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Effect of thermal annealing on the luminescent characteristics of CdSe/ZnSe quantum dot heterostructure

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Abstract. Effect of post-growth thermal annealing within the temperature range 200 to 430 ºC for 15 min on the luminescent characteristics of CdSe/ZnSe quantum dot (QD) heterostructure was studied. Annealing at lower temperatures ($T_{ann} \leq 270$ ºC) results in an increase by a factor of 2-3 of the intensity of two photoluminescence bands observed, the first being caused by excitonic transitions in QDs and the second one being connected with the defect complex including a column II vacancy. The effect is supposed to be caused by annealing of as-grown nonradiative defects. Annealing at higher temperatures ($T_{ann} > 270$ ºC) stimulates a decrease of the QD photoluminescence band intensity and up to 100 meV blue shift of its peak position. The former is explained by generation of extended defects and reduction of the QD density. The blue shift observed at 370-430 ºC is ascribed to diffusion of cadmium from QDs that also results in reduction of the QD density. It is found that the energy of excitonic transitions in the wetting layer does not change upon annealing. Lower thermal stability of QDs as compared to that of the wetting layer has been explained by strain-enhanced lateral Cd/Zn interdiffusion via vacancies. The presence of column II vacancies in the wetting layer is proved by characteristics of defect-related PL band and its excitation spectra.

Keywords: self-assembled quantum dots, thermal annealing, Cd diffusion, CdSe, photoluminescence.

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1. Introduction

In the recent decade, the processes of quantum dot (QD) formation in CdSe/ZnSe heterostructures grown by molecular beam epitaxy (MBE) as well as their structural, optical and luminescent properties have been extensively studied [1-4]. In particular, it was found that self-organization of CdSe QDs via Stranski-Krastanow growth mode is hindered by cadmium segregation [5, 6] and Cd/Zn interdiffusion [2, 7, 8]. It was shown that because of significant intermixing of CdSe and ZnSe layers a CdSe sheet transforms into cadmium enriched CdZnSe QDs of different sizes buried into 3-4 nm thick two-dimensional CdZnSe wetting layer [2, 4, 7]. The peculiarities of structural properties of epitaxial CdSe/ZnSe QD heterostructures determine their optical and luminescent characteristics in many respects [1, 3, 4].

An interest to CdSe QDs grown by MBE was stimulated by their potential application in optoelectronic devices, in particular in green laser diodes [9-11] instead of CdZnSe quantum wells (QWs) [12, 13]. Green laser diodes based on II-VI compound low-dimensional structures are still of interest because of both absence of commercially available alternatives and high demands for such devices. Specifically, they can be a new light source for plastic optical fibres with PMMA, compact full colour projector screens, laser TV projectors, etc.

The first injection lasers and optically pumped lasers that used the sheets with CdSe QDs as an active media demonstrated several advantages over QW-based devices, namely a reduced threshold for optical pumping and higher degradation stability [10, 11]. Heterostructures with CdSe QDs were found to be more stable against photo-degradation as compared to CdZnSe QWs [14]. These advantages were explained by effective

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localization of carriers in QDs that hinders their diffusion to relaxed QDs and other regions where carriers can recombine nonradiatively and stimulate defect multiplication in the active region. However, degradation processes in CdSe QD heterostructures have not been studied in details.

In particular, the peculiarities of Cd/Zn interdiffusion stimulated by external influences in as-grown CdSe QD heterostructures have not been studied at all. At the same time, it is known that degradation of light-emitting devices based on CdZnSe QWs is accompanied not only by noticeable reduction of QW emission caused by dislocation multiplication in active region, but also by the shift of QW emission band towards high energy spectral region (blue shift) due to Cd/Zn interdiffusion across QW heterointerface [12, 15, 16]. Study of the processes of Cd/Zn interdiffusion in CdZnSe/Zn(S)Se QW heterostructures by applying thermal annealing revealed that diffusion of Cd from the QW is governed by column II vacancies (V_{Zn} or V_{Cd}) [17] and the diffusion coefficient of Cd can be varied by about two orders of magnitude by varying the concentration of column II vacancies [18]. It was shown also that intermixing of the materials of QW and the barriers under thermal annealing occurs via the vacancies generated at the surface of the sample and diffuse into the structure [17]. In addition, we have found earlier in CdSe/ZnSe QD heterostructures that column II vacancies during the growth gather in the CdSe layers and influence significantly the QD self-organization process up to its full suppression [19]. It can be supposed that presence of the vacancies in the wetting layer will influence degradation of QD luminescent characteristics, too.

In this paper, we report photoluminescence (PL) study of CdSe/ZnSe QD heterostructure subjected to thermal annealing with the aim to find a method for improvement of QD luminescent characteristics and to obtain additional information about their degradation connected with Cd/Zn interdiffusion.

2. Experimental details

The studied structure was grown on (001) GaAs substrate by MBE and contained 250-nm thick ZnSe buffer layer, 12 vertically stacked CdSe inserts separated by ZnSe spacers of about 15 nm thickness and 150-nm thick ZnSe cap layer. Nominal thickness of CdSe inserts was 5 monolayers.

The growth rate was 5 nm/min. The growth temperature was 280 ºC for ZnSe buffer layer and 230 ºC for the rest of ZnSe layers as well as for CdSe layers. To stimulate QD formation, after the deposition of each CdSe layer the Cd beam was blocked, and the structure was heated up to 340 ºC and then cooled down to 230 ºC under Se flux. The duration of both steps was 4 min. The reflection high-energy electron diffraction (RHEED) was used for *in situ* control of three-dimensional island formation.

The PL spectra and PL excitation spectra were measured at 77 K. The PL spectra were excited by the light of 250-W halogen lamp at the excitation wavelength λ_{exc} = 440 nm and by 365-nm line of 500-W Hg-lamp. The PL excitation spectra were measured using a light of halogen lamp passing through a grating spectrometer as an excitation source. The PL signal was dispersed using a prism spectrometer (when the PL was excited by the light of a halogen lamp) or a grating spectrometer (when the PL was excited by the light of an Hg-lamp) and collected by photoelectronic multiplier.

Samples cut from wafer were thermally treated for 15 min at 200, 220, 270, 300, 335, 370 and 430 °C in nitrogen ambient to avoid surface oxidation.

3. Experimental results

The PL spectrum of the as-grown sample is shown in Fig. 1a (curve 1). In the spectrum, the band I_{QD} peaking at 544 nm (2.277 eV) and caused by radiative recombination of excitons in QDs dominates. The full width at a half maximum (FWHM) of this band is ~100 meV and is related with dispersion of QDs both in composition and in size. In the PL spectrum, a defect related band I_D peaking at 670 nm (1.844 eV) and of ~300 meV FWHM is also present. The intensity of I_D band is more than 10 times lower than that of I_{QD} band.

Fig. 1a also shows the excitation spectra of both the QD and defect related bands (curves 2 and 3, correspondingly). The excitation spectrum of I_{QD} band was detected in the low energy tail of the band, while the excitation spectrum of I_D band was measured in the band maximum. In the spectra, in addition to the region caused by absorption of excitation light in ZnSe layers ($\lambda \le 445$ nm) two peaks can be distinguished: (i) the peak *WL* at ~505 nm (2.455 eV), and (ii) the peak *X* at ~470 nm (2.638 eV). Our previous investigations of similar multistack QD structures have shown that the peak *WL* is caused by ground state heavy-hole-like exciton absorption in the wetting layer, while the peak *X* can be ascribed to ground state light-hole-like exciton absorption in the wetting layer [19-21]. We have found earlier the linear dependence of the I_D band maximum position versus the spectral position of *WL* peak in I_D band excitation spectra [20,21]. Approximation of this dependence to the value of the ZnSe energy gap revealed that I_D band is caused by defect complex including column II vacancy and shallow donor. Thus, the excitation spectra of I_D band indicate the presence of column II vacancies in the wetting layer of the as-grown sample.

The changes introduced to the PL and PL excitation spectra by thermal treatment at 270, 300 and 370 °C are also depicted in Fig. 1b, c and d, correspondingly. Fig. 1b shows that annealing at 270 °C results in a noticeable increase in the intensity of both the PL bands and in no change of their spectral position and excitation spectra. However, in the sample annealed at 300 °C the

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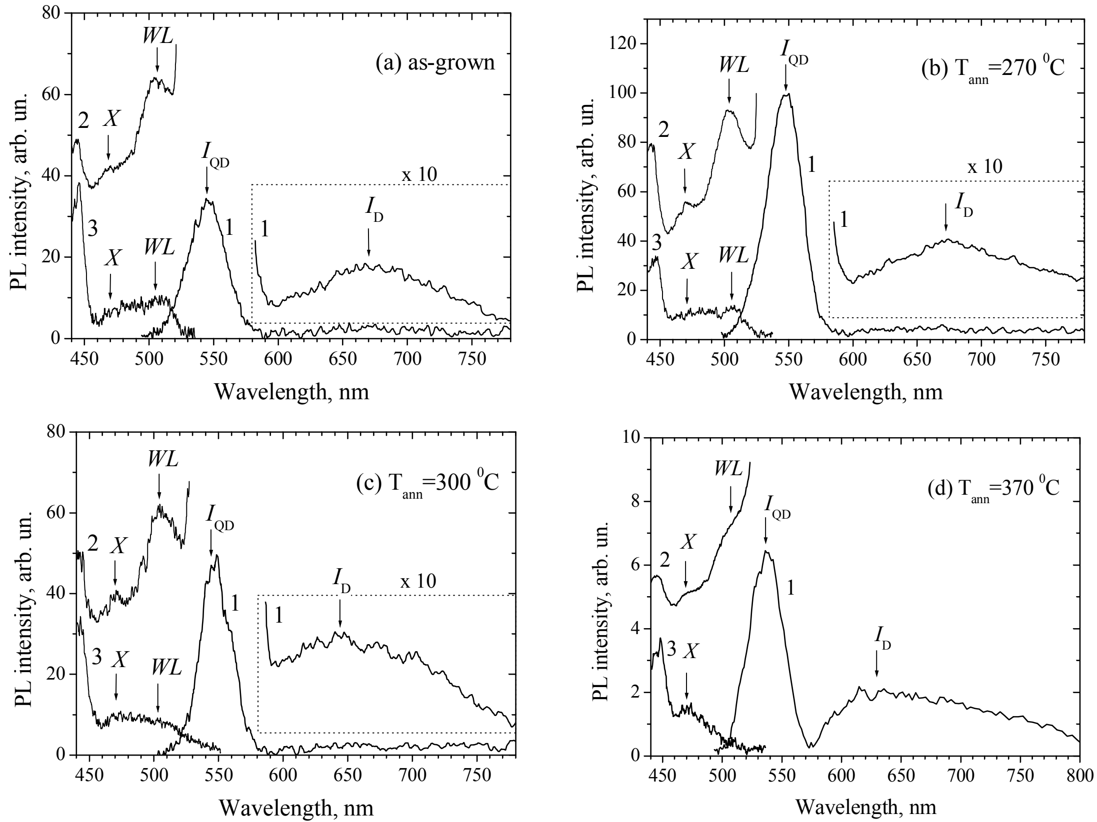

Fig. 1. PL (curves 1) and excitation spectra of I_{QD} band (curves 2) and of I_D band (curves 3) of the as-grown sample (a) and of the sample annealed at 270 (b), 300 (c) and 370 ^{0}C (d). The excitation spectra of I_{QD} and I_D bands are detected in low energy tail of the band and in the band maximum, respectively. T=77 K, λ_{exc}=440 nm.

I_D band intensity stops growing, while the intensity of I_{QD} band starts to decrease (Fig. 1c). These are accompanied by the shift to shorter wavelengths of the spectral position of the I_D band maximum and the decrease of *WL* peak intensity in its excitation spectrum. At the same time, no change is found in the excitation spectrum of the I_{QD} band. In the sample subjected to thermal annealing at 370 °C, the intensity of both PL bands decreases and their spectral position shifts to shorter wavelengths (blue shift) (Fig. 1d). The excitation spectrum of I_{QD} band still does not change, but in the excitation spectrum of I_D band the intensity of *WL* peak keeps decreasing.

Fig. 2 shows the way in which both the spectral position (a) and the intensity (b) of the PL bands change under annealing in the whole range of the annealing temperatures. This range can be divided by 2 regions: (i) low annealing temperatures ($T_{ann} \leq 270$ °C), and (ii) higher annealing temperatures ($T_{ann} > 270$ °C).

At low annealing temperatures ($T_{ann} \leq 270$ °C), the spectral position of both I_{QD} and I_D bands does not change, while their intensity increases. It should be noted that PL intensity starts growing already at 200 °C and 2 to 3 times increases in the sample annealed at 270 °C.

At higher annealing temperatures ($T_{ann} > 270$ °C), the intensity of the I_{QD} band falls down and decreases more than by the order of the value after thermal treatment at 430 °C. The intensity of the I_D band decreases too, but starting from 370 °C and more slowly than that of I_{QD} band.

Spectral position of the I_{QD} band does not change upon annealing up to 335 °C and is blue-shifted within the range 370-430 °C. The shift amounts to ~100 meV in the sample annealed at 430 °C (Fig. 2a). The blue shift of I_D band position is found in the range of annealing temperatures lower than that of the I_{QD} band (300-370 °C) and is accompanied by the changes in the excitation spectrum mentioned above (Fig. 1c). The spectral position of the I_D band in the sample annealed at 370 °C comes to ~630 nm (1.965 eV) and is not influenced by annealing at 430 °C. The excitation spectrum of the I_D band does not change at 430 °C, too.

Fig. 2a also shows the dependence of the spectral position for both *WL* and *X* bands on the annealing temperature. The spectral positions were extracted from the I_{QD} band excitation spectrum. As one can see, contrary to the I_{QD} band position, they are not influenced by annealing at least up to 335 °C. This is also true for the spectral position of *WL* and *X* bands extracted from the excitation spectrum of the I_D band. However, in this case the *WL* peak intensity decreases noticeably as opposed to the *X* band (see Fig. 1c,d). This is the evidence that the *WL* and *X* peaks are due to light absorption in different parts of heterostructure. We suppose that *WL* peak is indeed caused by ground state

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exciton absorption in the wetting layers, while the *X* peak is probably due to light absorption in ZnSe layers doped with cadmium during the growth. Specifically, similar layers have been found by us in MBE-grown CdZnSe/ZnSe quantum well heterostructures with high resolution X-ray diffraction and low temperature PL spectroscopy [22].

Unfortunately, *WL* peak was not detected in the I_{QD} band excitation spectrum of the sample subjected to thermal annealing at 430 °C. Most likely, this is caused by both the noticeable decrease of the I_{QD} band intensity and blue shift of its peak position. Therefore, to obtain the energy of the ground state exciton transition in the wetting layers, the PL spectra at higher excitation levels were studied. Fig. 3 presents normalized PL spectra of as-grown (curves 1, 2) and annealed at 430 °C (curves 1′, 2′) samples under excitation by 365-nm line of Hg-lamp, where the increase of a curve number corresponds to the ten-fold increase of the excitation power density. As the excitation level rises, a FWHM of the I_{QD} band increases approximately up to 1.3 times in the sample

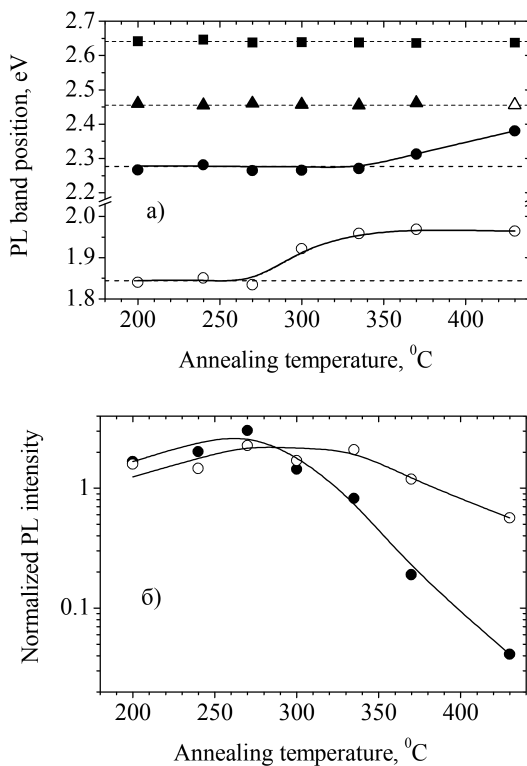

Fig. 2. Spectral position (a) and intensity normalized to that of the as-grown sample (b) of the I_{QD} (filled circles), I_D (open circles), *WL* (triangles) and *X* (filled squares) bands versus annealing temperature. The spectral position of the *WL* and *X* bands is extracted from the excitation spectra of I_{QD} band (filled triangles and squares) and PL spectrum excited by 365-nm line of Hg-lamp (open triangle). Dashed lines present spectral position of the I_{QD}, I_D, *WL* and *X* bands of the as-grown sample. T=77 K, λ_{exc}=440 nm.

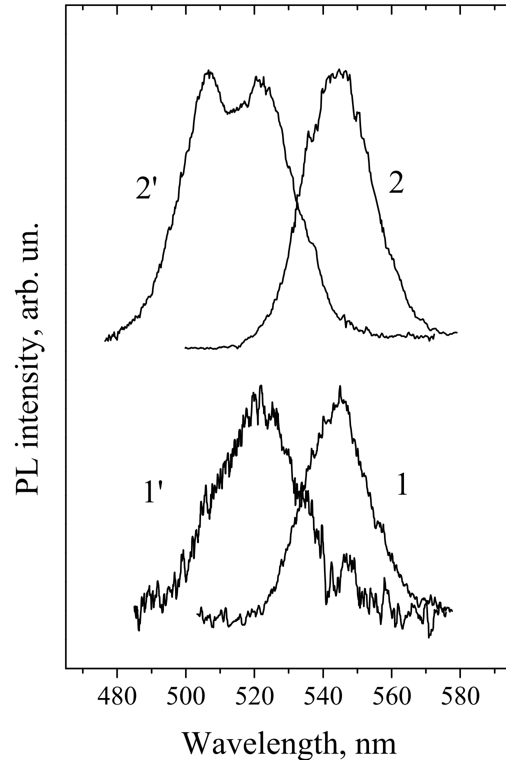

Fig. 3. Normalized photoluminescence spectra of the as-grown (curves 1, 2) and annealed at 430 ^{0}C (curves 1’, 2’) samples excited by 365-nm line of 500-W Hg-lamp at the excitation power density of P_0 (curves 1, 1’) and $10P_0$ (curves 2, 2’). T=77 K.

annealed at 430 °C, but does not change in as-grown sample. In addition, in annealed sample a new band at ~505 nm arises in the shortwave wing of the I_{QD} band. Generally, this band can be due to radiative transitions through the QD excited states as well as through the ground states in smaller QDs or wetting layer. Taking into account a more than by an order of magnitude decrease in the QD luminescence intensity as a result of thermal treatment at 430 °C, this band has to be ascribed to the wetting layer emission. Spectral position of this emission is marked in Fig. 3 by the open triangle. The total dependence presented in Fig. 3 by triangles indicates that the energy of the ground state exciton transition in the wetting layers is not modified by thermal treatment in the whole range of annealing temperatures.

4. Discussion

Thus, the post-growth thermal treatment of CdSe/ZnSe QD heterostructures results in changes in the PL intensity (at first the increase and then the decrease) and in the shift of PL band position to the high-energy spectral region (blue shift).

The increase of the PL intensity is observed at low annealing temperatures ($T_{ann} \le 270$ ^{0}C) and is not

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accompanied by any change in the spectral position of PL bands or in their excitation spectra. The effect of PL intensity increase has been found earlier in CdZnSe/ZnSe QW heterostructures subjected to post-growth thermal annealing at 250-700 ^{0}C [23-27] and explained by interfacial smoothing resulting from the small-scale lateral diffusion. The increase in the intensity of QW luminescence band was observed without any changes in its spectral position [24-26] or with a noticeable blue shift [23, 27]. A similar effect was also found in the InGaAs/GaAs heterostructures with QWs [28, 29] or QDs [30-32] subjected to thermal treatment and was ascribed to QW interface smoothing [28] or nonradiative defect annealing [29, 32]. We suppose that the increase in intensities of both I_{QD} and I_D bands is the result of the annealing of as-grown defects (point defects, for example) that act as the centers of nonradiative recombination and are located in different layers of heterostructure.

The decrease in intensity of both PL bands observed at higher temperatures (T_{ann}>270-335 ^{0}C) is probably caused by generation of the centers of nonradiative recombination under thermal treatment. In particular, it can be due to multiplication of extended defects (dislocations) nearby the stacking faults at ZnSe buffer layer/GaAs substrate interface and their following growing into the active layers (QD layers). It was proposed earlier to explain both quenching of CdZnSe QW emission after rapid thermal annealing treatment [33] and rapid degradation of blue-green laser diodes based on CdZnSe QWs [12].

However, the only rise of nonradiative defect concentration in the result of annealing can not explain different rates of the decrease of the I_{QD} and I_D band intensities. As it was mentioned above, quenching of the QD emission occurs much sharply than that of defect-related band. This can be due to the increase of concentration of defects giving rise to I_D band and/or the decrease of QD concentration.

Of the two mechanisms, the former can be realized if column II vacancies are generated during annealing at the surface of the sample and then diffuse into the structure as it was observed in [17]. This explanation agrees with the blue shift of I_D band position and the decrease of *WL* peak in its excitation spectra. Both of these are observed in the same range of annealing temperatures (T_{ann}=300-335 ^{0}C) and are very likely caused by the increase of contribution of emission of vacancy-related defects localized in the ZnSe layers to the I_D band (Fig. 1c, d). At the same time, the blue shift of defect-related band is accompanied by the increase of its FWHM, which in the sample annealed at 430 ^{0}C is 1.5 times larger than that in the as-grown one. In addition, the *WL* peak decreases but not disappears in the I_D band excitation spectrum upon annealing. These indicate that even after thermal treatment at 430 ^{0}C the I_D band remains mutlicomponent and the contribution of defects localized in the wetting layers to the I_D band is large enough. The data obtained imply that the total concentration of column II vacancies increases in the structure.

On the other hand, a noticeable blue shift of the QD emission band is found at higher annealing temperatures (T_{ann}=370-430 ^{0}C). The shift is apparently caused by Cd outdiffusion from the QDs, which results in gradual dissolution of QDs in the surrounding matrix. The latter means a decrease in the QD concentration. Diffusion of Cd from the QDs will also result in the growth of QD sizes and further disappearance of spatial confinement for carriers in the QDs. This also implies the decrease of QD concentration. Reduction of the QD concentration causes predominant decrease of the I_{QD} band intensity as well as the increase in the intensity of defect-related and wetting layer PL bands as recombination channels competing with the QD emission. This agrees well with the supposition made above of the appearance of the wetting layer PL band in the emission spectra after annealing at 430 ^{0}C (Fig. 3 curve 2').

Therefore, it can be assumed that a difference in the degradation rates of the I_{QD} and I_D band intensities is caused by the influence of two effects that dominate in different ranges of the annealing temperatures: (i) the increase of column II vacancy concentration (T_{ann}=300-430 ^{0}C), and (ii) the decrease of QD concentration (T_{ann}=370-430 ^{0}C).

The presented results make it possible to suppose that just the column II vacancies, that diffuse from the surface of the sample into the structure stimulate the process of Cd/Zn interdiffusion across the ZnSe/QD layer interface and cause a blue shift of the I_{QD} band. But in this case, not only Cd outdiffusion from the QDs but also Cd outdiffusion from the wetting layers should occur. However, we did not found any increase in the energy of excitonic transition in the wetting layers as opposed to the one in QDs (Fig. 2a). A similar result, namely much larger blue shift of the QD emission band as compared to that of the wetting layer and QW emission bands, has been observed during rapid thermal annealing of InGaAs/GaAs QD heterostructures [30, 31, 34, 35]. Structural investigations have proved fast dissolution of the QDs in surrounding matrix during annealing [31, 34] and predominant increase of the lateral sizes of the QDs [34] that implies lateral interdiffusion. We suppose that in our samples lateral diffusion, i.e. diffusion of Cd from the QDs in surrounding wetting layer, prevails over Cd/Zn interdiffusion across the QD/ZnSe layer interface. The diffusion occurs via vacancies generated during the growth process in the wetting layers. This is the non-Fickian strain-enhanced interdiffusion, which has been proposed as an explanation of some results of rapid thermal annealing treatment of InGaAs/GaAs QWs and QDs [34, 36]. Since the lattice constant of CdSe exceeds that of ZnSe, the CdSe layer is under compressive strains that relax partially during QD formation [37]. The compressive strain enhances the vacancy concentration [36], for example by stimulating the process of vacancy gettering in the QD layer. Thus, we can suppose that the

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column II vacancy concentration in the CdZnSe wetting layers should be much higher than that in the ZnSe layers. It should be noted that in the InGaAs/GaAs QD heterostructures the presence of cation vacancies in the QD layer was only supposed to explain experimental results but not proved directly [34, 36]. Our data, namely such characteristics of defect-related band as its spectral position, FWHM and its excitation spectrum indicate directly the presence of column II vacancies in the wetting layers and confirm proposed mechanism of interdiffusion.

5. Conclusions

In conclusion, we have found that post-growth thermal treatment of CdSe/ZnSe QD heterostructures influences the QD luminescence intensity and results in up to 100 meV blue shift of the QD luminescence band position. It is revealed that annealing of the samples at temperatures up to 270 ^{0}C allows raising the QD luminescence intensity by 2 to 3 times with no changes in other QD luminescent characteristics. The effect is supposed to be due to annealing of as-grown centers of nonradiative recombination. The blue shift occurs at annealing temperatures of 370-430 ^{0}C concurrently with the decrease in the QD luminescence intensity and is not accompanied by the changes in the energy of the ground state excitonic transition in the wetting layer. This effect is ascribed to strain-enhanced lateral Cd/Zn interdiffusion in the QD layers through the vacancies generated during the growth of the structure.

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