# MAGNETIC PROPERTIES OF Mn-DOPED InSe

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The static magnetic susceptibility (MS) and the electron paramagnetic resonance (EPR) spectra of a layered semiconductor InSe doped with manganese (about 5 at.%) have been studied in the temperature range 4.2—300 K, as well as the crystal structure of this compound (at $T = 300$ K). The formation of a solid solution $In_{1-x}Mn_xSe$ (about 90 vol.%), ferromagnetic clusters with the Curie temperatures higher than 350 K, and inclusions of the antiferromagnetic phase MnSe (about 10 vol.%) has been established. The positive background MS practically does not depend on the temperature and partially originates from those ferromagnetic states and atypical Van-Vleck ions $Mn^{3+}(d^4)$ and $Mn^{+}(d^6)$ . The maxima of the MS at about 160 and 270 K are associated with antiferromagnetic transitions in the MnSe inclusions. A wide EPR line is caused by weakly interacting $Mn^{2+}$ ions which are not included into ferromagnetic and antiferromagnetic clusters.

## 1. Introduction

At present, diluted ferromagnetic (FM) semiconductors (DFMSs) attract a more and more attention of scientists. As DFMSs, we mean semiconductors, whose ferromagnetism is due to magnetic ions which belong to $d$-transition metals and are coupled by indirect exchange interaction. After work [1], where the Curie temperature higher than the room one was predicted for ZnO and GaN doped with manganese (provided that the hole concentration is high), has been published, the interest to the problem of the FM state formation in semiconductors at $T > 300$ K and to the implication of the latter in spin electronic (spintronic) devices has grown. The application of DFMSs in spintronic devices would enable one to take advantage of two degrees of freedom — charge and spin — of an electron, thus providing the spintronic devices with new functionalities in comparison with traditional microelectronics.

In this connection, the layered crystals, which belong to group III—VI and are characterized by a high anisotropy of the chemical bond — strong ionic-covalent coupling in the plane of atomic layers and weak van der Waals one between the layers, — draw a particular attention. InSe is the only crystal in this group, where the sufficiently high electron and hole conductivities can be induced. Bond anisotropy leads to two-dimensional (2D) electron conductivity at temperatures below the critical one. In this case, current carriers with the average Hall concentration of about $10^{12} - 10^{14}$ cm$^{-3}$ in the crystal [2] become accumulated in 2D regions of ionic-covalent layers near the van der Waals gaps, thus inducing the surface concentration of electrons of about $10^{11}$ cm$^{-2}$, which enables an opportunity for the Shubnikov—de Haas effect to be observed [3]. Therefore, the researches of conditions, under which the magnetic states are formed in such specific objects as InSe crystals, are of special interest. In addition, the realization of magnetic interactions depends on the ability of layered crystals to redistribute impurities in the bulk, when the latter can be accumulated in the van der Waals gap, forming 2D aggregates or grids [4].

Studies of $A^{III}B^{VI}$ crystals (A = In or Ga; B = S or Se) doped with $d$-transition metals (Mn, Fe) revealed some nonconventional magnetic properties of these semiconductors. In layered InSe:Fe crystals, magnetically ordered clusters of iron emerge even at room temperature [5, 6]. The behavior of the measured

angular, field, and temperature dependences of the rotational momenta of specimens embedded into a homogeneous magnetic field turned out to be the features typical of ferromagnets. In works [7, 8], two magnetic subsystems were detected in unannealed InSe specimens that contained 1.25 at.% of Mn. Mn ions are located in both the ionic-covalent layers ($L$) and the interlayer van der Waals space ($I$). This statement is confirmed by the presence of two Mn lines – a narrow, $L$, and a wide one, $I$ — in the EPR spectrum. If one anneals the specimen, Mn ions diffuse into the interlayer space. The EPR spectrum transforms into a single broadened line $I$. The authors of works [7, 8] supposed that Mn ions in unannealed specimens interact ferromagnetically in the temperature range 140–300 K and antiferromagnetically at 77–140 K. At $T < 77$ K, a three-dimensional FM order arises. The results of experiments with annealed specimens [8] were explained by the authors as a manifestation of 2D ferromagnetism which is connected with the formation of ferromagnetic clusters in the interlayer space.

In work [9], a temperature hysteresis of magnetization in the interval $T = 90 \div 290$ K was observed for InSe crystals with Mn contents of 1 and 10 at.%. Below and above the hysteresis region, the paramagnetic signal obeyed the Curie–Weiss law. According to the results of work [10], InS with Mn content of about 2 at.% shows the behavior of the Curie–Weiss type; the authors consider that the system is in the spin glass state below 20 K. At the same time, according to the data of work [11], this substance does not reveal a ferromagnetic state and, below 10 K, has also the spin glass features. In GaSe with 5 at.% of Mn [12], a broad magnetization peak was observed in the interval 100–200 K; the amplitude of this peak decreased as the magnetic field grew, and the authors associated this phenomenon with a short-range antiferromagnetic ordering. GaS with Mn content of 6.6 at.% [13] obeys the Curie–Weiss law in the interval 77–325 K, but, at $T = 10.9$ K, it is characterized by a sharp maximum of magnetization, which is interpreted by the authors as a spin glass state. The authors of work [14] found the FM-like behavior of GaSe with 5 at.% of Fe at $T = 300$ and 400 K. Below 5 K, magnetization becomes constant and evidences for Van Vleck paramagnetism.

Therefore, Mn-doped InSe is an interesting object for studying. However, the experimental data discussed above demonstrate both a large divergence between the behaviors of diluted magnetic layered crystals and some difficulties in the revealing of their general regularities. In addition, there is no information concerning the influence of the doping with transition metals on the crystal structure and the phase composition of III-VI semiconductors, as well as except works [7, 8] about the thermal treatment of specimens; the latter issue, in our opinion, is extremely important for the objects under consideration.

In this work, we studied the magnetization and EPR spectra of InSe⟨Mn⟩ single crystals in wide ranges of temperature and magnetic field. We also studied the phase and element composition of those objects.

## 2. Specimens and Experimental Technique

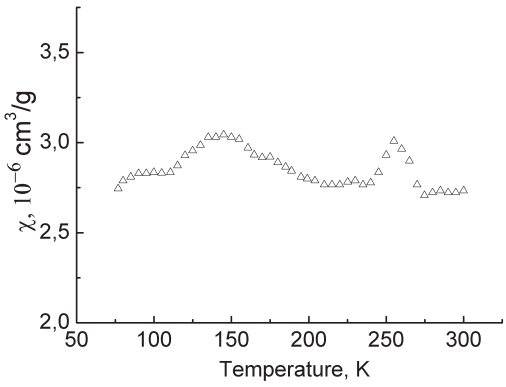
InSe single crystals grown up by the Bridgman method were doped by adding 1 at.% of manganese into the blend. An ingot was cut into disks normally to its growth axis. Specimens for further researches were cut out from the disks. The solubility threshold of Mn in $A^{III}B^{IV}$ crystals has not been determined till now. Therefore, we do not designate a doped crystal by the formula of a solid solution $In_{1-x}Mn_xSe$, as it occurs in the literature, but only specify that a certain amount of Mn is contained in the InSe crystal.

The content of Mn in the specimens obtained was determined by the x-ray fluorescence method. Our results testify that the impurity was distributed nonuniformly along the ingot, with the Mn concentration being increased toward its end. The experiments were carried out using the specimens cut out from a disk with the manganese content of about 5 at.% and annealed at a temperature of 320 °C for 70 h.

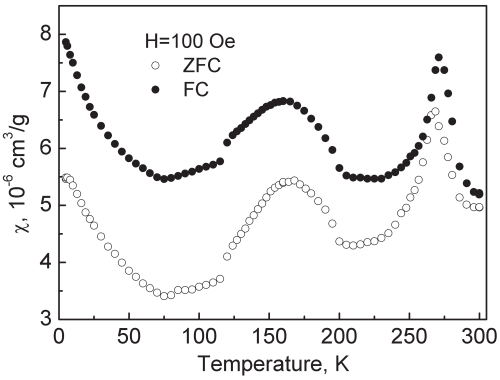
The analysis of the results obtained in the course of the x-ray phase studies of the specimen content showed that the hexagonal phase ($P63/mmc$) with the lattice constants $a = 4.0026$ Å and $c = 16.634$ Å is the main phase (about 90 vol.% of the crystal). In addition, the specimens also contained inclusions (about 10 %) of a second (cubic) phase MnSe ($Fm3m$, $a = 5.456$ Å).

The magnetic researches were carried out using a SQUID magnetometer (in the temperature range 2.4—350 K) and by the relative method of Faraday with the help of an electronic microbalance with automatic compensation (in the range 77—300 K) [15].

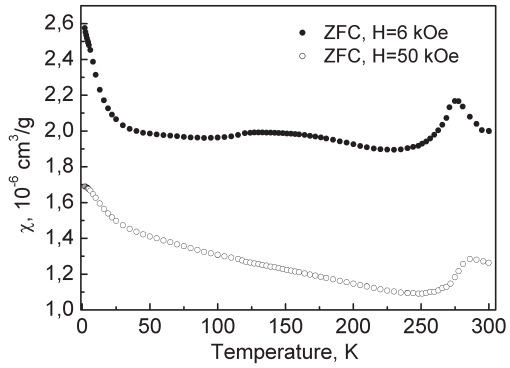
The EPR spectra were recorded in the interval 4.4–300 K using an EMX Bruker ER083CS spectrometer operating at a fixed frequency of 9.38 GHz in the X-band.




**a**




**b**




**c**

Fig. 1. Temperature dependences of the static magnetic susceptibility of an InSe specimen doped with 5 at.% of Mn. The dependences were measured ($a$) by means of an electronic microbalance following the Faraday method; ($b$) with the help of a SQUID magnetometer: *ZFC* indicates the zero-field cooling to the minimal temperature, *FC* cooling in the field of 100 Oe; ($c$) with the help of a SQUID magnetometer in the fields $H = 6$ and 50 kOe

## 3. Experimental Results

### *3.1. Magnetic properties*

The measurements of the static MS of the specimens in a magnetic field $H \approx 2.5$ kOe showed that it is paramagnetic in the temperature interval 77–300 K and does not reveal any dependence on the temperature of the Curie—Weiss type (Fig. 1,$a$). Its practically constant, independent of the temperature value serves as a background, against which two spikes with the amplitudes of about 8 and 15% are observed at 159 and 256 K, respectively.

The measurement of the MS $\chi = M/H$ making use of the SQUID magnetometer in the temperature interval 2.4—300 K at the zero-field cooling (ZFC) showed that its behavior is similar to that observed in the range 77—300 K and described above (Fig. 1,$b$). Namely, two well-pronounced peaks of MS with the amplitudes of about 40% were registered at approximately the same temperatures (163 and 267 K). At the same time, the MS grows in the interval from 77 to 2.4 K, and this growth (of about 62%) considerably exceeds that of the amplitudes of the above-described spikes.

Cooling the specimen down to 2.4 K in a field of 100 Oe (FC) gives rise to the MS growth in the whole temperature interval (Fig. 1,$b$). In this case, the background MS ceases to depend on the temperature.

After the ZFC, the measurements of the MS in strong fields $H = 6$ and 50 kOe (Fig. 1,$c$) revealed a substantial variation in the character of its temperature dependence, as compared with the MS dependence in the 100-kOe field at the ZFC. The peak at 163 K became strongly broadened in the field of 6 kOe and disappeared at $H = 50$ kOe, while the peak at 267 K became shifted to 276 and 286 K, respectively, in those fields.

The dependence of the magnetization on the magnetic field $M(H)$ was studied at a temperature of 2.4 K from the interval, where the temperature reduction is accompanied by the MS growth (see Fig. 1,$b$); at a temperature of 75 K, at which the MS has a minimum; at temperatures of 160 and 271 K, where it was maximal; as well as at 300 and 350 K. The results are exhibited in Fig. 2. Taking into account that both the magnetically ordered fraction responsible for the availability of the hysteresis loop and the paramagnetic one responsible for the linear magnetic dependence $M(H)$ at high fields contribute to the magnetic moment of the specimen, we subtracted the paramagnetic contribution from the total magnetic moment. As a result, we obtained a family of

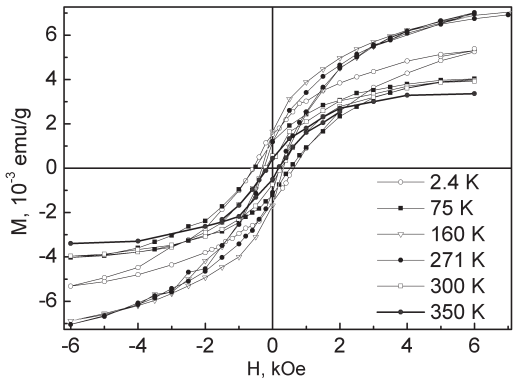



Fig. 2. Dependences of the magnetic moment of an InSe specimen with 5 at.% of Mn on the magnetic field at various temperatures

magnetic hysteresis loops which are characterized by the following parameters: the residual magnetization at various temperatures varies from $0.45 \times 10^{-3}$ to $1.65 \times 10^{-3}$ emu/g, the coercive force amounts to 0.21—0.62 kOe, and the saturation magnetization to $(3.3 \div 7) \times 10^{-3}$ emu/g, the latter being maximal at the MS maxima and minimal at the MS minima.

Summarizing the results of experiments, it should be noted that our study of the MS has discovered the following anomalous effects:

1) the existence of a background paramagnetic component in the MS, which is practically independent of the temperature;

2) the paramagnetic spikes appear at temperatures of about 160 and 260 K;

3) the magnetic prehistory (ZFC or FC) of the specimen affects the MS magnitude.

### *3.2. EPR Spectra*

The EPR spectra in the temperature interval 10—300 K are depicted in Fig. 3. Their decomposition was carried out by means of a special computer program. The spectrum can be presented as a superposition of two resonant lines: the first line ($I$) is observed at a resonant field of about 3.3 kOe, the second line ($L$), which is much broader, at a resonant field of 1 kOe. Provided the temperature is reduced from 300 to 155 K, line $I$ becomes two times and line $L$ four times more intensive (Fig. 4). The width of line $L$ does not change at that, while the width of line $I$ increases from 1 to 2 kOe. As the temperature diminishes from 155 to 75 K, the intensities of both lines decrease and become equal by amplitude. We obtained a satisfactory decomposition of the spectrum into two lines only for $T > 50$ K, because

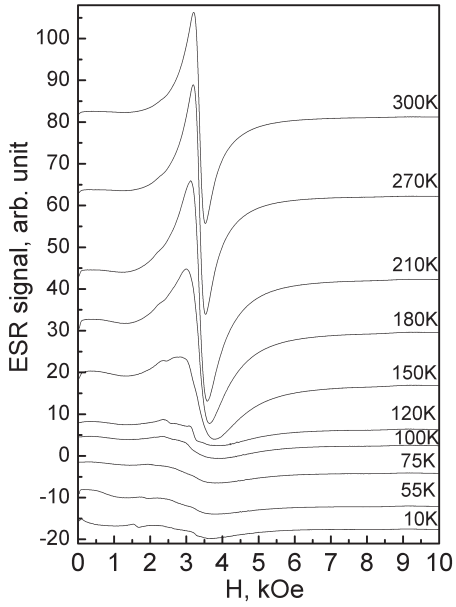



Fig. 3. EPR spectra of an InSe specimen with 5 at.% of Mn at various temperatures

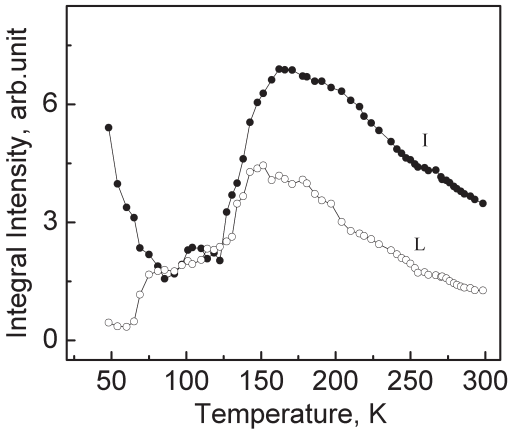



Fig. 4. Integrated intensities of the EPR lines of an InSe specimen with 5 at.% of Mn

of the non-resonance absorption inherent to undoped InSe starts to manifest itself at lower temperatures.

For the sake of comparison, we studied the EPR spectra of Mn-free InSe. In this case, the EPR signal was not observed in the interval $T = 300 \div 100$ K. At lower temperatures, the strong absorption of the unknown nature has been registered.

## 4. Discussion of the Results

Doping a layered InSe crystal with manganese can give rise to the formation of a complicated magnetic

structure. During a specimen growth from the liquid phase, manganese becomes distributed among the layers and the interlayer space. One may expect that a prolonged annealing should result, on the one hand, in a more uniform doping of the ionic-covalent Se-In-In-Se layers owing to the statistical substitution of In ions by Mn ones. On the other hand, Mn becomes displaced from the layers into the interlayer space; it also concentrates near the defects of the crystalline structure.

According to the results of x-ray diffraction (XRD) studies, the crystal lattice constants are smaller in the InSe⟨Mn⟩ crystal ($a$ = 4.0026 Å and $c$ = 16.634 Å [16]) as compared with those in the undoped InSe ($a$ = 4.005 Å and $c$ = 16.64 Å [16]), so that $\Delta a = 0.0024$ Å and $\Delta c = 0.006$ Å. The variation of the lattice constants evidences for the formation of the main phase – a solid solution $In_{1-x}Mn_xSe$, where Mn ions substitute In ones. The reduction of the lattice constants in the main phase, which accompanies such a substitution, is associated with a larger value of the In covalent radius $r = 1.44$ Å [17] in comparison with that of $Mn^{2+}$ ($r$ = 0.92 Å). Therefore, the replacement In → Mn has to reduce the lattice constants of the solid solution, which explains the results of x-ray researches. In this case, according to the data of spin-muon relaxation measurements, the excess In forms aggregates that reveal superconducting properties [18].

Provided that the 5-at.% manganese impurity is distributed statistically uniformly over the crystal, its concentration $N$ amounts to $8.6 \times 10^{20}$ cm$^{-3}$, and the distance $R = N^{-1/3}$ between ions is approximately equal to 23 Å. The activation of the ordinary Ruderman—Kittel—Kasuya—Yosida (RKKY) mechanism of indirect exchange interaction between magnetic ions is impossible, because the average concentration of free charge carriers is low (of about $10^{14}$ cm$^{-3}$). In this case, the hypothesis [7, 8] that the ferromagnetic order is established only in some regions of the crystal — clusters — remains as the most probable one. This occurs owing to the superexchange between Mn ions by means of anions (Se) located in the neighbor sites of the crystal lattice [19]. The exchange interaction in such clusters can take place in Mn—Se—Mn pairs and, probably, in Mn—In—Se—Mn ones [12]. The ferromagnetism of those clusters was discovered owing to the appearance of hysteresis loops in the magnetic moments of the clusters at $T < 350$ K.

The experimentally found value of the average magnetic moment per manganese ion amounts to $0.005\mu_B$ instead of $5.9\mu_B$. That is, only some portion of manganese ions participates in the ferromagnetic interaction, while the rest of them is in the paramagnetic state and belongs to either the antiferromagnetic phase MnSe or the pairs of antiferromagnetically coupled manganese ions.

We may suppose that the ferromagnetic state arises also owing to the interaction between Mn ions through the 2D electron gas located at the interface between the Se—In—In—Se layer and the van der Waals gap (2D ferromagnetism).

The low solubility of transition metals in InSe and the emergence of structural defects are the origin of the non-uniform distribution of manganese in this crystal. This can favor the formation of magnetically ordered clusters.

The number of isolated $Mn^{2+}$ ions in the crystal should be small, because no temperature dependence of the Curie—Weiss type was observed for the MS.

We may suppose that the peculiarities of the chemical bond and the superexchange in InSe⟨Mn⟩ lead to the generation of $Mn^{3+}(d^4)$ and $Mn^{+}(d^6)$ ions, which can be of the Van Vleck type. Owing to the even number of $d$-electrons, these ions are in a non-magnetic singlet state in the field of a local crystalline environment. Such ions can be responsible for the temperature-independent paramagnetism. The weak dependence of the paramagnetic MS on the temperature in low and average magnetic fields or its absence support such a hypothesis.

The fact that manganese can exist in several states, which play independent roles in the formation of magnetic properties, can be proved by means of thermoelectric studies. Earlier, we made such researches for PbSnMnTe [20] and GaMnAs [21].

The exchange interaction between manganese ions in the crystal and the nonequivalence of their local symmetry result in the absence of the group of six lines of superfine splitting (this group is characteristic of $Mn^{2+}$ ions) in the EPR spectrum of InSe. We may assume that those lines become broadened, and the fine structure disappears. The EPR spectra registered for the annealed specimens of InSe with 5 at.% of Mn are similar to those of the unannealed specimen of InSe with 1.25 at.% of Mn, which were obtained in works [7, 8]. It is obvious that the time of annealing was insufficiently long in our case for replacing all the Mn ions (about 5 at.%) into the interlayer space. Thus, a part of them should remain to stay in the layers. Therefore, the more intensive line ($I$) is caused by Mn ions which are located in the interlayer space, while the other ($L$) by the ions in crystal layers.

It should also be noticed that, in some cases, we have registered an extremely narrow line with $g = 2$ in the EPR spectra, the appearance of which is associated with a possible pollution of the specimen surface.

While discussing the features of the temperature dependence of MS, one should bear in mind that, according to the conclusion of work [22], the MnSe phase (its fraction of 10% was revealed making use of the XRD method) is antiferromagnetic with the Néel temperatures of 130 and 266 K. The authors of work [22] observed distinct peaks in the temperature dependence of the MS at $T = 150$ and 250 K.

The temperature position of a sharp spike in the MS of InSe⟨Mn⟩ at $T \approx 260$ K is close to the temperature of structural phase transition from a cubic modification of the NaCl type into a hexagonal modification of the NiAs type, which is characteristic of the MnSe phase at 266 K. The hexagonal phase is antiferromagnetic at temperatures $T < 266$ K [22]. We associate the smeared paramagnetic maximum of the MS at about 160 K with the second antiferromagnetic transition in the cubic modification of the MnSe phase. It is probable that the magnetic structure of antiferromagnetic MnSe phases becomes noncollinear in an anisotropic solid solution, and, as a result, weak ferromagnetism appears.

The broadening of the peak at 160 K in the magnetic field of 6 kOe and its disappearance at $H = 50$ kOe evidence for the decay of the antiferromagnetic state of the MnSe phase and the inclusion of the corresponding domains into the ferromagnetic system of the crystal. As concerning the dynamics of the MS spike at $T \approx 260$ K, a similar temperature-induced shift of the structural phase transition in strong external magnetic fields was observed for $Pb_{0.99}Ge_{0.01}Te$ in work [23] and was considered there in the frames of the interband electron-phonon interaction theory.

An enhancement of the magnetic response of the system at its cooling in a magnetic field, which was registered in the whole temperature interval, testifies to the superparamagnetism in the InSe⟨Mn⟩ crystal; the appearance of the phenomenon of superparamagnetism is connected with the formation of nano-sized single-domain ferromagnetic clusters, which occurs owing to a non-uniform distribution of manganese in the lattice. Hence, the magnetic structure of the crystal is diverse, being governed by the superposition of various magnetic states: ferromagnetic clusters with various dimensions, MnSe phase inclusions, and Van Vleck ions $Mn^{3+}(d^4)$ and $Mn^{+}(d^6)$.

## 5. Conclusions

The crystalline structure of a layered InSe semiconductor doped with 5 at.% of the manganese impurity has been studied for the first time. The formation of the solid solution $In_{1-x}Mn_xSe$, the inclusions of the MnSe phase, which is antiferromagnetic at low temperatures, and ferromagnetically ordered clusters with $T_C > 350$ K has been established. The saturation magnetization of about $(3.3 \div 7) \times 10^{-3}$ emu/g in the field of 6 kOe, the residual magnetization of $(0.45 \div 1.65) \times 10^{-3}$ emu/g, and the coercive force that reaches 620 Oe were registered.

We assume that there may exist $Mn^{3+}(d^4)$ and $Mn^{+}(d^6)$ ions in the crystal. Those ions are characterized by the Van Vleck behavior rather than the Curie—Weiss one and give rise to the paramagnetic MS component which is independent of the temperature.

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МАГНІТНІ ВЛАСТИВОСТІ InSe, ЛЕГОВАНОГО МАРГАНЦЕМ

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Р е з ю м е

У температурному інтервалі 4,2 — 300 K досліджено статичну магнітну сприйнятливість (МС) і спектри електронного парамагнітного резонансу (ЕПР), а також кристалічну структуру при $T$=300 K шаруватого напівпровідника InSe, який містив ∼5 ат. % марганцю. Встановлено утворення твердого розчину $In_{1-x}Mn_xSe$ (∼90 об. %), феромагнітних кластерів з температурою Кюрі, вищою за 350 K, і включень антиферомагнітної фази MnSe (∼10 об. %). Фонова позитивна МС практично не залежить від температури і частково зумовлена цими феромагнітними станами в області насичення магнітного момента і нетиповими ван-флеківськими іонами $Mn^{3+}(d^4)$ і $Mn^{+}(d^6)$ . Максимуми МС при 160 і 270 K пов'язані з антиферомагнітними переходами у фазі MnSe. Широка лінія ЕПР зумовлена слабовзаємодіючими іонами $Mn^{2+}$, які не входять до складу феромагнітних і антиферомагнітних кластерів.