

**For citation:** Sitnikov M. N., Kharkov A. M., Aplesnin S. S., Romanova O. B. Influence of the magnetic field on transport properties of holmium – manganese sulfide. *Siberian Journal of Science and Technology*. 2020, Vol. 21, No. 3, P. 451–458. Doi: 10.31772/2587-6066-2020-21-3-451-458

**Для цитирования:** Влияние магнитного поля на транспортные свойства гольмий-марганцевого сульфида / М. Н. Ситников, А. М. Харьков, С. С. Аплеснин, О. Б. Романова // Сибирский журнал науки и технологий. 2020. Т. 21, № 3. С. 451–458. Doi: 10.31772/2587-6066-2020-21-3-451-458

## INFLUENCE OF THE MAGNETIC FIELD ON TRANSPORT PROPERTIES OF HOLMIUM – MANGANESE SULFIDE

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*Holmium-manganese sulfide with giant magnetoresistance refers to new magnetic sulfide compounds of holmium and manganese that have the effect of giant magnetoresistance (i. e., with special magnetoelectric properties), which can be used as components of sensor technology, magnetic memory, and spintronics. The technology of manufacturing polycrystals  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  grown by crystallization from the melt of the obtained powdered sulfides with a purity not lower than 99,9 %, in glass-carbon crucibles and a quartz reactor in an argon atmosphere is presented. According to the results of x-ray diffraction analysis,  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  holmium-manganese sulfides have a HCC structure of the NaCl type. As the degree of cationic substitution increases, the unit cell parameter increases linearly with the concentration. No concomitant impurity phases are detected in the synthesized samples. To determine the state of the spin glass, magnetic moment measurements are conducted at several frequencies  $\omega = 1$  kHz, 10 kHz and 100 kHz. The dependence of magnetic characteristics on the frequency of measurements is found. The damping of the magnetic moment and its increase with a decrease in temperature is revealed, which is connected with the formation of metastable states. Measurements of electrical resistance without a field and in a magnetic field are conducted. Anomalies in the temperature dependence of the conductivity are found. A change in the magnetoresistance sign is detected with the increase of temperature below and above room temperature.*

*Keywords: solid solutions, resistance, magnetic permeability, the effect of giant magnetoresistance.*

## ВЛИЯНИЕ МАГНИТНОГО ПОЛЯ НА ТРАНСПОРТНЫЕ СВОЙСТВА ГОЛЬМИЙ-МАРГАНЦЕВОГО СУЛЬФИДА

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*Гольмий-марганцевый сульфид с гигантским магнитосопротивлением относится к новым магнитным сульфидным соединениям гольмия и марганца, обладающим эффектом гигантского магнитосопротивления, то есть с особыми магнитоэлектрическими свойствами, которые могут быть использованы в качестве составляющих компонент сенсорной техники, магнитной памяти для спинтроники. Приведена технология изготовления поликристаллов  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$ , выращенных кристаллизацией из расплава полученных порошковых сульфидов чистотой не ниже 99,9 % в стеклоуглеродных тиглях и кварцевом реакторе в атмосфере аргона. Согласно результатам рентгеноструктурного анализа, гольмий-марганцевые сульфиды  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  имеют ГЦК структуру типа NaCl. С увеличением степени катионного замещения параметр элементарной ячейки линейно увеличивается с концентрацией. Сопутствующих примесных фаз в синтезированных образцах не обнаружено. Для установления состояния спинового стекла проведены измерения магнитного момента на нескольких*

частотах  $\omega = 1, 10$  и  $100$  kHz. Обнаружена зависимость магнитных характеристик от частоты измерений. Найдено затухание магнитного момента и его увеличение с понижением температуры, что связывается с образованием метастабильных состояний. Проведены измерения электросопротивления без поля и в магнитном поле. Найдены аномалии в температурной зависимости проводимости. Обнаружена смена знака магнитосопротивления с ростом температуры ниже и выше комнатной температуры.

*Ключевые слова:* твердые растворы, электросопротивление, магнитная проницаемость, эффект гигантского магнитосопротивления.

**Introduction.** The microelectronics element base, functioning on the basis of the magneto resistive effect [1], can operate under extreme conditions. Materials with the effect of giant magnetoresistance (GMR) can be used as components of sensor technology [2], magnetic memory [3] and are able to change their electrical resistance in several times under external magnetic field application [4–7].

Summing up, it is possible to say that all currently known GMR substances are complex (oxide) phases based on manganese oxides, the ferromagnetism of ions of which is responsible for the GMR effect emerging [8]. Oxide compounds of manganese type  $\text{La}_{1-x}\text{A}_x\text{MnO}_3$  ( $\text{A} = \text{Ca}, \text{Sr}, \text{Ba}, \text{etc.}$ ) and methods for their preparation [8; 9] are known. The maximum amplitude of this effect is observed in the immediate vicinity of the transition to the ferromagnetic state, at high values of the Curie temperature  $T_C \sim 250\text{--}400$  K and opens up broad prospects of their technological application.

The drawback of these substances is the high sensitivity of lanthanum manganite to concentration of a divalent impurity, a high melting point  $T \sim 1800\text{--}1900$  °C, the cost of their constituent elements and realization of the GMR effect in a narrow temperature interval near the magnetic transition temperature. NaCl-type fcc lattice.

The vanadium chromium disulfide – copper  $\text{CuVXCr}_1\text{-XS}_2$  is also known (rhombohedral structure, space group R3m), which belongs to the class of mixed electron-ion semiconductors and is an antiferromagnetic with critical temperatures of superionic ( $T_{\text{su}} = 670$  K) and magnetic ( $T_N \sim 40$  K) transitions [10]. The effect of negative magnetoresistance in this compound is observed at 77 K in a magnetic field of 10 kOe and is – 40 %. This effect is observed only in polycrystalline samples but not in single-crystal samples with a copper deficiency.

The drawback of disulfides  $\text{CuVXCr}_1\text{-XS}_2$  is not a simple layered structure, the complexity of the crystal growth technology because of the high mobility of copper ions and low temperatures at which negative magnetoresistance arises. NaCl-type fcc lattice

In the initial manganese monosulfide  $\alpha\text{-MnS}$  (an antiferromagnetic with NaCl-type fcc lattice), anisotropy of the electrical resistivity for two crystallographic directions [111] and [100] in the temperature range of 77–300 K [11–13] was found. A negative magnetoresistance was also found, the value of which in a field of 10 kOe is –12 % and most clearly showed in the (111) plane [14]. With an increase of the magnetic field, the magnitude of the magnetoresistance does not change, but the minimum shifts to the low temperature range.

The main drawbacks of manganese monosulfide are the small magnitude of the magneto resistive effect, the energy consumption of the synthesis technology, and the

low operating temperatures of magneto resistive elements on the basis on such materials.

The technically closest to the claimed invention is ferromanganese sulfide  $\text{FeXMn}_1\text{-XS}$  [15–17], containing components in the following ratio, atom%: Fe 12.5–20; Mn 30–37.5 and S-50 and having a simple cubic structure such as NaCl. With an increase in the degree of cationic substitution (X) in the  $\text{FeXMn}_1\text{-XS}$  system, a semiconductor – semimetal transition with  $X_c = 0.4$  and an increase in magnetization are observed, with the Néel temperature increasing from 150 K for X = 0 and to 210 K for X = 0.2. Ferromanganese sulfide has GMR in the temperature range of 50 K – 250 K with the maximum development of the HMS effect ( $\delta_H = -83$  %) at 160 K in the magnetic field  $H = 10$  kOe and  $\delta_H = -450$  % at 50 K in the field  $H = 30$  kOe.

The drawback of the known ferromanganese sulfides  $\text{FeXMn}_1\text{-XS}$  is the poor repeatability of the compounds obtained, the complexity and duration of the synthesis.

The aim of this work is to obtain magnetic compounds of holmium – manganese sulfides with a cubic NaCl lattice type, which have a stable and repeating effect of giant magnetoresistance in a wide temperature range.

**Experimental results and their discussion.** *Obtaining method and radiograph.* Three compositions of  $\text{HoXMn}_1\text{-XS}$  were prepared; they are shown in tab. 1 in atomic%.

$\text{HoXMn}_1\text{-XS}$  crystals were grown by crystallization from a melt of the obtained powder sulfides with a purity of at least 99.9 %, in glassy carbon crucibles and a quartz reactor in an argon atmosphere;  $\text{NH}_4\text{CNS}$  was used as sulfiding reagents. The calculated mixture of oxides in a glassy carbon boat was placed in a quartz tube. After the air was displaced by argon and the decomposition products of ammonium thiocyanate, the furnace was turned on from a separate reactor. The synthesis was carried out in two stages: heating the mixture to 500 °C with keeping at this temperature for 1 hour; after grinding – repeated sulfiding for 3 hours at 7500–8000 °C. For completeness of sulfiding and homogenization of the resulting powder sulfide, annealing was carried out for 30 hours in a sulfiding atmosphere at 800 °C with repeated grinding of sulfides. The completeness of sulfidation was controlled by X-ray phase analysis and weight control.

For crystallization from sulphide melt, high-frequency heating of a graphite crucible 10 mm in diameter filled with 6–7 g of sulphide powder was used. A quartz reactor with a crucible was pulled at a speed of 0.5 to 1 cm / h through a single-turn inductor. The inert atmosphere in the reactor was maintained with argon. To obtain a sulphide melt, the necessary parameters of the power supplied to the inductor were determined experimentally. As a result of the synthesis, substances were obtained in the

form of dense ingots. The obtained samples were homogeneous in composition and were used for physical measurements.

According to the results of X-ray diffraction analysis, the holmium-manganese sulfides  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  have a NaCl-type fcc lattice [18]. With an increase of the degree of cationic substitution, the unit cell parameter  $a$  increases (fig. 1). No accompanying impurity phases were found in the synthesized samples.

*Magnetization and magnetoresistance.* The replacement of manganese with holmium ions leads to a significant change of the magnetic properties of the samples of the holmium-manganese system  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$ . A sharp decrease in the paramagnetic Curie temperature as a result of the competition of exchange interactions and a decrease in the effective magnetic moment occurs. The microscopic mechanism of a decrease of exchange and magnetic moment is associated with a change in the electronic structure of manganese ions interacting with holmium ions. Ferromagnetic exchange interaction between man-

ganese and holmium ions leads to an increase in the magnetic susceptibility with decrease of the temperature.

The competition of exchange interactions results in two possible variants: the disappearance of long-range magnetic order with the formation of a spin glass state [19; 20], or preservation of magnetic order in one of the spin components and with frozen transverse spin components (asperromagnetic state) [21; 22]. To determine the nature of this state, we are to measure the magnetic moment at several frequencies.

In the spin glass state, the time during which thermodynamic equilibrium is set depends on the temperature; the magnetic characteristics depend on the measurement frequency. The magnetic moment in an alternating magnetic field, shown in fig. 2, below  $T = 30$  K depends on the frequency. Thus, the relative change of  $\text{Re}(M(\omega = 10 \text{ kHz}) - M(\omega = 100 \text{ Hz})) / \text{Re}(M(\omega = 100 \text{ Hz}))$  increases with the holmium concentration growth and  $\text{Re}(M(T))$  reveals its maximum at  $T = 5$  K, which is missing in HoS.

Table 1

Composition	Ho, %	Mn, %	S, %
I	10	40	50
II	17.5	32.5	50
III	20	30	50

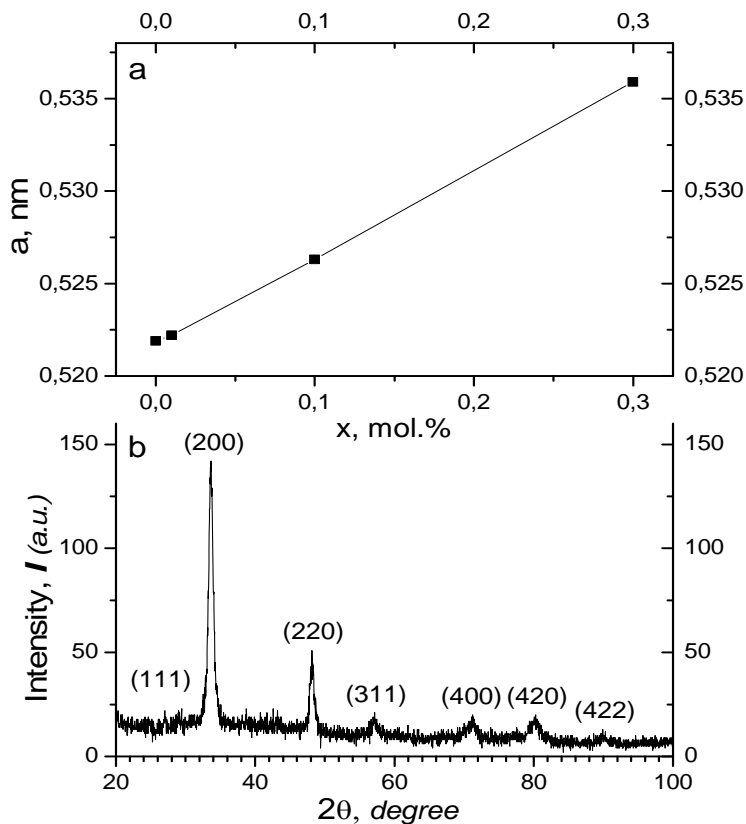


Fig. 1. The lattice constant of holmium-manganese sulfide  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  versus concentration and x-ray for  $x = 0.3$

Рис. 1. Постоянная решетки гольмий-марганцевого сульфида  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  от концентрации и рентгенограмма для  $x = 0,3$

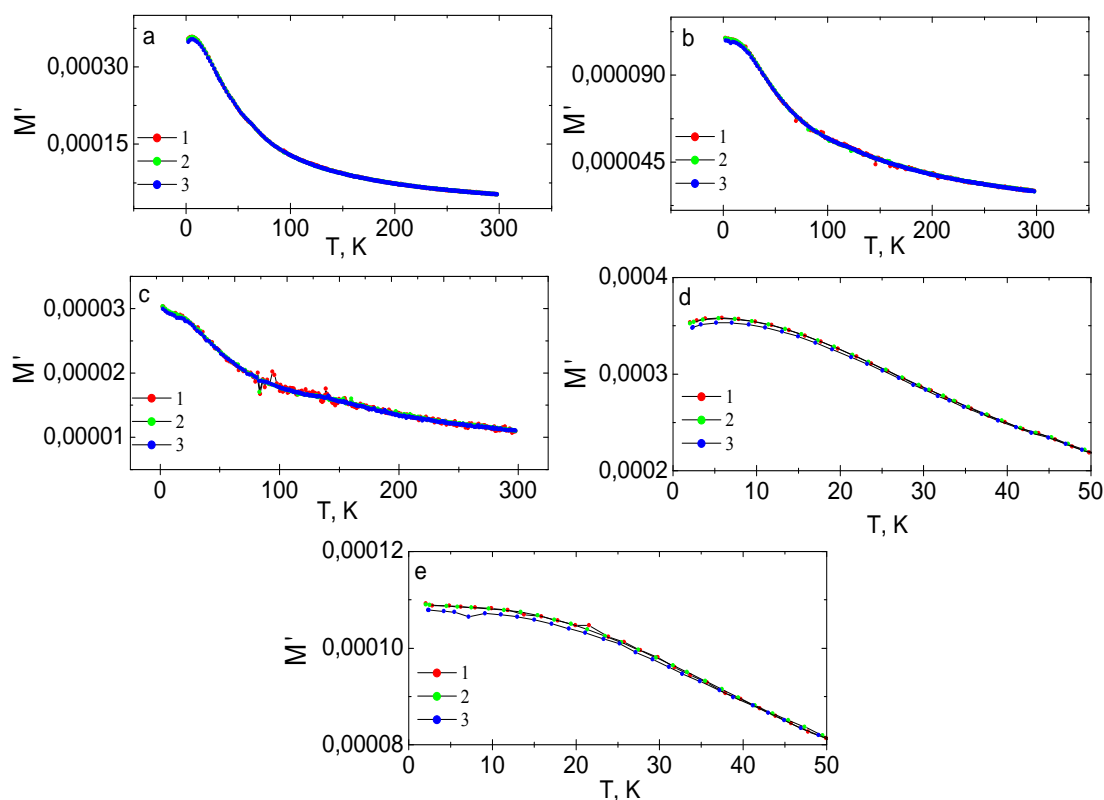


Fig. 2. The Real part of the magnetic permeability (a, b, c) and the imaginary part (d, e) of the temperature for  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  with  $x = 0.3$  (a, d), 0.1 (b, e), 0.05 (c) at three frequencies  $\omega = 1$  kHz (1), 10 kHz (2), 100 kHz (3)

Рис. 2. Реальная часть магнитной проницаемости (a, b, c) и мнимая часть (d, e) от температуры для  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  с  $x = 0,3$  (a, d), 0,1 (b, e), 0,05 (c) на трех частотах  $\omega = 1$  kHz (1), 10 kHz (2), 100 kHz (3)

The imaginary part is practically independent from temperature and has the value  $\text{Im}(M(T)) \sim 10^{-7}$  for concentrations  $x \leq 0.1$  and  $\text{Im}(M(T))$  is qualitatively different in the low-temperature range (fig. 2, d) for a composition with  $x = 0.3$ . The quantity  $\text{Im}(M(T))$ , which characterizes the decay of the magnetic moment, increases with temperature decreasing, and the derivative  $d\text{Im}(M)/dT$  passes through a maximum at  $T = 39$  K at a frequency of  $\omega = 1$  kHz, at  $T = 44$  K for  $\omega = 10$  kHz. An increase in the temperature of the maximum of the derivative of the imaginary part of the magnetic moment with frequency increasing is also connected with the formation of metastable states and is a characteristic feature of spin-glass behavior. It is possible that the spin relaxation mechanism is connected with the exchange interaction of localized and delocalized electrons, and the energy from the spin system is converted into the kinetic energy of current carriers [23; 24].

Replacement of manganese with a rare earth element in  $\text{ReXMn}_1\text{-XS}$  ( $\text{Re} = \text{Gd}, \text{Sm}, \text{Ho}$ ) compounds [25; 26] will result in a shift of the f-level. Several variants are possible in this case, the f-level falls into the conduction band, and the electron passes from the Re ion not to the d-level of the rare-earth ion, but to the conduction band, remaining connected with the donor. If the concentration of such centers is low, less than the critical concentration for impurity band or the percolation threshold formation, then the substance will remain a semiconductor. If the

f-level is located in the forbidden band near the chemical potential, extremum may appear in the temperature dependence of the resistance when the chemical potential is shifted with temperature increase [27; 28].

Substitution of manganese by a trivalent ion will cause electron doping and may induce orbital ordering [29–31], which will result in the splitting of the electron density of states. Depending on the location of the Fermi level relative to the split density of states, the electrical resistivity can change several times [32; 33].

In fig. 3, 4 the temperature dependences of the electrical resistance measured without and in a magnetic field are shown. The magnetoresistance is determined by the formula,

$$\delta_H^{\text{ex}} = \frac{\rho(H \neq 0) - \rho(H = 0)}{\rho(H \neq 0)} \cdot 100\%,$$

where  $\rho(H = 0)$  is the electrical resistance in a zero magnetic field,  $\rho(H \neq 0)$  is the electrical resistance in a given magnetic field.

The magnetoresistance for compositions with  $x = 0.05$  and  $x = 0.1$  indicates that in the synthesized substances in the temperature range 100 K – 300 K, the effect of giant negative magnetoresistance is observed with the maximum effect of GMR in a magnetic field  $H = 8$  kOe; the value of  $\delta_H, \%$  is  $-100$  and  $-80$  %, respectively.

Tab. 2 shows the main physical characteristics of the  $\text{HoXMn}_1\text{-XS}$  holmium-manganese sulfide system.

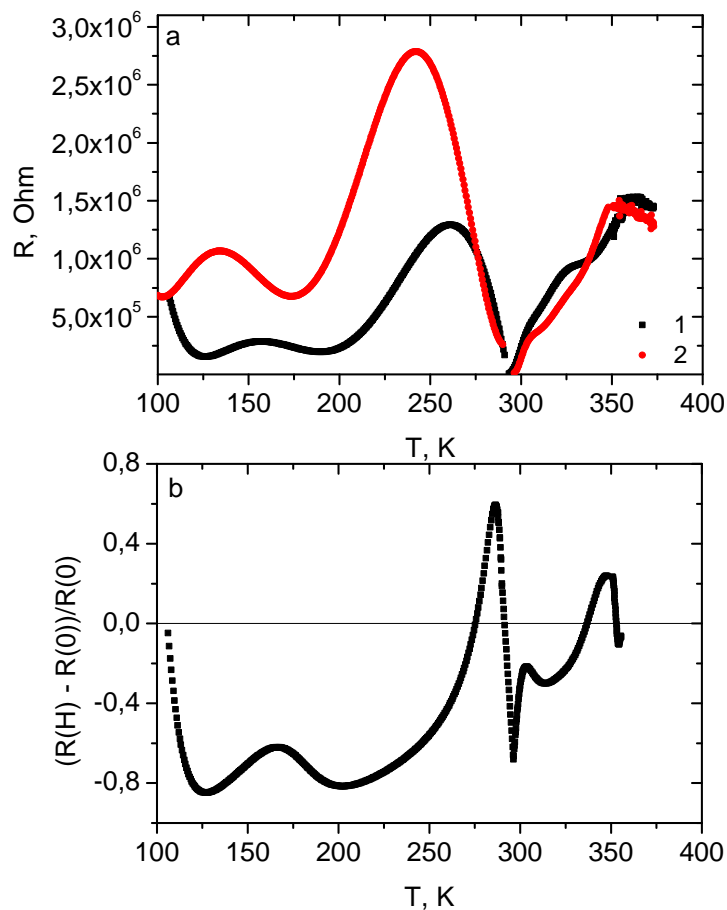


Fig. 3. Resistance  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  with  $x = 0.1$  measured without a field (2) and in a magnetic field  $H = 8 \text{ kOe}$  (1) (a). Magnetoresistance at temperature (b)

Рис. 3. Сопротивление  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  с  $x = 0,1$ , измеренное без поля (2) и в магнитном поле  $H = 8 \text{ kOe}$  (1) (a). Магнитосопротивление от температуры (b)

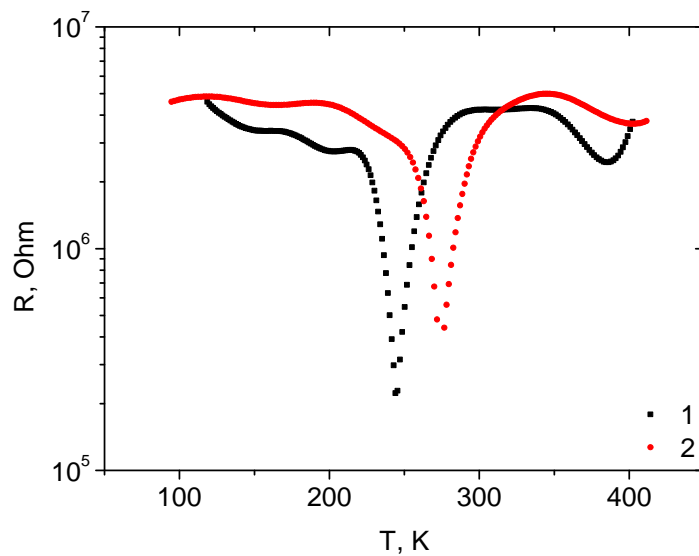


Fig. 4. Resistance  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  with  $x = 0.05$  measured without a field and in a magnetic field  $H = 8 \text{ kOe}$

Рис. 4. Сопротивление  $\text{Ho}_x\text{Mn}_{1-x}\text{S}$  с  $x = 0,05$ , измеренное без поля и в магнитном поле  $H = 8 \text{ kOe}$

Table 2

$\text{Ho}_x\text{Mn}_{1-x}\text{S}$	$a$ , nm	$-\Theta$ , K	$T_N$ , K	$\delta_H$ , % ( $H = 8$ kOe)	$\rho$ , Ом·см $T = 300$ K $H = 0$
I ( $X = 0.05$ )	0.5242	140	145	100	$2 \cdot 10^7$
II ( $X = 0.1$ )	0.5256	100	134	80	$3 \cdot 10^5$
III ( $X = 0.2$ )	0.5303	60	112	30	$10^4$

Where  $a$ , nm is the crystal lattice parameter;  $\Theta$ , K – paramagnetic Curie temperature;  $E_a$ , eV – activation energy;  $T_N$ , K is the Neel temperature;  $\delta_H$ , % – magnetoresistance;  $\rho$ , Ohm cm – resistivity at 300 K.

**Conclusion.** The replacement of manganese with holmium leads to a sharp decrease in the paramagnetic Curie temperature as a result of the competition of exchange interactions in the solid solution and to a reduction of the effective magnetic moment in the concentration range  $0 < X < 0.1$ . The microscopic mechanism of exchange and magnetic moment decrease is connected with the change of the electronic structure of manganese ions interacting with holmium ions. Ferromagnetic exchange interaction between manganese and holmium ions causes an increase of the magnetic susceptibility with decreasing temperature in the magnetically ordered phase. The frequency dependence of the magnetic permeability at low temperatures and the maximum of the imaginary part of the magnetic permeability for  $X = 0.3$ , which is connected with freezing of the transverse spin components, is found.

In solid solutions  $\text{HoXMn}_{1-X}\text{S}$ , a sharp maximum in the conductivity with respect to temperature is found, which shifts towards low temperatures in a magnetic field. A change in the sign of the magnetoresistance is reviled with an increase in temperature below and above room temperature. The results obtained can be used as perspective materials for spintronic, as components of sensor technology, magnetic memory based on the GMR effect for a wide range of temperatures and magnetic fields.

**Acknowledgments.** The work was supported by the Siberian State University youth grant, SibGU, 2020.

**Благодарности.** Работа выполнена при поддержке молодежного гранта СибГУ им. М. Ф. Решетнева, 2020.

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