Luminescence in Quantum-Confined Cadmium Selenide Nanocrystals and Nanorods in External Electric Fields

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Abstract—It is found that the absorption and luminescence spectra of CdSe nanocrystals and nanorods depend on the external electric field. It is shown that the external electric field quenches the *P*-polarized photoluminescence of CdSe nanorods to a degree higher than the degree of field-induced quenching of the *S*-polarized photoluminescence. It is established that the nanocrystals are more sensitive to the external electric field than the nanorods. The effect of the external electric field on the luminescence properties of the semiconductor nanorods is discussed.

1. INTRODUCTION

The dependence of the energy of optical transitions in semiconductor nanocrystals on the geometric dimensions of the nanocrystals opens up opportunities for development of up-to-date luminescent materials that are produced by synthesizing structures in the form of polymer films or sol–gel glasses containing nanocrystals of II–VI (CdSe, CdTe, CdS, etc.) or III–V (InP, GaAs, etc.) compounds. In these structures, the luminescence spectrum is controlled not only by the chemical composition, but even to a larger measure by the spatial dimensions of the active region as well [1]. The use of the quantum confinement effect in semiconductor nanocrystals brings to life the potential possibility of developing optimally selected luminescent materials in order to solve a large variety of applied problems, from the engineering of units for optoelectronic imaging and data transmission in information–computing systems [2] to the development of biological fluorescent markers used in diagnostics and pharmacology.

The possibility of controlling the luminescence properties of semiconductor nanocrystals embedded in thin insulator films and separated from each other with external electric fields can be used for the development of radically new optoelectronic devices operating in the visible spectral region. When combined with Coulomb effects, the quantum-confinement effects in semiconductors provide technological advantages in adjusting the devices to the wanted spectral region and preset operating voltages. Since the size

confinement of motion of charge carriers is a governing factor in this case, it is important to obtain a comparative estimate of the effectiveness of the influence of the electric field upon structures with different degrees of freedom of motion of electrons and holes.

In this study, we compare the absorption and luminescence spectra of CdSe nanocrystals and nanorods in thin insulator films in external electric fields. We consider the dependence of polarized components of photoluminescence of the nanorods on the orientation of the electric vector of the electromagnetic wave of excitation light with respect to the external electric field.

2. EXPERIMENTAL

In experiments, we used structures containing CdSe nanocrystals and nanorods embedded in thin polymethylmethacrylate (PMMA) insulator films. The total thickness of the insulator layer of the sample containing monodisperse CdSe nanocrystals, whose average dimension (diameter) is $d_{av} = 4$ nm, is $h_{gap} \approx 20$ μm. The samples containing CdSe nanorods are characterized by the average length–diameter ratio (aspect ratio) of the nanorods l_{rod} (nm)/d_{av} (nm) = 17 : 10, 23 : 7, 20 : 5, and 25 : 4. The PMMA insulator layer containing the nanoparticles is sandwiched between planar conducting indium–tin oxide (ITO) layers with electric contacts to apply the voltage. Apart from the PMMA layer, there is an epoxy resin layer between the ITO layers. The epoxy resin layer serves, on the one hand, as a glue and, on the other hand, as a separator. In the samples with nanorods with the above
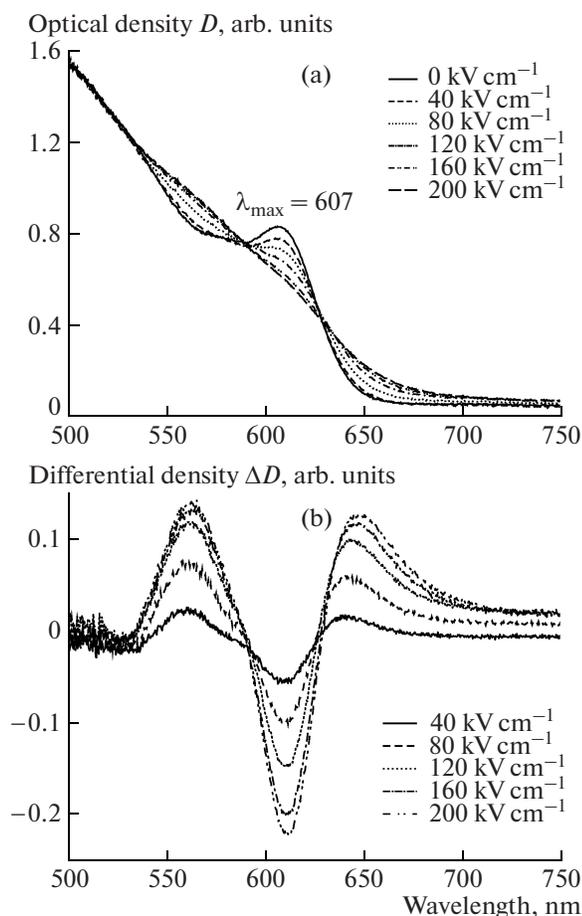
Fig. 1. (a) Absorption spectra ($D = \log I_0/I$) and (b) differential absorption spectra ($\Delta D = D - D_{0V}$) at different strengths of the external electric field (indicated) for CdSe nanocrystals embedded in the PMMA film.

Fig. 2. (a) PL spectra (I) and (b) differential PL spectra ($\Delta I = I(0) - I(U)$) at different external electric field strengths (indicated) for CdSe nanocrystals embedded in the PMMA film. The excitation wavelength is $\lambda_{exc} = 488$ nm.

indicated aspect ratios, the entire gap between the ITO layers is $h_{gap} \sim 30$, ~35, ~40, and ~20 μm, respectively.

The absorption and luminescence spectra of the nanocrystals were recorded with the use of the diffraction spectrograph S-3801, Solar TII, and the cooled CCD matrix LN/CCD-1162-E PROD FG, Princeton Instrument Inc. Polarization studies of the luminescence spectra of the CdSe nanorods were carried out with the use of the specrofluoremeter CM 2203 Solar, the spectral width of the slit was 5 nm. The polarization characteristics of the absorption spectra were studied with the use of the spectrophotometer Carry 500, Varian, with the spectral resolution 1 nm. The voltage was applied to the contacts from the stabilized voltage source HVPS-320, Spetspribor.

3. ABSORPTION AND LUMINESCENCE OF CdSe NANOCRYSTALS IN EXTERNAL ELECTRIC FIELDS

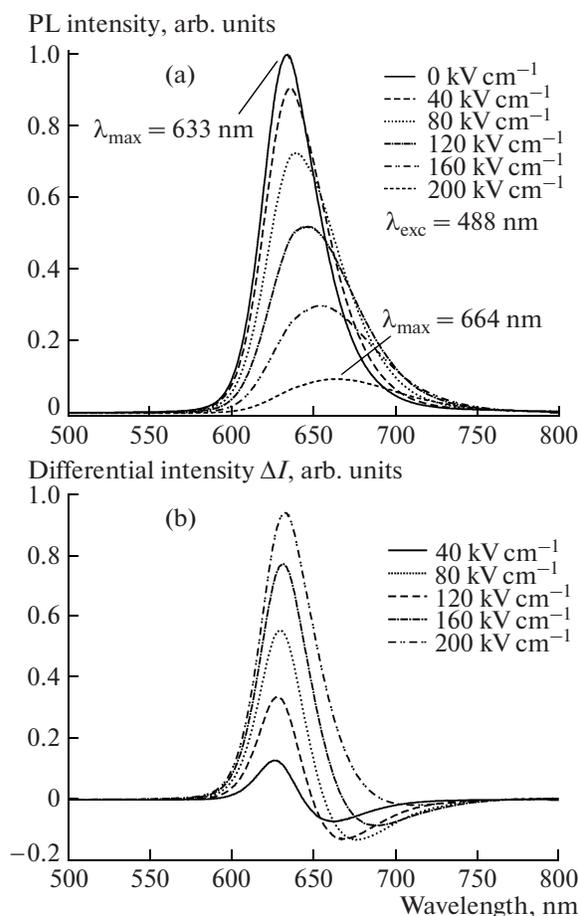The absorption spectra of the sample containing the CdSe nanocrystals, whose average dimension is ~4 nm, are shown in Fig. 1 for different external electric-field strengths. The absorption edge of the nanocrystals is shifted from the edge of the corresponding bulk semiconductor to shorter wavelengths and involves an excitonic line with a peak at the wavelength $\lambda_{max} = 607$ nm. On application of the external electric field, the amplitude of the excitonic peak decreases, and in the fields above 200 kV cm^{-1}, the excitonic peak practically disappears. The broadening of the excitonic peak in the absorption spectra is a typical manifestation of the Stark effect in quantum-confined semiconductor nanostructures [3, 4].

The luminescence spectrum is also modified by the external electric field (Fig. 2). As the field is increased from zero to 200 kV cm^{-1}, the luminescence-band intensity becomes more than ten times lower. The luminescence band steadily broadens, and band's peak shifts to longer wavelengths by 31 nm, from $\lambda_{max} = 633$ nm in a zero electric field to $\lambda_{max} = 664$ nm at the field strength 200 kV cm^{-1}. The coef-
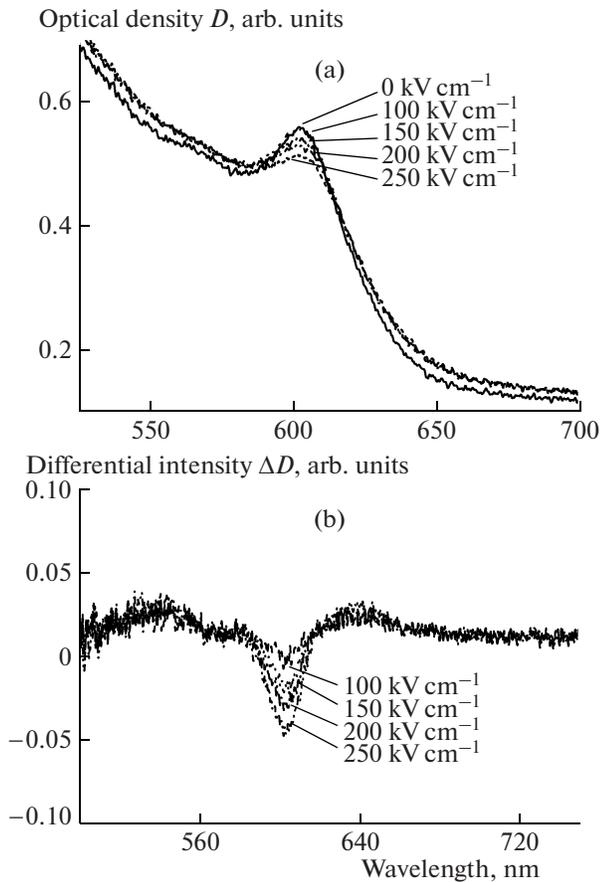
Optical density D, arb. units

Differential intensity ΔD, arb. units

Fig. 3. (a) Absorption spectra ($D = \log I_0/I$) and (b) differential absorption spectra ($\Delta D = D - D_{0V}$) at different strengths of the external electric field (indicated) for the sample containing CdSe nanorods with the length–diameter ratio 25 : 4.

ficient of shifting of the peak is $\Delta\lambda_{max}/\Delta E_{ext} \approx 1.55 \times 10^{-4}$ nm cm V^{-1}.

In considering the evolution of the absorption and luminescence spectra in the external electric field, we can suggest the following:

(i) the excitonic transitions represent most likely the basic mechanism of luminescence (this is supported by the field-induced changes in the absorption spectrum);

(ii) the probability of recombination of electron–hole pairs decreases in the external electric field because of the decrease in the overlapping between the wave functions of the electron and hole in the excited nanoparticle; and

(iii) the nanostructure studied here is apparently constituted of an ensemble of nanocrystals different in electronic and optical properties, and a certain part of the nanocrystals of the ensemble is excluded from luminescence by the electric field (the corresponding mechanism is unclear).

It should be noted that the field-induced change (decrease) in the coefficient of the absorption edge near the excitonic peak for the ensemble of semiconductor nanocrystals and the profound quenching of photoluminescence can be also due to injection of electrons to one of the unoccupied electron levels of the nanocrystal. The complete or partial occupation of this level yields a substantial decrease in the excitonic peak's intensity in the spectrum of the optical density. At the same time, electrons in the conduction band of the semiconductor are responsible for the pronounced quenching of photoluminescence via the Auger recombination of excitons. These and some other effects related to charging of nanocrystals were studied in detail previously [5, 6]. In [5, 6], nanocrystals were charged in electrochemical processes, which required the electric contact of the nanocrystals with one of the electrodes. In the case under consideration here, injection of electrons into nanocrystals is hampered by the matrix of the PMMA polymer. Therefore, we believe that the optical properties of the nanocrystals are modified basically due to the Stark effect, whereas the injection has only a minor effect.

An insight into the mechanism of quenching of luminescence of the nanocrystals in the external electric field could be gained in experimental studies of the kinetic properties of luminescence and the luminescence of individual nanocrystals in electric fields. Some studies in this field are now in progress [7–9].

4. ABSORPTION AND LUMINESCENCE OF CdSe NANORODS IN EXTERNAL ELECTRIC FIELDS

The CdSe nanorods are larger in geometric dimensions than the above-considered nanocrystals, and therefore, the effects of quantum confinement in the nanorods are less pronounced. Specifically, the excitonic band in the absorption spectra of the nanorods is pronouncedly broadened, and its spectral position is closer to the fundamental absorption edge of the bulk semiconductor. The aspheric shape of the nanorods (for example, the length–diameter ratio is in the range from 1.7 to 6.25 for different samples) enhances the effect of inhomogeneous broadening of the spectrum. This additionally makes the analysis of the shape of the spectrum more difficult. Since the structure is represented by a thin film with the thickness ~300 μm and with the optically flat surfaces, the absorption spectrum is superimposed by the spurious interference pattern. The absorption spectra of the CdSe nanorods in different external electric fields are shown in Fig. 3.

The PL spectrum of the CdSe nanorods, like the PL spectrum of the CdSe nanocrystals, is modified by the external electric field. However, these modifications are less substantial: as the field strength is increased to 200 kV cm^{-1}, the PL band's intensity decreases by a factor only slightly larger than 2, and the spectral position of the PL band's peak shifts to longer wavelengths only by several nanometers. With
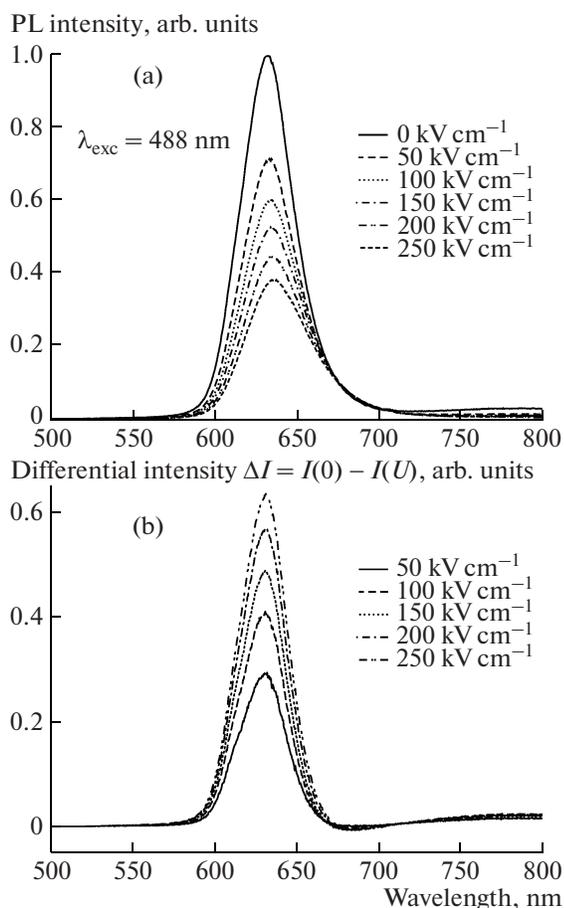
PL intensity, arb. units

Fig. 4. (a) PL spectra and (b) differential PL spectra of the sample containing CdSe nanorods with the length–diameter ratio 25 : 4 at different strengths of the external electric field from 50 to 250 kV cm^{-1}. The PL was excited by the short-wavelength emission mode of an argon laser (λ_{exc} = 488 nm).

Fig. 5. Diagram of the S- and P-polarized light beams in studies of the PL spectra of CdSe nanorods in external electric fields.

PL intensity, arb. units

Fig. 6. PL spectra of CdSe nanorods (with the length–diameter ratio 17 : 10) at different strengths of external electric field for the two independent polarizations (a) S and (b) P. The PL signal was excited with the second harmonic of the Nd laser emission (λ_{exc} = 531 nm) in the cw mode of operation. The time of signal accumulation was 3 s. The excitation radiation was cut off at the entrance of the photodetector with an OS-13 optical filter.

increasing the strength of the external electric field, the PL band's width increases too. The PL spectra of the CdSe nanorods at different strengths of the external electric field are shown in Fig. 4.

5. POLARIZATION OF LUMINESCENCE OF CdSe NANORODS IN EXTERNAL ELECTRIC FIELDS

For extended structures, such as nanorods, the optical properties are bound to manifest some differences, in accordance with the spatial orientation of the axes of the nanoparticles. One of the naturally distinguished directions, for which the physical processes of interaction of light with the semiconductor are expected to exhibit the most-pronounced difference from the processes for other directions, is the direction of the external electric field. The external field plays the role of a selecting field. In fact, from the ensemble of randomly oriented nanorods, the field selects parti-
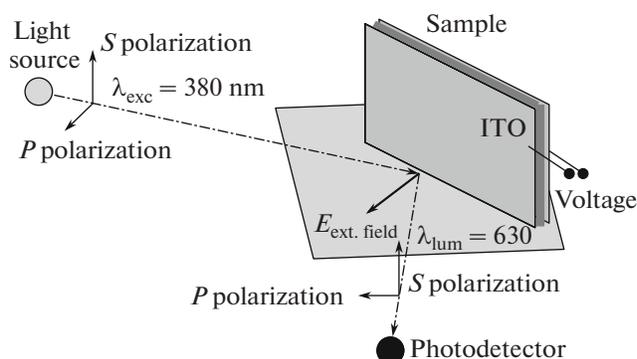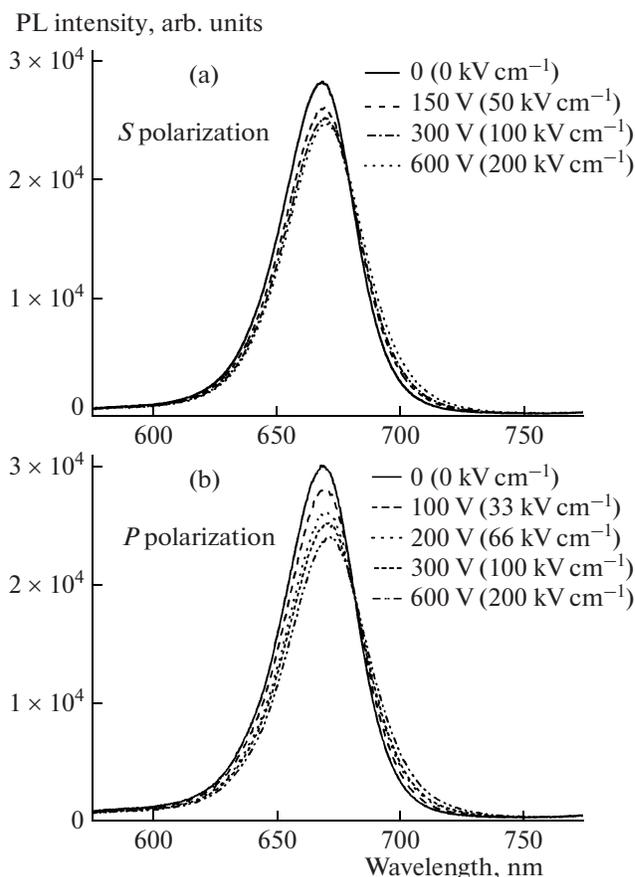
cles oriented along the field, so that the Coulomb effects of external forces are most pronounced exactly in these particles. When excitation light interacts with
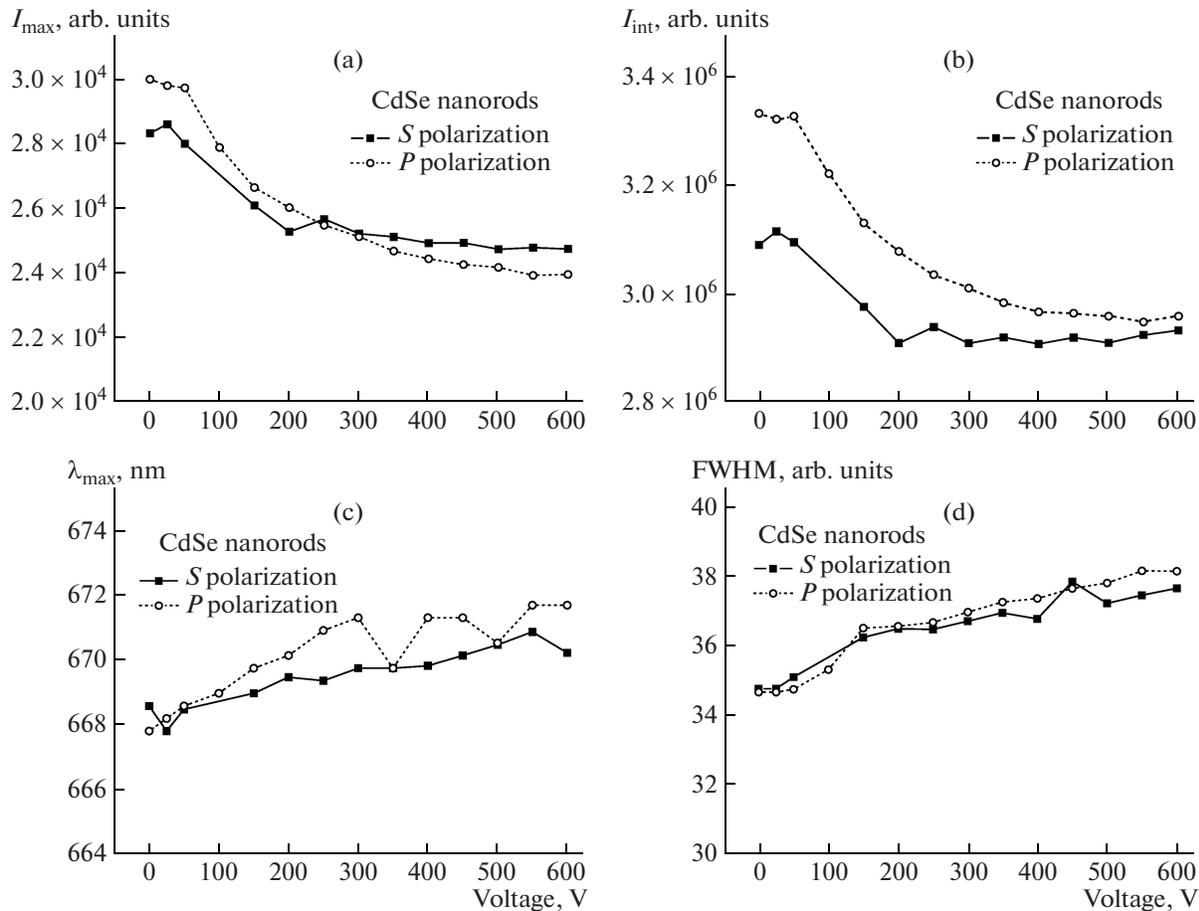
Fig. 7. Dependences of (a) the PL peak intensity I_{max}, (b) the integrated PL intensity I_{integr}, (c) the wavelength λ_{max} corresponding to the PL peak, and (d) the FWHM of the PL band of the CdSe nanorods (with the length−diameter ratio 17 : 10) on the external voltage for the two independent polarizations S and P.

a nanorod in the external electric field, two independent situations are possible: the component of the electric field of the light wave along the direction of the external electric field is zero (the S polarization) and nonzero (the P polarization) (Fig. 5).

In the case of the S polarization, at which the $\mathbf{E}$ vector of the electromagnetic wave of excitation light is orthogonal to the strength of the external electric field, the geometric layout of the system is such that the direction of the $\mathbf{E}$ vector is parallel to the sample plane. In the case of the P polarization, at which there always exists a nonzero component of the $\mathbf{E}$ vector of the electromagnetic wave of excitation light along the external electric field, the system is arranged in such manner that the $\mathbf{E}$ vector lies in the plane formed by the incident (reflected) beam of excitation light and the normal sample's surface.

The polarization of luminescence of the CdSe nanorods was studied for the sample (l_{rod}/d_{av} = 17 : 10, $h_{gap} \approx 30$ μm) that exhibited the highest degree of quenching. The studies were performed in the transmittance mode of measurements: the tilt angle of the sample with respect to the excitation beam was 45°,

and the PL signal emitted from the backside was recorded at the angle 90° with the excitation beam. The optical arrangement in the studies of polarized luminescence at different strengths of the electric field is illustrated in Fig. 5. The S- and P-polarized components of excitation and emission to be detected were separated with prism polarizers. The PL spectra of the CdSe nanorods in different electric fields for the two independent polarizations are shown in Fig. 6.

From Fig. 6, it can be seen that the P-polarized luminescence is modulated by the external electric field more efficiently than the S-polarized luminescence, for which the external electric field is transverse. This systematic feature is observed also for the other characteristics of luminescence spectra recorded for the independent S and P polarizations.

6. POLARIZED OPTICAL ABSORPTION SPECTRA OF CdSe NANORODS IN EXTERNAL ELECTRIC FIELDS

To study the polarized absorption spectra, we chose the longer CdSe nanorods, with the length−diameter

ratio 23 : 7. In the sample, the thickness of the PMMA insulator layer was ~35 μm. The absorption spectra were recorded using a Cary-500 spectrophotometer. In the measurement channel, two film polarizers were used, one placed in front of the sample and the other behind it. The tilt angle of the sample with the measurement beam was ~60°. The absorption spectrum of the CdSe nanorods for two independent polarizations is shown in Fig. 8.

From Fig. 8, the difference in absorption for the S and P polarizations can be clearly seen. The absorption spectra of the CdSe nanorods exhibit a typical excitonic structure for both S and P polarizations. However, in the S-polarized spectrum, one can notice a second excitonic peak, in contrast to what can be seen in the P-polarized spectrum. For both polarizations, the effect of the external electric field on the spectra is very slight and negligible compared to the corresponding effect in the case of the nanocrystals. The P-polarized absorption spectrum is substantially influenced also by the Fresnel reflection; at angles close to the Brewster angle, the Fresnel reflection makes a noticeable contribution to the magnitude of the experimentally measured optical density. The dissimilarity in shape between the absorption spectra in the region of the excitonic peak for different polarizations can also arise, e.g., from the relative difference in the behavior of the reflectance from the sample surface for the S and P polarizations.

7. THE DEGREE OF POLARIZATION OF LUMINESCENCE OF CdSe NANORODS AT DIFFERENT WAVELENGTHS

In the measurements of the degree of polarization, we used the CdSe nanorods with the length−diameter ratio 20 : 5 and with the thickness of the PMMA insulator layer ~40 μm. The PL signal from the nanorods at different strengths of the external electric field was excited at the wavelength $\lambda_{exc} = 380$ nm for the two independent S and P polarizations.

For these CdSe nanorods (longer than those in the samples with $l_{rod}/d_{av} = 17 : 10$ and 23 : 7), the PL spectrum is modified by the external electric field to a lesser extent. Specifically, as the external electric field 100 kV cm^{-1} is applied to the sample, the PL band's intensity decreases only by about 17%. Similar to how it happens in the samples considered above, the decrease is more pronounced for the P-polarized PL band.

The degree of polarization of PL of the CdSe nanorods in the spectral range from 580 to 700 nm was determined from the relations between the PL intensities for the $S-S$ and $S-P$ polarizations by the formula [10]

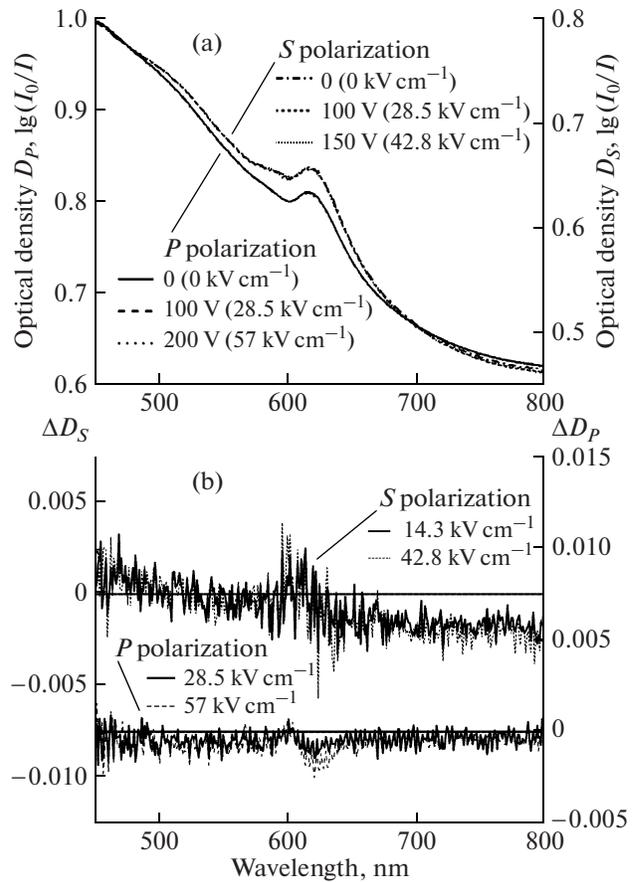$$P_{CdSe} = \frac{I_{\parallel} - I_{\perp}}{I_{\parallel} + I_{\perp}}. \qquad (1)$$

Fig. 8. (a) Absorption spectra and (b) differential absorption spectra of CdSe nanorods (with the length−diameter ratio 23 : 7 in the external electric field at different polarizations of transmitted light (indicated). The sample was oriented at an angle of ~60° to the (light source)−detector axis.

Here, $I_{\parallel}$ and $I_{\perp}$ are the intensities of the PL components polarized parallel ($S-S$) and orthogonal ($S-P$) to the vector **E** of the plane-polarized excitation radiation (S). The dependence of the degree of polarization on the wavelength of light emitted by the CdSe nanorods at different strengths of the external electric field is shown in Fig. 9.

From the dependences plotted in Fig. 9, it can be seen that the PL of the nanorods of the CdSe semiconductor with the length−diameter ratio 20 : 5 is partially polarized, with the average degree of polarization 0.13, and does not change its polarization in any external electric fields. The degree of polarization steadily increases with a decreasing wavelength. In the dependence, there are two flat portions, in the ranges from 600 to 630 nm ($P_{CdSe} = 0.145$) and from 640 to 680 nm ($P_{CdSe} = 0.11$), corresponding to the short- and long-wavelength regions of the PL band of the CdSe nanorods. The two different values of the degree of polarization within one PL band can be a consequence of superposition of two independent PL lines corresponding apparently to different mechanisms of emission. These can be, e.g., a narrow short-wavelength
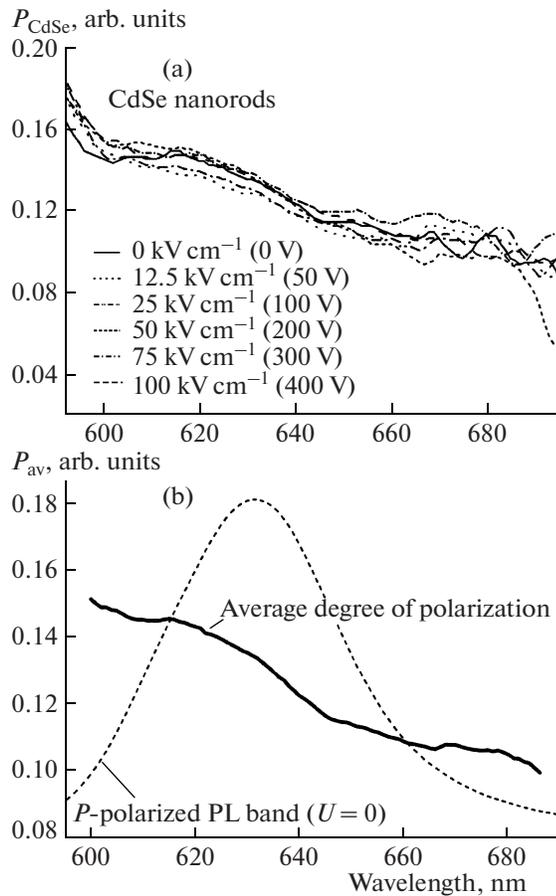
Fig. 9. The dependences of (a) the degree of polarization of PL of CdSe nanorods (with the length–diameter ratio 20 : 5), P_{CdSe}, in different external electric fields and (b) the degree of polarization arithmetically averaged over the data of all measurements, P_{av}, on the PL wavelength. b: Dashed line shows the contour of the PL band of the sample at zero voltage.

line of intrinsic luminescence and a wide long-wavelength line of extrinsic luminescence related to impurities or defects. More accurate conclusions could be drawn from the analysis of the data on the luminescence kinetics in the nanorods at the wavelengths 610 and 660 nm.

Similar behavior of the degree of polarization was observed in porous silicon, wherein luminescence is associated with emission from particles elongated in the direction of the etching current (i.e., from practically elliptic nanocrystals or nanorods) [1, 11, 12].

According to the theory of polarized luminescence, the ensemble of randomly oriented nanocrystals, each optically anisotropic, emit partially polarized radiation under the condition of excitation with plane-polarized light. Such an effect is known in molecular spectroscopy and referred to as photoselection. Polarization of luminescence is commonly described by the degree of polarization defined by expression (1), in which $I_{\parallel}$ and $I_{\perp}$ are the intensities of emission compo-

nents polarized parallel and transversely to the polarization of excitation radiation. In the linear oscillator model, the degree of polarization satisfies the relation [13]

$$P = \frac{3\cos^2\alpha - 1}{\cos^2\alpha + 3}, \qquad (2)$$

where α is the angle of the absorbing oscillator with the emitting one. Thus, the maximal value of P, $P_{max} = 1/2$ ($\alpha = 0$), corresponds to totally anisotropic oscillators, whereas the minimal value of P is at the angle $\alpha = 90°$ and corresponds to $P_{min} = -1/3$. It should be noted that the ensemble considered here is macroscopically isotropic because of the random orientation of the particles.

Partial polarization can also be a consequence of the asymmetric shape of randomly arranged nanocrystals. It was found that this is the case for extended silicon crystallites in porous silicon films. The authors of [11] observed polarization of emission with a noticeable positive degree, although silicon nanocrystals, nearly spherical in shape, emit nonpolarized radiation. The explanation suggested for this effect was based on the concept of permittivity confinement in ellipsoid-shaped nanocrystals [12, 14]. The anisotropy of emission is due to the anisotropy of the distribution of the electric field of the light wave inside the permittivity ellipsoid embedded in a medium with the permittivity different from that of the ellipsoid.

8. LONG-TERM EFFECT OF A HIGH EXTERNAL ELECTRIC FIELD

Long-term effect of the external electric field on the semiconductor can induce ordered motion of charge carriers in the form of drift of charged dislocations and traps. In fact, even in a 17 nm nanorod oriented along the external electric field, the energy of about 330 meV can be imparted to a free electron from the field 200 kV cm^{-1}. Even if we take into account the permittivity of CdSe $\langle \varepsilon_0 \rangle_{CdSe} = 9.4$ [15] and the energy losses by collisions associated with thermal motion of electrons and atoms of the crystal lattice, we can conclude that the above energy is high enough for electrons, if captured at traps with not so high activation energies (below 35 meV), to be set in motion. Thus, by applying the external electric field for a long time, it is possible to modify the optical properties of a compositionally inhomogeneous ensemble of semiconductor nanorods. Obviously, such modifications can be responsible for some degradation or for some sort of partial aging of the sample from the standpoint of its optical properties; however, the modifications can also be partially reversible due to the reverse diffusion drift by thermal motion, when there is no external electric field. At the same time, the long-term effect of the high external electric field on nanocrystals, whose dimensions are very small, is bound to be less noticeable.
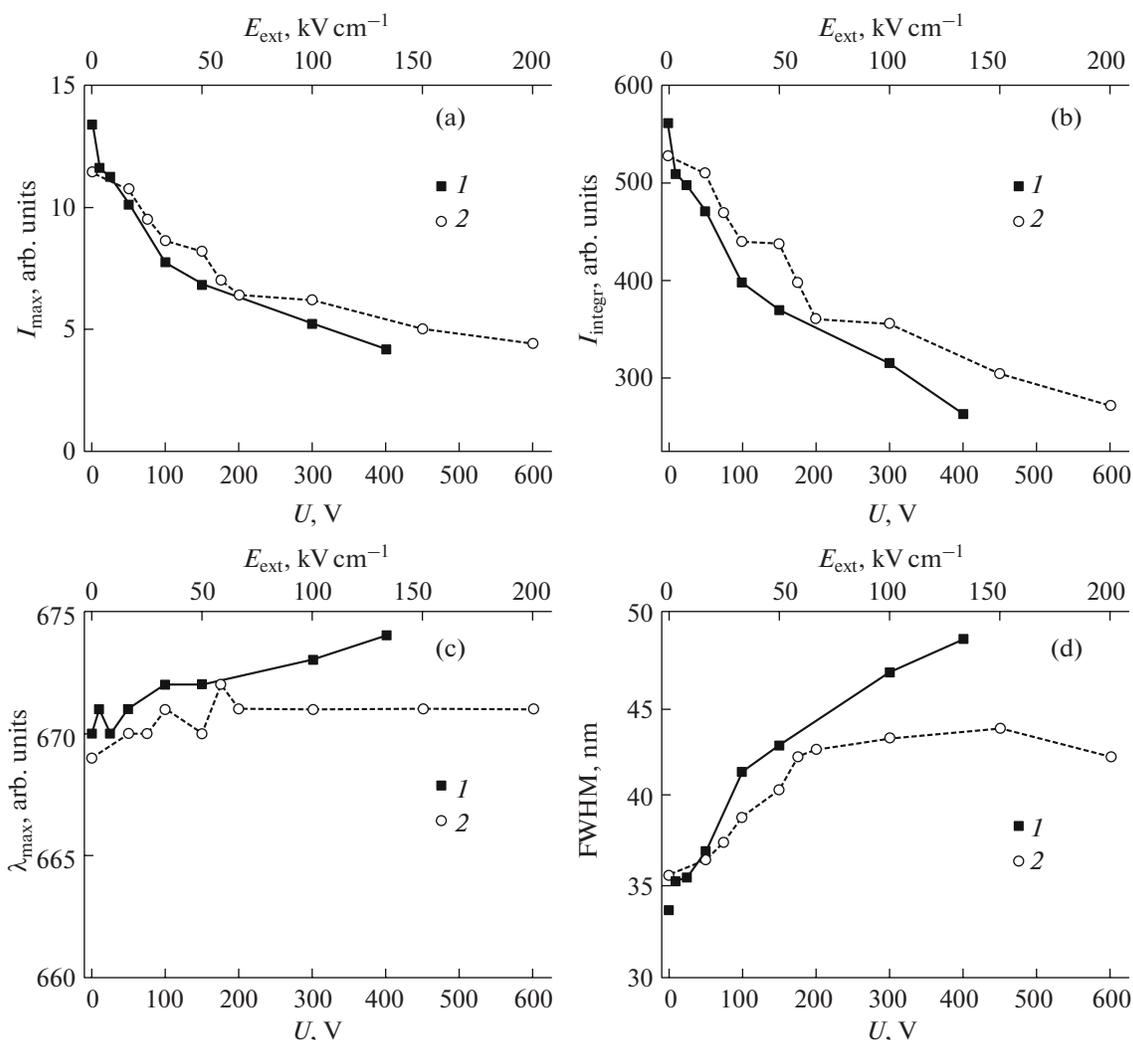
Fig. 10. (a) The PL peak intensity I_{max}, (b) the integrated PL intensity I_{integr}, (c) the wavelength λ_{max} corresponding to the PL peak, and (d) the FWHM of the PL band of the CdSe nanorods (with the length–diameter ratio 17 : 10) at different voltages U (*1*) before and (*2*) after long-term (9 h) application of the external electric field $E_{ext} = U/h_{gap}$ at different voltages U. The excitation wavelength is $\lambda_{exc} = 450$ nm. The thickness of the insulator layer between electrodes is $h_{gap} \approx 30$ μm.

In the experiment, we used the sample ($l_{rod}/d_{av} = 17 : 10$, $h_{gap} \approx 30$ μm) that exhibited the highest degree of quenching of luminescence. The sample was at the voltage 400 V for 9 h. This voltage corresponded to the electric field 133 kV cm^{-1} (the permittivity was disregarded). Then, we compared the main characteristics of the PL spectra of this sample before and after the application of the electric field. The PL signal was excited at the wavelength $\lambda_{exc} = 450$ nm.

Analyzing the experimental data, we can highlight a number of regular trends in the behavior of the PL signal. For the nanorods, the intensity in the PL peak, I_{max}, and the integrated intensity of the PL band, I_{integr}, decrease in inverse proportion to the strength of the applied external electric field $\mathbf{E}_{exc}$. The relative change in these intensities with an increasing electric field after the long-term application of the field is smaller than the change observed without the long-term effect (Figs. 10a, 10b).

The shift of the wavelength corresponding to the PL bands peak, λ_{max}, and the change in the full width at half-maximum (FWHM) of the PL band in the external electric field are also different for the sample before and after long-term application of the external electric field (Figs. 10c, 10d). The slowing-down of broadening of the PL band after long-term treatment of the sample in the external field is most pronounced at voltages above 175 V ($E \approx 58$ kV cm^{-1}): the PL band practically ceases to broaden. This can suggest in particular that, after the long-term effect of the field on the sample, there are fewer radiative centers characterized by a wide spread of luminescence parameters. The high external electric field can induce also electrochemical and thermal effects.

9. CONCLUSIONS

External electric fields can be efficiently used for modulation of PL in quantum-confined semiconductor nanostructures. The modulation depth depends on the type and geometric parameters of the nanostructures. The absorption and luminescence spectra are most efficiently modulated for quantum dot nanocrystals, wherein the effect of quantum confinement is well pronounced in three dimensions. The PL spectra of nanorods are partially polarized and exhibit an inhomogeneous internal structure controlled at least by two different mechanisms, the intrinsic emission and the defect- or impurity-related emission. The extended shape of the nanorods defines the selectivity of their luminescence properties with respect to the direction of the external electric field.

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