
PHASE
TRANSITIONS

Correlation of the Magnetic and Transport Properties with Polymorphic Transitions in Bismuth Pyrostannate $\text{Bi}_2(\text{Sn}_{1-x}\text{Cr}_x)_2\text{O}_7$

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Abstract—The correlation of the electrical, magnetic, and structural properties of $\text{Bi}_2(\text{Sn}_{1-x}\text{Cr}_x)_2\text{O}_7$ solid solutions has been investigated for two compositions with $x = 0.05$ and 0.1 . The temperatures of structural phase transitions and anomalies of the electrical resistance in the vicinity of these transitions at temperatures in the range $300 \text{ K} < T < 1000 \text{ K}$ have been determined using differential scanning calorimetry. It has been found that the thermoelectric power changes sign upon the polymorphic transition. The paramagnetic Curie temperature and the chromium-ion spin have been found from the temperature dependence of the magnetic susceptibility in the range $4 \text{ K} < T < 1000 \text{ K}$ and from the field dependence of the magnetic moment in magnetic fields of up to 6 T.

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1. INTRODUCTION

The search for new catalysts and gas sensors based on inorganic semiconductor materials is an important problem [1, 2]. On the catalyst surface, apart from the adsorption predominantly caused by physical interactions (van der Waals forces), there occurs a chemical reaction, chemisorption, associated with the overlap of the wave functions of the gas molecules and ions of the crystal lattice of the catalyst surface. The chemisorption is accompanied by an increase in the concentration of electrons with a high enough energy to overcome the barrier created by the negatively charged surface, which leads to an increase in the coverage of the surface with chemisorbed oxygen, as well as to a change in the surface charge and, hence, in the reactivity of the semiconductor oxide upon interaction with reductant gases (electron donors) and oxidant gases (electron acceptors). This determines the sensor signal in the detection of different gases. Thus, the control of the electronic structure of a semiconductor sensor material makes it possible to stimulate the interaction of this material with the gas to be determined and provides additional opportunities for

increasing the selectivity of semiconductor gas sensors.

The highest sensitivity to changes in the concentration of adsorbed molecules and, hence, to variations in the composition of the gas phase is observed in materials with predominantly ionic bonds (for example, ZnO and SnO_2). However, the oxides characterized by an extremely high electrical resistance (SiO_2 , Al_2O_3 , etc.) in the pure form are not used in gas sensors of the resistive type because of the difficulties encountered in performing conductivity measurements [3]. Titanium dioxide is used as a sensitive element in sensors and multisensors for determining the concentration of harmful and dangerous substances, such as CH_4 , CO, and NO_2 . The operating temperatures of such sensors lie in the range from 750 to 900°C. Sensitive elements of sensors and multisensors based on the Bi_2O_3 – SnO_2 system [4, 5] are used to control the concentration of carbon monoxide, ammonia, and hydrogen sulfide at temperatures of the heater in the range from 100 to 400°C.

The ferroelectric order is revealed in compounds with the orthorhombic lattice of the pyrochlore struc-

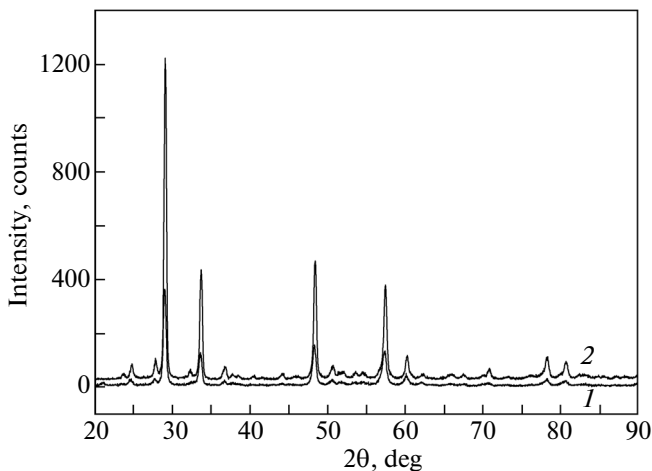


Fig. 1. X-ray diffraction patterns of $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$: (1) before high-temperature measurements and (2) after high-temperature measurements.

ture; for example, in $\text{Gd}_2\text{Nb}_2\text{O}_7$, the critical temperature of ferroelectric ordering is equal to 198 K [6]. In bismuth pyrostannate, there is no center of symmetry, and the substitution of chromium ions for tin ions can induce local polarization due to the redistribution of charges between the bismuth and chromium ions. Chromium is a magnetically active ion, and, by varying the magnetoelastic interaction, it is possible to change the polarization and the magnitude of the exchange interaction.

It was found that, in bismuth pyrostannate, there exist three crystallographic phases [4, 7]: tetragonal α - $\text{Bi}_2\text{Sn}_2\text{O}_7$ ($a = 21.328 \text{ \AA}$, $c = 21.545 \text{ \AA}$), which exists to 90°C ; cubic β - $\text{Bi}_2\text{Sn}_2\text{O}_7$ ($a = 21.4 \text{ \AA}$), which exists in the range between 90 and 680°C ; and face-centered cubic γ - $\text{Bi}_2\text{Sn}_2\text{O}_7$ ($a = 10.73 \text{ \AA}$), which exists above 680°C . In [8], Raman spectroscopy was used to investigate the temperature dependence of the intensity of phonon modes, which revealed the structural transition $\alpha \rightarrow \beta$ at a temperature of approximately 127°C . The IR spectrum of $\text{Bi}_2\text{Sn}_2\text{O}_7$ [9] differs significantly from typical spectra of stannates with a cubic structure of the pyrochlore type. In $\text{Bi}_2\text{Sn}_2\text{O}_7$, two polymorphic (orthorhombic and cubic) modifications can exist simultaneously [10].

The theoretical calculations of the electronic structure by the electron density functional method [7] for stannates with a cubic lattice revealed a gap of approximately 2 eV in the spectrum of electronic excitations; i.e., stannates are semiconductors and should possess semiconductor conductivity. The semiconductor character of the bulk conductivity of metal oxides is determined to a large extent by the impurity conductivity, which can be purposefully changed by substitution in the cation sublattice. Thus, the substitution of 3d metal ions for tin ions in bismuth pyrostannate $\text{Bi}_2\text{Sn}_2\text{O}_7$ will lead to a change in the magnetic and

electrical properties and will make it possible establish a correlation of the structural phase transitions with the change in the electronic structure.

The purpose of this work was to determine the correlation of the magnetic and transport properties in bismuth pyrostannate $\text{Bi}_2\text{Sn}_2\text{O}_7$ with the structural phase transitions upon cation substitution of chromium ions for tin ions.

2. SYNTHESIS AND CHARACTERIZATION OF SAMPLES

The synthesis of $\text{Bi}_2(\text{Sn}_{1-x}\text{Cr}_x)_2\text{O}_7$ ($x = 0.05, 0.1$) was performed by the solid-phase reaction with repeated careful and prolonged intermediate grindings in alcohol. The initial substances were Bi_2O_3 , SnO_2 , and Cr_2O_3 oxides (special-purity grade). The initial oxide mixture pressed into pellets was placed in a furnace and heated at temperatures in the range from 700 to 950°C . The exposure time was varied from 8 to 24 h. The X-ray diffraction analysis of the synthesized samples showed that the compound does not contain impurities and has two polymorphic (tetragonal and orthorhombic) modifications, which exist simultaneously; moreover, the percentage of the tetragonal phase is higher than that of the orthorhombic phase, as in the $\text{Bi}_2\text{Sn}_2\text{O}_7$ compound [10]. The X-ray diffraction pattern of $\text{Bi}_2(\text{Sn}_{1-x}\text{Cr}_x)_2\text{O}_7$ with $x = 0.1$ is shown in Fig. 1. The X-ray diffraction analysis of $\text{Bi}_2(\text{Sn}_{1-x}\text{Cr}_x)_2\text{O}_7$ was performed both before the measurements with heating of the sample to $T = 1000 \text{ K}$ and after the measurements. No changes in the positions of the reflections were observed.

The structural transitions occurring in the compounds are accompanied by the release or absorption of heat, as can be determined from calorimetric measurements using differential scanning calorimetry. The complex thermal analysis of $\text{Bi}_2\text{Sn}_2\text{O}_7$ and $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$ samples was performed on a NETZSCH STA 449 C Jupiter thermal analysis system combined with a NETZSCH QMS 403 C Aeolos quadrupole mass spectrometer for the analysis of gases released upon heating of the samples. This technique combines the simultaneous detection of changes in the mass (thermogravimetry), and heat flux (differential scanning calorimetry (DSC)). The investigations were carried out in platinum–rhodium crucibles with caps having a hole at temperatures ranging from 310 to 1073 K during heating and from 1073 to 523 K during cooling in a dynamic argon atmosphere at a flow rate of 40 mL/min. The heating and cooling rates were 10 K/min. The DSC curves for $\text{Bi}_2\text{Sn}_2\text{O}_7$ (Fig. 2a) and $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$ (Fig. 2b) exhibit several endothermic peaks during heating of the samples.

The performed mass spectrometric analysis of the gases released during heating of $\text{Bi}_2\text{Sn}_2\text{O}_7$ and $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$ demonstrated that, when scanning

over the mass numbers of oxygen-containing ions (16, 17, 18, 32), no change in the signal intensity was detected.

The DSC curve measured during heating of the $\text{Bi}_2\text{Sn}_2\text{O}_7$ sample (curve 1 in Fig. 2a) contains three endothermic effect at temperatures of 370, 548, and 872 K. In the subsequent cooling of $\text{Bi}_2\text{Sn}_2\text{O}_7$ (curve 2 in Fig. 2a), the DSC curve exhibits only one peak at a temperature of 865.4 K. The presence of peaks in the DSC curve indicates the occurrence of phase transitions. It is known that bismuth stannate $\text{Bi}_2\text{Sn}_2\text{O}_7$ [4, 11] undergoes a series of polymorphic transitions: α - $\text{Bi}_2\text{Sn}_2\text{O}_7$ is a thermodynamically stable phase at room temperature; β - $\text{Bi}_2\text{Sn}_2\text{O}_7$ exists in the temperature range of 130–630°C; and above 630°C, the face-centered cubic phase γ - $\text{Bi}_2\text{Sn}_2\text{O}_7$ dominates. According to the X-ray diffraction data, our sample at room temperature already exists in two crystallographic polymorphic (cubic and orthorhombic) modifications. Therefore, the phase transition $\alpha \rightarrow \beta$ at approximately 370 K is smeared in temperature. The next effect, which is accompanied by the absorption of heat at 548 K, suggests another phase transition, in which the orthorhombic phase completely transforms into the cubic phase. In the vicinity of this temperature, anomalies were revealed in the temperature dependences of the imaginary part of the permittivity and the electrical resistance of $\text{Bi}_2\text{Sn}_2\text{O}_7$ [10]. The third distinct endothermic peak at $T = 872$ K is reversible. In the DSC curve (curve 2 in Fig. 2a) measured during cooling of the sample, the corresponding exothermic peak is observed at $T = 865$ K. The heat released in this case $Q = 0.48$ J/g is close in magnitude to the heat absorbed during heating $Q = -0.54$ J/g. The close values of the temperatures and heats of transformation of the third effect allow us to interpret it as a reversible phase transition in which the $\text{Bi}_2\text{Sn}_2\text{O}_7$ cubic phase exists.

The DSC curve measured during heating of the $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$ sample is presented in Fig. 2b. It can be seen from this figure that the DSC curve has several endothermic peaks at temperatures of 367, 473, 553, and 646 K, which correlate with anomalies in the temperature dependence of the electrical resistivity. The close values of the temperatures of the peaks $T = 367$ and 553 K for $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$ and $T = 370$ and 548 K for $\text{Bi}_2\text{Sn}_2\text{O}_7$ suggest that they have the same nature: the structural transition from the α phase to the β phase and from the orthorhombic phase to the cubic phase. The presence of two endothermic peaks in the DSC curve is possibly associated with local deformations of the orthorhombic and cubic structures, which are not correlated with each other and are not revealed by the X-ray diffraction analysis; however, they can manifest themselves in the transport properties. The reverse DSC curve (cooling) exhibits poorly pronounced exothermic effects at temperatures of 924 and 977 K.

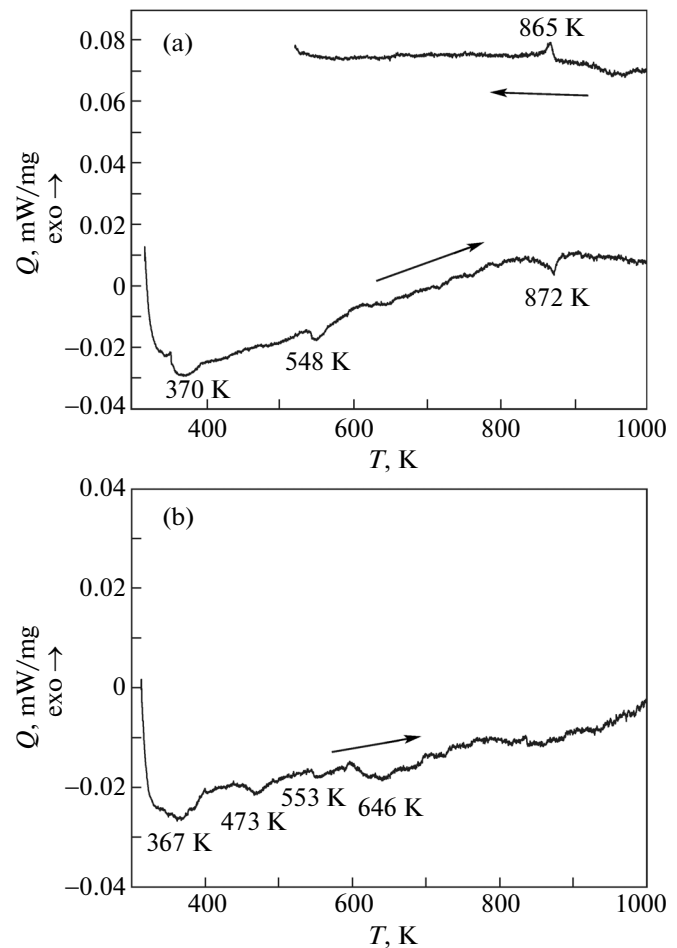


Fig. 2. DSC data: (a) the DSC curve for $\text{Bi}_2\text{Sn}_2\text{O}_7$ and (b) the DSC curve for $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$.

3. ELECTRICAL AND MAGNETIC PROPERTIES

The electrical properties of $\text{Bi}_2(\text{Sn}_{1-x}\text{Cr}_x)_2\text{O}_7$ ($x = 0.05, 0.1$) were investigated using the four-probe method on an Agilent Technologies 34410A multimeter in the temperature range of 300–900 K. Figure 3 shows the temperature dependences of the electrical resistance for two compositions. For $x = 0.05$, there are oscillations of the electrical resistance in the vicinity of the structural transitions. The highest maximum in the temperature dependence of the electrical resistance is observed upon the structural transition from the α phase to the β phase and also persists for the composition with $x = 0.1$. The temperatures of the transitions observed in the DSC curves (Fig. 2b), i.e., $T = 473$ and 553 K, coincide with the temperatures of minima in the temperature dependence of the electrical resistance. The symmetry of the crystal structure determines the electronic structure of a semiconductor, the band gap, and the positions of the impurity levels in it. In the case when there are several phases, the electron density of states has maxima and minima.

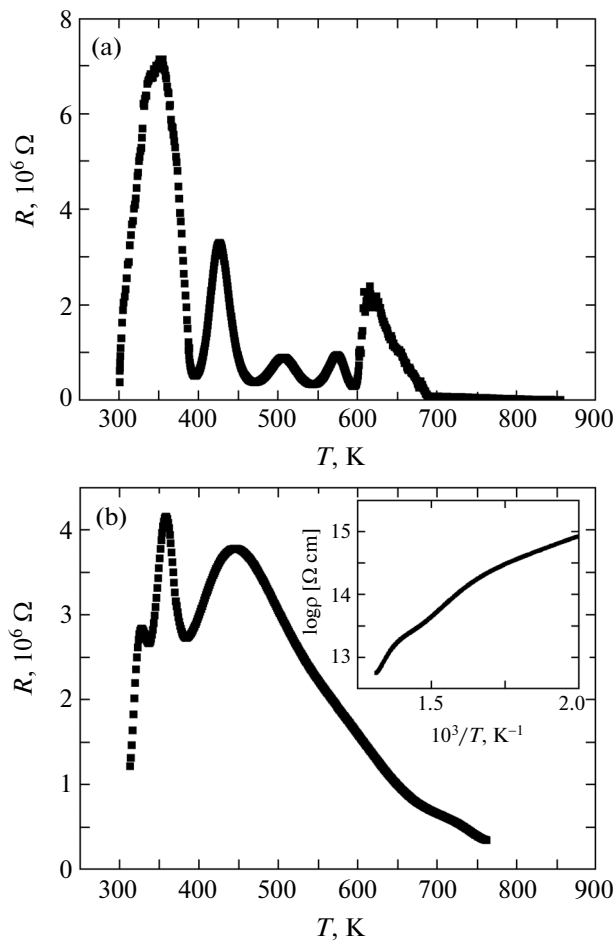


Fig. 3. Temperature dependences of the electrical resistance: (a) $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$ and (b) $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$. The inset shows the temperature dependence of the electrical resistivity $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$.

A change in the crystal structure leads to a shift in the mobility edge by several times and to a change in the density of states of charge carriers, the charge carrier mobility, and the electrical resistance. In the $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$ solid solution, the substitution of chromium for tin causes a shift of the boundaries of the phase transition from the orthorhombic to cubic state toward the low-temperature range up to $T = 445$ K. In the high-temperature range, the dependences have two portions in the regions of the temperatures $T = 570$ and 670 – 720 K, which are characterized by a change in the activation energy. The activation energies calculated from the linear dependence $\log\rho(10^3/T)$ on the inverse temperature for $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$ are as follows: $\Delta E_1 = 0.6$ eV in the temperature range of 450 – 570 K and $\Delta E_2 = 0.8$ eV in the temperature range of 570 – 650 K, which is higher than the activation energy $\Delta E = 0.23$ eV for $\text{Bi}_2\text{Sn}_2\text{O}_7$ [10].

For bismuth stannate $\text{Bi}_2\text{Sn}_2\text{O}_7$ with a cubic structure, we calculated the electron density of states with the energy gap of 2 eV. Electrons of the Bi 6s and O 2p shells are predominantly located in the upper valence band, whereas electrons of the Sn 5s, O 2p, and Bi 6p shells occupy the bottom of the conduction band. Different contributions from electrons of the Sn 5s and Bi 6s states of the valence band and the conduction band manifest themselves in both the atomic binding energy and the strength of the interaction of metal cations with oxygen anions [12]. Chromium ions predominantly occupy octahedral sites [13–15]. Therefore, we can assume that, in our sample, Cr^{3+} ions substitute for tin ions. Chromium ions in the trivalent state were observed in LiNdO_3 crystals [16]. When embedding in the niobium octahedron, the Cr^{3+} ion causes its deformation, which can occur in two ways [17]: (i) with a change in the positions of the oxygen ions near the immobile Cr^{3+} ion and (ii) as a result of the displacement of the Cr^{3+} ion from the Nb^{5+} site toward the center of the octahedron. In [16], the experimental and theoretical data confirmed the deformation of the niobium octahedron due to the embedding of a trivalent chromium ion in it.

The stable trivalent state of chromium ions leads to a change in the valence of bismuth according to the scheme $\text{Bi}^{3+} + 2\text{Sn}^{4+} \rightarrow \text{Bi}^{5+} + 2\text{Cr}^{3+}$. This is favored by the configuration of the pyrochlore lattice, because one bismuth ion has two nearest neighbor tin ions. As a result, the formation of an impurity subband of chromium ions is possible near the bottom of the conduction band.

The position of the impurity subband with respect to the valence band and the type of charge carriers can be determined from the thermoelectric power measured by the standard dc four-probe compensation method in the temperature range of 80 – 1000 K. Figure 4 shows the temperature dependence of the Seebeck coefficient α for the $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$ sample in the range of 400 – 1000 K. At the temperature of the transition from the orthorhombic phase to the cubic phase $T_{\text{OC}} = 450$ K, we observe a minimum in the dependence $\alpha(T)$ and a change in the sign of the thermoelectric power from negative to positive in the temperature range of 550 – 680 K, where the activation energy increases and the type of charge carriers becomes electronic.

The magnetic properties of $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$ were investigated on a PPMS-9 magnetometer at temperatures up to 300 K and in magnetic fields -6 T $< H < 6$ T. The magnetic properties of $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$ were also investigated on a high-temperature setup using the Faraday method at temperatures up to 1400 K and in magnetic fields up to 0.86 T. From the temperature dependence of the inverse magnetic susceptibility (inset in Fig. 5a), which is described by the Curie–Weiss law in the range 0 K $< T < 40$ K, we deter-

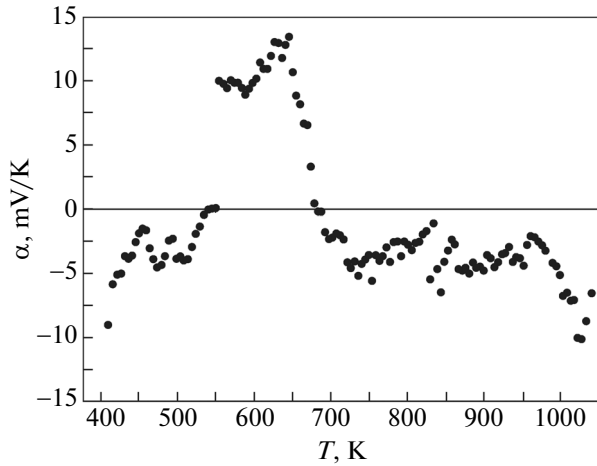


Fig. 4. Temperature dependence of the Seebeck coefficient α for $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$.

mined the paramagnetic Curie temperature $\Theta \approx 2$ K. The field dependence of the magnetic moment in the field range $-6 \text{ T} < H < 6 \text{ T}$ at $T = 4.2 \text{ K}$ (Fig. 6) has a nonlinear character. In the case when this dependence is described by the expression $M(H) = N_A \mu_B S B_S(g S \mu_B H / k_B T)$, where the $B_S(x)$ is the Brillouin function, the best agreement with the experimental data is achieved for the chromium-ion spin $S = 3/2$. Here, μ_B is the Bohr magneton, N_A is the Avogadro's number, k_B is the Boltzmann constant, and $g = 2$ is the g -factor. The magnetic measurements confirmed that the chromium ions are trivalent. The difference between the theoretical and experimental results is associated with the presence of a uniaxial anisotropy induced by the distortion of the octahedron due to the displacement of oxygen ions between the tin and chromium ions. An increase in the concentration of substitutional ions leads to an increase in the magnetic moment in a magnetic field (Fig. 5b). The slope of the temperature dependence of the inverse magnetic susceptibility in $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$ is changed (inset in Fig. 5b) upon the transition from the α phase to the β phase at $T = 370 \text{ K}$. The paramagnetic Curie temperature increases by a factor of 3. For example, $\Theta_T = 50 \text{ K}$ for the tetragonal phase in the range $150 \text{ K} < T < 300 \text{ K}$ and $\Theta_C = 150 \text{ K}$ for the cubic phase at $T > 400 \text{ K}$. In the tetragonal phase, the lattice constant changes by $(a - c)/a = 0.01$. Therefore, we can ignore the difference between the exchange interactions in the plane perpendicular to the tetragonal axis, as well as along the tetragonal axis, and estimate changes in the exchange parameters $J_T/J_C = \Theta_T/\Theta_C = 1/3$. In the cubic phase, the ferromagnetic exchange increases by a factor of 3, whereas the electrical resistance sharply decreases. Possibly, there is an additional mechanism of the exchange interaction through charge carriers, for example, the kinetic exchange.

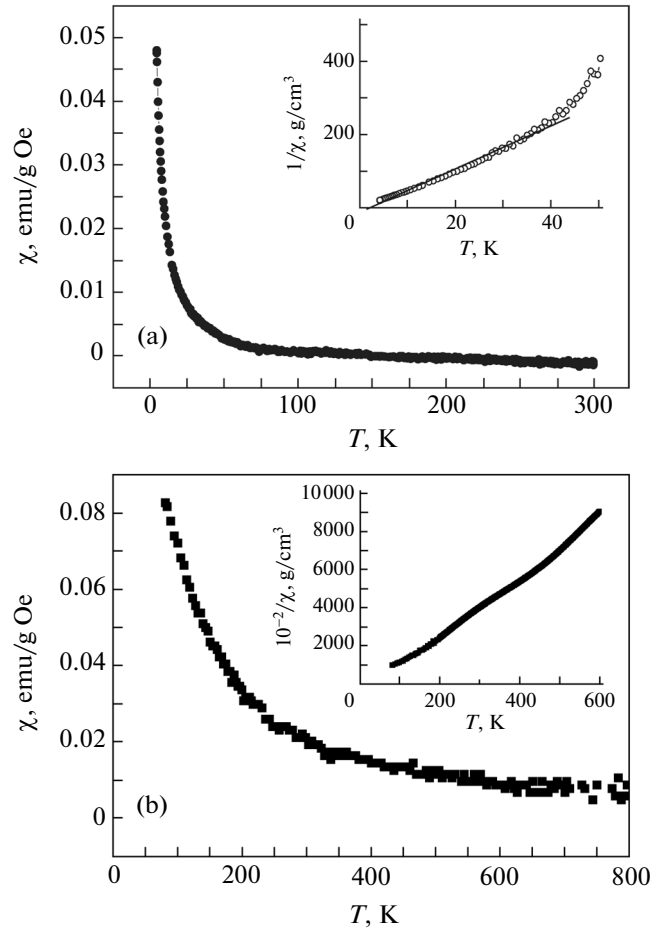


Fig. 5. Temperature dependences of the magnetic susceptibility: (a) $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$ and (b) $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$. The insets show the temperature dependences of the inverse magnetic susceptibility for the same compounds.

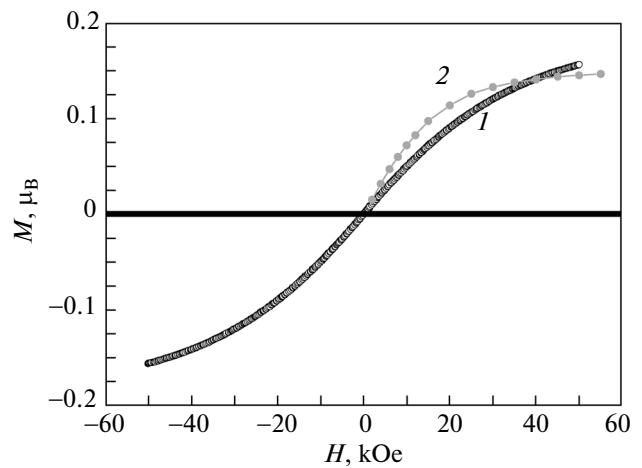


Fig. 6. Field dependence of the magnetization of $\text{Bi}_2(\text{Sn}_{0.95}\text{Cr}_{0.05})_2\text{O}_7$ at $T = 4.2 \text{ K}$: (1) experimental data and (2) approximation by the dependence $M/N_A \mu_B = S B_S(g S \mu_B H / k_B T)$, where $B_S(x)$ is the Brillouin function.

4. CONCLUSIONS

In $\text{Bi}_2(\text{Sn}_{1-x}\text{Cr}_x)_2\text{O}_7$ solid solutions with $x = 0.05$ and 0.1 , two new structural transitions, as compared to the bismuth stannate $\text{Bi}_2\text{Sn}_2\text{O}_7$, were revealed using differential scanning calorimetry. The polymorphic transitions and the transition from the orthorhombic to cubic phase are accompanied by the appearance of maxima in the temperature dependence of the electrical resistance due to the transformation of the electronic structure. According to the thermoelectric measurements, the charge carriers are electrons, except for the temperature region of the transition from the orthorhombic to cubic phase. From the field dependences of the magnetic moment, it was found that chromium ions are in the trivalent state with spin $S = 3/2$. The ferromagnetic exchange interaction between the chromium-ion spins is enhanced by a factor of 3 upon the transition from the tetragonal to cubic phase. The enhancement of the exchange correlates with an increase in the electrical conductivity of $\text{Bi}_2(\text{Sn}_{0.9}\text{Cr}_{0.1})_2\text{O}_7$.

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