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THE EFFECT OF ENHANCING THE RECOMBINATION ASSEMBLY OF EXCITON-LIKE LUMINESCENCE IN KCl-Na CRYSTALS WITH A DECREASE IN LATTICE SYMMETRY

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Abstract: The radiative relaxation of exciton-like creations in KCl-Na single crystal was studied by experimental methods of luminescent spectroscopy. In the X-ray luminescence spectra of KCl-Na crystals, the emission band with a maximum at 2.8 eV has been detected for the first time at room temperature.

The effect of multiple amplification of the emission intensity with a maximum at 2.8 eV in KCl-Na crystals is maximum after hardening (400-700^{0}C), a rise in sodium impurity concentration (for example, 10÷1000 ppm), and when exposed to uniaxial elastic deformation along crystallographic directions (<100 >, <110>).

At high concentrations of sodium (1000 ppm) in KCl-Na crystals, intense additional emission has been also detected with a maximum at 3.1 eV, which is typical for paired sodium ions, surrounded by which emissive relaxation of electronic excitations occurs.

It has been experimentally established that lowering symmetry of KCl-Na lattice by thermoelastic stress during hardening, by a light cation (Na) during doping, as well as by elastic compression during uniaxial deformation (<100>, <110>) leads to an increase in the mean free path of the unrelaxed hole before self-trapping. As a result, the probability of recombination assembly of electron-hole pairs increases with the formation of an exciton-like creation, the emissive relaxation of which occurs in the field of thermoelastic, local and uniaxial deformation with intense luminescence.

Keywords: alkali halide crystals, X-ray luminescence, hardening, local and uniaxial deformation, self-trapped exciton, exciton-like luminescence, electron-hole recombination, exciton-like creation.

Introduction. In the process of studying the fundamental regularities of relaxation of electronic excitations (EE) in alkali halide crystals (AHCs), the channels for the creation of primary radiation defects (*F*, -*H*- pairs) and the appearance of intrinsic lattice luminescence originate from the self-trapped state of anionic excitons [1–4]. AHCs are traditionally used as ionizing radiation dosimeters and scintillation detectors [4–6].

A series of original researches have been carried out under direct impact on the channels of emissive relaxation of electronic excitations (EE) in AHC by lowering symmetry of lattice by local deformation due to various sizes of both homologous cations [7,8] and homologous anions [9,10], uniaxial [11-17] and comprehensive deformation [18-23].

This paper presents original experimental results on the ignition of intensity of exciton-like luminescence in KCl matrix during the recombination formation of electron-hole pairs in a sodium field, stimulated by thermoelastic hardening and uniaxial (<100>, <110>) deformation.

Objects of research and technique of experiment. KCl-Na crystals were grown at the Institute of Physics of the University of Tartu, Estonia, using the Stockbarger method in an evacuated quartz ampoule based on zone-purified raw materials with the addition of sodium chloride powders preliminarily dried in vacuum [24-26]. The concentration of sodium ions in KCl-Na samples, taking into account the incorporation coefficient of sodium into KCl lattice was 10 ppm, 100 ppm and 1000 ppm.

Local deformation of the lattice was achieved by purposefully doping KCl with a light sodium cation, the ionic radius (0.98 Å) of which is noticeably smaller than the intrinsic cation of the potassium matrix (1.33 Å).

Hardening of KCl-Na crystals was performed by rapid cooling to room temperature on a quartz substrate of crystals, previously calcined in a quartz ampoule to high temperatures – $400\div700^0$C with exposure for 15 minutes at these temperatures in the «Programix TX 25» electric muffle furnace.

Uniaxial deformation ($\varepsilon = 0.8\div0.9\%$) of crystals was carried out along crystallographic directions <100> and <110> in a special cryostat at low temperatures (85 K) under conditions of high technical vacuum (10^{-6} Torr) [27].

Scanning of the X-ray luminescence spectra (XRL) of the crystals was held in the range of 1.8÷6.0 eV using a high-aperture MSD-2 monochromator and H8259-01 type «Hamamatsu» photomultiplier operating in the photon counting mode controlled by the special SpectraScan program.

For recording the XRL spectra of the crystals, bremsstrahlung from a RUP-120 X-ray machine with a W-anticathode operating in the 3 mA and 100 kV mode was applied as ionizing radiation.

Experimental results. The experiments were conducted in successive impacts of local (Na), thermoelastic and uniaxial deformation on the XRL spectra of KCl crystalline matrix.

The essence of the effect of thermoelastic deformation consists in recording the X-ray luminescence spectra of KCl-Na crystals (50 ppm) depending on the hardening temperature, which are realized by a sharp cooling of the crystals calcined in a quartz ampoule to various high temperatures – 170ºC, 320ºC, 470ºC, 520ºC and 700ºC. The hardening of crystals according to this procedure not only regulates the homogeneity of the sodium impurity distribution, but also simultaneously increases the lattice thermoelasticity, which is an important element for the recombination assembly of exciton-like luminescence in a sodium field.

It should be specially emphasized that all X-ray luminescence spectra of hardened crystals were recorded at room temperature under the same conditions under the action of isodose X-ray emission.

The main indicator as a reference signal is the XRL spectra of unhardened KCl-Na crystal, shown in Figure 1 (curve 1′), which has a maximum at 2.8 eV. In KCl-Na crystals, a weak emission band was registered with a maximum at 2.8 eV, the intensity of which remains unchanged despite a significant increase in the sodium concentration in KCl matrix from 10 to 1000 ppm.

It is known that during long-term operation of scintillation detectors developed on the basis of AHC by doping with cationic impurities (NaI-Tl, CsI-Tl and CsI-Na), their high luminescence quantum yield constantly decreases with time. This effect is associated with the accumulation of impurities and with the transformation of stable aggregates, which reduces the concentration in the lattice of dispersedly distributed single impurity cations, which are centers of luminescence [28]. Because of the creation of aggregates in KCl-Na crystal, the concentrations of single sodium ions located in the cationic lattice site sharply decrease, which is the main reason for the extinguishing of the luminescence intensity of KCl-Na crystal, regardless of the sodium concentration.

To maintain a uniform (homogeneous) distribution of sodium impurity in KCl-Na lattice, a method was developed for calcining samples in a wide temperature range (170°C ÷ 700°C) in order to obtain a stable intense signal characterizing the placement of single sodium ions in the cationic lattice sites.

Figure 1 presents the X-ray luminescence spectra at room temperature of KCl-Na crystals (50 ppm), hardened by calcination to high temperatures (curve 1 - at 400°C, curve 2 - at 520°C, curve 3 - at 620°C, curve 4 - at 700°C) followed by cooling on a quartz substrate in air up to room temperature.

It follows from Figure 1 that the intensity of the luminescence band with a maximum at 2.8 eV begins to grow with an increase in the hardening temperature: at 400°C 44.8 times than before hardening (compare curves 1 and 1′), at 520°C 296.6 times than before hardening (compare curves 2 and 1′), at 620°C by 430.5 times than before hardening (compare curves 4 and 1′), and at 700°C by 511.5 times than before hardening (compare curves 4 and 1′). At high hardening temperatures of KCl-Na crystals (50 ppm), in addition to an intense band with a maximum at 2.8 eV, an emission band appears with a maximum at 3.1 eV (curve 4), as the emission of exciton-like creation in the field of paired sodium ions.

Thus, two intense luminescence bands with maxima at 2.8 eV and 3.1 eV were recorded in hardened KCl-Na crystals (50 ppm) in terms of spectral composition as a result of emissive relaxation of the exciton-like creation in the field of single and paired sodium ions, respectively.

The inset in Figure 1 shows the complete contour of the dependence of the intensity of the luminescence band with a maximum at 2.8 eV on the hardening temperature - *I~f(t)*, constructed on the basis of direct experimental data on the registration of X-ray luminescence spectra (curves 1-4 of Figure 1) of hardened KCl-Na crystals. Points (1-8) correspond to the maximum intensity of the luminescence band at 2.8 eV of hardened KCl-Na crystals (50 ppm).

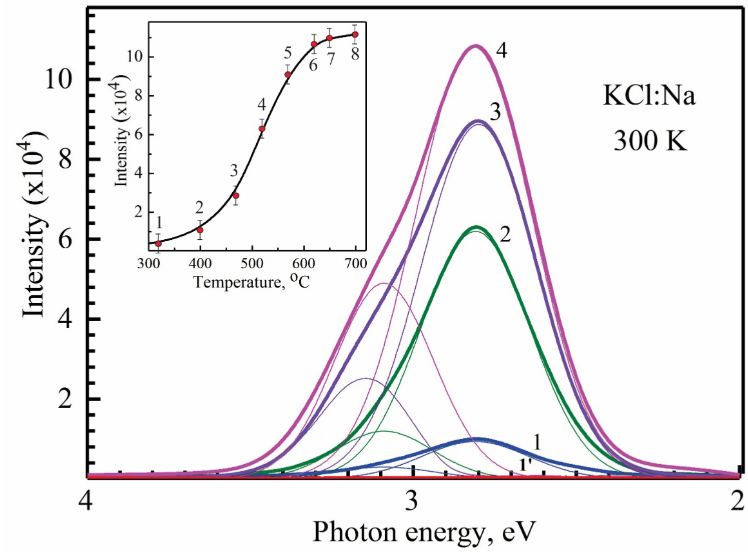

Figure 1 - X-ray luminescence spectra of KCl-Na crystals (50 ppm) measured at room temperature before (curve 1′) and after hardening at 400°C (curve 1), at 520°C (curve 2), at 620°C (curve 3), and at 700°C (curve 4).

Inset: Dependence of the intensity of the luminescence band with a maximum at 2.8 eV on the hardening temperature – *I~f(t)* of KCl-Na crystals (50 ppm) after hardening at 320°C (point 1), at 400°C (point 2), at 470°C (point 3), at 520°C (point 4), at 570°C (point 5), at 620°C (point 6), at 650°C (point 7) and at 700°C (point 8).

It can be seen from the full contour of the dependence *I~f(t)* that, starting from 400°C of hardening (point 2), the luminescence intensity at 2.8 eV noticeably increases, and in the temperature range from 470°C (point 3) to 570°C (point 5) a sharp (almost linear) increase in intensity, after which saturation occurs (point 7, 8). At high hardening temperatures (600÷700°C), apparently, the deformation in the crystal lattice increases due to an increase in the thermoelasticity of the material, and the creation of vacancy defects as a result of the interaction of various types of dislocation is not excluded.

The effect of high-temperature ignition of the intensity of exciton-like formation in the sodium field with a maximum at 2.8 eV in the hardening temperature range of 500-700°C occurs due to an increase in the thermoelasticity of crystals, as a result of which the probability of successive recombination of electrons located in the field of a relaxed hole near sodium increases under the action of X-ray radiation.

In this regard, all samples were preliminarily hardened before experiments to study the concentration dependence of the sodium impurity and the directional effect of uniaxial deformation.

Very weak non-elementary emission bands with maxima at 3.05÷3.2 eV and 2.6÷2.8 eV in the X-ray luminescence spectrum of the zone-purified KCl crystal were registered by us (curve 1 in Figure 2). The latter seem to be associated with uncontrolled defects or impurities. Also the emissive relaxation of anionic excitons localized in the field of anionic vacancies and divacancies is known [29-31], having maxima at 2.7 eV and 3.1÷3.4 eV, respectively. It is possible that traces of homologue cations (Li, Na) remain in the zone-purified KCl crystals, which manifest themselves with a maximum at 2.8 eV during emissive relaxation of excitons in their environment.

From the comparison of curves 1 and 2 in Figure 2, it can be seen that in the XRL spectrum of KCl-Na crystal (10 ppm), an emission band appears with a maximum at 2.8 eV, which is 15 times more intense than the X-ray luminescence spectrum of KCl crystal. With a rise in sodium impurity concentration in KCl-Na crystal to a level of 100 ppm, the intensity of the emission band at 2.8 eV continues to grow (curve 3 in Figure 2). Now, the ratio with the XRL spectrum of KCl crystal is 25 times. Curve 3 in Figure 2 shows that with increasing sodium concentration, in addition to emission with a maximum at 2.8 eV, an additional emission band appears with a maximum at 3.1 eV, which will become dominant at a sodium concentration of 1000 ppm in KCl-Na (curve 4 Figure 2).

In conclusion, we note that in KCl-Na crystal (1000 ppm), the emission bands at 3.1 eV and 2.8 eV (curve 4 in Figure 2) are more intense than in KCl crystal (curve 1 in Figure 1) by more than 55 times and 35 times, respectively.

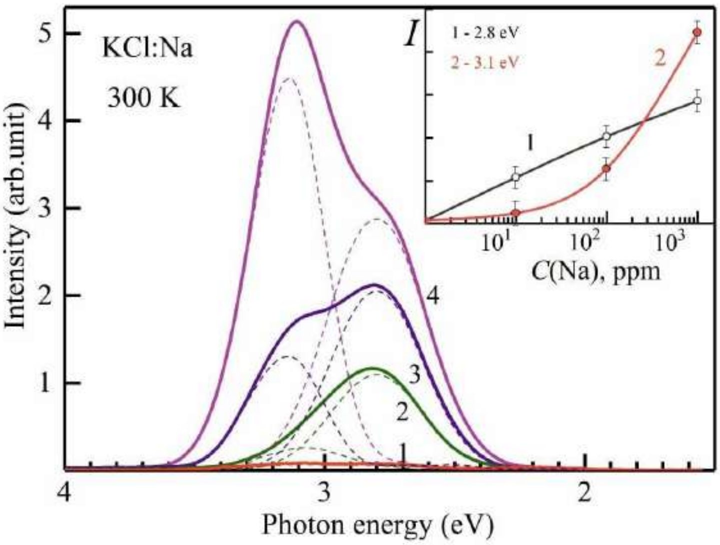

Figure 2 – X-ray luminescence spectra of crystals at 300K:
1– KCl, 2 – KCl-Na (10 ppm), 3 – KCl-Na (100 ppm), 4 – KCl-Na (1000 ppm).

The emission bands in KCl-Na crystals are decomposed into their constituent components, as a result of which the maxima at 3.1 eV and 2.8 eV are clearly established, and the emission band half-widths are 0.37 eV and 0.4 eV, respectively.

These experimental results unambiguously show that the emission bands with maxima at 2.8 eV and 3.1 eV, which correlate with an increase in the sodium concentration in KCl-Na crystal, can be attributed with a high probability to the emission of a self-trapped exciton in the field of single and paired sodium ions, respectively.

Taking into account the decomposed bands into constituent emission components with maxima at 3.1 eV and 2.8 eV, the dependences of their intensity on the sodium concentration in KCl-Na were plotted (Figure 3). It follows from the figure that in KCl-Na crystal, the emission intensity with a maximum at 2.8 eV (curve 1), which is absent in a pure KCl crystal, increases linearly with increasing sodium concentration, which is typical for single cationic impurities located in the cationic lattice site.

It also follows from Figure 3 that in KCl-Na crystal, the emission intensity with a maximum at 3.1 eV (curve 2) increases superlinearly, close to quadratic, with increasing sodium concentration, which is typical for paired impurity centers, in the environment of which the emissive annihilation of the exciton-like creation.

It is known that the accumulation of impurities in the form of aggregates is thermally dissociated by hardening crystals by calcination to high temperatures, at least not lower than half of the AHC melting point [32]. However, the radiation with a maximum at 3.1 eV, which is interpreted by the emissive relaxation of an exciton-like creation in the field of paired sodium ions, after hardening KCl-Na crystal increases in intensity even more, which is not typical for the classical understanding of the behavior of aggregates in AHC matrix. The thermal stability of sodium aggregates in KCl matrix seems to be associated with the creation of NaCl phase, the melting point being higher (801^0C) than the main KCl lattice (770^0C).

From these experimental data, the main effect stands out - a multiple increase in the emission intensity with maxima at 2.8 eV and 3.1 eV in KCl-Na crystals depending on the sodium concentration, compared with a pure KCl crystal at 300 K.

As a working hypothesis, it can be assumed that the detected effect of high-temperature (300 K) ignition of the intensity of exciton-like creation with maxima at 2.8 eV and 3.1 eV in KCl-Na crystals occurs through the mechanism of successive recombination of electrons located in the field of a «relaxing hole» close to cations.

The structure of such an exciton-like creation can be considered as the structure of a self-trapped exciton, formed by recombination of electron-hole pairs, the probability of which is sharply increased by the local deformation created by cationic impurities (Na).

For KCl crystal, the hole component of the exciton-like creation is located at two anionic nodes of the lattice, which has the structure X_2^- of a molecule oriented along <110> crystallographic direction [1,3,4].

This circumstance creates a unique opportunity for a targeted impact on the core of an exciton-like creation by forming an external elastic deformation of crystals (for example, KCl-Na) along the <100> and <110> crystallographic directions.

Experiments show that there is a further increase in the emission intensity at 3.1 eV and 2.8 eV in KCl-Na crystals (curves 2, 3 of Figure 3) in the X-ray luminescence spectra recorded under two directions of uniaxial deformation ($\varepsilon = 0.9\%$). It should be noted that with uniaxial deformation in the <100> direction, a further increase in the emission intensity is 2 times (curve 2 in Figure 3), and with <110> - 3.5 times.

These differences are logically compatible with the AHC deformation theory [32] based on the sliding of anions by the <110> direction due to their elastic shell. Even with deformation <100>, sliding occurs along <110>.

Therefore, the maximum emission intensity at 3.1 eV and 2.8 eV in KCl-Na crystals was registered in the case when the direction of the external elastic deformation (<110>) coincides with the orientation of the X_2^- molecule (<110>), which is the core-relaxed component of the exciton-like creation.

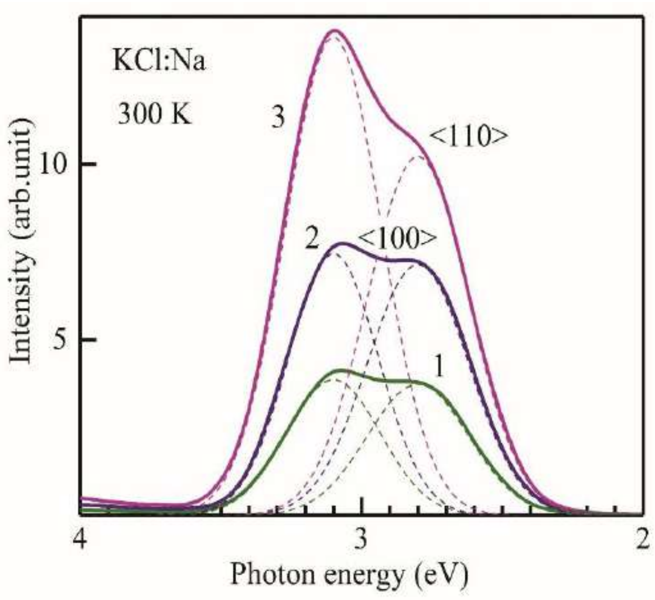

Figure 3. X-ray luminescence spectra of KCl-Na crystals (100 ppm) at 300 K in the absence of deformation (1), with deformation (ε = 0.9%) along crystallographic directions <100> (2) and <110> (3).

Thus, internal local (Li, Na, Sr) and external elastic deformations enhance the luminescence intensity of an exciton-like creation formed by recombination from electron-hole pairs.

Figure 3 (curve 2, 3) shows that an identical increase in the emission intensity is observed at 3.1 eV and 2.8 eV, as evidenced by an increase in the mean free path of an unrelaxed hole.

As a result, the probability of localization near single (Na) and paired (Na, Na) sodium ions have the same character, which is controlled by the emission intensity at 2.8 eV and 3.1 eV, respectively.

It is also possible that the lowering lattice symmetry of local (Na), thermoelastic and uniaxial deformation (<100> and <110>) affects the mean free path of unrelaxed holes up to the formation of spatially separated electron-hole pairs with the transformation of an exciton-like creation.

A comparative analysis of the mean free paths of an unrelaxed hole and a free exciton in KCl crystals shows that at 80 K the mean free path of an unrelaxed hole before self-trapping is up to 30–90 a [2, 4], i.e., it significantly exceeds the free path of an exciton ($2a$) in a crystal KCl [2, 4]. Where a is the lattice constant of crystal. Moreover, with increasing temperature, the mean free path of excitons decreases and the unrelaxed hole apparently increases, as evidenced by the increase in the emission intensity with maxima at 2.8 eV and 3.1 eV at room temperature (Figures 2, 3, 5). Recall once again that emissions with maxima at 2.8 eV and 3.1 eV, which correlate with an increase in the sodium concentration in KCl-Na crystal, are the result of emissive relaxation of an exciton-like creation in the field of single and paired sodium ions, respectively.

The concentrations of sodium, taking into account the coefficient of incorporation into KCl matrix, as the estimate shows, are - 10 ppm, 100 ppm and 1000 ppm, which correspond to the distance between single sodium ions located in the cationic lattice site - $16\div20a$, $8\div10a$ and $5\div7a$. Obviously, the distances of paired sodium ions are significantly greater than the distances of single ions in KCl-Na crystal. However, the X-ray luminescence spectrum of KCl-Na crystals deformed in different directions (<100>, <110>) shows that the emission intensities with maxima at 2.8 eV and 3.1 eV increase at the same rate (curves 2 and 3 of Figure 3). This suggests that the mean free path of an unrelaxed hole stimulated by thermoelastic, local and uniaxial deformation reaches the distance between paired sodium ions, as well as single sodium ions in KCl-Na lattice.

Conclusion. The effect of a multiple increase in the emission intensity with maxima at 2.8 eV and 3.1 eV in KCl-Na crystals, compared with a pure KCl crystal, after hardening (500-600^{0}C), an increase in the concentration of Na ions (for example, 1000 ppm), as well as under the influence of uniaxial deformation along crystallographic directions (<100> and <110>).

Lowering lattice symmetry of KCl matrix by light cations (Na), thermoelastic and uniaxial deformation (<100>, <110>) leads to an increase in the mean free path of an unrelaxed hole to self-trapping.

As a result, the probability of recombination assembly of electron-hole pairs with the formation of an exciton-like creation increases, the emissive relaxation of which occurs in the field of local, thermoelastic and uniaxial deformation with intense luminescence.

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ТОР СИММЕТРИЯСЫНЫҢ ТӨМЕНДЕУІМЕН KCl-Na КРИСТАЛДАРЫНДАҒЫ ЭКСИТОН ТӘРІЗДІ ЛЮМИНЕСЦЕНЦИЯНЫҢ РЕКОМБИНАЦИЯЛЫҚ ЖИЫНЫН КҮШЕЙТУ ӘСЕРІ

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Аңдатпа: KCl-Na монокристалындағы экситон тәрізді түзілулердің радиациялық релаксациясы люминесценттік спектроскопияның тәжірибелік әдістерімен зерттелді. KCl-Na кристалдарының рентгенлюминесценция спектрлерінде алғаш рет бөлме температурасында 2,8 эВ максимумы бар сәулелену жолағы анықталды.

KCl-Na кристалдарындағы 2,8 эВ кезінде сәулелену қарқындылығының көп реттік күшейту әсері қыздырудан кейін (400-700°C), Na иондары концентрациясының артуынан кейін (мысалы, 1000 ppm), сондай-ақ кристаллографиялық бағыттар бойынша (<100> және <110>) бір осьті деформация әсер еткенде барынша жоғары болады.

Сондай-ақ, натрийдің жоғары концентрациясында (1000 ppm) KCl-Na кристалдарында 3,1 эВ-да максимумы бар қарқынды қосымша сәулелену анықталды, бұл қоршаған ортада электронды қозулардың эмиссиялық релаксациясы жүретін жұптасқан натрий иондарына тән.

KCl-Na тор симметриясының термосерпімді кернеумен қыздырылған кезінде, жеңіл катионмен (Na) қоспалау кезінде, сондай-ақ бір осьті деформация кезінде (<100> және <110>) сығумен төмендеуі өздігінен қармалғанға дейін босаңсымаған кемтіктің еркін жүру жолының артуына әкелетіні эксперименттік түрде анықталды. Нәтижесінде экситон тәрізді түзілудің пайда болуымен электрон-кемтік жұптарының рекомбинациялық жиын ықтималдығы артады, оның радиациялық релаксациясы қарқынды люминесценцияға ие термосерпімді, нүктелік және бір осьті деформация өрісінде жүреді.

Түйін сөздер: сілтілі галоидты кристалдар, рентгенлюминесценция, күшею, нүктелік және бір осьті деформация, өздігінен қармалған экситон, экситон тәрізді люминесценция, электрон-кемтік рекомбинациясы, экситон тәрізді түзілу.

ЭФФЕКТ УСИЛЕНИЯ РЕКОМБИНАЦИОННОЙ СБОРКИ ЭКСИТОНОПОДОБНОЙ ЛЮМИНЕСЦЕНЦИИ В КРИСТАЛЛАХ KCl-Na ПРИ ПОНИЖЕНИИ СИММЕТРИИ РЕШЕТКИ

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Аннотация: Экспериментальными методами люминесцентной спектроскопии исследована излучательная релаксация экситоноподобных образований в монокристалле KCl-Na. В спектрах рентгенолюминесценции кристаллов KCl-Na впервые обнаружена полоса излучения с максимумом при 2,8 эВ при комнатной температуре.

Эффект многократного усиления интенсивности излучения с максимумом при 2,8 эВ в кристаллах KCl-Na максимален после закалки (400-700^{0}C), увеличения концентрации ионов Na (например, 1000 ppm), а также при воздействии одноосной деформации по кристаллографическим направлениям (<100> и <110>).

При больших концентрациях натрия (1000 ppm) в кристаллах KCl-Na обнаружено также интенсивное дополнительное излучение с максимумом при 3,1 эВ, что свойственно для парных ионов натрия, в окружении которых происходит излучательная релаксация электронных возбуждений.

Экспериментально установлено, что понижение симметрии решетки KCl-Na термоупругим напряжением при закалке, легким катионом (Na) при допировании, а также сжатием при одноосной деформации (<100> и <110>) приводит к возрастанию длины свободного пробега нерелаксированной дырки до автолокализации. В результате увеличивается вероятность рекомбинационной сборки электронно-дырочных пар с формированием экситоноподобного образования, излучательная релаксация которого происходит в поле термоупругой, локальной и одноосной деформации с интенсивной люминесценцией.

Ключевые слова: щелочногалоидные кристаллы, рентгенолюминесценция, тушение, локальная и одноосная деформация, автолокализованный экситон, экситоноподобная люминесценция, электронно-дырочная рекомбинация, экситоноподобное образование.