_2S_3 –TmS cross section was plotted after providing an equilibrium state for the alloys (Fig. 3).

Three intermediate phases $Tm_3As_4S_9$, $TmAs_2S_4$ and $TmAs_4S_7$ were formed along the studied cross section. $TmAs_2S_4$ and $TmAs_4S_7$ compounds melted congruently at the temperatures of 915K and 1175K, respectively, while compounds of $Tm_3As_4S_9$ composition were formed by the following subsequent peritectic reaction:

$$L + TmS \overset{1173}{\Longleftrightarrow} Tm_3As_4S_9$$

The results of XRD showed that new diffraction patterns appear on the diffractograms of the alloys. XRD analysis data are given in Table 3. The Table shows the relationship between inter-planar distances and intensities of diffraction lines.

The XRD results showed that new diffraction lines appear on the diffraction patterns of the alloys in addition to the line of the initial As_2S_3 and TmS. The data of XRD are given in Table 3.

Table 3.Results of XRD analysis for intermediate phase obtained in the As_2S_3 –TmS system

Formula of compounds	Θ	$İ_{exp.}$	$İ_{\%}$	d_α
$TmAs_4S_7$	13.21	10	48.48	3.374
	16.56	16	69.57	3.712
	16.85	18	78.26	2.661
	17.44	24	100	2.621
	19.55	23	98	2.220
	20.73	16	78.13	2.710
$TmAs_2S_4$	12.31	18	37.14	3.622
	15.62	13	37.16	2.871
	16.12	15	42.36	2.780
	16.62	33	94.29	2.713
	16.18	35	100	2.504
	22.34	11	31.43	2.102
	25.76	5	14.29	1.775
$Tm_3As_4S_9$	10.71	15	22.41	4.152
	12.27	18	26.89	8.634
	15.7	19	28.42	2.851
	16.5	32	47.81	2.801
	16.57	46	68.72	2.710
	19.1	67	100	2.403
	19.6	29	43.3	2.310
	22.11	16	23.88	2.051
	25.83	9	13.43	1.813

Parameters of crystalline lattice, singonies and volume of elemental cells were determined based on the calculated data (table 4).

Table 4. Crystallographic and physicochemical properties of thulium ternary compounds (intermediate phases)

Compound	Space group	Singony	Type of structure	Parameters of lattice, nm				Density, g/cm^3		Micro-hardness MPa
	Pbnm	Rhomb.	Sb_2S_3	a	b	c	z	$P_{calc.}$	$P_{pc.}$	
$TmAs_4S_7$				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
$Tm_3As_4Se_9$				2.681	2.438	0.402	4	4.65	4.62	2025
$TmAsSe_3$				1.115	1.194	0.403		5.09	5.06	1865

As_2S_3 –Tm_2S_3 cross section

Synthesis and cooling of alloys were carried out similar to the above systems. In this case, alloys containing up to 15 mol% Tm_2S_3 were obtained in a glassy form. The values of some macroscopic properties of glasses are presented in Table 5.

Table 5. Some physicochemical properties of the glasses of the system (rate of cooling v=7-10 deg/min.)

№	Composition of alloys		Thermal effects T,K			Micro-hardness H_{μ}, g/mm^2	Density d, g/cm^3	Results of MSA
	As_2S_3	Tm_2S_3	T_g	T_{crys}	T_{melt}			
1	100	0	440	475	580	120	3.75	Single-turbid phase
2	97	3	448	480	565	125	3.83	Single-turbid phase
3	95	5	455	485	510	128	3.85	Single-turbid phase
4	93	7	460	490	485	135	3.94	Single-turbid phase
5	90	10	465	510	470	143	4.01	Single-turbid phase
6	85	15	480	515	485	140	4.25	Single-turbid phase
7	80	20	485	520	505	145	4.30	Crystalline glass

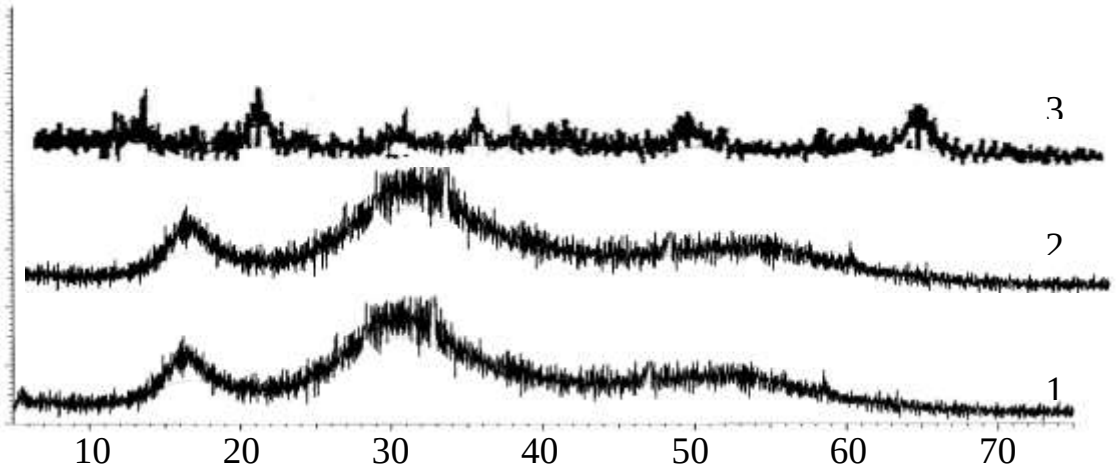
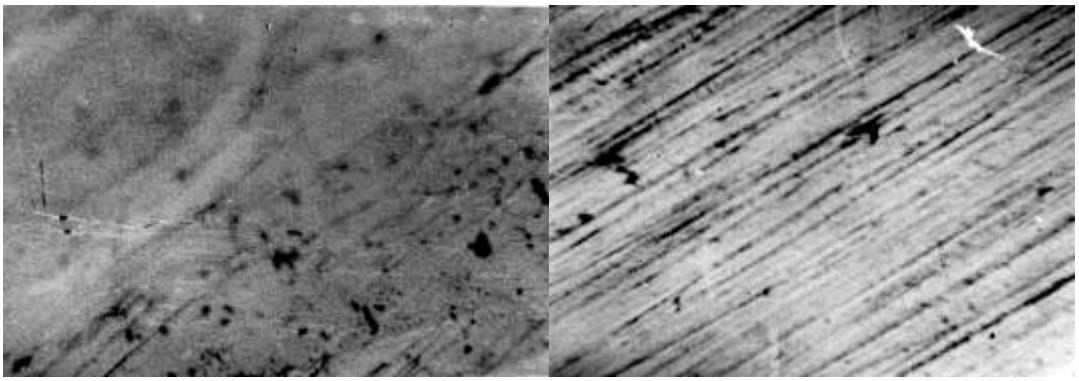


Fig. 4. X-ray diffraction patterns of the alloys of the As_2S_3 -Tm_2S_3 system (before annealing) 1-5 mol%, 2-10 mol%, 3-20 mol% Tm_2S_3

The XRD results showed that there were no intense X-ray diffraction lines on the diffraction patterns of alloys from the glass formation area (Fig. 4).

Single-turbid phase appeared from the microstructural analysis; micrographs of the alloys are shown in Fig. 5.



a) b)

Fig. 5. Microstructures of the alloys of the As_2S_3 -Tm_2S_3 systems
a) 5 mol% As_2S_3 b) 15 mol% Tm_2S_3

Results of integrated methods of physicochemical analysis showed that one incongruently melting compound of $TmAsS_3$ composition was formed in the system at the temperature of 1123K according to the following reaction:

$$L+\gamma\text{-}Tm_2S_3 \overset{1123}{\Longleftrightarrow} TmAsS_3$$

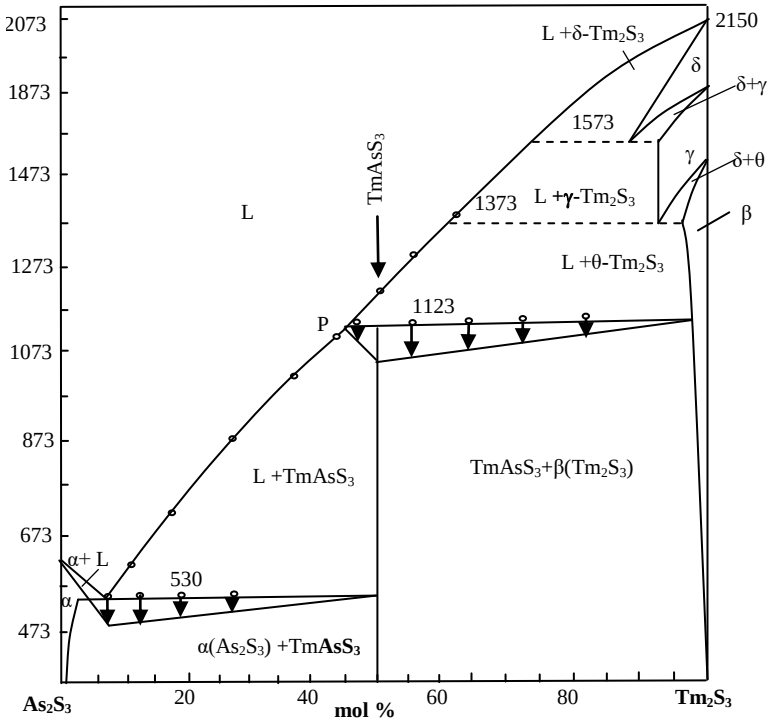


Fig. 6. Phase diagram of the As_2S_3 -Tm_2S_3 system

AsSe –Tm cross section

The alloys were synthesized by the direct ampoule method; the synthesis mode was stepwise. The systems based on AsSe were annealed at the temperature of 475K for 500 hours, and as a result of that alloys were obtained. The phase diagram of AsSe – Tm cross section was plotted on the basis of the results obtained from the integrated methods of PCA. As is seen from Fig.7, one congruently melting compound of TmAsSe composition was formed in the system at 1125K. The solubility range of up to 2 mol Tm % was found on the basis of AsSe.

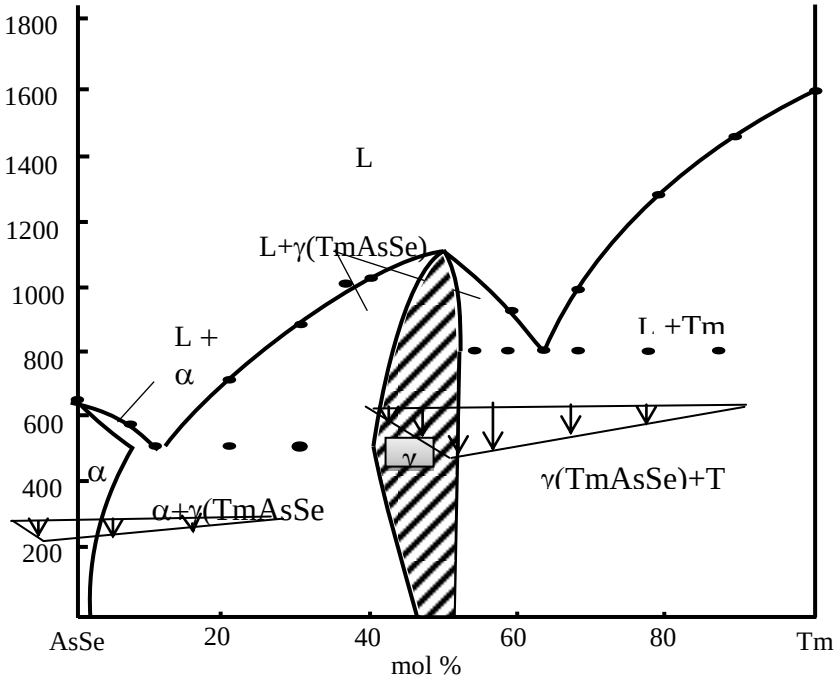


Fig. 7. Phase diagram of the AsSe –Tm system

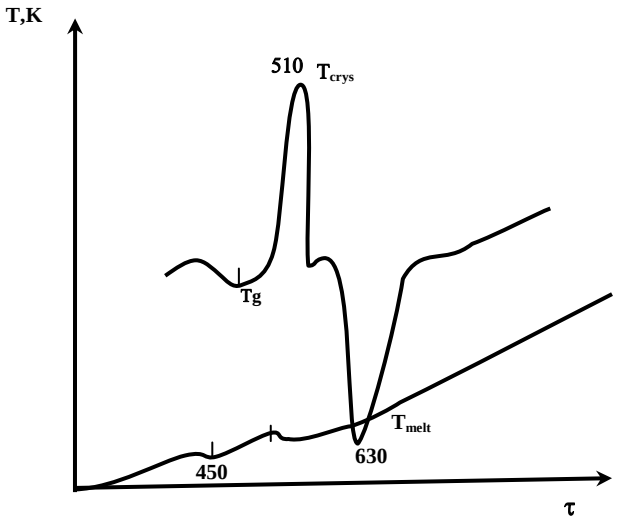


Fig. 8. Thermogram of alloys of $[As_2Se_3]_{0.90}[TmSe]_{0.1}$ composition

As_2Se_3 –TmSe cross section

Alloys of this system were synthesized similarly to the above systems and cooled with the rate of v=7-10 deg/min.

Alloys of up to 30-40 mol% TmSe were obtained in the form of a black powder; while alloys of 12 mol% TmSe in the form of conchoidal fracture. The obtained alloys were investigated by the integrated methods of physical-chemical analysis. The study showed that these alloys were glasses. Thermograms of the alloys containing 10 mol% TmSe are shown in Fig.8.

Results of XRD analysis are shown in Fig.9.

As is seen from the diffraction pattern of alloys of the As_2Se_3-TmSe system, there are no intense diffraction lines special to crystalline substances (Fig. 9)

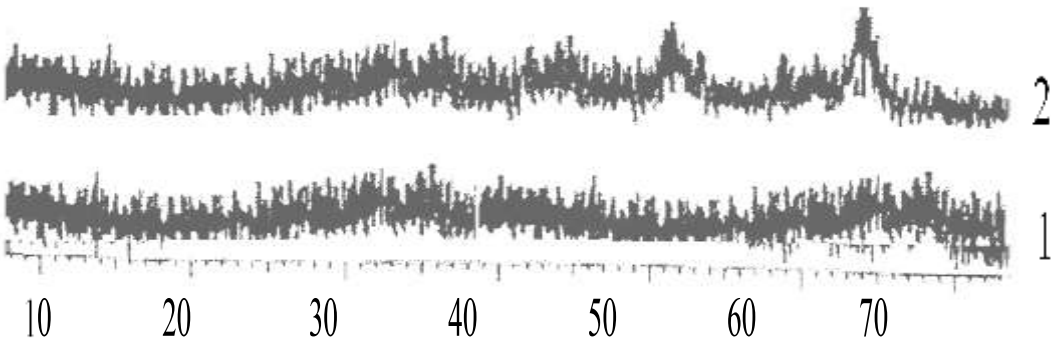


Fig. 9. Diffractograms of alloys of 1) $[As_2Se_3]_{0.90}[TmSe]_{0.1}$ 2) $[As_2Se_3]_{0.85}[TmSe]_{0.15}$ systems

Table 6. Some physicochemical properties of the alloys of As_2Se_3-TmSe system (Rate of cooling v=7-10 deg/min)

№	Composition of alloys		Thermal effects T,K			Microhardness H_{μ}, g/mm^2	Density d, g/cm^3	Results of MSA
	As_2Se_3	TmSe	T_g	T_{crys}	T_{melt}			
1	100	0	450	-	650	130	4.58	Glass
2	99	1	456	520	645	130	4.61	Glass
3	97	3	465	515	640	135	4.70	Glass
4	95	5	476	500	635	140	4.75	Glass
5	90	10	480	510	630	148	4.83	Glass
6	85	15	485	518	615	155	4.85	Glass
7	80	10	493	500	610	160	4.95	Crystalline glass

After providing the equilibrium state for the alloys, they were studied again by means of the physicochemical analysis, the phase diagram of the As_2Se_3-TmSe system was plotted (Fig. 10).

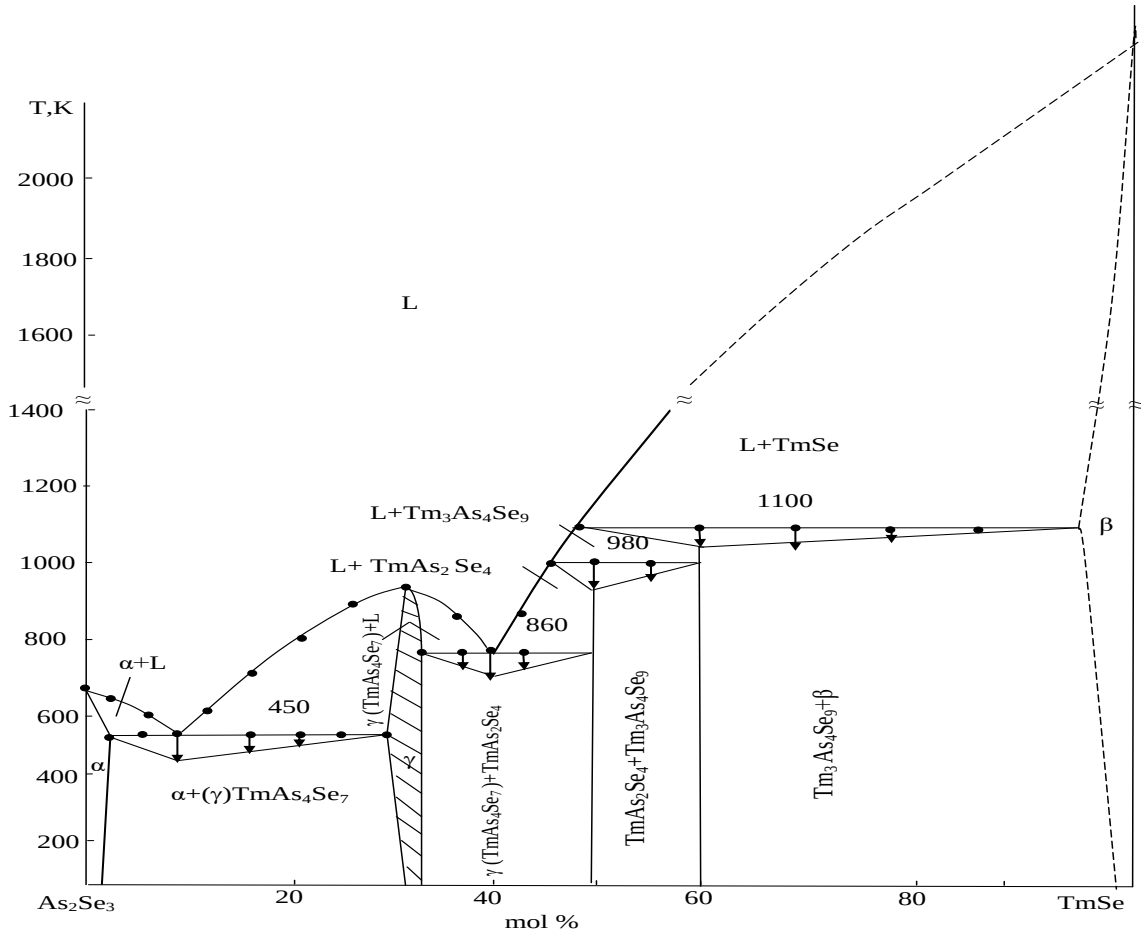


Fig. 10. Phase diagram of the As_2Se_3 –TmSe system

Three intermediate phases $Tm_3As_4Se_9$, $TmAs_2Se_4$ and $TmAs_4Se_7$ are formed in the system. $TmAs_4Se_7$ melts congruently, while $TmAs_2Se_4$ and $Tm_3As_4Se_9$ melt incongruently.

As_2Se_3 –Tm_2Se_3 cross section

Synthesis of the alloys of this system was carried out similarly to the alloys of the As_2Se_3 –TmSe system. The synthesis process was stepwise: firstly, the temperature of the furnace was raised to 750K, then up to 900-1000K; the alloys were kept at this temperature for 2 hours, and then they were cooled together with the furnace. After synthesis, the alloys were obtained in the compact form up to 70% Tm_2Se_3, and then they turned into the spec form and had porous. The alloys were annealed at the temperature of 750K for 300 hours to provide the equilibrium state.

The phase diagram of the As_2Se_3 – Tm_2Se_3 cross section was plotted using the results of physical and chemical analysis (Fig. 11).

As is seen from the Fig. 11, the cross section is a quasi-binary section of the Tm-As-Se ternary system.

After the separation of the intermediate phases, their temperature dependence of the electrical conductivity was determined individually (Fig. 12,13). It has been established that they are p-type semiconductors.

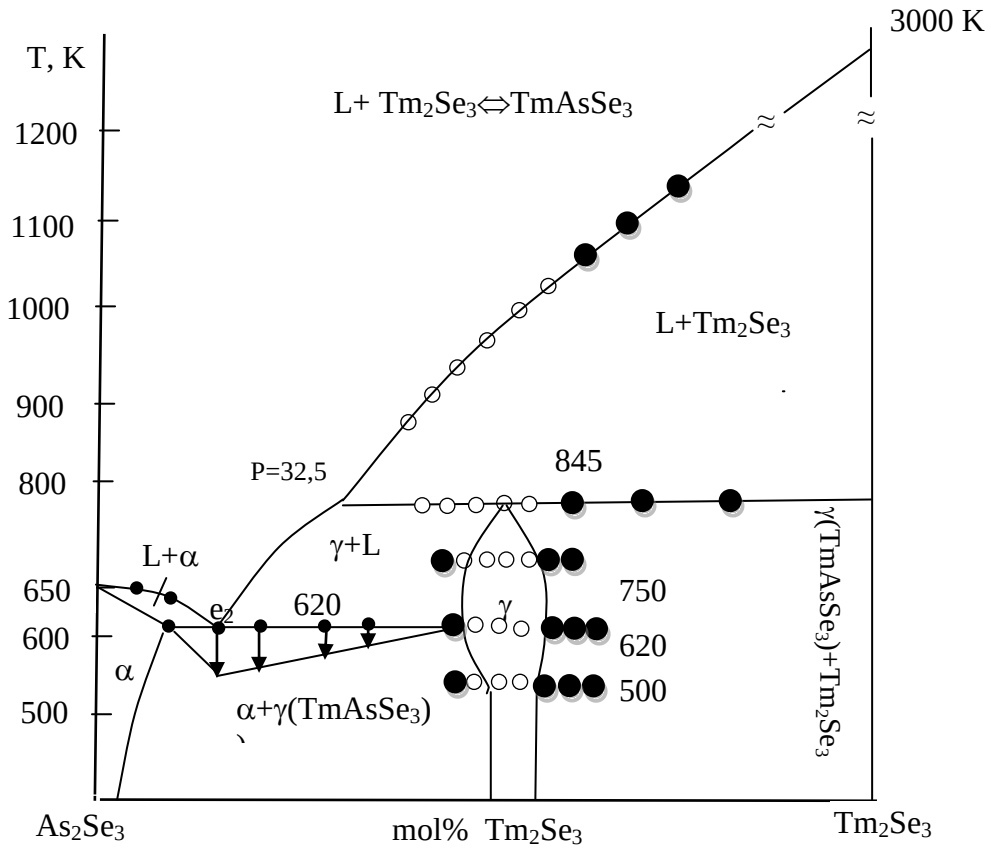


Fig. 11. Phase diagram of the As_2Se_3 – Tm_2Se_3 system

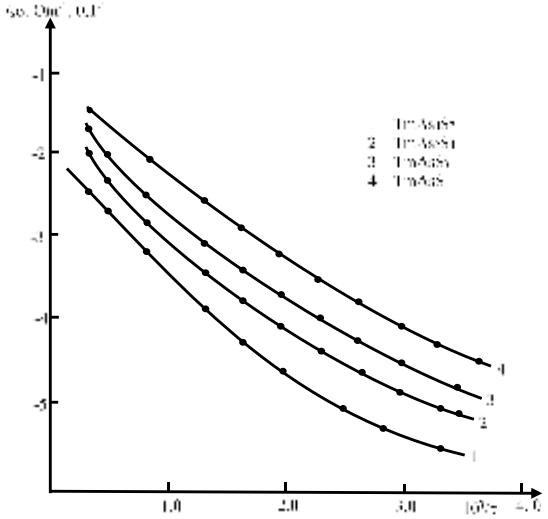


Fig. 12. Temperature dependence of the electrical conductivity of sulfide compounds

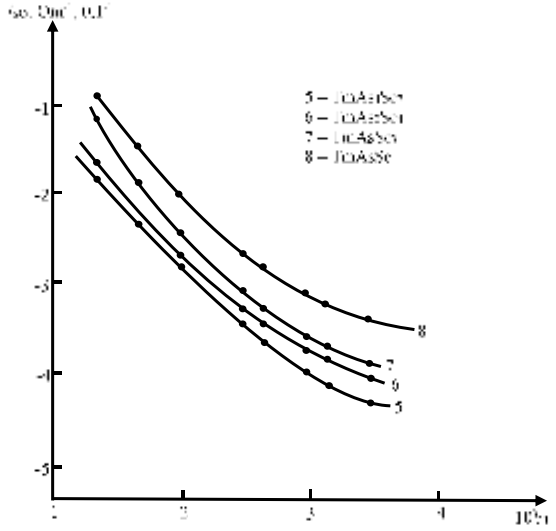


Fig. 13. Temperature dependence of the electrical conductivity of selenide compounds.

Conclusions

1. When studying the cross sections of the Tm-As-S and Tm-As-Se ternary systems, glass formation areas were revealed in them and their boundary of existence was outlined.
2. When studying Tm-AsS, Tm_2S_3-As_2S_3, TmS-As_2S_3 Tm-AsSe, TmSe-As_2Se_3, Tm_2Se_3-As_2Se_3 cross sections, intermediate phases TmAsS, $TmAsS_3$, $TmAs_4S_7$, $TmAs_2S_4$, $Tm_3As_4S_9$, TmAsSe, $TmAs_4Se_7$, $TmAs_2Se_4$, $Tm_3As_4Se_9$ and $TmAsSe_3$ also appeared in these cross sections; their formation and melting nature was determined. It was established that the cross sections were quasi-binary sections of the Tm-As-S and Tm-As-Se ternary systems.
3. Parameters of the crystal lattice and intermediate phases have been calculated. It is established that they crystallize in a rhombic syngony.
4. Study into the electrical conductivity of the glasses and intermediates revealed that they are semiconductors of p-type conductivity.

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Tm-As-S VƏ Tm-As-Se SİSTEMLERİNDƏ ŞÜŞƏLƏŞMƏ, FAZA TARAZLIĞI VƏ ARALIQ FAZALARIN XASSƏLƏRİ

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Xülasə*: Tm-As-S və Tm-As-Se sistemlərində fiziki-kimyəvi qarşılıqlı təsirin xarakteri müxtəlif en kəsiylər boyunca Diferensial Termiki (DTA), X-şüalarının difraksiyası (XRD) və Mikroquruluş (MQA) analizləri ilə öyrənilmiş, onların mikrosərtlikləri və elektrofiziki xassələri müəyyən edilmişdir. Sistemin ərintilərinin tədqiqinə 7-10dər/dəq soyutma rejimlərində, ilkin komponentlərin xassələrinin öyrənilməsi ilə başlanmışdır. Sistemlərdə şüşə əmələgəlmə sahəsinin sərhədi müəyyən edildikdən sonra şüşələrin və aralıq fazaların fiziki-kimyəvi xassələri tədqiq edilmişdir. $TmAsS_3$, $TmAs_4S_7$, $TmAs_2S_4$, $Tm_3As_4S_9$ birləşmələri As_2S_3-Tm_2S_3, As_2S_3-TmS sistemlərində əmələ gəlir. $TmAs_4S_7$, TmAsS və $TmAs_2S_4$ kompozisiyalarının birləşmələri konqruent olaraq əriyir, $Tm_3As_4S_9$ və $TmAsS_3$ birləşmələri isə inkonqruent əriyir. Tm-As-Se sistemində TmAsSe, $TmAsSe_3$, $TmAs_4Se_7$, $TmAs_2Se_4$ və $Tm_3As_4Se_9$ fazaları əmələ gəlir. $Tm_3As_4Se_9$ peritektik reaksiya ilə $TmSe + L \leftrightarrow Tm_3As_4Se_9$ əmələ gəlir. Mikroquruluş təhlili göstərdi ki, aralıq fazaların tərkibi istisna olmaqla, Tm-As-S, Tm-As-Se kəsiklərinin bütün ərintiləri ikifazalıdır. XRD-nin nəticələrinə əsasən TmAsS, $TmAsS_3$, $TmAs_4S_7$, $TmAs_2S_4$-ün qəfəs parametrləri, həmçinin selen tərkibli birləşmənin parametrləri hesablanmışdır. Müəyyən edilmişdir ki, birləşmələr rombik sinqoniyada kristallaşırlar. Tədqiqat nəticələrinə əsasən müəyyən edilmişdir ki, tədqiq olunan kəsiklər Tm-As-S və Tm-As-Se üçlü sistemlərinin kvazibinar kəsikləridir. Fiziki-kimyəvi analizin nəticələrindən istifadə etməklə kükürd və selenin iştirakı ilə kəsiklərin faza diaqramı qurulmuşdur; müəyyən edilmişdir ki, tədqiq olunan kəsiklər Tm-As-S və Tm-As-Se üçlü sistemlərinin kvazibinar kəsikləridir.*

Açar sözlər*: analiz, kəsik, mikrobərklik, ərinti, temperatur, kristallaşma*

СТЕКЛООБРАЗОВАНИЕ И ФАЗОВЫЕ РАВНОВЕСИЯ В СИСТЕМАХ Tm-As-S И Tm-As-Se И ИХ СВОЙСТВА

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Аннотация: *Методами дифференциально-термического (ДТА), рентгенофазового (РФА) и микроструктурного (МСА) анализов, измерением микротвердости и электрофизических свойств изучен характер физико-химического взаимодействия в системах Tm-As-S и Tm-As-Se по различным разрезам. Исследование сплавов системы было начато изучением свойств исходных компонентов в режимах охлаждения со скоростью 7-10 град/мин. После установления в системах границы области стеклообразования изучали физико-химические свойства стекол и промежуточных фаз. Анализ термограмм показал, что все эффекты на термограммах обратимые. В системах As_2S_3-Tm_2S_3, As_2S_3-TmS образуются соединения состава $TmAsS_3$, $TmAs_4S_7$, $TmAs_2S_4$, $Tm_3As_4S_9$. Соединения $TmAs_4S_7$, TmAsS, $TmAs_2S_4$ плавятся конгруэнтно, а соединения $Tm_3As_4S_9$ и $TmAsS_3$ - инконгруэнтно. В системе Tm-As-Se выявлено образование фаз TmAsSe, $TmAsSe_3$, $TmAs_4Se_7$, $TmAs_2Se_4$ и $Tm_3As_4Se_9$. $Tm_3As_4Se_9$ образуется по перитектической реакции $TmSe + Ж \leftrightarrow Tm_3As_4Se_9$.*

Микроструктурный анализ показал, что все сплавы разрезов Tm-As-S, Tm-As-Se двухфазные кроме состава промежуточные фаз. По результатам РФА рассчитаны параметры решетки соединений TmAsS, $TmAsS_3$, $TmAs_4S_7$, $TmAs_2S_4$ а также соединений с участием селена и установлено, что они кристаллизуется в ромбическом сингонии. По результатам физико-химического анализа построена диаграмма состояния разрезов с участием серы и селена и установлено, что исследованные разрезы являются квазибинарными сечениями тройных систем Tm-As-S и Tm-As-Se.

Ключевые слова: *анализ, разрез, микротвердость, сплав, температура, кристаллизация*