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## PHYSICO-CHEMICAL AND X-RAY DIFRACTION INVESTIGATION OF SOLID SOLUTIONS $(PbS)_{1-x}(YbGa_2S_4)_x$

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**Abstract**

To determine the region of solid solutions $(PbS)_{1-x}(YbGa_2S_4)x$ (x=0.01; 0.02; 0.03) complex methods of physicochemical analysis were used: differential thermal analysis (DTA), X-ray phase analysis (XRD), microstructural analysis (MSA), as well as measuring microhardness and density. The nature of interaction between PbSe and $YbGa_2S_4$ was studied, it was found that introduction of $YbGa_2S_4$ into PbS leads to formation of solid solutions and change of some physicochemical properties. It was found that solid solutions based on PbSe at room temperature reach 3% $YbGa_2S_4$. For alloys of solid solution $(PbS)_{1-x}(YbGa_2S_4)_x$ (x = 0.01; 0.02; 0.03) 2 and 3 mol. % $YbGa_2S_4$ the X-ray diffraction data were studied and lattice parameters were calculated. It was established that the obtained alloys crystallize with cubic syngony with lattice parameters: *a*= 6.03 and *a*=6.10 Å, respectively

**Keywords:** solid solution, density, component, microhardness, density

### Introduction

The creation of new multicomponent semiconductor materials based on lead chalcogenides with specific properties requires obtaining reliable information on phase equilibria in the corresponding systems.

Lead sulfide has a high permittivity, an absorption coefficient in the visible and near infrared spectral regions of ~ 105 cm-1) [1], as well as the possibility of significantly expanding the band gap from 0.4 to 5.2 eV [2], and is of interest to fundamental researchers. The PbS compound has a wide range of functional properties and is used in various fields, such as lasers [3], infrared (IR) detectors [4], optical switches and amplifiers [5].

In work [6] the luminescent properties of the compound of the composition $YbGa_2S_4$:5%$Er^{+3}$ were investigated. Compounds of the composition $YbGa_2S_4$:5%$Er^{+3}$, activated by other trivalent rare earth ions, are promising for optical excitation of activated centers through fundamental absorption bands [7-9].

The aim of this work is to synthesize alloys of solid solutions $(PbS)_{1-x}(YbGa_2S_4)x$ (x = 0.01; 0.02; 0.03) and to study their physicochemical and X-ray studies.

The following information is available for the main components. The compound PbS melts congruently at 1113°C and crystallizes in the form of a cubic syngony with the lattice parameters: *a*=5.935 Å, Z=4, sp. gr. Fm3m-$O^5_h$, density ρ=7.68 g/cm$^3$, microhardness Hμ=720 MPa [10]. The compound $YbGa_2S_4$ melts at 1180°C with an open maximum and crystallizes in the orthorhombic syngony with the lattice parameters: *a*=20.14; *b*=20.12; *c*=12.15 Å, sp. gr. $D^{24}_{2h}$-Fddd, density ρ=5.12 g/cm$^3$, microhardness Hμ=2250 MPa [11].

### Experimental part

The synthesis of the initial compounds and alloys of the studied section was carried out by melting high-purity elemental components in quartz ampoules evacuated to 0.133 Pa at a temperature of 700-1200°C. Yb-99.98 %, Pb -99.998 %, Ga-99.999 %, sulfur of special purity grade were used as initial elemental substances. For homogenization of alloys, annealing was carried out at a temperature of 600°C for 240 hours.

The interaction in the $(PbS)_{1-x}(YbGa_2S_4)_x$ system was studied by methods of differential-thermal analysis (DTA), X-ray phase analysis (XPA), microstructural analysis (MSA), as well as measuring microhardness and determining density.

DTA of the alloys of the system was carried out on an NTR-73 device with a heating rate of 9 deg/min. Chromel-alumel thermocouples were used for calibration, and $Al_2O_3$ was used as a standard. X-ray analysis was performed on a D2 PHASER X-ray device using CuKα radiation and a Ni filter. The MSA of the system's alloys was studied using a MIM-8 metallographic microscope on pre-etched sections polished with GOI paste. When studying the microstructure of the alloys, an etchant of the composition conc. $HNO_3$: $H_2O_2$ = 1:1 was used, the etching time was 20 s. The microhardness of the system's alloys was measured on a PMT-3 microhardness tester under loads of 0.10 and 0.20 N. The density of the system's alloys was determined by the pycnometric method, with toluene used as the working fluid.

### Results and discussion

Solid solution alloys $(PbS)_{1-x}(YbGa_2S_4)_x$ (x= 0.01; 0.02; 0.03) are obtained in the form of compact light-black ingots. The alloys are stable with respect to air and water. Concentrated mineral acids (HCl, $HNO_3$)

decompose them. Annealed alloys were studied by physicochemical methods.

DTA of the alloys of the system shows that two endothermic effects related to solidus and liquidus were found on the thermograms of the alloys. When studying the microstructure, it was revealed that the alloys of the system $(PbS)_{1-x}(YbGa_2S_4)_x$ (x= 0.01; 0.02; 0.03) in the range of 0-3 mol % $As_2S_3$ are single-phase, the remaining alloys are two-phase.

To confirm the results of DTA, MSA, X-ray phase analysis was performed. In Fig. 1 shows the X-ray diffraction patterns of the alloys of solid solutions $(PbS)_{1-x}(YbGa_2S_4)_x$ (x = 0.01; 0.02; 0.03). As can be seen from Fig. 1, the diffraction lines on the diffraction pattern of the sample of 2 and 3 mol % $YbGa_2S_4$ are identical to the X-ray diffraction pattern of the PbS compound, i.e. the sample is a solid solution based on PbS. On the diffraction pattern of the alloy of 5 mol % $YbGa_2S_4$, the diffraction lines consist of a mixture of the diffraction lines of the original compounds PbS and $YbGa_2S_4$. This shows that the alloy of 5 mol % $YbGa_2S_4$ is two-phase.

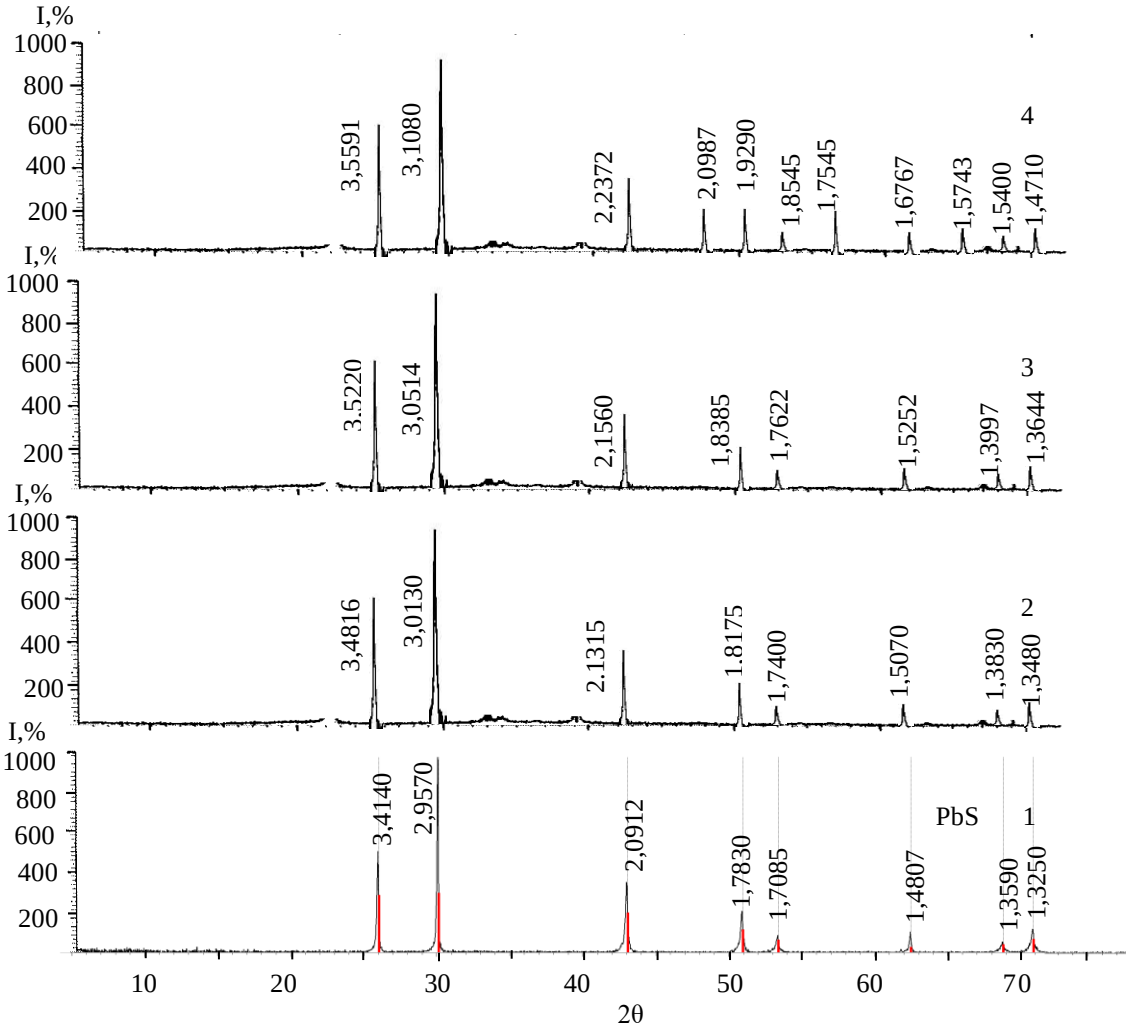


Fig. 1. Diffraction patterns of solid solution alloys $(PbS)_{1-x}(YbGa_2S_4)_x$ (x = 0.01; 0.02; 0.03). 1-PbS, 2-2 mol %, 3-3 mol %, 4-5 mol % $YbGa_2S_4$.

Table 1.

Compositions, DTA results, microhardness measurement values and density determination of solid solutions $(PbS)_{1-x}(YbGa_2S_4)_x$.

| Composition mol % | | Thermal effects, ˚C | Density 10 q/cm$^3$ | Microhardness, MPa | |
|---|---|---|---|---|---|
| PbS | $YbGa_2S_4$ | | | α | $YbGa_2S_4$ |
| | | | | P=0,10N | P=0,20 N |
| 100 | 0,0 | 1113 | 7,68 | 720 | - |
| 98 | 2,0 | 1105 | 7,65 | 730 | - |
| 97 | 3,0 | 1095,1100 | 7,60 | 735 | - |
| 95 | 5,0 | 1080,1095 | 7,54 | 740 | - |
| 93 | 7,0 | 1070,1090 | 7,50 | 760 | - |
| 90 | 10 | 1050,1070 | 7,40 | 790 | - |

The values of the dependences of some physicochemical properties of solid solutions $(PbS)_{1-x}(YbGa_2S_4)_x$ (x = 0.01; 0.02; 0.03). on the amount of $YbGa_2S_4$ are given in Table 1. When $YbGa_2S_4$ is introduced into the composition of PbS, the microhardness increases compared to pure PbS (720 MPa). In the region of the solid solution $(PbS)_{1-x}(YbGa_2S_4)_x$ (x = 0.01; 0.02; 0.03) for alloys of 2 and 3 mol % $YbGa_2S_4$, the lattice parameters were calculated (Table 2). It was found that the obtained alloys crystallize with cubic syngony with the lattice parameters: $a$ = 6.03 and $a$ = 6.10 Å, respectively.

Table 2.

X-ray data of solid solution alloys $(PbS)_{1-x}(YbGa_2S_4)_x$

| 98 % PbS+ 2 % $PbGa_2S_4$ | | | | 98 % PbS +3 % $PbGa_2S_4$ | | | |
|---|---|---|---|---|---|---|---|
| I, % | d, $_{exp.}$Å | d, $_{cal.}$Å | hkl | I, % | d,$_{exp.}$Å | d,$_{cal.}$Å | Hkl |
| 55 | 3,4816 | 3,4815 | 111 | 55 | 3,5220 | 3,5223 | 111 |
| 100 | 3,0130 | 3,0151 | 200 | 100 | 3,0514 | 3,0514 | 200 |
| 30 | 2,1315 | 2,1320 | 210 | 30 | 2,1560 | 2,1572 | 210 |
| 15 | 1,8175 | 1,8182 | 311 | 15 | 1,8385 | 1,8393 | 311 |
| 5 | 1,7400 | 1,7408 | 222 | 5 | 1,7622 | 1,7612 | 222 |
| 3 | 1,5070 | 1,5075 | 400 | 3 | 1,5252 | 1,15252 | 400 |
| 2 | 1,3830 | 1,3834 | 331 | 2 | 1,3997 | 1,3994 | 331 |
| 5 | 1,3480 | 1,3484 | 420 | 5 | 1,3644 | 1,3641 | 420 |

**Conclusion**

In order to determine the region of PbS-based solid solutions in the PbS-$YbGa_2S_4$ system, alloys containing 1, 2, 3, and 5 mol. % $YbGa_2S_4$ were synthesized in the temperature range of 1000-1200°C. Using the methods of physicochemical analysis: differential thermal analysis (DTA), X-ray phase analysis (XRD), microstructural analysis (MSA), as well as density and microhardness measurements, it was established that at room temperature, PbS-based solid solutions reach 3 mol % $YbGa_2S_4$. As a result of X-ray structural analysis, the parameters of the alloys of solid solutions containing 2 and 3 mol % $YbGa_2S_4$ were calculated. The obtained alloys crystallize with cubic syngony with the lattice parameters: $a$ = 6.03 and $a$ = 6.10 Å, respectively. In the concentration range of 0-10 mol % $YbGa_2S_4$ alloys of the PbS-$YbGa_2S_4$ system were studied for microhardness and density depending on the composition.

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