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Radiative and nonradiative recombination kinetics of indirect bound excitons studied by time-resolved level anticrossing experiments

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Abstract
Time-resolved investigation of the level anticrossing effect in exciton emission is first used to determine separately the radiative and nonradiative indirect bound exciton lifetimes in a semiconductor crystal using GaSe as an example. A theoretical treatment of the experimental level anticrossing signal is carried out for different instances of the indirect bound exciton lifetime. The indirect bound exciton radiative lifetime $\tau_r = 4.8\ \mu$s is found to be about five times shorter than the nonradiative lifetime $\tau_0 = 19\ \mu$s and about two orders of magnitude longer than the radiative lifetime of direct bound excitons in the same compound. Within the two-level scheme, the value of the indirect bound exciton spin relaxation time T_1 is assessed to be 100 μs.

1. Introduction

Crystal defects serve as effective centres of radiative and nonradiative electron–hole recombination. The corresponding processes influence appreciably the optical and electrical properties of semiconductors. In particular, annihilation of excitons bound to crystal defects contributes largely to low-temperature emission in real crystals [1]. An important characteristic of the annihilation process is the radiative lifetime of the emitting state. To determine the bound exciton (BE) radiative lifetime, Thomas and Hopfield [2] suggested a procedure based on measuring BE optical absorption along with evaluating the concentration of the defects which bind excitons. However, measuring BE optical absorption is a hard problem in the case of thin films and/or indirect BEs which are characterized by low absorptance. In the circumstances, procedures based on the analysis of BE emission become of special interest. There are two experimental methods commonly used in emission spectroscopy for lifetime measurements: time-resolved spectroscopy and the phase-modulated technique. (The modification of the latter, known as quadrature frequency-resolved spectroscopy [3], has been used successfully for exciton lifetime measurements in amorphous semiconductors [4, 5].) However, the traditional experimental methods for measuring exciton lifetimes provide information on the *total* lifetime of excitons [4–14]. To determine the exciton radiative lifetime in this situation, additional knowledge of the exciton nonradiative lifetime or of the ratio between the exciton radiative and nonradiative lifetimes is required. This complementary information has to be gleaned either from further experiments (e.g. measuring the quantum efficiency of exciton emission) or from a theoretical treatment of radiative and nonradiative processes in the system under investigation [6–13, 15]. At the same time, it has recently been shown that, taken alone, a time-resolved study of the level anticrossing effect in BE emission can yield information on both radiative and nonradiative lifetimes of the excited states [16]. In this work, the procedure described in [16] was first applied to assess the radiative lifetime of indirect BEs in semiconductor crystals in the specific case of GaSe.

2. Experimental results

The GaSe crystals were Bridgman-grown and were not doped intentionally. The samples for the study were cleaved from

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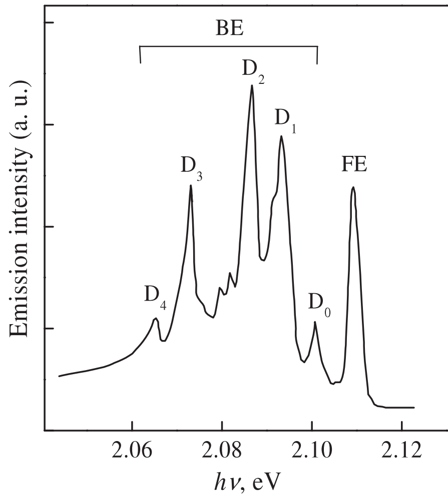

Figure 1. Emission spectrum of a type I GaSe crystal under band-to-band CW excitation: $T = 2$ K.

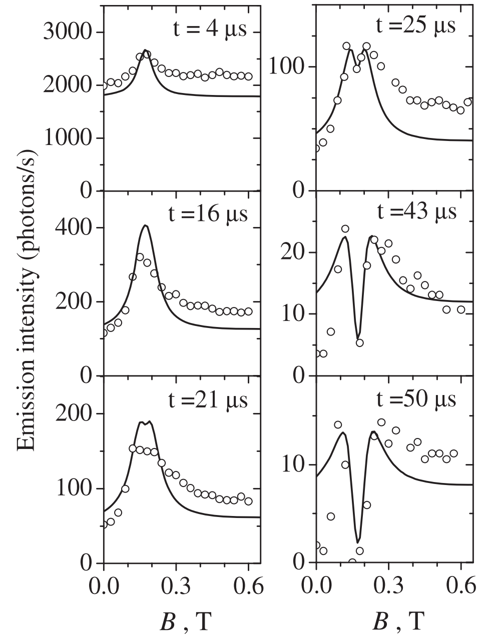

Figure 2. Level anticrossing signal in the D_1 indirect bound exciton emission, $I_{\sigma^-}(B, t)$, measured at different times t during the excited-state lifetime. The time t is specified in the figure. The points are experimental data, and the solid lines are plots of the theoretical relation (7).

ingots perpendicular to the crystal optical axis c. The exciton luminescence was excited by a pulsed copper-vapour laser with a pulse duration $\tau_p = 20$ ns. The excitation density was about 200 W cm^{-2}. Pump light with a photon energy $h\nu_{exc} = 2.428$ eV $> E_g$ (where E_g is the crystal bandgap) was incident on the crystal at a small angle to the sample surface normal, and the radiation was detected along the normal, i.e. parallel to the optical axis. The spectra were measured by a grating spectrometer operating in photon counting mode with a time resolution of ~30 ns. The radiation was scanned over the exciton lifetime by appropriately gating the photon counting system with respect to the pump pulse. During the experiment, the samples were kept immersed in liquid helium at 2 K. The magnetic field was generated by a superconducting coil. The field was oriented parallel to the crystal optical axis.

In GaSe crystals, the absolute maximum of the valence band lies at the centre of the Brillouin zone. The conduction band is characterized by two minima, close in energy: one is at the centre of the Brillouin zone; another is at the border of the zone (at the M point) [17]. The edge emission spectra of GaSe, at liquid helium temperature, consist primarily either of the emission of indirect BEs (type I GaSe crystals) or of the emission of direct BEs (type II GaSe crystals), depending on crystal imperfections. In this study, type I GaSe crystals were used.

Figure 1 presents the GaSe emission spectrum in the region of the fundamental absorption edge, which was obtained under conditions of continuous wave (CW) excitation. The shortest-wavelength line FE (free exciton) of the spectrum at $h\nu = 2.109$ eV is derived from the annihilation of direct free triplet excitons. (A direct free singlet exciton emission is polarized with $\mathbf{E} \parallel c$ and does not contribute to the spectrum in figure 1 which corresponds to $\mathbf{E} \perp c$ emission polarization.) The intense lines D_j ($j = 0, 1, 2, \ldots$), in the spectral range 2.105–2.060 eV, are assigned to the annihilation of indirect triplet excitons bound to ionized centres (or isoelectronic traps) [18, 19]. The D_0 line corresponds to zero-phonon transitions; the longer-wavelength lines are phonon replicas of it. Indirect zero-phonon optical transitions between electron states located at different Brillouin-zone points are forbidden by the quasi-momentum conservation rule. In the case of indirect exciton transitions, the 'forbiddenness' is lifted in part by the promotion of phonons involved in the transitions. The presence of the zero-phonon BE line, D_0, is explained by a breakdown of lattice periodicity in the vicinity of the crystal defect, which relaxes the quasi-momentum conservation relations. In either case, the probability of indirect exciton optical transitions is low, so that the indirect BE transitions in the GaSe absorption spectrum are not observed.

When placed in a longitudinal magnetic field $B \geqslant 2$ T ($\mathbf{B} \parallel c \parallel \mathbf{k}_{photon}$ is the Faraday geometry), the BE lines split into doublets, whose components are strictly right (σ^+) or left (σ^-) circularly polarized [18, 19]. While in weaker fields no splitting of the lines is observed because of their relatively large widths, the magnetic-field dependence of the exciton steady-state emission intensity exhibits a hump [20], which is due to the BE Zeeman sublevel anticrossing in the magnetic field. (A level-anticrossing-produced singularity in the magnetic-field dependence of the emission intensity is generally called a level anticrossing signal [21].)

The steady-state signal $I(B)$ is made up of level anticrossing signals $I(B, t)$ which arise from excitons generated at different times t preceding the signal registration: $I(B) = \text{const} \cdot \int_0^\infty I(B, t)\, dt$. With the purpose of determining the radiative and nonradiative lifetimes of the indirect BEs, the temporary evolution of the level anticrossing signal, $I(B, t)$, was studied. The shape of the indirect BE Zeeman sublevel anticrossing signal at different instances of the excited-state

lifetime was found to differ substantially from the shape of the signal observed under CW excitation. Figure 2 shows $I_{\sigma^-}(B,t)$ dependences of the D_1 line ($h\nu_1 = 2.093$ eV) intensity in σ^- polarization ($0 \leqslant B < 1$ T, $\mathbf{B} \parallel c \parallel \mathbf{k}_{\text{photon}}$) obtained at various delay times t with respect to the excitation pulse. (The magneto-optical effects on all the D_i lines ($i = 0, 1, 2, \ldots$) have the same pattern, so we shall restrict ourselves in what follows to an analysis of the D_1 line behaviour.) The detection gate width was $\Delta t \approx 30$ ns. As seen from figure 2, at $t = 4$ μs the $I_{\sigma^-}(B,t)$ plot exhibits a weak maximum at $B = 0.169$ T $\equiv B'_c$. (For $t \approx 0$, the maximum in $I_{\sigma^-}(B,t)$ is practically indistinguishable.) As the delay time is increased to 16 μs, the relative intensity of the maximum increases (although the absolute D_1-line intensity measured at $B = 0$ naturally decreases with increasing delay), to produce a pronounced peak in the $B = B'_c$ region (see figure 2). Thus, in this delay-time interval the $I_{\sigma^-}(B,t)$ relation obtained at a fixed t is similar to the $I_{\sigma^-}(B)$ dependence of the D_1-line σ^--emission intensity observed in the case of CW luminescence excitation. As the delay time is progressively increased, a shallow dip forms in the region of the peak maximum (figure 2). A further increase in t entails an increase in the dip width and depth, so that at $t = 43$ μs the $I_{\sigma^-}(B,t)$ dependence already exhibits two well-resolved peaks, whose separation continues to grow with increasing t (figure 2). Thus, the experimental data presented demonstrate that the shape of the level anticrossing signal measured at different instants within the BE lifetime essentially varies.

3. Discussion

To account for the magneto-optical effects that are observed, consider the energy structure of the indirect BE in gallium selenide. From an analysis of the Zeeman effect for the BE it follows that, for $B > 2$ T, the energy structure of the indirect BE emitting states is similar to that of a triplet direct exciton [18]. In GaSe crystals, the ground state of the free direct exciton is split by exchange interaction into two states: a singlet state and a triplet state [22]. For free excitons, the splitting between these states is $\Delta_1 = 2$ meV [22]. The total electron and hole spin in a singlet exciton is zero, and transitions to this state are allowed for radiation polarized with $\mathbf{E} \parallel c$. In our experiment ($\mathbf{E} \perp c$), the singlet state does not manifest itself. Triplet excitons have a total spin of $S = 1$ and spin projections on the c axis of $S_z = 0, \pm 1$. Transitions to the $S_z = \pm 1$ states are allowed in the $\mathbf{E} \perp c$ polarization, while the $S_z = 0$ state is optically inactive. Because of the crystal anisotropy, the $S_z = 0$ state is split from the $S_z = \pm 1$ states by an amount $\Delta \ll \Delta_1$ (figure 3). A longitudinal magnetic field $\mathbf{B} \parallel c$ splits the $S_z = \pm 1$ level into two sublevels with $S_z = +1$ and $S_z = -1$, which yield right-hand and left-hand circularly polarized emissions, respectively. The energies of the triplet exciton states in a longitudinal field are given by the expressions

$$E_{1,2} = E_0 \pm 0.5 g_{zz} \mu_0 B, \qquad E_3 = E_0 - \Delta, \tag{1}$$

where g_{zz} is the longitudinal component of the exciton g-factor, and μ_0 is the Bohr magneton. According to (1), in the field $B = 2\Delta / g_{zz}\mu_0 \equiv B_c$, the energies of states 2 and 3 become equal, i.e. the corresponding energy levels cross (figure 3). We further assume the existence in the crystal of a perturbation (i.e. a local field near the defect) V which mixes the exciton states $|0\rangle$ and $|\pm 1\rangle$. The existence of a static perturbation mixing various spin states of BEs is suggested by observation of spin sublevel anticrossing of triplet BEs in a magnetic field [23, 24] as well as by magnetic resonance experiments [25, 26]. In this case, the crossing of levels 2 and 3 will be replaced by their anticrossing (figure 3). For $g_{zz}\mu_0 B \approx 2\Delta$, one will observe the mixing primarily of states 2 and 3 (i.e. states $|0\rangle$ and $|-1\rangle$), because the energy difference between these states will be substantially smaller than that between states 1 and 3. Taking into account the perturbation V, the wavefunctions $\Psi_{\alpha,b}$ of the states originating from states 2 and 3 can be written as

$$\Psi_a = C_2\Psi_2 + C_3\Psi_3, \qquad \Psi_b = C_3\Psi_2 - C_2\Psi_3. \tag{2}$$

Recalling that state 3 is optically inactive, one finds that the radiative transitions from states a and b are polarized in the same way as those from state 2. The coefficients $C_{2,3}$ have the form

$$C_{2,3}(B) = \frac{1}{\sqrt{2}}\left\{1 \pm \frac{\Delta' - 0.5 g_{zz}\mu_0 B}{[(\Delta' - 0.5 g_{zz}\mu_0 B)^2 + 4|V_{23}|^2]^{0.5}}\right\}^{0.5}, \tag{3}$$

where $V_{ik} = \langle\Psi_i|V|\Psi_k\rangle$ $(i,k = 2,3)$, $\Delta' = \Delta + V_{22} - V_{33}$. The fraction of the optically active state 2 in the wavefunctions (2) determines the radiative lifetimes $\tau_{ar,br}$ of the states of triplet excitons with wavefunctions $\Psi_{a,b}$: $\tau_{ar}(B) = [C_2^2(B)\tau_r^{-1}]^{-1}$, $\tau_{br}(B) = [C_3^2(B)\tau_r^{-1}]^{-1}$, where τ_r is the radiative lifetime of state 2. Assuming the nonradiative lifetime of bound triplet excitons τ_0 to be independent of their spin state, we obtain for the total effective lifetimes of states a and b: $\tau_a(B) = [\tau_{ar}^{-1}(B) + \tau_0^{-1}]^{-1}$, $\tau_b(B) = [\tau_{br}^{-1}(B) + \tau_0^{-1}]^{-1}$. Taking into account that the radiative decay of an indirect BE can occur with the assistance of different phonons and through phononless optical transitions, the probability of the decay can be written as $\tau_r^{-1} = \sum_i \tau_{r(j)}^{-1}$, where $\tau_{r(j)}^{-1}$ is the probability of indirect exciton radiative decay with the promotion of the

Figure 3. Energy level diagram of the triplet exciton in GaSe in the presence of a magnetic field: $\boldsymbol{B} \parallel c$.

jth phonon ($j = 0, 1, 2, \ldots$; $j = 0$ corresponds to the zero-phonon optical transition). The relation between $\tau_{r(j)}$ and τ_r can be written in the form:

$$\tau_{r(j)}^{-1} = A_j \tau_r^{-1}, \quad (4)$$

where $A_j = \tau_{r(j)}^{-1}/\tau_r^{-1}$ is the relative probability for the exciton to radiatively decay with the promotion of the jth phonon. In the indirect BE emission spectrum (figure 1), parameters A_j determine the relative intensities of the D_j lines ($j = 0, 1, 2, \ldots$). When the crystal is excited by unpolarized light with $h\nu_{exc} > E_g$, and neglecting the exciton spin relaxation, the intensities of the emission components $I_i^{(j)}(B,t)$ ($i = a, b$) of the D_j line can be written as

$$I_i^{(j)}(B,t) = A_j n_i(B,t)\tau_{ir}^{-1}(B), \quad (5)$$

with

$$n_i(B,t) = n_i(B,0)\exp[-t/\tau_i(B)]. \quad (6)$$

Here the initial population of the ith state, $n_i(B,0)$, is assumed to be proportional to the exciton generation rate, independent of B, and is the same for all i. (Equation (6) was derived under the assumption that the excitation pulse duration $\tau_p \ll \tau_i(B)$.) Because optical transitions from states a and b are not spectrally resolved, one observes experimentally the total emission

$$I_{\sigma^-}^{(j)}(B,t) = I_a^{(j)}(B,t) + I_b^{(j)}(B,t). \quad (7)$$

The $I_{\sigma^-}^{(j)}(B,t)$ dependence calculated using equation (7) is in good qualitative agreement with the experimental results. It is essential that, starting from a certain value of $t = 4\tau_r$, the calculated dependence exhibits two maxima separated by a minimum (dip) at $B = B_c'$, whose relative depth increases with t. This allows direct measurement of the exciton radiative lifetime: $\tau_r = 4.8 \times 10^{-6}$ s. If the exciton radiative lifetime τ_r is established, then the exciton nonradiative lifetime τ_0 can easily be evaluated using the relation:

$$\ln I_{\sigma^-}^{(j)}(B_c', t) = \text{const} - 0.5(2\tau_0^{-1} + \tau_r^{-1})t. \quad (8)$$

The experimental $\ln I_{\sigma^-}^{(j)}(B_c', t)$ dependence (for $j = 1$) and its approximation by equation (8) with $\tau_0 = 18.6 \times 10^{-6}$ s are presented in figure 4. (Notice that the analysis of the steady-state level anticrossing signal $I_{\sigma^-}^{(j)}(B) = \text{const.}\int_0^\infty I_{\sigma^-}^{(j)}(B,t)\,dt$ yields information on the $\tau_r\tau_0$ ratio only.) The other parameters of the model can also be easily found practically independently of each other. At a given g_{zz}, Δ' determines the position of the peak (for $t = 4$, 16 μs) or of the dip (for $t \geqslant 21$ μs) in the $I_{\sigma^-}^{(j)}(B,t)$ dependence (figure 2); $|V_{23}|$ is responsible for the peak width. The theoretical curves in figure 2 were obtained for the parameters $g_{zz} = 3.6$ [18], $\tau_r = 4.8 \times 10^{-6}$ s $\cong \tau_{ar}(0)$, $\tau_0 = 19 \times 10^{-6}$ s, $\Delta' = 0.018$ meV, and $|V_{23}| = 0.002$ meV.

In principle, indirect BE spin relaxation can also influence the population of states a and b. In this case, within the framework of the two-level scheme under consideration, the magnitudes of $n_a(B,t)$ and $n_b(B,t)$ can be obtained by

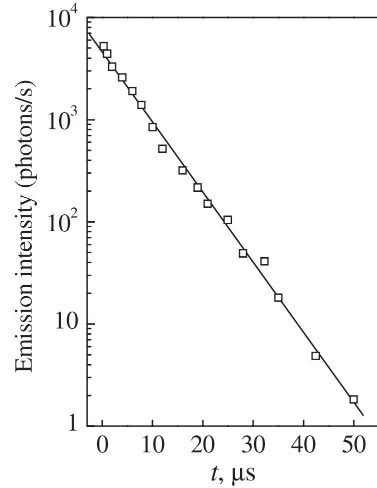

Figure 4. The temporary dependence of the intensity of the D_1 indirect bound exciton emission line, $I_{\sigma^-}(B,t)$, measured at a fixed value of $B = B_c'$. The points are experimental data, and the solid lines are plots of the theoretical relation (8).

solving the appropriate rate equations [27]. Taking indirect BE spin relaxation into account, the best fit of the calculated $I_{\sigma^-}^{(j)}(B,t)$ dependence to the experimental data in question was obtained with the effective spin–lattice relaxation time $T_1 = 100 \times 10^{-6}$ s $\gg \tau_r, \tau_0$. If $T_1 \gg \tau_r, \tau_0$, the calculated values of $I_{\sigma^-}^{(j)}(B,t)$ differ little from the values calculated using equation (6).

Thus, taken alone, the study of the level anticrossing signal in the time-delayed indirect bound exciton emission can yield information about both the radiative and nonradiative lifetimes of the excited states. It should be noted that in the present work we do not consider any microscopic mechanisms of the indirect triplet bound exciton radiative decay, and hence we do not calculate a triplet exciton radiative lifetime by itself. Therefore we cannot apply the recent theory [15] and assess its validity for the case presented in this paper.

GaSe crystals present a unique possibility to compare the radiative and nonradiative lifetimes of direct and indirect BEs of approximately equal energies in the same compound. As one would expect, the comparison shows that the radiative lifetime of indirect triplet BEs far exceeds the respective lifetime of direct triplet BEs [16]. The radiative lifetime of the indirect BEs is found to be about 50 times longer then that of the direct BEs, while their nonradiative lifetimes differ to an essentially smaller extent (19 μs and $\sim$7 μs, respectively). We attribute the relatively large radiative lifetime of indirect BEs to the 'forbiddenness' of the indirect optical transitions, as mentioned above. The total lifetime of the direct BEs is determined by their radiative lifetime rather than by their nonradiative lifetime (because of the condition $\tau_r \ll \tau_0$). In the case of the indirect BEs, their radiative and nonradiative lifetimes are comparable, so that their total lifetime is essentially dependent on both τ_r and τ_0.

It is rewarding to compare the ratio between the radiative lifetimes of indirect and direct BEs, as measured in GaSe, with that in other semiconductors. The energy separation between direct and indirect minima of the conduction band

of a semiconductor can appreciably influence the radiative lifetime of indirect BEs (varying the separation changes the fractions of electron states with different wavevectors in the bound electron wavefunction [28]). From this point of view, of interest, in our case, are perfect GaAs crystals where the variation of the band structure from direct gap to indirect gap can be induced under hydrostatic pressure, and both direct and indirect BE emission lines can be detected in a certain pressure range. So, if both direct and indirect minima of the conduction band in GaAs are close in energy (like GaSe), the ratio between the radiative lifetimes of indirect and direct BEs was found to be >20 [29], which is in agreement with the result obtained above for GaSe. Though the ratio relates to the properties of *singlet* BEs in GaAs, the prohibition of intercombined optical transitions can be supposed to affect the annihilation probabilities of both direct and indirect triplet BEs to approximately the same extent. The lifetimes of triplet BEs in GaSe are about two orders of magnitude longer than the lifetimes of singlet BEs in GaAs, and nearly the same relationship between the lifetimes of triplet and singlet BEs was found in GaP crystals [8].

4. Conclusion

A time-resolved study of the level anticrossing signal in the indirect BE emission of a type I GaSe crystal has been carried out. The study has permitted the first determination of the individual values of the radiative and nonradiative lifetimes of indirect triplet BEs by means of emission spectroscopy. It has been demonstrated that, in the specific case of GaSe, the radiative lifetime of indirect BEs differs from that of direct BEs by about two orders of magnitude, with their nonradiative lifetimes being of the same order.

Acknowledgment

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