

## NEUTRON DIFFRACTION STUDY OF MAGNETIC TRANSFORMATIONS IN InSe (Mn) LAYERED SEMICONDUCTOR

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The analysis of magnetic properties and neutron diffraction spectra for an InSe layered semiconductor doped with Mn has been carried out. The neutron diffraction structural studies show that the InSe(Mn) semiconductor is a multiphase material consisting of several phases, namely InSe, MnSe, and In<sub>4</sub>Se<sub>3</sub>, which is a result of a poor solubility of manganese in InSe. A nonmonotonic temperature dependence of magnetic susceptibility (MS) for the InSe(Mn) specimen within a temperature range of 2.4–270 K is due to the antiferromagnetic properties of the MnSe cubic phase. A quantitative agreement between the experimental and simulated data is achieved when the MnSe phase content is about 0.15 mass percent in InSe. The observations of the neutron reflections from the MnSe phase indicate that the typical sizes of its inclusions exceed the coherence area size for neutron radiation. It is revealed that the magnetic lattice period for the MnSe cubic phase is doubled in comparison with that for a crystal lattice. For the first time, the decrease in the magnetic lattice parameter was observed with the temperature reduction below 70 K, where MnSe is in the antiferromagnetic state. It is concluded that this effect leads to an enhancement of the interaction between magnetic moments in the magnetic sublattice and probably to the appearance of a new ferromagnetic (FM) state. This is believed to lead to a drastic increase in MS of MnSe with decrease in the temperature from 70 to 2.4 K, whose nature remained unknown till now.

### 1. Introduction

Diluted magnetic semiconductors (DMS) belong to a new kind of magnetic materials which don't contain a magnetic sublattice.

In recent years, DMS have attracted the more and more attention of scientists. These materials have been known for about 40 years. In 1984, a FM ordering with the Curie temperature  $T_C = 10$  K was attained in  $\text{Pb}_{1-x-y}\text{Sn}_x\text{Mn}_y\text{Te}$  solid solutions [1]. It is the RKKY mechanism that provides the exchange

interaction between magnetic manganese ions. Later on, the FM state was revealed in  $\text{Ga}_{1-x}\text{Mn}_x\text{As}$ . The advances in technology made it possible to increase the Curie temperature for this semiconductor compound to 175 K [2], but no further progress has been observed in recent years.

A shift of the FM transition temperature to the values higher than 350 K would allow one to use DMS in spin electronics devices. In this case, the preference should be given to the materials which are non-toxic and can be easily integrated into modern microelectronic technology.

In this regard, layered crystals of groups III–VI have attracted the much attention. They are characterized by a high anisotropy of chemical bonds: a strong ion-covalent bond is characteristic of the planes of atomic layers and a weak one, of the van der Waals kind, is between the layers. InSe is the only crystal of groups III–VI, in which a sufficient level of electron and hole conductances can be created. At the same time, the current carriers characterized by an average Hall concentration of  $10^{12} - 10^{13} \text{ cm}^{-3}$  in a crystal [3] are concentrated within the 2D regions of the ion-covalent layers in the vicinity of van der Waals gaps. This results in a surface concentration of electrons of  $\approx 10^{11} \text{ cm}^{-2}$ , which allows the observation of the Shubnikov–de Haas effect [4]. The magnetic interactions is also influenced by the inclination of the layered crystals to a spatial redistribution of impurities: they are accumulated in the van der Waals gaps by forming two-dimensional aggregates or nets [5].

The studies of the  $\text{A}^{\text{III}}\text{B}^{\text{VI}}$  (A – In, Ga; B – S, Se) crystals doped with *d*-transition metals (Mn, Fe)

have shown that these semiconductors display nontrivial magnetic properties. Two magnetic subsystems were revealed in non-annealed InSe specimens doped with 1.25 at. % of Mn, which were studied in works [6,7]. This phenomenon was concluded to result from a location of Mn ions both within ion-covalent layers and in interlayer van der Waals gaps. Annealed specimens, as the authors of [7] inferred, display the two-dimensional ferromagnetism related to the formation of FM clusters in the interlayer space.

The temperature hysteresis of MS was observed in  $\text{In}_{1-x}\text{Mn}_x\text{Se}$  ( $x = 0.01; 0.10$ ) DMS in the temperature range 90–290 K [8]. The origin of the magnetic ordering is thought to result from the exchange interaction in a chain Mn–Se–Mn and/or Mn–Se–In–Mn. Beyond this temperature range, the MS behavior is characteristic of a paramagnet.

According to the results of work [9], the features characteristic of InS doped with 2 at. % of Mn are the Curie–Weiss behavior of MS at high temperatures and the spin glass state below 20 K. On the other hand, the authors of [10] did not reveal any evidence for the FM state in the material of the same nominal composition, although they found the spin glass features below 10 K. In GaSe doped with 5 at. % of Mn, the authors of [11] observed a wide magnetization peak in the temperature range 100–200 K. As the peak height decreased with increase in the magnetic field, it was concluded that this peak was related to the short-range antiferromagnetic ordering. In GaSe doped with 5 at. % of Fe, the indication of FM ordering was revealed at 300 and 400 K [12]. Below 5 K, the magnetization was constant, which was ascribed to the van Fleck paramagnetism. As was shown in work [13], GaS doped with 6.6 at. % of Mn obeys the Curie–Weiss law in the temperature range 77–325 K. However, it displayed a sharp magnetization maximum at 10.9 K which was explained as that originating from a transition to the spin glass state.

All the above facts provide convincing arguments in favor of the importance of the investigation of layered crystals doped with manganese. At the same time, the aforementioned experimental data also demonstrate a great variety of the behavior features of diluted magnetic layered crystals and the difficulties in specifying their regularities. To turn DMS from hypothetical materials to those which can actually be used in applications, it is necessary to elucidate a question about a relation between the high- $T_C$  ferromagnetism, the characteristics of a parent semiconductor, and the nature and the concentration of a magnetic impurity.

In this respect, the study of an InSe layered semiconductor doped with a magnetic component, namely Mn, is of particular interest. If impurity ions are expelled in an interlayer gap, a model for a layered structure of the kind of diamagnetic matrix–magnetic component is obtained. On the contrary, if the magnetic component substitutes for indium, a solid solution with the DMS properties is obtained. The task is the comprehensive study of the magnetic properties and neutron diffraction spectra of  $\text{InSe}(\text{Mn})$  specimens over a wide temperature range with the aim to investigate the magnetic interactions in layered systems. The chosen type of a layered system assumes a strong reduction of the current carrier scattering at the layer boundaries for the case of the exchange interaction between the layers, in contrast to layered systems of the same kind which would be created by means of a successive deposition of atomic layers with the use of one of the known methods. However, the low level of a dilution limit for the magnetic components in indium chalcogenides leads to a separation of the surpluses of these components, as well as to the formation of other magnetic phases. For this reason, the study of magnetic properties should necessarily be accompanied by the phase analysis of specimens. To date, we have not known the works devoted to the investigation of the neutron diffraction in layered indium chalcogenides doped with magnetic impurities, as well as in solid solutions on their basis, particularly in  $\text{In}_{1-x}\text{M}_x\text{Se}$  compounds, where M is a magnetic impurity.

The studies of the neutron diffraction was carried out in two directions: 1) the spatial scanning of a single crystal with the aim of the specification of a lattice type, structure features, and impurity phases; 2) the study of temperature effects to reveal the presence of a magnetic structure.

## 2. The Neutron Diffraction Studies

The InSe single crystals were grown by the Bridgman technique. The crystal doping was performed by means of the addition of 1 at. % of manganese in a furnace charge. The ingot was cut into slices perpendicularly to the growth axis. The specimens for the investigations were cut out of these slices.

The Mn content in the specimens obtained was determined by the X-ray fluorescence analysis. The results of the analysis testify to an inhomogeneous distribution of the impurity along the ingot length with increase of the Mn concentration towards its end. The neutron diffraction investigations were carried out on the

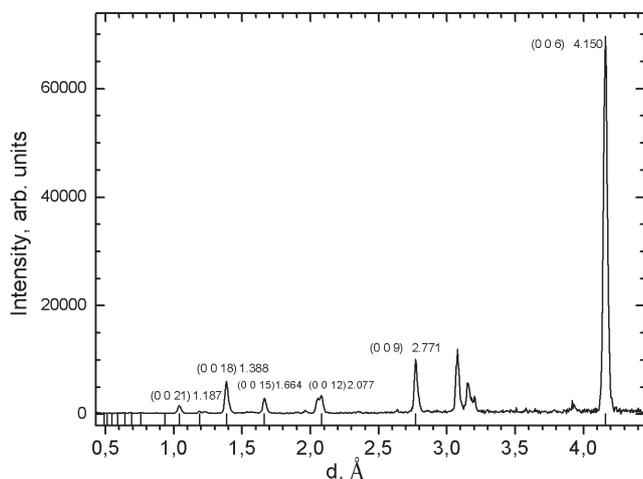


Fig. 1. Neutron diffraction spectrum for the InSe(Mn) crystal. The bars on the abscissa axis show the position of the peaks which belong to the InSe structure (S.G.  $P6_3/mmc$ , No. 194,  $a = 4.005$ ,  $c = 16.640$ , and  $\gamma = 120^\circ$ ). The Miller indices for atomic planes and the interplanar distances are shown above these peaks

specimens which were cut out of the slice with about 0.4 atomic percent of manganese and annealed at  $320^\circ\text{C}$  for 140 h.

The X-ray phase composition analysis for the specimens under study showed that the main phase, which occupied up to 90 % of the crystal volume, was the  $P6_3/mmc$  hexagonal phase with the lattice dimensions  $a = 4.0026$  Å and  $c = 16.634$  Å. In addition, the specimens contained the inclusions ( $\sim 10\%$ ) of a cubic MnSe phase ( $Fm\bar{3}m$ ,  $a = 5.456$  Å).

The neutron diffraction studies were carried out on an IBR-2 fast neutron pulse reactor in the Joint Institute for Nuclear Research in Dubna (Russia) by the method of time-of-flight neutron diffractometry with the use of a DN-2 diffractometer [14,15]. A single crystal was mounted on a remotely controlled movable goniometer. The neutron diffraction spectra were recorded with the use of a position-sensitive detector. The scanning was performed in various geometries, which made it possible to clear up the features of the reflections at different angles. The specimen orientation with respect to a neutron flow was changed with the use of a goniometer. There was a possibility to change the fixation angle  $\theta$  of a detector. It was found experimentally that the optimal orientation for the investigations corresponded to the specimen position, where the neutron flow was reflected from the planes of crystalline layers (reflections from the  $C$  planes). At the same time, two kinds of the relative orientation of a specimen and a detector were used.

The first one was most suitable for the detection of the peaks from the planes with small interplanar distances. The second type was optimized for the detection of the reflections from the planes with greater interplanar distances, although this made it impossible to observe the peaks from the former planes. Such restrictions were caused by the installation features and the peculiarities of the energy distribution within a neutron flow.

At the first stage, the neutron diffraction spectra for the parent (undoped) InSe single crystal were studied in the temperature range 10–290 K with a step of 10 K on the cooling and heating of a specimen. By analyzing the temperature dependence of the intensity for the peak which corresponds to the crystallographic direction  $c$ , it is possible to trace the temperature changes in the interplanar distance proportional to the period  $c$  of the crystalline lattice. The studies carried out revealed no critical changes in the lattice symmetry. The lattice was found to shrink in the  $\langle 001 \rangle$  direction with decrease in the temperature. The crystalline structure was identified as a hexagonal lattice (space group  $P6_3/mmc$  (N 194);  $a = 4.005$  Å,  $c = 16.640$  Å, and  $\gamma = 120^\circ$ ).

Figure 1 shows the neutron diffraction spectrum for an InSe specimen with 0.4 at. % of Mn. It is seen that not all the reflections belong to the InSe-type hexagonal lattice. The additional peaks can be identified as those that belong to  $\text{In}_4\text{Se}_3$  (Table 1) and  $\alpha\text{-MnSe}$  (Table 2).

**Table 1. Comparison of the reflection positions in the neutron diffraction spectra for the specimen under study with the reference data for  $\text{In}_4\text{Se}_3$**

$hkl$	$d$ , Å (PDF)	$d$ , Å (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

Note.  $\text{In}_4\text{Se}_3$  (PDF 83-0039)

Indium Selenide

Sys: Orthorhombic S.G.  $Pnmm$  (58)

a: 15.296(1) b:12.308(1) c: 4.0806(5)

**Table 2. Comparison of the reflection positions in the neutron diffraction spectra for the specimen under study with the reference data for  $\alpha\text{-MnSe}$**

$hkl$	$d$ , Å (PDF)	$d$ , Å (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 $\times$ 2	$3.152 \times 2 = 6.304$	6.275
		“magnetic” reflection

Note. Alpha MnSe (PDF N 11-0683)

Manganese Selenide

Sys: Cubic S.G.  $Fm\bar{3}m$  (225)

a: 5.462

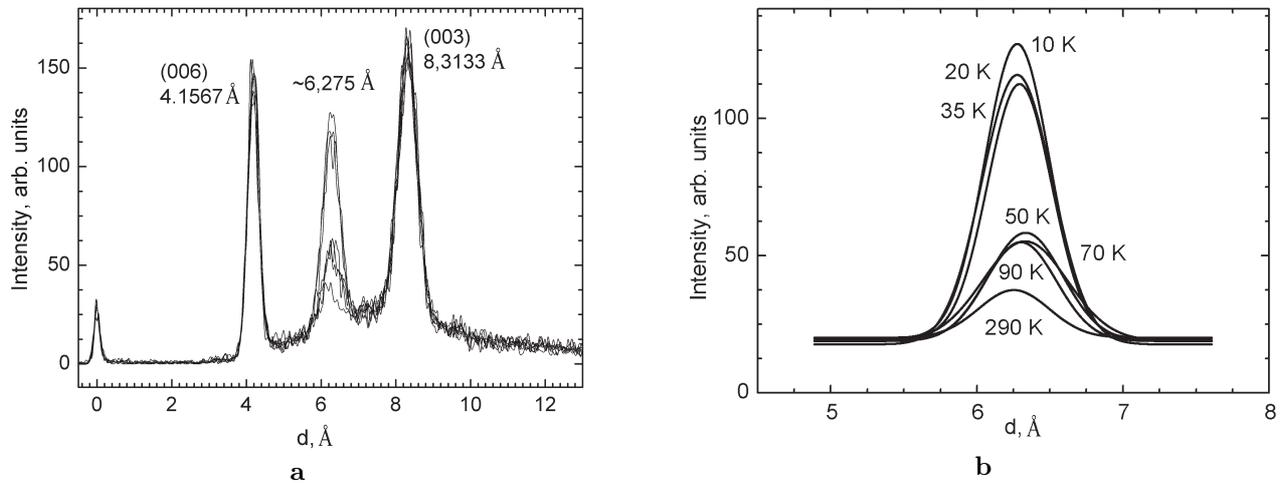


Fig. 2. Intensity of the neutron diffraction reflections for the InSe(Mn) crystal at different temperatures: *a* – experiment; *b* – the results of the mathematic modeling for the peak at 6.303 Å ( $2\theta = 161.03^\circ$ )

To reveal the manifestations of the magnetic ordering, the experiments were performed at different temperatures with focusing on the reflections from the plane *C*.

The spectra obtained contain a peak at 6.275 Å which doesn't belong to the InSe structure. Its intensity increases almost twice as the temperature is lowered from 50 K to 10 K. At the same time, the intensity of the neighboring peaks of the InSe structure with the (006) and (003) Miller indices ( $d = 4.1567$  Å and 8.3133 Å, respectively) rises by less than 10%. The features of the peaks' intensity change with the temperature variation are clearly seen from Fig. 2, *a*.

The peak at 6.275 Å is a superposition of two reflections: one of them is a weakly temperature dependent peak at 6.161 Å ( $\text{In}_4\text{Se}_3$ , see Table 1) and the second one – a temperature sensitive peak at 6.303 Å (MnSe, see Table 2). Basing on the data of Fig. 2, *a*, the temperature dependence of the intensity of the latter peak was separated by means of a mathematical modeling (Fig. 2, *b*). By extrapolating the low-temperature region of the intensity to the intersection with the temperature axis (see the straight line in Fig. 3), we get the temperature of the peak appearance:  $T_0 = (70 \pm 7)$  K.

The change in the experiment geometry (a switch from  $2\theta = 161.03$  to  $90.00^\circ$ ) made it possible to reveal the temperature-dependent peak at  $d = 6.303$  Å well separated from the other peaks.

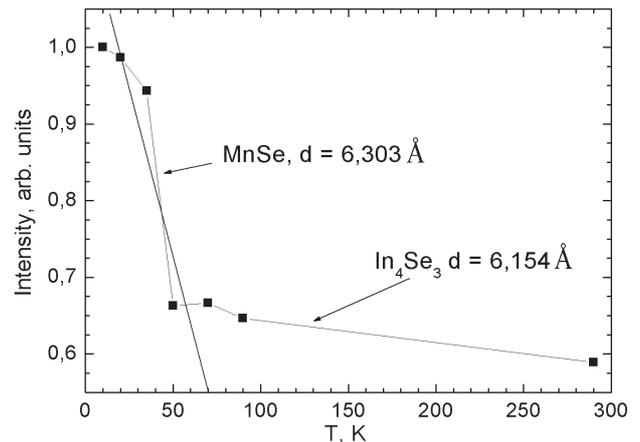


Fig. 3. Temperature dependence of the intensity of the magnetic peak at 6.303 Å ( $2\theta = 161.03^\circ$ )

### 3. Discussion of the Experimental Results

The analysis of the results of neutron diffraction studies and the magnetic properties, carried out on the InSe(Mn) specimen ( $x_{\text{Mn}} = 0.4$  at. %) over wide ranges of temperatures and magnetic fields, testifies to the specimen's multicomponent composition.

Proceeding from the assumption that the MS components are additive, we can calculate the MS for a hypothetical specimen which consists of an InSe matrix, for which the static susceptibility is diamagnetic and weakly dependent on temperature [16], and a MnSe impurity (up to 0.15 mass %) which is antiferromagnetic [17]. The comparison of the MS of such a specimen (let

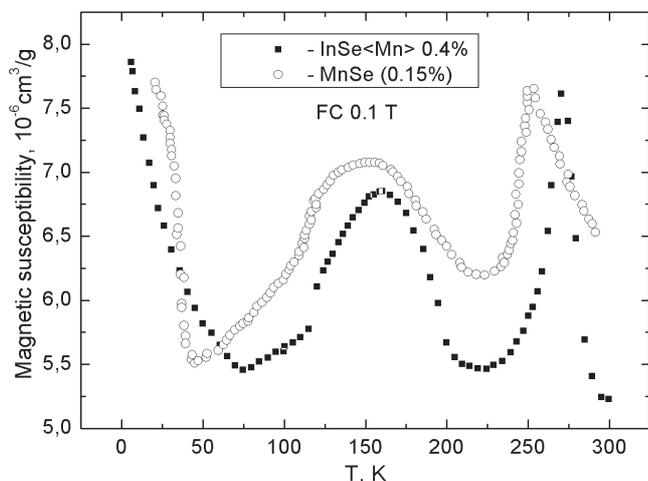


Fig. 4. Temperature dependence of the static MS: solid squares – InSe(Mn); open circles – the model specimen containing 0.15 mass percent of MnSe in the InSe diamagnetic matrix

us denote it as MnSe (0.15 mass %) with the results of our investigation of the InSe specimen doped with 0.4 at. % of Mn [18,19] is most pertinent, since the experiment conditions were identical: in both cases, the specimens were cooled down to low temperatures in a zero magnetic field. The results of such a comparison are shown in Fig. 4. As is seen from the figure, there is not only the qualitative, but also quantitative correlation between the temperature dependences of MS for both the specimens.

The differences between these dependences can be noticed only in details. Namely, for the case of the MnSe (0.15 mass %) specimen, the extrema on the MS vs  $T$  dependence are shifted by 15–30 K towards the lower temperatures. What is more, with increase in the temperature, the paramagnetic component of a background part of MS grows with a rate of  $0.39 \times 10^{-8} \text{ cm}^3/(\text{g}\cdot\text{K})$ , whereas it remains almost constant for the InSe(Mn) specimen. The origin of these differences lies likely in the dependence of the phase transformation temperatures on the manganese concentration. The other features of the MS in the range 4.2–300 K are completely determined by the MnSe fraction, which was identified in the neutron diffraction spectra for the InSe(Mn) specimen under study. The MS maximum at 260 K reflects a partial structural phase transition from a paramagnetic cubic (NaCl-type) modification to the antiferromagnetic hexagonal (NiAs-type) one which is observed in pure MnSe at 266 K. In our case, only  $\sim 30$  mass % of the MnSe cubic phase undergoes such a transition [17]. The MS peak at 160 K is caused by the antiferromagnetic ordering of the remainder of the MnSe

cubic modification ( $\sim 70$  mass %) which eventually forms a magnetic unit cell of the MnO type. What further distinguishes the behavior of our specimen from that of pure MnSe is a shift by 20 K of the sharp MS drop in the vicinity of 50 K. This feature was not discussed in [17].

According to [20], a magnetic material, which consists of magnetic and nonmagnetic atoms, is characterized by the magnetic scattering factor by analogy with the nuclear one. A magnetic unit cell does not coincide with a crystallographic one: it is 2, 3 or more times greater than the latter. Such a material can be regarded as one consisting of a series of separate crystallographic cells. If the neutron scattering in this material is elastic, the coherent intensity maxima are observed at the angles which correspond to the Bragg reflections from the magnetic lattice. If the magnetic unit cell is greater than the crystallographic one, there should appear the purely magnetic maxima which do not coincide with the nuclear ones. They do not exist above the temperature of magnetic ordering, i.e. above the Curie or Neel point. This means that the investigation of the temperature evolution of the neutron diffraction spectra makes it possible to characterize the magnetic ordering in a crystal which manifests itself by the appearance of additional peaks and the change of their intensity.

The peak at  $6.303 \text{ \AA}$ , which shows the strong temperature dependence of the intensity in the region of 7–70 K, corresponds to a doubled period of the  $\alpha$ -MnSe lattice, as the interplanar distance for (111) planes is  $3.152 \text{ \AA}$  (see Table 2). This gives grounds to state that the “magnetic” peak observed at  $6.303 \text{ \AA}$  belongs to the  $\alpha$ -MnSe phase.

The sharp MS growth at  $T \approx 70 \text{ K}$ , which is observed as the temperature is lowered and characterized by a rate of  $3.5 \times 10^{-8} \text{ cm}^3/(\text{g}\cdot\text{K})$  (see Fig. 4), can be regarded as a manifestation of the magnetic phase transformation which occurs in  $\alpha$ -MnSe clusters and is accompanied by a change in the volume of a magnetic unit cell. This transition survives in a field of  $H = 50 \text{ kOe}$ , contrary to the case of the known antiferromagnetic phase transition at  $T_N = 160 \text{ K}$  which disappears in such a field [18,19].

An additional confirmation of this conclusion follows from the temperature dependence of the magnetic lattice period observed by us in the neutron diffraction spectra for the InSe(Mn) specimen below 70 K (Fig. 5). The fact that the anomalous decrease in the magnetic cell period occurs in this range of temperatures testifies to the existence of the aforementioned magnetic phase transformation. In the temperature range 2.4–70 K, the

thermal expansion coefficient is  $1.5 \times 10^{-2} \text{ K}^{-1}$ , which is four orders higher than the usually observed value. It is likely that, as the magnetic interaction between the magnetic moments is enhanced with decrease in the temperature, the antiferromagnetic cubic phase of MnSe with a magnetic unit cell of the MnO type undergoes changes. This could lead to the formation of a new FM state and eventually results in a sharp MS rise below 70 K. It should be noted that the studies on pure MnSe [17] did not reveal any peculiarities in the neutron diffraction spectra over the temperature range 10–130 K. This fact can result from the insufficient technical capabilities of the equipment used in work [17]. It should also be stressed that the present work is aimed at a purposeful search for “magnetic” reflections by means of the use of different experiment geometries for the specimen exposure. Thus, the facts mentioned above allows us to state that, for  $T < 70 \text{ K}$ , the enhancement of the magnetic interaction between the magnetic moments of a magnetic cell is characteristic of both the pure MnSe and its inclusions into the InSe matrix.

The comparison of the data of Fig. 1 and Table 2 leads to the conclusion that the inclusions of the MnSe cubic phase in the layered structure of the InSe(Mn) matrix are correlated by their axes with the matrix: the  $\langle 111 \rangle$  direction of the inclusions coincides with the  $c$  axis of the matrix (the  $\langle 001 \rangle$  direction).

Each of the layers has a thickness greater than the size of the coherence area for both X-ray and neutron radiations. This allows the observation of the reflections from the MnSe phase in the X-ray and neutron diffraction spectra. As follows from the data of the X-ray phase analysis, the ratio of the volume fractions of these layers in the crystal approximately equals 9.

#### 4. Conclusions

According to the data of neutron diffraction studies, the InSe(Mn) layered crystal with the Mn doping level higher than the solubility limit consists of InSe, MnSe, and  $\text{In}_4\text{Se}_3$  phases.

The nonmonotonous character of the temperature dependence of MS for the InSe(Mn) specimens is caused by the properties of the  $\alpha$ -MnSe phase, whose fraction reaches 0.15 mass percent. The presence of the neutron diffraction peaks from the MnSe phase indicates that the size of its inclusions exceeds that of the coherence area for the neutron radiation.

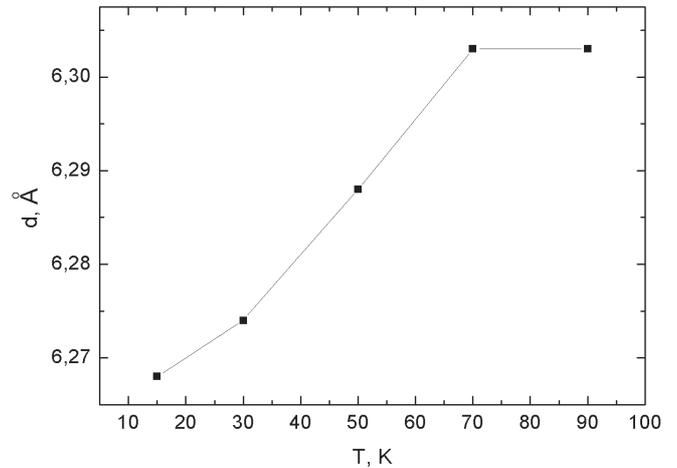


Fig. 5. Temperature dependence of the magnetic lattice period,  $2\theta = 90.00^\circ$

It is found that the magnetic lattice period of the MnSe cubic phase is doubled in comparison with the period of its crystallographic lattice. For the first time, the decrease of the magnetic lattice parameter was observed with the temperature reduction below 70 K, where MnSe is in the antiferromagnetic state. It is concluded that this effect leads to an enhancement of the interaction between magnetic moments in the magnetic sublattice and, probably, to the appearance of a new FM state. This is believed to lead to a drastic increase in MS with decrease in the temperature from 70 to 2.4 K, whose nature has remained unknown to date.

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#### НЕЙТРОНОГРАФІЧНІ ДОСЛІДЖЕННЯ МАГНІТНИХ ПЕРЕТВОРЕНЬ ШАРУВАТОГО НАПІВПРОВІДНИКА InSe(Mn)

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#### Резюме

Проаналізовано результат досліджень магнітних властивостей та нейтронографічних спектрів шаруватого напівпровідника InSe, легованого марганцем (InSe(Mn)). Нейтронографічні структурні дослідження показали, що зразки складаються з декількох фракцій, а саме InSe, MnSe, In<sub>4</sub>Se<sub>3</sub>. Це є наслідком низького рівня розчинності Mn. Немонотонний хід температурної залежності магнітної сприйнятливості (МС) InSe(Mn) в інтервалі температур 2,4 – 270 К визначається антиферомагнітними властивостями кубічної фази MnSe. Кількісний збіг досягається при її вмісті 0,15 ваг. % в InSe. Наявність нейтронних піків MnSe вказує на те, що характерні розміри його включень перевищують довжину когерентності нейтронного випромінювання. Виявлено подвоєння періоду магнітної комірки кубічної фази MnSe в порівнянні з періодом її кристалохімічної ґратки. Вперше для антиферомагнітного стану MnSe виявлено зменшення періоду магнітної комірки при  $T < 70$  К, що приводить до посилення взаємодії між магнітними моментами в магнітній комірці і, можливо, до виникнення нового феромагнітного стану. Це зумовлює різке зростання МС MnSe при зниженні температури від 70 до 2,4 К, природа якого до теперішнього часу була невідома.