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ELECTRONIC STRUCTURE CHANGE AT CATIONIC SUBSTITUTION OF MANGANESE SULFIDE BY ELEMENTS WITH VARIABLE VALENCE

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Cation-substituted solid solutions $Yb_XMn_{1-X}S$ were prepared by the melt method from polycrystalline sulfide powders. The synthesized samples are antiferromagnetic semiconductors and, according to the results of X-ray structural analysis, have an FCC structure of the NaCl type. Structural, electrical, optical, and acoustic properties of the chalcogenide system $Yb_XMn_{1-X}S$ were studied in the temperature range 80–500 K. The effect of variable valence elements on the electronic structure of cationic substitution of manganese sulfide has been studied. The change in the electronic structure in the $Yb_XMn_{1-X}S$ system occurs due to the electron-phonon interaction. Samples with variable valence have anomalous compressibility, which is confirmed by the data on the thermal expansion coefficient and the change in the attenuation coefficient. As a result of inelastic interaction with d- electrons, the density of states at the Fermi level changes, this is reflected in the temperature dependence of the conductivity. The positions of the f-level and two electronic transitions were determined from the IR spectra. A zone of temperatures and concentrations was found, where a correlation of structural, electrical, optical and acoustic properties is observed. To explain the experimental results, the electronic structure of the semiconductor is considered and a model is proposed that qualitatively describes the experiment.

Keywords: elements with variable valence, structure, IR spectroscopy, attenuation coefficient, conductivity, electronic structure.

ИЗМЕНЕНИЕ ЭЛЕКТРОННОЙ СТРУКТУРЫ ПРИ КАТИОННОМ ЗАМЕЩЕНИИ СУЛЬФИДА МАРГАНЦА ЭЛЕМЕНТАМИ С ПЕРЕМЕННОЙ ВАЛЕНТНОСТЬЮ

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Методом расплава из поликристаллических порошков сульфидов приготовлены катион замещенные твердые растворы $Yb_XMn_{1-X}S$. Синтезированные образцы являются антиферромагнитными полупроводниками и согласно результатам рентгеноструктурного анализа имеют ГЦК структуру типа NaCl. Проведены исследования структурных, электрических, оптических и акустических свойств халькогенидной системы $Yb_XMn_{1-X}S$ в интервале температур 80–500 К. Исследовано влияние на электронную структуру элементов переменной валентности при катионном замещении сульфида марганца. Изменение электронной структуры в системе $Yb_XMn_{1-X}S$ происходит за счет электрон-фононного взаимодействия. Образцы с переменной валентностью обладают аномальной сжимаемостью, что подтверждается данными коэффициента теплового расширения и изменением коэффициента затухания. В результате неупругого взаимодействия с d-электронами меняется плотность состояний на уровне Ферми, что отражается на температурной зависимости проводимости. Из ИК спектров определены положения f-уровня и два электронных перехода. Обнаружена область темпера-

тур и концентраций, где наблюдается корреляция структурных, электрических, оптических и акустических свойств. Для объяснения экспериментальных результатов рассмотрена электронная структура полупроводника и предложена модель, качественно описывающая эксперимент.

Ключевые слова: элементы с переменной валентностью, структура, ИК-спектроскопия, коэффициент затухания, проводимость, электронная структура.

Introduction. Recently, active research has been carried out in the field of spin electronics or spintronics, which is one of the priority areas of modern electronics [1–3]. In spintronics, not only the charge degree of freedom of the electron, but also the spin is used to convert an electrical signal, which makes it possible to create fundamentally new spintronic devices based on materials with a magnetoresistive effect. These substances include the cation-anionic semiconductors which we investigate, and which are synthesized on the basis of manganese chalcogenides and they demonstrate phase transitions, and a number of effects associated with changes in the electronic structure under the influence of external factors [4–10]. A similar correlation of changes in the electronic and crystal structures was observed in bismuth pyrovskites [11–13]. The study of these phenomena in manganese chalcogenides seems to be relevant, both from the point of view of technical applications and from the point of view of fundamental physics.

Manganese monosulfide, on the basis of which the studied compound $Yb_XMn_{1-X}S$ was synthesized, is an antiferomagnetic (T_N = 150 K) p-type semiconductor [14; 15]. It has an fcc structure of the NaCl type with the unit cell parameter a = 5.22 nm and undergoes a number of structural distortions at temperatures below the Néel temperature [16]. It reveals the anisotropy of the electrical resistance, which in the (111) plane is two orders of magnitude greater than in the (100) plane and depends on the applied magnetic field. The negative magnetoresistance found in the (111) plane for MnS was – 12 % in a magnetic field of 10 kOe at T = 230 K [17].

When manganese cations are replaced by ytterbium ions, the pressure exerted by the nearest environment will lead to a change in the valence of ytterbium ions and to the formation of a metal bond. This was observed in YbS compounds under pressure [18]. Substitution of ytterbium for manganese will cause an f-level shift. Several options are possible here, the f-level falls into the conduction band, and the electron passes from the f-to the d-level of the rare-earth ion, or remains below the bottom of the conduction band, and bound to the donor. The transition of an electron from the f-level to the t_{2g} state will lead to electronic degeneracy, which can be lifted due to the spin-orbit or Jahn–Teller interaction. In the work [19], the effect of a magnetic field on the electrical and magnetic properties in $Yb_XMn_{1-X}S$ ($0.05 < X < 0.2$) was studied. A change in the type of conductivity from the Poole–Frenkel law to the Mott law was found from the current-voltage characteristics and a change in the sign of the current carriers, both in temperature and in concentration. The effect of prehistory under the action of a magnetic field on the conductivity, impedance, and magnetic susceptibility was found in a wide temperature range. The trivalent state of ytterbium ions from EPR was found. The experimental data are satisfactorily explained by the condensation of lattice polarons with the formation of orbital glass with successive freezing of interstitial orbital angular momentum and with orbital angular momentum at the site.

In solid solutions with a disordered arrangement of ytterbium ions, a situation is possible when ions of different valences occupy crystallographically equivalent positions, but the exchange of electrons between them turns out to be relatively slow and is associated with thermally activated hopping with emission and absorption of phonons. At high temperatures, the exchange between ions of different valence occurs rather quickly, which causes an increase in conductivity, and with decreasing temperature, the characteristic times increase sharply and the situation becomes close to static. This will manifest itself in the study of acoustic properties, in softening and splitting of sound vibration modes, in particular, in damping of acoustic phonons, which is sensitive to valence fluctuations and to local deformations accompanying them. Acoustic properties which main parameters are elastic moduli and ultrasound damping are very sensitive to the defect structure of the crystal [20]. In studies of high-temperature superconductors [21–23], it was found that the structure (grain size, presence of pores, etc.), as well as defects of the grain itself, can largely determine the acoustic characteristics of ceramics. The experience with ceramic compounds has shown that acoustic measurements make it possible to effectively control the quality of synthesized samples. In the work [24], a new method was proposed for studying the metal-insulator transition using acoustic effects. They were measured by successive irradiation of the sample in the absence of a magnetic field and in a magnetic field of 6 T at 4.2 K. By successive irradiation of the *n*-GaAs/AlGaAs heterostructure, the conductivity can be increased fivefold. The absorption of ultrasound in metals has been studied for a long time [25]. This phenomenon is closely related to electron-phonon heat exchange [26], which determines the possible scale of violation of thermodynamic equilibrium of the electron gas and phonons and the lattice.

Thus, the aim of this work is to establish possible phase transitions and changes in the electronic structure caused by the substitution of manganese by ytterbium ions as a result of a comprehensive study of optical, electrical, structural and acoustic properties. It should be noted that optical and acoustic studies are sensitive to local distortions of the structure; therefore, the selected methods will allow obtaining detailed information on phase transitions in $Yb_XMn_{1-X}S$ systems.

Experimental results and discussion. *Synthesis and experimental technique.* Cation-substituted solid solutions of the $Yb_XMn_{1-X}S$ system with a substitution concentration ($0 \le X \le 0.2$) were grown in a quartz reactor from a melt of polycrystalline sulfide powders. A reactor with a charge in glassy carbon crucibles was pulled through

a single-turn inductor of the HF unit. High-purity argon was used as an inert medium [7]. Determination of the phase composition and crystal structure of the synthesized samples was carried out at 300 K on a DRON-3 *X*-ray facility using CuK_α radiation after their preparation and measurements of their acoustic and optical properties. The electrical resistance measurements were carried out by a four-probe direct current compensation method in the temperature range of 77÷1000 K. IR spectroscopy studies of $Yb_XMn_{1-X}S$ samples by IR spectroscopy were performed on an 2202 IR Fourier spectrometer (FSM) with a spectral resolution of 1 cm^{-1} in a temperature range of 80–500 K and frequencies of 400–7000 cm^{-1} on a polycrystalline sample in the form of tablets with a diameter of 13 mm in a KBr matrix. Acoustic properties were measured directly on the tablets with two piezoelectric sensors glued with silver paste to the tablet planes, one of which was a generator, the other was a receiver of ultrasonic waves. The sound propagation time was $\tau = 10(-6)$ seconds at a frequency of 5 MHz, the sample thickness was 0.4 cm. The sound wave attenuation coefficient was calculated by the formula: $\alpha = \ln(U_0/U)/d$, where U and U_0 are the voltage amplitudes recorded by the generator and the receiver of vibrations, d is the thickness of the tablet.

X-ray structural analysis. According to the results of *X*-ray structural analysis, the synthesized $Yb_XMn_{1-X}S$ samples with a low substitution concentration $X \leq 0.05$ have a cubic lattice of the NaCl type, characteristic of α-MnS (fig. 1, *a*). With an increase in concentration to $X = 0.2$, along with diffraction reflections of the cubic structure, very weak reflections of the impurity Yb_2S_3 phase with a cubic structure (space group *Ia3*) are observed, which do not disappear upon heat treatment (fig. 1, *b*). The result of exposure to temperature is redistribution of the intensity of diffraction reflections on *X*-ray diffraction patterns. The main peak [200] in the *X*-ray diffraction pattern for the $Yb_XMn_{1-X}S$ solid solution splits into two with increasing *X*, which is possibly associated with the cooperative Jahn – Teller effect and a decrease in cubic symmetry. Cationic substitution with ytterbium leads to a linear increase in the unit cell parameter in accordance with an increase in the radius of the substituting element ($r = 0.91$ nm for Mn and $r = 0.101$ nm for Yb). The concentration dependence of the lattice parameter for the ytterbium-substituted system is shown in the inset to fig. 1, *a*.

Investigation of samples by IR spectroscopy and acoustic method. The substitution of ytterbium for manganese in the $Yb_XMn_{1-X}S$ system leads to structural distortions accompanied by a change in the electronic spectrum, which can be established using IR spectroscopy. Distortion of octahedra, due to the Jahn-Teller effect, was observed in the IR spectra in the form of splitting of the intensity lines shown in fig. 2. In the frequency range of 900–1100 cm^{-1} in $Yb_{0.15}Mn_{0.85}S$, a splitting of the absorption peak into two maxima at frequencies $\omega_1 = 953$ cm^{-1} and $\omega_2 = 994$ cm^{-1} was found. When heated to $T > 200$ K, the first maximum detected at the frequency ω_1 disappears, and the intensity of the second one decreases several times and disappears at 400 K. With increasing temperature, ω_1 and ω_2 shift by 3 cm and 12 cm, respectively. Substitution of manganese by ytterbium ions leads to the formation of electrons in the t_{2g} subsystem and to the degeneracy of electronic states, which are removed due to the Jahn-Teller effect. Two electronic transitions were found from the IR spectra for $X = 0.15$, one disappears at 200 K and the other at 400 K, which are caused by the successive growth of the octahedral symmetry and the splitting of the t_{2g} states of the ions in the zone with electronic phase separation.

Studies of the magnetic properties of the $Yb_XMn_{1-X}S$ system presented in [19] showed that the synthesized compounds are antiferromagnets with T_N decreasing with increasing concentration from 150 K for $X = 0.05$ to 102 K for $X = 0.2$. In the paramagnetic region, two features were observed in the temperature behavior of the magnetization measured in the magnetic field of 8.6 kOe, associated with the local minimum $d\sigma/dT$ at T_1 and T_2, the values of which are in the ranges 224 K < T < 234 K and at 336 K < T < 386 K. Above T_2, the temperature dependence of the magnetic susceptibility is described by the Curie–Weiss law with a negative paramagnetic Curie temperature θ, which sharply decreases when manganese is replaced by ytterbium ions. This can be explained by the formation of superparamagnetic clusters with a strong ferromagnetic interaction between manganese ions, which are the nearest neighbors of the ytterbium ion.

The anomalous behavior of the attenuation coefficient (α) shown in fig. 3 for the sample $Yb_{0.05}Mn_{0.95}S$ is observed in two temperature regions (in the magnetic ordering region and paramagnetic one). The first temperature region correlates with the temperatures of structural distortions (100, 120, 150 K) observed in the initial matrix of manganese monosulfide [27] and with the data of the temperature dependence of the thermal expansion coefficient measured for samples of the $Yb_XMn_{1-X}S$ system [19]. According to which anomalies in the temperature range of the magnetic phase transition 110K ≤ T ≤ 140 K are established on the temperature dependence of the coefficient of thermal expansion. This is due to the fact that when moving to a magnetically ordered region, the lattice begins to contract anomaly, as a result of magnetoelastic interaction.

Above 150 K, the α (T) curve reaches a plateau. An exponential decrease in the value of the attenuation coefficient is observed at $T = 220$ K; at this temperature, the first local minimum of magnetization was detected. The anomalous behavior of the attenuation coefficient in the high temperature range above 400K correlates with the temperatures detected in the IR spectra and is most likely connected with the rearrangement of the electronic spectrum due to the Jahn–Teller effect.

Fig. 4, *a*, *b* illustrates the behavior of the frequency dependence of the sound attenuation coefficient in samples $Yb_{0.05}Mn_{0.95}S$ and $Yb_{0.2}Mn_{0.8}S$ measured at a pulse sequence t = 50 sec in the temperature range 300–450 K. At temperatures of 300 and 320 K for concentrations 0.05 and 0.2, respectively, the α (ω) curves are almost linear. With increasing temperatures, maxima were found on the frequency dependences of the attenuation coefficient. For both the $Yb_{0.05}Mn_{0.95}S$ and $Yb_{0.2}Mn_{0.8}S$ samples, the frequency range 5–5.5 MHz for which anomalies are ob-

served at T = 350 and 400 K coincides (fig. 4, *a*, *b*). A further increase in temperature to 450K results in a step on the α (ω) curve in the low-frequency region at 3 MHz for $Yb_{0.05}Mn_{0.95}S$ and 2.5 MHz for $Yb_{0.2}Mn_{0.8}S$. It is assumed that energy absorption in the room temperature region is caused by internal friction, and the approach to 400 K is due to scattering on crystallographic domains.

When acoustic waves propagate in polycrystalline solid media, reflection, refraction, and transformation occur at grain boundaries, domain boundaries, and structure inhomogeneities. Thus, attenuation is due to both absorption and dissipation of energy. In solids, absorption can be caused by: internal friction (in this case, the sound attenuation coefficient α is proportional to the frequency ω); thermal conductivity (α is proportional to ω^2); elastic effects (α is proportional to ω).

Electrical property. The rearrangement of the electronic structure at the Mn-Yb interface as a result of lowering the local crystal structure and splitting the t_{2g} levels will manifest itself as a result of the study of electrical properties, namely, conductivity. The temperature dependence of the conductivity for solid solutions of $Yb_{0.15}Mn_{0.85}S$ is shown in fig. 5. It has a typical semiconductor type and does not differ qualitatively from the temperature dependence σ(T) for the initial manganese monosulfide. A correlation is found between the temperatures at which anomalies are detected in the IR spectra, the attenuation coefficient, and the temperature dependence of the conductivity (σ). The first jump in the curve σ(T) is observed in the room temperature region. Further along the temperature, a slight increase in conductivity is observed, which turns into a sharp increase in the region of 400 K. This behavior of σ correlates with anomalies detected in the frequency and temperature dependences of the attenuation coefficient and IR spectroscopy data, which confirms the assumptions made above about changes in the electronic structure during cationic substitution of manganese with ytterbium. In the high temperature range of 880 < T < 1020 K, a small maximum in electrical resistance is detected (inset in fig. 3).

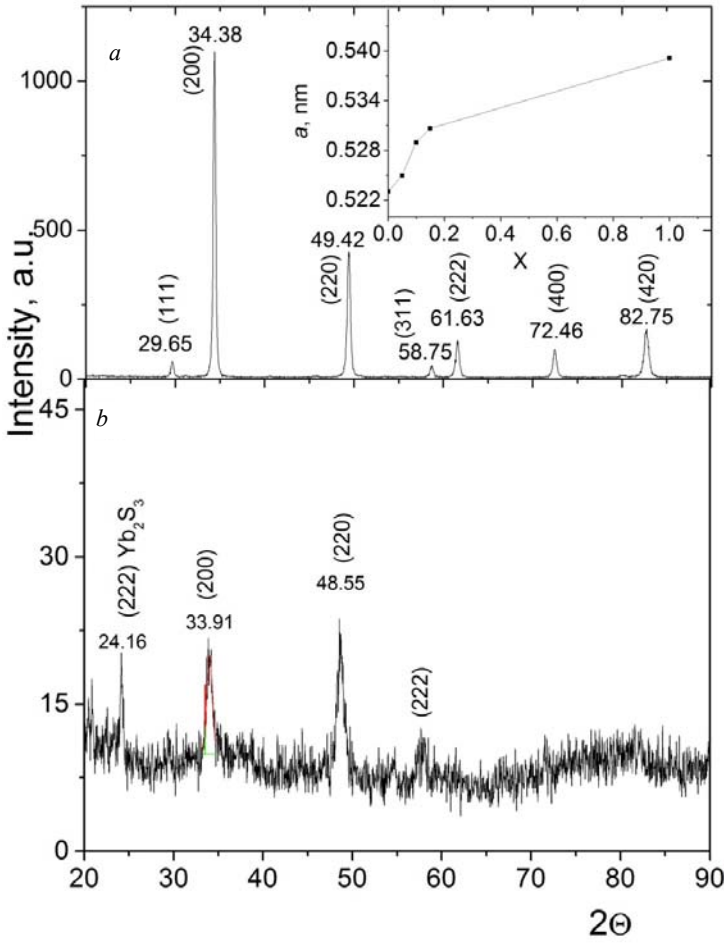


Fig. 1. X-ray diffraction patterns of $Yb_{0.05}Mn_{0.95}S$ (*a*) and $Yb_{0.2}Mn_{0.8}S$ (*b*) samples. The inset shows the concentration dependence of the lattice parameter of the system $Yb_XMn_{1-X}S$

Рис. 1. Рентгенограммы образцов $Yb_{0,05}Mn_{0,95}S$ (*a*) и $Yb_{0,2}Mn_{0,8}S$ (*b*). На вставке концентрационная зависимость параметра решетки системы $Yb_XMn_{1-X}S$

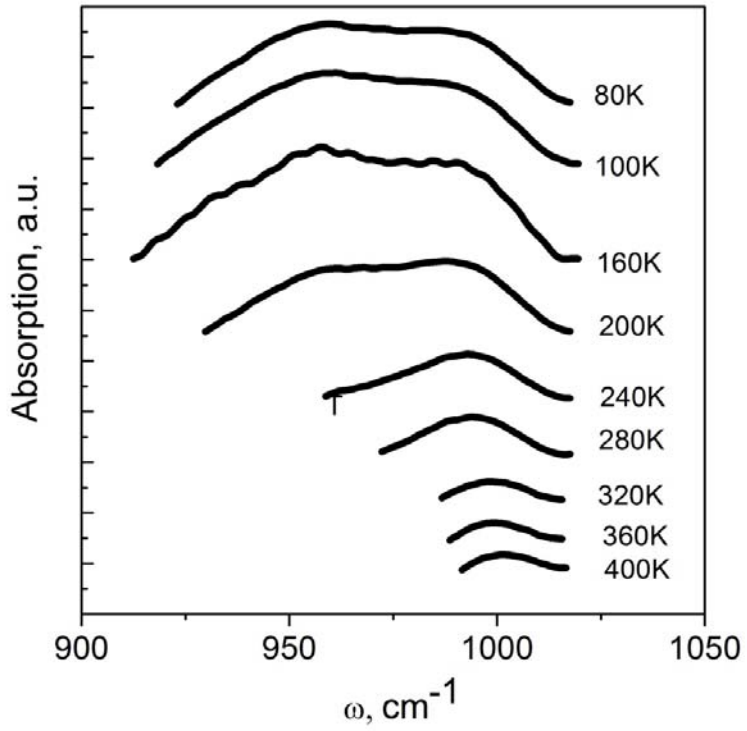


Fig. 2. Temperature dependences of the intensity of the IR spectra of the sample $Yb_{0.15}Mn_{0.85}S$

Рис. 2. Температурные зависимости интенсивности ИК спектров образца $Yb_{0.15}Mn_{0.85}S$

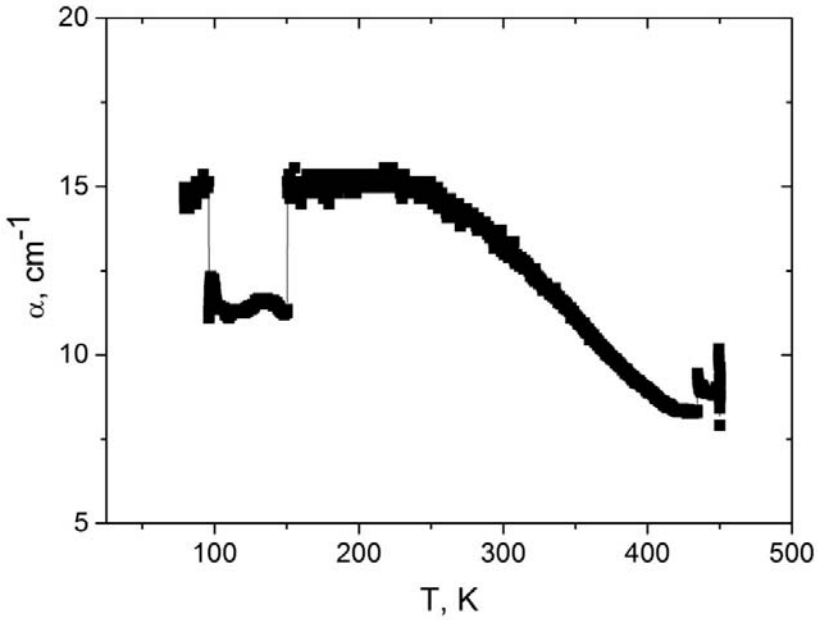
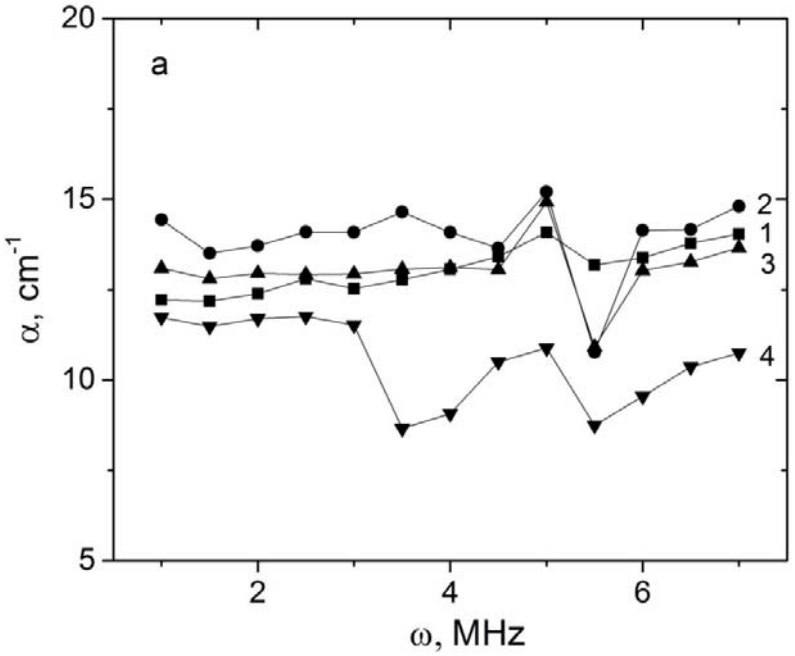
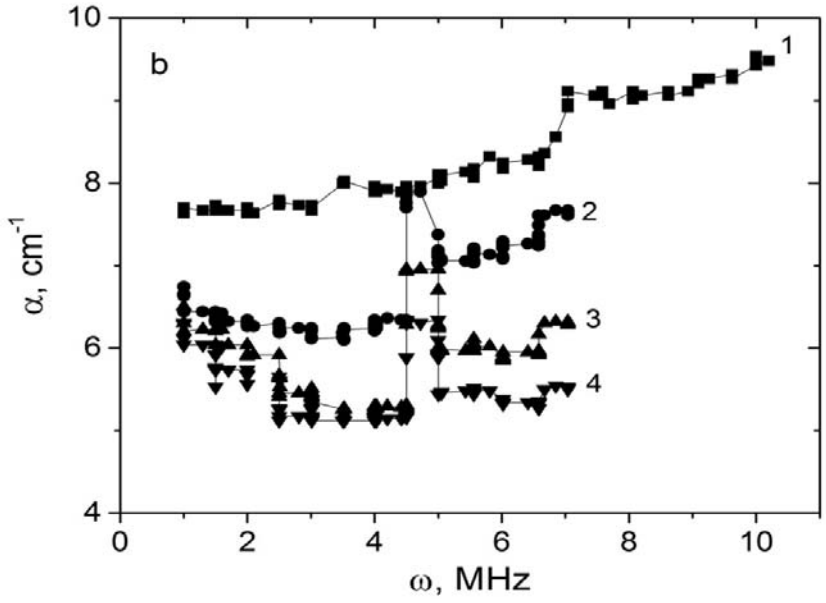


Fig. 3. Temperature dependence of the attenuation coefficient for the sample $Yb_{0,05}Mn_{0,95}S$

Рис. 3. Температурная зависимость коэффициента затухания для образца $Yb_{0,05}Mn_{0,95}S$



a



b

Fig. 4. Frequency dependence of the sound attenuation coefficient for $Yb_{0.05}Mn_{0.95}S$ (*a*) measured at T = 300 K (*1*); 350 K (*2*); 400 K (*3*); 450 K (*4*) and for $Yb_{0.2}Mn_{0.8}S$ (*b*) at T = 320 K (*1*); 360 K (*2*); 400 K (*3*); 440 K (*4*)

Рис. 4. Частотная зависимость коэффициента затухания звука для $Yb_{0,05}Mn_{0,95}S$ (*a*), измеренного при T = 300 К (*1*); 350 К (*2*); 400 К (*3*); 450 К (*4*) и для $Yb_{0,2}Mn_{0,8}S$ (*b*) при T = 320 К (*1*); 360 К (*2*); 400 К (*3*); 440 К (*4*)

This maximum is due to the coincidence of the 4f-level with the chemical potential when it is shifted from the bottom of the conduction band to the middle of the band gap.At low temperatures, the 4f-level is filled and located below the chemical potential, and at high temperatures, electrons are scattered at the f – centers as a result of df-exchange, which leads to a maximum in resistance. It should be noted that with an increase in the concentration to X = 0.2, a jump in the electrical resistance is observed at T = 700 K and the reason for this is the electron-phonon interaction, which induces splitting of the 4f subband. Another reason is the presence of ytterbium ions of different sizes (+2 and +3) with different (up to 10–15 %) ion radii. The electron-lattice interaction

consists of the interaction of electrons with a homogenous strain and with phonons at a given strain.

To explain the experimental results obtained for solid solutions of $Yb_XMn_{1-X}S$, we consider the electronic structure shown in fig. 6. In the band gap below the bottom of the conduction band is the donor level corresponding to 4f ytterbium electrons. The electron concentration at the 4f-level is equal to the concentration of ytterbium ions. In the temperature range 390–440 K, condensation of local octahedral modes is possible, i. e., sulfur ions shift from octahedral positions in the direction of manganese at the Mn-Yb boundary.

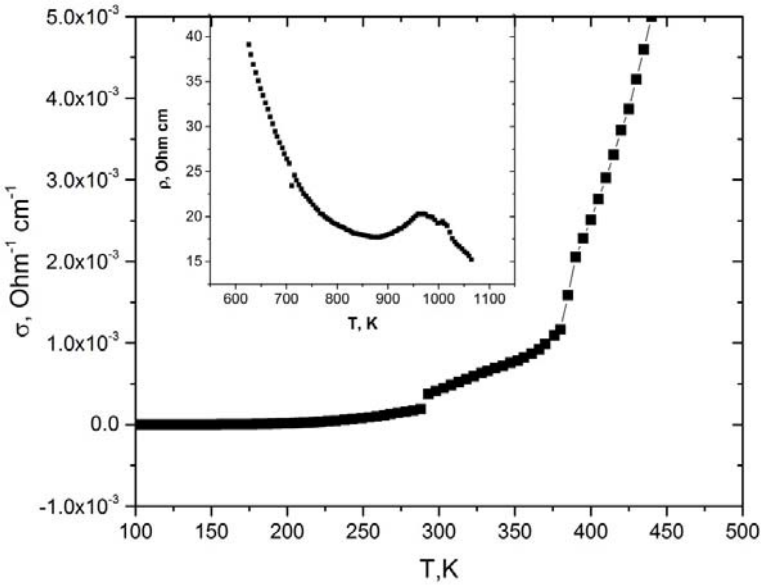


Fig. 5. Temperature dependence of conductivity for the $Yb_{0.15}Mn_{0.85}S$ sample. Inset: temperature dependence of electrical resistivity for the same concentration

Рис. 5. Температурная зависимость проводимости для образца $Yb_{0,15}Mn_{0,85}S$. На вставке: температурная зависимость удельного электросопротивления для этой же концентрации

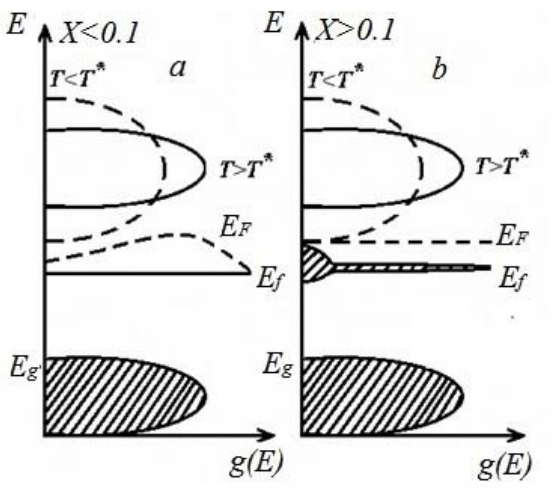


Fig. 6. Temperature dependence of the 4f-level energy E_f and Fermi energy E_F. T^* – temperature at which the activation energy increases, taken from the data on electrical resistance

Рис. 6. Температурная зависимость энергии 4f-уровня E_f и энергии Ферми E_F. T^* – температура, при которой энергия активации увеличивается, взята из данных по электросопротивлению

The bending mode of the octahedron is likely to lead to splitting of the t_{2g} states of electrons and to a shift in the conduction band, as well as a decrease in the energy interval between the f-level and the bottom of the conduction band. There is a shift of the f-level relative to the bottom of the conduction band, which leads to a rearrangement of the electronic structure. At high temperatures, the Fermi energy and the 4f-level intersect at high temperatures for all concentrations.

Conclusion. Cation-substituted solid solutions of $Yb_XMn_{1-X}S$ ($0 \le X \le 0.2$) were synthesized, which mainly have a face-centered cubic lattice of the NaCl type according to the results of *x*-ray diffraction analysis. An increase in the substitution concentration $X = 0.2$ leads to splitting of the main peak on the *x*-ray image, which is associated with the cooperative Jahn–Teller effect and a decrease in cubic symmetry. Structural changes and rearrangements of the electronic spectrum were confirmed by IR spectroscopy. Two electron transitions were detected from the IR spectra, one of which disappears at 200 K and the other at 400 K. These transitions are caused by a sequential increase in the symmetry of the octahedron and with the splitting of the t_{2g} states of the ions. A structural transition for $X = 0.05$ is detected in the magnetically ordered phase, which is accompanied by compression of the crystal lattice and minima on the temperature dependence of the attenuation coefficient. A temperature range is found in which the correlation of structural, magnetic, electrical, optical, and acoustic properties is observed. Comparison of acoustic parameters with the behavior of the temperature dependence of the thermal expansion coefficient of the sample, IR spectroscopy, *x*-ray diffraction data, and conductivity in the paramagnetic region allows us to assume that the detected anomalies indicate the existence of both structural phase transitions and changes in the electronic structure in the sample. The change in the electronic structure as a result of substitution of manganese with elements of variable valence is caused by a shift in the bottom of the conduction band with an increase in temperature as a result of electron-phonon interaction

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