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RESEARCH ON ELECTRICAL PROPERTIES OF MANGANESE SULPHIDES DOPED BY THULIUM AND YTTERBIUM IONS

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Materials exhibiting connection between electrical and magnetic properties are attractive for possible use as an element base in microelectronics, spintronics, and sensor devices. Compounds with mixed valence exhibit a number of metal-insulator phase transitions, magnetic phase transitions, including changes in magnetic properties without changing magnetic symmetry.

Promising materials for studying these effects are cation-substituted $Mn_{1-x}Re_xS$ compounds ($Re = 4f$ elements) synthesized on the basis of the antiferromagnetic semiconductor of manganese monosulfide. The latter is of practical importance in the development of new materials for temperature sensors, widely used in the metallurgical industry.

The structural and electrical properties of compounds with mixed valences $Tm_xMn_{1-x}S$ ($0 \leq x \leq 0.15$) and $Tm_xMn_{1-x}S$ ($0 \leq x \leq 0.25$) were studied in the temperature range 80–1100K. The regions of existence of solid solutions of $Tm_xMn_{1-x}S$ sulfides with an fcc (face-centered cubic) lattice of the NaCl type were determined. It was found that conductivity decreases upon the substitution of manganese cations with thulium ions and the lattice constant increases more sharply in comparison with Vegard's law. When ytterbium ions are substituted, the conductivity increases with increasing concentration and the temperature dependence has the form typical of semiconductors.

Key words: manganese sulfide, mixed valence, conductivity, X-ray diffraction analysis.

ИССЛЕДОВАНИЕ ЭЛЕКТРИЧЕСКИХ СВОЙСТВ СУЛЬФИДОВ МАРГАНЦА ДОПИРОВАННЫХ ИОНАМИ ТУЛИЯ И ИТТЕРБИЯ

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Материалы, обнаруживающие связь между электрическими и магнитными свойствами, являются привлекательными для возможного использования в качестве элементной базы в микроэлектронике, спинтронике, сенсорных устройствах. Соединения с переменной валентностью проявляют ряд фазовых переходов металл-диэлектрик, магнитные фазовые переходы, включая изменения магнитных свойств без изменения магнитной симметрии.

Перспективными материалами для изучения этих эффектов являются катион замещенные соединения $Mn_{1-x}Re_xS$ ($Re = 4f$ элементы), синтезированные на основе антиферромагнитного полупроводника моносulfида марганца. Последнее имеет практическую значимость в разработке новых материалов для датчиков температуры, широко используемых в металлургической отрасли.

Проведены исследования структурных и электрических свойств соединений с переменной валентностью $Tm_xMn_{1-x}S$ ($0 \leq x \leq 0.15$) и $Tm_xMn_{1-x}S$ ($0 \leq x \leq 0.25$) в области температур 80–1100K. Определены области существования твердых растворов сульфидов $Tm_xMn_{1-x}S$ с ГЦК решеткой типа NaCl. Установлено уменьшение проводимости при замещении катионов марганца ионами тулия и более резкое увеличение постоянной решетки по сравнению с законом Вегарда. При замещении ионами иттербия проводимость увеличивается с ростом концентрации и температурная зависимость имеет вид, типичный для полупроводников.

Ключевые слова: сульфид марганца, переменная валентность, проводимость, рентгеноструктурный анализ.

Introduction. Compounds containing rare-earth chemical elements with mixed valence such as Sm, Yb, Ce, Eu, Tm have a number of unique properties. Phase transitions of purely electronic nature and associated with change in the filling of 4f electronic levels [1] often occur in them when external conditions (temperature, pressure, composition) change. At the same time the magnetic properties also change [2–4] (localized magnetic moments disappear), i. e. the transitions are of the “magnetic – nonmagnetic” state type [5]. Change in the type of conductivity from semiconductor to metal type and a significant magnitude of magnetoresistance (of the order of 100 %) in the paramagnetic region at room temperatures and above [6–11] is observed in manganese sulfides substituted by samarium [6] and gadolinium [7] ions.

Thulium sulfide has a cubic crystalline structure with a lattice parameter of 5.412 Å. This compound is characterized by a metallic type of conductivity at $T > 100$ K with electron concentration of about 10^{22} cm⁻³ and resistivity of about 10^{-6} Ohm / cm at room temperature [2]. Thulium whose electronic configuration of the 4f – shell is unstable and close to a filled one, can enter the compounds with other elements, be in the state of $\text{Tm}^{2+} 4f^{13}$ term $^2F_{7/2}$ and $\text{Tm}^{3+} 4f^{12}$ term 3H_6 . In TmS a thulium ion is in the trivalent state with a 4f level filling of $n_f = 0.65$ and the energy difference between the divalent and trivalent states $E^{2+} - E^{3+} = 0.3$ eV [12]. The proximity of the energies of thulium aliovalent states leads to the fact that TmS exhibits a Condo effect in which band electrons group around thulium ions screening its magnetic moment [13]. Under the action of pressure “quasilocalized” states expand and pass into the conduction band, which will manifest itself in the form of transition to the usual metallic state. This is confirmed by the baric dependence of thulium thermopower, which decreases under pressure to 20 GPa, and ceases to change at higher pressures [14]. Pressure leads to changing magnetic characteristics and magnetic structure [15–18]

Ytterbium sulfide at normal pressure is a semiconductor with a direct gap in the spectrum of electronic excitations ~ 1.3 eV and an indirect gap ~ 1.0 eV between the fully occupied *f*-state and free *sd*-band states [19], which are 4 eV higher in energy than the 3 *p*-valence band of sulfur ions. Under pressure the gap monotonically decreases $dE_g / dp = -6 \pm 1$ eV / kbar [20], at 8 GPa the zones overlap and a metallic state arises [21]. At 10 GPa quantum resonance, that is, a superposition of f_{13} and f_{14} states and change in valence from 2 to 4 is observed. The density of current carriers per an ytterbium ion is 0.4 [22]

Materials and methods of research. The synthesis of $\text{Mn}_{1-x}\text{Re}_x\text{S}$ samples is described in detail in [8]. Solid solutions $\text{Mn}_{1-x}\text{Tm}_x\text{S}$ and $\text{Mn}_{1-x}\text{Yb}_x\text{S}$ were obtained by solid-phase synthesis, degrees of substitution 0.05; 0.10; 0.15 and 0.05; 0.10; 0.15; 0.2; 0.25 respectively.

X-ray diffraction analysis of $\text{Mn}_{1-x}\text{Tm}_x\text{S}$ ($x = 0.05; 0.15$) and $\text{Mn}_{1-x}\text{Yb}_x\text{S}$ ($x = 0.1; 0.2$) sulfides was carried out on a DRON-3 installation in $\text{CuK}\alpha$ -radiation at temperature of 300 K after they were obtained and their transport properties were measured. X-ray diffraction

patterns obtained after the measurements indicate that all the substances studied have a face-centered cubic (fcc) structure of the NaCl type typical of manganese monosulfide.

The conductivity was measured in the temperature range 80–1100 K using the four-probe method. The four-probe method for measuring electrical resistivity is the most common. The method is very convenient since there is no need to create ohmic contacts; it is possible to measure the resistivity of bulk samples of the most diverse shapes and sizes, as well as the resistivity of layers of semiconductor structures, for example, during ion implantation. In this case, one condition must be fulfilled as far as the shape is concerned, that is, the presence of a flat surface whose linear dimensions exceed the linear dimensions of the probe system (the distance between them).

Four metal probes with a small contact area (fig. 1) the distances between which are s_1, s_2, s_3 , are placed on the flat surface of the sample along a straight line. Electric current I_{14} is passed through two external probes 1 and 4, and the potential difference U_{23} is measured on two internal probes 2 and 3.

Results and discussion. X-ray diffraction patterns of the synthesized sulfides were obtained (fig. 2, 3). X-ray diffraction patterns obtained after measurements indicate that all the substances studied have a stable crystalline state up to temperatures of the order of 1100 K. X-ray diffraction analysis showed that the synthesized compounds are single-phase and have a face-centered cubic (fcc) structure of the NaCl type, typical of manganese monosulfide. With increase in the degree of cationic substitution (x), the unit cell parameter a increases linearly, which indicates the formation of $\text{Mn}_{1-x}\text{Tm}_x\text{S}$ and $\text{Mn}_{1-x}\text{Yb}_x\text{S}$ solid solutions (fig. 2, 3). Increase in the lattice constant, compared with linear growth according to Vegard's law, is possibly associated with the localization of electrons at the interface of manganese ions with substituted ones and with weak hybridization of 4f-3d orbitals, which is described by an exponential dependence on distance.

The temperature dependence on conductivity was measured for the synthesized samples $\text{Mn}_{1-x}\text{Tm}_x\text{S}$ ($0.01 \leq x \leq 0.15$) and $\text{Mn}_{1-x}\text{Yb}_x\text{S}$ ($0.05 \leq x \leq 0.25$) (fig. 4). The behavior of the $\ln \sigma$ ($10^3 / T$) dependences is characteristic of substances with semiconductor conductivity. Fig. 4, *a* shows the electrical conductivity of $\text{Tm}_x\text{Mn}_{1-x}\text{S}$ solid solutions. A sample with a thulium substitution degree of $x = 0.05$ has a conductivity plateau from 310 to 380 K. For $x = 0.1$ there is an anomaly in the conductivity behavior from 330 to 360 K. For $x = 0.15$ there is an anomaly in the conductivity behavior from 650 K to 690 K. All of the studied samples with thulium are characterized by a high resistance state even at room temperature in comparison with the electrical resistance observed in manganese monosulfide [8]. Fig. 4, *b* shows the electrical conductivity of $\text{Tm}_x\text{Yb}_{1-x}\text{S}$ solid solutions. When the temperature increases the conductivity grows significantly faster in the case of ytterbium than in the case of thulium.

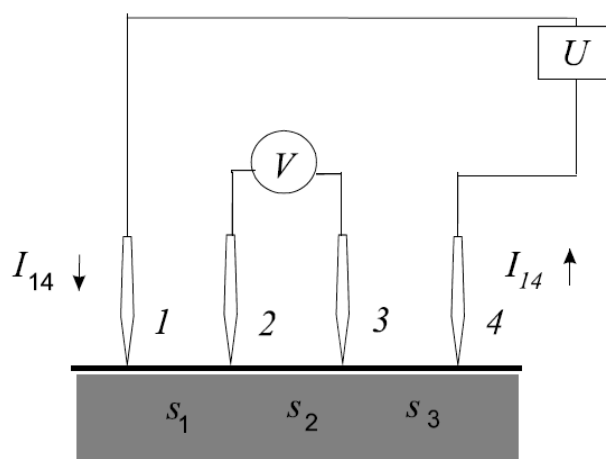


Fig. 1. A schematic diagram of measurements using the four-probe method

Рис. 1. Принципиальная схема измерений четырёхзондовым методом

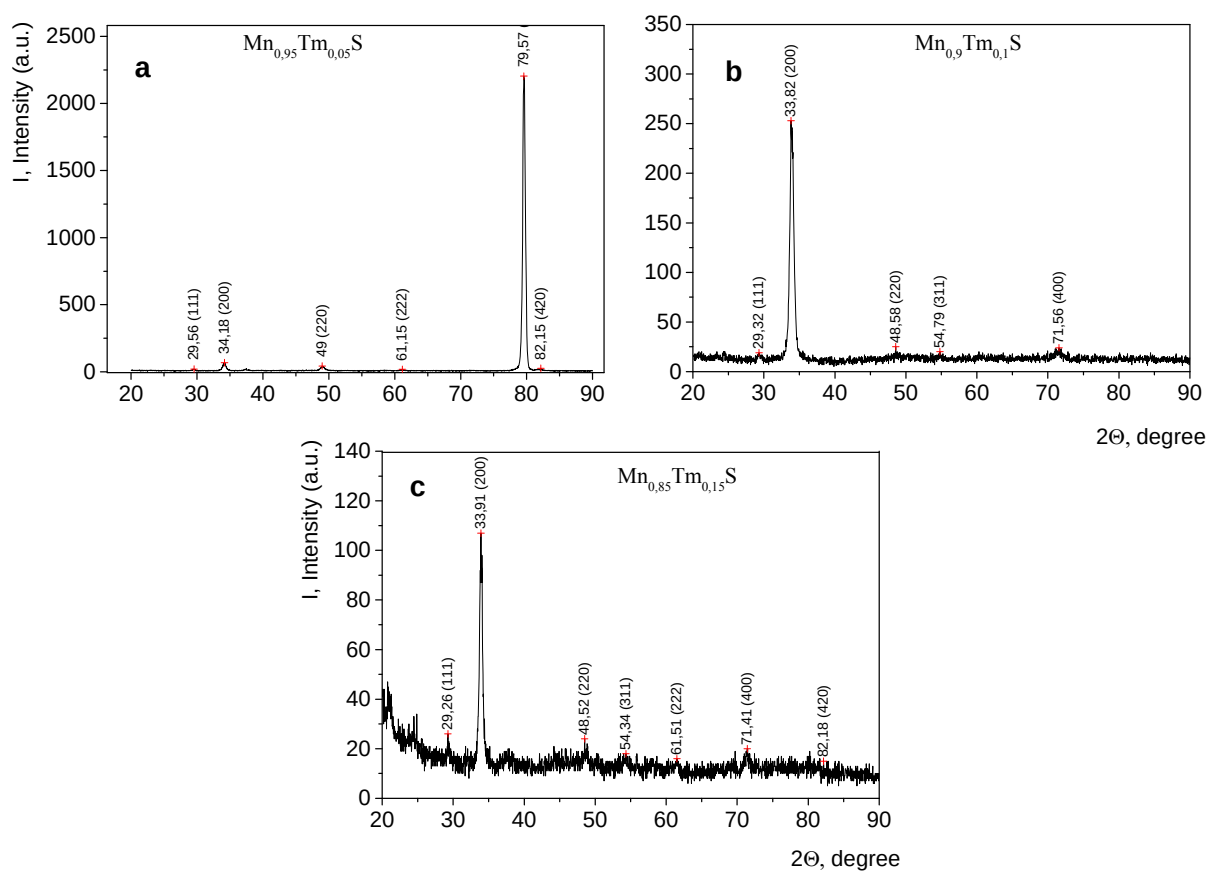


Fig. 2. X-ray diffraction patterns of $\text{Mn}_{1-x}\text{Tm}_x\text{S}$:
a - $x = 0.05$; b - $x = 0.1$; c - $x = 0.15$

Рис. 2. Рентгенограммы $\text{Mn}_{1-x}\text{Tm}_x\text{S}$:
a - $x = 0.05$; b - $x = 0.1$; c - $x = 0.15$

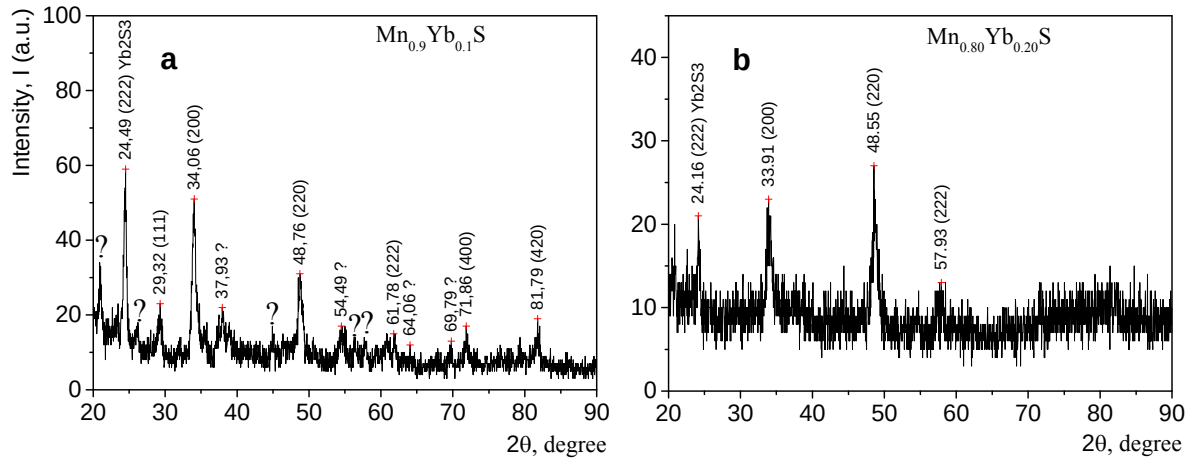


Fig. 3. X-ray diffraction patterns of $\text{Mn}_{1-x}\text{Yb}_x\text{S}$:
 a – $x=0.1$; b – $x=0.2$

Рис. 3. Рентгенограммы $\text{Mn}_{1-x}\text{Yb}_x\text{S}$:
 a – $x=0.1$; b – $x=0.2$

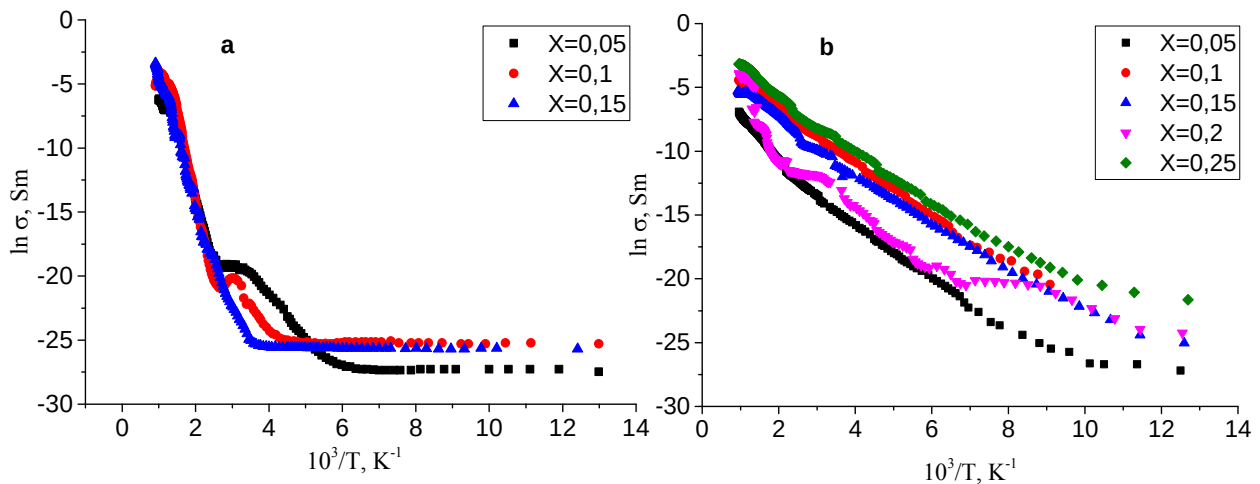


Fig. 4. Temperature dependence on conductivity:
 a – $\text{Mn}_{1-x}\text{Tm}_x\text{S}$ ($0.05 \leq x \leq 0.15$); b – $\text{Mn}_{1-x}\text{Yb}_x\text{S}$ ($0.05 \leq x \leq 0.25$)

Рис. 4. Зависимость проводимости от температуры:
 a – $\text{Mn}_{1-x}\text{Tm}_x\text{S}$ ($0.05 \leq x \leq 0.15$); b – $\text{Mn}_{1-x}\text{Yb}_x\text{S}$ ($0.05 \leq x \leq 0.25$)

The dependences of the conductivity on the substitution concentration of thulium and ytterbium ions at room temperature are shown in fig. 5. With increase in the degree of samples doping with thulium the conductivity decreases (fig. 5, a), and when doping with ytterbium a similar conductivity behavior occurs, but there is a section in the concentration range from $x = 0.1$ to $x = 0.2$ in which, on the contrary, growth is observed. In general, there is a nontrivial picture that is different from the behavior of impurity semiconductors, in which substitution with an alloying element increases the concentration of charge carriers and, as a result, the conductivity.

This behavior can be explained by the fact that an ytterbium ion is trivalent and, when a divalent manganese ion is substituted in a solid solution, both electrons and holes are formed by non-stoichiometry of the obtained samples. With increase in the substitution concentration, electrons and holes accumulate at intercrystalline boundaries and form a carrier-depleted layer similar to the p-n junction. Substitution with ytterbium ions leads to the formation of holes in the cationic subsystem; as a result, the conductivity decreases sharply compared to manganese sulfide. As the concentration of hole current carriers increases, the conductivity increases (fig. 5, b).

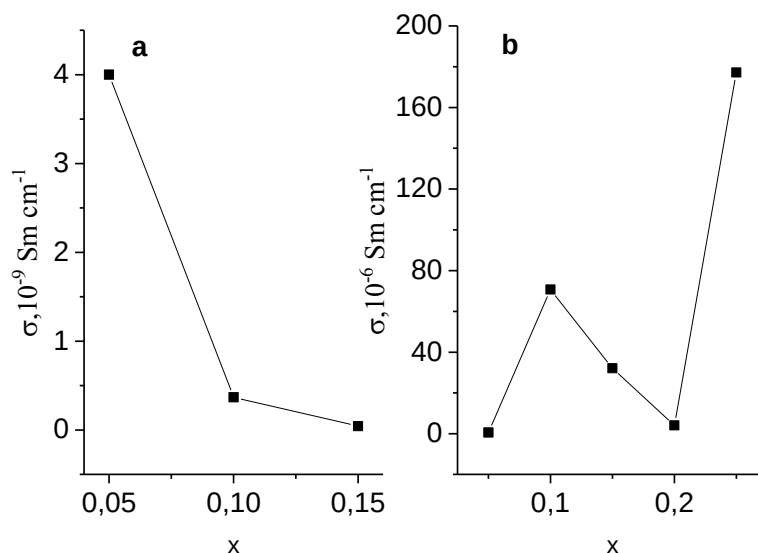


Fig. 5. Dependence of conductivity on the temperature of samples $\text{Mn}_{1-x}\text{Yb}_x\text{S}$ ($0.05 \leq X \leq 0.25$)

Рис. 5. Зависимость проводимости от температуры образцов $\text{Mn}_{1-x}\text{Yb}_x\text{S}$ ($0.05 \leq X \leq 0.25$)

Conclusion. X-ray diffraction analysis of solid solutions of manganese sulfides substituted by rare-earth ions of thulium and ytterbium was carried out. It was found that the synthesized compounds are single-phase ones and have a face-centered cubic (fcc) structure. There is increase in the unit cell when substituted by thulium and ytterbium.

Decrease in the conductivity upon substitution with thulium and increase in the conductivity of solid solutions upon substitution with ytterbium were found. The temperature dependence of substituted sulfides has a semiconductor form. The concentration dependence of the conductivity for $\text{Mn}_{1-x}\text{Tm}_x\text{S}$ is explained by the formation of a space charge at the boundaries of intercrystalline grains.

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