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MAGNETIC RESONANCE IN FERROMAGNETIC DILUTED MAGNETIC SEMICONDUCTORS

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We overview the typical properties of the EPR spectra of the most representative $A^{II}B^{VI}$ diluted magnetic semiconductors (e.g. $Ga_{1-x}Mn_xAs$, $Cd_{1-x}Mn_xTe$, $Cd_{1-x}Cr_xSe$). We study the magnetic anisotropy in $Cd_{1-x}Cr_xTe$ from angular dependence of the resonance field in FMR experiment . The samples were obtained from the melt with nominal concentration x≈0.04. The temperature dependence of the EPR linewidth points on a room Curie temperature.

INTRODUCTION

The most investigated diluted magnetic semiconductors (DMS) are based on a classical semiconductors of the types: $A^{II}B^{VI}$ (A=Cd, Zn, B=S, Se, Te), $A^{III}B^{VI}$ (A=Ga, In, Al, B=As, P, N). In DMS a fraction of cations is replaced by magnetic ions (Mn, Fe, Co). Magnetic properties of such system arise from the strong exchange interaction between delocalized s and p electrons (forming conduction and valence bands), and d localized electrons of the magnetic impurity. The s-d exchange is positive (ferromagnetic) for all DMS and its value is near $N\alpha \approx 0.2$ *eV*. The exchange interaction between valence band p electrons (formed by p electrons of anions) and d electrons is antiferromagnetic one ($N\beta \approx -1{,}6 \text{–} 1.9$ *eV*) almost in all DMS's containing Mn ions. In this case the p-d exchange is indirect. Due to much greater value of this interaction, the resulting magnetism is of the antiferromagnetic ordering in the materials containing Mn, Fe as well as Co. However, Dietl et al. demonstrated in the frame of mean-field approximation (Dietl, Haury & Merle d'Aubigne, 1997) that at sufficiently high hole densities, the ferromagnetic phase in bulk and low dimensional II-VI structures can be attained. The Curie temperatures for many III-V and II-VI compounds were calculated (Dietl, Ohno, Matsukura, Cibert & Ferrand, 2000; Dietl, Ohno & Matsukura, 2001). In this paper we present a typical EPR (FMR) properties of ferromagnetic dilute magnetic semiconductors (Section 3). For comparison in section 2 we overview the EPR antiferromagnetic properties of $Cd_{1-x}Mn_xTe$ which is the most popular DMS. In section 4 we present our measurements of ferromagnetic properties of $Cd_{1-x}Cr_xTe$.

EPR LINEWIDTH IN $Cd_{1-x}M_xTe$

The dependence of EPR spectra in $Cd_{1-x}Mn_xTe$, $Cd_{1-x}Mn_xSe$ and $Cd_{1-x}Mn_xS$ on the concentration x and the temperature have been studied in many papers (see e.g. (Oseroff, 1982; Kremer & Furdyna, 1985; Sayad & Bhagat, 1985; Furdyna & Kossut, 1988) and References therein). The general features of the spectra are:

1) For small Mn concentrations ($x<0.001$) the interaction between Mn^{2+} spins can be neglected and the fine structure of the spectra is visible. The Mn^{2+} ions have a $3d^5$ configuration. The ground state $^6S_{5/2}$ is not pure S state, therefore crystal field splits this level into three doublets. This degeneracy is removed completely by an external magnetic field, therefore five lines of the fine structure are observed. Further, these lines are split into six lines due to hyperfine interaction (nuclear magnetic moment of Mn is 5/2). While $Cd_{1-x}Mn_xTe$ crystallites in a zinc-blende structure, the EPR spectrum is described by the spin Hamiltonian (Abragam & Bleaney, 1970):

$$H = g\,\mu_B \boldsymbol{H}\boldsymbol{S} + (a/6)\,(S_x^4 + S_y^4 + S_z^4) + A\,\boldsymbol{I}\boldsymbol{S} \qquad (1)$$

where the first, second and third terms represent, respectively, the electronic Zeeman interaction, the crystal field and the hyperfine interaction. The gyromagnetic factor *g* is nearly equal to the free electron $1.99 < g < 2.02$.

The Mn ion levels are visible at sufficiently low temperatures (kT less then levels spacing: *0.1 K* at zero magnetic field) or at high magnetic fields $H>10$ *kOe* Then the system with uniformly spaced levels fulfils a Curie law

$$\chi = [N g^2 \mu_B^2 S(S+1)]/3k_BT \qquad (2)$$

where N is the number of ions in the unit volume, S is the total spin.

2) For Mn concentration above $x \approx 0.005$, the clusters of various sizes of Mn atoms appear rapidly (Nagata, Galazka, Mullin, Akbarzadeh, Khattak, Furdyna & Keesom, 1980; Okada, 1980). Then the susceptibility χ is expressed as

$$\chi = \Sigma N_i \chi_i \tag{3}$$

where N_i is the number of clusters (per unit volume) of various sizes (nearest, next-nearest neighbor etc.), χ_i is a susceptibility of the cluster of the i-th type (Oseroff & Keesom, 1988)

$$\chi_i = \frac{g^2 \mu_B^2}{3 k_B T} \frac{\sum_S \$(S+1)(2S+1) exp(-E_S / k_B T)}{\sum_S (2S+1) exp(-E_S / k_B T)} \tag{4}$$

The theory of the Mn clustering explains the critical concentration $x=x_c$ at which the size of cluster is comparable with the size of the sample. For the CdMnTe $x_c=0.2$ what is in good agreement with value $x_c=0.195$ obtained from percolation theory for the nearest neighbor interaction in fcc structure.

In CdMnTe materials the nearest–neighbor interaction between Mn spins is antiferromagnetic (Galazka, Nagata & Keesom, 1980; Shapira, Foner, Ridgley, Dwight & Wold, 1984) resulting in spin-glass or long range antiferromagnetic phases of the system.

3) For $x > 0.005$ the EPR spectrum is a single line which could be approximated by Lorentzian function very well.

In the concentration range $0.005 < x < 0.03$ the linewidth decreases significantly with the concentration increase reaching the minimum at $x = 0.03$.

4) For $x > 0.03$, the increasing of the concentration, results in significant monotonic broadening of the line (Oseroff, 1982; Sayad & Bhagat, 1985) at room temperature, while the g factor is almost unchanged (Webb, Bhagat & Furdyna, 1984) and independent on microwave frequency (Webb et al., 1984).

The large increase of the linewidth with decrease in temperature (Grochulski, Leibler, Sienkiewicz & Galazka, 1979; Oseroff, Calvo & Giriat, 1979) (Manoogian, Chan, Brun del Re, Donofrio & Woolley, 1982). was attributed to an increase of the internal field formed by finite clusters and was described by Huber law (Huber, 1972).

$$\Delta H_{pp} = A\left(\frac{T_C}{T - T_C}\right)^{\alpha} + B \tag{5}$$

where α is a critical exponent, T_c is an order- disorder transition and B is the high temperature linewidth.

The values of T_c, α and B obtained from this equation depend clearly on composition x. However, the values of T_c were to large compared with the temperature $T_{f\Delta H}$ of the cusp in the ZFC susceptibility measurements. A better agreement was achieved by Oseroff et al. (Oseroff, Calvo, Frisk & Acker, 1980; Oseroff, Calvo, Giriat & Fisk, 1980; Oseroff, 1982) using an equation (Dormann & Jaccarino, 1974)

$$\Delta H_{pp} = A\left[\frac{T_{f\Delta H}}{T - T_{f\Delta H}}\right]^{\alpha_{\Delta H}} + B\left[\frac{\Theta}{T} + 1\right] \tag{6}$$

where the symbols correlates with Huber's law and Θ is The Curie-Weiss temperature. The second term becomes dominant at $T >> T_{f\Delta H}$.

Similar equation was used for the change of resonance field H_R with temperature due to internal field H_i

$$H_i = H_0 - H_R = C \tag{7}$$

where H_0 is the field corresponding to $g = 2$.

Values of parameters describing the temperature dependence of EPR linewidth as well as resonance field for CdMnTe were collected by Oseroff (Oseroff, 1982) in several tables for a wide range of concentration $0.01 < x < 60$.

Sayad and Bhagat (Sayad & Bhagat, 1985) explained the line broadening with lowering T by a Lorentzian distribution of random fields centered at zero. In this model the linewidth depends on temperature according to expression proposed by Bhagat et al. (Bhagat, Spano & Lloyd, 1981) for several ferromagnetic systems showing spin-glass phase at low temperatures:

$$\Gamma = \Gamma_0 + \Gamma_1 exp(-T/T_o) \tag{8}$$

where Γ_o is the high temperature linewidth, Γ_1 and T_0 are empirical parameters.

Equation (8) fits the experimental data in the range of temperature 50–100 K very well. However, for Mn concentration below the threshold concentration, x < 0.2, the discrepancy between experimental and theoretical behavior is significant (see Fig.1 in (Sayad & Bhagat, 1985)). Such inconsistency does not exist in the case of the fitting with the Huber law (see e.g. Fig.3 in (Oseroff & Keesom, 1988)).

FERROMAGNETIC CHROMIUM BASED DMS'S

Peculiar group of diluted magnetic semiconductors is that in which ions with less than half filled shell (e.g. V, Ti, Cr) are incorporated into a semiconductor matrix.

The IV-VI chromium based DMS were investigated widely by Dobrowolski, Story, Wilamowski et al (Story, Eggenkamp, Swuste, Swagten & de Jonge, 1993;

Grodzicka, Dobrowolski, Kossut, Story & Witkowska, 1994; Story, Wilamowski, Grodzicka & Dobrowolski, 1995; Wilamowski & Story, 1995; Dobrowolski, Arciszewska, Brodowska, Domukhowski, Dugaev, Grzeda, Kuriyliszyn-Kudelska, Wojcik & Slynko, 2006).

The ferromagnetic properties of Cr-based II- VI DMS's were theoretically predicted by Blinowski et al (Blinowski, Kacman & Majewski, 1995; Blinowski, Kacman & Majewski, 1996). Such properties results mainly from location of the unoccupied t_+ and t_- d orbitals above the top of the valence band (Bhattacharjee, 1992; Blinowski et al., 1996).

The first observation of the ferromagnetic p-d exchange in II-VI type DMS , $Zn_{1-x}Cr_xSe$ *x=0.005* , was reported by Mac et.al (Mac, Khoi, Twardowski & Gaj, 1993) in free exciton spectroscopy experiment, where band splitting measured was different from those observed for Mn-, Co- , Fe-, based DMS. These properties were supported by studies of other zinc chalcogenides: $Zn_{1-x}Cr_x$ Se $_{0.95}S_{0.05}$, $Zn_{1-x}Cr_x$ S and, $Zn_{1-x}Cr_xTe$ (Mac, Twardowski & Demianiuk, 1996). The p-d exchange constants were positive for these compounds as well as for $Zn_{1-x}Cr_xSe$. (Table 1). Herbich et al measured in this same way the p-d coupling in $Cd_{1-x}Cr_xTe$ for *x =0.002 -0.003*, see Table 1.

Table 1. Exchange constants for zinc and cadmium chalcogenides doped with Cr.

Compound	Sample method of growth substrate thicknes	$N_0\alpha$, *eV (assumed values)*	$N_0\beta$, *eV*	Method	T_C, *K*	Ref.
$Zn_{1-x}Cr_xS$ *x< 0.007*		(0.2)	0.57 0.62 (corrected)	Polarized Magnetorefl., Magnetization T=2K	2	A
$Zn_{1-x}Cr_xSe_{0.95}S_{0.05}$ x<0.007	Single crystal, Bridgman	(0.2)	0.83 0.93 (corrected)	-//-		A
$Zn_{1-x}Cr_xSe$ *x= 0.0011* *x= 0.0025* *x= 0.0035* *x= 0.0045*	Single crystal, modified Bridgman	(0.2)	0.85 0.95 (corrected)	Free excitation spectroscopy		B
$Zn_{1-x}Cr_xTe$ *x< 0.001*	Single crystals, modified Bridgman	(0.2)	3.6 4.25 (corrected)	Polarized Magnetorefl., Magnetization		A
$Zn_{1-x}Cr_xS$ *x= 0.003*	Single crystal, Bridgman			Magnetization M(B), M(T), 1.5-300 K, B=0-6T	~300 (for the minority phase)	G
$Zn_{1-x}Cr_xS$ *x= 0.17*	Single crystals, modified Bridgman, thin films MBE				275	C
$Zn_{1-x}Cr_xTe$ *x= 0.0026* *x= 0.141* *x>0.18*	Thin fims MBE, ZnT/GaAs			Magnetization M(T), M(B)	120 (x=0.026) 265 (x=0.14) 365 (from $Cr_{1-\delta}Te$ precipitates)	H
$Zn_{1-x}Cr_xTe$ *x-0.20*	Films MBE, ZnTe/GaAs, 200-400 nm			Transmission TMCD	300±10	E
$C_{1-x}Cr_xS$ x=0.002	Single crystals, High-pressure Bridgman	0.22 (T=1.9 K, B≤10 T)		Magnetization M(B), T=1.9 K Spin-flip Raman scattering		I
$Cd_{1-x}Cr_xS$ *x=0.0024* *x=0.0031* *x=0.0033*	Single crystals	(0.22±0.01)	0.48±0.05	-//-	275	D
$Cd_{1-x}Cr_xTe$ *x=0.04*	Crystals (vertical solidification)			Magnetization M(T), M(B), T=5-600 K	395	F

A - (Mac et al., 1996), B - (Mac et al., 1993) C - (Kuroda, Ozaki, Nishizawa, Kumekawa, Marcet & Takita, 2005), D - (Herbich, Mac, Twardowski, Ando, Shapira & Demianiuk, 1998), E - (Saito, Zayets, Yamagata & Ando, 2003), F - (Ko & Blamire, 2006), G-(Pekarek, Luning, Miotkowski & Crooker, 1994), H- (Hou, Teo, Sreenivasan, Liew & Chong, 2006), I- (Twardowski, Heiman, Liu, Shapira & Demianiuk, 1996)

The $Zn_{1-x}Cr_xTe$ DMS with greater concentration of chromium *x<0.17* was investigated by Kuroda et al (Kuroda et al., 2005) by magnetization measurements. The linear increasing of the Curie temperature was observed reaching $T_c = 275$ K at $x = 0.17$.

In Table 1 we have collected other results on Cr based II-VI DMS.

In the case of cadmium telluride crystals the doping with Cr introduces a deep donors, whose levels lie in the forbidden band gap in the energy range $(E_v+0.19)–(E_v+0.39)$ *eV* (Nikonoyuk, Zakharuk, Kuchma, Kovalets, Rarenko & Yurichuk, 2007) (see Fig.1).

Vallin (Vallin & Watkins, 1974) studied ZnS, ZnSe, ZnTe, CdTe, CdTe for small Cr concentration. A significant static tetragonal Jahn-Teller distortion was observed from the changes of the fine structure of the spectra due to the axial pressure applied to the sample. The Jahn-Teller effect is not essential in the DMS with Mn. Therefore, it is new feature which contributes to the ferromagnetic properties of $A^{II}B^{VI}$ semiconductors with chromium.

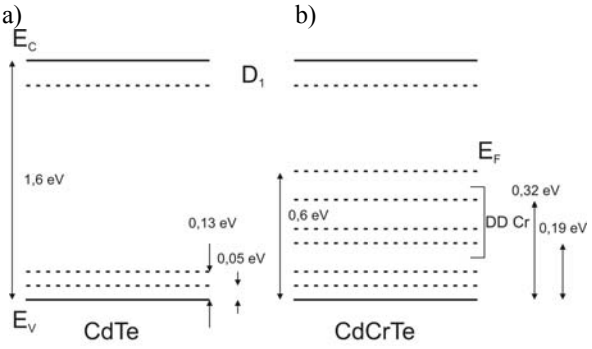

Fig. 1. Energy diagram of CdTe (a) and CdTe:Cr (b) crystals.

MAGNETIC ANISOTROPY IN $Cd_{1-x}Cr_xTe$ CRYSTALS

We have grown polycrystalline $Cd_{1-x}Cr_xTe$ solid solution by liquid-state synthesis of stechiometric (*x=0.04*) powered quantities Cd and Cr_2Te_3 (Aldrich 99.9). Synthesis was accomplished in vacuum in quartz ampoule at *1320 K*. The ingot obtained was cut into disks of *3 cm* diameter and 1 mm thick. The averaged chemical composition of the wafer was checked by X-ray fluorescence analyses revealing a homogenous distribution of Cr in ingot. Samples in the averaged shape of about $1x2x3\ mm^3$ cuboids were formed by the proper breaking of the wafer.

The FMR experiments were carried out in 9.4 GHz EPR spectrometer at room temperature. Angular dependence of the spectra was measured using one-axis goniometer. The EPR experiments on $A^{II}B^{VI}$ semiconductor crystals with low concentration of Cr have been reported previously by several authors (Vallin & Watkins, 1974; Cieplak, Godlewski & Baranowski, 1975; Biernacki, 1978). The EPR results are well described by the spin Hamiltonian:

$$\hat{H} = \mu_B g_{\parallel} S_z H_z + \mu_B g_{\perp}\left(S_x H_x + S_y H_y\right) + DS_z^{\,2} + E\left(S_x^{\,2} - S_y^{\,2}\right) + \frac{F}{180}\left[35S_z^{\,4} - 30S(S+1)S_z^{\,2} + 25S_z^{\,2}\right] + \frac{1}{6}a\left(S_1^{\,4} + S_2^{\,4} + S_3^{\,4}\right) \quad (9)$$

where μ_B is the Bohr magneton, $\vec{H}$ is the magnetic field, and S is the spin quantum number of Cr^{2+} ion ($S = 2$) and (1,2,3) are cubic axes. The D, E, F and a parameters are fine structure constants taking values (Vallin & Watkins, 1974):

$g_{\parallel} = 1.90 \pm 0.15$, $g_{\perp} = 1.980 \pm 0.015$,

$D = 0.260 \pm 0.004\ cm^{-1}$ $E = 0$, $a = 0.05 \pm 0.01\ cm^{-1}$,

$F = -0.05 \pm 0.02\ cm^{-1}$.

These Cr based DMS (with small *x: 0.001<x<0.01*) show paramagnetic properties and fulfil the intermediate case between Brillouin and Van Vleck paramagnetism while the ground state consist of multiplet of the nearby located states.

Our samples contain substantial amount of Cr ions: *x=0.04*. Therefore, we expect the ferromagnetic properties according to the theoretical works of Blinowski, Kacman and Majewski (Blinowski et al., 1996). Our measurements of magnetic resonance seem to confirm the presence of ferromagnetism in the CdCrTe samples studied. The spectra reveal typical features of the FMR: anisotropy of the resonance field as well as the anisotropy of the peak-peak linewidth of the EPR signal. The Lorentzian shape of the lines contributes to this conclusion as well.

The twofold symmetry of the line position changes in the angular dependence (Fig. 2a) indicates that the plane *"a"*, in which the anisotropy was registered, is the crystallographic plane (110). We compare these data with formulae derived in previous our paper (Stefaniuk, Bester & Kuzma, 2008)

$$H_{res} = -\frac{1}{2}NM_z + \frac{H_1}{2}\left(4 - 5\sin^2\theta - \frac{15}{4}\sin^2 2\theta\right) + \frac{H_2}{2}\left(\frac{1}{2}\sin^6\theta + 3\sin^2\theta\cos^4\theta - 7\sin^4\theta\cos^2\theta - \sin^2\theta\cos^2\theta\right) + \sqrt{R^2 + \left(\frac{\omega}{\gamma}\right)^2} \quad (10a)$$

where

$$R = -\frac{1}{2}NM_z + \frac{H_1}{2}\left(\frac{9}{4}\sin^2 2\theta - 3\sin^2\theta\right) + \frac{H_2}{2}\left(-\frac{1}{2}\sin^6\theta - 3\sin^2\theta\cos^4\theta + 4\sin^4\theta\cos^2\theta - \sin^2\theta\cos^2\theta\right) \quad (10b)$$

There are three unknown parameters in equations 10a and 10b: NM_z, $H1=K_1/M_z$, and $H_2=K_2/M_z$.

A general graphical view of the angular dependence of the resonance field (equation 10a) in the plane (110) is

presented in Fig. 2b. This plot was obtained for (100) plane in which three nonequivalent axes [001], [111] and [110] were indicated.

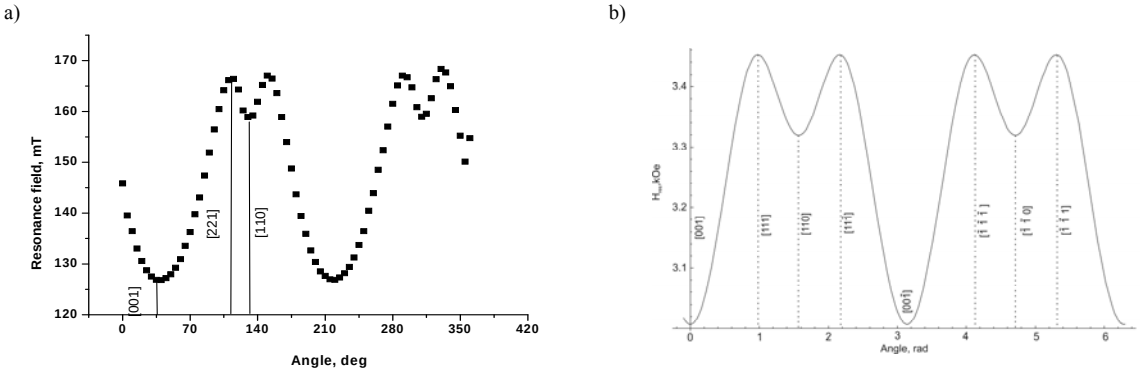

Fig. 2. Experimental angular dependence of the resonance magnetic field of $Cd_{1-x}Cr_xTe$ (*x=0.04*) in the plane (110) with principal axes [001], [221] and [110] (a); Angular dependence of the resonance magnetic field in the plane (110) of a cubic crystals (according to equation (10a) for the following parameters: H_1=0.12 kOe, H_2=0.1 kOe, NM_z=1.2 kOe, ω/γ=3.8 kOe (b).

The shape of the curve in Fig. 2a resembles that in Fig. 2b. However, the distance between the two axes is about 20^o rather than 54^o. This is in agreement with the presumption that in our crystal the axis [221] is more significant than [111] direction (Stefaniuk et al., 2008).

The temperature dependence of the linewidth was studied in our previous paper (Kuzma, Stefaniuk & Bester, 2010). The dependence did not relate to that observed for Cd or Zn based antiferromagnetic DMS with Mn (e.g. $Zn_{1-x}Mn_xS$, $Zn_{1-x}Mn_xSe$, $Zn_{1-x}Mn_xTe$, $Cd_{1-x}Mn_xS$, $Cd_{1-x}Mn_xSe$, $Cd_{1-x}Mn_xTe$, (Kremer & Furdyna, 1985)). For samples with Mn there is a dramatic broadening of the resonance at low temperatures and then the resonance narrows continuously as the temperature increases. Such behaviour is typical for antiferromagnet DMS. In our sample we noted that the linewidth decreases starting from high temperature until temperature T_{min}= *350 K* at which the linewidth takes minimal value. Below this temperature the linewidth increases with temperature decreasing. Such behaviour was observed in studies of manganites (Ivanshin, Deisenhofer, Krug von Nidda, Loidl, Mukhin, Balbashov & Eremin, 2000) and is not fully understood. The same type of the temperature dependence of the EPR linewidth was observed by Story at al (Story et al., 1993) in $Pb_{1-x}Sn_yMn_x$ Te in the regime of high carrier concentration exceeding the threshold value (see Fig.8 in (Story et al., 1993)).

CONCLUSIONS

We reviewed the main features of the ferromagnetic resonances (EPR, AFM, FMR) in magnetic semiconductors. In our material investigated ($Cd_{1-x}Cr_xTe$, *x=0.04*) the resonance field decreases strongly at temperatures *T< 350 K*, the linewidth become large and the shape of lines is asymmetric considerably. Therefore at low temperatures the magnetic resonance of the material studied is addressed as ferromagnetic resonance (FMR). The temperature dependence of the linewidth points on significant p-d exchange interaction as in ferromagnetic phase of model $Pb_{1-x}Sn_yMn_xTe$ DMS presented in (Story et al., 1993).

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