

Journal of Alloys and Compounds 384 (2004) 33-38

Journal of ALLOYS AND COMPOUNDS

www.elsevier.com/locate/jallcom

Influence of the Mn intercalation on magnetic properties of TiSe₂

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Received 12 March 2004; received in revised form 19 March 2004; accepted 25 March 2004

Abstract

Using the measurements of the magnetic susceptibility and magnetization we have investigated the magnetic properties of Mn-intercalated $Mn_x TiSe_2$ compounds having a hexagonal crystal structure of a quasi-two-dimensional type. Unlike other 3d-transition metals, intercalation of Mn ions between Se–Ti–Se tri-layers increases the *c*-parameter of the lattice. All compounds of $Mn_x TiSe_2$ exhibit a paramagnetic behavior above ~15 K. The effective magnetic moment μ_{eff} of Mn in $Mn_x TiSe_2$ is found to be less than μ_{eff} for the free Mn^{2+} ion. In a low-temperature region a spin-glass or cluster-glass behavior depending on the intercalant concentration is observed. The paramagnetic Curie temperature θ_p takes negative values and decreases monotonically with increasing *x*, while the freezing temperature shows non-monotonous change at the intercalation. The obtained results are discussed taking into account Mn 3d–Ti 3d hybridization effects and variations of intralayer and interlayer exchange interactions with intercalation.

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Keywords: Titanium dichalcogenides; Intercalated compounds; 3d-Transition metals; Itinerant magnetism; Magnetic susceptibility; Spin-glass

1. Introduction

The intercalated compounds based on layered titanium dichalcogenides TiX₂ (X = S, Se, Te) are attractive objects during two last decades owing to the rich variety of the physical properties which may be produced in these materials upon insertion of different atoms or molecules between X–Ti–X layers [1-6]. In particular, the TiX₂ compounds intercalated by 3d-transition (M) metals can be considered as an analog of artificial multi-layer structures with magnetic layers consisting of M-atoms, which are separated by non-magnetic layers. The intercalation of some 3d-metals in $M_{x}TiX_{2}$ leads to appearance of the different magnetic states depending on the type and concentration of M-atoms as well as on the parent TiX_2 matrix. While the compounds TiX_2 are Pauli paramagnets, the intercalated compounds M_xTiX₂ exhibit a spin-glass or cluster-glass behavior at the low concentration of M-atoms and antiferromagnetic or ferromagnetic order at the high M-content [4,5,7]. In particular, the compounds $Fe_x TiSe_2$ (x = 0.25, 0.33, 0.5) have an antiferromagnetic order below corresponding Néel temperatures of 62, 95 and 129 K [5]. The antiferromagnet-like behavior was found to exist in $Cr_{0.5}TiSe_2$ below $T_N = 38 K$ [7].

Distinctive feature of the intercalated compounds M_xTiX₂ is the appreciable change of the lattice parameter c and consequently the distance between X-Ti-X layers with increasing M-content. For the M_x TiSe₂ series the intercalation of Cr, Fe or Co up to $x \approx 0.25$ leads to contraction of the lattice in the *c*-direction, which is ascribed to the formation of covalent links between X-Ti-X layers via intercalated M-ions [2,6,8]. The 3d-electrons of the intercalated M-ions are presumably involved in these links and hybridized with Ti 3d-electrons and p-electrons of chalcogen atoms. Further increase of the M-concentration up to x = 0.5 is accompanied by the growth of the *c*-parameter in the case of M =Cr, while the *c*-parameter remains constant for M = Fe and decreases more slowly at the intercalation of Co. Such a behavior of c(x) at x > 0.25 was attributed to the saturation of the covalent links in these intercalated compounds [8]. Another feature of M_x TiSe₂ compounds is a lower value of the effective magnetic moment μ_{eff} of the intercalated M-ion in comparison with the free 3d-transition metal ion, which may be also associated with the contribution of 3d-electrons of intercalated ions to the bonding of Se-Ti-Se layers and with

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hybridization effects of 3d-electrons of M-ions with TiSe₂ bands [8,9].

According to preliminary investigations of $Mn_x TiSe_2$ compounds, unlike other 3d-transition metals the intercalation of Mn-ions increases the *c*-parameter and consequently the distance between Se–Ti–Se layers [2]. One can therefore assume that $Mn_x TiSe_2$ should reveal a higher degree of two-dimensionality (2D) than $M_x TiSe_2$ compounds intercalated by other 3d-transition metals.

The aim of the present paper is to study the influence of Mn intercalation on the magnetic properties of the titanium diselenide by means of magnetic susceptibility and magnetization measurements of the Mn_xTiSe_2 compounds with the different Mn-content.

2. Experimental

Polycrystalline $Mn_x TiSe_2$ samples ($0 < x \le 0.5$) were prepared by an ampoule synthesis method from the constituent elements. The starting materials are Ti (99.9), Se (99.99) and Mn (99.9). The method of the synthesis includes two stages. At first, the parent compound TiSe₂ was synthesized by heat treatment of a mixture of starting materials Ti and Se at 800 °C during 150 h. At the second stage the mixtures of Mn and TiSe₂ powders were sealed in evacuated quartz tubes and annealed under the same conditions. The obtained specimens were milled, compacted into pellets and then homogenized for 1 week at 800 °C followed by cooling down to room temperature during about 10 min. Powder X-ray diffraction investigations have shown that all samples have the CdI₂ structure. Lattice parameters were measured by X-ray diffraction with an error of 3×10^{-3} Å.

The static magnetization and magnetic susceptibility were measured by means of a Quantum Design SQUID magnetometer in the temperature interval from 2 up to 300 K in a magnetic field up to 50 kOe.

3. Results and discussion

As it seen from Fig. 1, the compounds $Mn_x TiSe_2$ with the Mn-content up to x = 0.5 exhibit a paramagnetic behavior with a nonlinear temperature dependence of the inverse susceptibility at the temperatures above ~15 K. The phase transition to the superlattice with the charge density wave, which was observed in the parent TiSe₂ [10] with decreasing temperature below ~200 K, is not observed in the Mn-intercalated compounds even at low *x*. This finding is in agreement with other investigations [6,8,11], which show that the intercalation of d-metals suppresses the superlattice formation in TiSe₂. The temperature dependencies of the magnetic susceptibility of $Mn_x TiSe_2$ compounds in a wide temperature range can be described well by the expression

$$\chi = \chi_0 + \frac{C}{T - \theta_p}.$$
 (1)



Fig. 1. Temperature dependencies of the inverse magnetic susceptibility of $Mn_x TiSe_2$ at various Mn content. Inset: $(\chi - \chi_0)^{-1}$ vs. *T* dependence for the sample with x = 0.1.

Here χ_0 is a temperature-independent term, which includes both the diamagnetic contribution from the ions with full-filled electron shells (χ_d) and paramagnetic contribution from the delocalized electrons (χ_p). As it follows from estimations in Ref. [7], the diamagnetic contribution to the total magnetic susceptibility of M_x TiSe₂ should not change significantly upon intercalation. Therefore, the change of χ_0 may be associated mainly with the concentration dependence of the Pauli contribution χ_p . The second term in Eq. (1) relates to the Curie-Weiss contribution. The inset in Fig. 1 shows the $1/(\chi - \chi_0)$ versus T dependence for Mn_{0.1}TiSe₂ as an example. Such linear dependencies we have obtained for the compounds with other Mn-content. However, the magnetic susceptibility of Mn_{0.5}TiSe₂ was found to follow the expression (1) only above 80 K. One can assume that the deviation of $\chi(T)$ from Eq. (1) with decreasing temperature below 80 K results from the influence of the magnetic short-range Mn-Mn correlations. Using Eq. (1), the parameters χ_0 , C and θ_p were determined for each compound. The effective magnetic moment μ_{eff} per Mn ion was calculated from the Curie constant C. All these data are collected in Table 1.

As it turned out, the intercalation of Mn increases the temperature-independent term χ_0 . Our data for Mn_{0.1}TiSe₂ ($\chi_0 = 1.5 \times 10^{-6} \text{ emu g}^{-1} \text{ Oe}^{-1}$) are something less than that obtained by Tazuke and Takeyama

Table 1

The temperature-independent contribution to the magnetic susceptibility (χ_0), effective magnetic moment of the Mn-ion (μ_{eff}), paramagnetic Curie temperature (θ_p) and freezing temperature (T_f) for Mn_xTiSe₂ compounds

x	$\chi_0 ~(\times 10^{-6} \text{emu} \text{g}^{-1} \text{Oe}^{-1})$	$\mu_{\rm eff}~(\mu_{\rm B})$	$\theta_{\rm p}$ (K)	$T_{\rm f}$ (K)
0.1	1.50	3.6	-2	~ 2
0.17	1.47	4.3	-12	7.0
0.25	1.80	4.4	-30	10.0
0.5	3.20	4.3	-105	5.0

 $(\sim 2 \times 10^{-6} \text{ emu g}^{-1} \text{ Oe}^{-1}$ [6]). Nevertheless, the value of χ_0 for Mn_{0.1}TiSe₂ obtained in the present work exceeds χ_0 for other M_{0.1}TiSe₂ (M = V, Cr, Fe, Co, Ni) [6], which supports qualitatively the data obtained in Ref. [8]. The enhancement of χ_0 with increasing Mn-content in the Mn_xTiSe_2 system we attribute to the growth of the density of electronic states (DOS) at the Fermi level. This assumption is confirmed by X-ray photoelectron spectroscopy, which shows the appearance of a narrow band near the Fermi level in the intercalated compounds with M = Cr, Co [12] in comparison with the parent compound TiSe₂. Such a band is formed by 3d orbitals of inserted M-atoms, which are hybridized with Ti 3d and Se 4p orbitals. It should be noted that the calculations of the energy band structure and the photoemission spectroscopy of analogous systems M_x TiS₂ have revealed a strong hybridization of M 3d-states with Ti 3d- and S 3p-states [9,12].

At the first gaze, the conclusion about an increase of the DOS value at the Fermi level at the intercalation of TiSe₂ by 3d-transition metals contradicts with experimental data on the conductivity of M_x TiSe₂ (where M = Cr, Fe, Co), which show a drastic fall of σ (about of one order [8]) with increasing M-content especially in a low concentration range [8], whereas the additional s- and d-electrons are put into the matrix with inserted M-atoms. As is shown in Ref. [8], these results can be understood if the localization of charge carriers on the Ti-M-Ti covalent centers will be taken into consideration. Such a localization of charge carriers may occur on potential wells formed by the lattice contraction in the vicinity of intercalants. According to Ref. [2], the conductivity of Mn_xTiSe₂ measured at room temperature shows an opposite behavior with the intercalant concentration: the σ value increases about twice with increasing x up to x = 0.2and then decreases with further growth of the Mn content. An upturn of σ in the low concentration range is not surprising in this case bearing in mind that the intercalation of Mn increases the c parameter (see Fig. 2) and the concentration of charge carriers. The charge carriers are apparently less localized in Mn_xTiSe_2 than in other M_xTiSe_2 compounds (M = Cr, Fe, Co). It should be noted that the temperature dependence of the electrical conductivity of Mn_xTiSe_2 shows a metallic character [2]. The metallic type of $\sigma(T)$ dependencies was also observed for many other M_xTiSe₂ compounds intercalated by 3d-transition metals [8].

The values of μ_{eff} per Mn-ion in Mn_xTiSe₂ compounds are found to be lower than $\mu_{\text{eff}} = 2\mu_{\text{B}}[S(S+1)]^{1/2} =$ $5.9\,\mu_{\text{B}}$ for the free Mn²⁺ ion. As is seen from Fig. 2, μ_{eff} increases from ~3.6 μ_{B} for x = 0.1 up to 4.3 μ_{B} for x =0.17 and then μ_{eff} remains practically constant with further increasing x. As it was mentioned above, the lower value of μ_{eff} per intercalated 3d-metal ion in comparison with μ_{eff} for the free 3d-metal ion is a common feature of M_xTiX₂ compounds. According to the band structure calculations such a difference may originate from hybridization effects between 3d-electronic states of inserted M-ions and 3d-states of Ti [6]. In magnetically ordered state the hy-



Fig. 2. Concentration dependencies of the effective magnetic moment μ_{eff} per M-ion (a) and the lattice parameter *c* (b) for M_xTiSe₂ systems (M = Mn and Cr).

bridized M 3d-band should be split by an exchange interaction. Ab initio calculation of the magnetic moment per Mn in the compound $Mn_{1/3}TiSe_2$ has given the value of $3.35 \mu_B$ only [9]. Moreover, the high-field magnetization measurements performed at low temperatures for some intercalated compounds $Cr_{0.5}TiSe_2$ [7], Cr_xTiTe_2 [13] and M_xTiS_2 (M = Fe, Mn, Co, Ni [14]) have shown that the magnetic moment per intercalated guest atom in saturation, μ_S , is appreciably lower than that expected from the localized electron model. These data indicate the itinerant character of the M 3d-electrons. According to the spin-fluctuation theory for itinerant electron magnets [15], the Curie–Weiss contribution to the total paramagnetic susceptibility is associated with the temperature dependence of the amplitude of the local spin density [15].

In Fig. 2 we also plotted the concentration dependences of both μ_{eff} and *c* for Cr_xTiSe₂ (the data are taken from Ref. [2]). The comparison of these results with that obtained in the present work for Mn_xTiSe₂ shows that in both these systems a pronounced correlation between $\mu_{eff}(x)$ and c(x) exists. In our opinion, such a correlation indicates that the value of μ_{eff} in M_xTiSe₂ compounds is controlled by the hybridization degree of the M 3d- and Ti 3d-states indeed. The growth of the Pauli contribution to the total magnetic susceptibility and increase of the distance between the Se–Ti–Se layers together with the growth of the conductivity with increasing *x* in Mn_xTiSe₂ may be also indicative of a lower Mn 3d–Ti 3d hybridization degree than in the case of the intercalation by other 3d-transition metals.

As it follows from Table 1, the paramagnetic Curie temperature θ_p is negative for all Mn_xTiSe₂ compounds and the absolute value of θ_p increases with increasing Mn content, which may be associated with enhancement of the exchange interaction when the distance between Mn atoms reduces. As to the origin of the exchange interaction one can suppose that at a low Mn concentration the exchange interaction between 3d-electrons of Mn atoms situated within one plane may be mediated by conduction electrons, i.e. this exchange interaction is apparently of the RKKY type. Here we take into account that these compounds reveal a metallic type of the conductivity, while the Mn 3d-electrons remain guite localized because of the low hybridization degree. The exchange interaction between Mn atoms located in the neighbor planes may be considered as a superexchange type, which occurs presumably through the intervening Se atoms because of the large Mn-Mn distance and significantly lower conductivity along *c*-axis. A huge anisotropy of the conductivity was revealed by the measurements of single crystalline samples of the TiX₂-based compounds (see Ref. [14] for example). One may suggest that the sign of the coupling of the M-magnetic moments in the *c*-direction will depend on the hybridization degree and on the Ti-M-Ti and X-Ti-X distances too. The maximal energy of the M-M coupling via superexchange interaction is expected when the M-atoms are situated in neighbor octahedral sites along the c-direction. Such an alignment of M-atoms will be dominant at x > 0.5. Since the value of θ_p is associated with an average of the sum of exchange interactions acting in the magnetic system we can conclude that the antiferromagnetic exchange dominates in Mn_xTiSe_2 . However, we should note that in some $M_x TiX_2$ systems the θ_p value was found to change its sign at the intercalation. Thus, the concentration dependence of $\theta_{\rm p}$ in Cr_xTiSe₂ shows non-monotonous variation at the intercalation: θ_p is negative at x = 0.25, while it becomes positive at x > 0.25 [8,9]. A sign-varying behavior of $\theta_p(x)$ was also observed for $Fe_x TiSe_2$ [8].

Fig. 3 shows the temperature dependencies of the susceptibility of $Mn_x TiSe_2$ measured in a low-temperature region on a sample cooled in zero field (ZFC) or on a sample cooled in an applied field (FC). An essential difference between ZFC and FC curves is observed for the compounds with x > x0.1, which may be indicative of a spin-glass or cluster-glass behavior below the freezing temperature, $T_{\rm f}$. The value of $T_{\rm f}$ for each sample we determined as the temperature that corresponds to the position of the maximum on the temperature dependencies of the ZFC susceptibility. A small hysteresis below 3 K may be also seen on the $\chi(T)$ dependencies for Mn_{0.1}TiSe₂, which implies a low spin freezing temperature ($T_{\rm f} \sim 2 \,\rm K$) in this compound. The FC susceptibility has the same value as the ZFC susceptibility above $T_{\rm f}$, while for the sample with x = 0.17 it shows a nearly constant value below $T_{\rm f}$. These behavior is characteristic of spin-glass found in various systems [4]. In the samples with x = 0.25 and x = 0.5 the ZFC susceptibility exhibits a broad peak, while the FC one increases below $T_{\rm f}$ with decreasing temperature. Such a behavior one observes in cluster-glass phases. As it is seen from Fig. 4, an increase of an applied field up to 1000 Oe does not strongly influence the shape of $\chi(T)$ dependencies. The inset in Fig. 3 displays the concentration dependence of the freezing temperature obtained



Fig. 3. Temperature dependencies of the ZFC (solid symbols) and FC (open symbols) magnetic susceptibility for Mn_xTiSe_2 at $\mu_0H = 200$ Oe. Inset: concentration dependence of the freezing temperature $T_f(x)$.

from $\chi(T)$ curves. The $T_{\rm f}$ value increases with increasing x up to x = 0.25, while further growth of the Mn content reduces the freezing temperature. Bearing in mind that the absolute value of the paramagnetic Curie temperature $\theta_{\rm p}$



Fig. 4. Temperature dependencies of the ZFC (solid symbols) and FC (open symbols) magnetic susceptibility χ for the sample with x = 0.5 at various magnetic fields.



Fig. 5. Magnetization curves measured at T = 30 K (a) and at T = 2 K (b) on Mn_xTiSe₂ compounds with different Mn content.

increases monotonously with increasing Mn content such a non-monotonous change of $T_{\rm f}$ is not understood yet. The spin-glass or cluster-glass state in these compounds results apparently from the oscillating character of the RKKY interaction and the possible competition between the RKKY-type exchange (in the Mn layer) and superexchange interaction (between Mn layers). One can suggest that owing to the high degree of two-dimensionality of Mn_xTiSe_2 the spin-glass or cluster-glass state has mainly 2D character at a low Mn concentration (x < 0.5), while the magnetic ordering becomes three-dimensional at a higher intercalant content. The change of the dimensionality $(2D \rightarrow 3D)$ of the magnetic ordering with increasing x may presumably influence the freezing temperature owing to the possible difference in the relaxation rate of the magnetization of 2D and 3D clusters in cluster-glass state. It should be noted that our suggestion about the 2D nature of the magnetic ordering in Mn_xTiSe₂ at the low Mn concentration is supported by Monte Carlo simulations of a cluster-glass state performed for Fe_xTiS_2 compounds (x = 1/4 and 1/3) in a simple site-percolation model [4].

The magnetization curves of $Mn_x TiSe_2$ measured at T = 30 K (above freezing temperatures) and at T = 2 K (below T_f) are presented in Fig. 5. In the paramagnetic region (T = 30 K) the M(H) dependencies are nearly linear in magnetic fields up to 10 kOe. The slope of the magnetization curves increases gradually with increasing Mn content, while such a graduality is not observed at 2 K. As can be seen from



Fig. 6. The full-cycle field dependencies of the magnetization for the samples with x = 0.25 (a) and x = 0.5 (b) at T = 2 K. Insets show the *M* vs. *H* behavior in a low-field region in detail.

Fig. 5b, the magnetization measured at 2 K does not show saturation with increasing field up to 50 kOe. In a low-field region the compound $Mn_{0.1}$ TiSe₂ exhibits the higher magnetic susceptibility than the samples with x = 0.17 and 0.25. Such a behavior may be attributed to the difference in the measuring temperature (2 K) in relation to spin-freezing temperatures of these compounds. The measuring temperature for the sample with x = 0.1 is close to $T_f \sim 2$ K, while it is significantly lower than T_f for the compounds with x = 0.17 and x = 0.25 (7 and 10 K respectively).

The full-cycle field dependencies of the magnetization at T = 2 K for the samples with x = 0.17, 0.25, 0.5 is found to reveal a visible hysteresis in a low-field region. Fig. 6 displays hysteresis loops for $Mn_x TiSe_2$ with x = 0.25and 0.5. The insets in Fig. 6 show M versus H behavior in detail within the field range $-1000 \,\text{Oe} < H < 1000 \,\text{Oe}$. For Mn_{0.25}TiSe₂ a step-like behavior of the magnetization is observed near a coercive field $H_{\rm c} \sim 500$ Oe, which may be associated with the coherent magnetization reversal of groups of Mn-clusters having the ferromagnetic short-range order. An analogous M(H) dependence was observed for the compound $Mn_{0.17}$ TiSe₂. As it seen from the inset in Fig. 6b, the compound $Mn_{0.5}$ TiSe₂ exhibits nearly the same value of H_c (~500 Oe), while its magnetization does not reveal any jumps in the vicinity of H_c and changes gradually with variation of a magnetic field. Such a change of the shape of the hysteresis loop with increasing Mn-content may

be attributed to the above-discussed changes of the ratio between intralayer and interlayer exchange interactions.

4. Conclusion

The intercalation of TiSe₂ by manganese atoms results in the monotonous increase of the c-parameter of the hexagonal CdI₂-type lattice, unlike the intercalation of other 3d-transition metal. An increased distance between Se-Ti-Se tri-layers suggests a lower degree of the hybridization of Mn 3d-electrons with Ti 3d and Se p electrons and higher degree of two-dimensionality in comparison with M_xTiSe₂ compounds intercalated by other 3d-transition (M) metal. According to study of the magnetic susceptibility performed in the present work, the Mn_xTiSe₂ compounds are paramagnets above $\sim 15 \text{ K}$ with nonlinear temperature dependence of the inverse susceptibility. The temperature-independent contribution χ_0 to the total magnetic susceptibility increases monotonously with increasing Mn-content, which may be indicative of enhancement of the DOS value at the Fermi level in these compounds as in other M_x TiSe₂.

The effective magnetic moment per inserted Mn-ion is found to be lower than that expected for the Mn²⁺ free ion (5.9 μ_B). The value of μ_{eff} increases from 3.6 μ_B for x =0.1 up to 4.3 μ_B in saturation above x = 0.17. The change of μ_{eff} with intercalation in Mn_xTiSe₂ correlates with the concentration dependence of the lattice parameter *c* as well as in other M_xTiSe₂. These results allow us to conclude that the Mn 3d-electrons in Mn_xTiSe₂ have an itinerant character and that the value of μ_{eff} in M_xTiSe₂ systems is controlled by the Mn 3d–Ti 3d hybridization degree.

The temperature and field dependencies of the magnetic susceptibility and magnetization of $Mn_x TiSe_2$ at $0.17 \le x \le 0.5$ are found to be typical for spin-glass and cluster-glass state below the freezing temperatures T_f . These magnetic states may result from oscillating character of the RKKY-type exchange interaction that dominates in the Mn-layer and from interlayer interaction of the superexchange type. The non-monotonous change of the freezing temperature with increasing Mn concentration we attribute to the possible change of the dimensionality $(2D \rightarrow 3D)$ of the magnetic ordering resulting from the competition between intralayer and interlayer exchange interaction.

Acknowledgements

We thank Dr. A.N. Titov for helpful discussions. This work was supported by the Program "Universities of Russia".

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