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SYNTHESIS AND CRYSTAL STRUCTURE OF SOLID SOLUTIONS AND THIN FILMS OF THE $Mn_{1-x}Tm_xSe$ SYSTEM

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Abstract. *Solid solutions of the $Mn_{1-x}Tm_xSe$ ($0 \le x \le 0.7$) system with the cubic structure of the $Fm\bar{3}m$ space group have been obtained. An increase in the concentration of Tm cations in $Mn_{1-x}Tm_xSe$ solid solutions leads to an increase in the parameter a form 0.547 nm for the $Mn_{0.975}Tm_{0.025}Se$ composition to 0.566 nm for the $Mn_{0.3}Tm_{0.7}Se$ composition. Thin films of solid solutions $Mn_{1-x}Tm_xSe$ (x=0.05; 0.1; 0.3) were synthesized by the "flash" method on standard substrates of optically transparent glass. It has been established that the film thicknesses are in the range of values: from 0.8 µm to 3.2 µm*

Keywords: *$Mn_{1-x}Tm_xSe$, solid solutions, cation, concentration, thin films, optically transparent glass, synthesis.*

INTRODUCTION

The search and study of new substances that exhibit the properties of multiferroics and a strong relationship between the magnetic, electronic, and elastic subsystems, which manifests itself in the form of magneto electric and magneto resistive effects at room temperatures, is of interest from both fundamental and applied points of view. This will make it possible to create efficient sensors and element base in microelectronics, in particular in spintronic, which uses the advantages of both non-volatile magnetic memory and high-speed electrical information processing systems. In spintronic, to convert an electrical signal, not only the charge degree of freedom of an electron, but also the spin is used, which makes it possible to create fundamentally new spintronic devices. The electron has orbital degrees of freedom, by acting on which it is also possible to regulate the transport and dielectric characteristics in a magnetic field. Semiconductors with inhomogeneous electronic states and with orbital ordering of electrons can be used as such materials. The transfer of charge carriers is carried out along a certain type of orbitals, the position of which relative to the chemical potential can be varied by electric, magnetic fields and elastic stresses. The electronic structure and electric current in systems with orbital charge ordering can be controlled by elastic stresses based on the piezoelectric effect. Magneto electric materials can be used in non-volatile magnetic memory, in microwave devices. Promising materials for solving these problems are magnetic semiconductors based on manganese chalcogenides.

MATERIALS AND METHODS.

Synthesis of polycrystalline compositions and thin films of the system $Mn_{1-x}Tm_xSe$

Solid solutions $Mn_{1-x}Tm_xSe$ are obtained by solid-phase reactions from powders of initial chemical elements in evacuated quartz ampoules in a single-zone resistance furnace. Powders of manganese, thulium, and selenium, calculated and weighed in the required quantities, were thoroughly mixed and loaded into quartz ampoules, followed by evacuation to 10^{-2} Pa. Since selenium has a boiling point of 950 K, above which the vapor pressure sharply increases, a stepwise synthesis regime was adopted to avoid the explosion of the ampoules. For the first 24 hours, the temperature was maintained at 670–720 K. Then, for 70 hours, the ampoules were kept at a temperature of 870–970 K. After that, the temperature rose to 1370 K and was maintained at this level for 2 hours. This was followed by hardening of the ampoules from this temperature into cold water. Substances synthesized in this way were subjected to homogenizing annealing. The resulting ingots are greyish-silver in color and are quite hard.

Thin layers of different thicknesses of $M_{n1-x}Tm_xSe$ solid solutions were also synthesized. The films were synthesized by the method of thermal evaporation on an industrial plant.

The deposition of $M_{n1-x}Tm_xSe$ films was carried out by the “flash” thermal evaporation method with partial supply of a powder of the evaporated substance with a grain size of 0.1-0.3 mm to a tantalum evaporator having a temperature of ~2000°C. The patented development of the Laboratory of Physics of Magnetic Materials of the State Scientific and Practical Center of the National Academy of Sciences of Belarus for Materials Science, which ensures the production of thin layers’ uniform in thickness and composition on various substrates, was used [1].

Standard glass plates 0.5 x 18 x 18 (mm^3) of optically transparent glass served as substrates in the synthesis of $M_{n1-x}Tm_xSe$ films. Films of solid solutions $M_{n1-x}Tm_xSe$ (x=0.05; 0.1; 0.3) with a thickness of 0.8 - 3.2 μm were obtained.

After each stage of synthesis, X-ray diffraction studies were carried out in Cu*K*α - radiation in order to identify the optimal, energy-saving conditions for the synthesis of new materials that are promising for practical applications in instrumentation.

RESULTS AND DISCUSSION.

Study of the crystal structure and phase state of solid solutions of the $M_{n1-x}Tm_xSe$ system.

Manganese monoselenide has a cubic system of the NaCl type, space group $Fm\bar{3}m$ with a unit cell parameter a = 0.5456 nm. It is known that thulium selenide also crystallizes in the cubic syngony of NaCl type, space group $Fm\bar{3}m$ with unit cell parameter a=0.564 nm. Melting point Tm. ~ 2060ºC. At temperatures ~ 1730°C and ~ 1100°C, crystalline phase transformations of the second kind take place. It has been established that the compound is an antiferromagnetic with a Neel temperature of about 3 K. A difference of ~4% in the unit cell parameters suggests the possibility of the existence of solid solutions in the MnSe – TmSe quasi-binary section. This justifies the synthesis, study of the crystal structure and phase state of solid solutions of the $M_{n1-x}Tm_xSe$ system obtained under various conditions of reactions in the solid phase and annealing of samples after primary sintering [2-8].

Below are the results of studying the crystal structure of solid solutions of the $M_{n1-x}Tm_xSe$ (x=0.025; 0.05; 0.075; 0.1; 0.3; 0.5) system at various substitution concentrations in the cationic sub lattice after homogenizing annealing (Figure 1).

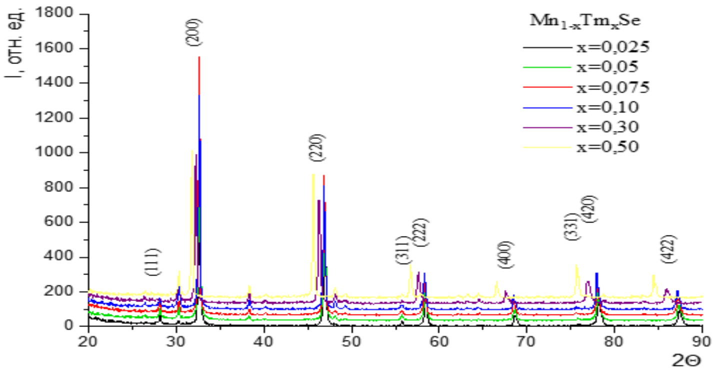

Figure 1. X-ray patterns of solid solutions of the $M_{n1-x}Tm_xSe$ system after homogenizing annealing.

Solid solutions of the $M_{n1-x}Tm_xSe$ ($0 \leq x < 0.7$) system with the cubic structure of the space group $Fm\bar{3}m$ were obtained using the solid-phase synthesis technique. Based on the results of the analysis of X-ray diffraction patterns, the parameter α of the cubic unit cell was determined.

It has been established that an increase in the concentration of Tm cations in $M_{n1-x}Tm_xSe$ solid solutions leads to an increase in the parameter a form 0.547 nm for the $Mn_{0.975}Tm_{0.025}Se$ composition to 0.566 nm for the $Mn_{0.3}Tm_{0.7}Se$ composition. Accordingly, the volume of an elementary crystal cell also increases.

The main prerequisites and reasons for limiting the solubility limits are: a rather large difference in the melting points of manganese and thulium; different activity of chemical interaction of Se chalcogen with Mn and Tm cations; the difference in the ionic radii of Mn and Tm; the presence of multivalent states in Mn; preferential formation of the $TmSe_2$ phase during diffusion processes of high-temperature synthesis.

Study of the Features of the Crystal Structure and Composition of Thin Films of the $M_{n1-x}Tm_xSe$ System.

The study of the crystal structure of $M_{n1-x}Tm_xSe$ thin films was carried out in CuKα radiation using a modified DRON-2 diffractometer in the mode of taking X-ray patterns by points in the angle range $20° \leq 2\theta \leq 90°$ at room temperature. The diffraction patterns of all compositions were obtained in the shooting mode: the time of collecting information at the point $\Delta\tau = 3$ sec., the scanning step along the angle $\Delta 2\theta = 0.03$ degrees.

Figure 2, using the x=0.05 composition as an example, shows X-ray patterns of thin films of $\Delta\tau$ solid solutions.

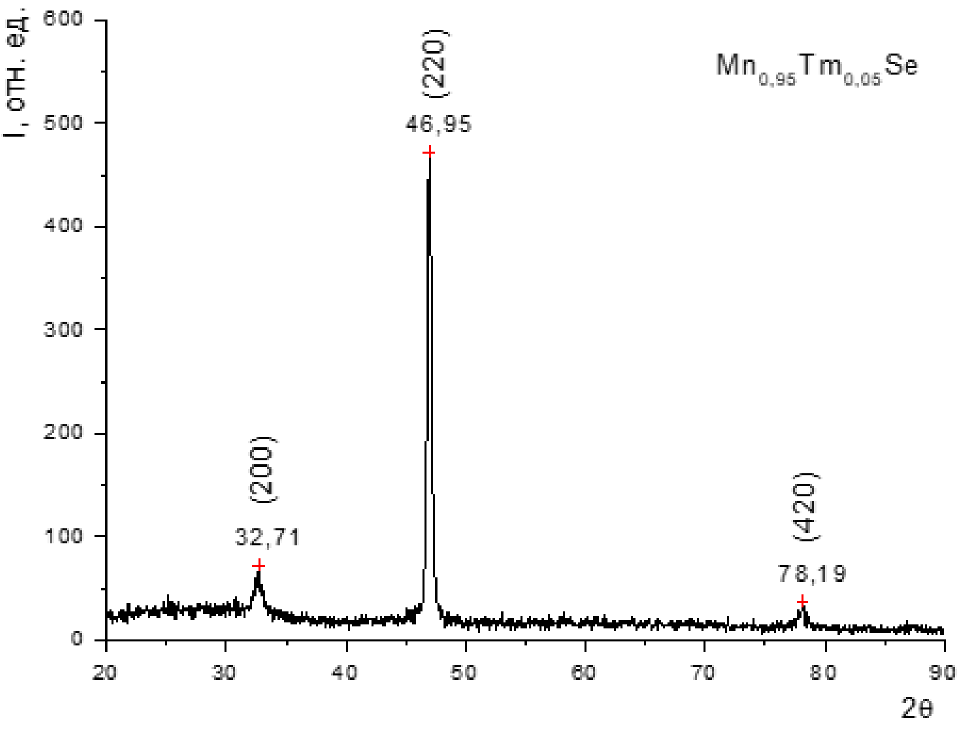

Figure 2. X-ray of a thin film $Mn_{0.95}Tm_{0.05}Se$

X-ray analysis showed that thin films of $M_{n1-x}Tm_xSe$ (x=0.05; 0.1; 0.3) have the same crystal structure of the NaCl type, space group $Fm\bar{3}m$. Reflections of diffractograms are indexed in accordance with the database PCPDFWIN 1998, V.2.00 [9-11].

The surface morphology and chemical composition of the films were studied by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). The results showed that the films are homogeneous without any significant structural damage. The composition of the Mn1-xTmxSe thin films corresponds to the chemical composition of the initial powders, in particular, this is evidenced by the results of the analysis of the composition of the $Mn_{0.95}Tm_{0.05}Se$ film (Figure 3).

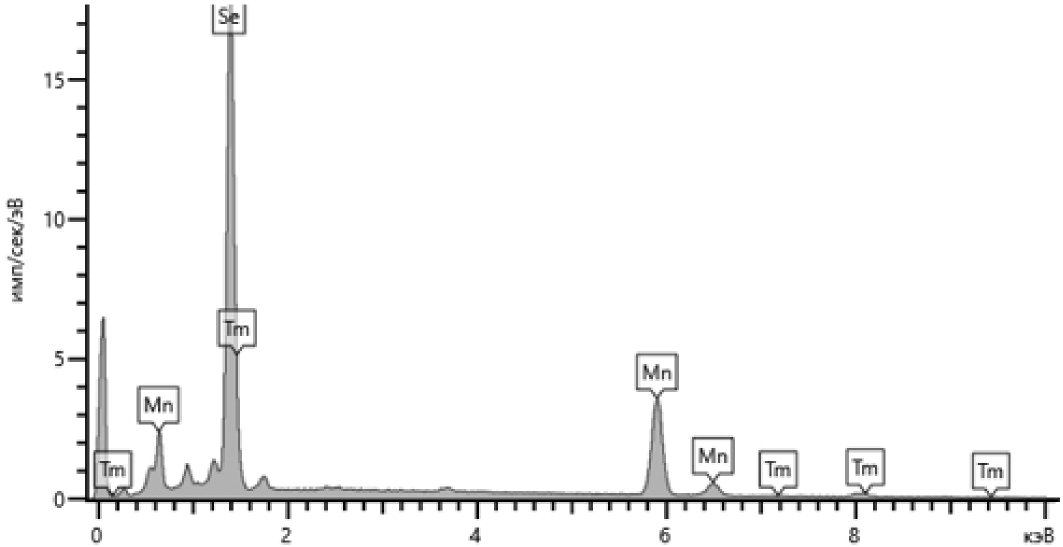

Figure 3. Electron microanalysis of the $Mn_{0.95}Tm_{0.05}Se$ film

CONCLUSION

Solid solutions of the Mn1-xTmxSe (0≤x≤0.7) system with the cubic structure of the space group $Fm\overline{3}m$ were obtained using the solid-phase synthesis technique. The crystal structure of solid solutions of the $M_{n1-x}Tm_xSe$ system has been studied by X-ray phase analysis. An increase in the concentration of Tm cations in $M_{n1-x}Tm_xSe$ solid solutions leads to an increase in the parameter a form 0.547 nm for the $Mn_{0.975}Tm_{0.025}Se$ composition to 0.566 nm for the $Mn_{0.3}Tm_{0.7}Se$ composition.

Thin films of Thin films of $M_{n1-x}Tm_xSe$ solid solutions were synthesized using the flash method on standard substrates of optically transparent glass. It has been established that the film thicknesses are in the range of values: from 0.8 μm to 3.2 μm solid solutions were synthesized using the flash method on standard substrates of optically transparent glass. It has been established that the film thicknesses are in the range of values: from 0.8 μm to 3.2 μm.

X-ray phase analysis of thin films of Mn1-xTmxSe solid solutions was performed at room temperature in CuKα radiation. It has been established that $M_{n1-x}Tm_xSe$ (x=0.05; 0.1; 0.3) films have a cubic system of the NaCl type, space group $Fm\overline{3}m$. The composition of $M_{n1-x}Tm_xSe$ thin films corresponds to the chemical composition of the initial powders.

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