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Features of Localization of Radiation Defects and Electronic Excitations in the Field of Light-weight Sodium in Potassium Chloride Single Crystals

© K.Sh. Shunkeyev[1], A.S. Tilep[1], Sh.Zh. Sagimbayeva[1,¶], Zh.K. Ubaev[1], A.Ch. Lushchik[2]

[2] K. Zhubanov Aktobe Regional University, 030000 Aktobe, Kazakhstan
[2] Institute of Physics, University of Tartu, Tartu, Estonia
¶e-mail: sshynar.2021@gmail

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The stability of KCl single crystals doped with sodium impurity ions was analyzed for the first time using absorption and luminescence spectroscopy methods. An indicator for detecting the central position of the sodium ion in the cation lattice site of a KCl$-$Na single crystal is the optical absorption bands induced by X-ray radiation with maxima at 6.35 and 3.5 eV (respectively, interstitial ions and chlorine atoms localized near impurity Na$^+$ ions), as well as characteristic luminescence at 2.8 eV and 3.1 eV of exciton-like formations created by recombination near single Na$^+$ or pair Na$^+-$Na$^+$ impurity ions, respectively. It has been shown that in KCl:Na crystals stored for a long time at room temperature, Na$^+$ ions leave the regular cation sites and form nano-sized clusters. However, subsequent quenching of the „decayed“ crystal at high temperatures (400$-$700°C) leads to partial reverse incorporation of sodium ions into the cation sites of the KCl:Na lattice. With increasing quenching temperature, the degree of restoration increases and reaches saturation (80% of the characteristics of freshly grown crystals).

Keywords: KCl:Na single crystal, decay of KCl:Na — precipitation of sodium ion, thermal quenching — restoration of the KCl:Na lattice, X-irradiation, near-sodium radiation defects and luminescence.

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1. Introduction

To date, it has been proven to satisfaction that the act of annihilation of an anionic self-trapping/self-trapped exciton (STE) in alkali halide crystals (AHC) leads both to the creation of primary point defects in the crystal lattice and to the emission of a characteristic luminescence quantum (see [1–3] and references therein). AHCs traditionally serve as the basis for the development of effective phosphors, scintillators, and laser materials [4–10]. In the context of these applications, in recent years, intensively researched are spectroscopic characteristics of AHCs with a reduced lattice symmetry because of local [11–17], uniaxial [18–20], or hydrostatic deformation [21–24]. Low-temperature uniaxial deformation has been successfully applied to determine the nature of E_x-luminescence in alkali metal iodides [18,19].

Many years of experimental research in AHCs have resulted in well-established spectral characteristics and structures of radiation defects, as well as the nature of the luminescence of the excitons localized in the field of various point defects/impurities [1–3,11–13,15–19] .

This study investigates and analyzes in detail the effect of instability of the substitutional sodium impurity ion - its removal from a regular cation site of a KCl:Na crystal. The study was carried out using absorption and luminescence spectroscopy methods and was based on the well-established structures of radiation defects and the nature of the luminescence of exciton-like formations.

2. Experimental results

2.1. Objects of the study and experimental set-ups

KCl and KCl:Na crystals were grown at the Institute of Physics of the University of Tartu (Estonia) using the Stockbarger method and raw materials additionally purified via halogen gas flow and manifold zone melting. As a result of this comprehensive purification method, KCl crystals with impurity ion concentrations in the range of $10^{-4}-10^{-5}$ mol% were produced.

Doped KCl:Na crystals were grown from zone-purified KCl raw materials with the addition of NaCl powder previously dried in vacuum [1,3,14,15] . The concentration of sodium ions in the KCl:Na samples, taking into account the coefficient of sodium integration into the KCl lattice, was 0.1$-$1.0 mol%.

The crystals under study were annealed in a Programix TX 25 electric muffle furnace. During the programmed quenching procedure, KCl:Na crystals were heated at a constant rate of 15°C/min to a specified temperature (in the range from 300 to 700°C), held at this temperature for 15 min, and naturally cooled on a quartz substrate to room temperature [25].

Characteristic X-ray radiation from a Spellman DF60N3X4596 generator (20 mA, 45 kV, W-anticathode) was used as ionizing radiation. The optical absorption of radiation defects created in KCl:Na crystals was recorded with a „Evolution-300“ spectrophotometer in a wide range of spectrum (6.5–1.1 eV) and temperature (85–400 K) [26]. Thermal annealing of radiation-induced absorption was carried out in a stepwise mode: the pre-irradiated crystal was heated to a specified temperature, held at this temperature for 2 min, and then cooled down to 85 K (at this temperature all optical absorption spectra were recorded).

To study the X-ray luminescence (XRL) spectra of crystals, bremsstrahlung radiation from a RUP-120 unit with a W-anticathode operating in the 3 mA and 100 kV mode was used. The XRL spectra of crystals were recorded in the range of 6.0–1.5 eV using a MSD-2 high-aperture monochromator, a H 8259-01 photomultiplier by Hamamatsu and a special SpectraScan software [14 ,27]. The scanning speed of the XRL spectra was 10 nm/s. All necessary corrections have been made to the XRL spectra. Integral thermally stimulated luminescence (TSL) curves were measured with heating a pre-irradiated crystal at a constant rate of $\beta = 0.15$ K/s using a H 8259-01 detector and a specially developed ThermoScan program [14,28]. Both above-mentioned software programs were developed by the authors together with programmers from Yanka Kupala State University of Grodno (The Republic of Belarus).

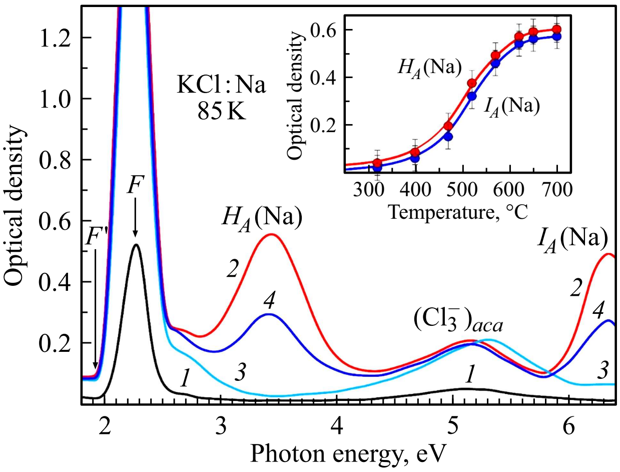

Figure 1. Absorption spectra of radiation defects in KCl (curve *1*) and KCl : Na (1 mol%, curves *2–4*) crystals after isodose irradiation with X-rays at 85 K (20 mA, 45 kV, W-anticathode, 3 h). Curves *1* and *2* — for freshly grown KCl and KCl : Na (1 mol%) crystals, respectively; *3* — for KCl : Na (1 mol%) after 3–4 months of storage at room temperature („decayed crystal"); *4* — after additional quenching of the „decayed" KCl : Na at 600°C. All spectra were measured at 85 K. The inset shows dependences of the absorption bands of I_A(Na) and H_A(Na) centers induced by isodose irradiation (3 h) on the temperature of additional quenching of the „decayed" KCl : Na.

2.2. Radiation defects in the field of sodium impurity ions in a KCl matrix

In AHCs, a light homologue cation located in a regular cation site (Li_c^+ or Na_c^+) creates an energetically favorable situation for stabilizing around it the mobile defects created by radiation [11–15, 14.27], which include both interstitial halogen ions (for example, I_A(Na)) and atoms (H_A(Na)). As a result, the temperature stability of anion radiation defects localized in the field of light cations increases compared to the case of their location in the regular AHC lattice sites. For example, I- and H-centers in a pure KCl crystal have temperatures of maximum destruction at 20 and 50 K, respectively, and I_A(Na)- and H_A(Na)-centers in KCl : Na are stable at temperatures up to 140–150 K [3,4,13–15], i. e. the stability of anion interstitials increases by more than 100 K.

In the KCl : Na crystal, the structure of I_A(Na)- and H_A(Na)-centers is unambiguously established using EPR [11–13] and optical [14,29] spectroscopy methods: an interstitial chlorine ion or an atom, respectively, localized in the field of an impurity sodium ion located in a regular cation lattice site. Structural models of I_A(Na)- and H_A(Na)-centers are presented in the conclusion of this study. Optical absorption bands with maxima at 6.35 eV and 3.5 eV, corresponding to I_A(Na)- and H_A(Na)-defects, can be considered as an indicator of the central positioning of sodium ions in the cation site of KCl : Na crystal lattice.

Fig. 1 shows the optical absorption spectra of radiation defects in freshly grown KCl and KCl : Na (1 mol%) crystals subjected to isodose irradiation with X-rays at 85 K. Compared to the spectrum of a pure KCl crystal (curve *1*), in the KCl : Na crystal (curve *2*) there is a sharp enhancement of the absorption bands peaked 6.35 eV, 5.2 eV, 3.5 eV, 2.3 eV, and associated with the number of radiation-induced anion defects, in particular, I_A(Na), trihalide quasi-molecules $(\mathrm{Cl}_3^-)_{\mathrm{aca}}$, H_A(Na), as well as F- and F'-centers — an anion vacancy with one ($v_a^+e^-$) or two ($v_a^+e^-e^-$) captured electrons, respectively. Subscripts indicate that the quasi-molecule occupies two anion (a) and one cation (c) lattice sites; the creation processes of these defects via $H{-}H_A$(Na) or $H{-}V_{KA}$(Na) pair interactions of two-halide molecules of X_2^- type are described in detail in [1,3,15,30]).

The creation efficiency of radiation defects, both near-sodium-localized anion interstitials — I_A(Na)- and H_A(Na)-centers, and intrinsic defects of hole ($(\mathrm{Cl}_3^-)_{\mathrm{aca}}$-centers) and electronic (F- and F'-centers) type increases with increasing sodium impurity concentration [14]. It should be noted that the concentration of F-centers, calculated by the well-known Smakula formula based on the corresponding optical absorption band in the KCl : Na crystal (curve *2*), increased compared to pure KCl (curve *1*) more than 10 times and amounts to $1.8 \cdot 10^{17}\,\mathrm{cm}^{-3}$.

The efficiency of creating a classical point vacancy defect (F-center) in KCl : Na crystals, which increases with the sodium impurity concentration, is primarily due to the stabilization of mobile interstitial ions and atoms of halogen near sodium ions with the formation of I_A(Na)- and H_A(Na)-centers, as well as the effective formation of trihalide $(\mathrm{Cl}_3^-)_{\mathrm{aca}}$-centers (see [1,3,15,30] for details).

The study of thermal annealing of sodium-related radiation defects in KCl : Na crystals showed that the defects anneal synchronously, and the temperatures of the fastest destruction of I_A(Na)- and H_A(Na)-centers are determined as 145 K and 135 K, respectively (see also [29]). Annealing of I_A(Na)- and H_A(Na)-centers is also accompanied by a decrease in the concentration of electron-type F- and F'-centers and some enhancement of the absorption band with a maximum at 5.2 eV, corresponding to $(\mathrm{Cl}_3^-)_{\mathrm{aca}}$-centers [1–3,15,30]. It is worth reminding that electronic F'-centers (absorption band at 1.8 eV) have a low ionization temperature (130 K) [14].

Repeated spectroscopic investigation of KCl : Na crystals after long-term storage of samples at room temperature allowed us to conclude that the crystal under study was unstable. As already noted, in the radiation-induced absorption spectrum of a doped KCl : Na crystal freshly grown from an ultrapure matrix, bands at 6.35 eV and 3.5 eV are clearly visible (Fig. 1, curve *2*), associated with I_A(Na)- and H_A(Na)-centers. However, these bands are practically absent after isodose irradiation of the KCl : Na crystal stored for a long time (3–4 months) in a desiccator at room temperature (Fig. 1, curve *3*).

It should be immediately noted that quenching such a „decayed " KCl : Na crystal after a long-term storage according to the method described in section 2.1 leads to a partial reverse incorporation of sodium ions into the cation lattice sites. This is evidenced by the newly appeared absorption bands of I_A(Na)- and H_A(Na)-centers in the additionally quenched KCl : Na crystal (Fig. 1, curve *4*).

We have investigated in detail the process of restoration of absorption bands of I_A(Na)- and H_A(Na)-centers in isodose-irradiated „decayed" crystals of KCl : Na pre-quenched to a certain temperature in the range from 300 to 700°C (inset in Fig. 1). After additional quenching to temperatures in the range from 450 to 600°C, an almost linear increase in the absorption of I_A(Na)- and H_A(Na)-centers is observed, and with quenching above 600°C the saturation of the recovery process can be seen. Thus, it can be assumed that when heated to 450–600°C, light sodium ions are released from nanoscale clusters and are again incorporated into regular cationic sites of the KCl : Na lattice. It should be noted that with increasing quenching temperature, a parallel increase in the optical absorption of I_A(Na) and H_A(Na) centers is observed (inset in Fig. 1).

It follows from the phase diagram that KCl : NaCl crystallizes from the melt into a continuous series of solid solutions, however, at $T = 495°\mathrm{C}$ their spinodal decomposition begins, which is associated with the occurrence of fluctuations in the distribution of impurities over the lattice and the formation of clusters of impurity ions without separating them into an independent phase [31]. For temperatures close to room temperature, the solubility limits of NaCl in KCl have not been established. At the same time, a significant difference (by approximately 35%) in the ionic radii of potassium (1.33 Å) and sodium (0.98 Å) suggests a very low miscibility limit of these systems, which is consistent with the inhomogeneity of sodium distribution in KCl established by us even at an impurity concentration of 0.1 mol%.

2.3. Exciton-like luminescence in the field of an impurity sodium ion

The effect of the KCl : Na (0.1 mol%) crystal decay, associated with the removal of sodium ions from regular cation sites, was also studied using a highly sensitive luminescence technique based on changes in the XRL spectra of the decayed and quenched crystals.

We used the XRL band with a maximum at 2.8 eV as the main luminescent characteristic of sodium impurity ions in the KCl : Na crystal, which was interpreted as radiative annihilation of an exciton-like formation in the field of a sodium ion located in the cation site [14]. In other words, the luminescence with a maximum at 2.8 eV is due to recombinationally generated exciton-like formation near sodium in a regular cation site according to the following scheme:

$$\mathrm{e}^- + V_{KA}(\mathrm{Na}) \rightarrow \mathrm{e}_\mathrm{s}^0(\mathrm{Na}) = h\nu\ (2.8\,\mathrm{eV})$$

The hole component of the bound exciton is the valence hole, which quickly loses its mobility in KCl — a self-trapped hole (or V_K center) near the impurity cation (sodium).

Based on the previously presented results of absorption spectroscopy, the suppression of luminescence at 2.8 eV in the „decayed" crystals of KCl : Na was expected, because after the removal of sodium from the regular cation sites the corresponding near-sodium bound excitons cannot be longer formed as well. In „decayed" (stored for a long time after growth) crystals of KCl : Na (0.1 mol%), a weak XRL band was recorded at 2.8 eV (curve *1* in Fig. 2), which intensity does not depend on the sodium concentration in the range from 0.01 to 1.0 mol%.

The dependence of the peak XRL intensity at 2.8 eV (normalized to that in the „decayed" KCl : Na crystal) on the quenching temperature is given in the table. It can be seen that intermediate quenching at 400°C enhances the luminescence of near-sodium bound excitons by approximately 37 times, quenching at 520°C enhances it by 246 times, and further increase in quenching temperatures up to 700°C increases the characteristic luminescence by 525 times.

In KCl : Na (0. mol%) crystals quenched at high temperatures, along with an intense XRL band at 2.8 eV, a band with a maximum at 3.1 eV is clearly visible (Fig. 2, curve *4*), which is associated with the exciton-like formation in the field of a pair of impurity ions $\mathrm{Na}^+{-}\mathrm{Na}^+$ [14].

Thus, in quenched KCl : Na (0.1 mol%) crystals, two intense luminescence bands with maxima at 2.8 eV and 3.1 eV were recorded, which are the result of radiative annihilation (decay) of an exciton-like formation in the field of a single or pair sodium impurity center, respectively.

The inset in Fig. 2 shows the peak intensity of the XRL band at 2.8 eV as a function of the quenching temperature

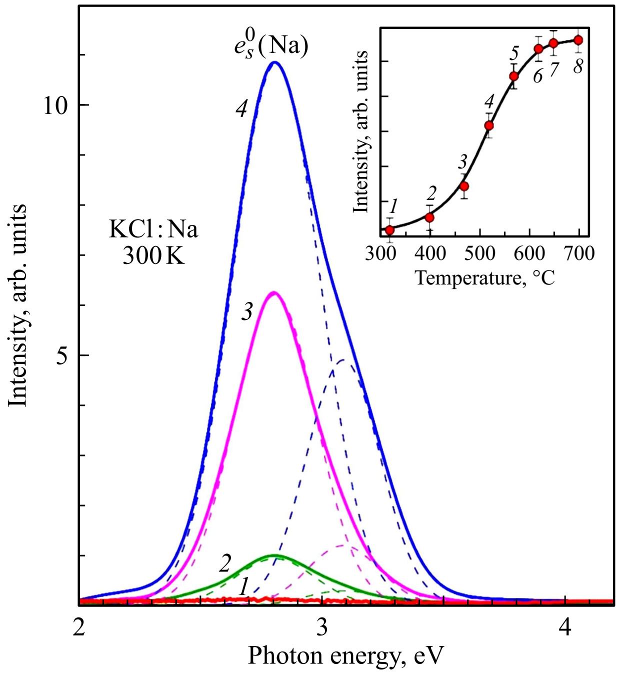

Figure 2. XRL spectra measured for „decayed “ KCl:Na crystals (0.1 mol%) before (curve *1*) and after additional quenching of such a crystal at 400°C (curve *2*), 520°C (curve *3*), and at 700°C (curve *4*). All spectra were measured at room temperature. Elementary Gaussians obtained by decomposing the XRL spectra are shown by dashed lines. The inset shows the dependence of the peak intensity of the 2.8 eV band on the crystal quenching temperature: point *1* — quenching at 320°C, *2* — 400°C, *3* — 470°C, *4* — 520°C, *5* — 570°C, *6* — 620°C, *7* — 650°C and *8* — 700°C.

Dependence of the peak XRL intensity with a maximum at 2.8 eV on the quenching temperature of the „decayed“ KCl : Na crystal (according to Fig. 2, curves *2*–*4*)

Curve Num in Fig. 2	Temperature of quenching, °C	XRL intensity at 2.8 eV in „decayed“ and quenched KCl : Na, arb.units
1	before quenching	1
2	400	37
3	520	246
4	700	525

(from 320 to 700°C). It can be seen from the $I \sim f(T)$ dependence that from the 400°C quenching (point *2*) a band enhancement starts, the peak XRL intensity at 2.8 eV grows sharply and almost linearly in the quenching range from 470 to 570°C (points *3*–*5*), and at higher quenching temperatures (points *6*–*8*) a saturation of the $I \sim f(T)$ dependence takes place. At quenching temperatures of 600–700°C, the deformation of the crystal lattice apparently increases due to an increase in the thermoelasticity of the material, and the creation of vacancy defects is also possible during the interaction of different types of dislocations.

2.4. Thermally stimulated luminescence of radiation defects localized in the sodium field in the KCl matrix

One of the highly sensitive methods for studying the recombination-type luminescence of radiation defects is thermally stimulated luminescence (TSL), which provides complete information about the destruction temperatures of radiation defects localized in the sodium field in KCl : Na crystals. Such defects include I_A(Na)-, H_A(Na)-, V_{KA}(Na)- and V_{KAA}(Na ,Na) - centers.

From the TSL curves shown in Fig. 3, measured for two isodose-irradiated crystals of KCl : Na — „decayed“ crystal (curve *1*) and crystal that is additionally quenched at 600 °C (curve *2*) — it follows that after quenching, the high-temperature peak at 400 K associated with the thermal destruction of Cl_3^- - and F-centers becomes considerably weaker, but the main lower-temperature peaks are significantly enhanced. The nature of these TSL peaks has been carefully studied for a long time, and based on literature data [1,3,14,20,32] in Fig. 3 the correspondence of peaks to the thermal destruction of corresponding anion defects is indicated above the peaks (V_F is a V_K-center localized near a cation vacancy).

It should be noted that to date, for many AHCs, all TSL peaks have been identified that are caused by the radiative recombination of charge carriers released from sequentially thermally destroyed defects with still stable defects that include a charge carrier of the opposite sign (for LiF, see, for example, [32]). Therefore, the emergence of certain TSL peaks in irradiated crystals that have been additionally quenched can also serve as an indicator of a change in the state of the impurity in the crystal. It is important to emphasize that the sharp enhancement of TSL peaks at 130 K (H_A), 145 K (I_A(Na)), 265 K (V_{KA}), 310 K (V_{KAA}) in the quenched crystal is indicative of the reverse incorporation of sodium impurity ions into regular cation positions.

Thus, along with the previously noted characteristic optical absorption bands, the listed TSL peaks serve as a direct indicator of the restoration of a homogeneous distribution of sodium impurity over regular cationic lattice sites in a quenched KCl : Na crystal. The structure of the I_A(Na)- and H_A(Na)-centers is shown in Fig. 4.

Also, it should be noted that the comparison of optical absorption spectra and TSL curves for freshly grown (or quenched) and „decayed“ KCl : Na crystals (Fig. 1, 2, and 3) shows that $(Cl_3^-)_{\text{aca}}$-centers in freshly grown (or quenched) crystals start destructing at rather low temperatures and there are very few of them left by a temperature of 400 K (unlike the case of the „decayed“ crystal). Such low-temperature destruction of trihalide quasi-molecules can be associated with a significant difference in the mobility of single cation vacancies in freshly grown (quenched) and „decayed“ KCl : Na crystals [29].

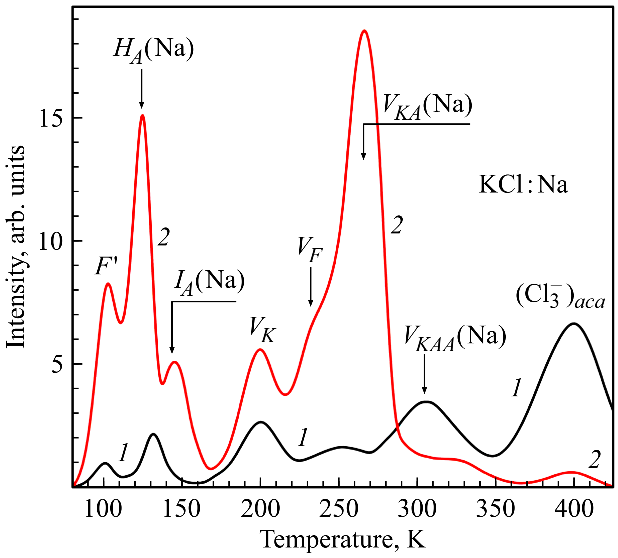

Figure 3. Thermally stimulated luminescence of KCl : Na crystals (1.0 mol%) isodose-irradiated with X-rays at 85 K (3 h, 20 mA, 45 kV, W-anticathode). *1* — „decayed" crystal, *2* — additionally quenched at 600°C. Heating rate is $\beta = 0.15$ K/s.

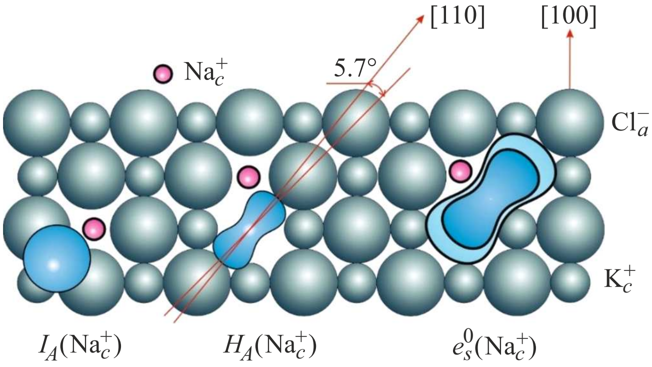

Figure 4. Structural models of radiation-induced defects and exciton-like formation (bound exciton) in the sodium field. The subscript indicates the place occupied by the defect/ion in the crystal lattice (*a, c* — anion and cation sites, respectively), the superscript indicates the charge relative to the lattice.

Numerous experimental results show that the efficiency of $(\mathrm{Cl}_3^-)_{\mathrm{aca}}$-center formation in decayed and freshly grown KCl : Na crystals is the same. In a decayed KCl : Na crystal, the sodium ion apparently moves from a regular site to an interstice, because sizes of the sodium ion (0.98 Å) and the tetrahedral void (0.9 Å) of the KCl lattice are close to each other.

As a result, an interstitial sodium ion (Na_i^+) and a cation vacancy (v_c^-) can form in the decayed KCl : Na crystal, the latter being a seed for the association of two mobile halogen atoms with the formation of $(\mathrm{Cl}_3^-)_{\mathrm{aca}}$-centers. Thus, in a decayed KCl : Na crystal, all cation vacancies are involved as a building material for the creation of $(\mathrm{Cl}_3^-)_{\mathrm{aca}}$-centers, and, as a consequence, this crystal lacks the low-temperature annealing stage of destruction of $(\mathrm{Cl}_3^-)_{\mathrm{aca}}$-centers, which is clearly recorded in a freshly grown KCl : Na crystal.

3. Conclusion

The effect of instability of sodium-doped KCl : Na crystals is investigated in this study based on the structures and characteristics of radiation defects clearly established in the literature, which are localized near impurity sodium ions (H_A(Na)-, I_A(Na)-, V_{KA}(Na)-, V_{KAA}(Na,Na)-centers), as well as near-impurity-localized (near-sodium bound excitons, e_s^0(Na)). Structural models of near-sodium radiation-induced defects — $I_A(\mathrm{Na}_\mathrm{c}^+)$ and $H_A(\mathrm{Na}_\mathrm{c}^+)$, $V_{KA}(\mathrm{Na}_\mathrm{c}^+)$ and exciton-like formations — $\mathrm{e}_\mathrm{s}^0(\mathrm{Na}_\mathrm{c}^+)$ in the KCl : Na lattice are shown in Fig. 4.

The comparison of optical absorption, XRL and TSL spectra for isodose X-ray irradiated KCl : Na crystals — freshly grown, decayed (after long-term storage at room temperature), and also subjected to additional quenching crystals — made it possible to isolate indicators, which are indicative of the presence of impurity sodium ions in regular cation lattice sites or the absence of a homogeneous distribution of impurity ions in decayed samples. Such indicators are optical absorption bands with maxima at 6.35 eV and 3.5 eV (caused by interstitial chromium ions or atoms localized near Na^+ — I_A(Na)- and H_A(Na)-centers, respectively), as well as the luminescence band at 2.8 eV, arising due to the radiative decay of recombination-generated bound excitons near Na^+ impurity ions.

Based on the disappearance of characteristic indicators, it was established that long-term storage of doped crystals leads to the removal of impurity sodium ions from regular cation lattice sites and, probably, the formation of their nanoscale clusters. However, subsequent quenching at high temperatures (400–700°C) ensures partial incorporation of sodium ions back into the cation sites and restores the homogeneous distribution of sodium ions in the KCl : Na lattice. With increasing quenching temperature, the degree of restoration increases and reaches saturation (approximately 80% of the characteristics of freshly grown crystals). At an extremely high quenching temperature (700°C), an additional band at 3.1 eV appears in the XRL spectra, which dominates in crystals with a high sodium concentration; we recently attributed this XRL band to exciton-like formations near $\mathrm{Na}^+{-}\mathrm{Na}^+$ pairs of impurity ions [14].

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Conflict of interest

The authors declare that they have no conflict of interest.

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