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Abnormal Electron Transport in Graphite Intercalation Compounds with Iron

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The paper presents the results of experimental studies of the structure, Hall coefficient, and thermopower of a graphite intercalation compound (GIC) with iron which was obtained by the reduction of metal chloride in the GIC by C₈K. It is shown that the intercalated compound of graphite with iron demonstrates the effect of charge transfer between metal and graphite layers. The revealed sharp rising of the Hall coefficient at low values of the magnetic induction is considered as the onset of the abnormal Hall effect that is inherent to the structures with an essential interaction between charge carriers and magnetic moments of intercalated atoms. The thermopower for GICs with iron is shown to contain not only the diffusion and phonon terms but also a constituent related with the onset of the Kondo effect in this system.

Keywords Abnormal Hall effect; charge transfer; graphite intercalation compound; Kondo effect; thermopower

Introduction

Recently, due to the extensive development of spintronics, tremendous efforts of researchers are directed to the creation of layered structures consisting of the alternating magnetic and nonmagnetic layers. Therein, the creation of graphite intercalation compounds (GICs) looks to be the exclusively promising line of development. GICs are known to be natural two-dimensional electronic systems that are formed at the introduction of monoatomic or monomolecular layers of different agents (e.g., intercalates) between graphite sheets [1]. The characteristic features of the formation of GICs are the alternation of intercalate layers between graphite sheets (the special type of a superstructure) and the charge transfer from intercalate to graphite layers. Therewith, the donor-type GICs are formed, when graphite layers gain a negative extra-charge; otherwise the acceptor compounds are formed [2].

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The engineering of the intercalated compounds containing cobalt, iron, and nickel as intercalates is of a particular interest. According to the data of theoretical simulations, the properties of charge carriers are changed significantly. In particular, the degree of electron's polarization can reach even 100% due to the interaction between graphite layers enriched by additional charge carriers from layers of a metal [3,4]. Just this defines the suitability of these materials for spintronics. GICs attract a certain interest also due to the possibility to produce a transition metal in the form of a monolayer encapsulated in the graphite matrix, which prevents the oxidation of a metal.

The metal-graphite layered compounds have been synthesized for the first time by Vol'pin et al. in the former Soviet Union in the mid-1970s. Vol'pin et al. have succeeded to synthesize metal-graphite compounds such as Fe-, Ni-, Co-, Cu-, Mo-, W-, and Cr-graphite ones [5,6]. Although they have obtained interesting results on the structural and magnetic properties of these compounds, no special attention has been directed toward such studies. In 1996, Touzain *et al.* [7] have reported on their success in the synthesis of an electrochemically reduced cobalt-graphite layered compound. Cobalt atoms form an island structure between adjacent graphene sheets. The in-plane structure of such compounds is a commensurate structure, where atoms of cobalt are situated at the center of each graphite hexagon. In 1998–2004, Walter has succeeded in synthesizing a number of metal-graphite compounds with zinc, molybdenum, palladium, and other metals by the reduction of the corresponding metal-chloride GIC's [8–12]. This method of synthesis is rather different from that used by Vol'pin et al. According to the appropriate schemes of synthesis, GICs intercalated with metal chlorides were obtained at the first technological stage. At the second stage, the metal was reduced from a metal salt in liquid Li diphenylide at room temperature [13] or in the gaseous phase by contact with the hydrogen gas at a high temperature of $\sim 350\text{--}400^\circ\text{C}$ [12–13]. The materials synthesized according to these schemes were found to be the layered systems. However, in contrast to GICs, a polyatomic, rather than monoatomic, metal layer is formed between graphite sheets. This is related to fact that the synthesis of these compounds is performed at high temperatures, so the diffusion processes occur to be significant. Moreover, the metal layer is composed by separate nanoparticles in a form of either 1-D chains or 2-D scales. In this case, the main peculiar feature of the GIC formation is not observed. Namely, no charge transfer between graphite and metal layers is observed. The van der Waals interaction arising between graphite and metal layers is significantly lower than that in GICs, which is determined by the absence of the interlayer Coulomb interaction.

So, in spite of extensive possibilities to use GICs with transition metals in spintronics, the problems of their synthesis and the investigation of transport and magnetic characteristics are far from the solution. Our previous study has shown that the production of graphite intercalation compounds, particularly with Co, is possible, in principle. The abnormal behavior of transport and magnetic properties of these compounds related to the formation of GICs clusters with cobalt has been reported in [14,15].

The aim of this work was the testing of the possibilities of the production of bulk fine crystalline graphite intercalated with iron, the analysis of peculiarities of the Hall coefficient and the thermopower, and the ascertainment of the characteristics of charge transfer on this basis.

Experimental

Anisotropic pyrolytic fine crystalline graphite (PAFG) (crystallite size $L_a \sim 30$ nm, interplanar spacing $d_{002} = 0.34$ nm) was used as a source material for the GICs synthesis. The latter were synthesized by the two-stage method described in [14], the intercalated compound of PAFG with C_8K being used as a precursor. The intercalated compound with potassium according to the reaction: $C + K \rightarrow C_8K$ was obtained at the first stage. At the second stage, the reduction of iron chloride to iron metal with the subsequent substitution of K atoms by Fe in the interplanar spacing was performed. The reaction of iron chloride reduction runs in the tetrahydrofuran (THF) medium: iron chloride in 50 ml of THF was heated in vacuum up to 350–400 K, and then C_8K was added to the mixture. The solution was thoroughly stirred for 3 days in a magnetic blender and then repeatedly washed in THF and a water solution of alcohol (1:1 ratio). The reaction runs according to the scheme: $C_8K + FeCl_3$ (in a THF solution) $\rightarrow C-Fe + KCl$. As a result of the interaction of C_8K and $FeCl_3$, K was substituted with Fe, and C_8K compound was transformed to the graphite – Fe compound.

The structure and the phase composition of synthesized GIC specimens were studied by a DRON-4-07 automated X-ray diffractometer. The Hall coefficient was determined by the routine procedure at temperatures of 77 and 293 K and at a magnetic field up to 1.8 T. The thermopower was measured within the temperature range (4.2–293) K, by using the method described in [16].

Results and Discussion

Figure 1 presents parts of the X-ray diffraction patterns for the source PAFG used in the intercalation process, as well as for the intercalated compound containing iron that was prepared by the method described earlier.

As is easily seen from Figure 1, the X-ray diffraction pattern for the GIC contains a series of additional peaks as compared with that for the source graphite. Reasoning from the performed analysis, these peaks evidences the formation of a superlattice due to the presence of iron layers which are placed between graphite layers in a certain order. Using data of the X-ray diffraction, the identity parameter

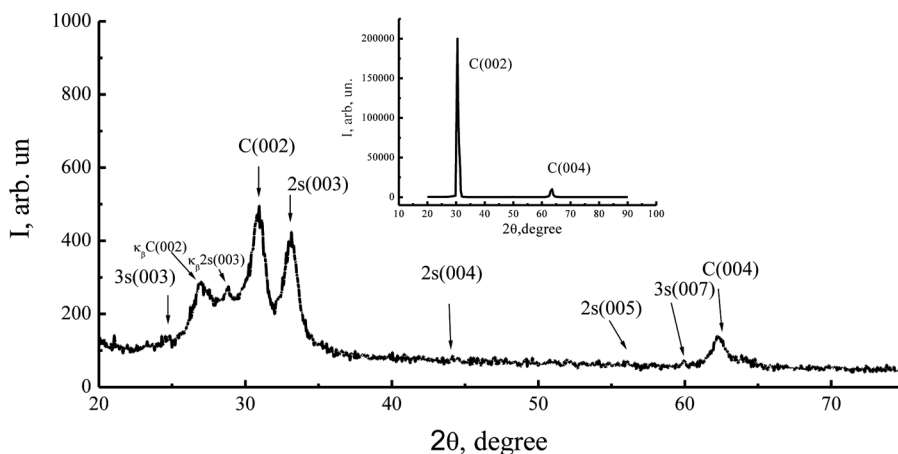


Figure 1. X-ray diffraction patterns for the GIC PAFG-Fe and for the source PAFG (insert).

I_s of the compound has been determined as the distance between the subsequent intercalate layers separated by S graphite layers (S is the number of a GIC intercalation stage). This distance was found to be equal to 9.81 nm.

Thus, the X-ray diffraction data evidence the formation of a periodic layered structure as a result of the performed reactions of intercalation. The intercalate layers alternate with graphite layers, which is inherent to the intercalated compounds of graphite. Since the interplanar distance in a source disperse graphite is equal to 0.34 nm, the synthesized compound is a compound of the second stage.

A peculiar feature of the GICs of low stages is the metallic type of conductivity. Reasoning from the signs of the Hall coefficient and the thermopower, the alkaline metals intercalated into graphite act as electronic donors which donate the valent s-electrons that are localized in the π -bands of graphite. *Vice versa*, the signs of the Hall coefficient and the thermopower in molecular GICs prove that the intercalate acts as an acceptor capturing the electrons from graphite. As a result, the delocalized holes are formed.

The performed structural studies of the synthesized specimens directly indicate the formation of layered compounds. However, these studies do not give the answer to the main question: whether the charge transfer from iron to graphite layers occurs, since just this process is a specific feature of the formation of GICs and results in qualitatively new properties of GICs as compare with the source graphite.

The investigation of the temperature and field dependences of the Hall coefficient has been performed in order to detect a change of the charge carriers concentration in the GICs as compare with the source graphite.

The temperature dependence of the Hall coefficient of pyrolytic fine crystalline graphite is known to be rather complicated [15]: the Hall coefficient decreases with increase in the temperature from 4.2 K to an ambient value indicating an increase in the total charge carriers concentration. Meanwhile, the sign of the Hall coefficient remains positive within the whole temperature range. This is determined by the exceeding concentration of holes as compared with that of electrons in PAFG.

Figure 2 presents the experimental temperature and field dependences of the Hall coefficient for the synthesized GICs with iron.

Two ranges are observed in the field dependences of the Hall coefficient for GICs. The first of them corresponds to a sharp decrease of the Hall coefficient, and the second one exhibits the absence of the Hall coefficient changes with the field. The latter is similar to the $R_H(B)$ dependence for GICs with nonmagnetic materials. Additionally, the value of R_H does not depend on the temperature, which is inherent to degenerated electronic systems, among which are GICs of low stages. The estimation of the charge carriers concentration within the model of conductivity of systems containing one type of carriers gives the value $n = 3.87 \cdot 10^{26} \text{ m}^{-3}$ that is as 35 times as higher than the total charge carrier concentration in the source PAFG.

Let us ascertain the nature of the abnormal dependence of the Hall coefficient in low fields. It is known that the Hall coefficient, which is determined by the action of the Lorentz force on the charge carriers in a magnetic field directed perpendicularly to their flow, does not depend on the magnetic field. The presence of an internal magnetic field in magnetic materials leads to an additional term in the expression for the Hall coefficient [17]:

$$R_H = R_{H0} + R_a \mu_0 M / B, \quad (1)$$

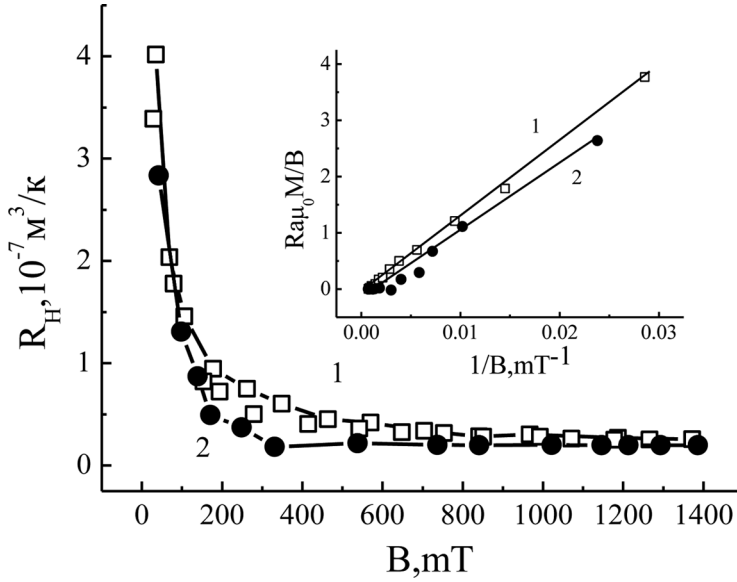


Figure 2. $R_H(B)$ and $R_a \mu_0 M / B(1/B)$ (insert) dependences for the GICs PAFG-Fe, $T = 293$ K (1), $T = 77$ K (2).

where R_{H0} is the normal Hall coefficient, R_a is the abnormal Hall coefficient, μ_0 is the magnetic permeability in vacuum, and M is the magnetization that depends on the magnetic field. The contribution to the Hall effect that is proportional to the magnetization of a material is known as the ferromagnetic, spontaneous, or abnormal Hall effect (AHE). AHE is observed in paramagnets, ferromagnets, antiferromagnets, and Kondo systems. It is caused by the spin-orbit coupling which is proportional to the magnetization and produces the asymmetry of the charge carriers scattering. The latter leads to the “Hall-like” twisting under the absence of a magnetic field. R_a is called usually the AHE constant, since it is field-independent for all ferro-, antiferro-, and paramagnetic materials. However, according to [18], R_a for magnetic granulated alloys exhibits, in general, a nonmonotonous variation with magnetic field due to its influence on the charge carriers scattering, field dependence of R_a being determined by the size of granules.

Thus, taking into account a high concentration of charge carriers in graphite layers, on the one hand, and the presence of magnetic impurities in the studied GIC specimens, on the other hand, one can suppose that just the “asymmetric scattering” that involves the spin-orbit coupling between conduction electrons and magnetic moments of intercalated atoms leads to the onset of AHE.

The dependences of $R_a \mu_0 M / B$ on the reciprocal magnetic induction at two temperatures are presented in the insert in Figure 2.

The dependence of $R_a \mu_0 M / B$ versus $1/B$ is seen to be linear, i.e., $R_a \mu_0 M = \text{const}$. Since the studied GICs with iron have been synthesized on the base of fine crystalline graphite and are consisted of GIC nanoclusters 10–15 nm in size [15], one can suppose that the field dependence of the magnetization is described as $M = M_0 L(B)$, where $L(B)$ is the Langevin function. Then it is obvious that R_a for the GIC containing iron at low fields essentially depends on the magnetic field, and its behavior is determined by the field dependence of the magnetization.

As is clearly seen from the figure, the value of $R_a\mu_0 M/B$ weakly depends on the temperature. The temperature dependence of $R_a\mu_0 M/B$ is determined by the temperature dependences of the magnetization and the R_a coefficient. However, the temperature dependence of R_a is strongly correlated with the temperature dependence of the resistivity [17]. Due to the influence of the spin-orbit interaction during the electron scattering on impurity centers, the arising asymmetry of the probability of the charge carriers scattering leads to the following expression: $R_a \sim a\rho + b\rho^2$. As was shown in [15], the resistivity for GIC with iron within the temperature range (77–300) K does not depend on the temperature: the ratio of resistivities at 77 and 293 K is equal to 0.95. This is due to the strong scattering of charge carriers on crystallites boundaries and defects. That is why one can assume that R_a for the studied GIC also weakly depends on the temperature. Moreover, the temperature independence of $R_a\mu_0 M/B$ evidences also the weak temperature dependence of the magnetization for this compound.

Let us estimate a part of the additional charge f that is transferred from intercalate molecules to a graphite layer and, respectively, the Fermi energy E_F within the ordinary two-dimensional model of the electron structure of a GIC (the model of “metallic sandwich” [19]), by using the data on the Hall coefficient. Within this model, the GIC electronic structure is considered as a sequence of bands with low and high free charge carrier densities along the c axis [19,20]. One of these bands corresponds to pure 3D graphite, and another one corresponds to a 2D “metallic sandwich” consisting of the intercalate layer and two neighboring graphite layers. The unique properties of GICs are caused just due to the existence of a 2D “metallic sandwich”. The electrons captured from graphite at the intercalation occur to be localized in the intercalate layer. They form the electrostatic potential barrier that totally shields the neighboring graphite layers, the hole being formed are delocalized charge carriers in the graphite layers neighboring to the intercalate layer.

The estimation of f and E_F within this model according to the equations listed in [19] gives $f \sim 0.026$ and $E_F \sim 0.20$ eV. The corresponding values for the second-stage intercalated compounds with stibium chloride are equal to $f = 0.67$, $E_F = 1$ eV in a case of HOPG and $f = 0.15$, $E_F = 0.40$ eV in the case of PAFG. The part of an additional charge for PAFG-Fe GICs calculated within the ordinary 2D model of GIC electronic structure is seen to be substantially lower than that for GIC containing stibium chloride.

The temperature dependence of the thermopower has been also studied for the synthesized GICs containing iron.

Among other kinetic properties, just the thermopower is known to provide a profound information on the type of charge carriers, the nature and the temperature dependence of the dominated mechanisms of charge carriers and phonon scattering. The introduction of an intercalate in graphite leads to the transformation of the temperature dependence of the thermopower as compared with that for the source graphite.

Figure 3 presents the temperature dependence of the thermopower for PAFG-Fe GIC. Additionally, the temperature dependence of the thermopower for the source PAFG used for the intercalation is also presented in this figure.

The thermopower for the studied compound exhibits a complicated temperature dependence and essentially differs from that for the source PAFG. As was shown in [18], the character of a temperature dependence of the thermopower for PAFG is determined by the contribution of only the diffusion constituent of the thermopower.

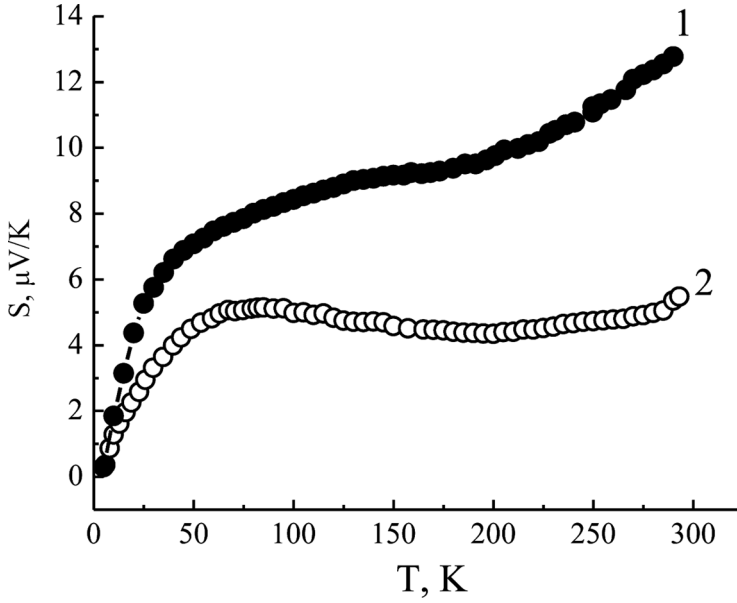


Figure 3. $S(T)$ dependences for the GIC PAFG-Fe (1) and for the source PAFG (2).

The maximal value of the phonon constituent of the thermopower $\sim 0.5 \mu\text{V/K}$ is observed at 5 K. Such a value of the phonon constituent of the thermopower is essentially lower than that observed experimentally.

As was shown, the thermopower for GIC based on both HOPG [21,22] and PAFG [23,24] is described by the sum of two components: the diffusion thermopower S_d and the thermopower associated with the effect of phonon drag of charge carriers S_{ph} . The diffusion thermopower S_d for these materials depends linearly on the temperature [21]:

$$S_d = \frac{\pi^2 k_B^2 T}{3eE_F} (1 + p). \quad (2)$$

Here, k_B is the Boltzmann constant, E_F is the Fermi energy, and p is a parameter that is determined by the contributions of all scattering mechanisms if several scattering mechanisms with close relaxation times contribute to the thermopower, some of which being temperature-dependent [21,23].

The phonon constituent of the thermopower, S_{ph} , can be expressed as [22]

$$S_{ph} = \frac{\langle C_{ph} R(q) \rangle}{3eN}, \quad (3)$$

where C_{ph} is the heat capacity of the phonon system participating in the phonon drag process, N is the density of charge carriers, and $R(q)$ characterizes the relaxation processes of phonons participating in the phonon drag of charge carriers. The temperature dependence of S_{ph} is totally determined by the temperature dependence of $\langle C_{ph} R \rangle$.

The temperature dependences of the diffusion and phonon constituents of the thermopower presented in the insert in Figure 4 have been simulated according the procedure that has been successfully applied in [23,24] to calculate thermopower constituents for an acceptor-type GIC using the data on the Hall coefficient and the Fermi energy $E_F = 0.20$ eV. The total calculated thermopower for PAFG-Fe GIC is also presented in Figure 4.

As is clearly seen from Figure 4, the total calculated thermopower is in a good agreement with experimental data at low temperatures and in a temperature region higher than 200 K. However, an essential deviation between these two curves is observed within the range (30–200) K. Thus, an additional contribution to the thermopower differing from the diffusion and phonon constituents should be supposed to exist in this temperature region. Let us consider possible mechanisms of such a deviation.

It is well known that high experimental values of the thermopower have been observed in highly diluted solutions of transition elements (Ti, V, Cr, Mn, Fe, Co, Ni) in a noble metal matrix (Au, Ag, Cu). Similar maxima on the temperature dependences of the thermopower have been observed for the specimens consisted of carbon nanotubes filled with iron and nickel. They are commonly related to the interaction between magnetic moments of the impurity atoms and electron spins of the matrix, which leads to the spin-dependent scattering and to the appearance of a narrow hybridization peak of the density of electronic states near the Fermi level, i.e., to the Kondo-resonance [25]. The temperature where the Kondo-resonance is observed (the Kondo temperature) within the first approximation can be written as:

$$T_k = \frac{E_F}{k_B} \exp(-|j|D(E_F))^{-1}, \quad (4)$$

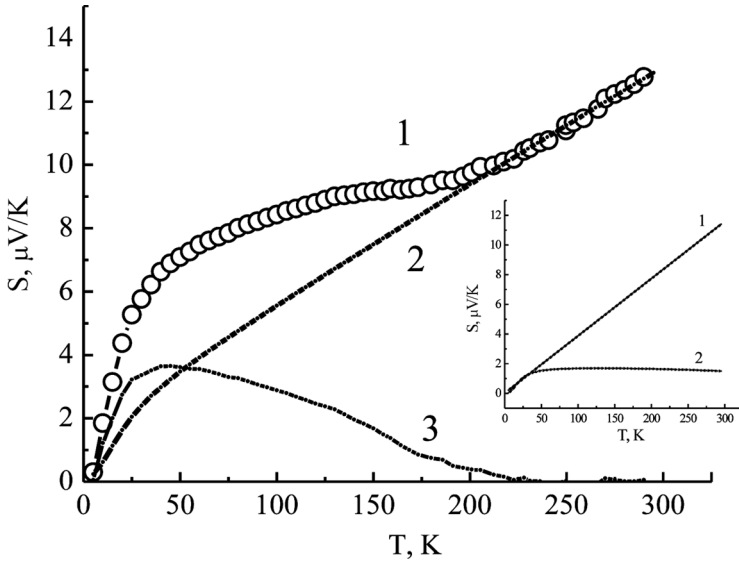


Figure 4. $S(T)$ dependences for GIC PAFG-Fe: 1 – experimental $S(T)$; 2 – calculated $S(T)_{\text{calc}} = S_d + S_{ph}$; 3 – $S_K = S - (S_d + S_{ph})$ insert: calculated $S_d(T)$ and $S_{ph}(T)$.

where \mathbf{j} is the integral of the antiferromagnetic exchange interaction between localized electrons of impurity atoms and conduction electrons of the matrix ($\mathbf{j} < 0$); \mathbf{D} is the polarized density of matrix electronic states (per the magnetic moment of an atom). Relation (4) shows that T_K is sensitive to \mathbf{j} and $\mathbf{D}(E_F)$. The hybridization peak width near E_F is about $k_B T_K$. For the specimens studied in [26], T_K is ranged within (80–100) K and depends on the properties of impurity atoms. In general, T_K in the Kondo systems can vary from several Kelvins to 1000 K.

Thus, reasoning from the structure of the studied specimens (layered systems with alternative magnetic and nonmagnetic graphite layers) and anomalous transport properties, the abnormal Hall coefficient related to the interaction between charge carriers of graphite layers and magnetic moments of intercalate atoms, in particular, we can assume that the total thermopower contains not only the diffusion and phonon terms but also a constituent arising from the interaction between magnetic moments of iron atoms and spins of charge carriers of the graphite matrix.

Figure 4 presents the temperature dependence of $S_K = S - (S_d + S_{ph})$. It is clearly seen that the $S_K(T)$ dependence is represented as a peak with the maximum at $T = 45$ K which falls down to zero at 200 K.

According to [26], where the transport properties were investigated, in particular, the temperature dependence of the thermopower of SWCNT mats produced using transition metal particles as a catalyst, the similar peaks of the temperature dependence of the thermopower have been related to the Kondo effect. The corresponding temperatures are ranged between 80 and 100 K depending on the type of a magnetic metal.

Conclusion

The investigation of the Hall coefficient and the thermopower for PAFG-Fe compounds has shown that the intercalated compound of graphite with iron, in contrast to those with other metals such as vanadium and palladium, demonstrates the effect of charge transfer between metal and graphite layers. This leads to an essential increase of the charge carriers concentration in graphite layers and, subsequently, to a modification of the temperature dependences of the Hall coefficient and the thermopower. The revealed sharp rising of the Hall coefficient at low values of the magnetic induction is considered as the onset of the abnormal Hall effect that is inherent to the structures with an essential interaction between charge carriers and magnetic moments of intercalated atoms. The latter is distinctly observed for graphite-based intercalated compounds with magnetic metals. The thermopower for GICs with iron is shown to contain not only the diffusion and phonon terms, but also a constituent related to the onset of the Kondo effect in this system.

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