Review

Growth and magnetic properties of novel ferromagnetic semiconductor (Zn, Cr)Te

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Abstract

Magnetic properties of a novel ferromagnetic semiconductor (Zn, Cr)Te were investigated. Zn$_{1-x}$Cr$_x$Te thin films, both without and with the additional hole doping by nitrogen, were grown by molecular beam epitaxy. In the magnetization measurement on Zn$_{1-x}$Cr$_x$Te without carrier doping, the ferromagnetic behaviors such as a hysteresis loop in the magnetization vs. magnetic field curve were observed. Similar hysteretic behaviors in the field dependence were reproduced in the magnetic circular dichroism measurement. The ferromagnetic transition temperature $T_C$ deduced from Arrott plot increased almost linearly with Cr composition with the maximum $T_C = 275$ K at a Cr composition of $x = 0.17$. The ferromagnetic behaviors observed in the undoped samples were found to be suppressed upon the $p$-type doping with nitrogen. These experimental findings are discussed based on the double exchange mechanism and the suppression of ferromagnetism by the hole doping is interpreted as due to the shift of the Fermi level in the Cr 3$d$ level with the acceptor doping.

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Keywords: Magnetic semiconductors; Spintronics; Molecular beam epitaxy; II–VI Compounds; Ferromagnetism; Magnetic circular dichroism; Carrier doping; Double exchange interaction

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1. Introduction

Today there is a growing interest for the realization of new technologies utilizing the spin degree of freedom in semiconductor devices. This new frontier of the electronics is called ‘spintronics’ [1]. Diluted magnetic semiconductors (DMSs), semiconductors containing magnetic elements (transition metals or rare earths) in the substitutional site, are expected to be key materials for controlling the electron spins in semiconductor devices. More favorable are DMSs that exhibit...
the ferromagnetism in order to realize the spin-polarized electron source. Since the discovery of the ferromagnetism in Mn-doped III–V semiconductors [2], the search for novel semiconductors exhibiting the ferromagnetism above room temperature has been stimulated. Indeed, DMSs consisting of various combinations of host semiconductors and magnetic elements have been studied so far, and some of them have been reported to become ferromagnetic above room temperature [3,4]. However, it has been still controversial whether these materials are truly ferromagnetic semiconductors or not; that is, whether the ferromagnetism observed is an intrinsic property of the DMS studied, or it originates from precipitations of other ferromagnetic compounds, which was unintentionally synthesized.

Among numerous candidates of ferromagnetic semiconductors, we have chosen (Zn, Cr)Te as a target of the experimental study. Cr-doped II–VI compounds attracted attention due to the sp–d exchange interaction which is different from the Mn-doped counterpart [5], and further the ferromagnetic d–d interaction was theoretically predicted [6]. Later, ferromagnetic behaviors were observed in Zn₁₋ₓCrₓTe [7] and Zn₁₋ₓCrₓSe [8] with Cr contents x reaching a few percent, and then the room-temperature ferromagnetism was reported in Zn₁₋ₓCrₓTe with x=0.20 [9]. However, the mechanism responsible for the ferromagnetism in these Cr-doped II–VI’s has not yet been clarified. The ferromagnetism in Mn-doped III–V’s is understood as due to the carrier-induced mechanism; the ferromagnetic interaction between Mn spins is mediated by holes supplied from Mn²⁺ acting as acceptors in the substitutional site of cation atoms. On the other hand, it is considered that Cr-doped II–VI compounds are insulating with Cr²⁺ in the cation site. Therefore another mechanism has to be examined as an origin of the ferromagnetism in (II, Cr)VI.

In the present article, we report our recent experimental studies on (Zn, Cr)Te [10–13]. We have investigated the detailed magnetic properties of (Zn, Cr)Te thin films grown by molecular beam epitaxy (MBE), with the intention to elucidate the physical origin of the ferromagnetic interaction. In this study, special emphasis is laid on (1) detailed magnetic properties of (Zn, Cr)Te without carrier doping, particularly with the systematic analysis of the dependence on Cr composition [10–12], and (2) the effect of the hole doping on the ferromagnetism [13]. We performed various procedures of magnetization and magneto-optical measurements on undoped and hole-doped (Zn, Cr)Te with nitrogen as an acceptor. The experimental findings are discussed based on the double exchange mechanism as an origin of the ferromagnetic interaction.

2. Experimental procedures and results

2.1. MBE growth

The growth of (Zn, Cr)Te was performed by conventional MBE using solid sources of Zn, Te, and Cr. A piece of GaAs (100) wafer was used as a substrate. Firstly a thick buffer layer of ZnTe (≈700 nm) was grown on the substrate in order to relax a large lattice mismatch and then a (Zn, Cr)Te layer of 300 nm was grown on it. The growth of the both layers was performed under Te-rich condition (Zn/Te ≈ 0.5) at a substrate temperature of 300 °C. The surface during the growth was monitoring in situ using reflection high-energy electron diffraction (RHEED). The RHEED pattern during the growth was changed depending on the amount of Cr flux added. The (2×1) streak pattern was maintained for the addition of a small Cr flux corresponding to Cr compositions x less than x=0.01. However, with the increase of Cr flux, the streak image became spotty during the growth. With the further increase of Cr flux, a ring pattern appeared and finally the RHEED image disappeared during the growth.

The Cr composition was estimated using electron probe microanalyzer (EPMA) with a low acceleration voltage in order to probe only the Zn₁₋ₓCrₓTe layer, and it was checked by secondary ion mass spectroscopy (SIMS) in some of the samples. The obtained Cr composition x was larger than the ratio of Cr flux to the total cation flux ρ=ΦCr/(ΦZn+ΦCr). The factor ρ/p larger than unity, which could be considered as the relative efficiency of the incorporation of Cr atoms, indicates that the sticking coefficient of Cr on the Te-covered surface is larger than that of Zn.

2.2. Crystal characterization

The crystallographic analyses of the grown films were performed using X-ray diffraction (XRD) and high-resolution transmission electron microscopy (HRTEM). In order to exclude the possibility of minute precipitates of other phases, the XRD analysis was performed in the configuration intended for the high-sensitivity measurement [14]. Fig. 1 shows the diffraction pattern of θ–2θ scan for two Zn₁₋ₓCrₓTe films with x=0.07 and 0.17. In both samples, any other phase than the pure diluted phase of Zn₁₋ₓCrₓTe and/or ZnTe did not appear even at a dynamic range larger than six order of magnitude. The lattice constant along the growth direction was deduced from the diffraction angle of the (400) peak and was plotted against Cr composition in Fig. 1(b). As seen in the figure, the lattice constant a increased almost linearly with Cr composition x up to x=0.02, but it decreased again beyond x=0.02 and finally returned to the original value of ZnTe. The linear relation between a and x in the range of x≤0.02 suggests that Cr atoms are incorporated in the substitutional site of Zn. On the other hand, the deviation from it above x=0.02 might be related to some kind of disorder in the substitutional incorporation of Cr.

Microscopic crystalline structure was examined by HRTEM analysis. Fig. 2 shows the cross-sectional HRTEM images of three Zn₁₋ₓCrₓTe samples with different Cr compositions. In contrast to an almost perfect zinc-blende crystal in (a) x=0.01, many stacking faults were observed along the {111} plane in (b) x=0.07. Further, in (c) x=0.17, there appeared multi-domain structure consisting of nanometer-scale domains of zinc-blende structure with different orientations. However, any apparent precipitates of different phases than zinc-blende crystal were not observed in all these samples.
2.3. Magnetic and magneto-optical properties

The magnetic properties of (Zn, Cr)Te films were investigated in detail using superconducting quantum interference device (SQUID) magnetometer. Fig. 3 shows the magnetization versus magnetic field ($M-H$) curves in Zn$_{1-x}$Cr$_x$Te with Cr compositions of $x=0.01$, $0.07$, and $0.17$. These measurements were performed with magnetic fields perpendicular to the film plane at $T=2$ K. A diamagnetic response from a GaAs substrate was subtracted. The measurements with magnetic fields parallel to the film plane exhibited little differences. A clear hysteresis loop was observed in all the samples shown here. With the increase of Cr composition, the coercive field $H_c$ increased from $2H_c = 57$ mT at $x=0.01$ to $2H_c = 260$ mT at $x=0.17$.

Fig. 4 shows the magnetization versus temperature ($M-T$) curves of the same samples shown in Fig. 3. A magnetic field of 500 Oe was applied perpendicular to the plane. These $M-T$ curves in this figure are characterized by the irreversibility between the zero-field-cooled (ZFC) process and the field-cooled (FC) process and the appearance of a cusp in the ZFC process.

Fig. 1. (a) The diffraction pattern of XRD $\theta-2\theta$ scan of Zn$_{1-x}$Cr$_x$Te with two different Cr compositions of $x=0.07$ (lower curve) and $x=0.17$ (upper curve). (b) The lattice constant along the growth direction $a$ vs. Cr composition $x$. The inset shows a magnified plot of the range of $x=0–0.02$.

Fig. 2. Cross-sectional HRTEM images of Zn$_{1-x}$Cr$_x$Te layers with Cr compositions of (a) $x=0.01$, (b) $x=0.07$, and (c) $x=0.17$. The cross section is in the (110) plane and the upward direction in the figure is parallel to (001) (growth direction).

Fig. 3. Magnetization versus magnetic field ($M-H$) curves of Zn$_{1-x}$Cr$_x$Te films with Cr compositions of $x=0.01$, $0.07$ and $0.17$. In the measurement, magnetic fields were applied perpendicular to the film plane and the temperature was 2 K.
of this linear dependence of \( T_{310} \) with the increase of Cr composition from \( x \) from Arrott plot, the paramagnetic Curie temperature \( q \) of \( T_{q} \). The blocking temperature \( T_{B} \) deduced from Curie–Weiss plot, the blocking temperature \( T_{B} \) of the cusp peak in the ZFC curve, increased with Cr composition.

In Fig. 5, several critical temperatures deduced from various magnetization measurements are plotted against Cr composition; the ferromagnetic transition temperature \( T_{C} \) deduced from Arrott plot, the paramagnetic Curie temperature \( \theta_{p} \) deduced from Curie–Weiss plot, the blocking temperature \( T_{B} \) of the cusp in the ZFC curve. As shown in the figure, \( T_{C} \) increased almost linearly with Cr composition with the maximum value of \( T_{C} = 275\,\text{K} \) at \( x = 0.17 \). The extrapolation of this linear dependence of \( T_{C} \) on \( x \) is in agreement with the data of \( T_{C} = 300\,\text{K} \) at \( x = 0.20 \) reported by Saito et al. [9]. On the other hand, \( \theta_{p} \) was much higher and the dependence on Cr composition \( x \) was less steep than \( T_{C} \); \( \theta_{p} \) increased from 130 to 310 K with the increase of Cr composition from \( x = 0.01 \) to 0.17. The discrepancy between \( T_{C} \) and \( \theta_{p} \), the former representing the thermodynamic ferromagnetic transition of the whole system and the latter representing the ferromagnetic interaction with localized spins in a microscopic scale, is one of the features of the magnetic properties of this material.

Magneto-optical measurement was performed in order to derive intrinsic properties of (Zn, Cr)Te as a DMS. Magnetic circular dichroism (MCD), differential absorption between left- and right-circular polarized lights under magnetic fields perpendicular to the layer (Faraday configuration), was measured using a quartz stress modulator and a lock-in technique. The intensity of MCD signal is proportional to the Zeeman splitting at energy levels corresponding to the relevant optical transition. Therefore MCD spectra allow us to identify the material from which the magnetization originates.

Fig. 6 shows the MCD spectra of ZnTe and Zn\(_{1-x}\)Cr\(_{x}\)Te with \( x = 0.07 \) at 2 K. In the spectra of ZnTe, a sharp peak appeared at 2.38 eV, which corresponds to the Zeeman splitting at \( \Gamma \) point. On the other hand, the MCD spectra of Zn\(_{1-x}\)Cr\(_{x}\)Te exhibited a broad band of negative MCD signals which covered the whole spectral range in the figure. A steep drop at 2.37 eV is considered due to an enhanced Zeeman splitting at \( \Gamma \) point through the \( sp-d \) exchange interaction in Zn\(_{1-x}\)Cr\(_{x}\)Te. In Fig. 6(b), the MCD intensity at these critical points was plotted against magnetic field for ZnTe and Zn\(_{1-x}\)Cr\(_{x}\)Te. A linear field dependence of the MCD intensity in ZnTe is typical of the intrinsic Zeeman splitting in a non-magnetic semiconductor. On the other hand, the field dependence of MCD in Zn\(_{1-x}\)Cr\(_{x}\)Te exhibited a hysteresis loop, which was exactly the same shape as that of the magnetization in Fig. 3. This indicates that the magnetization measured by SQUID has the same origin as that causing the Zeeman splitting at the band-gap of ZnTe; that is, Cr spins contributing to the observed magnetization have the exchange interaction with \( sp \) electrons at the \( \Gamma \) point of ZnTe. This corroborates that the origin of the ferromagnetism is nothing but a DMS which is expressed as (Zn\(_{1-x}\)Cr\(_{x}\))Te, with Cr ions substituting the Zn site in ZnTe.

2.4. Effect of hole doping

As described in the preceding section, ferromagnetic properties were observed in (Zn, Cr)Te without additional carrier doping. Indeed, the electric measurement revealed that these undoped samples were insulating with high resistivities. Therefore the mechanism responsible for the observed ferromagnetism could not be attributed to the carrier-mediated interaction, differently from (In, Mn)As and (Ga, Mn)As. Then it is expected to provide a substantial clue to the origin of ferromagnetism to study how the magnetic properties are affected by the additional carrier doping in (Zn, Cr)Te. In this
study, we have investigated the magnetic properties of hole-doped (Zn, Cr)Te with nitrogen as an acceptor. The MBE growth of (Zn, Cr)Te:N films was performed in a similar way to the case of undoped samples, except the addition of nitrogen gas excited by radio-frequency (rf) plasma. The nitrogen doping concentration was controlled by changing the excitation power of rf-plasma in a range of 100–300 W with a fixed flow rate of nitrogen gas. In the examination of the crystallinity using XRD and HRTEM, precipitates of any other phases than the zinc-blende structure were not detected, similarly to undoped samples.

The electrical measurements were performed with In electrodes on the surface of the grown films. Fig. 7 shows the temperature dependence of resistivity of Zn$_{1-x}$Cr$_x$Te:N films with different Cr compositions $x$. All these samples were grown in the same condition of nitrogen doping. The concentration of nitrogen atoms in the substitutional site [N$_{Te}$] was estimated from the variation of the lattice constant $a$ following the linear relation between $a$ and [N$_{Te}$] reported in ZnTe:N [15] and it was found that [N$_{Te}$] was in the same order of $10^{20}$ cm$^{-3}$ in all the samples shown here. Nevertheless, the hole concentration $p$ deduced from the Hall measurement at room temperature decreased with the increase of Cr composition by more than three order of magnitudes; from $p=4.7\times10^{20}$ cm$^{-3}$ at $x=0.004$ to $p=3.4\times10^{17}$ cm$^{-3}$ at $x=0.032$. In accordance with this variation of the hole concentration, the temperature dependence of resistivity changed from metallic to insulating one with the increase of $x$, as shown in Fig. 7. This variation of the electric properties with Cr composition could be explained by the balance of the density between the shallow acceptor level of nitrogen and a deep level which is considered to be formed by Cr; that is, with the increase of Cr composition, the transition takes place from the degenerate semiconductor with a large doping concentration to the insulator in which the holes supplied from the nitrogen acceptors are trapped by the Cr deep level.

The magnetization measurements revealed that the hole doping significantly changes the magnetic properties of (Zn,Cr)Te. Fig. 8 shows $M$–$H$ curves of undoped and p-doped Zn$_{1-x}$Cr$_x$Te samples with the same Cr compositions of (a) $x=0.01$ and (b) $x=0.05$. The measurement was performed at $T=2$ K with magnetic fields perpendicular to the film plane.

In Fig. 8(a), a clear hysteresis loop was observed in the undoped sample #0, but this hysteresis was reduced and disappeared with the increase of hole concentration from sample #1 to #3. At the same time, the magnitude of magnetization became smaller in the same order. Fig. 8(b) compares $M$–$H$ curves between the undoped and p-doped samples at a higher Cr composition of $x=0.05$. As shown in the figure, a hysteretic behavior disappeared and the magnetization was reduced significantly in the p-doped sample with a nitrogen concentration of $\sim 10^{20}$ cm$^{-3}$. Analysis by Arrott plot showed that the ferromagnetic transition did not take place down to the lowest temperature of 2 K in samples #2 and #3 in Fig. 8(a) and also in the p-doped sample in Fig. 8(b). This is in clear contrast to the ferromagnetic transition temperature $T_C$ in the undoped samples of the same Cr compositions $x$; $T_C=5$ K in sample #0 in Fig. 8(a) and $T_C=30$ K in the undoped sample in Fig. 8(b).

The suppression of ferromagnetism by the nitrogen doping was confirmed in the range of Cr compositions $x\leq0.09$ studied here; with the nitrogen doping of the order of
$10^{20}$ cm$^{-3}$, hysteretic behaviors in $M$–$H$ curves disappeared, the magnetization was reduced, and the ferromagnetic transition was not observed down to 2 K.

3. Discussion

As described above, Zn$_{1-x}$Cr$_x$Te without carrier doping exhibited apparent ferromagnetic behaviors such as hysteresis in the $M$–$H$ curve. At the same time, the irreversibility in the $M$–$T$ curve, which is characteristic of magnetic disorder system such as superparamagnetism, was observed. Since any precipitates of other phases were not detected in the scrupulous examination of crystallinity using XRD and TEM, ferromagnetic clusters of different compounds such as spinel [8] could be excluded as an origin of the observed superparamagnetic behavior. In addition, the coincidence of the field dependence between the magnetization (Fig. 3) and the MCD signal (Fig. 6) corroborates that the magnetization measured by SQUID originates from the pure diluted phase of Zn$_{1-x}$Cr$_x$Te.

As shown in Fig. 5, the paramagnetic Curie temperature $\theta_p$ deduced from Curie–Weiss plot did not agree with the ferromagnetic transition temperature $T_C$ deduced from Arrott plot. It is considered that a high $\theta_p$ reflects a strong ferromagnetic interaction between Cr spins in a microscopic scale. In contrast, $T_C$ is much lower, particularly in low Cr compositions. This could be explained by assuming that this ferromagnetic interaction between Cr spins is of short range order; in low Cr compositions below the percolation, Cr spins are coupled with their neighbors due to the strong short-range interaction to form ferromagnetic clusters, but they are isolated with each other. Therefore the ferromagnetic alignment of all Cr spins in the whole system is difficult to take place and $T_C$ remains low in contrast to much higher $\theta_p$. Following the above picture, it could be reasonably understood that $T_C$ shows a tendency to approach $\theta_p$ towards $x=0.2$, which is the percolation threshold in the zinc-blende crystal.

Concerning the origin of the ferromagnetic interaction in (Zn, Cr)Te, the carrier-induced mechanism is denied from the observation of the ferromagnetism in the undoped samples and its suppression due to the hole doping. Other than the carrier-induced mechanism, the superexchange [6] or the double exchange [16] has been known as the origin of the ferromagnetic interaction. Sato et al. proposed the double-exchange interaction as an origin of the ferromagnetism in (Zn, Cr)Te based on the first-principle calculation [17]. According to their calculation of the density of states (DOS), non-bonding state $e$ and anti-bonding state $ta$ are created from the Cr 3d level in the band gap of ZnTe and the Fermi level is located in the middle of the upper $ta$ state. With the half-filled $ta$ state, the ferromagnetic coupling between 3d spins is at work due to the band broadening (double exchange mechanism). This double exchange interaction is of short-range due to well-localized character of the Cr 3d state in the gap. The result of calculation beyond mean-field approximation predicted the ferromagnetic transition temperature, which agrees well with our experimental data shown in Fig. 5 [18].

The suppression of the ferromagnetism in p-doped samples could be explained qualitatively following the above double exchange mechanism; upon the doping of acceptor impurities, the Fermi level shifts downward from the middle of the $ta$ state, which results in a significant decline of the ferromagnetic interaction due to the decrease of the density of states at the Fermi level. The validity of the above picture is to be examined by probing directly the valence of Cr ions in the doped samples using experimental technique such as infrared spectroscopy [19].

4. Conclusion

Recent results of our experimental studies on the novel ferromagnetic semiconductor (Zn, Cr)Te were described. Thin films of Zn$_{1-x}$Cr$_x$Te with Cr composition up to $x=0.17$ were successfully grown by MBE, without any trace of precipitates of other phases detected by XRD and HRTEM. In the magnetization measurements on Zn$_{1-x}$Cr$_x$Te without carrier doping, a hysteresis loop in the $M$–$H$ curve and the irreversibility in the $M$–$T$ curve were observed. From the similar hysteretic field dependence of the MCD signal at the band-gap energy of ZnTe, it was confirmed that the ferromagnetism is an intrinsic property of Zn$_{1-x}$Cr$_x$Te as a DMS. The ferromagnetic transition temperature $T_C$ deduced from Arrott plot increased almost linearly with Cr.
composition with the maximum $T_C = 275$ K at $x=0.17$. In p-doped Zn$_{1-x}$Cr$_x$Te with nitrogen concentrations of the order of $10^{20}$ cm$^{-3}$, the ferromagnetic behaviors were suppressed. The above experimental findings could be explained qualitatively by considering the double exchange mechanism as an origin of the ferromagnetic interaction. In particular, the suppression of the ferromagnetism due to the hole doping could be understood as a result of significant decline of the ferromagnetic double exchange interaction, which is caused by the downward shift of the Fermi level from the middle of the 3$d$ impurity level upon the acceptor doping.

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