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NEUTRON DIFFRACTION RESEARCHES OF SPINTRONIC LAYERED SEMICONDUCTOR InSe<Mn>

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Abstract

The analysis of magnetic properties and neutron diffractometry of layered semiconductor InSe doped by Mn was carried out. Neutron diffraction structural researches shown that semiconductor InSe<Mn> consists of several phases namely $In_{1-X}Mn_XSe$, α-MnSe, In_4Se_3. It is a result of low solubility of Mn in InSe. Nonmonotonic temperature dependence of magnetic susceptibility for InSe<Mn> within temperature range 2.4 – 270 K is due to antiferromagnetic properties of α-MnSe cubic phase. Quantitative coincidence is achieved at its content about 0.15% weight in InSe. For the first time the lowering of magnetic sublattice parameter for α-MnSe was discovered. It leads to an appearance of weak ferromagnetism. The assumption of existence selforganization superlattices ferromagnetic $In_{1-X}Mn_XSe$ / antiferromagnetic MnSe is stated.

Keywords: *"Neutron diffractometry", "Diluted magnetic semiconductors", " Spintronics", "Magnetic susceptibility"*

1. INTRODUCTION

Diluted magnetic semiconductors (DMS) refer to new magnetic materials in which a magnetic sublattice is absent. Their appearance provokes the development a new area of electronics, namely spin electronics or spintronics.

Semiconductor spintronics is a science about the coexistence of charge and spin extent of freedom in semiconductors and nanostructures doped with d-transition metals, the nature of their ferromagnetism, the fabrication of devices operating on the basis of spin and electron properties. Nanospintronics studies the behavior of spins in nanoparticles; by now their

indirect interaction has not been well investigated yet. Today the key problem of spintronics is the absence of ferromagnetic materials with required combination of magnetic, semiconductor and optic properties at room temperature.

DMS are attracting more and more attention of scientists. Such materials have been known for about forty years: $Ge_{1-X}Mn_XTe$ since 1966 [1] and $Sn_{1-X}Mn_XTe$ since 1968 [2]. The RKKI mechanism provides exchange interaction between magnetic manganese ions through degenerate hole gas. In papers [3,4] are worth noticing as they first reported the formation of magnetically ordered clusters of impurity ions in layered crystals InSe:Fe at room temperature. Publication [5] is a classical one, it is devoted to investigation of magnetic ordering of impurities in CdTe:Fe. In the mentioned crystals, control over the process of cluster formation from impurity ions and dissociation of magnetically ordered clusters was performed for the first time in [6]. In 1984 ferromagnetism was obtained in solid solutions $Pb_{1-X-Y}Sn_XMn_YTe$ with Tc=10K [7]. These semiconductors were further investigated in detail [8]. Later a ferromagnetic state was found in $Ga_{1-X}Mn_XAs$, where the Curie temperature was increased up to 175 K thanks to improvement of technology [9]. However, over last years the progress has stopped.

Increasing interest in the problem of formation of a ferromagnetic state in semiconductors over 300 K arose after the publication of paper [10], which forecasts the Curie temperature over room temperature in ZnO and GaN doped with manganese provided with the availability of high hole concentration. The authors [11], however, consider this idea to be mistaken.

The high temperature of ferromagnetic transition will make possible to use DMS in devices of spin electronics. Here nontoxic materials with Tc> 350 K that integrate in modern microelectronics technology are preferable.

Therefore layered III-Y1 crystals, characterized by high anisotropy of chemical bond, which consists of strong ion-covalent bonds in the plane of atomic layers and weak Van-der-Waals bonds between layers, attract much attention. This is especially important in the case of InSe where such anisotropy leads to two-dimensional (2D) electronic conductance below critical temperatures. Furthermore, InSe is a unique crystal of group III-YI in which sufficient electron and hole conductance is possible. Herein current carriers with an average Hall concentration in the crystal of about ~ $10^{12} – 10^{13}$ $см^{-3}$ [13] are concentrated in 2D regions of ion-covalent layers near Van-der-Waals gaps, creating a surface electron concentration of about 10^{11} cm^{-2}, which provides a possibility to observe the 2D Shubnikov-de-Haas effect [13]. Control over the transition from 2D to 3D conductance is possible via varying temperature, doping, intercalation, pressure, and illumination of crystal [12,14,15], which provides electronic devices with new potentials. Hence, investigation of conditions for formation of magnetic states in such a specific object as InSe crystals are of particular interest. The existance of strong anisotropy of chemical bonds and 2D conductance cannot help affecting the magnetic interaction of introduced magnetic ions both directly between themselves and via the other crystal ions. The indirect exchange interaction between magnetic ions through two-dimension electronic gas is also of interest. In addition, the occurrence of magnetic interactions will be also influenced by the capability of layered crystals to space rearrangement of impurities when the latter are concentrated in the Van-der-Waals gaps and form there 2D clusters or grids [16].

The investigation of AIIIBVI (A is In or Ga and B is S or Se) doped with d-transition metals (Mn, Fe), has found the presence of nontrivial magnetic properties in these semiconductors. Thus, in non-annealed InSe samples containing 1.25 at.% Mn, two magnetic d-subsystems were revealed, which is connected with the position of Mn ions inside the ion-covalent layers and interlayer Van-der-Waals space [17,18]. This is confirmed by the presence of two lines (broad and narrow) of manganese, in the EPR spectrum. In the opinion

of the authors [18], 2D magnetism in non-annealed samples relates to the formation of ferromagnetic clusters in the interlayer space.

Temperature hysteresis of magnetic susceptibility (MS) in the diluted magnetic semiconductor $In_{1-x}Mn_XSe$ (x=0.01; 0.10) was observed in the temperature range 90-290 K [19]. The magnetic states are considered to appear thanks to the exchange interaction in the chain Mn-Se-Mn and/or Mn-Se-In-Mn. Beyond the limits of this range MS is paramagnetic.

InSe with 2 at.% Mn [20] demonstrates a Curie-Weiss type behavior. The authors suggest that below 20 K the system should be in the state of spin glass. InS with 2 at.% Mn does not show up a ferromagnetic behavior either and below 10 K has features of spin glass [21]. In GaSe with 5 at.% Mn [22], a broad peak of magnetization is observed in the interval 100-200 K, which amplitude decreases with increasing the magnetic field. The authors associate this bihavior with the presence of short-range antiferromagnetic ordering. In [23], the ferromagnetic behavior of GaSe with 5 at.% Fe was revealed at 300 and 400 K. Below 5 K magnetization became constant and was prescribed to the Van-Fleck paramagnetism. GaS with 6.6 at.% Mn obeys the Curie-Weiss law in the range 77-325 K, but at 10.9 K it is characterized by sharp maximum of magnetization, which the authors attribute to the transition into a spin glass state.

All the mentioned facts are a clench to the importance of investigation of layered crystals doped with manganese. However, the available experimental data demonstrate significant discrepancy in the behavior of magnetically dissoluble layered crystals and difficulties in finding their common regularities. In order to relate DMS from hypothetic materials to really used in spintronics, it is necessary to solve the problem of relation between ferromagnetism with high T_C and characteristics of the basic semiconductor as well as the nature and concentration of magnetic impurity.

It is of interest the investigation of layered InSe semiconductor doped with the magnetic component Mn(InSe<Mn>). In the case of displacement of impurities into the interlayer space, a model of a layered structure of a diamagnetic/magnetic component type is obtained. When indium is replaced with a magnetic component, a solid solution with properties of DMS is obtained. The task is to study a complex of magnetic properties and neutron diffraction spectra of InSe<Mn> in a wide temperature range with the aim to establish the peculiarities of magnetic interaction in layered systems. The chosen model of a layered system assumes sufficient reduction in scattering of current carriers on the layer boundaries at exchange interaction between magnetic layers opposite to the layered system formed by series of depositions of atomic layers by any available method. However, the small solubility (about 7 %) of magnetic components, in indium chalchogenides results in creating their inclusions and also in forming other magnetic phases. That is why investigation of such systems requires a phase analysis of samples.

Investigation of neutron diffraction was carried out in two directions: the one was neutron diffraction for phase analysis of InSe doped by Mn; the other was temperature studies aimed at revealing effects that can indicate the presence of magnetic structure.

2. NEUTRON DIFFRACTION INVESTIGATION

InSe single crystals grown by the Bridgeman method were doped with 1 at.% Mn in a charge. The ingot was cut into wafers perpendicular to the axis of growth. The samples for further studies were cut from the wafers.

The content of Mn in the samples was determined using an X-ray fluorescent analysis, the results of which indicate a nonuniform distribution of impurities along the ingot with

increasing them at the ingot end. Investigation of neutron diffraction was made on samples cut from ingot with a Mn content of about 0.4 at. % and then annealed at 320^{O}C during 140h.

The X-ray analysis of the sample phase composition showed that the main phase (about 90 vol. %) was the hexagonal phase InSe ($P6_3/mmc$) with lattice parameters of a=4.0026 Å and c=16.634 Å. Additionally, inclusions of another phase (about 10 vol.%), namely the cubic phase MnSe (Fm3m, a=5.456 Å), were detected. Neutron diffraction analysis was performed on a nuclear impulse reactor operating on fast neutrons IBR-2 at the Joint Institute for Nuclear Research (Dubna, Russia) using a DN-2 diffractometer by a method of time-of-flight neutron diffractometry [25,26]. A single crystal was mounted on a moving remotely-operating goniometer. Neutron diffraction spectra were obtained with using a positionally-sensitive detector. Scanning was performed in various geometries in order to show up the peculiarities of reflexes at various angles. The sample orientation related to the neutron flow was changed with help of the goniometer. The measurements were carried out at different fixed angles θ of detector. It was experimentally established that an optimal position for investigation of sample is the orientation at which the neutron flow reflects from the layer plane *C*. Herein two systems of mutual orientation of the sample and the detector were used. At the first geometry peaks from small interplane distances are clearly seen, at the second one from big distances (with no reflections from small ones). This is connected with the peculiarities of the apparatus used and the distribution of neutron flow energies.

We have studied neutron diffraction spectra of the undoped InSe single crystal in the temperature range 10-290 K with a step of 10 K under cooling and heating of the sample. The analysis of the temperature dependence of the peak corresponding to the crystallographic direction *c* makes it possible to observe the temperature changes in the interplane distance, which is proportional to the period *c* of the crystal lattice. No critical changes in the crystal lattice symmetry were revealed. The lattice linearly reduces with decreasing temperature in the <001> direction. The crystal structure was identified as a hexagonal lattice (S.G. $P6_3/mmc$, №194); a=4.005 Å; c= 16.640 Å, $\gamma = 120^{O}$. Fig. 1 presents the neutron diffraction spectrum of the InSe sample with 0.4 at.% Mn. As it is seen, not all peaks belong to the hexagonal lattice of InSe. The additional peaks were established to belong to In_4Se_3 (Table 1) and to α -MnSe (Table 2).

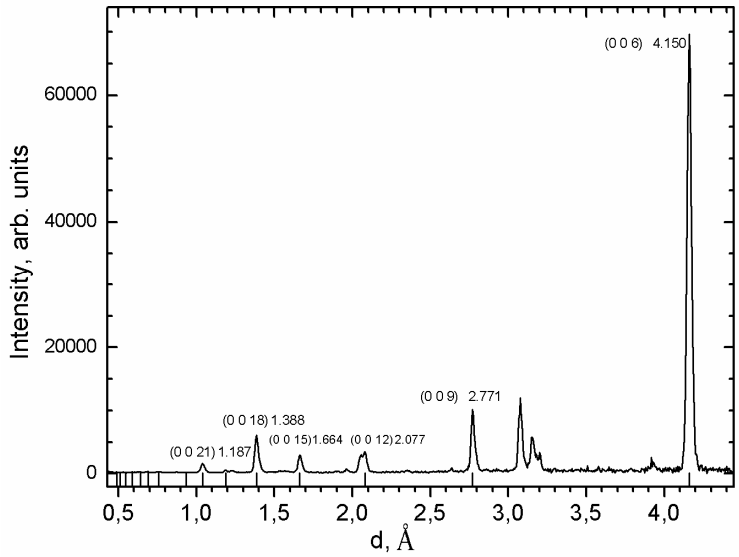

Figure 1. The neutron diffracton spectrum from the InSe<Mn> crystal. The marks below denote the position of peaks that belong to the InSe structure (S.G. $P6_3/mmc$, №194); a=4.005 Å; c= 16.640 Å, $\gamma = 120^{O}$. Designations above peaks: (Muller indexes of nuclear planes) distance between nuclear planes.

To study the effects of magnetic ordering, experiments at various temperatures were conducted in the geometry of reflection from the *C* plane. The peak 6.275 Å was observed, which does not belong to the InSe structure. Its intensity nearly doubles with decreasing temperature from 50 down to 10 K, while the intensity of the neighboring peaks with Miller indexes (006) and d=4.1567 Å as well as (003) and d=8.3133 Å increases by less than 10%. The temperature dependence of the peak 6.275 Å intensity is clearly seen in Fig. 2.

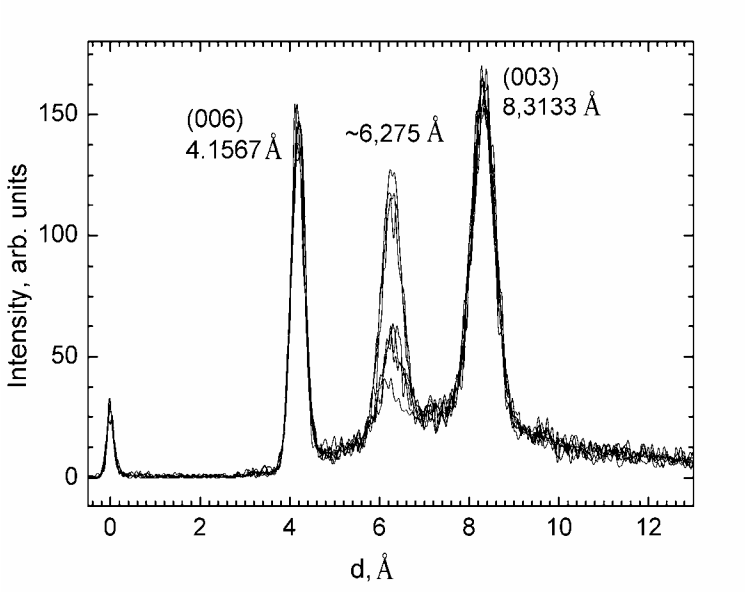

Figure 2. The intensity of neutron diffraction reflexes of the InSe<Mn> crystal for various temperatures. 2θ= 161,03^0

The peak 6.275 Å is a superposition of the following peaks: weakly temperature-dependent peak 6.161 Å (In_4Se_3) (see Table 1) and temperature-sensitive peak 6.303 Å (MnSe) (see Table 2). From the data of Fig. 2, the temperature dependence of the peak 6.303 Å (MnSe) intensity was obtained (Fig. 3a, 3b), using the method of mathematic modeling. By extrapolating the low-temperature region of the 6.303 Å peak intensity up to the intersect with the temperature axis (see the straight line), the temperature of this peak appearance can be determined, T_o=(70 ± 7)K.

The change in the experiment geometry (transition from 2θ = 161,03^O to 90,00^O) made it possible to reveal a temperature-dependent peak near d=6.303 Å not mixed with the other peaks.

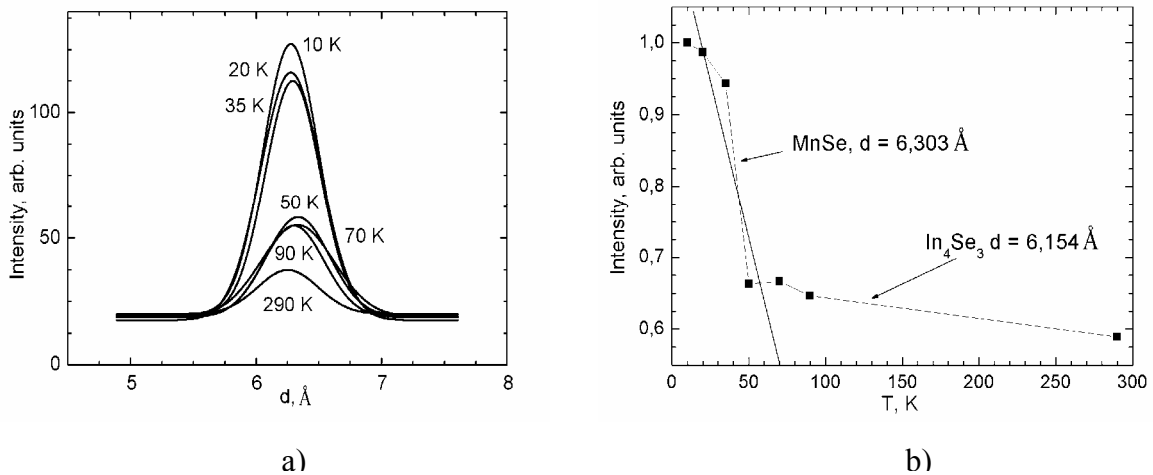

Figure 3. The temperature dependence of the magnetic peak intensity; . 2θ= 161,03^0.

Table 1. Comparison of the found reflexes in the neutron diffraction spectra with reference data for $In_4Se_3.In_4Se_3$ (PDF № 83-0039) Indium Selenide Sys: Orthorhombic S.G. Pnnm (58) a: 15.296(1) b:12.308(1) c: 4.0806(5)

h k l	d, A (PDF)	d, A (EXP)
0 2 0	6.154	6.161
0 4 0	3.077	3.078
0 6 0	2.0513	2.352
0 8 0	1.5385	1.531

Table 2. Comparison of the found reflexes in the neutron diffraction spectra with reference data for α- MnSe Alpha MnSe (PDF № 11-0683) Manganese Selenide Sys: Cubic S.G. Fm3m (225) a: 5.462

h k l	d, A (PDF)	d, A (EXP)
1 1 1	3.152	3.152
2 2 2	1.577	1.568
4 4 4	0.788	0.880
1 1 1 *2	3.152*2 =6.304	6.275 the magnetic peak

3. DISCUSSION ON THE EXPERIMENTAL RESULTS

The analysis of our neutron diffraction studies and magnetic properties of the InSe<Mn> samples (Mn=0.4 at.%) in wide ranges of temperatures and magnetic fields indicates its multicomponent composition.

Bearing in mind the additivity of MS components one can calculate MS of an imaginary sample composed of a InSe matrix which MS is diamagnetic and weakly depends on temperature [27]. This sample is doped with 0.15 mass % MnSe, which has antiferromagnetic properties [28]. Comparison of MS of this sample (let us name it MnSe (0.15 mass %) with the results of our investigation of MS of the InSe sample with 0.4 at.% Mn [29, 30] is the most visual in as much as the experimental conditions were identical, namely cooling down to low temperatures in magnetic field H = 0.1 T (FC 0.1T). The results of the comparison are shown in Fig. 4. Practically complete not only qualitative, but also quantitative agreement between the temperature dependences of MS for comparative InSe<Mn> samples and MnSe (0.15 mass %) is seen.

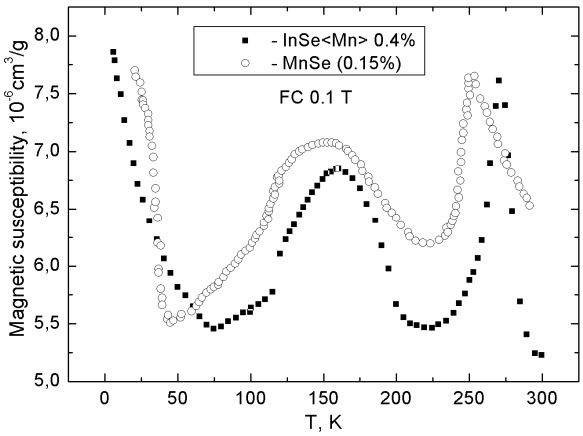

Figure 4. The temperature dependence of the static magnetic susceptibility:
■ - InSe <Mn>; o - model MnSe (0.15 mass %) in the diamagnetic InSe matrix.

Only some details are different. For example, the MS extremuma are shifted toward low temperatures by (15-30) K, and with rising temperature the paramagnetic component of the background part of the MS increases with a rate of $0.39.10^{-8}$ cm^3/(g.deg), with the background component being constant for the InSe<Mn> sample. The reason for these differences is connected with the dependence of phase transformations on Mn content. In the rest, the MS of the InSe<Mn> sample in the temperature range 4.2-300K is completely determined by the MnSe phase, which we identified in the neutron diffraction spectra. The MS maximum at about 260 K is caused by partial structural phase transition from a cubic NaCl-type modification into an antiferromagnetic hexagonal NiAs modification, which is intrinsical for the MnSe phase at 266 K [28]. The sharp rise of MS at about 160 K is associated with the antiferromagnetic ordering of inclusions of the NaCl cubic modification of MnSe.

According to [31], in the case of a sample composed of magnetic and nonmagnetic atoms, there exists a factor of magnetic scattering similar to nuclear scattering. The elemental magnetic cell does not coincide with the crystallographic one being larger than the latter by a factor of 2, 3 or n. Such a sample can be considered as composed of a number of individual crystallographic cells. Under elastic neutron scattering, coherent intensity maxima at angles corresponding to the Bragg reflections from magnetic lattice of the crystal appear. When the elemental magnetic cell is larger than the crystallographic one, pure magnetic maxima of scattering which do not coincide with nuclear ones must appear. At temperatures higher than the Curie or Noel ones they are absent, which means that investigation of temperature dependence of neutron diffraction spectra permits one to observe magnetic ordering in crystals thanks to the appearance of additional peaks and change in their intensity.

The 6.303 Å peak, which showed a strong temperature dependence of intensity in the range 7-70 K, corresponds to the double α-MnSe lattice parameter for the interplane distance (111) 3.152 Å (see Table 2). This permits one to conclude that the observed “magnetic” 6.303 peak is related to the α-MnSe phase.

The sharp increase in MS with decreasing temperature from about 70 K with a rate of $3.5.10^{-8}$ cm3/(g.deg) (Fig. 4), which does not disappear at high magnetic field H=5,0T can be attributed to the new phase transformation with a change in the magnetic cell volume of α-MnSe clusters in contrast to the antiferromagnetic phase transition at T_N=160 K, which disappears at H=5,0 T [29, 30].

This conclusion is confirmed by the temperature dependence of the magnetic cell parameter below 70 K in the neutron diffraction spectra from the InSe<Mn> sample (Fig. 5). Therefore in the region of such low temperatures anomalous reduction of the magnetic lattice parameter takes place, which indicates the fact of magnetic phase transformation. The thermal

expansion coefficient in the temperature range 2.4-70.0 K equals to $1.5.10^{-2}$ deg^{-1}, that is, is four orders of magnitude higher than usual one. This means that with decreasing temperature below 70 K magnetic interaction of ferromagnetic type occurs, which critically decreases the magnetic lattice parameter of MnSe inclusions in the <111> direction (self-consistent problem).

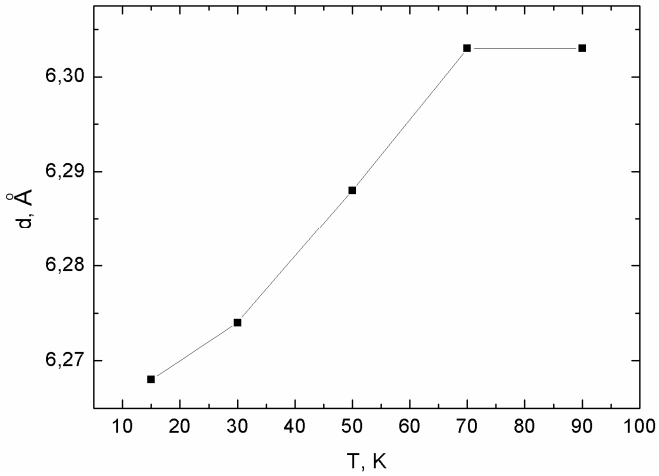

Figure 5. The temperature dependence of the magnetic cell parameter; $2\theta = 90{,}00^0$

Perhaps, herein weak paramagnetism arises as a result of the transformation of the antiferromagnetic MnSe phase into a noncompensated antiferromagnetic state.

Comparison of Fig. 1 and Table 2 leads to the conclusion that inclusion of the cubic MnSe phase in the layered structure of the InSe<Mn> matrix is regular: the <111> direction of the inclusions coincides with the matrix *c* axis (<001> direction).

Hence, the InSe<Mn> single crystal can be considered as a self-arranged superlattice composed of $In_{1-x}Mn_xSe$ and MnSe layers. The thickness of each layer is larger than the size of the coherence region of X-ray radiation, that is equal to hundreds of units of the corresponding lattice parameter. This permits observation of the MnSe phase in X-ray and neutron diffraction spectra. According to the x-ray phase analysis data, the relationship between the volume fractions of these layers is about 9. The alternation of $In_{1-x}Mn_xSe$ and MnSe layers is probably periodic similar to the case of InSe intercalation [32]. Thus, in the temperature range 4-270 K, the described superlattice should be considered to be composed of alternating ferromagnetic (or ferrimagnetic $In_{1-x}Mn_xSe$) and antiferromagnetic (MnSe) layers. This must result in periodically changing in the manganese content in the cross direction and in specifically interacting magnetic layers as well as in high magnetic resistance. The antiferromagnetism of the EuS/PbS superlattice, which magnetic characteristics depend on the non-magnetic PbS layer thickness [33,34], is an example of such interaction.

4. CONCLUSIONS

According to the neutron diffraction analysis, the layered InSe crystal doped with manganese above the level of its dissolution is composed of the phases, $In_{1-x}Mn_xSe$, In_4Se_3, and MnSe.

The nonmonotonous path of the MS temperature dependence of for InSe<Mn> is due to the properties of the α-MnSe phase, whose content reaches 0.15 mass %. The presence of MnSe neutron peaks indicates that the dimensions of these inclusions are equal to hundreds of the lattice parameter units.

For the first time a decrease in the magnetic sublattice parameter with decreasing temperature below 70 K was established. This results in arising weak ferromagnetism, probably, owing to noncompensated antiferromagnetism, which causes a sharp rise in the MS.

The existence of the self-arranged magnetic superlattice "ferromagnetic $In_{1-x}Mn_xSe$/antiferromagnetic MnSe" is suggested.

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