

# Synthesis and Investigation of Titanium Diselenide Intercalated with Ferrocene and Cobaltocene

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**Abstract**—Single crystals and polycrystals of titanium diselenide  $\text{TiSe}_2$  intercalated with ferrocene  $\text{Fe}(\eta^5\text{-C}_5\text{H}_5)_2$  and cobaltocene  $\text{Co}(\eta^5\text{-C}_5\text{H}_5)_2$  are synthesized. The magnetic susceptibility and electrical resistivity of the intercalation compounds are measured. The results obtained demonstrate that the intercalation brings about the formation of an impurity band with a temperature-dependent width.

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## 1. INTRODUCTION

Over the past forty years, titanium dichalcogenides with a layered structure have been a subject of considerable interest owing to both the strong anisotropy of their physical properties and the possibility of modifying them through intercalation with quite different guest objects, such as alkali, noble, transition-element, and rare-earth metals; organic molecules; and fragments of crystal lattices of simple and rare-earth metal monochalcogenides. The structural unit of titanium dichalcogenides is a so-called sandwich consisting of a hexagonal monoatomic titanium layer coordinated by hexagonally packed chalcogen layers. The crystal structure of titanium dichalcogenides is formed by a stack of these sandwiches separated by wide interlayers, i.e., van der Waals gaps into which the above guest objects can be intercalated [1].

Compounds intercalated with transition metals possessing magnetic moments have attracted the particular attention of researchers, because they can serve as natural analogs of artificially produced layered systems in which magnetic metal layers alternate with nonmagnetic interlayers. However, it turned out that intercalation of transition metals into titanium dichalcogenides brings about the formation of  $\text{Ti-M-Ti}$  covalent centers, where  $M$  is an intercalated metal. This leads to a shortening of the distance between the sandwiches and, hence, to a decrease in the anisotropy, on the one hand, and to a reduction of the magnetic moment of the intercalated atom due to the sharing of  $3d$  electrons with

ligands, on the other hand. As a result, the formation of monoatomic layers of magnetic metals leads to a loss of all the most interesting properties of titanium dichalcogenides [2].

The intercalation of magnetic metal atoms preliminarily embedded in organic molecules preventing direct overlap of  $3d$  orbitals of titanium and intercalated metal atoms seems to be an appropriate way out of the current situation. These molecules can be, for example, molecules of metallocenes, namely, dicyclopentadienyl metal compounds of the general formula  $M(\eta^2\text{-C}_5\text{H}_5)_2 = \text{MCp}_2$  with a structure consisting of two parallel pentagons with CH groups located at vertices and a metal atom at the center of the “barrel” formed by these pentagons (Fig. 1) [3].

To date, it has been established that metallocenes can be intercalated into many materials with a layered structure, including titanium disulfide. Unfortunately, the majority of papers concerned with this problem have noted only the mere fact of the intercalation of metallocenes into layered compounds. Little is known about the intercalation compounds themselves. In their review, Friend and Yoffe [4] argued that, in all cases, the intercalation of metallocenes brings about an approximately identical increase in the van der Waals gap (by 5.5 Å) and initiates charge transfer to the host lattice, as is the case with the intercalation of alkali metals. A comparison between the broadening of the van der Waals gap and the size of the metallocene molecules allows one to determine their spatial orientation: the

pentagon planes are perpendicular to the sandwich planes. Evans et al. [5] demonstrated that the intercalation of metallocenes into layered materials of different types results in an identical (or close) stoichiometry of the intercalation materials in equilibrium with the intercalated metallocene ( $\sim 0.3$  metallocene molecule per formula unit of the host lattice). The above circumstance suggests that the geometric size of the metallocene molecule is a factor controlling the amount of metallocene intercalated. This makes it impossible to elucidate the nature of chemical bonding between intercalated organometallic molecules and the host lattice. However, it is clear that the charge transfer can produce a substantial effect on the magnetic moment of the  $MCp_2$  metallocene.

In this work, titanium diselenides intercalated with ferrocene  $FeCp_2$  and cobaltocene  $CoCp_2$  were synthesized for the first time. In order to elucidate the nature of chemical bonding between intercalated organometallic molecules and the host lattice and to determine the effect of intercalation on the energy spectrum of charge carriers, we investigated the electrical conductivity and magnetic susceptibility of the synthesized compounds.

## 2. SAMPLE PREPARATION AND EXPERIMENTAL TECHNIQUE

Intercalation was performed using the following initial components: ferrocene purchased from Aldrich, cobaltocene synthesized according to the standard procedure described in [6], and titanium diselenide in the form of a powder and single crystals. The metallocenes to be intercalated were additionally purified by recrystallization from hexane solutions. The technique used for preparing titanium diselenide and growing single crystals was described in detail in [7]. Direct thermal intercalation in evacuated Pyrex tubes proved possible only for ferrocene. We succeeded in preparing a homogeneous intercalated material after heat treatment at a temperature of  $200^\circ\text{C}$  for approximately one month in an evacuated Pyrex tube. A lower decomposition temperature of cobaltocene made use of this technique impossible. It seems likely that, despite the thermodynamic favorability, the inhibition of the intercalation reaction is associated with the specific dielectric properties exhibited by the above compounds, which hinder the ionization required to form chemical bonds between these compounds and titanium diselenide. This circumstance suggests that the metallocene– $TiSe_2$  interaction is ionic or covalent in character but is not provided by van der Waals forces.

This difficulty was obviated with the use of tetrahydrofuran solutions of metallocenes in the course of intercalation. It was assumed that solvation of metallocene molecules by the solvent can encourage their ionization due to an increase in the permittivity of the medium. In actual fact, the intercalation of cobaltocene

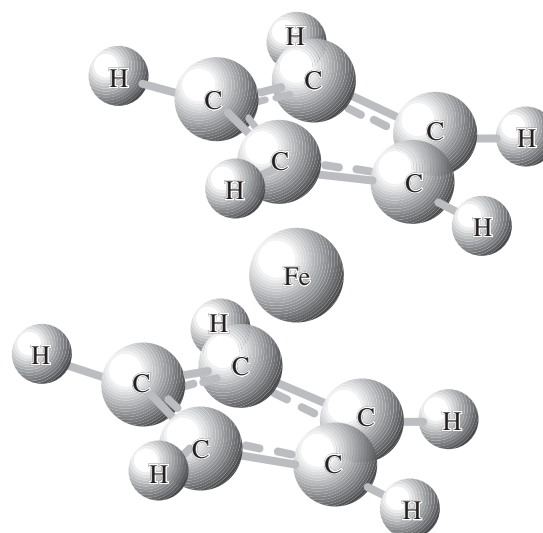


Fig. 1. Molecular structure of ferrocene (dicyclopentadienyliron).

into a  $TiSe_2$  powder under these conditions proceeded at a very high rate and titanium diselenide changed in color from violet to black over several hours. In the process, a solid pressed pellet transformed into a fine powder. Probably, this phenomenon was caused by the strong stresses arising in the lattice upon the intercalation of large molecules. For reliability, after the visible changes were complete, the sample was held in contact with the solution for one week and was then washed with xylene in order to remove excess metallocene and dried in a nitrogen stream with a residual oxygen pressure of no higher than  $10^{-10}$  Torr. It is worth noting that, under these conditions, there was no interaction between ferrocene and titanium diselenide and no indication of intercalation was revealed after storage for even several months.

It should be noted that the intercalation reaction in the solution did not accelerate with an increase in the temperature from  $20$  to  $60^\circ\text{C}$ . Moreover, the reaction rate decreased during heating: the sample completely changed color in two or three days at  $60^\circ\text{C}$  rather than in a few hours at  $20^\circ\text{C}$ . The revealed phenomenon indicates that the key role in the acceleration of the reaction is played by an increase in the permittivity, which decreases during heating due to the destruction of the solvate shells. This confirms the assumption that the inhibition of ionization is responsible for the retardation of the kinetic processes and suggests that charge transfer occurs between the intercalant and the host lattice in the course of the intercalation.

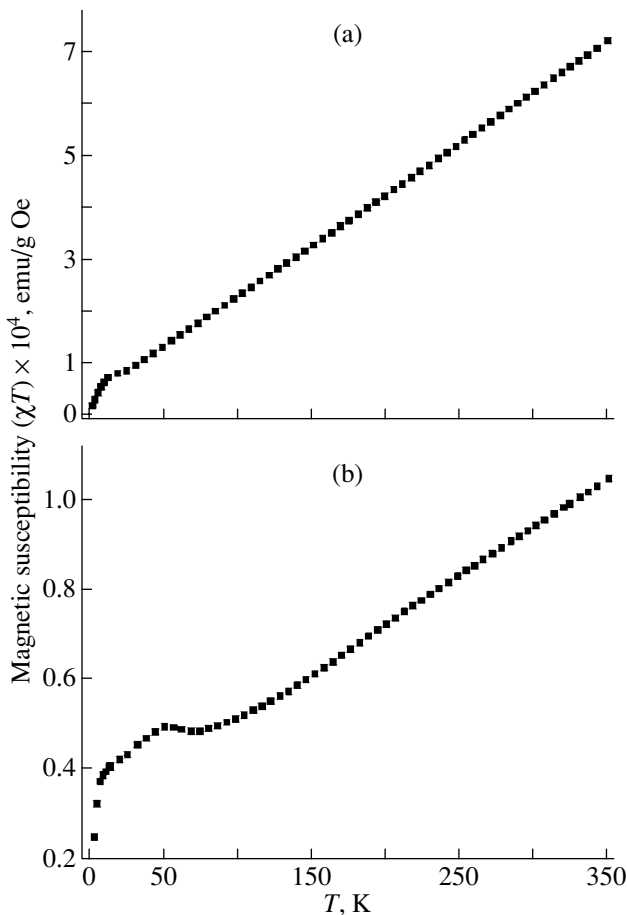
The composition of the material in equilibrium with the corresponding metallocene was determined using electron microprobe analysis with a JEOL-733 microscope. It should be noted that the masses of hydrogen and carbon atoms were too small to be determined by this method. Therefore, the presence of these elements

**Table 1.** Lattice constants  $c_0$  of the  $\text{TiSe}_2$  diselenide and its changes upon intercalation of metallocenes

Compound	$c_0$ , Å	$\Delta c_0$ , Å
$\text{TiSe}_2$	6.008	
$(\text{Cp}_2\text{Co})_{0.25}\text{TiSe}_2$	11.701	5.693
$(\text{Cp}_2\text{Fe})_{0.25}\text{TiSe}_2$	11.685	5.677

was judged from the concentration ratio between either iron and titanium or cobalt and titanium. This ratio turned out to be equal to  $0.25 \pm 0.03$  (irrespective of the nature of the metallocene and the specific features of the intercalation reaction).

The structural investigations were performed on a DRON-3M x-ray diffractometer ( $\text{CuK}_\alpha$  radiation, Ni filter). The magnetic measurements were carried out on a SQUID magnetometer in the temperature range 2–370 K. The electrical resistivity was measured using the four-point probe method along the basal plane of  $\text{TiSe}_2$  single crystals intercalated in the same way as the powdered materials.

**Fig. 2.** Temperature dependences of the magnetic susceptibility  $\chi T$  for (a)  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  and (b)  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  compounds.

### 3. RESULTS AND DISCUSSION

The x-ray diffraction patterns of the  $\text{TiSe}_2$  compounds intercalated with  $\text{MCp}_2$  ( $M = \text{Fe}, \text{Co}$ ) metallocenes proved too complicated to be indexed uniquely. It is possible to interpret uniquely only the (001) lines corresponding to reflections from the basal planes of the material. For this purpose, the powder was pressed to prepare a sample with a strong texture. Diffraction from the sample thus prepared was interpreted as diffraction from the basal planes of the intercalated single crystals. The results obtained are presented in Table 1. No lines of the initial compounds are revealed.

It can be seen from Table 1 that the intercalation leads to an increase in the lattice constant  $c_0$ , which characterizes the periodicity in the direction normal to the layer planes of the host lattice of the titanium diselenide. It is reasonable to attribute this increase to the increase in the distance between the sandwiches in the lattice of the  $\text{TiSe}_2$  initial compound due to the intercalation of  $\text{MCp}_2$  molecules into the host lattice. The increase in the van der Waals gap is close to a mean value of 5.5 Å observed upon intercalation of different metallocenes into layered materials [4].

The temperature dependences of the magnetic susceptibility for the  $(\text{MCp}_2)_{0.25}\text{TiSe}_2$  ( $M = \text{Fe}, \text{Co}$ ) compounds were approximated by the standard Curie-Weiss relationship

$$\chi = \chi_0 + \frac{C}{T + \Theta}, \quad (1)$$

where  $\chi_0$  is the temperature-independent magnetic susceptibility,  $\Theta$  is the paramagnetic Curie temperature, and  $C$  is the Curie constant. The fitting of the experimental data with the use of relationship (1) demonstrated that the condition  $\Theta = 0$  is satisfied with a good accuracy. This implies that the magnetic interaction in the intercalant sublattice is absent. Hence, it follows that relationship (1) can be simplified. For this purpose, both sides are multiplied by the temperature  $T$ . The obtained dependences of the quantity  $\chi T$  on the temperature  $T$  are plotted in Fig. 2. As can be seen from this figure, the temperature dependences of the quantity  $\chi T$  at temperatures above 50 K for the  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  compound and above 10 K for the  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  compound exhibit a linear behavior (and a kink at lower temperatures). The temperatures of the anomalies revealed in the dependences  $\chi T(T)$  are close to those of the anomalies in the heat capacity of the  $\text{FeCp}_2$  and  $\text{CoCp}_2$  metallocenes [8], whose origin, however, is still not clearly understood. The observed coincidence of the temperatures can indicate changes in the internal structure of the molecules, for example, due to a change in the mutual orientation of the pentadienyl rings. The fitting parameters for the dependences  $\chi T(T)$  in the high-temperature and low-temperature (2–10 K for the  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  compound and 6–50 K for the  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  compound) ranges are given in Table 2.

It can be seen from Table 2 that the intercalation of the  $\text{CoCp}_2$  metallocene leads to a considerable decrease in the effective magnetic moment  $\mu_{\text{eff}}$ . Note that the magnetic moment in the intercalation compound can be attributed only to the cobalt atom. Therefore, the above circumstance suggests a change in the charge state of the cobalt atom as a result of charge transfer between the intercalant molecule and the host lattice. This change itself does not permit us to determine the direction of charge transfer, because any change in the charge state of the cobalt atom with a  $3d^7$  configuration leads to a decrease in the magnetic moment. However, owing to the strong electron-donating ability of the  $\text{CoCp}_2$  molecule and the impossibility of intercalating acceptor compounds into titanium dichalcogenides [1, 4], it is reasonable to assume that the intercalation brings about a transfer of electrons from the  $\text{CoCp}_2$  molecule to the host lattice of the titanium diselenide. Similar reasoning holds for the  $\text{FeCp}_2$  metallocene, whose diamagnetic nature originates from the low-spin state of the iron atom with a  $3d^6$  configuration [9].

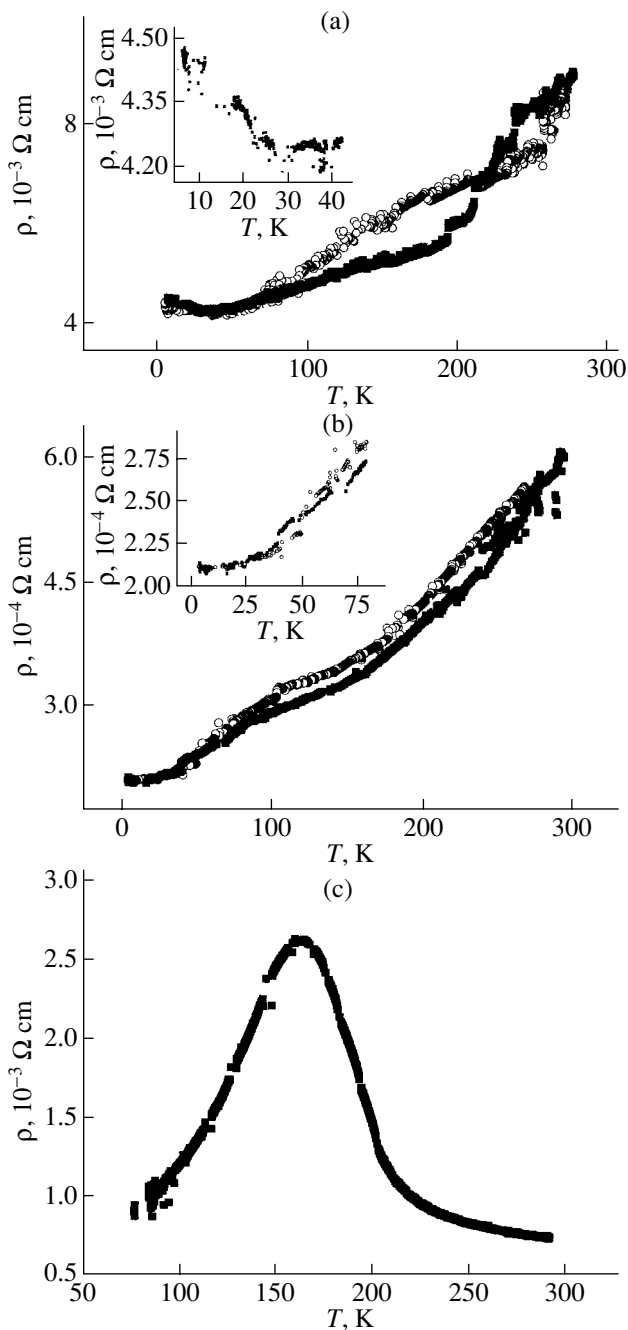
The above inference is in good agreement with the increase observed in the magnetic susceptibility  $\chi_0$  upon intercalation. Since the value of  $\chi_0$  is positive, this quantity can be related to the magnetic susceptibility of free electrons (the Pauli susceptibility). The initial compounds  $\text{FeCp}_2$  and  $\text{CoCp}_2$  are dielectrics and, hence, do not make a positive temperature-independent contribution. It is evident that the charge carriers responsible for this contribution for the intercalation compound can be localized only in the host lattice of titanium diselenide. A comparison between the Pauli susceptibilities  $\chi_0$  of the initial and intercalated materials demonstrates that the intercalation leads to an increase in the Pauli susceptibility. Note that the magnetic susceptibility  $\chi_0$  is directly proportional to the density of states  $\rho(E_F)$  at the Fermi level. Therefore, this behavior suggests that, upon intercalation of metallocenes, the density of states  $\rho(E_F)$  at the Fermi level increases drastically. According to Krasavin et al. [10], the Pauli susceptibility  $\chi_0$  is scarcely affected by simple electron transfer to the conduction band. Hence, it follows that the intercalation leads to a transformation of the energy spectrum of charge carriers in the  $\text{TiSe}_2$  diselenide. In our previous study [2], we also revealed a similar effect upon the intercalation of transition metals into the  $\text{TiSe}_2$  diselenide. For these materials, the observed effect was explained by the formation of a band of localized impurity states generated through the hybridization of the valence states of the impurity atom with the Ti  $3d$  states of the host lattice [7]. Most likely, the intercalation of the  $\text{FeCp}_2$  and  $\text{CoCp}_2$  compounds gives rise to a similar effect with the formation of covalent bonds between the metallocene molecules and the host lattice. Therefore, the inference can be made that an increase in the Pauli susceptibility  $\chi_0$  upon cooling is associated with the increase in the degree of localiza-

**Table 2.** Effective magnetic moments  $\mu_{\text{eff}}$  and Pauli susceptibilities  $\chi_0$  for titanium diselenide  $\text{TiSe}_2$  and its intercalation compounds

Compound	Temperature range, K	$\mu_{\text{eff}}/\mu_B$	$\chi_0, 10^{-6}$ emu/(mol Oe)
$\text{TiSe}_2$	>200		8.23 [8]
$\text{CoCp}_2$	77–300	1.81 [10]	
$\text{FeCp}_2$	77–300	Diamagnetic	
$(\text{CoCp}_2)_{0.25}\text{TiSe}_2$	12–350	0.54	439
	2–10	0.24	1203
$(\text{FeCp}_2)_{0.25}\text{TiSe}_2$	70–350	0.45	49
	6–50	0.52	53.6
$\text{Co}_{0.25}\text{TiSe}_2$ [2]	77–350	2.3	240
$\text{Fe}_{0.25}\text{TiSe}_2$ [2]	77–350	4.2	327

tion of impurity states and that the change in the effective magnetic moment  $\mu_{\text{eff}}$  is caused by the shift (due to this increase) of the Fermi level and, hence, by the change in charge transfer between the metallocene molecules and the host lattice.

Figure 3 shows the temperature dependences of the electrical resistivity for the intercalation compounds  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  (Fig. 3a) and  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  (Fig. 3b) and the initial compound  $\text{TiSe}_2$  (Fig. 3c). It can be clearly seen from Fig. 3 that the intercalation leads to suppression of the resistive anomaly, which is observed for the  $\text{TiSe}_2$  diselenide [10] and can be associated with the transition of the material to a charge-density-wave state at temperatures below 202 K. It should be remembered that the condition of this transition is the equality of the electron and hole concentrations [11]. Therefore, the suppression of the transition to a charge-density-wave state is consistent with the inference that there occurs charge transfer between the intercalant and the host lattice and that this transfer disturbs the above balance. The temperature dependence of the electrical resistivity exhibits a metallic behavior. This is also in good agreement with the electron transfer between the intercalant and the host lattice. The temperatures of the anomalies observed in the temperature dependences of the electrical resistivity at 150 K for the  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  compound and at  $\approx 100$  K for the  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  compound are close to those of the structural anomalies revealed in the initial molecules due to freezing of the rotational motion of the cyclopentadienyl rings around the fivefold axis [8]. It can be seen that this transition does not manifest itself in the temperature dependences of the quantity  $\chi T$  and, hence, cannot be related to the change in the degree of ionization of the intercalant molecules or to the change in the degree of delocalization of charge carriers. The mutual orientation of the  $\text{C}_5\text{H}_5$  pentagons admits two non-equivalent positions [3]: (i) position *I*, in which the CH



**Fig. 3.** Temperature dependences of the electrical resistivity measured for (a)  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  and (b)  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  intercalation compounds and (c) the  $\text{TiSe}_2$  initial compound during heating (closed symbols) and cooling (open symbols). Insets show the low-temperature portions of the dependences. The temperature dependences of the electrical resistivity measured during cooling exhibit a semiconductor behavior for the  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  compound and an insignificant decrease in the slope for the  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  compound.

groups are located one above another; and (ii) position 2, in which the pentagons are inverted. At high temperatures, the two positions are equally probable due to the

rotation of the pentagons. Freezing of the rotation fixes one of these positions. The rotation is hindered in the vicinity of the transition. Quite probably, this circumstance can lead to the formation of regions in which molecules are located positions 1 and 2. It seems likely that the resistive anomaly in the transition temperature range results from scattering of charge carriers by boundaries of these regions. This is also indicated by the hysteresis observed in the temperature dependence of the electrical resistivity in the transition range. The hysteresis suggests kinetic hindrances and, consequently, a possible spatial inhomogeneity of the material.

In the low-temperature range, the temperature dependences of the electrical resistivity for both materials deviate from the metallic behavior. This deviation is especially pronounced for the  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  compound, which, at temperatures below  $\approx 30$  K, is characterized by a semiconductor-type dependence of the electrical conductivity on the temperature. By contrast, the deviation from the metallic behavior is less pronounced for the  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  compound; however, a decrease in the slope of the temperature dependence of the electrical resistivity is observed in this case also. Apparently, the observed effects indicate that charge carriers are localized upon cooling. The temperatures of localization are close to the temperatures of the kinks in the temperature dependences of the quantity  $\chi T$  for both materials. A comparison between the magnitude of the resistive effect of localization and the change in the Pauli susceptibility  $\chi_0$  for both materials demonstrates good agreement: the effect is clearly pronounced for the  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  compound and is insignificant for the  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  compound. This circumstance allows us to attribute the localization not to the formation of a band gap in the density of states at the Fermi level but to the increase in the degree of localization of impurity states and the corresponding decrease in the width of the impurity band. It is obvious that this mechanism would be impossible in the case of conventional electron transfer (which has been proposed in the literature) from the intercalant to the conduction band of the  $\text{TiSe}_2$  diselenide.

Therefore, the analysis of the temperature dependences of the magnetic susceptibility and the electrical resistivity of the titanium diselenide intercalated with the  $\text{CoCp}_2$  and  $\text{FeCp}_2$  metallocenes demonstrates that the intercalation brings about the formation of an impurity band in which electrons transferred from intercalated molecules are localized.

#### 4. CONCLUSIONS

Thus, the results obtained have demonstrated that the intercalation of metallocenes into titanium diselenide  $\text{TiSe}_2$  is accompanied by charge transfer from the intercalant to the host lattice. However, the mechanism of this transfer is far short of the simplified models

currently accepted for describing the intercalation of organic molecules into layered dichalcogenides [12]. The observed properties of the  $(\text{CoCp}_2)_{0.25}\text{TiSe}_2$  and  $(\text{FeCp}_2)_{0.25}\text{TiSe}_2$  intercalation compounds can be explained under the assumption that the chemical bonding between the intercalant and the host lattice is covalent in nature. This allows one to understand the absence of a correlation between the donating ability and the limiting concentration of molecules that can be intercalated into the lattice under consideration. Actually, this concentration should be determined by the number of positions in which the intercalated molecules can form covalent bonds with the host lattice. However, the electronic properties of intercalation compounds of this type should depend on the characteristics of both (intercalant and host) sublattices. The relative easiness of affecting the properties of organometallic molecules offers new possibilities for modifying an intercalation material under relatively weak actions.

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