

Recombination of conduction electrons with trapped-hole centres in MgO:Be and MgO:Ca single crystals

MgO:Be және MgO:Ca монокристалдарындағы өткізгіштік электрондардың жинақылған кемтіктермен рекомбинациясы

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Кең саңылаулы ($E_g = 7,8$ эВ) MgO:Be және MgO:Ca монокристалдарында Be^{2+} и Ca^{2+} иондары шапшаң қозғалатын кемтіктер үшін қармағыштар қызметін атқаратыны көрсетілді. Олар MgO кристалының көлемінде автолокалданған күйге өтпейді. MgO:Be және MgO:Ca кристалдарында 10 К температурада кемтіктік орталықтардың электрондармен рекомбинациялануы кеңжолақты люминесценцияның (максимумдары 7,2 және 6,8 эВ) пайда болуына әкеледі. Аталған жолақтар сәйкесінше 190 және 50 К температураларда жылулық сөнеді. Энергиялары 5–10 кэВ электрондармен немесе 25 эВ фотондармен алдын ала сәуленген монокристалды қыздыру барысында термостимульденген люминесценция шыңдары тіркелді. Ол шыңдар кемтіктік орталықтардың термоионизациясына және қозғалмалы кемтіктердің ақауларға жинақылған электрондармен рекомбинациялануына байланысты. $[Be]^+$ және $[Ca]^+$ кемтіктік орталықтар кулондық зарядқа және ыстық электрондармен сәуле шығармай өтетін рекомбинацияланудың тиімді қимасына ие. Осындай ыстық рекомбинацияларды френкелдік жұптың құрылу энергиясы E_g -дан асатын жүйелердегі Френкель ақауларының радиациямен құрылуы механизмдерінің бірі ретінде қарастыруға болады.

Показано, что в широкощелевых ($E_g = 7,8$ эВ) монокристаллах MgO:Be и MgO:Ca ионы Be^{2+} и Ca^{2+} служат ловушками для высокоподвижных дырок, которые в объеме MgO не переходят в автокализованное состояние. Рекомбинация дырочных центров в MgO:Be и MgO:Ca с электронами проводимости при 10 К приводит к появлению широкополосной люминесценции (максимумы при 7,2 и 6,8 эВ), испытывающей тепловое тушение при 190 и 50 К соответственно. При нагреве монокристалла, предварительно облученного электронами 5–10 кэВ или фотонами 25 эВ, зарегистрированы пики термостимулированной люминесценции, связанные с термоионизацией дырочных центров и рекомбинацией подвижных дырок с локализованными на дефектах электронами. Дырочные центры $[Be]^+$ и $[Ca]^+$ имеют кулоновский заряд и большое эффективное сечение безызлучательной рекомбинации с горячими электронами. Такие горячие рекомбинации можно рассматривать как один из механизмов радиационного создания дефектов Френкеля в системах, где энергия создания френкелевой пары превышает E_g .

MgO is the simplest representative of the wide band gap oxide materials ($E_g = 7.8$ eV) and is often regarded as their model system. Furthermore, it itself has found various practical applications as an isolator material, irradiation resistant construction material, laser material, and even as a luminescent material in the UV spectral region (5–7 eV).

Similarly to the majority of alkali halide crystals (AHC), magnesium oxide has a cubic face-centred Bravais lattice, where O^{2-} anions form a close-packed structure. Anions and cations are located at octahedral sites, the ionic radius for 6-coordinated O^{2-} and Mg^{2+} is 1.26 Å and 0.86 Å, respectively. In spite of the similar crystal lattice, the properties of electronic excitations in MgO and AHC are different. In all AHC, at sufficiently low temperatures a hole after vibronic relaxation inside a valence band undergoes transformation into the self-trapped state in the form of an immobile V_K centre, the structure of which was determined via the EPR method — a two-halide quasi-molecule X_2^- located at two anion sites along $\langle 110 \rangle$ direction [1,2]. Because of a strong electron-phonon interaction, the self-trapping of excitons also takes place in regular lattice regions of AHC at low temperatures (see, e.g., [3, 4]). In AHC, the maxima of broad luminescence bands related to self-trapped excitons are located at 2.2–6 eV. On the other hand, only the manifestations of large-radius free excitons and highly mobile conduction electrons (e) and valence holes (h) have been detected in the reflection and luminescence spectra of highly pure MgO single crystals at low temperatures. In our opinion, this fundamental difference in the behaviour of the holes in the regular regions of AHC and MgO can also influence the processes of hole trapping by different impurity centres.

The optical properties of MgO depend strongly on the presence of various defects. The prevailing defects in nominally pure MgO crystals not subjected to an irradiation with heavy particles, are the defects in-

troduced during the crystal growth: mainly cation impurities, both bi- (Ca^{2+} , Mn^{2+}) and trivalent (Fe^{3+} , Cr^{3+} , Al^{3+}), and, as a consequence, the cation vacancies (v_c) to neutralize the excess charge of the trivalent impurities. If one subjects the MgO crystals to an ionizing radiation (VUV-radiation, X-rays), free holes are created and then become localized near the cation vacancies (the so-called V-centres, see, e.g., [5, 6]). The free electrons are trapped mainly by the impurity cations. In the accordingly doped crystals, the holes can be localized also near the monovalent (e.g., Li^+) or bivalent (Be^{2+}) impurity cations. If the resulting trapped-hole centre does not contain the cation vacancy, a different notation is used: [impurity atom]^{effective center charge}, e.g., $[\text{Li}]^0$, $[\text{Be}]^+$ are, respectively, a hole localized near a Li^+ and a Be^{2+} ion. For MgO, such notations are widely accepted since [5]. The main constituent of the hole centres is the O^- , the hole localized on a regular anion (see., e.g., [5–8]).

Doping of MgO single crystals with Be results in the formation of a number of new Be-containing centres. In the context of this paper, one of them is relevant (see also [7, 8]): the $[\text{Be}]^+$ centre ($\text{Be}^{2+}-\text{O}^-$, i.e. a hole trapped at an oxygen ion nearby an impurity Be^{2+} ion). The $[\text{Be}]^+$ centre is created by X-irradiation at 77 K, its EPR spectrum is best observed at 4 K and at high microwave powers. The $[\text{Be}]^+$ centre has a tetragonal symmetry with a slight orthorhombic distortion. This distortion is caused by the off-centre position of the small Be^{2+} ion (the ionic radius equals 0.41 Å and 0.3 Å for 4- and 3-coordination, respectively) in an Mg^{2+} cation site.

The low-temperature irradiation of the MgO single crystals containing purposely introduced impurity hole traps with photons of $h\nu \geq E_g = 7.8$ eV, X-rays or an electron beam causes the formation of electron-hole (e-h) pairs. Highly mobile valence holes undergo rapid localization near dominant hole trapping centres, while conduction electrons lose their energy excess down to the bottom of the band via fast vibronic relaxation. Thereupon totally relaxed electrons mainly recombined with the trapped holes. Figure 1 demonstrates the corresponding wide luminescence bands detected via a double VUV monochromator at the excitation of doped MgO single crystals by 10-keV electrons at 10 K. The same luminescence bands have been detected under crystal excitation by synchrotron radiation of 9–30 eV at 6–10 K. The wide emission band peaked at the highest energy of ~6.8 eV is related to the electron recombination luminescence in MgO:Ca. This emission is not detected in pure MgO and, therefore, could not be ascribed to the luminescence of self-trapped excitons. The maximum of the recombination luminescence band in MgO: Be is located at a lower energy (~6.2 eV [9]). The intensity of both recombination emissions drastically decreases (more than by three orders of magnitude) in ~1 s after an electron irradiation is stopped. The continuously weak phosphorescence is typical of the tunnel recombination between spatially separated but trapped electrons and holes.

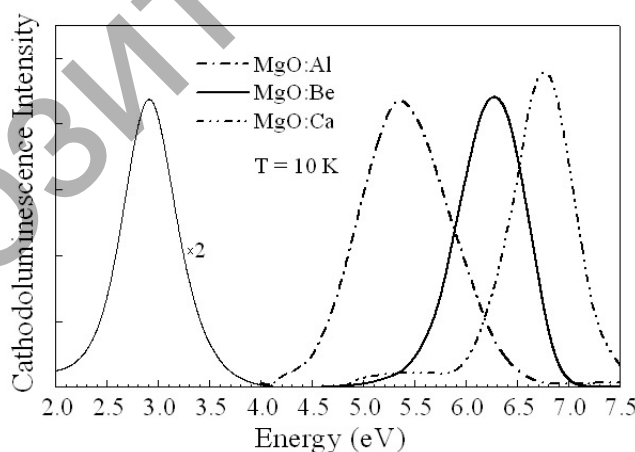


Fig. 1. Emission spectra of MgO: Be (solid line), MgO: Al (dashed-dotted line) and MgO: Ca single crystals (dashed-double-dotted line) under the excitation by 10-keV electrons at 10 K

The heating of an X-irradiated MgO: Be crystal with a constant rate of $\beta = 10$ K/min from 10 K to $T \geq 190$ K causes the destruction of $[\text{Be}]^+$ centres via hole release and restoration of Be^{2+} at cation sites. The released holes recombine with the electrons still localized at different defect/impurity centres, for instance with the appearance of the 2.9 eV emission, which is especially intensive in plastically deformed samples and is tentatively connected with the presence of bivacancies. Some holes are also localized at deeper traps, for instance, VAl centres in MgO:Al^{3+} are stable up to 375 K [10].

Figure 2 shows the thermoactivation characteristics of MgO:Be²⁺ (150 ppm) single crystals. The pulse annealing of the EPR signal of [Be]⁺ centres was measured in the sample X-irradiated at 10 K. The curve of thermally stimulated luminescence ($\beta = 10$ K/min) was measured for 2.9 eV emission in a MgO:Be²⁺ crystal irradiated for 1 hour by 5 keV electrons at 5.2 K. The main TSL peak at ~ 190 K accompanies a sharp decrease of the EPR signal intensity. The thermal quenching of the 6.2 eV emission (under excitation by 25 eV photons), connected with the release of electrons from some traps and their recombination with the holes from [Be]⁺ centers, takes place in the same temperature region (see Fig. 2). So, there is no doubt that we have a hole process at about 190 K.

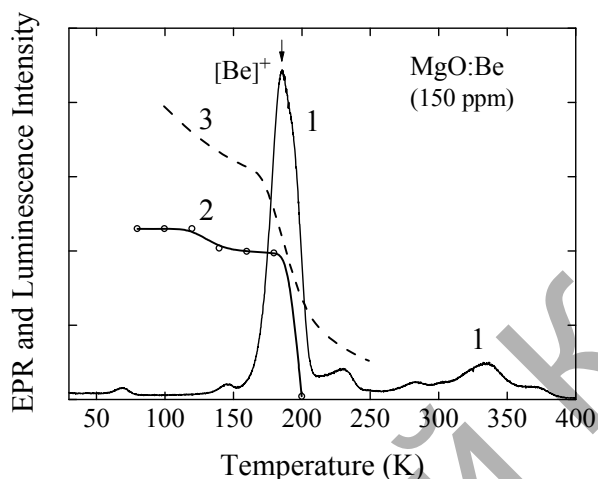


Fig. 2. Thermoactivation characteristics of MgO:Be²⁺ (150 ppm) single crystals. TSL of 2.9 eV (curve 1, $\beta = 10$ K/min) for MgO:Be²⁺ (150 ppm) irradiated for 1 hour by 5 keV electrons at 5.2 K. The annealing of the EPR signal of [Be]⁺-centers in the sample X-irradiated at 10 K (curve 2) and the temperature dependence of 6.2 eV emission under excitation by 25 eV photons (curve 3)

The problem of the hole self-trapping in MgO has long been under consideration. In [11] the 6.9 eV luminescence band in MgO single crystals was interpreted as a luminescence of self-trapped or at least relaxed excitons. On the other hand, the theoretical calculations have shown that there can not occur any self-trapping of holes in a bulk MgO crystal, while the process is possible at low-coordinated sites (corners, kinks etc.) at MgO (100) surface [12, 13]. This conclusion is also consistent with the experimental results [14] that showed a high hole mobility in the bulk MgO.

In MgO, Ca²⁺ impurity ions replace regular cations and form substitutional solid solutions with the concentration of calcium ions up to several at%. The ionic radius of Ca²⁺ surrounded by six O²⁻ is larger than that for Mg²⁺ (1.14 Å and 0.86 Å, respectively). So, the doping of MgO with Ca²⁺ causes the expansion of the crystal lattice. It is obvious that the Ca²⁺ situated at cation lattice sites cannot serve as traps for conduction electrons because the ionisation energy E_{ion} of a free Ca⁺ is about 3 eV lower than of Mg⁺. On the other hand, the value of E_i for Be⁺ is by ~ 3 eV higher than that for Mg⁺ and, in principle, electrons can be trapped at Be²⁺. However, the effective cross-section of electron trapping by Be²⁺ is at least by dozens of times lower than the effective cross-section of the recombination for the complex Coulomb centres formed at a hole trapping nearby Be²⁺ ions.

In [15], a 6.8 eV luminescence peak was reported in Ca-doped MgO crystals and assigned presumably to [Ca]⁺ centre (Ca²⁺ substituting Mg²⁺ next to the hole trapped on an oxygen ion). To verify this conclusion which is in contradiction with the assignment of the ~ 6.9 eV emission in nominally pure MgO to self-trapped excitons [11], additional investigations of MgO:Ca (~ 200 ppm) were performed in our laboratory. The crystal was firstly grown from highly pure starting materials by a variation of arc fusion technique and thereafter the second growth was performed using the purest part of the crystal grown in the first stage. Such procedure allowed to reduce additionally the concentration of uncontrolled impurities in MgO:Ca. According to [16], a quasiline emission of free excitons at 7.69–7.70 eV dominates in the photoluminescence spectrum of highly pure MgO, while the emission band peaked at 6.8 eV is absent. On the other hand, quasiline emission at 7.65 eV (i.e. shifted toward low-energy region with respect to the emission of free excitons) and intense broadband emission peaked at 6.8 eV were detected in MgO:Ca crystals. The intensity of broadband emis-

sion decreases parallel to the decrease of the concentration of calcium impurity ions. The 6.8 eV luminescence is efficiently excited by synchrotron radiation of 7–40 eV, i.e. both below the edge of fundamental absorption and at the formation of free excitons or separated electrons and holes (band-to-band transitions) [17]. The efficiency of 6.8 eV emission is especially high at 25–30 eV, where the values of absorption constant are relatively low thus decreasing the outcome of conduction electrons and valence holes at the surface. In addition, the multiplication of e-h pairs, when one exciting photon forms two or even more e-h pairs occurs if the photon energy exceeds ~24 eV [17, 18].

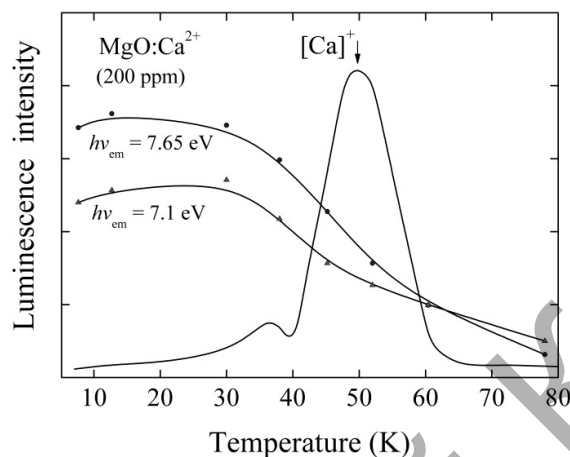


Fig. 3. The temperature dependences of the steady luminescence intensity measured for quasiline emission at 7.65 eV (filled circles) and broadband emission at 7.1 eV (triangles) at the excitation of MgO:Ca by synchrotron radiation of 25 eV and the TSL curve (solid line) measured after irradiation of MgO:Ca by X-rays at 6 K ($\beta = 10$ K/min).

Figure 3 demonstrates the temperature dependences of the luminescence intensity measured for quasiline emission at 7.65 eV and broadband emission with the maximum at 6.8 eV (bandwidth of ~0.8 eV, measurement was performed at 7.1 eV) at the steady excitation of MgO:Ca by synchrotron radiation of 25 eV, which selectively forms separated electrons and holes. The absorption constant at 25 eV is significantly lower than those at the beginning of band-to-band transitions and the 25-eV photons excite crystal regions located rather far from the surface. The thermal quenching of the emissions starts above ~40 K and the intensities are decreased by half at about 50 K. To elucidate the processes responsible for the thermal quenching, the thermally stimulated luminescence (TSL) has been measured after irradiation of MgO:Ca by X-rays (50 kV, penetration depths exceeds the thickness of our sample) at 6 K. The TSL was measured for 2.9 eV emission with the heating rate of $\beta = 10$ K/min. The 48 K TSL peak is dominant for 2.9 eV emission, while a weak TSL peak at ~36 K is most noticeable for ultraviolet emission. The TSL has been detected on the background of a weak temperature-independent (8–80 K) tunnel phosphorescence with a complex spectral composition. A similar TSL curve ($\beta = 10$ K/min) has been detected in MgO:Ca previously irradiated by 6-keV electrons at 6 K. The analysis of the dependences of steady photoluminescence on temperature and the TSL curves testify that $[\text{Ca}]^+$ centres are formed after irradiation of MgO:Ca resulting on the hole trapping by Ca^{2+} impurity ions located at regular cation sites. The thermal ionisation of $[\text{Ca}]^+$ centres takes place at about 50 K. The hole delocalisation causes a sharp attenuation of the 6.8 eV emission because the holes released from $[\text{Ca}]^+$ centres migrate to still localized electrons. The latter recombination process manifests itself in the TSL peak at ~48 K in previously irradiated MgO:Ca: thermally released holes recombine with the localized electrons via 2.9 eV emission.

Hole trapping in MgO single crystals can happen only if there exists an initial distortion or irregularity in the crystal. It should be noted that the $[\text{Be}]^+$ hole centre does not have any obvious coulombic precursor for the hole trap. However, due to the small ionic radius, the Be^{2+} that substitutes for regular cation Ca^{2+} is hopping inside the cation vacancy site polarizing the surrounding oxygen ions. When MgO:Be is subjected to irradiation, such polarizations act as shallow traps for the holes. At some point, when a hole localizes at the O^{2-} neighbouring the Be^{2+} , the latter relaxes away from the former, hence, the potential well is deepened so that its depth is sufficient for localizing the hole and formation of Be^{2+} trapped-hole centre.

In view of this, it could be very instructive to investigate hole trapping in MgO:Ca crystals. Ca is also isovalent with Mg, but its ionic radius $r_{\text{Ca}} = 1.14 \text{ \AA}$ is much bigger than that of magnesium. Such a big ion will produce local distortions at the cation vacancy site, thus, possibly allowing a hole to be trapped on the neighboring O^{2-} , forming a $[\text{Ca}]^+$ centre. According to our experimental data, a 48 K TSL peak is related to the thermal destruction of $[\text{Ca}]^+$ centre and the 6.8 eV luminescence peak is assigned to it (see Figs. 1 and 3). However, the decisive experiment would be the EPR detection of the $[\text{Ca}]^+$ centre but it is technically complicated because the MgO:Ca crystal must be irradiated and kept before the measurements at $T < 30 \text{ K}$.

It is of interest to compare the processes of a hole localization nearby impurity Be^{2+} and Ca^{2+} ions in MgO and nearby Na^+ or Rb^+ impurity ions (the ionic radius is smaller or bigger than that of K^+ , respectively) in KCl. The so-called V_{KA} centres (a self-trapped hole near an impurity cation, see, e.g., [2]) are efficiently formed in KCl:Na, while such centres are not detected in a KCl:Rb crystal. According to luminescent studies, the hole localization near a Ca^{2+} that substitute for a Mg^{2+} with a smaller ionic radius in a regular cation site takes place in MgO:Ca. Conceivably the effect of a lattice polarization by a hole in the region of an impurity ion in metal oxides is significantly stronger than that in AHC doped with isovalent alkali metals. As a result the localization of a hole (or an exciton) in MgO occurs even near an impurity which ionic radius is bigger than that of a regular cation. Since Ca^{2+} do not serve as the electron traps, the recombination of mobile conduction electrons, formed at MgO:Ca irradiation by photons of $h\nu > E_{\text{g}}$, with the trapped holes causes the intense wideband luminescence peaked at 6.8 eV (see also figure. 3).

The cross-section for the recombination of electrons with charged (coulombic) $[\text{Be}]^+$ and $[\text{Ca}]^+$ centres is at least two orders of magnitude as high as that for neutral trapped-hole centres. In MgO, the energy released at the recombination of a cold electron (relaxed down to the bottom of the conduction band) with $[\text{Be}]^+$ and $[\text{Ca}]^+$ is not sufficient for the creation of a pair of Frenkel defects, i.e. the formation energy of a Frenkel pair exceeds the energy gap ($E_{\text{FH}} > E_{\text{g}}$). However, non-relaxed (hot) conduction electrons participate in the recombination with $[\text{Be}]^+$ and $[\text{Ca}]^+$ as well providing, in principal, the formation of Frenkel defects. Such hot recombination are in competition with the multiplication process of e-h pairs, when a sufficiently hot conduction electron (or a hot valence hole) is able to create a secondary e-h pair (see, e.g., [18]). In MgO, the width of the valence band is about 6 eV [19] and hot valence holes do not cause the multiplication of e-h pairs. According to detailed experimental investigations, the efficient creation of secondary e-h pairs occurs if the energy of exciting photons is $h\nu \approx 25\text{--}30 \text{ eV}$. So, the energy of non-relaxed conduction electrons in the crystal bulk, which can be involved in hot recombination instead of the multiplication process, is limited by about 11–16 eV. The energy released at the recombination of such electrons with $[\text{Be}]^+$ and $[\text{Ca}]^+$ centres ranges up to 19–24 eV, i.e. is basically sufficient for the non-impact creation of Frenkel pairs nearby $[\text{Be}]^+$ or $[\text{Ca}]^+$. Behaviour of oxygen interstitials in MgO was theoretically calculated in [20] and the experimental manifestations of these interstitials were detected in X-irradiated MgO crystals. Oxygen interstitials, stable up to 700 K [21], can be formed, for instance, with the participation of hot electrons and $[\text{Be}]^+$, $[\text{Ca}]^+$ or other trapped-hole centres. It is worth noting that the favourable conditions for the defect creation via hot e-h recombination are formed at the irradiation of MgO with swift heavy ions due to the extremely high density of electronic excitations (e-h pairs) within ion tracks. The further investigation of such processes lies ahead.

Acknowledgements

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УДК 537.35

Спектрально-люминесцентные характеристики твердых растворов сульфатов калия с органическими красителями

Spectral-luminescent features hard solution sulphate potassium with organic dye staff

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Кристалдан бояғыштардың мөлшері ертіндідегіден үш есе аз екені көрсетілді. Жұтылу және люминесценция спектрлерін өлшеу үшін жасалған автоматты құрылғының блок-сұлбасы мен жұмыс ұстанымы келтірілген. Алынған кристалдардың оптикалық сипаттамалары зерттелді. Жұту және люминесценция спектрлері су ертілеріндегі спектрлерге ұқсас екендігі көрсетілді.

Potassium sulfate crystals doped by organic dye stuffs are researched in the article. It is shown the dye contents in the crystals is less than one in the solution by three orders of magnitude. Optic characteristics of the obtained crystals are investigated. It also shown, that absorption and luminescence spectrums are analogous to ones of their water solutions. The flow block and operation principle of the automatic plant for measurement of the absorption and luminescence spectrums are cited.

Введение

В последние годы увеличился интерес к таким твердым растворам, в которых при синтезе или выращивании кристаллов используются неорганические компоненты в сочетании с органическими. Это связано, прежде всего, с потребностями оптоэлектроники и лазерной техники. В настоящей работе мы рассмотрим такие твердые растворы, используя идеологию работ [1–7].