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ELECTRONIC PROPERTIES OF SOLID

Effect of Cobalt Impurity Ions on the Magnetic and Electrical Properties of Iron Monosilicide Crystals

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Abstract—The results of experimental investigations of $Fe_{1-x}Co_xSi$ crystals in the impurity limit with $x =$ 0.001, 0.005, and 0.01 are reported. The temperature and field dependences of the magnetic susceptibility have been studied. According to the experimental data, the introduction of cobalt impurity leads to a change in the energy structure, which is most pronounced in a change in the electrical properties. The temperature, field, and concentration dependences of the resistivity have been measured. The results have been interpreted in the framework of the Kondo model.

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1. INTRODUCTION

The physical properties of 3*d* metal monosilicide crystals are diverse [1]. It is known that the ε-FeSi phase with the B20 structure in the iron–silicon system exists in a narrow region near the equiatomic composition. Figure 1a shows the unit cell of this FeSi phase, as well as a fragment in the form of the cluster with the central iron atom in the environment of the nearest silicon atoms [2]. Iron and cobalt monosilicides are of special interest among 3*d* metal monosilicides. For the former, it is characteristic that with an increase in the temperature, the resistivity ρ first decreases monotonically by about four orders of magnitude, reaches a minimum at $T \approx 300$ K (in this case, the temperature dependence $\rho(T)$ is not described by a simple thermoactivation dependence), and then increases slowly. According to this attribute, the FeSi crystal belongs either to Kondo compounds [3] or to materials exhibiting a semiconductor–metal transition [4]. The magnetic properties of this material are not described by the traditional scheme of the behavior of the known magnets. The magnetic susceptibility ($\chi \sim 10^{-6}$–10^{-4}) first decreases when the temperature is increased to $T \approx 90$ K and then increases strongly, reaching a maximum at $T \approx 500$ K [5]. Such a behavior is attributed to the formation of iron clusters (owing to nonstoichiometry) and to their superparamagnetic behavior at low temperatures [6, 7], and the high-temperature dependences are explained by the thermally induced transition of electrons in the d^4 configuration from the e_{2g}^4 ($S = 0$) state to the $e_{2g}^3 t_{2g}^1$ ($S = 1$) state [8]. The temperature dependence of the resistivity of the CoSi crystal corresponds to the metallic conduction. The resistivity ρ depends on the stoichiometry of the crystal and its purity, lies in the range of $(1.4–2.8) \times 10^{-4}\ \Omega$ cm at $T = 300$ K [1], and increases slightly with the temperature. According to the magnetic properties, stoichiometric high-quality cobalt monosilicide crystals belong to diamagnetic materials [1, 9]. Deviation from perfection leads to the appearance of weak paramagnetism. However, the addition of only several percent of cobalt to iron monosilicide is accompanied by the appearance of attributes of the ferromagnetic order and metallic conduction. This property is likely the most astonishing property in the formation of the

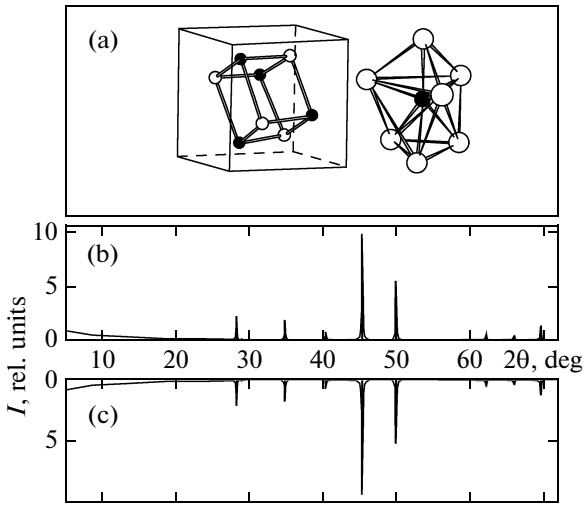

Fig. 1. (a) Unit cell of the FeSi crystal and the fragment of the structure with (●) the central iron atom surrounded by (○) silicon atoms, (b) the diffraction pattern of the $Fe_{1-x}Co_xSi$ crystal ($x = 0.02$), and (c) the diffraction pattern of the nominally pure reference FeSi sample.

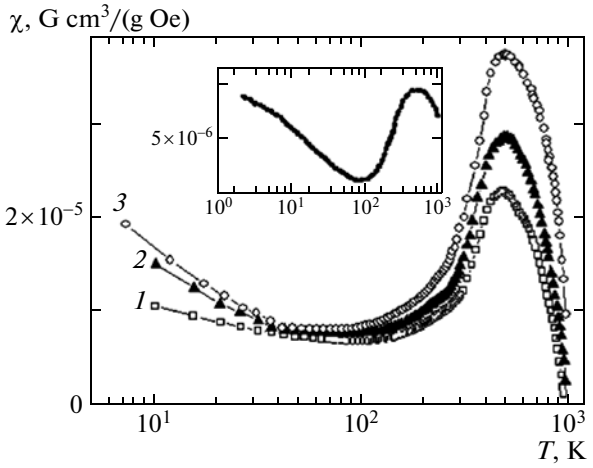

Fig. 2. Temperature dependences of the magnetic susceptibility for $Fe_{1-x}Co_xSi$ crystals with x = (*1*) 0.001, (*2*) 0.005, and (*3*) 0.01. The inset shows the magnetic susceptibility for the nominally pure FeSi sample.

magnetic and electrical properties of mixed $Fe_{1-x}Co_xSi$ crystals. However, the clear understanding of the mechanisms determining the properties of nominally pure iron and cobalt monosilicide crystals, as well as of mixed crystals and, in particular, the causes responsible for the appearance of the magnetic order is absent.

In this work, we attempt to study a change in the magnetic properties and conductivity in $Fe_{1-x}Co_xSi$ mixed crystals in the impurity limit.

2. EXPERIMENTAL PROCEDURE

The synthesis of $Fe_{1-x}Co_xSi$ crystals was described in [7]. The polycrystalline samples were obtained by cooling a melt with a rate of 3 K/h. Preliminarily, the mixture of iron, cobalt, and silicon in the necessary ratio was thoroughly milled, then was heated to the necessary temperature, and was aged at this temperature for 5 h. After cooling, the resulting substance was again milled and the entire procedure was repeated. Three or four cycles were carried out. The cobalt content determined by the X-ray fluorescence method was $x = 0.001$, 0.005, and 0.01. X-ray measurements were performed on several samples taken from various regions of the crucible. For all synthesized compositions, the data of different X-ray measurements coincide. The magnetic measurements were performed at low temperatures ($T = 4.2$–300 K) on a SQUID magnetometer and at high temperatures ($T = 250$–950 K) on a vibration magnetometer. The dependences of the resistivity were obtained by the four-probe method. To more clearly identify the features in the temperature dependences, the temperature axis is given in the logarithmic scale.

According to the X-ray spectra shown in Figs. 1b and 1c, nominally pure crystals and crystals doped with cobalt ions have an identical structure and correspond to the ε-FeSi phase with the B20 structure [10].

When a small amount of impurity cobalt ions was introduced, the qualitative temperature behavior of the magnetic susceptibility remains almost unchanged (see Fig. 2). However, the absolute value of ρ increases with the concentration of cobalt ions. The difference between the susceptibilities of the doped and nominally pure crystals near the absolute minimum of susceptibility at $T \sim 90$ K can be accepted as the reference point. In this case, the observed increases in the susceptibility are different in the low- and high-temperature regions. As is seen in Fig. 2, the high-temperature maximum increases with the concentration of impurities stronger than the susceptibility in the low-temperature region and is noticeably larger than the value for the nominally pure crystal (see the inset in Fig. 2).

The field dependences of the magnetization at low temperatures in low magnetic fields have the form shown in Fig. 3. It is seen that the $M(H)$ dependence for studied concentrations is almost linear, exhibiting weak hysteresis under the reversal of the magnetic field; the width of hysteresis increases with the concentration of impurities. The tangent of the slope angle of the magnetization curves increases with the concentration. At room temperature, the $M(H)$ dependences for concentrations $x = 0.001$ and 0.005 are linear and nonlinearity asymptotically passing to a straight line is observed for $x = 0.01$ in fields $H < 5$ kOe (see Fig. 4). However, hysteresis in this case is almost absent.

The electrical properties of doped crystals also change noticeably. Figure 5 shows the temperature dependences of the resistivity of $Fe_{1-x}Co_xSi$ crystals. It is seen that two sections with different dependences are observed in the low-temperature region. A step feature in the $\rho(T)$ dependence is observed for all crystals. The introduction of cobalt ions leads to a general decrease in the resistivity and to a more pronounced manifestation of this step feature. The magnetic field reduces the resistivity. The inset in Fig. 5 shows the temperature dependences of the magnetic-field-induced addition $\Delta\rho = \rho(H) - \rho(0)$. As the concentration of the impurity increases, the sensitivity to the magnetic field decreases. Figure 6 illustrates the behavior of the resistivity as a function of the external magnetic field at various temperatures. A weak quadratic dependence on the magnetic field is observed at low temperatures; this dependence is weaker for a higher concentration of the impurity. The field dependence becomes weaker with an increase in the temperature and almost disappears at $T \geq 15$ K.

3. DISCUSSION OF THE RESULTS

3.1. Magnetic Properties

First, the data of the magnetic measurements for both polycrystalline and single-crystal samples of

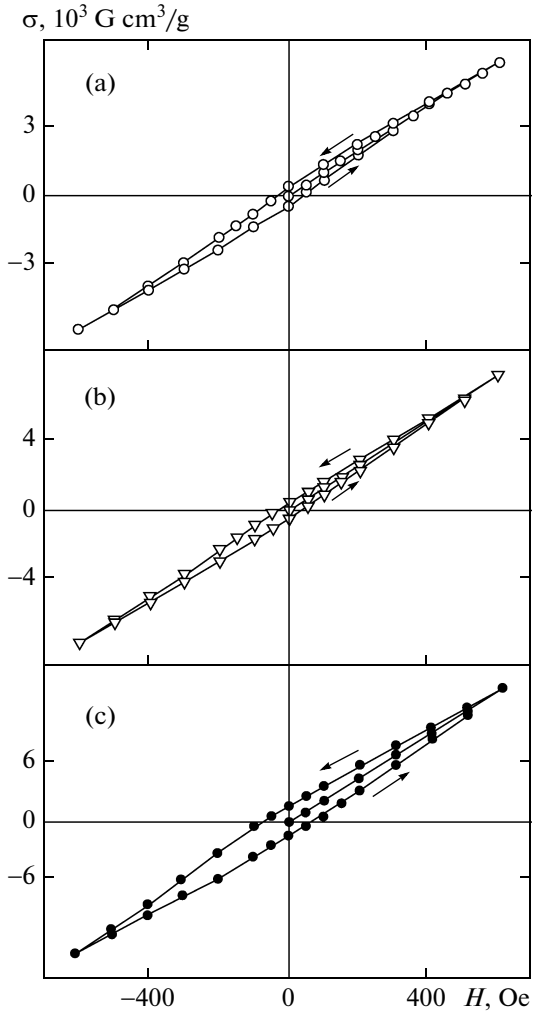

Fig. 3. Field dependences of the magnetization of $Fe_{1-x}Co_xSi$ crystals with x = (a) 0.001, (b) 0.005, and (c) 0.01 at $T = 4.2$ K.

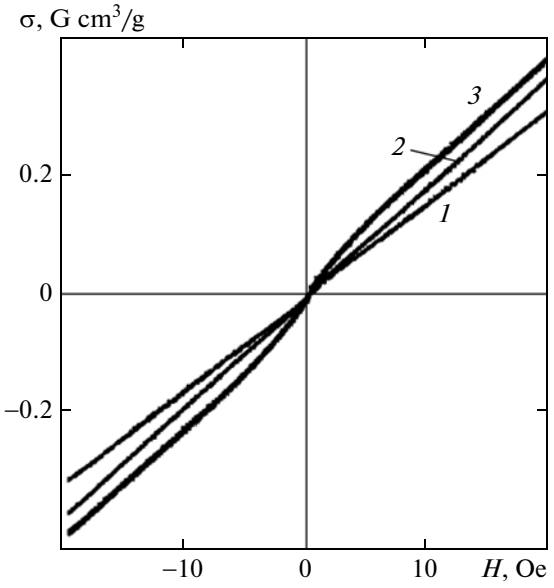

Fig. 4. Field dependences of the magnetization of $Fe_{1-x}Co_xSi$ crystals with x = (*1*) 0.001, (*2*) 0.005, and (*3*) 0.01 at $T = 300$ K.

nominally pure FeSi crystals of different series have a spread within 5% [7]. This circumstance was one of the reasons in favor of the impurity mechanism for the nonstoichiometry-induced low-temperature behavior of the magnetization of FeSi crystals [6, 7]. The same approach was applied when considering the physical properties of iron monosilicide crystals doped with dysprosium ions [11]. When determining the number n of atoms in a cluster, we used the results reported in [12], where the magnetic properties of iron clusters were calculated as functions of the number of particles. According to the calculation, the magnetic moment per atom increases with the size of the cluster and the magnetic moment per iron atom for clusters containing 15 or more atoms is about $m_{Fe} = 3\mu_B$, as in bulk iron. The crystal structure of the Co-ion-doped FeSi crystal remains unchanged until the complete replacement of iron by cobalt. At small concentrations of the impurity, the cobalt ions can occupy positions of iron or, owing to nonstoichiometry, positions that should be occupied by silicon. The earlier NMR investigation [13] indicates that the cobalt ion in $Fe_{1-x}Co_xSi$ crystals acquires a magnetic moment when the nearest neighbor of the cobalt ion is one or several iron ions; otherwise, the cobalt ion remains nonmagnetic. It is also known [14] that the metallic iron–cobalt melt for investigated concentrations of the impurity has a bcc structure and the magnetization of the alloy with $x \le 0.3$, as well as partial magnetizations of the iron and cobalt subsystem, increases. Therefore, an increase in the magnetic susceptibility in $Fe_{1-x}Co_xSi$ crystals is likely associated with the formation of Fe–Co complexes. In this case, the total magnetic moment consists of two contributions:

$$M(H, T) = mN\{\coth(mH/k_BT) - k_BT/mH\} + M_{Fe-Co}, \quad (1)$$

where the term in the braces is the contribution of the subsystem of superparamagnetic iron clusters [7] and clusters earlier appearing owing to nonstoichiometry (m is the magnetic moment of the cluster and N is the number of clusters) and the last term is the contribution from complexes containing cobalt ions. The approximation of the low-temperature part of the experimental susceptibility curves ($\chi = M/H$) in Fig. 2 with the simulation of the magnetization by Eq. (1) gives satisfactory agreement at the values presented in the table. Comparison of these data with similar data obtained earlier [7] indicates that the magnetic moment of the iron cluster in the studied polycrystals is approximately equal to that observed in FeSi single crystals ($m \approx 2.13 \times 10^{-18}$ G or about $230\mu_B$) and the

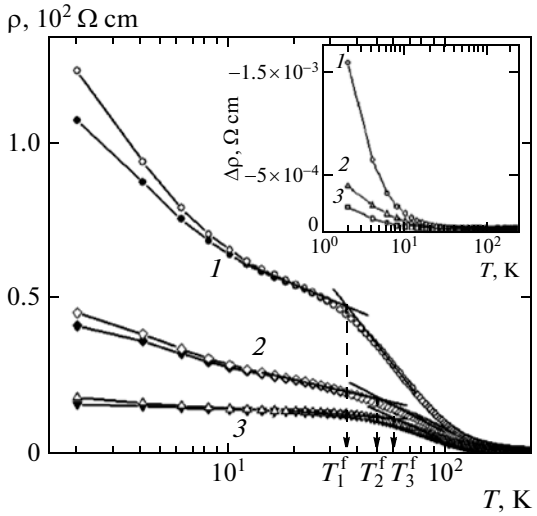

Fig. 5. Temperature dependences of the resistivity of $Fe_{1-x}Co_xSi$ samples with x = (*1*) 0.001, (*2*) 0.005, and (*3*) 0.01 at H = (open points) 0.6 and (closed points) 5000 Oe. The inset shows the magnetic-field dependent contribution $\Delta\rho(T) = \rho(H = 500) - \rho(H = 0)$.

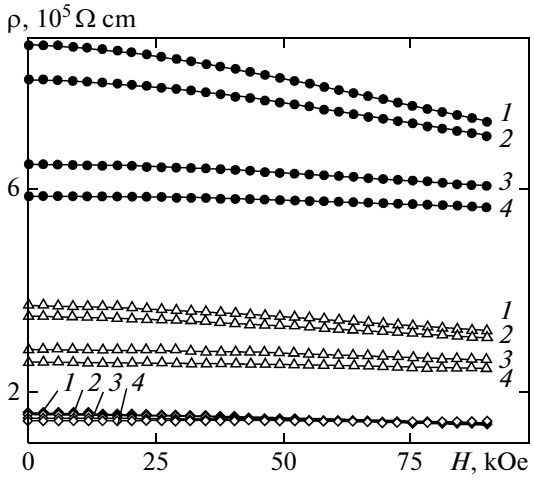

Fig. 6. Field dependences of the resistivity of the $Fe_{1-x}Co_xSi$ samples with x = (●) 0.001, (△) 0.005, and (◇) 0.01 at T = (*1*) 4, (*2*) 5, (*3*) 10, and (*4*) 15 K.

number of clusters N is typical for nominally pure iron monosilicide polycrystals.

As was shown in earlier experiments [15, 16], the magnetic moment in mixed iron–cobalt monosilicides appears at the cobalt concentration $x \geq 0.05$. It is also known [17] that the magnetic moment on an impurity in metals appears under the condition

$$I\nu(\varepsilon_F) > 1 \qquad (2)$$

where I is the effective interatomic exchange integral and $\nu(\varepsilon_F)$ is the density of states at the Fermi level. The theoretical calculation [18] of the electronic structure of $Fe_{1-x}Co_xSi$ crystals by the LMTO method shows that when cobalt ions are introduced, the spin-up subband first downshifts and, then, the spin-down subband begins to shift at $x > 0.4$. In this process, the band gap Δ remains almost unchanged. Figures 7a and 7b illustrate such a change in the density of states. Figure 7c shows the positions of the tops of the valence band E_V for both subbands as functions of the cobalt content. (The difference between lines *1* and *2* in Fig. 7c is a line similar to the saturation magnetization curve in mixed $Fe_{1-x}Co_xSi$ crystals [16].) According to the optical measurements [19], the band gap is $\Delta \approx 60$ meV. Using line *1* in Fig. 7c, it is easy to find that the shift of the bottom of the conduction band for the spin-up subband to ε_F at this Δ value occurs when $x \approx 0.044$, which is in agreement with an experimental value of 0.05. Thus, according to Eq. (2), for considered cobalt impurity concentrations, $\nu(\varepsilon_F) \approx 0$ and the isolated cobalt impurity should have no magnetic moment.

Table

Cobalt content	Magnetic moment of the cluster m, 10^{-18} G	Number of the clusters N, 10^{15} cm^{-3}
$x = 0.001$	1.85	6.6
$x = 0.005$	2.2	7.3
$x = 0.01$	2.3	8.5
$M_{Fe-Co} = 2.95 \times 10^{-3}$ G		

An increase in the magnetic susceptibility in the high-temperature region is due, first, to the addition M_{Fe-Co} and, second, possibly to the features of the electronic structure of the $Fe_{1-x}Co_xSi$ crystal (see, e.g., Fig. 7b). Comparison of the behaviors of the magnetic susceptibility in the high-temperature region for the nominally pure and doped samples indicates that the position of the high-temperature maximum remains unchanged and only the height of the peak increases with the cobalt concentration (see Fig. 2). Such a behavior can be attributed to a doping-induced decrease in the energy gap separating the e_{2g}^4 and $e_{2g}^3 t_{2g}^1$ iron states, leading to a larger population of the excited level under the same conditions. However, another scenario is also possible. As is known [20], a local thermally induced magnetic moment can appear on an impurity in view of the s–d interaction of conduction electrons with the impurity in the impurity limit. This moment begins to be manifested at temperatures above the Kondo temperature (T_K) [21], whereas at low temperatures, the s–d interaction (at $J < 0$) suppresses the local magnetic moment. In addition, it was shown theoretically [22] that the introduction of cobalt to metallic iron is accompanied by a change in the magnetic properties of the alloy because of the shift of the spin-up subband with respect to the spin-down subband (similar to the

behavior shown in Fig. 7); this shift leads to an increase in the magnetization. Such a mechanism can explain the modification of the magnetic properties accompanying the formation of FeCo complexes.

The magnetic-field dependence of the magnetization at low temperatures is explained in the framework of the model of superparamagnetic clusters. Weak hysteresis phenomena are due to the small anisotropy and to the disorientation of clusters, as well as to the weak exchange interaction between clusters under their segregation [7]. An increase in hysteresis with the cobalt concentration is explained by an additional contribution to anisotropy from cobalt-containing complexes (see Fig. 3). At room temperature, when anisotropy almost disappears, magnetization curves exhibit no hysteresis (see Fig. 4).

3.2. Electrical Properties

The behavior of the electrophysical properties is similar to the behavior of a Kondo system. In the framework of such an approach, we analyze the experimental results under the assumption that the appearing magnetic impurity clusters serve as scattering centers for conduction electrons. As is known [21], the existence of a minimum in the temperature dependence of the resistivity is explained by the competition between the electron–phonon scattering ($\rho_{s-ph} = bT^5$, where $b = \text{const}$) and spin-dependent scattering (ρ_s) of conduction electrons on magnetic impurities, which is described by the expression

$$\rho_s = \rho_0\left[1 - 2JN\nu(\varepsilon_F)\ln\frac{\varepsilon_F}{T}\right], \qquad (3)$$

where N is the concentration of the magnetic particles, J is the constant of the exchange interaction of a conduction electron with the magnetic moment of a particle, $\rho_0 \sim (Jm)^2$, and m is the magnetic moment of the scattering particle. It is easy to determine the temperature $T_{min} \sim N^{1/5}m^{2/5}$ at which the resistivity $\rho = \rho_{s-ph} + \rho_s$ is minimal.

Since the increasing section of the $\rho(T)$ dependence for these compounds is of no special interest, we focus on the section below the temperature at which the resistivity is minimal. First, we consider the nominally pure FeSi crystal. The number of intrinsic charge carriers at a band gap of $\Delta \approx 60$ meV in the low-temperature region $T < 30$ K is very small, because the Gibbs factor is no more than 10^{-8}. For this reason, a noticeable conductivity can be due to the existence of tails in the band gap because of the imperfection of the crystal. Since the concentration of impurity clusters N is approximately the same for various samples, this does not affect the position of the minimum in the temperature dependence of ρ. At the same time, the magnetic moment of an iron cluster is $m \sim 250\mu_B$ [7] and, taking into account that $m_{Fe} \approx 3\mu_B$, the gain for scattering by the iron cluster is $(m/m_{Fe})^{2/5} \sim 6$. This explains a quite high temperature at which the resistivity of nominally pure FeSi crystals is minimal as compared to compounds in which scattering occurs by single impurity ions [23].

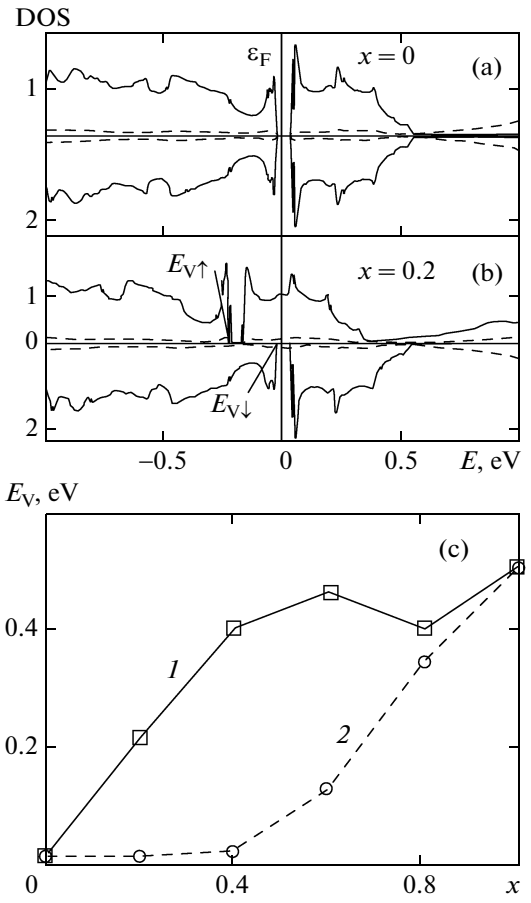

Fig. 7. (a, b) Calculated densities of states in the $Fe_{1-x}Co_xSi$ crystals [16]; ε_F is the Fermi energy and $E_{V\uparrow}$ and $E_{V\downarrow}$ are the tops of the spin-up and spin-down valence subbands, respectively. (c) Cobalt-concentration dependence of the position of the top of the (*1*) spin-up and (*2*) spin-down valence subbands (according to data from [16]).

In doped crystals, there is an additional scattering channel by cobalt-containing complexes. Nevertheless, the resistivity decreases. This circumstance can be attributed to an increase in the density of states in the band gap owing to the shift of subbands with different directions of the electron spins. The effect of a change in the density of states in the band gap is likely prevailing. However, the existence of the additional magnetic subsystem is manifested in the resistivity of the sample. According to Eq. (3), a decrease in the temperature is accompanied by an increase in the resistivity of the Kondo system at $J < 0$; the experiment confirms this behavior. As the temperature decreases, the increase in the resistivity ceases when the interaction between magnetic impurities becomes significant. In our case (see Fig. 5), this is manifested in the formation of a step in the temperature dependence $\rho(T)$

at the temperature T_i^f ($i = 1, 2, 3$). At this temperature, the directions of the magnetic moments are "frozen." As is known [21], the spin-flip scattering of electrons is impossible when the orientation of impurity spins is fixed. According to the estimates made in [7], the average distance between magnetic particles in the nominally pure FeSi crystal is about 50 nm and the interaction between these particles can be disregarded when the electron density is low. In this case, the effect of the magnetic field leads to the fixation of the direction of the magnetic moment of the particle, counteracts the effect of the temperature, and thus reduces the probability of spin-dependent scattering. Additional cobalt-containing magnetic scattering complexes appear in $Fe_{1-x}Co_xSi$ crystals. Although the magnetic order does not yet appear in the crystal, but in addition to an increase in the conduction electron density, the distance between various magnetic structures decreases and additional correlation appears; i.e., magnetic interactions become stronger. For this reason, the spin-freezing temperature increases with the cobalt ion content; this increase is confirmed in the experiment, see Fig. 5 ($T_1^f = 34$ K, $T_2^f = 48$ K, and $T_3^f = 57$ K). The resistivity of doped crystals decreases in the magnetic field and an increase in the cobalt content suppresses the magnetic field dependent contribution to ρ (see the inset in Fig. 5). This is most clearly seen in Fig. 6, where the field dependences of the resistivity are shown. According to the Kondo scattering mechanism, the resistivity is a decreasing quadratic function of the magnetic field [21]. In our case, the experimental results are well described by the function

$$\rho(H) = \rho_0 - AH - BH^2, \quad (4)$$

where H is measured in oersteds, $A = 3.3 \times 10^{-11}$ and $B = 3.01 \times 10^{-16}$ for the crystal with $x = 0.001$ at $T = 4$ K. For all analyzed impurity concentrations, the ratio of the linear term to the square term decreases by a factor of 5 with an increase in the temperature from 4 to 15 K. Dependences given by Eq. (4) are observed in inhomogeneously magnetized ferromagnets and antiferromagnets [23], where various signs of the constants A and B are possible. In this case, the linear term can be attributed to the appearance of local regions with spontaneous magnetization in view of the introduction of cobalt impurities.

4. CONCLUSIONS

The performed investigations indicate that the introduction of cobalt impurity ions in the FeSi crystal modifies the magnetic and electrical properties. The low-temperature behavior of the magnetic susceptibility is explained by the existence of superparamagnetic iron clusters and by the formation of Fe–Co complexes. The magnetic moment on the Co ion appears because of a change in the energy structure owing to the relative shift of the spin-up and spin-down subbands.

The magnetotransport properties are satisfactorily described in the framework of the Kondo model. The presence of the cobalt subsystem leads to the appearance of spin-dependent channel in the scattering of conduction electrons. An increase in the cobalt concentration is accompanied by the reduction of the magnetic field dependent contribution and an increase in the spin freezing temperature. The linear term in the field dependences of the resistivity indicates the beginning of the formation of magnetically ordered microregions.

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