Phase transition and electron localization in $1T$-TaS_2

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The magnetic properties and phase transitions of $1T$-TaS_2 and $1T$-$Fe_{0.07}Ta_{0.93}S_2$ have been studied in the interval of 1.5–300 K and over the range of 100 Oe–60 kOe. Experimental results show that at high temperatures the compounds are in a diamagnetic state and the commensurate-charge-density-wave–triclinic-nearly-commensurate transition temperature of $1T$-TaS_2 decreases with increasing magnetic field. The amount of variation is a function of the magnetic field. At low temperatures both $1T$-TaS_2 and $1T$-$Fe_{0.07}Ta_{0.93}S_2$ are in a paramagnetic state owing to the localized moments that come from the single Anderson-Mott localization state. The curves of magnetization versus temperature do not follow the Curie law or Curie-Weiss law, but can be described fairly well as $M=M_0+\gamma T^{-n}$. The fitting parameters of experimental curves show that a part of the neighboring moment appears as antiferromagnetic coupling due to exchange interaction between the moments. The magnetic-field dependence of magnetization exhibits a complicated feature at low temperature. It shows that the compounds may undergo a phase transition at the maximum value of magnetization and then they are probably in a mixed charge-density-wave–spin-density-wave (CDW-SDW) state or SDW state due to the coherent superposition of the antiferromagnetic coupling.

I. INTRODUCTION

The study of the layered compound $1T$-TaS_2, whose complicated features in phase transformations are well known to many, has become a much pursued subject of research in solid-state physics and chemistry. This compound is generally considered as a quasi-two-dimensional material owing to the strong anisotropy of its macroscopic properties, e.g., electrical resistivity. It structurally consists of strongly (covalent) bonded S-Ta-S layers, which are held together mainly by weak van der Waals interaction between adjacent sulfur layers. The sulfur atoms are arranged in closely packed hexagonal places with the Ta atoms situated at the octahedral holes. The Ta atoms constitute a metal-atomic layer of two dimensions. In cooling, $1T$-TaS_2 exhibits three first-order phase transitions, each associated with an appearance or a change in the charge-density wave (CDW). These transitions are, namely, from a metallic phase ($T>543$ K, without the CDW) to an incommensurate CDW (IC) phase, from the hexagonal IC phase ($353 \le T \le 543$ K) to a nearly commensurate CDW (NC) phase, and from the NC phase (either 353–187 K on cooling or 285–353 K on warming) to a commensurate CDW (CC) phase ($T<187$ K). Furthermore, on warming from the CC phase, it produces a triclinic nearly commensurate phase (T phase) at about 230 K and undergoes a transition back to the NC phase at about 285 K. In all the phase transitions, the modulated structure of the CDW changes is either incommensurate to commensurate or vice versa. A change is involved not only in the period of modulation but also in the angle of modulation direction relative to the fundamental structure. Some excellent work on this subject has been done in studying the electronic,[1] magnetic,[2] optical[3] properties, microstructure,[4] and the photoelectron spectrum,[5] but a lot of problems still remain unresolved. Among them, its magnetic behavior at low temperature is particularly important, because it is related to the magnetic interaction between atoms in a quasi-two-dimensional system. In order to study their magnetic properties and the effect of magnetic field on these transitions, the magnetization of $1T$-TaS_2 and $1T$-$Fe_{0.07}Ta_{0.93}S_2$ has been measured in the temperature interval of 1.5–300 K and over the field range of 100 Oe–60 kOe. Experimental results show that both compounds are diamagnetic at high temperatures. It was also demonstrated that the CC-T phase transition temperature is a function of the applied magnetic field. At low temperatures the aforementioned compounds are in a paramagnetic state. The temperature dependence of their magnetization does not follow the Curie law or Curie-Weiss law, but experimental curves can be described fairly well by $M_0+\gamma T^{-n}$. In addition, the atom in a two-dimensional system can be in different states depending on its electronic structure. When an atom is in a singly occupied Anderson-Mott localized state as a result of the disorder and Coulomb-repulsive interaction between the electrons, the atom has a localized spin, or localized moment. The distribution of the moments is stochastic in direction and magnitude. Some of the moments may have antiferromagnetic coupling due to exchange interaction between neighboring moments, while the remaining others are free (uncoupled) moments, the latter's density being proportional to the parameter γ. When a magnetic

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field is applied to the sample at low temperatures, the curve of magnetization versus field shows a complicated functional relationship. On the basis of the experimental evidence, we assume these compounds undergo a phase transition at the maximum value of magnetization. The resulting phase of 1T-TaS_2 and 1T-$Fe_{0.07}Ta_{0.93}S_2$ can be considered as a mixed state of CDW and spin-density wave (SDW) or a SDW state. The magnetic behavior of this phase depends on the low-lying magnetic excitation of the SDW ground state, since the CDW ground state is nonmagnetic.

II. EXPERIMENT

Both 1T-TaS_2 and 1T-$Fe_{0.07}Ta_{0.93}S_2$ crystals were grown by using iodine-vapor transport method. The stoichiometric mixture of highly pure Ta, Fe, and S powders with a small amount of iodine was sealed in a quartz tube under vacuum ($\sim 10^{-3}$ Torr), put in a zone where the temperature gradient was about 5 K/cm, and then allowed to react at 1223 K for about 340 h. Following the reaction, the tube was rapidly cooled in cold water. The golden single crystals were obtained in this way. The 1T-type structure was verified by x-ray and electron diffraction and the composition was determined by chemical analysis. The electrical resistivity of the sample was measured by the standard four-probe method in the absence of any remaining magnetic field. The resistivity-temperature (R-T) curve of 1T-TaS_2 presented sharp steps at about 180 K on cooling and at about 230 and 285 K on warming, which are associated with NC-CC, CC-T, and T-NC transitions,[5] but these variations did not occur on the curves of 1T-$Fe_{0.07}Ta_{0.93}S_2$. At low temperatures the resistivity of both 1T-TaS_2 and 1T-$Fe_{0.07}Ta_{0.93}S_2$ increases rapidly with decreasing temperature. In order to study the magnetic behavior of the compounds, the magnetization was measured in the range of 100 Oe–60 kOe on an extracting sample magnetometer, made by Néel Laboratory in France. The sample used for the magnetic measurement had the shape of a pressure cylinder, where single crystals were stacked and oriented in parallel. During the measurement of magnetization, the magnetic history of the sample was found to be a factor that would influence the experimental result. To circumvent such a magnetic aftereffect, the sample was cooled down to the starting temperature without being exposed to any remnant magnetic field; after which the data of the magnetization were automatically taken with a heating rate of about 2 K/min. Finally, the sample was demagnetized at room temperature for the next cycle. At the end of measurement, all experimental data were corrected in order to make allowances for the sample holder.

III. RESULTS AND DISCUSSION

In order to study the magnetic behavior and phase transition of 1T-TaS_2, its magnetization as a function of both the temperature and the magnetic field has been measured. The curves of the magnetization versus temperature (Fig. 1), being similar to the temperature dependence of susceptibility,[2] show that there is a turning point at temperature T_p. When $T > T_p$, the compound is in a diamagnetic state with its magnetization approximately independent of temperature. However, the magnetization increases rapidly with decreasing temperature in the region of $T < T_p$. The T_p's value is also a function of magnetic field. As regards the M-T curve at 60 kOe, T_p is about 100 K and the T-NC structural transition is revealed by a small jump of the magnetization at about 280 K. Another significant jump occurs at about 222 K, where the CC phase transforms into the triclinic-nearly-commensurate state upon warming. The temperature of the jump depends on the applied magnetic field. For 5, 10, 20, 30, 40, and 60 kOe the corresponding transition takes place (half way through the jump) at about 228, 228, 227, 226, 225, and 222 K, respectively. The influence of magnetic field on the CC-T phase transition is similar to the electron irradiation effect that lowers the IC-NC transition temperature.[6] The CC-T transition is well known as a part of the CC-NC transition. The NC-CC transition had been considered as a Mott transition by Fazekas and Tosatti.[8] Measurements using a photoelectron spectroscope[5] have shown that this transition is a gapless Mott transition. In addition, the CC phase of 1T-TaS_2 consists of small "molecules" with long-range order, where all the "molecules" take the perfect star-shape of 13 unit cells, while the NC phase is on the average incommensurate. The NC phase consists of commensurate hexagonal domains and a discommensurate network. Within a domain the CDW is commensurate with the underlying lattice, and the discommensuration is considered to be line defects or a disturbed region of the CDW where both the phase and amplitude of the CDW change almost discontinuously. When the NC phase makes a transition to the CC state, all discommensurations of the CDW disappear. Investigators working on semiconductors[7] have pointed out that the variation in disorder (impurities and defects) density could induce a metal-insulator transition. As in the case with disorder,

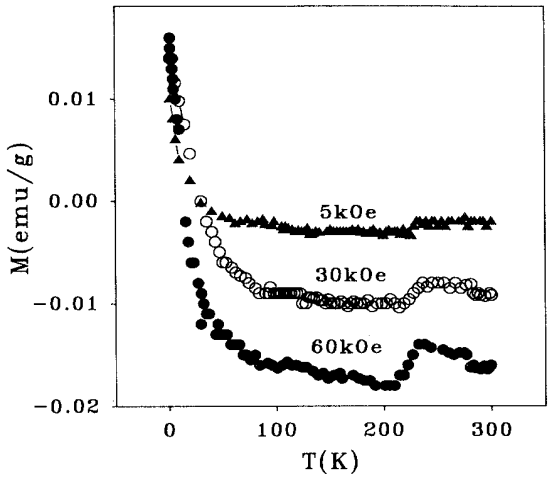

FIG. 1. Temperature dependence of magnetization in 1T-TaS_2.

when a strong magnetic field or a high hydrostatic pressure is applied to a sample, the transition of metal to nonmetal would be induced. On the basis of some well-known calculations, an analogous results has been obtained by Yafet, Keyes, and Adams.[9] For an anisotropic system where the Fermi surface does not completely disappear, Balseiro and Falicov[10] have performed a good theoretical analysis in this respect. They pointed out that the magnetic field can induce a more effective nesting of the Fermi surface, and therefore it is possible to drive the phase-transition temperature of the system increase with increasing magnetic field. However, for a stable phase of density wave, e.g., the CC phase of $1T$-TaS_2, the field enhancement effect of critical temperature would be a small one. On the other hand, when a magnetic field is applied to an electronic system, the electron wave function will localize on a length scale of the order $L_H=(ch/eH)^{1/2}$. It leads to an increase in the local nonhomogeneity of electronic distribution. As the L_H is shorter than the shortest localization length, where the eigenfunction of the atom is localized a transition takes place and the system is in a new localized state. Besides, other localization parameters such as the mobility edge E_c ($\sim H^{1/2}$) and the critical density of disorder would also be changed by the field. In the present case, among all factors that exert influence the effect of the localization is the predominant one capable of breaking up the commensurate structure of the CDW. Similar to the case of defects, the localization effect not only creates and reinforces the discommensurations, but also stabilizes the NC phase toward low temperature. As a result, the external magnetic field lowers the phase-transition temperature of $1T$-TaS_2, despite the fact that both the two-dimensionality of structure and the strong electron-phonon coupling are the driving forces that produce the CDW periodic lattice distortion and the metal-insulator transition.

At high temperatures, the magnetizing curves of $1T$-TaS_2 (Fig. 2) show that the magnetization is a linear function of magnetic field and that it may approximately be described as $M=M_0+\chi H$. The fitting parameters are listed in Table I, where M_0 is a monotonous function of temperature. However, the differential susceptibility χ is a nonlinear function of temperature and its value shows a transition in the interval of 250–160 K. As above, all experimental results show that at high temperatures the main component of the magnetization of $1T$-TaS_2 is a diamagnetic one due to the compound's quasiclosed electronic structure. The Pauli-like paramagnetic component of metallic electrons, which come from those portions of Fermi surface not removed by gap formation, is a smaller one as compared with the diamagnetic component.

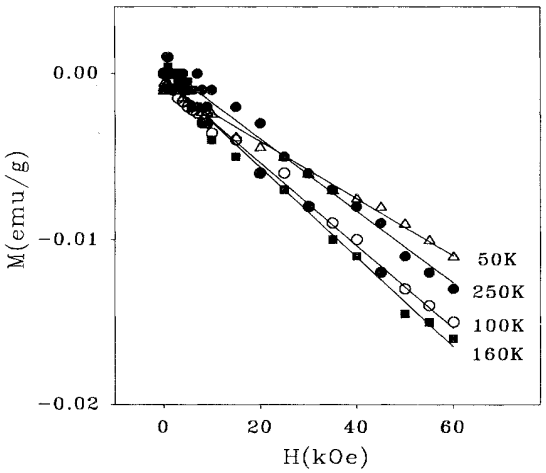

FIG. 2. Magnetic-field dependence of magnetization in $1T$-TaS_2 at high temperatures.

At low temperatures, the physical properties of $1T$-TaS_2 are different from those observed at high temperatures. It is well known that below 60 K the resistivity of $1T$-TaS_2 increases rapidly as the temperature decreases. There are two opinions advanced to explain the origin of this phenomenon. One ascribes it to the occurrence of electron (Anderson) localization in the random potential created by impurities and defects. The other stresses the Mott localization of electron wave function due to electron-electron correlation. In practice, the localization could be considered as an Anderson-Mott one, because the disorders and Coulomb-repulsive interaction between electrons are always existent simultaneously in a real crystal. The divergent resistivity at low temperature is due to the conduction by variable range hopping associated with these localizations. The localization of electrons may also influence the magnetic behavior of $1T$-TaS_2. At low temperatures, it is in a paramagnetic state. Its magnetization increases rapidly with decreasing temperature (Fig. 1), similar to the dependence of its susceptibility on temperature and due to defects, as has been described by DiSalvo and co-workers.[2] This phenomenon bears some resemblance to the temperature dependence of susceptibility of semiconductors Si, Ge, and InSb as well. One may regard this phenomenon occurring in semiconductors as evidence for the presence of localized magnetic moment, or spin localization in these materials, even on the metallic side of a metal-insulator transition.[7,11] The exchange interaction among these localized states could be considered antiferromagnetic.[12] Now, at $T<T_p$, experimental results show that the temperature dependence of magnetization (in a given magnetic field) does not follow the simple Curie law or Curie-Weiss law, but can be described fairly well by

$$M=[\chi_0+\chi(T)]H=M_0+M(T)\ ,\quad M(T)=\gamma T^{-n}\ ,$$

where M_0 is the sum of the Pauli-like paramagnetic and diamagnetic contributions, and $M(T)$ is the contribution of the uncoupled localized moments that originate from the singly occupied Anderson-Mott localized states. For

TABLE I. Fitting parameters of the magnetizing curves.

T (K)	250	160	100	50
$M_0\times10^4$ (emu/g)	4.418	−2.341	−3.571	−6.428
$\chi\times10^7$ (emu/g Oe)	−2.179	−2.715	−2.501	−1.722

the M-T curve at 60 kOe (Fig. 1), the values of parameters obtained using the least-squares fitting of curve are given in Table II. For other experimental curves, their fitting parameters yield values that vary analogously. The difference between n and 1 represents the deviation of the behavior of magnetic moments from the Curie law or Curie-Weiss law. Below 8 K, the magnetic behavior shows a more pronounced departure from the Curie law, indicating that the exchange interaction plays a more important role. As mentioned above, the localized moments do exist in 1T-TaS_2. Mutka and Molinie[13] claimed that their experimental results do not clearly demonstrate the existence of magnetic defects, whereas ours demonstrate beyond any doubt the presence of magnetic moments. Furthermore, the distribution of local magnetic moments is stochastic in direction and magnitude due to the random distribution of the localized center and the short-range nature of the exchange interaction. The distribution also depends on external perturbations that interact with electronic spin. On the basis of the temperature dependence of parameter γ and the nonsaturation behavior of magnetization at low temperatures, we assume that the neighboring Anderson-Mott moments take the form of an antiferromagnetic pair as the exchange interaction between moments becomes sufficiently strong. In 1982, Bhatt and Lee[14] made a more extensive study of the pair model of Andres *et al.* and found that the susceptibility could approximately be described by the T^{-n} law. In the Bhatt-Lee model the only relevant variable that describes the temperature variation is the effective number $N(T)$ of free (unoccupied) moments that survive at temperature T. If the diamagnetic component does not change with temperature and the temperature variation arises only from the free spins, the magnetization $M(T)$ is then expressed when $N(T)$ is implicitly used as

$$M(T)=HN(T)\mu_b{}^2/\kappa_b T\ ,\quad \gamma=HN(T)\mu_b{}^2/\kappa_b T^{1-n}\ .$$

The parameter γ is proportional to the density of free (uncoupled) moments; its decrease is caused by the antiferromagnetic coupling of neighboring moments as the temperature is lowered. Furthermore, we think that a SDW has probably appeared in 1T-TaS_2 with the provision that it is at low temperature and in a strong field. Both $\gamma \ll 1$ and $n \ll 1$ may be regarded as evidence for the presence of a SDW in 1T-TaS_2.

At low temperatures, the magnetizing curves of 1T-TaS_2 show that the magnetization is a nonlinear function of magnetic field (Fig. 3). Although our data on magnetization are in the same order of magnitude as that measured by DiSalvo and co-workers,[2] the nonlinear behavior of the magnetizing curve obtained by us is different from theirs. At 1.5 K, the magnetization is a nonlinear function of magnetic field. It increases with the field at the beginning and gradually approaches a maximum value at about 30 kOe, then the value descends with further increase of the field. At 10 K the maximum of magnetization moves to about 45 kOe (Fig. 3). As mentioned above, the distribution of exchange interaction between moments is stochastic in this compound. When a field is applied to 1T-TaS_2, a part of the coupled moments is decoupled by the field and thermal energies, and the uncoupled magnetic moments tend to align with the external magnetic field. Thus, the magnetization increases with field at the beginning of the M-H curves and approaches its maximum value gradually at about 30–40 kOe. In the same interval of magnetic field, a maximum in the magnetoresistance curve of 1T-TaS_2 has also been observed.[15] As a result, 1T-TaS_2 may undergo a phase transition induced by external magnetic field at the maximum of magnetization. The threshold field of the transition changes with temperature. At 50 K, 1T-TaS_2 is already in the diamagnetic state and the magnetization is a linear function of magnetic field (Fig. 2).

TABLE II. Parameters of M-T curve of 1T-TaS_2 at 60 kOe.

T (K)	1.5–8	9–40	40–100
γ (emu K/g)	0.0378	0.1007	0.1763
n	0.1108	0.6696	0.8279

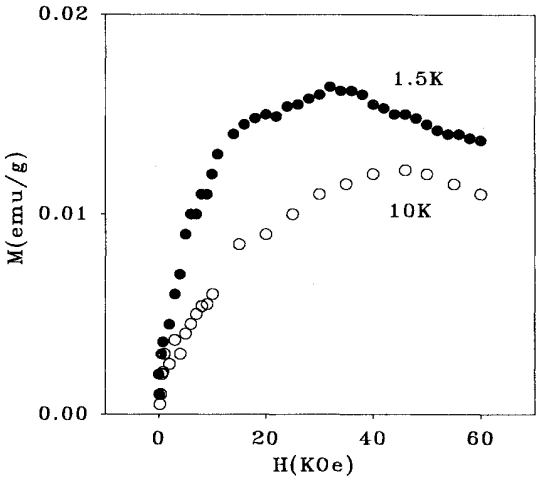

FIG. 3. Magnetization of 1T-TaS_2 as a function of magnetic field shows nonlinear behavior at low temperatures.

In order to further understand more of the magnetic behavior of low-dimensional materials, the single crystal of 1T-$Fe_{0.07}Ta_{0.93}S_2$ was scrupulously prepared by the iodine-vapor transport method and its magnetization carefully measured with the extracting sample magnetometer. Experimental curves show that its magnetic properties are very similar to those of 1T-TaS_2. At high temperatures, the curves of magnetization versus temperature of 1T-$Fe_{0.07}Ta_{0.93}S_2$ (Fig. 4) show that the compound is in a diamagnetic state and the magnetization does not vary very much with temperature or field. Moreover, no jumps in the temperature curve of resistivity and magnetization were observed. Consequently, the Fe atom is probably in low spin state ($S=0$) and the NC-CC or CC-T phase transition may have been completely suppressed. Thus 1T-$Fe_{0.07}Ta_{0.93}S_2$ may be in a metallic state when $T > 70$ K. In this respect, our result is different from that of Eibschutz and DiSalvo.[16] They reported that 1T-$Fe_xTa_{1-x}S_2$ had unusual magnetic

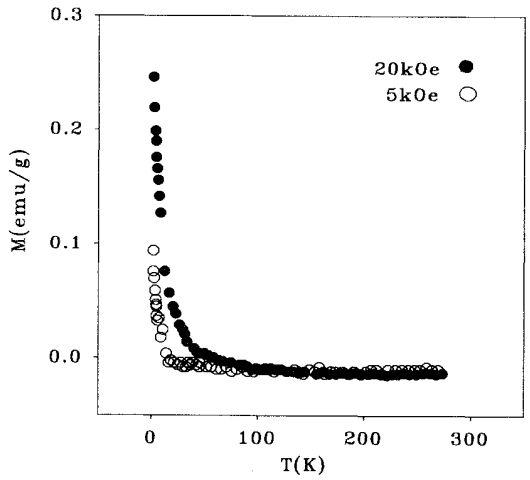

FIG. 4. Temperature dependence of magnetization in $1T$-$Fe_{0.07}Ta_{0.93}S_2$.

behavior and was completely unlike any of the pure, substituted, or intercalated layered compounds. At low temperatures, the magnetization of $1T$-$Fe_{0.07}Ta_{0.93}S_2$ is about 10 times larger than that observed in $1T$-TaS_2. It thus implied that the Fe atom is probably in high spin state and that the temperature of spin flip is approximately equal to that of the turning point of magnetization, that is, T_p. The temperature dependence of magnetization (Fig. 4) can also be fitted by $M_0+\gamma T^{-n}$. Good fitting to the data curve at 20 kOe has been obtained and the fitting parameters are listed in Table III. In the temperature range of 31–70 K, the M-T curve shows a stronger temperature dependence of magnetization than that observed at high temperatures. It probably originates in the localized moments on the metallic-state side near the metal-insulator transition. In the interval of 13–35 K, the magnetization follows approximately the Curie law, which means the contribution of the Pauli-like component has almost disappeared. At $T < 12$ K, on the basis of experimental results of resistivity, we assume that the compound is already in an insulating state. The magnetization increases rapidly with the decrease of temperature, while both parameters γ and n decrease quickly with temperature. The origin of this phenomenon could be traced back to the strengthening of exchange interaction between the neighboring spins and the increase of antiferromagnetic coupling. Moreover, a noticeable reduction in γ appears visible at about 30 K, which implies some changes in its band structure have probably taken place, but all experimental curves are smooth. As a result, $1T$-$Fe_{0.07}Ta_{0.93}S_2$ is like a semiconductor; its magnetization is changing smoothly during the metal-insulator transition. Besides, the magnetizing curves of $1T$-$Fe_{0.07}Ta_{0.93}S_2$ are similar to that of $1T$-TaS_2. At 100 K, $1T$-$Fe_{0.07}Ta_{0.93}S_2$ is in a diamagnetic state and the magnetization may approximately be considered as a linear function of magnetic field (Fig. 5). The differential susceptibility is about -2.91×10^{-6} emu/g Oe. At 1.5 K, this compound is paramagnetic (Fig. 5). In a weak field the magnetization increases with the field, reaching a maximum value at about 500 Oe. With further increase of the field the magnetization of $1T$-$Fe_{0.07}Ta_{0.93}S_2$ decreases and goes down to a minimum value at about 1.2 kOe, after which it increases again with the field and then gradually approaches saturation. As described above, the magnetization is a nonlinear function of magnetic field. In the interval of 500 Oe–60 kOe, it is similar to the magnetic-field dependence of chromium's susceptibility.[17] It is well known that Cr is a typical material of the SDW phase.

TABLE III. Fitting parameters of M-T curve of $1T$-$Fe_{0.07}Ta_{0.93}S_2$ at 20 kOe.

T (K)	1.5–12	13–30	31–70
γ (emu K/g)	0.3557	1.3435	8.9245
n	0.4365	1.0711	1.6778

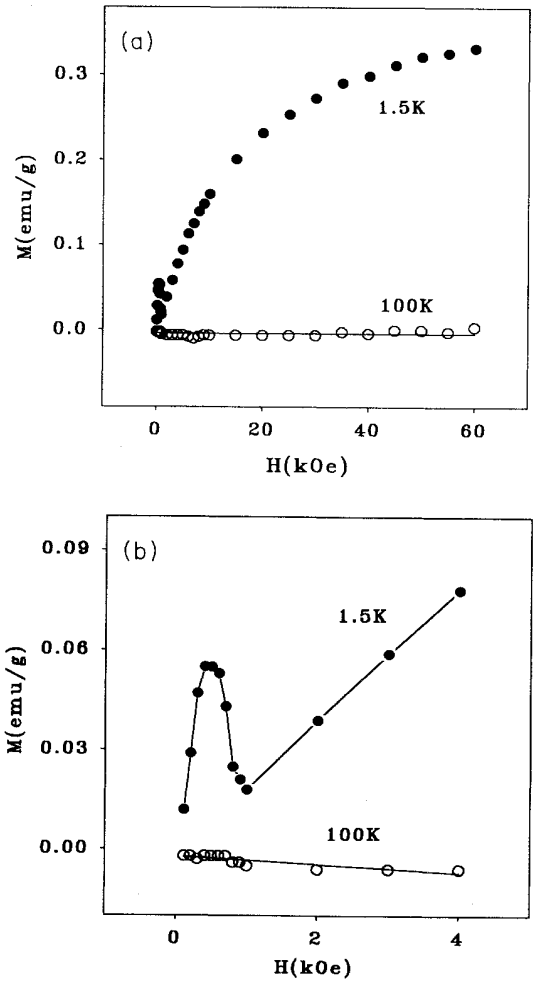

FIG. 5. Magnetization of $1T$-$Fe_{0.07}Ta_{0.93}S_2$ as a function of magnetic field exhibits a complicated behavior at low temperatures, while its magnetization is a linear function of the field at high temperatures. (a) In the magnetic field interval of 100 Oe–60 kOe. (b) In the magnetic field interval of 100 Oe–4 kOe.

Theoretical investigations of a two-dimensional one-electron system[18] showed that the electron-phonon coupling and interaction between electrons could lead to a mixed CDW-SDW ground state and the Q vector of the SDW would change with magnetic field. On the other hand, for the ground state of a two-dimensional magnetic system there are four possibilities: paramagnetic, ferromagnetic, antiferromagnetic, and a SDW state. Now, the magnetic behavior of both 1T-TaS_2 and 1T-$Fe_{0.07}Ta_{0.93}S_2$ does not follow either the Curie law or Curie-Weiss law, and neither of them shows any Curie or Néel transition. Consequently, a phase transition may take place at the maximum value of the magnetization of 1T-TaS_2 and 1T-$Fe_{0.07}Ta_{0.93}S_2$. The resulting phase may be a mixed state of SDW-CDW or a SDW state due to the coherent superposition of antiferromagnetic coupling. The magnetic behavior of these compounds then is associated with the low-lying magnetic excitation of the SDW ground state, because the CDW ground state is nonmagnetic. Furthermore, the experimental results show that Fe atoms suppress the CDW and stabilize the SDW phase of 1T-TaS_2. In these respects, 1T-TaS_2 and 1T-$Fe_{0.07}Ta_{0.93}S_2$ are probably similar to other low-dimensional materials.[19,20] The ground state of such low-dimensional compounds is in the mixed state of SDW and CDW or the single SDW state at low temperatures, and their behavior was strongly influenced by magnetic impurities.

IV. CONCLUSION

In summary, experimental studies have demonstrated that, at high temperatures, 1T-TaS_2 is in a diamagnetic state and its magnetization is a linear function of the magnetic field. Under the influence of external magnetic field, the occurring temperature of the CC-T phase transition shifts to lower temperature. At low temperatures 1T-TaS_2 and 1T-$Fe_{0.07}Ta_{0.93}S_2$ are in a paramagnetic state. The origin of the paramagnetism is the localized spins, or localized moments, which come from the singly occupied Anderson-Mott localized states. The temperature dependence of magnetization does not follow Curie law or Curie-Weiss law, but it may adequately be described by $M = M_0 + \gamma T^{-n}$, where parameter γ is proportional to the density of free moments. The difference between the parameter n and 1 represents the fact that the behavior of uncoupled moments deviates from that given according to Curie law or Curie-Weiss law. On the other hand, the magnetic-field dependence of magnetization displays some complicated features at low temperatures. We assume that a phase transition may take place at the maximum value of the magnetization, and then the compounds are probably in a mixed state of SDW and CDW or a SDW state. The threshold field of the phase transition is a function of temperature and impurities.

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