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EFFECT OF THE CdSe NANOPLATELETS CONCENTRATION IN A COLLOIDAL SOLUTION ON THE NONLINEAR CHANGE IN ABSORPTION

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Abstract. Nonlinear absorption features of CdSe nanoplatelets colloidal solutions with a thickness of 2.5 and 3.5 monolayers were experimentally studied depending on the concentration in the case of resonant stationary excitation by nanosecond laser pulses. An increase in the amplitude of differential transmission and absorption saturation intensity at the wavelengths of excitonic transitions associated with heavy holes was detected for two series of samples with increasing concentration of nanoplatelets in the colloidal solution and explained by the process of excitons phase space filling. For colloidal solutions of high-concentration nanoplatelets, a region of negative differential transmission values was revealed at a sufficiently high pump intensity and explained by the transition from the absorption saturation regime to the optical amplification regime.

Keywords: *Nanoplatelets*, *CdSe*, *nonlinear absorption*, *exciton*, *heavy holes*, *light holes*, *optical amplification*

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1. INTRODUCTION

In the last few decades, semiconductor structures of reduced dimensionality have been of particular practical and scientific interest. The improvement of the methods of colloidal growth of nanocrystals has led to the possibility of building nanocrystals with specified optical and electronic properties due to the possibility of controlling the parameters of the crystal lattice, morphology, size and dimensionality, as well as the possibility of creating heterostructural nanocrystals: quantum dots [1], nanotetrapods [2, 3], nanoplatelets [4-6] and nanorolls [7].

Quantum-dimensional semiconductor crystals are characterized by a spatial limitation of the motion of charge carriers, which determines a significant modification of their energy spectrum from the initial spectrum of the bulk semi-conductor. By controlling the size and dimensionality of nanocrystals, it is possible to create objects with a given effective band gap, with a controlled discrete optical spectrum. In addition, the determining difference between quantum-dimensional semiconductor crystals and bulk semiconductors is a significantly large oscillator force and exciton binding energy. A common feature of all low-dimensional structures is a special type of absorption and photoluminescence (PL) spectra due to the possibility of the influence in them of the form of nanostructures on nanostructures on the binding energy of excitons [8], charge localization [9], charge-induced Stark effect [10].

The above-mentioned features of nanocrystals open up exceptional opportunities for the design

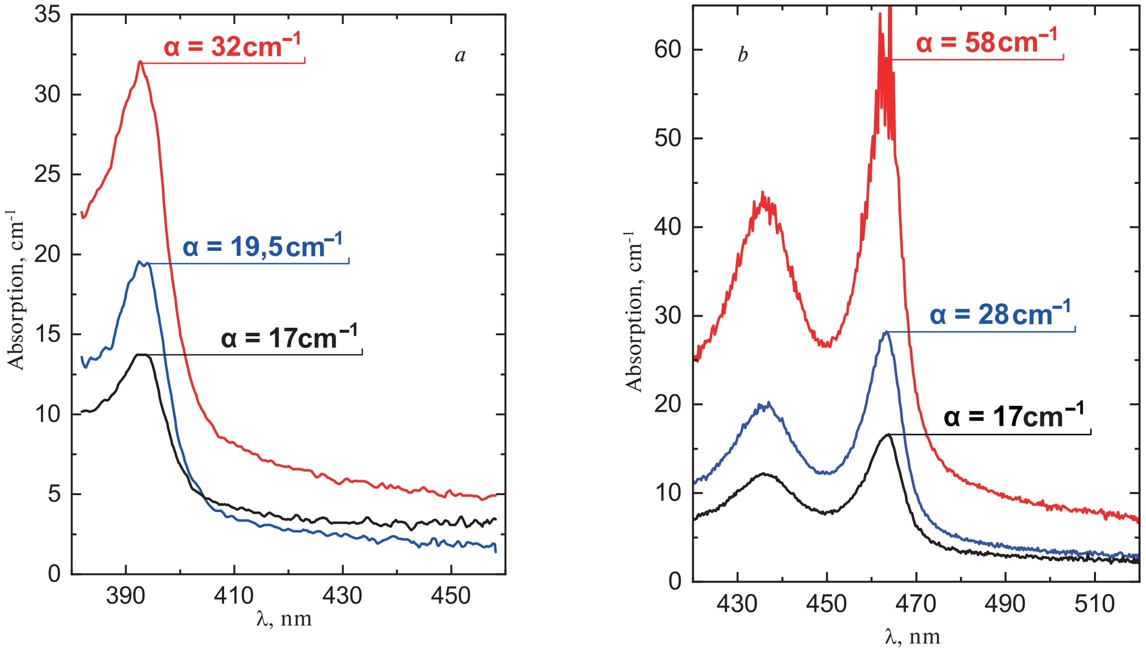

Fig. 1. (In color online) Linear absorption spectra of colloidal nanoplatelets: CdSe393 (*a*) and CdSe463 (*b*)

of efficient optoelectronic devices, the principle of operation of which is determined by exciton processes. Examples include the successful creation of phototransistors [11], highly efficient solar concentrators [12-14], LEDs [15-17], optical elements of the laser active medium [18], Q-factor modulators [19], biomarkers [20].

Recently, a new type of nanoparticles was synthesized for the first time [4] — semiconductor two-dimensional nanocrystals of planar geometry (CdSe, CdS, CdTe, CdSe / CdS, CdSe / CdZnS), called “nanoplatelets”. Due to the lateral dimensions of 30–200 nm and a thickness not exceeding several atomic layers (3–7 monolayers), the effect of dimensional quantization in these objects is realized only in one selected direction. An important feature of such nanocrystals is the increasing ratio of surface atoms to atoms in volume, which leads to an increase in the influence of surface states on the optical properties of nanostructures [21, 22]. When charge carriers are captured to localized surface states as a result of Auger recombination [23-26], both an increase in the relaxation times of PL during radiative relaxation from surface states and accelerated depopulation during the transformation of nonradiative processes are possible. This property is especially characteristic of ultrathin nanoplatelets. To control these properties using colloidal synthesis, heterostructural nanocrystals are grown [27], including the second kind, which are of particular interest due to the effect of photoinduced charge separation [28, 29].

In addition, all of the samples are characterized by significantly lower values of the Stokes shift and the attenuation time of luminescence in comparison with other low-dimensional structures (in particular, with quantum dots) [30]. The third effect, which distinguishes the nanoplastics from the rest of the nanostructures, is a narrow band of luminescence [31].

In this study we investigated the effect of the concentration of CdSe nanoplatelets in a colloidal solution on the nonlinear change in absorption in the case of stationary excitation of charge carriers by nanosecond laser pulses, as well as to establish the characteristics of the absorption saturation due to the effect of filling the exciton phase space [32].

2. EXPERIMENTAL SETUP

The object of the study was selected colloidal solutions of CdSe nanocrystals: CdSe393 and CdSe463 with thicknesses of 2.5 monolayer (1.2 nm) and 3.5 monolayer (1.5 nm) respectively (the number in the designation of the samples indicates the wavelength of the exciton transition 1_{hh}–1_e, associated with heavy holes, Fig. 1).

The samples were synthesized by colloidal chemistry and have a crystalline structure of sphalerite. Hexane, transparent in the optical range under study, was used as a solvent. To carry out measurements, colloidal solutions of CdSe nanoplatelets were placed in a glass cuvette with a thickness of 1 mm. All measurements were carried out at room temperature. The initial concentration of nanoplatelets in the colloidal solution was about 10^{15} cm^{-3}. A subsequent decrease in concentration by 2 and 3 times was carried out by adding a solvent to the initial colloidal solution. To characterize the colloidal solutions of CdSe nanoplatelets of various thicknesses and concentrations, their linear absorption spectra were measured. On the absorption spectra of colloidal solutions of CdSe nanocrystals with 2.5 monolayer thicknesses (Fig. 1*a*), the absorption maximum at a wavelength of 393 nm corresponds to the exciton transition 1_{hh}–1_e. The position of this peak does not depend on the concentration of the solution. Depending on the value of the absorption coefficient α (calculated from Booger's law $I = I_0 e^{-\alpha d}$) colloidal solutions of CdSe393 nanoplatelets would be re-designated as CdSe14, CdSe19 and CdSe32 for low, medium and high concentrations of solution, respectively. The linear absorption spectra of CdSe colloidal nanocrystals with a thickness of 3.5 monolayers (Fig. 1*b*) are characterized by two maxima, the position of which does not depend on the concentration, and correspond to exciton transitions associated with light (1_{lh}–1_e; 436 nm) and heavy (1_{hh}–1_e; 463 nm) holes. Similarly, for colloidal solutions of CdSe463 nanoplatelets, the designations CdSe17, CdSe28 and CdSe58 were used for low, medium and high concentrations of the solution respectively. The solvability of exciton peaks at room temperature indicates their significant binding energy [33] and the monodispersity of nanocrystals in thickness.

The Pump&Probe method was used to determine the nonlinear change in the absorption of colloidal solutions of nanoplatelets. The samples were excited by a pulse of the third harmonic (360 nm) Nd^{3+} : YAP (Nd^{3+} : $YAlO_3$; $\lambda = 1080$ nm), operating in Q-factor modulation mode (pulse duration $\tau \approx 9$ ns). Simultaneous probing was carried out with a broadband PL of organic BBQ dyes ($C_{48}H_{66}O_2$) for CdSe393, nanoplastic, as well as Coumarin-120 ($C_{10}H_9NO_2$) for CdSe463 nanoplatelets. The selection of dyes was determined according to their absorption spectra, in order to cover the wavelengths of the main exciton absorption bands of the studied nanoplatelets. In addition, the conditions of spatial and temporal alignment of the pumping and probing beams were taken into account. In particular, the duration of the dye PL is about 1 ns, which made it possible to fulfill the condition of simultaneous excitation and probing of samples.

The intensity of the exciting radiation of the I_{pump} varied from 0.3 to 2.5 MW/cm^2 using neural light filters. The transmission and PL spectra of CdSe nanocrystals were measured using a Pixis 256 CCD camera combined with a SpectraPro 2300 polychromatometer.

3. RESULTS AND DISCUSSION

The dependences of the wavelengths corresponding to the maximum of the PL and the values of the half-width of the peak of the PL on the pump intensity were plotted from the PL spectra of CdSe nanoplatelets (Fig. 2 and 3) (Fig. 4*a*, *b*).

The wavelengths of their maxima (402 ± 3nm and 472 ± 1 nm, respectively), which differ from the wavelengths of the absorption peaks (393 nm and 463 nm, respectively) due to the Stokes effect, are determined from the PL spectra of CdSe393 colloidal nanocrystals (Fig. 2), CdSe463 (Fig. 3) at different concentrations and pump intensities shift (about 10 nm; the emission of photons shifts relative to their absorption into the long-wavelength region, including due to the transfer of part of the energy into the radiation of an optical phonon). In addition, it was found that for CdSe463 samples, the width of the PL peaks increases with increasing pump intensity (Fig. 4*a*,*b*), and the maximum of the PL spectrum shifts to the red region (Fig. 4*e*,*f*). The broadening and shift of the PL peaks by about 4 nm (or 23 MeV) is explained by the emission of charged excitons (trions) [34] and biexitons [35]. In addition, it was noted that with an increase in the density of emitting nanoparticles, the amplitudes of the PL peaks at the same excitation levels have large values (Fig. *4c*,*d*).

To determine the features of nonlinear absorption of samples, the Pump&Probe method described in detail in [36] was applied. Differential transmission spectra (DT) were calculated from the measured transmission spectra [37]:

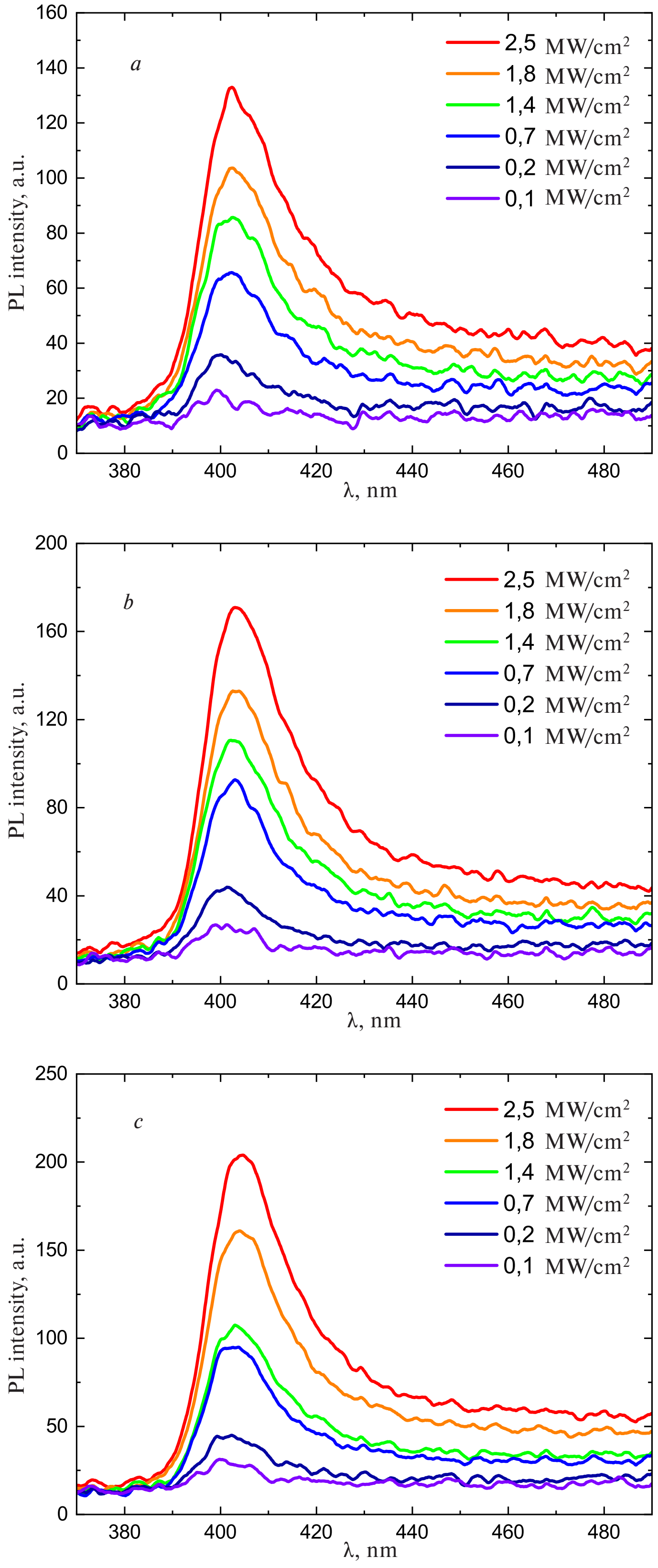

Fig. 2. (In color online) PL spectra of CdSe393 colloidal nanoplatelets at different pump intensities for solutions of low (*a*), medium (*b*) and high (*c*) concentrations (exposure time t = 3 s)

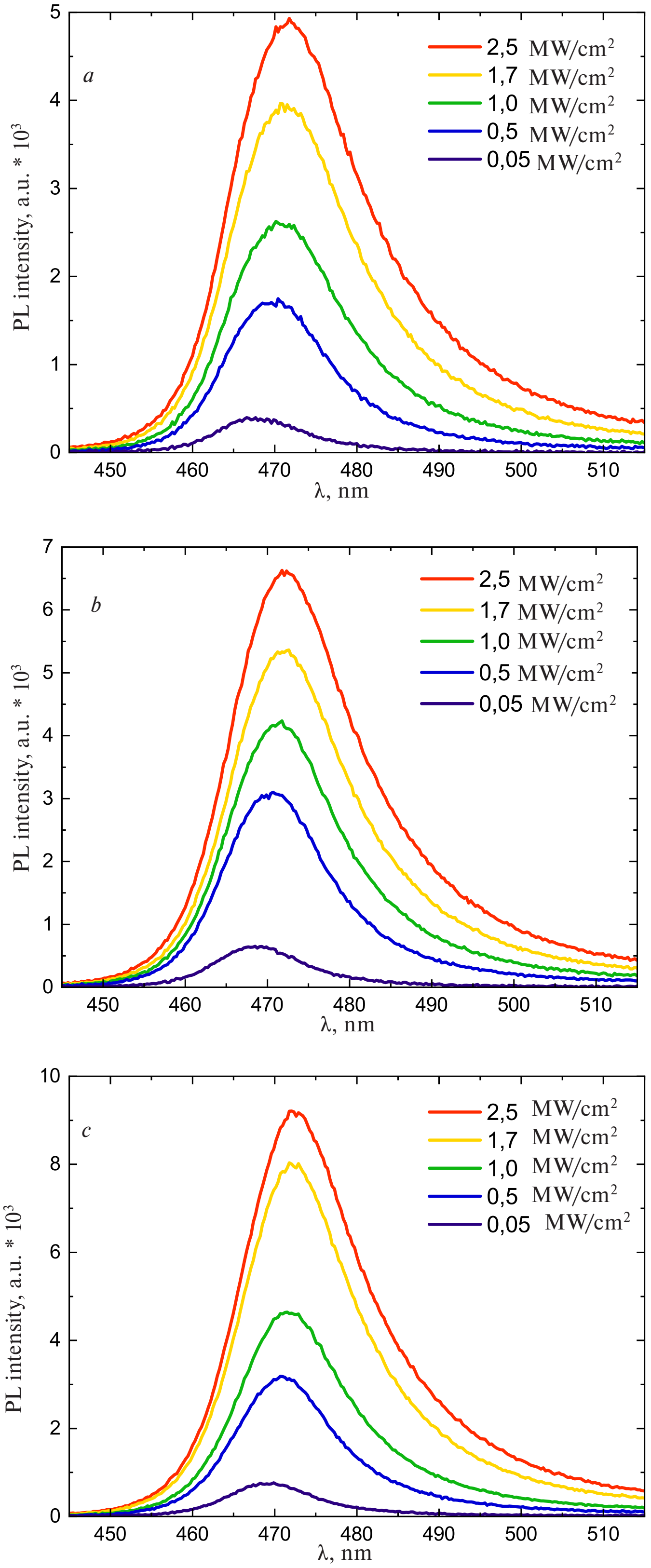

Fig. 3. (In color online) PL spectra of CdSe463 colloidal nanoplatelets at different pump intensities for solutions of low (*a*), medium (*b*) and high (*c*) concentrations (exposure time t = 3 s)

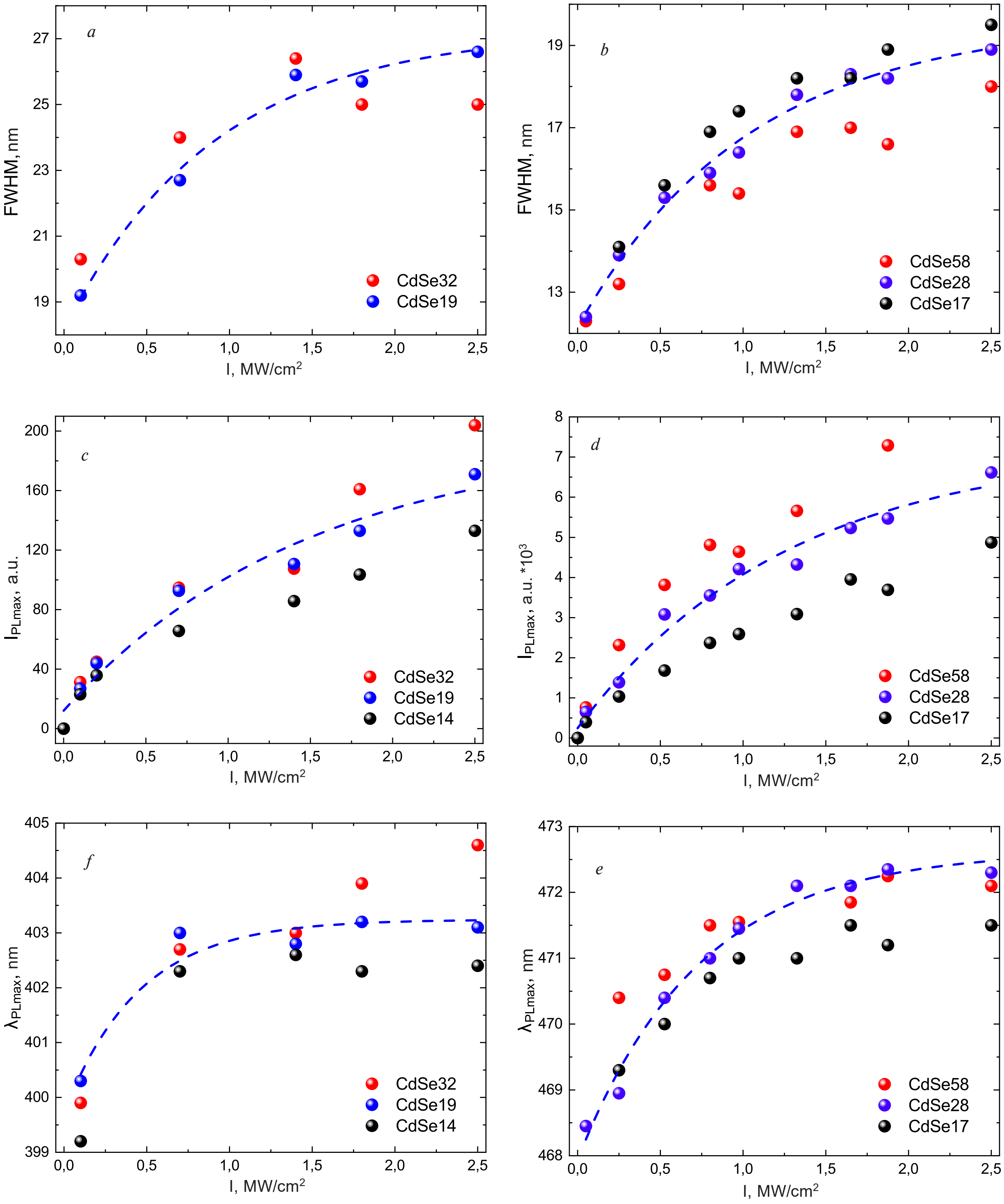

Fig. 4. (In color online) The dependence of the half-widths (*a*, *b*), intensities (*c*, *d*) and wavelengths (*f*, *e*) of the PL peaks of colloidal solutions CdSe393 (*a*, *b*, *e*) and CdSe463 (*b*, *d*, *e*) on the pump intensity. Blue dashed lines are presented to improve the visual perception of experimental data

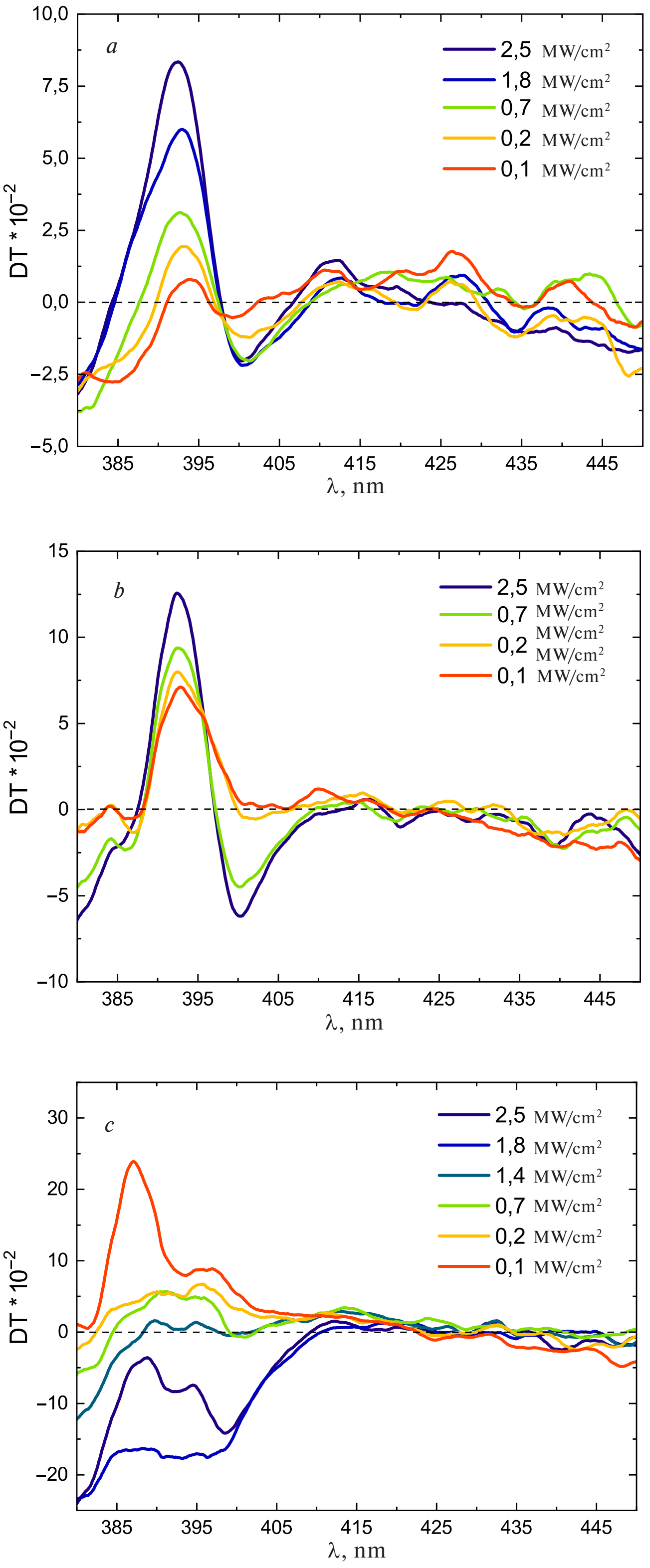

Fig. 5. (In color online) Differential transmission spectra of CdSe393 colloidal nanocrystals of low (*a*), medium (*b*) and high (*c*) concentrations at different pump intensities

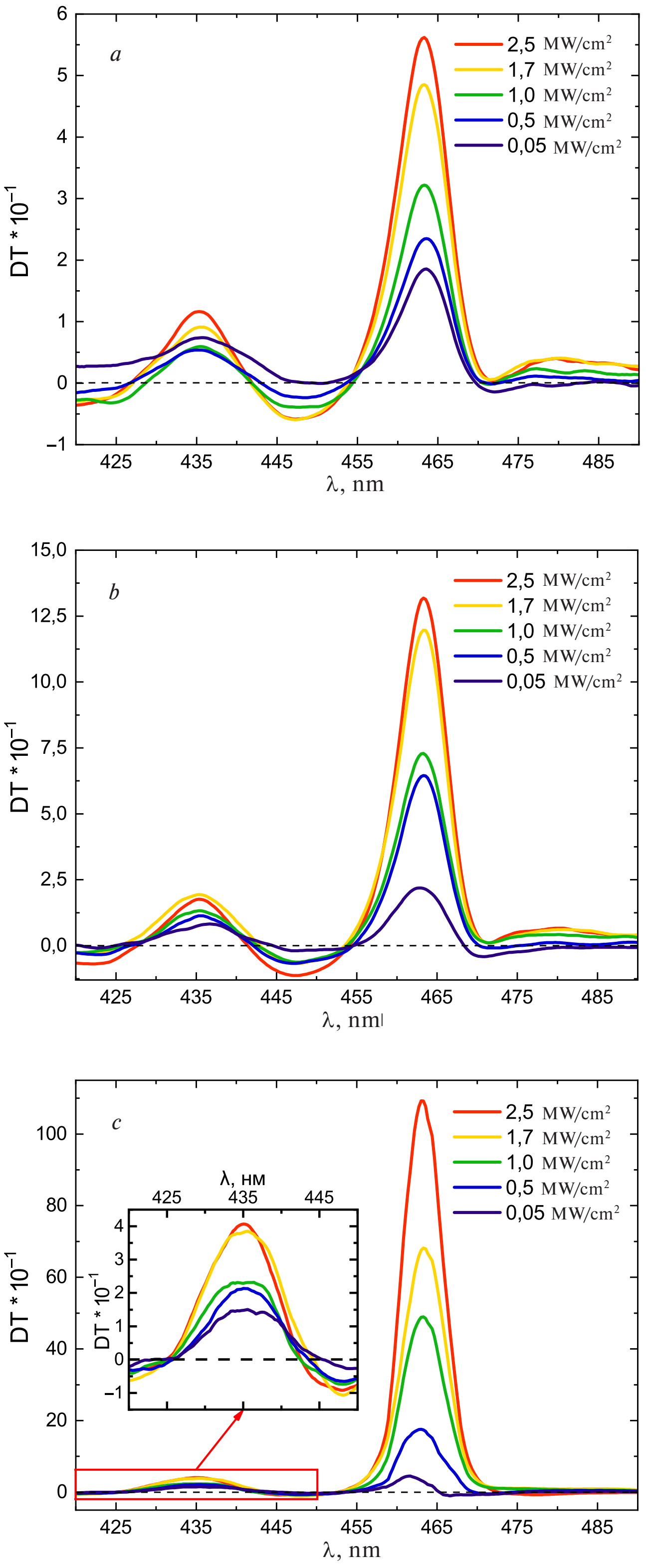

Fig. 6. (In color online) Differential transmission spectra of CdSe463 colloidal nanocrystals of low (*a*), medium (*b*) and high (*c*) concentrations at different pump intensities

$$\mathrm{DT}(\lambda) = \frac{T(\lambda) - T_0(\lambda)}{T_0(\lambda)}, \tag{1}$$

where $T(\lambda)$ and $T_0(\lambda)$ are the transmission spectra of excited and non-excited samples, respectively. Figures 5 and 6 show the DT spectra of colloidal nanoplatelets at different pump intensities. For CdSe393 series samples, the measured maximum of the DT spectrum is associated with the illumination of the exciton transition from the heavy hole sub-band. For samples of the CdSe463 series, the presence of two maxima corresponding to exciton transitions from the sub-band of light and heavy holes was established.

On the basis of these data, the dependences of the DT maxima on the intensity of the pitching were also defined (Fig. 7, 8). To determine the saturation intensities of I_s for each of the studied exciton transitions in CdSe nanoplatelets, a semi-empirical dependence of the amplification on DT values on the pump intensity [37] was used:

$$\mathrm{DT}(I) = \mathrm{DT}_{max}\left(1 - \frac{1}{1 + I / I_s}\right). \tag{2}$$

For CdSe393 nanoplatelets (Fig. 7), according to formula (2), the values $I_s \approx 0.5 \pm 0.1\,\mathrm{MW/cm^2}$ for a sample of low concentration CdSe14 and $I_s \approx 0.6 \pm 0.1\,\mathrm{MW/cm^2}$ for a sample of medium concentration CdSe19. For CdSe463 nanoplatelets (Fig. 8), the values $I_s(lh) \approx 0.9 \pm 0.5$ MW/cm^2 and $I_s(hh) \approx 1.4 \pm 0.4$ MW/cm^2 for a sample of low concentration CdSe17; $I_s(lh) \approx 0.7 \pm 0.2$ MW/cm^2 and $I_s(hh) \approx 1.6 \pm 0.6$ MW/cm^2 for sample of medium concentration of CdSe28. This indicates an increase in the amplitude of DT and the intensity of absorption saturation at the wavelengths of exciton transitions 1_{hh}–1_e for both samples CdSe393 and CdSe463, with an increase in the concentration of nanocrystals in colloidal solution, which is explained by the process of filling the exciton phase space.

Of particular interest was the case of high concentration for colloidal solutions of nanoplastics of both thicknesses. For a solution of CdSe393 nanocrystals in the case of high concentration (Fig.7*b*) at moderate values of pump intensity (up to 1.5 MW/cm²), an increase in transmission was detected, whereas with values of pump intensity above 1.5 MW/cm^2 an increase in absorption was detected.

Such a transition from an increase to a decrease in transmission can be explained by the manifestation of stimulated radiation during the resonant excitation of the exciton transition associated with heavy holes and the transition from the absorption saturation mode to the optical amplification mode [38]. Nevertheless, the presence of optical amplification or amplified spontaneous radiation is an essential, but not fully sufficient condition for the realization of forced radiation in highly limited systems. The absence of a negative DT signal for exciton transitions of 1_{hh}–1_e in colloidal solutions with a lower concentration of CdSe nanoplatelets

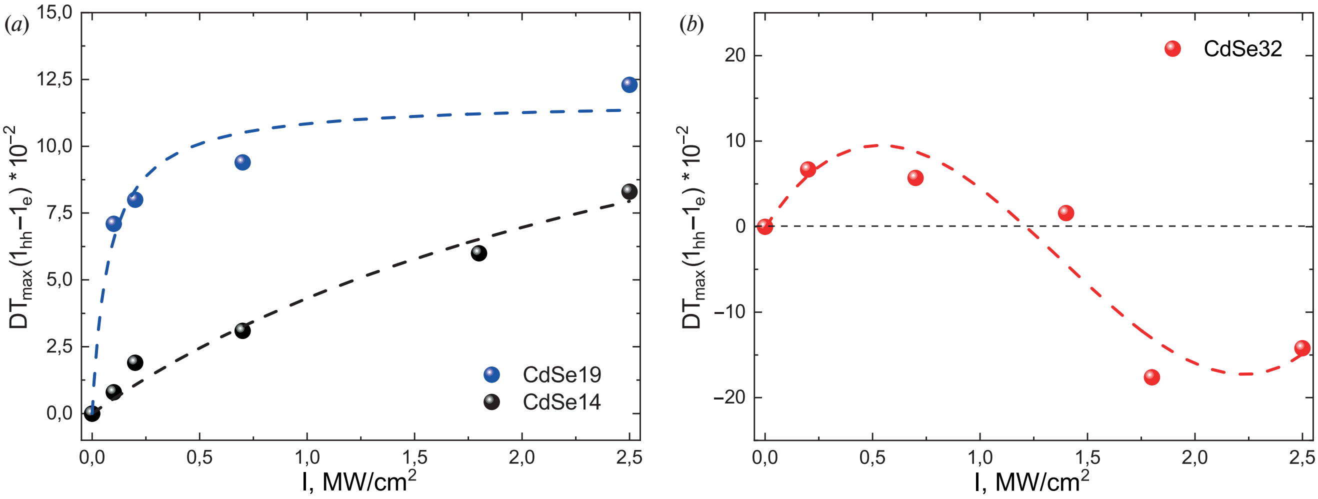

Fig. 7. (In color online) Dependence of the maxima of differential transmission corresponding to the transition 1_{hh}–1_e on the pump intensity for CdSe393 colloidal nanoplatelets of low/medium (*a*) and high (*b*) concentration

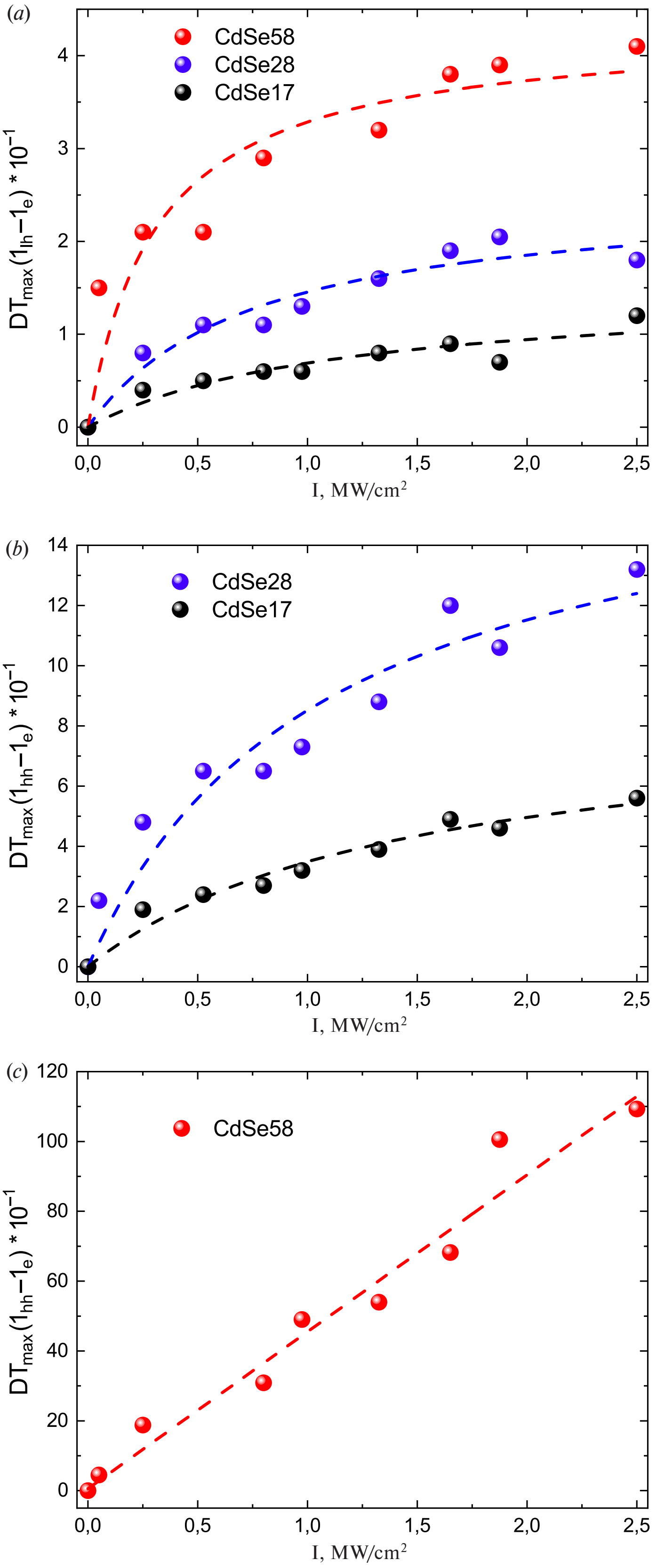

Fig. 8. (In color online) Dependence of the differential transmission maxima corresponding to peaks $1_{lh}-1_e$ (*a*) and $1_{hh}-1_e$ (*b*, *c*) on the pump intensity for CdSe463 colloidal nanoplatelets of low/medium (*a*, *b*) and high (*c*) concentrations

can be explained by insufficient pump intensity or a strongly limited length of the active medium (1 mm cuvette).

According to the DT spectra of a colloidal solution of CdSe463 nanocrystals of high concentration (Fig. 8*b*), an increase in transmission and absorption saturation was detected at the wavelength of exciton transitions associated with light and heavy holes, however, the transition from absorption saturation to forced radiation was not detected (the absence of a region with negative DT values). For the exciton transition 1_{lh}–1_e associated with light holes, according to formula (2), it was possible to determine the absorption saturation intensity $I_s(lh) \approx 0.5 \pm 0.1$ MW/cm^2.

In the case of the exciton transition 1_{hh}–1_e associated with heavy holes, it is not possible to determine this characteristic. With an increase in the excitation intensity, a sharp, almost linear increase in the amplitude of DT was revealed, which may indicate a boundary ratio between the excitation intensity and the concentration of the colloidal solution of CdSe463 nanoplatelets, exceeding which a transition to optical amplification mode is possible. Similar results were demonstrated and explained in [38]. The failure to achieve optical amplification in a colloidal solution of nanoplastics of the CdSe393 population is apparently explained by different excitation modes. In case of CdSe393 nanoplatelets, excitation is carried out at a wavelength corresponding to the resonant excitation of excitons 1_{lh}–1_e which transfer energy quite efficiently to excitons associated with heavy holes 1_{hh}–1_e [5, 6], while when excited at a wavelength of 360 nm in CdSe463 nanoplatelets Initially, free electrons and holes are born, which, in order to bind into excitons, must be given a total energy of $E_b \approx 0.77$ eV.

Thus, in the latter (non-resonant) case, high excitation intensities are required to achieve optical amplification. In addition, other difficulties may arise with the excess energy of free electrons and holes, which both lead to the transfer of energy to phonons and local heating of the nanoplatelets, and provoke nonradiative Auger recombination [23-26], which devastates the working state.

The process of Auger recombination in semiconductor nanocrystals can additionally lead to the capture of free carriers on the surface and manifest itself in PL with the participation of defects. In the studied nanoplastics with a thickness of 2.5 monolayers (CdSe393) and 3.5 monolayers (CdSe463) [39] the PL band associated with defects is located in the wavelength range of 430 –700 nm and 470 –700 nm, respectively. As a rule, the intensity of PL on defects increases at excitation levels low compared to the absorption saturation of the main exciton transition, due to the fact that the relaxation time exceeds units of microseconds

4. CONCLUSIONS

The effect of the concentration of colloidal CdSe nanoplatelets of various thicknesses on the nonlinear change in absorption during stationary excitation of charge carriers by nanosecond laser pulses has been determined. Resonant excitation of excitons associated with light holes was analyzed for samples of the CdSe393 series. An increase in the differential transmission (DT) of colloidal structures of CdSe nanocrystals of various concentrations has been detected. The enlightenment, which increases with the growth of optical excitation, is explained by the filling of the exciton phase space. An increase in the DT of a colloidal hole sub-band to the conduction sub-band was found only at pump intensities up to 1.5 MW/cm^2. At a pump intensity above 1.5 MW/cm^2, a negative DT was revealed, which can be explained by the transition from absorption saturation to optical amplification mode. During the excitation of free electrons and holes realized in the colloidal solution of CdSe463 nanoplatelets, an increase in DT was detected for all the concentrations studied. In the studied range of excitation intensities, the transition from absorption saturation to the optical amplification mode is not achieved, however, a sharp increase in the amplitude of DT is revealed, which may indicate a negative ratio of the excitation intensity and the concentration of the colloidal solution of CdSe463 nanoplatelets, if exceeded, a transition to the optical amplification mode is possible.

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