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System of localized excitons on oxygen complexes in CdS

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Intense CdS luminescence in the blue and green spectral regions is widely used in all areas of optoelectronics. In this spectrum band are working on lasers CdS. This paper presents the results of a study of the exciton region of the CdS spectrum based on the theory of anti-intersecting bands (bands anticrossing theory-BAC) with the involvement of broader initial data for the analysis of optical properties. Depending on the growth conditions, the presence and change of oxygen concentration and intrinsic point defects determining the composition of crystals are taken into account. The concept of the nonuniform distribution of isoelectronic oxygen centers in the bulk of CdS due to their predominant segregation on compensating stacking faults is introduced. Cathodoluminescence (CL) spectra were studied using various recording methods, excitation intensity and temperature, as well as pulsed CL at high excitation intensities. In a scanning electron microscope from local registration and a high excitation density, the emission of the edge luminescence components of CdS was detected at 300 K. To analyze the optical data, we used the capabilities of the method for constructing band models based on the BAC theory, which collects extensive and multilateral information about specific samples. A model of a $\mathrm{CdS \cdot O}$ multizone with stacking faults, which determines the spectrum of edge emission is presented. An explanation of the nature of the green edge emission of cadmium sulfide as excitons localized on oxygen-containing complexes in SF layers has been obtained for the first time.

It was found that the system of levels of localized excitons at stacking faults does not change either with temperature up to 300 K, or with a change in the oxygen solubility in the crystal to the limiting one. It is shown that the presence of isoelectronic oxygen centers appear itself in the electro-physical properties of crystals. Recommendations are given for the diagnostics of crystals suitable for the creation of luminescent systems or lasers that are stable in operation.

Keywords: isoelectronic centers, bands anticrossing theory, localized exciton, stacking faults, point defects, stimulated radiation.

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1. Introduction

Many researches have been attracted by narrow bands at the CdS absorption edge [1–11]. The well known studies of Gross et al. [2] presented the first results of studies of the excitons in $\mathrm{A^{II}B^{VI}}$. The main features of the group of bands that combine the components of the green edge emission CdS (edgeemission — EE) are described in fundamental work [1]. The CdS EE nature was explained by applying a recently established model of donor-acceptor (D-A) pairs [3]. According to this model, an acceptor could be a lattice defect, while various types of impurity donors determined the edge emission bands. The various bands were regarded as a set of D-A pairs. This trend, being a key one, is still the same up to now [4,5]. In doing so, it is not taken into account that only several CdS EE features are similar to those of the D-A pairs, but the others clearly contradict them [1]. All the researchers noted a dependence of the emission on a composition of the crystal [1,7–11]. The oxygen is not taken into account, but it is typical that the CdS edge emission is observed in the samples with excessive cadmium [1].

The development of the theoretical basis of the solid state physics and the creation of the quite recently established bands anticrossing theory (BAC) [6] resulted in deeper perception of an actual defect structure of the crystals.

2. Samples for the study

Considering the inevitable presence of oxygen and influence of the intrinsic point defects on the CdS radiation spectra, we have studied real single crystals for modificability of their composition depending on growth conditions [11–16]. The studies were based on a group of the crystals grown out of a gas phase within the CdS homogeneity [12]. In accordance with the crystal compositions, the equilibrium of the intrinsic point defects has been calculated and the oxygen concentrations [O], have been determined by data of a developed and tested procedure of the gas-chromatographic analysis (GCA) [13].

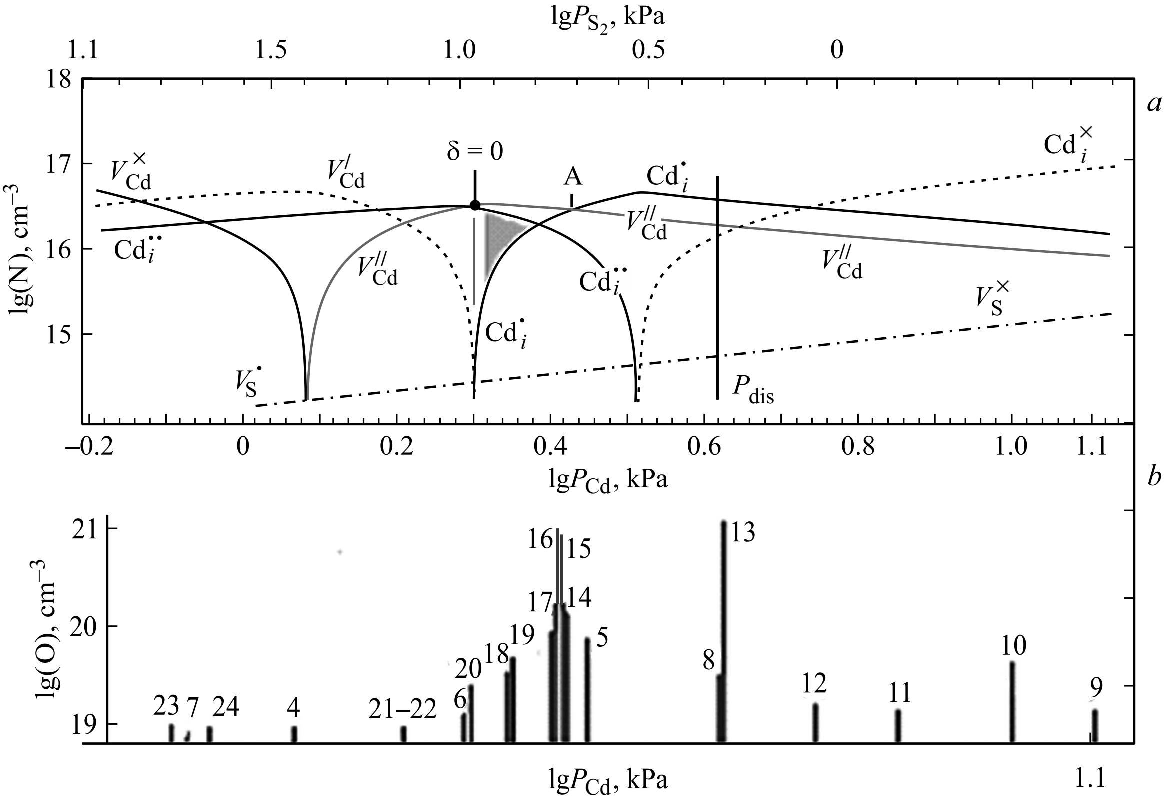

Figure 1. CdS crystals with indicating the growth conditions at 1100°C and the oxygen content therein (*b*) in comparison with a design equilibrium diagram of intrinsic point defects (IPD) (*a*). The „stoichiometry region" is blackened. The charge states of the defects 0, +, ++, −, -- correspond to the designations: ×, •, ••, /, // [1].

Fig. 1 shows these data. The conditions of the growth of the CdS crystals at 1100°C of the gas phase at the controllable sulfur vapor pressure (S) are described in [11]. A prime mark with the sample from number is located on the figure to determine the crystal composition as the set of intrinsic point defects. The oxygen concentration [O] is characterized by a prime mark length on Fig. 1, *b*. The „stoichiometry region" is blackened and limited by a sharp concentration drop of $Zn_i^{\bullet}$ on the diagram and the concentration of O_S in the crystals. This region has a minimum of the sum of the total number of the defects.

The defect formation was analyzed to give a base for studying the optical properties of these crystals taking into account their dependence on the type of the intrinsic point defects. Furthermore, the studies have also taken into account features of the crystal structure of the samples with stoichiometry deviations [11].

3. Cathodoluminescence spectra

Fig. 2 selectively shows the cathodoluminescence spectra (CL) of the area of group of the CdS crystals, which are grown within the homogeneity. The spectra are measured by the M1[11] procedure in a wide beam at the excitation density of $10^{22}\,\mathrm{cm}^{-3}\cdot\mathrm{s}^{-1}$ and the possibility of obtaining the data from separate areas of a radiated crystal 0.5−1 mm. A depth of the information layer is $\sim 0.5\,\mu$m. As it is clear from Fig. 2, there are evident intense series of the edge emission with main lines 514−516 nm and a LO motive. The typical spectrum is represented by the crystal № 15. The entire group of the crystals (Fig. 1) was studied [11] to demonstrate that these bands have the maximum intensity in regions closer to stoichiometry region (Fig. 1, *a*). Below, the present paper provides data disclosing a nature of these bands: their relation to the oxygen concentration and IPD type, the temperature dependence and the presence of the stacking faults [17].

As for the exciton band of 489 nm, it does not reveal the dependence on [O] for the whole group of the crystals (Fig. 2). This stability can be related to a quite constant composition of clean layers in the crystals with stacking faults (SF). The stacking faults, which are formed for compensation of the O_S-centers, hold almost the entire oxygen by being saturated to the limit. In doing so, the composition of the light layers is kept constant. Let us designate the band of 489 nm as the exciton of the light layers $(E_{ex}^{cc})^1$.

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[1] Position of the exciton A-band CdS as per the data [1] — 487 nm (80 K). In accordance with the spectrum position of the band of 489 nm, the light layers are quite clean in terms of oxygen $\sim 2\cdot 10^{19}\,\mathrm{cm}^{-3}$.

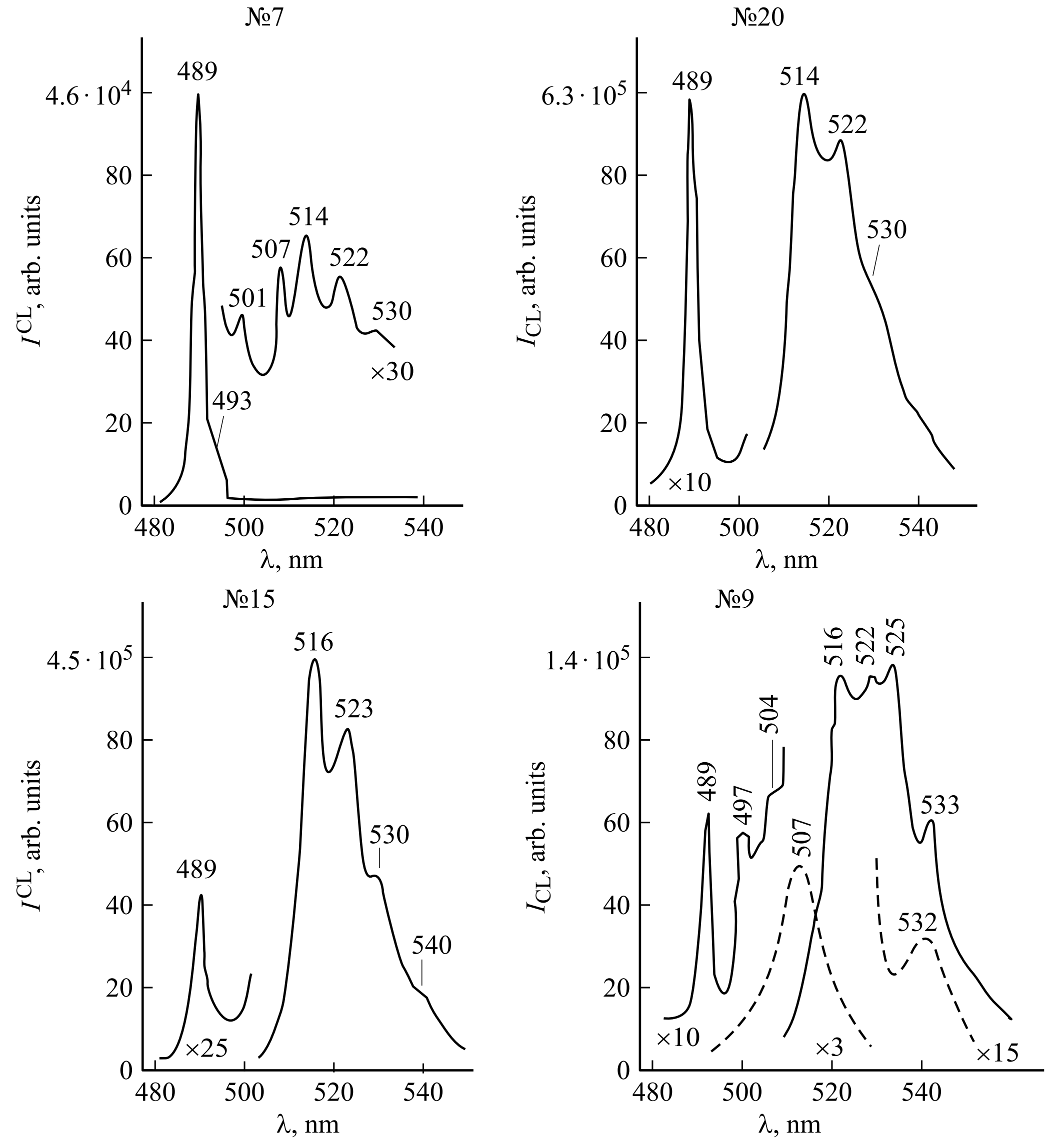

Figure 2. CL spectra measured by the M1 procedure at 80 K (dashed — 300 K). The excitation density $G = 10^{22}\,\text{cm}^{-3} \cdot \text{s}^{-1}$.

The nature of this CdS exciton band was refined by shooting as per the procedure M2[11] at the temperatures below the nitrogen ones and at the higher resolution. Fig. 3 shows typical spectra of the CdS single crystals grown at various vapor pressures of the components.

As it is clear from Fig. 3, the exciton region of the spectrum is resolved with two bands of 489 and 487 nm. The spectra of the entire group of the crystals were studied to show that each of the bands of 489 and 487 nm is accompanied with various „edge“ emission and depends variously on the composition of the crystals and the oxygen content.

The exciton band of 487 nm is observed in the spectra of most gas-phase samples, which are clean in terms of oxygen and grown at excessive sulfur vapor pressures.

In the edge region of the spectrum of these crystals, at 47 K we have observed the only series of the bands with the main line of 507 nm and LO repetitions (see the insert of Fig. 3). This most short-wave (SW) series in the edge spectrum region has been observed only at 47 K as an independent one at the minimum quantity of oxygen of $8.7 \cdot 10^{18}\,\text{cm}^{-3}$ in the sample № 7.

This type of the emission is detailed in the papers [11,14,16,18] for CdS ZnS and ZnSe as SAL[2]. The emission is observed at the low temperatures for the

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[2] These papers designate two kinds of self-activated emission which is different in terms of its nature $A^{II}B^{VI}$. This SA-emission at the deep recombination levels for CdS ~ 0.8 eV, due to the $\{O_SCd_i^{\bullet}V_{Cd}^{//}\}^{/}$ compounds. It is realized for the compositions of the crystals, which at big concentrations contain the compound components, in particular, $Cd_i^{\bullet}$ and O_S. Fig. 1, *a* shows these compositions from the border of „stoichiometry region“ to excessive cadmium.

The SAL-emission is realized by other compounds The emission is observed for the compositions outside the „stoichiometry region“, which are close to the point $\delta = 0$ on Fig. 1, *a*. The SAL-centers appear in accordance with low concentrations of oxygen.

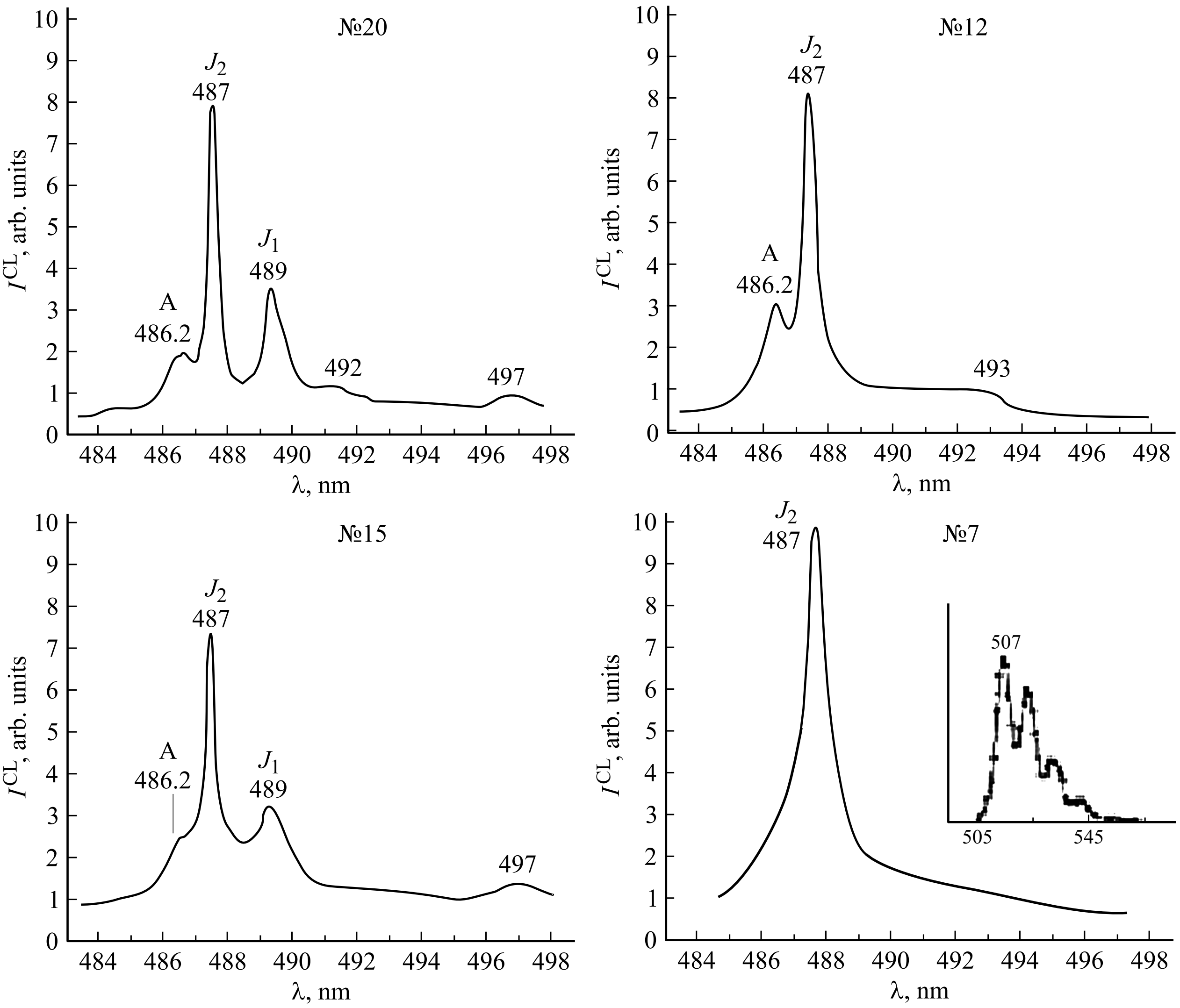

Figure 3. CL spectra recorded as per the procedure M2[3] at 47 K and at the excitation density of $10^{21}\,\text{cm}^{-3}\cdot\text{s}^{-1}$. The insert shows a series of the edge emission of the local region of the heterogeneous crystal № 7.

compositions which are close to the crystal clean in terms of oxygen.

As for the exciton band of 489 nm, most it is not observed in the spectra of the crystals, which are clean in terms of oxygen (Fig. 3, the samples Nos. 7, 12). In all the cases, at 80 K this band of 489 nm in the spectra of Fig. 2 was accompanied by lines of the CdS edge emission of 514, 515, 516 nm, which are not detected with decrease in the measurement temperature to 47 K (Fig. 3), even when the band of 489 nm is observed. However, the band of 489 nm is related by us to the stacking faults and, as shown below, it can be considered that its detection signifies a SF in the crystal.

We also note features of the spectra, which are revealed when investigating the entire group of the crystals by local shooting. For separate areas of the heterogeneous crystals, for example, the sample № 10, along with $E_{\text{ex}}^{\text{cc}}$ 489 nm, there is a band of 497 nm appearing and amplifying. And at the same time, the edge emission components are reducing up to a background level.

The band of 497 nm is also distinguished as an individual one (Fig. 4). Within the reflection spectra, it is accompanied by a distinct edge of the additional absorption of 496 nm (Fig. 4, the curve *2*), which is limited on the long-wave side by the selective band 495–497 nm of the half-width of 9–20 meV. According to the spectral position of the exciton and the results of the GCA analysis, the band approaches the level of limiting doping with oxygen.

When the band gap of the base is reduced due to the limit alloying by oxygen, almost all the edge emission components are overlapped with the band of 497 nm. The sample № 18 (Fig. 4) is resolved only with the most long-

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[3] The helium cryostat allowed getting to the temperature of 28–30 K, the excitation level $10^{22}\,\text{cm}^{-3}\cdot\text{s}^{-1}$. A depth of the information layer is $\sim 0.5\,\mu$m. The diffraction grating provided for the dispersion of 7.4 Å/mm.

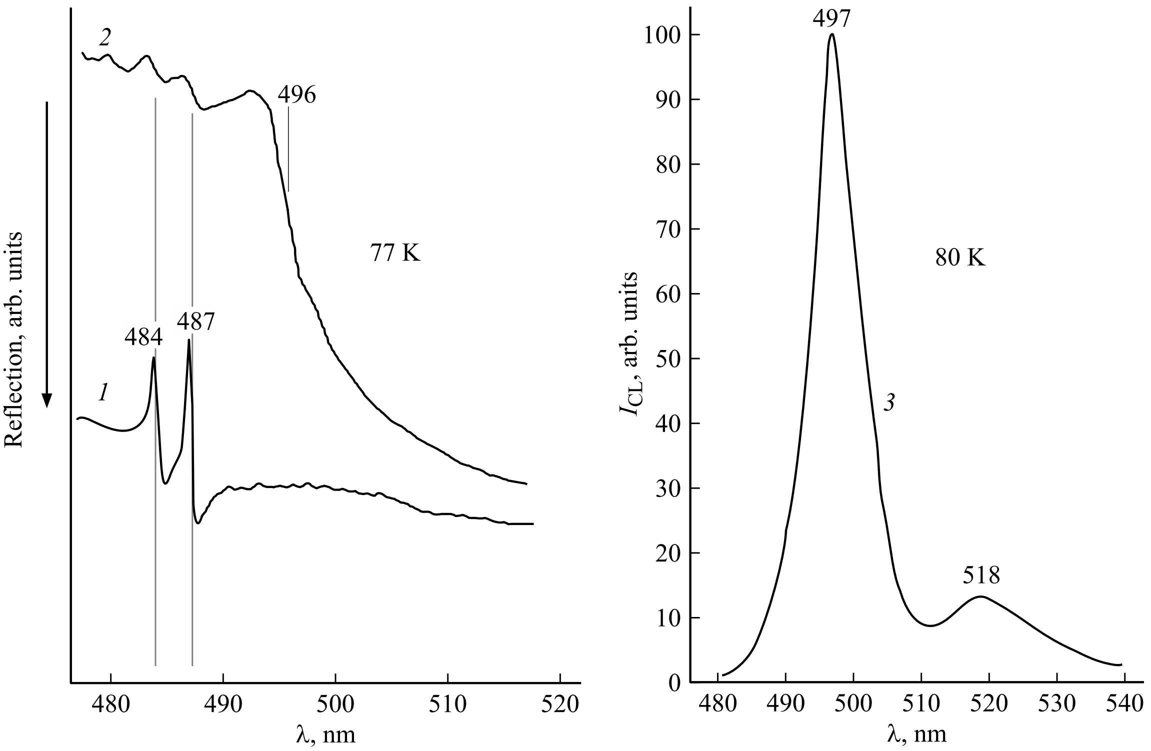

Figure 4. Microphotograms of the reflection spectra of the CdS(O) crystals with the total oxygen content [O] approximately at 10^{19} cm^{-3} (the curve *1*) and 10^{20} cm^{-3} (the curve *2*), and the CL spectrum of the crystal № 18 from the region of oxygen clusters (the curve *3*).

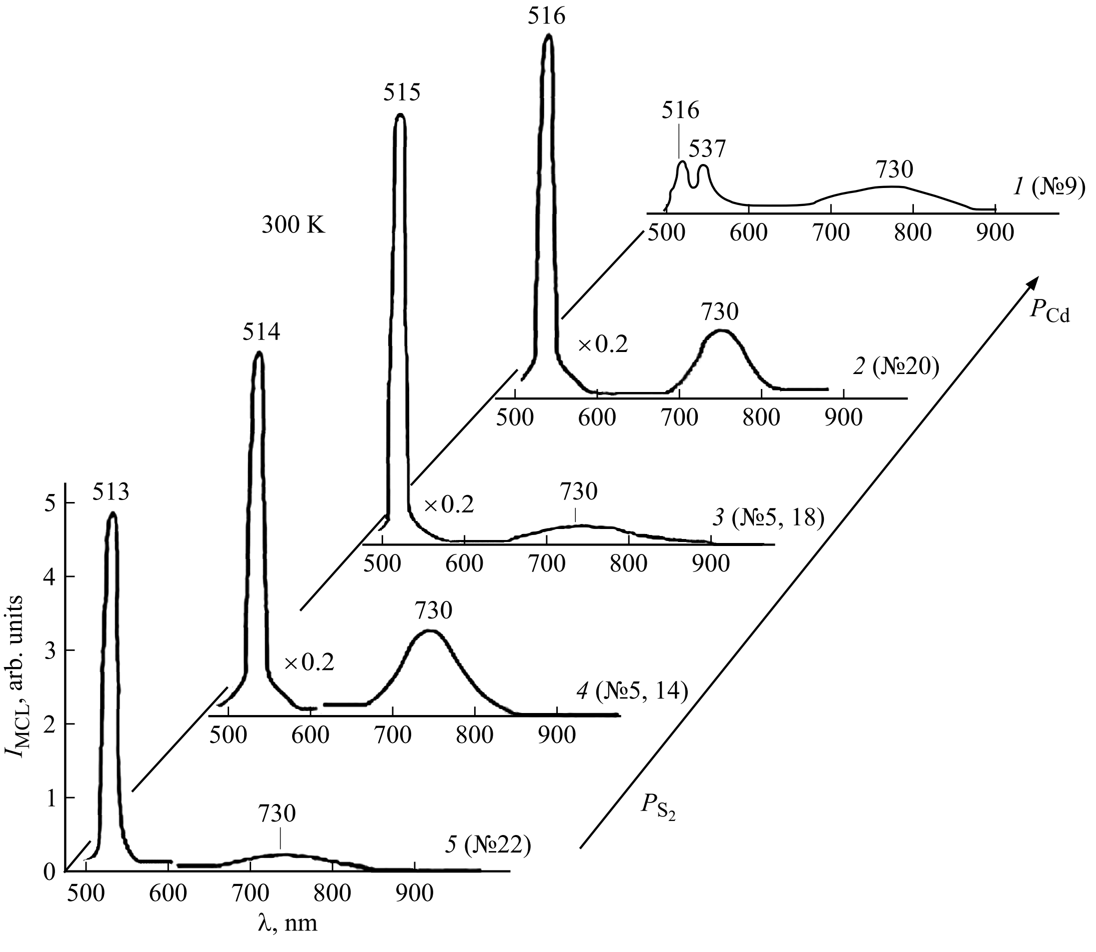

Figure 5. MCL spectra are shot in SEM at 300 K, the beam energy of 25 keV and the excitation density of 10^{24} cm^{-3} · s^{-1}.

wave component (LW) — 518 (520) nm. Thus, the exciton band at 496–497 nm indirectly indicates the band gap at the value of limiting doping of CdS with oxygen in volume and on stacking faults.

4. Micro-cathodoluminescence

In order to exclude the influence of structure heterogeneities in studying the dependence of the optical properties of the CdS crystals on the composition, the micro-cathodoluminescence spectra (MCL) have been studied in the scanning electron microscope (SEM) at 300 K and at higher excitation intensity. The MCL spectra have been shot from separate single blocks at the magnification of ×10000 and a grid size of $10 \times 10\,\mu$m (the beam diameter 1000 Å).

The spectra are shown of Fig. 5 selectively for the crystals grown within the CdS homogeneity (Fig. 1, *b*). As it is clear, with increase in the shooting temperature to 300 K, the spectra have evident bands of the green edge emission at the same crystals and at the same wavelengths of 513, 514, 515, 516 nm.

Fig. 5 shows the results corresponding to shooting of a central part of the single blocks[4]. Generally, it reveals the trend of high-energy shift of the band maximum with the crystal composition — increase in P_{S2} during the growth. In all the samples, the shift range is from 518 to 511 nm.

5. Model of the green edge emission

As it was shown in the paper [19] for the crystals ZnS · O with stacking faults, the bands within the region of the edge emission are exciton components of the bounded (localized) exciton spectra (BE_{SF}) on the SA-centers of luminescence in the layers of the stacking faults.

The features of „CdS green edge emission“ at the temperature of ∼ 20 K, corresponding to our results are described in the fundamental study [1]. Their nature was explained by applying a recently established model of donor-acceptor (D-A) pairs. According to this model, an acceptor could be a lattice defect, while various types of impurity donors determined the edge emission bands. The various bands were regarded as a set of D-A pairs. This trend, being a key one, is still the same up to now [4,5]. In doing so, it is not taken into account that only several CdS edge emission features are similar to those of the D-A pairs, but the others clearly contradict them [1]. The crystal oxygen has not been taken into account, but it is typical that the CdS edge emission is observed in the samples with excessive cadmium [1].

In accordance with our accepted analysis procedure based on the BAC theory [9–12], the multi-band model CdS · O with SF was built for the spectrum of the green edge emission. The model is shown on Fig. 6, *b* for CdS · O with SF.

Comparison of design [O_S] at $G = 80$ meV and data of the GCA analyses [O] in the studied CdS crystals

№ of the crystal	Band BE_{SF}, nm (eV)	[O_S], cm^{-3} calc. as per Δ	[O], cm^{-3} GCA data
9,13,8,17,19	516 (2.413)	$1.8 \cdot 10^{20}$	$1.2 \cdot 10^{20}$
18	515 (2.407)	$1.7 \cdot 10^{20}$	$1.2 \cdot 10^{20}$
14	514 (2.412)	$1.6 \cdot 10^{20}$	$1 \cdot 10^{20}$
5	513 (2.417)	$1.49 \cdot 10^{20}$	$8 \cdot 10^{19}$
20	512 (2.422)	$7 \cdot 10^{19}$	$3 \cdot 10^{19}$

The left part of the diagram (*a*) corresponds to the CdS · O clean crystals with [O] of approximately 0.1 mol% [11]. As per Fig. 1, *a*, for these compositions, the concentration of the $Cd_i^\bullet$ defects is decreasing almost to zero and, therefore, it is impossible to form the SA-centers $\{O_S^* Cd_i^\bullet V_{Cd}^{//}\}^/$ and stacking faults [18,19].

The right part of the diagram corresponds to the CdS · O samples with SF. The light layers by the position of the exciton band of 489 nm (2.535 eV) at 80 K or E_{ex}^{cc} correspond to the above-described CL spectra with [O_S] $\sim 2 \cdot 10^{19}$ cm^{-3}.

As it is already noted, the bands of the edge emission CdS · O with stacking faults are caused by the BE_{SF} excitons on the SA-centers in the layers of the stacking faults [18,19]. For separate bands of the edge emission, the levels of the (E_-^{BE}) bounded exciton are built on Fig. 6, *b* in accordance with a general model of radiat exciton transitions at the bond energy of 80 meV. For averaging for all closely located E_-^{BE} levels, we designate a bottom of the subband of the stacking fault as *E*-(SF).

The model of Fig. 6 determines the magnitudes $\Delta = E_-^{cc} - E_-^{BE}$. These data are tabularized. The results well agree with CL(M2)-spectra measured distances between the exciton bands of the light layers and the BE_{SF} bands. In the spectra of our studied crystals, these values are approximately 120–140 meV.

The table also contains the oxygen concentration [O_S], which is calculated by the value Δ and corresponds to a spectral position of a specific band of the bounded exciton BE_{SF}, as well as the GCA analyses [O].

The calculated oxygen concentrations by the value Δ for the group of our studied crystals with the edge emission within the range 513–518 nm are $(1.5{-}1.8) \cdot 10^{20}$ cm^{-3}. These data [O_S] are symbate to the results of the GCA analysis, but they exceed the latter because if there are stacking faults, the GCA analysis provides for the oxygen content per the unit crystal volume, i.e. it averages oxygen in the stacking faults and the clean layers.

The oxygen concentrations, which are calculated by the spectra of the green edge emission CdS · O, reveal a relatively narrow range of the crystal compositions, which corresponds to a region with the maximum intensity

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[4] Band of 537 nm due to CdO exciton emission in the heterogeneous crystals, and of 730 nm — SA(H) component of the CdS self-activated radiation are not described here [11,16,18].

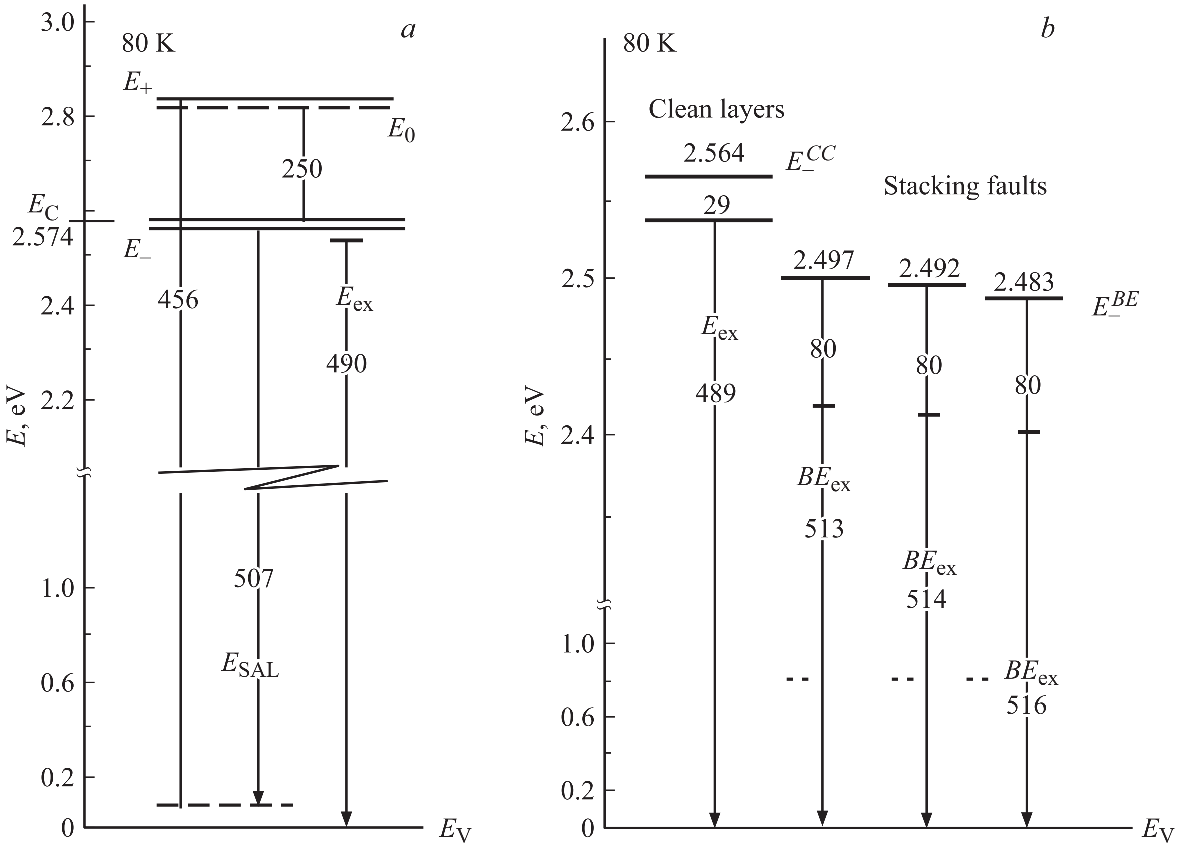

Figure 6. Band models of the CdS · O crystals: a clean one without SF (*a*), a multi-band model for the crystal with SF (*b*). The bond energy G of the bounded exciton for the hexagonal CdS · O is assumed to be 80 meV in accordance with that obtained in [17] for wurtzite. Dashes are used to outline deep levels of the recombination E_{SA}, of the self-activated SA-radiation, as at 80 K (Fig. 2).

of the SA-emission and the limit solubility of oxygen in CdS (a region of the A point on the diagram of Fig. 1, *a*). For this region of the compositions, the oxygen is full compensated with the intrinsic point defects of the lattice [20,21].

As follows from the analysis of the above-described CL spectra, the system of the levels of the localized excitons on the SF does not change along with the temperature. The BE_{SF} bands were observed at the same wavelengths both at 80 and at 300 K.

With change of the concentration $[O_S]$ in certain limits, the crystal still has the same spectrum, which is typical for SF. It includes the bands of the light layers with an equilibrium concentrations of dissolved oxygen, which accompany the bands of the edge emission BE_{SF} in the SF layers. At the same time, the exciton band of the light layers E^{cc}_{ex} was observed at the low measurement temperatures, when the band gap of the base is higher and the level of the base E^{cc}_- is higher than E^{BE}_- in the layers of the stacking faults. At 300 K, the relationship is changing and the radiation can be corresponded by separate bands of the edge emission BE_{SF}.

The similar situation was observed in the MCL spectra of the CdS · O crystals, which are saturated with oxygen to the limit and alloyed with ions [18,20][5] During their annealing, with decrease in the defectiveness, the bands of the exciton range were evidently substantially amplified. The oxygen extracting and the change of the layer composition during the annealing were accompanied by shifting the exciton band of the base E^{cc}_{ex}. In doing so, the exciton band of the base was evident only at 100 K, for example, in 495−494 nm. At 300 K, instead of it, the exciton region of the same samples is predominated by the bands BE_{SF} of the edge emission, for example, 513 → 512 nm. The spectrum position of these bands BE_{SF} and the base exciton E^{cc}_{ex} allows determining $[O_S]$ as per the table, on the SF and in the base, respectively. Good agreement of the experimental and calculated results confirms a capability of the presented model.

During annealing in transfer to excessive sulfur, the further change of the crystal composition had shown that already for the position of the band BE_{SF} 512 nm the oxygen concentration $[O_S]$ is leveled to $\leq 10^{19}\,\mathrm{cm}^{-3}$ in the whole crystal volume without SF formation.

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[5] Ion implantation to $\sim 4 \cdot 10^{20}\,\mathrm{cm}^{-3}$ resulted in shifting of the exciton MCL band of the base to 524−525 nm at 100 K.

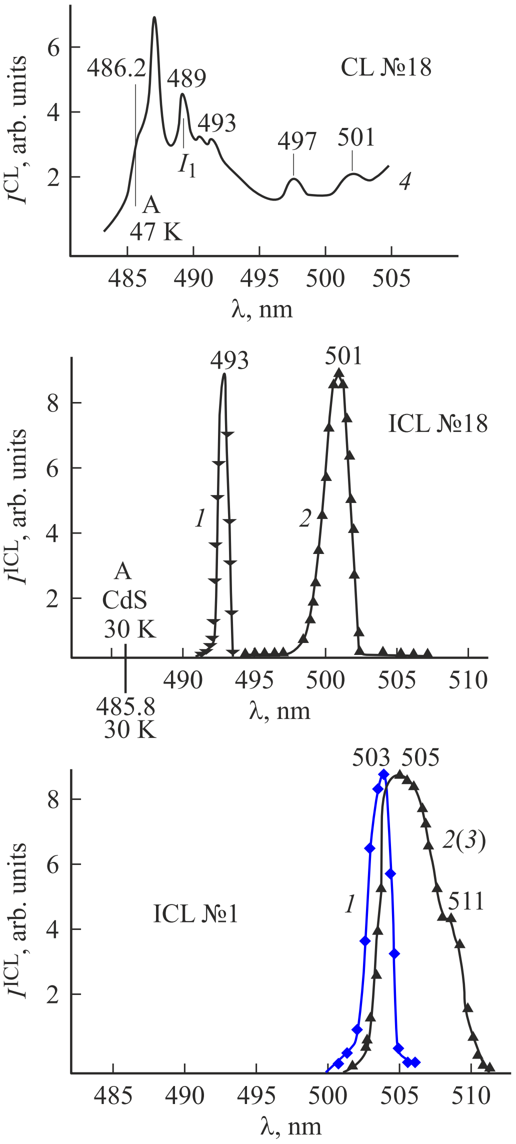

Figure 7. CdS(O) luminescence spectra were measured at 47 K — CL and at 30 K — ICL. The excitation density was: CL — $\sim 10^{21}\,\mathrm{cm}^{-3}\cdot\mathrm{c}^{-1}$ (*4*); ICL — $1.56\cdot 10^{26}$ (*1*); $6.52\cdot 10^{26}$ (*2*) and $7\cdot 10^{26}\,\mathrm{cm}^{-3}\cdot\mathrm{c}^{-1}$ (*3*).

6. Pulse cathodoluminescence

Let us consider the change of the CL spectrum of the crystals when being excited with a high-current electron beam, i.e. the pulse-impulse cathodoluminescence (ICL). The ICL measurements were carried out with the electron beam energy $0.1-0.02\,\mathrm{J/cm}^2$, corresponding to the excitation levels $(10^{26}-10^{27})\,\mathrm{cm}^{-3}\cdot\mathrm{s}^{-1}$, using a unit of the National Research Tomsk Polytechnical University. The depth of the electron beam penetration to the sample bulk was $125-200\,\mu$m, the beam diameter was $5-10$ mm [20,22].

Fig. 7 shows the ICL spectra of the crystal № 18 (the curves $1\rightarrow 2$) of the stoichiometric composition with the oxygen content $[\mathrm{O}] = 5\cdot 10^{19}\,\mathrm{cm}^{-3}$. The ICL spectra are given in comparison with the CL spectrum of spontaneous (exciton) radiation of the same crystal № 18 (the curve *4*), in order to establish a relation therebetween. Let us consider the exciton band of 489 nm, which is described above as $E_{\mathrm{ex}}^{\mathrm{cc}}$ of clean layers. It is well known in the literature as I_1 [16–18]. When the excitation level is increased by the electron beam, the ICL spectra have evident retuning of the spontaneous radiation with increasing the intensity and the half-width of this exciton band.

At the excitation frequency of $\leq 1.56\cdot 10^{26}\,\mathrm{cm}^{-3}\cdot\mathrm{s}^{-1}$, the ICL spectra of the sample № 18 have a narrow band of stimulated radiation of 493 nm (Fig. 7, the curve *1*) within the region of the first phonon repetition of the light layers' exciton band of 489 nm $E_{\mathrm{ex}}^{\mathrm{cc}}$. The lasing mode is achieved. In accordance with [22], the lasing (lasing radiation) at the band of 493 nm is observed at free excitons interacting with the optical phonons.

With increase in the excitation level of the crystal № 18 to $6.52\cdot 10^{26}\,\mathrm{cm}^{-3}\cdot\mathrm{c}^{-1}$, the band of 493 nm is amplified, widened and shifted to the long-wave region to the position of 501 nm, which is close to the second phonon repetition $E_{\mathrm{ex}}^{\mathrm{cc}}$ (Fig. 7, the curve *2*).

In order to establish a relation of the ICL bands with the concentration $[\mathrm{O_S}]$, Fig. 7 shows the spectrum of the CdS crystal № 1 of the stoichiometric composition, which is grown out of the melt, when the oxygen could be introduced in the higher concentration $(1.2\cdot 10^{20}\,\mathrm{cm}^{-3})$ in comparison with the above-discussed crystal № 18.

As it is clear from Fig. 7, at the same excitation density of $1.56\cdot 10^{26}\,\mathrm{cm}^{-3}\cdot\mathrm{s}^{-1}$ the crystal № 1 had evident lasing in the same way as for the sample № 18. However, the stimulated radiation corresponds to — 503 nm — the first phonon repetition A-LO of another exciton band of 497 nm. As we note above, the spectrum position of the band of 497 nm reveals the crystal with the limit concentration of dissolved oxygen within the CdS bulk. We must assume that in case of the crystal № 1 the bulk of the strongly alloyed sample is excited[6].

As per the spectral position of the exciton band of 497 nm, the concentration of oxygen in the crystal № 1 coincides with the results of the GCA analysis $[\mathrm{O}] \sim 1.2\cdot 10^{20}\,\mathrm{cm}^{-3}$, which corresponds to the oxygen alloying level of the stacking faults.

When increasing the excitation of the crystal № 1 to $6.52\cdot 10^{26}\,\mathrm{cm}^{-3}\cdot\mathrm{s}^{-1}$, the band of 503 nm is shifting to 505 nm. The maximum of 505 nm had evident amplification — super-luminescence. It is confirmed by the increased emission intensity and occurrence of directivity [22]. The amplification is at levels of the defects, which are responsible for the spontaneous luminescence band of 497 nm,

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[6] The limit alloying by oxygen means for the theoretically homogeneous crystal the same level E_{C} of the base and the crystal E_{-}^{BE} of the most LW components of the edge emission spectrum. For the real crystals, which are strongly alloyed with oxygen, at the high excitation density, the ICL spectra had the evident bands $\mathrm{BE_{SF}}$, which were the most long-wave ones. Specifically, the spectrum of the crystal № 1 at 15 K — these are the bands 517 and 519 nm [5], and for the crystal № 18 at 80 K — it is the band of 518 nm (Fig. 4).

i. e. for CdS sample № 1 it is determined by radiation of the exciton of the CdS(O) solid solution bulk, which is saturated with oxygen to the limit.

The spectral position, the intensity and the shape of the band of stimulated radiation out of the bulk of the CdS sample № 1 (the curve *3*) are stabilized, but this process is observed at higher excitation levels.

Based on the above-discussed results, it can be expected that the minimum thresholds of lasing and the maximum efficiency of the CdS lasers with the electron pumping at 80−300 K [22] can be achieved on the samples of cadmium sulfide with SF of the composition of the „stoichiometry region", i.e. with a less content of oxygen. In doing so, the clean layers are responsible for the lasing.

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Conflict of interest

The authors declare that they have no conflict of interest.

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