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Optically detected magnetic resonance investigations of diluted magnetic semiconductors

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Mechanisms responsible for optical detection of Mn^{2+} magnetic resonance in II-Mn-VI compounds are discussed. We describe several of these mechanisms. The most efficient one which is due to very efficient spin-flip interactions of localized magnetic moments of Mn^{2+} ions with free carriers is important at increased excitation density.

Keywords: magnetic resonance, diluted magnetic semiconductors, spin flip interactions, spin dynamics.

1. Introduction

Diluted magnetic semiconductors (DMS) of II-VI compounds [1, 2] are intensively studied due to their possible use in spintronics and in optoelectronics devices. In particular, spin-related relaxation processes are of importance, since these processes govern spin relaxation times crucial for applications mentioned above.

In this paper, we review the results of our recent magneto-optical and optically detected magnetic resonance (ODMR) investigations of a range of II-Mn-VI samples of different dimensionality. Mn^{2+}-related magnetic resonances in bulk samples of ZnMnS, ZnMnSe, CdMnTe and in quantum well (QW) structure of CdMnTe/CdMgTe are discussed. The ODMR method is employed not only to the studies of recombination processes in DMS systems, but also to evaluate the rate of some nonradiative recombination transitions, and the role of Mn^{2+}-free carriers interactions in energy dissipation processes. We shortly describe how dynamics of spin interactions in DMS samples can be studied with the ODMR using time-resolved study.

2. Principles of ODMR

Principles of ODMR were described in numerous papers (see, *e.g.*, [3, 4]). Formally, ODMR yields the same information as electron spin resonance (ESR). Lande factor

(so-called *g*-factor), its symmetry, spin multiplicity, *etc*., can be determined from both types of these magnetic resonance experiments. The essential difference relates to the mechanism of detection. In the ESR, we study resonances of systems in a ground state, by tracing microwave absorption at the resonance. Photo-excitation is necessary to extend this method to light-excited states [5]. In the ODMR, in turn excited states of a given system are studied in most of the cases. Magnetic resonances are detected via changes in light emission at resonance conditions. This helps to relate the observed magnetic resonance signals to recombination paths active in light emission processes. This is a unique property of the ODMR method.

Initially, the ODMR method was successfully applied to study recombination processes of donor-acceptor pairs (DAPs) in semiconductors [3]. We thus use DAP processes to introduce the ODMR method. DAP transitions between donor and acceptor species with spins ½ are discussed for simplicity. In an external magnetic field, spins of donor and acceptor species are aligned. For some of the donor and acceptor pairs spins are parallel (DAP in spin triplet state) for other antiparallel (DAP in spin singlet state). The latter pairs can decay readily, since recombination process from spin singlet excited state of the pair to a spin singlet ground state (no electron and no hole) is a spin allowed as an electric dipole transition between states of the same spin multiplicity. This spin selection rule results in depopulation of donor acceptor pairs with antiparallel spin orientation, and leaves an increased population of pairs with a parallel orientation of spins. We must assume here that spin relaxation processes due to spin-lattice interactions are rather inefficient, otherwise spin relaxation (thermalization) determines the population of spin substates of donors and acceptors. If we now flip the spin of a donor or acceptor, at magnetic resonance, we will allow radiative recombination for DAP pairs which initially had parallel spins. In consequence, an increase in intensity of the DAP emission will be observed at the magnetic resonance. Donor- and acceptor-related resonances will be observed via DAP emission, which allows not only center identification but also yields identity of recombination processes [3].

3. Mechanisms of ODMR detection in DMS systems

In the following paragraphs we will discuss ODMR mechanisms which are specific to a class of DMS samples of II-Mn-VI compounds. Several possible mechanisms were considered in the previous studies, such as: spin dependent energy transfer [6–8], spin dependent scattering [9–11], cross-relaxation [6, 12], effects caused by nonresonant backgrounds [13, 14], and magneto-optical or semimagnetic effects [2, 15–17]. Their relative importance remained however not clear [18]. We will demonstrate that due to the specific properties of this system ODMR signals observed are uncommonly large, compared to the signals detected in nonmagnetic samples, and often relate to few mechanisms mentioned above. Their relative importance depends on excitation conditions. We will also discuss how the ODMR can be used to evaluate the efficiency of Auger mechanism of bound excitons quenching.

3.1. Spin cross-relaxation processes

Energy transfer processes of spin excitation are discussed as first. These are often referred to as spin cross-relaxation processes. Two types of such processes are discussed below.

3.1.1. Mn-DAP cross-relaxation

Cross-relaxation is an energy transfer process, in which interacting centers exchange spin excitation energy. Such a mechanism of the ODMR detection in DMS samples was first proposed in references [6, 12]. In the processes discussed therein, Mn^{2+} ions relax their spin excitation energy by transfer to nearby components of the DAP, *i.e.*, either to donor or acceptor. In consequence, the spin of a donor or an acceptor is flipped and DAP recombination is speeded up, using the mechanism explained in

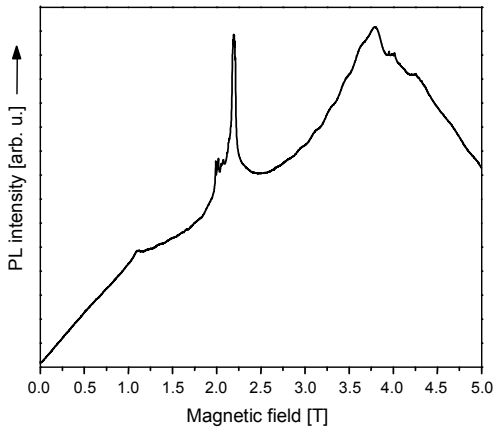

Fig. 1. Low temperature ODMR spectrum of bulk ZnMnSe sample measured with the 60 GHz ODMR system developed by us. For the PL excitation we used above band gap excitation.

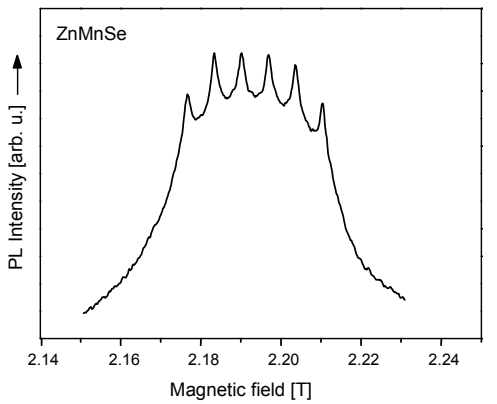

Fig. 2. Part of the ODMR spectrum shown in Fig. 1 from the region of the Mn^{2+} magnetic resonance. A well-resolved 6 line hyperfine structure of Mn^{2+} was observed.

the introduction to the ODMR method. Results of relevant experiments are shown in Figs. 1 and 2.

We observed the Mn-DAP cross-relaxation for ZnMnSe bulk sample with a fairly low Mn fraction (below 0.5%). For such a sample, the ODMR spectrum (observed via an increase in intensity or change in polarization rate of a green-red DAP photoluminescence (PL) emission) consists of three signals: a strong donor related resonance at about 3.8 T (see Fig. 1), a structured, Cu-acceptor related resonance at 2.04 T (studied by us earlier [19, 20]), and a fairly strong resonance at 2.19 T, with a well-resolved 6 line hyperfine structure, better seen in Fig. 2. The latter signal is due to Mn^{2+} ions and is detected via an increase in intensity of the DAP PL emission, *i.e.*, Mn^{2+} magnetic resonance speeds up DAP PL emission, as is expected in the case of efficient Mn-DAP cross-relaxation processes.

The magnitude of the ODMR signals shown in Figs. 1 and 2 is fairly typical, *i.e.*, similar to that observed in nonmagnetic ZnSe (0.1 to 0. 5% of PL intensity [19, 20]).

3.1.2. Mn-Mn cross-relaxation

For ZnMnS DMS samples with an increased Mn fraction (1% and more) DAP PL emission is no longer observed. Visible PL in such samples is dominated by an orange PL attributed to $^4T_1$ to $^6A_1$ intra-shell transition of Mn^{2+} ions. This intra-shell transition is a spin and parity forbidden process, which is characterized by fairly slow recombination rates (weak PL and ms decay times) [21, 22]. For this system we evaluated the importance of spin cross-relaxation interactions between Mn ions by studying the origin and intensity of the ODMR signal.

The experiments shown in Figs. 3 and 4 were performed using the Q-band (36 GHz) ODMR system developed by us. The broad signal of magnetic resonance (see Fig. 3) was detected in the ODMR study. We performed ESR investigations to determine the identity of this resonance. ESR signal was measured in the same system as the ODMR one. The ESR spectrum thus obtained is dominated by a very strong and broad magnetic resonance of Mn^{2+} ions. The hyperfine structure is not resolved, which is a typical feature of DMS samples with increased Mn fractions [2, 18]. This signal is identical to the one observed in the ODMR study. Thus, signal observed in ODMR is due to the magnetic resonance of Mn^{2+} ions.

The ODMR signal shown in Fig. 3 was detected via an increase in the PL intensity of the $^4T_1 \rightarrow {}^6A_1$ intra-shell PL of Mn^{2+} ions. The latter we proved by measuring the so-called ODMR-PL spectrum, which is shown in Fig. 4.

In the ODMR-PL we measured a spectral response of the PL to the magnetic resonance by setting magnetic field at the Mn^{2+} resonance conditions and by scanning the response of the PL spectrum to this resonance. In such a way we found that Mn^{2+} magnetic resonance is detected via an increase in intensity of Mn^{2+} intra-shell PL. The relevant results, shown in Fig. 4, mean that flip of a spin of Mn^{2+} ion in the ground state helps to relax spin selection rules for the intra-shell recombination at adjacent

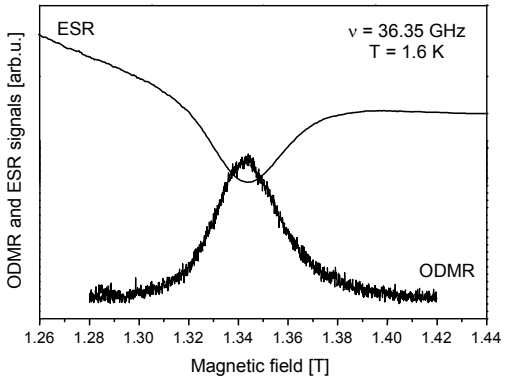

Fig. 3. Low temperature ESR and ODMR spectra of bulk ZnMnS (1% Mn fraction) measured with the Q-band ODMR system.

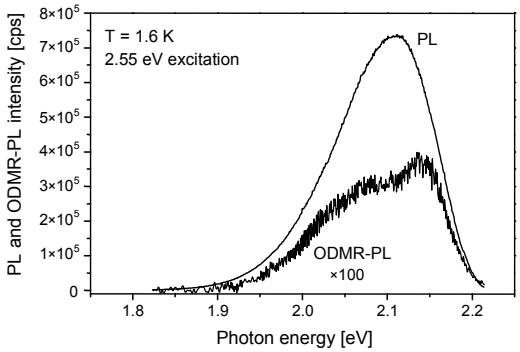

Fig. 4. Photoluminescence spectrum of ZnMnS (1% of Mn), as compared to the ODMR-PL spectrum taken with magnetic field set at Mn^{2+} magnetic resonance conditions.

excited Mn^{2+} ion. The ODMR investigations thus confirm that spin cross-relaxation interactions between adjacent Mn ions are present. The mechanism cannot however be very efficient, since ODMR-PL spectrum is about 200 times weaker than the PL spectrum.

3.2. Magnetization quenching at Mn^{2+} magnetic resonance

In our initial ODMR investigations of bulk CdMnTe samples [18] we observed a derivative-like response of band edge PL to the Mn^{2+} magnetic resonance. Excitonic PL bands were shifted up-in-energy at magnetic resonance. Such a response of the excitonic PL bands was related to magnetization quenching [18].

The present experiments were performed for a CdMnTe/CdMgTe single QW structure with 1% Mn fraction in the magnetic QW and with 20% of Mg in the CdMgTe barriers. The structure was grown by molecular beam epitaxy on a GaAs substrate

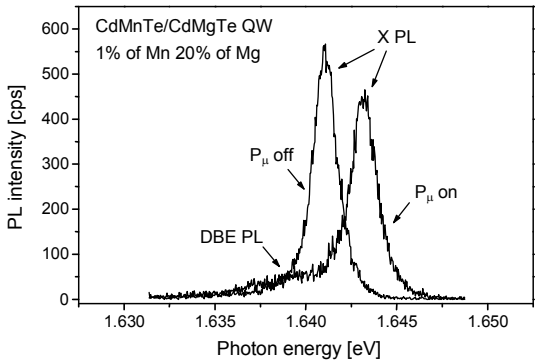

Fig. 5. Response of the QW PL emission of a single QW CdMnTe/CdMgTe structure to the Mn^{2+} magnetic resonance observed at low excitation density of about 0.01 W/cm^2.

covered with a thick CdTe buffer layer. Above band gap excitation (1.92 eV) and low excitation densities were used to avoid lattice heating.

In the ODMR study shown in Fig. 5 we studied PL response to the Mn^{2+} magnetic resonance. At the low excitation density free exciton PL (X PL in Fig. 5) decreases in intensity and shifts up-in-energy by about 2 meV at the Mn^{2+} magnetic resonance (Fig. 5). Such spectral shift of the X PL reflects a significant decrease of the sample magnetization at the magnetic resonance, as was also observed previously [16, 18]. The magnetization quenching is large and reaches 60% at 200 mW microwave power. The shift and decrease in intensity of the X PL is accompanied by the appearance of a weak neutral donor bound exciton (DBE) PL at the low energy wing of the QW PL.

3.3. Auger mechanism of ODMR detection

For bulk CdMnTe samples with about 10% Mn fraction PL of acceptor bound excitons (ABE PL) is observed only at low temperatures and at low magnetic field. At increased magnetic field ABE PL decreases first in the intensity, which is accompanied by an increase in intensity of the FE PL, and then for larger magnetic fields is replaced by the FE PL. Such a property of PL relates to different responses of the two excitonic PL emissions to external magnetic field. For the FE magnetic moments of an electron and hole can freely adjust to the direction of magnetic field. Then, Zeeman splitting for electron and hole adds up. In consequence, we observe large Zeeman splitting for free exciton PL in DMS samples. Thus, FE PL, which comes from the lowest spin sublevel of FE, shifts down-in-energy with increasing magnetic field by a few meV in the system under study.

ABE is an excited state of a neutral acceptor consisting of two holes and one electron. Magnetic moments of two holes are antiparallel. Thus, their contribution to the Zeeman splitting is cancelled. The resulting Zeeman splitting is only due to the electron and thus is smaller than that observed for the FE PL [1]. In consequence, at relatively small magnetic field the lowest spin sublevel of the FE passes the relevant

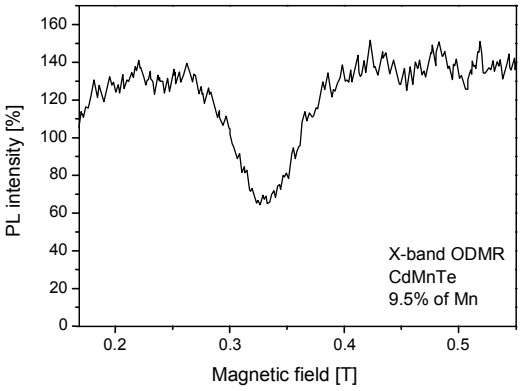

Fig. 6. Response of the "band edge" PL of bulk CdMnTe (9.5% of Mn) to the Mn^{2+} magnetic resonance (in X-band ODMR experiment). PL intensity is scaled as compared to 0 T value (100% intensity).

spin sublevel for the ABE and becomes the lowest excitonic state in the system. Then, the ABE PL becomes energetically unfavorable and is thus replaced by the FE PL. Therefor we can control the relative intensity of the FE and ABE PL by changing the magnetic field.

Field-induced ABE to FE conversion allows us to estimate the efficiency of an Auger-type nonradiative recombination, which is expected to be important for ABE excitons in CdMnTe [23]. We mean here the process in which energy of recombining electron-hole pair is transferred to a third carrier (hole for ABE, electron for DBE) bound at the same center. This third carrier is ionized into continuum of the valence (hole) or conduction (electron) band states [24, 25]. The process is responsible for a very efficient nonradiative recombination of DBE and ABE excitons in indirect band gap GaP and Si [24, 25].

In the experiment shown in Fig. 6, we first quenched the ABE PL by applying sufficient magnetic field. For Q-band ODMR system it is enough to set magnetic field at the Mn^{2+} resonance to deactivate the ABE PL. Thus, we reduce sample magnetization by inducing magnetic resonance of the Mn^{2+} and if the effect is sufficiently large the ABE PL recovers. We applied a large microwave power of 200 mW to induce significant quenching of magnetization, which in fact was large at low excitation density. A 60% magnetization quenching was achieved. This was enough to recover the ABE PL, the process for which we expected efficient nonradiative recombination. In the ODMR experiment we followed the changes in the intensity of the "band edge" PL when tuning magnetic field to the resonance conditions and turning on and off microwave power. The observed decrease of the overall intensity of the "band edge" PL at the Mn^{2+} magnetic resonance is very large. PL is quenched by about 40–50% when the ABE PL recovers on cost of the FE PL. It is a huge ODMR signal, compared to the above mentioned 0.1 to 0.5% PL change of the DAP PL.

The observed magnitude of the PL change only partly relates to a high efficiency of the Auger process for ABE excitons in CdMnTe. Below we introduce another mechanism of ODMR detection in DMS system. We show that carrier heating at the magnetic resonance conditions is very efficient and may strongly affect the rate of excitons formation and recombination.

3.4. Spin flip interactions between free carriers and Mn ions

Inequilibrium hot carriers in DMS samples (*e.g.*, due to nonresonant excitation) can relax their excess energy by energy transfer to magnetic ions [26–35]. In the process hot carriers relax their excess energy by fast spin-flip interactions with Mn^{2+} ions. These spin-flip processes are faster than spin-lattice relaxation processes [28], even in DMS samples with increased Mn fractions. For the latter samples spin-lattice relaxation processes are enhanced, which was explained by a diffusion of spin excitation to regions of an increased Mn concentration [36]. Even for such samples spin-lattice relaxation is too slow to pass efficiently spin excitation energy to the lattice, or to account for dynamics of magnetic polaron formation [37]. Consequently, several Mn^{2+} ions remain spin excited, which results in an increase of their effective spin temperature.

In the experiment shown in Fig. 7 we measured PL (change of intensity or shift of spectral position) of CdMnTe/CdMgTe single QW structure with excitation density varying between 0.01 and 15 W/cm^2. PL spectrum was measured with an external magnetic field set at 1.305 T, *i.e.*, at the Mn^{2+} magnetic resonance field in our Q-band ODMR system. At such a field QW PL is down shifted in the energy, as the consequence of large field-induced sample magnetization.

We observed several effects when excitation density varied between 0.01 and 15 W/cm^2. Increasing excitation density we observed up-in-energy shift of the dominant PL band, which is due to radiative recombination of FE excitons (denoted by X PL in Figs. 7 and 8). Such a shift means that the sample magnetization is reduced at increased excitation densities. We relate this effect to spin flip scattering of hot free carriers on localized spins of Mn^{2+} ions [38], resulting in an increase of an effective temperature of the Mn^{2+} spin subsystem [35, 36].

The increase in the excitation density results also in the appearance of PL of negatively charged excitons (trions) [38]. The trion PL (X^- PL) increases in the intensity with increasing excitation density and also shifts up-in-energy, as the X PL. We relate the increase in the intensity of the X^- PL to two effects. First, formation rate of negatively charged excitons is enhanced at increased excitation densities, which is due to increased concentration of free carriers. Second, spin-flip interactions between free carriers and Mn ions help to change the spin of some of electrons to an opposite one, which is required to form a trion in its singlet state, with two electrons with opposite spin directions. At high excitation densities an additional PL band, attributed to a radiative decay of neutral donor bound excitons (DBE PL), is also seen.

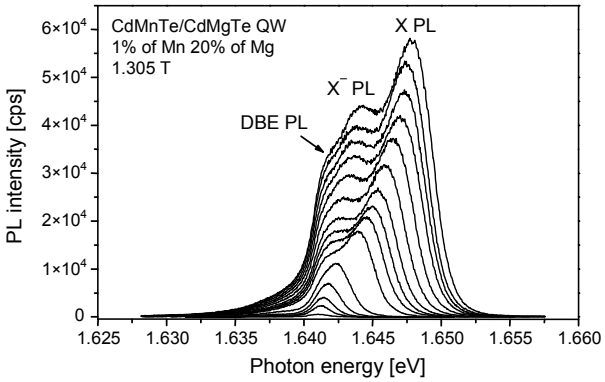

Fig. 7. PL of CdMnTe/CdMgTe QW structure measured with an excitation density varying between 15 and 0.01 W/cm^2 and magnetic field set at the Mn^{2+} magnetic resonance conditions (1.305 T).

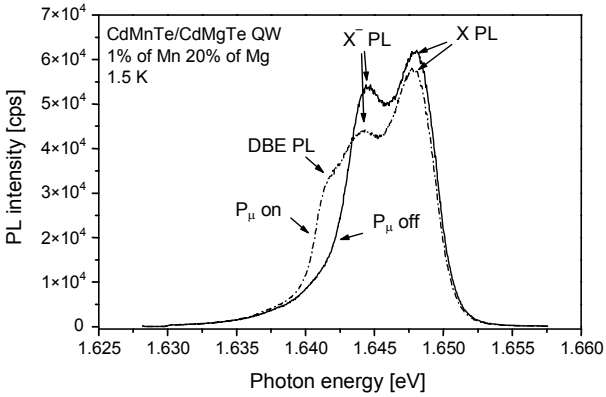

Fig. 8. Response of the QW PL emission of a single QW CdMnTe/CdMgTe structure to the Mn^{2+} magnetic resonance observed for the 15 W/cm^2 excitation density.

In the process described above inequilibrium hot carriers pass their excess energy to a spin subsystem of Mn^{2+} ions, which results in an increase of spin effective temperature. Much less attention was paid to the reverse process of spin relaxation of excited magnetic ions via interaction with free carriers. Several investigations indicated that spin relaxation cannot proceed via an inefficient spin-lattice interaction, mediated by acoustic phonons (see, *e.g.*, [28]). We will demonstrate below that spin-flip interactions between free carriers and Mn ions can be important for DMS samples, for which spin-lattice relaxation is fairly inefficient [36]. Such possibility was already indicated by our previous ODMR investigations [18, 31].

The magnetization-induced spectral shift of the X PL gradually disappears with increasing excitation density and is not observed for density of 15 W/cm^2. Spin-excited

Mn^{2+} ions recombine much faster at increased excitation densities, *i.e.*, in the presence of free carriers. Under such excitation conditions the ODMR signal is detected by only changes in the intensity of the QW PL, as seen in Fig. 8. The three excitonic PL bands do not change their spectral positions at the magnetic resonance. The X and X^- PLs are reduced in intensity, whereas the DBE PL is enhanced once microwave power is turned on at magnetic field set at Mn^{2+} magnetic resonance (Fig. 8). The dominant effect is now related to delocalization of X excitons, which enhances their binding at neutral donor sites and reduces the rate of X^- formation.

A similar PL response we observed setting conditions for the electron cyclotron resonance (CR) in the optically detected cyclotron resonance (ODCR, see, *e.g.*, [39] for the explanation of the technique). The X PL is reduced in intensity and the DBE PL is enhanced once we heat free electrons at the CR conditions. The ODCR investigations indicated that we must delocalize excitons to enhance the DBE formation in CdTe or CdMnTe QWs [40]. The fact that we observe identical PL responses in the ODMR (at high excitation densities) and ODCR indicates that free carriers are heated at the magnetic resonance conditions, similarly to the situation in the ODCR. The scattering of hot carriers (heated at either magnetic resonance or CR conditions) on excitons may result in their delocalization and/or dissociation, observed as changes of the PL intensities. This explains the ODMR-PL spectra observed by us at high excitation densities.

3.5. Concept of time-resolved ODMR

To explain a large decrease of the sample magnetization, observed in the ODMR (see Fig. 5), we must assume that spin lattice relaxation for Mn^{2+} ions (so-called T_1 time) is slow. The dynamics of spin relaxation can be evaluated using a time-resolved ODMR method. In this study, we measured characteristic time scales for magnetic resonance induced spectral shifts of the X PL. We applied pulsed microwave power to follow kinetics of the PL shifts. At low excitation density a characteristic time for PL spectral shifts is about 10 μs, which agrees well with a spin relaxation time (spin-lattice relaxation) in bulk CdMnTe with 1% Mn fraction [36, 37]. This time shortens by factor of a few at increased excitation densities, accounting for differences in the PL response shown in Figs. 5, 7 and 8. The time-resolved experiment thus confirms the significant role of spin flip Mn-free carriers interactions in DMS samples observed at increased excitation densities.

4. Conclusions

We have demonstrated fairly complicated mechanisms responsible for the optical detection of magnetic resonances in the ODMR study of DMS samples. Only at highly controlled experimental conditions we can resolve which of the mechanisms discussed above dominates. Their relevant importance depends on many parameters,

including, *e.g.*, excitation density, Mn fraction, character of PL transitions studied, *etc*. The present study indicates that spin flip interactions between Mn ions and free carriers are very efficient.

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