phys. stat. sol. (b) **189**, 247 (1995)

Subject classification: 71.35 and 78.55; S8.11

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Influence of Surface Electric Field on Exciton Photoluminescence of CdS

Self-Reversal of Spectral Lines

By

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The structure of luminescence spectra of free excitons in CdS single crystals at $T = 77$ K is investigated. The dip formation is found in the top part of free A and B exciton resonant peaks of photoluminescence (PL) spectra after heating of the CdS crystals followed by fast cooling down to 77 K. Similar effects are observed when an external electric field is applied. It is shown that the structure of exciton PL spectra is due to self-absorption of resonance radiation in the near-surface layer with low exciton concentration. Our calculations demonstrate that the formation of an exciton concentration gradient may be caused by the influence of a surface electric field on the binding of electron-hole pairs to excitons. It is assumed that the increase of surface electric fields after heat treatment is caused by the thermostimulated adsorption of oxygen, which results in the formation of electron trap levels.

Изучена структура спектров люминесценции свободных экситонов в CdS при $T = 77$ K. Обнаружено возникновение провалов на резонансных полосах после нагревания образцов от $T = 77$ K до 295–395 K с последующим быстрым охлаждением, а также в результате приложения внешнего электрического поля. Показано, что структура спектров люминесценции обусловлена самопоглощением резонансного излучения в приповерхностном слое с низкой концентрацией экситонов. Результаты расчетов показывают, что образование градиента концентрации экситонов может быть вызвано влиянием поверхностного электрического поля на связывание носителей заряда в экситоны. Предполагается, что увеличение поверхностного электрического поля при термообработке обусловлено термостимулированной адсорбцией кислорода, которая приводит к возникновению уровней захвата электронов.

1. Introduction

The exciton states are one of the fundamental phenomena in semiconductors. Their properties are determined by the basic parameters of crystals: significance of effective masses, dielectric constants, band gap width. The exciton spectroscopy (reflection, absorption, and luminescence) is a rather sensitive method to study the surface and bulk properties of semiconductors: a surface state, the exciton-free layers, the defect formation processes under various kinds of external actions. The observed PL spectra of free excitons are transformed because of the influence of polariton effects or absorption reversal effects in a bulk crystal.

In previous works [1, 2] it has been shown that in II–VI crystals, which have a sphalerite structure, the PL spectra of free excitons have a dip in the short-wave region relative to the maximum of the resonant band. The formation of such spectral structure was explained

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by the annihilation of polaritons from the top and bottom branches of the dispersion curve, by polariton effects, by reabsorption or self-absorption of exciton radiation [2 to 4] because in line with perturbation theory these crystals have narrow absorption peaks. The authors of [1] have made the conclusion that in CdS crystals the above-mentioned effect cannot take place since due to polariton effects the absorption spectrum has a flat trapezium shape in the top part.

In the present work the results of a detailed investigation of the found phenomenon of self-reversal of free exciton lines in the PL spectra of CdS single crystals after low-temperature heat treatment are included.

2. Experimental

In the CdS single crystals PL was excited by the light of continuous wave lasers and lamps ($\lambda = 325, 365, 436, 441.6$ nm). The power density of excitation was varied in the range of 0.01 to 1 W/cm². The free exciton emission was registered as viewed from the illuminated surface of a sample. For registration we used the grating monochromator MDR-23 with linear dispersion 1.3 nm/mm. The emission intensity was registered by using a photomultiplier.

The CdS crystals were flat parallel plates with surfaces, oriented parallel to the following planes: (0001), (11 $\bar{2}$ 0), or (10 $\bar{1}$ 0). To obtain reproducible surface conditions mechanical and chemical polishing or cleaving was used.

The electro-optical experiments were carried out by using optical quartz glasses covered with thin conducting SnO₂ layers as the first (field-) electrode. The resistivity of the SnO₂ layers was about 5 k Ω /cm. The copper foil was used as the second electrode pressed against the sample with a 2 mm thick teflon spacer. This arrangement was mounted into a cryostat and cooled to liquid nitrogen temperature. A dc high voltage of up to 25 kV was applied to the electrodes. The temperature of the sample was measured with the help of a thermocouple. By heating the cryostat we could obtain any temperature from 77 to 300 K. The temperature was kept constant by using a thermostat system. During PL spectra recording the temporal instabilities of the output of lasers and lamps were controlled, and we took also into account the absorbance of the SnO₂ layer.

3. Results

At the excitation of photoluminescence by the continuous emission of a He-Cd laser ($\lambda = 441.6$ nm, $P = 0.01$ W) there are only spectral bands of A and B free excitons in the PL spectra of initial high-resistance CdS crystals ($\rho > 10^6 \Omega$ cm) (solid curve 1 in Fig. 1). The spectra of low-resistance samples show also the band I₂ of bound excitons (solid curves 2, 3 in Fig. 1). The heating of crystals from $T = 295$ to 390 K followed by fast cooling down to liquid nitrogen temperature results in a dip formation in the contours of free exciton resonant lines (dotted curves in Fig. 1). The considerably deeper dips form as a result of heating the samples from $T = 77$ K to room temperature and fast cooling down to $T = 77$ K. In low-resistive samples the dip value is bigger than in high-resistive ones (Fig. 1). Also, the dip value is greater at the polar surfaces (0001) and (000 $\bar{1}$) comparable to the prismatic ones (10 $\bar{1}$ 0) and (11 $\bar{2}$ 0). The small dips (5 to 10% of the maximum intensity) may also form as a result of cleaving the samples in air or even in liquid nitrogen.

Fig. 1. PL spectra in the exciton region for CdS crystals with various values of conductance. Solid curves initial spectra; dotted curves after heat treatment

The dip value depends on the excitation level (I_{ex}). As the excitation intensity increases, the dip value rises up to its maximum and then decreases. The band profile becomes smooth again at $I_{ex} \approx 1 \text{ kW/cm}^2$ when the concentration of the electron-hole pairs and excitons is still small. Therefore, it may be concluded that the disappearance of a dip with increasing I_{ex} is not connected with the destruction of exciton states.

As time passes after the heat treatment and measurement, the dip values decrease and the initial spectrum shape is restored. The time taken for dip relaxation depends on the dip size and takes from several hours to several days. The formation of the dip does not depend on the polarization of the excitation emission, but its position on the contour of the spectral bands is determined by the relative orientation of vectors E and C . When $E \perp C$, the dip is localized either at the center of the PL band or slightly shifted to the short-wave region. When $E \parallel C$, it is placed in the long-wave region. The intensity and the shape of the spectral band of bound excitons as well as the reflection spectra do not change when there is a dip in the free exciton peak.

To elucidate the role of polariton effects in the formation of the structure of the radiation band of free excitons, the PL and reflection spectra were studied in the temperature region of 77 to 300 K in 5 to 10 K steps. The measurements have shown that one can find the dip in the free exciton resonant band up to $T = 130 \text{ K}$ as long as the exciton structure in the spectrum is distinctly observed (Fig. 2). So, in this case, we may conclude that polariton effects play no part since at such high temperatures they are insignificant [5, 6].

4. Discussion

The origin of the dip in the maximum of the resonant bands of exciton PL in CdS is largely similar to the self-reversal of spectral lines in the atomic spectroscopy [7]. The

Fig. 2. PL spectra of CdS crystals in the free-exciton region at various temperatures (after heat treatment)

radiation at the resonant frequency is subjected to strong absorption and in place of the smooth contour of the PL band the dip arises, if the light passes through layers with a small concentration of radiating atoms but with capability of light absorption [8, 9].

The absorption factor of wide band gap semiconductors in the region of the exciton resonance emission takes on very large values, 10^4 to 10^5 cm^{-1} [1]. The absorption and reflection spectra in such crystals are well interpreted in the framework of classical exciton theory or with help of the polariton model for pure samples at low temperatures. However, owing to a large value of the absorption factor, the reabsorption

and the resonance self-absorption of the luminescence light deform the shape of the free exciton spectral band [1, 2].

In [8] it has been shown that in CdS crystals the rate of exciton generation caused by the absorption of photons, radiated by free excitons, is negligibly small in comparison with the rate of generation of free excitons through electron-hole binding. Hence, the reabsorption of photons is a minor effect compared with their self-absorption and, in this case, the reabsorption can be disregarded.

A photon appearing with exciton recombination in CdS has sufficient energy for self-absorption. The formation of an exciton concentration gradient can be caused by the influence of the surface.

Because the reflection spectra of CdS in the exciton region at low-temperature heat treatment do not change, it is possible to assume that the change of surface exciton annihilation processes does not play a large role in this case. Therefore, the dip formation in the contour of the free exciton emission band cannot be stimulated by the occurrence of a defective ("dead") layer near the surface [10]. It has been shown experimentally [11] that the thickness of such a layer (about the radius of an exciton) can be caused by various effects, such as additional illumination, surface electrical fields with intensity more than the threshold of the exciton ionization ($F_{cr} \approx 10^5 \text{ V/cm}$ for CdS [12]), etc. The effect of self-absorption in such a layer is impossible because a significant part of exciton states is ionized, hence, there is no exciton absorption.

As our measurements of reflection spectra show, as well as measurements of photo-reflectance spectra from [13], heating in air to temperatures below 500 K does not result

in any structural changes in the near-surface areas. However, after the low-temperature heat treatment new surface states can occur, which are due to chemisorption of oxygen molecules [13]. If the surface of the crystal has been cooled fast, the high-temperature concentration of oxygen is “frozen”, that results in the change of conductivity of near-surface areas [14]. In this case, it is considered that the change of surface charge is caused by trapping of electrons in acceptor surface levels from the conduction band in low-resistance crystals [15, 16] or photoelectrons of high-resistance samples at optical excitation [13].

Thus, after low-temperature heat treatment, owing to thermostimulated adsorption of oxygen, at the sample surface an additional bending of energy bands arises. In this case the surface electrical field increases, but its value remains less than F_{cr} (this is evident from the analysis of reflectance spectra). As a result the motion characteristics of charge carriers change in the near-surface areas and, as a consequence, parameters which determine the probability of formation of exciton states change, too. Therefore, in this area a gradient of exciton concentration can arise outward from the surface and, thus, a layer with lowered concentration of radiating centers can be formed.

The experimental and theoretical study of the influence of adsorption on exciton luminescence spectra [17] shows that the actions of both adsorption and external electrical field (normal to the surface of a sample) under certain conditions lead to the same effects. To check our assumption concerning the field mechanism of dip formation we investigated the influence of an electrostatic external field of various polarity on the form of a free exciton emission band of CdS single crystals at liquid nitrogen temperature. To observe a self-absorption effect of exciton emission it is necessary that the depth of field penetration in the crystal should be less than or equal to the diffusion length of the exciton; then for measurements we used samples with rather large conductivity ($\rho \approx 10 \Omega \text{ cm}$). In Fig. 3 the results of these measurements are shown. For a series of samples the application of a negative field to the field electrode resulted in the formation of a dip in the contour of the band (curve 2 in Fig. 3). When the field polarity was changed a reduction of the dip depth was observed with increasing field intensity (curve 3 in Fig. 3). After achievement of the field value $F = 18.8 \text{ kV/cm}$ the dip disappeared completely (curve 4 in Fig. 3). Thus, the offered model of the formation of a dip may be considered as qualitatively correct.

We could not find the formation of a dip when an electrical field was applied to the surface of a high-resistance sample that was, obviously, due to the large value of the Debye shielding length of the electrical field. The dip, caused by the low-temperature heat treatment in such samples, is very small in comparison with that in low-resistance samples as already mentioned above.

With interaction between oxygen molecules and the surface of the crystals, the free energy of the surface and the accommodation factor show strong anisotropy relative to the crystallographical orientation of the surface and the degree of chemisorption [18, 19], that can be caused by the distinction of the packing density of faces and the pyroelectric properties at sample heating. As the polar planes (0001) and (000 $\bar{1}$) are the most active ones in terms of chemisorption, the dip at such orientation should be largest in comparison with other faces.

The evaluation of the levels of excitation used in our experiments shows that at the created concentration of carriers degeneracy does not take place. Thus, the radiation lineshape is described in the stationary case and in one-coordinate approximation by the following equation [20]:

$$I_{PL} = C(v) T(v) \int_0^d n_{ex}(x) \exp(-k(v)x) dx. \quad (1)$$

Fig. 3. PL spectra of low-resistance CdS crystal under applied dc electric fields of different strength. (1) $F = 0$, (2) -19.3 , (3) 10 , (4) 18.8 kV/cm

(The crystal has the form of a flat parallel plate, bounded by planes $x = 0$ and $x = d$. The light of frequency ν is incident perpendicularly on the plane $x = 0$ from the area $x < 0$.)

Here $n_{\text{ex}}(x)$ is the concentration of excitons at a point with coordinate x , $k(\nu)$ the absorption factor, $T(\nu)$ the transmission factor of light of frequency ν , incident from the inside on the crystal surface, $C(\nu)$ is related to the probability per unit time of emitting a photon with frequency ν at exciton annihilation.

To calculate the function $n_{\text{ex}}(x)$ the interaction between two subsystems was studied: a gas of free charge carriers and excitons in the presence of an electrostatic surface field. Electronic processes in such systems are described by four equations: three nonlinear differential equations of continuity for electrons, holes, and excitons and Poisson's equation. The general analysis of such a system is very complicated, so we have used the following approaches and simplifications to find the distribution function:

1. the excitation level has a value such that there is no degeneration and the concentrations of photogenerated carriers (δn and δp) are far less than the concentration of donors (n_0) in every point of the sample;
2. in low-resistance samples of CdS ($n_0 \approx 10^{21}$ to 10^{22} m^{-3}) the distribution of majority carriers (electrons) can be approximately calculated from Boltzmann's equation $n(x) =$

$n_0 \exp(e\varphi(x)/k_B T)$. In this case the electric potential distribution $\varphi(x)$ is practically independent of the processes of electron and hole photogeneration in the space-charge region [21] ($e\varphi < 0$ and $\varphi < 0$ at upwards bending of the energy bands).

Then, for the space-charge layer in the steady case, the given system of equations takes the following form:

$$I_0 k_0 \exp(-k_0 x) - \mu_p F(x) \frac{dp(x)}{dx} - \mu_p p(x) \frac{dF(x)}{dx} + D_p \frac{d^2 p(x)}{dx^2} - \gamma_e n(x) p(x) + (\gamma_F(x) + \alpha_e) n_{ex}(x) = 0, \quad (2)$$

$$\gamma_e n(x) p(x) - (\gamma_F(x) + \alpha_e) n_{ex}(x) - \frac{n_{ex}(x)}{\tau_{ex}} + D_{ex} \frac{d^2 n_{ex}(x)}{dx^2} = 0, \quad (3)$$

$$\frac{dF(x)}{dx} = \frac{e}{\varepsilon \varepsilon_0} (n(x) - n_0); \quad n(x) = n_0 \exp\left(\frac{e\varphi(x)}{k_B T}\right), \quad (4)$$

where I_0 is the generation intensity of electron–hole pairs, k_0 the absorption factor at the frequency of excitation emission, μ_p , D_p are the hole mobility and the diffusion coefficient of holes, respectively, γ_e is the probability of binding of electron–hole pairs in excitons, $\gamma_F(x)$ the probability of ionization of excitons in an external electric field (calculated according to [12]), α_e the thermal exciton ionization factor, $p(x) = \delta p(x)$ the concentration of nonequilibrium minority carriers (holes), $F(x)$ the electric field strength, ε the background dielectric constant, ε_0 the permittivity of vacuum, and e the electron charge.

Under conditions of quasi-equilibrium between electronic and excitonic subsystems for the relationship between the factor of binding of electron–hole pairs to excitons γ_e and the thermal exciton ionization factor α_e can be written [23, 24]

$$\alpha_e = N_{eff} \gamma_e = \gamma_e \left(\frac{\mu_r k_B T}{2\pi \hbar^2} \right)^{1/2} \exp\left(-\frac{\varepsilon_{ex}}{k_B T}\right),$$

where μ_r is the reduced mass of an exciton, ε_{ex} the binding energy of an exciton, N_{eff} the effective density of exciton states.

If the surface electrical field is small and the current density of nonequilibrium charge carriers (NCC) obeys Ohm's law, then the distributions of electron and hole velocities can be described by using a Maxwell distribution. In this case the temperature of the electron–hole gas is equal to that of the lattice and a slow drift of NCC is imposed on their chaotic motion. Because of the electron–phonon interaction in stronger electrical fields the mean energy of NCC increases and the electron–hole gas “heats up”, i.e. the heat equilibrium between NCC and the crystal lattice is disturbed [24]. With increasing field the drift velocity tends to saturate to V_d , and the mobility decreases from the maximum value μ_s in weak fields [25]. Until the drift velocity reaches its saturation (our calculation of these parameters for low-resistance CdS samples, following [24], gives roughly $F \approx 2 \times 10^4$ V/cm) the deviation from heat equilibrium can be described through the use of the “effective electron temperature” T_e , which in general is higher than the lattice temperature T . In this case the functions of the velocity distribution of NCC and the drift terms in the set (2) to (4) are the same.

The effective electron temperature may be calculated under the condition of equality of the velocity of energy build-up

$$\left\langle \frac{dE}{dt} \right\rangle_F = eV_d F$$

and the velocity of energy losses at interaction with LO-phonons [26],

$$\left\langle \frac{dE}{dt} \right\rangle_{LO} = B \left(\exp \left(-\frac{\hbar\omega_{LO}}{k_B T_e} \right) - \exp \left(-\frac{\hbar\omega_{LO}}{k_B T} \right) \right).$$

Here $\hbar\omega_{LO}$ is the energy of a LO-phonon, $B = (2m_e)^{1/2} (\hbar\omega_{LO})^{3/2} (e/h)^2 (1/\varepsilon - 1/\varepsilon_0)$.

Then

$$T_e = \frac{\theta_D}{\ln \left(eV_d F + B \exp \left(-\frac{\theta_D}{T} \right) \right)},$$

where $\theta_D = \hbar\omega_{LO}/k_B$ is the Debye temperature, T the temperature of the lattice, assumed to be equal to the thermostat temperature.

To find the field dependence of the drift velocity we have used the interpolation formula

$$V_d = \frac{\mu_s F}{1 + \frac{\mu_s F}{V_d^0}},$$

where

$$V_d^0 = \left(\frac{\hbar\omega_{LO}}{m_e} \tanh \left(\frac{\hbar\omega_{LO}}{2k_B T} \right) \right)^{1/2}.$$

If the field does not influence the lifetimes of carriers it can be shown [22] that the ratio of exciton concentration without field to that with applied field is equal to

$$\frac{n_{ex}(T)}{n_{ex}(T_e)} = \frac{T_e^{3/2} \exp \left(-\frac{\varepsilon_{ex}}{k_B T_e} \right)}{T^{3/2} \exp \left(-\frac{\varepsilon_{ex}}{k_B T} \right)} = \left(\frac{T_e}{T} \right)^{3/2} \exp \left(\frac{\varepsilon_{ex}}{k_B} \left(\frac{1}{T} - \frac{1}{T_e} \right) \right).$$

The calculation shows that, for fields $F = 10^4$ V/cm, the effective temperature $T_e = 170$ K. The concentration of excitons decreases under these conditions by 125 times. As the surface is approached the electrical field and, consequently, T_e increase and n_{ex} decreases.

The set of equations (2) to (4) was solved numerically by using the finite element approximation of differential equations and iterative method of Gummel [27]. At first, the Poisson equation for a given value of the surface field F_s was solved. Then, the calculated distribution of the potential was used to find the distribution of nonequilibrium holes and excitons. The solutions were sought separately for the space-charge region and for the quasineutral volume and then they were joined at the boundary. To realize a function $n_{ex}(x)$ we used a cubic spline for data approximation. By using (1) together with the found solution, the form of the A band of free exciton emission was calculated. The transmission factor was equal to $(1 - R(v))$, where $R(v)$ is the reflection factor [1].

Fig. 4. The computer-calculated concentration distribution of free excitons at different values of surface electric field. (1) $F_s = 0$, (2) 15, and (3) 30 kV/cm. In the inset the associated contour of the A-band emission is shown

On Fig. 4 one can see plots of the function $n_{ex}(x)$ for three values of the surface field F_s . From this curves one can notice that a thin layer with relatively low concentration of excitons forms when a surface electric field is present. The thickness of such a layer may be as large as several tens of nanometers. With increasing field, the maximum of the exciton distribution shifts into the crystal bulk. In the inset of Fig. 4 three corresponding contours of spectral lines are plotted. The asymmetrical form of the calculated spectral bands was caused by the form of the curves of the reflection spectra. If the transmission factor in (1) is constant, the form of the dip is symmetrical.

Our calculations have shown that the values of a surface field in the range of $\approx 7 \times 10^3$ to 2×10^4 V/cm are sufficient to form a dip in the emission spectra of free excitons. These values are far less than that of the critical field for exciton ionization. This fact explains the absence of changes in the reflection spectra at low-temperature heat treatment.

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(Received September 30, 1994)