Eurasian Journal of Physics and Functional Materials

2022, 6(2), 139-150

# Exciton-like luminescence of KCl matrix under thermoelastic, local and uniaxial deformation

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DOI: **10.32523/ejpfm.2022060206** Received: 05.05.2022 - after revision

The radiative relaxation of exciton-like formations in KCl-Na single crystal has been studied by experimental methods of luminescent spectroscopy. The amplifying effect of the radiation intensity with a maximum at 2.8 eV up to 500 times as compared to pure KCl has been detected in KCl-Na. The luminescence efficiency of an exciton-like formation increases with rise in: sodium ions concentration ( $10 \div 1000$  ppm), thermal exposure ( $500^{\circ}C \div 600^{\circ}C$ ) and the degree of uniaxial deformation along <100> and <110> crystallographic directions. Previously, such a scale of the luminescence enhancement effect has not been registered in KCl matrix at room temperature. At high sodium concentrations (1000 ppm) in KCl-Na crystals, the additional intense emission with a maximum at 3.1 eV has also been revealed, which is typical for pair sodium ions. It is interpreted that the exciton-like formation in the sodium field with the maximum quantum yield of luminescence is formed by recombination assembly of electron-hole pairs due to the mobility of unrelaxed holes.

**Keywords:** alkali halide crystals; X-ray luminescence; quenching; local and uniaxial deformation; self-trapped exciton; exciton-like luminescence; electron-hole recombination; exciton-like formation

### Introduction

The fundamental regularities of the relaxation of electronic excitations in alkali halide crystals (AHCs) have been studied to a fairly full extent by modern research methods and, as a result, of which the channels for the formation of primary radiation defects (F, -H- pairs) and the appearance of intrinsic luminescence of the lattice occurs from the self-trapping state of anionic excitons [1–4]. AHCs

are traditionally used as ionizing radiation dosimeters and scintillation detectors [4–6].

The colossal luminescent abilities of alkali halide crystals manifest themselves at very low temperatures (4.2 K), and are completely extinguished at room temperature, and this is the main constraint on the wide use of scintillation detectors based on ionic crystals.

In AHC scintillators (NaI-Tl, KI-Tl, CsI-Tl) it was established that the transfer of excitation energy to luminescence centers occurs due to the high mobility of free excitons and electron-hole pairs [1–4]. However, crystals with a high mobility of unrelaxed holes, which have a sufficient mean free path before self-trapping and are capable of forming exciton-like formations with a high luminescence quantum yield, are still ignored.

A series of original researches have been carried out under direct impact on the channels of radiative 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].

To determine the limiting yield of luminescence associated with exciton energy transfer, an important role is played by the mean free path of excitons before their self-trapping. In this regard, it is necessary to establish a physical parameter that makes it possible to influence the mean free path of electronic excitations before self-trapping.

In the future, it is advisable to develop a technology for influencing the formation of a scintillation impulse through exciton and electron-hole mechanisms.

This research work presents original experimental results on the ignition of intensity of exciton-like luminescence in a KCl matrix during the recombination formation of electron-hole pairs in a sodium field, stimulated by thermoelastic quenching and uniaxial (<100>, <110>) deformation.Recombination occurs due to mobile unrelaxed holes, in contrast to the recombination of electrons with self-trapped holes detected in alkali metal sulfates [24, 25].

# Objects of research and technique of experiment

KCl-Na crystals were grown at the Institute of Physics of the University of Tartu in Estonia using the Stockbarger method in a vacuumized quartz ampoule after a step-by-step preliminary purification [26–28].

The crystal purification algorithm consists of the following sequential steps:

1. The long-term vacuum drying of powdered raw materials KCl at  $80 \div 100^{\circ}C$  to remove oxygen-containing impurities;

2. Heating of dried raw materials under vacuum to the melting temperature  $(780 \div 800^{\circ}C)$ ;

3. Treatments of KCl melt in a chlorine gas current for purification from the impurity bromine from the melt in the form of gas. As a result, KCl melt was completely purified from anionic homologues (Br);

4. Multiple recrystallization of raw materials KCl "zone melting", based on

the difference in the coefficients of embedding (k) ions from the melt into the solid solution [26–28]. The more significantly the embedding coefficient (k«1) differs, the more efficiently KCl raw materials are cleaned from such impurities.

As a result of this complex purification method, KCl crystals with a low concentration of impurity ions –  $0.01 \div 1.0$  ppm were obtained.

KCl-Na crystals were grown on the basis of zone-purified KCl raw materials with additives of NaCl powders preliminarily dried in vacuum [26–28]. The concentration of sodium ions in the KCl-Na samples, taking into account the coefficient of incorporation of sodium into the 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).

Quenching 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 -  $400 \div 600$  °C with exposure for 15 minutes at these temperatures in the "Programix TX 25" electric muffle furnace.

Uniaxial deformation ( $\epsilon = 0.8 \div 0.9\%$ ) of crystals was carried out along <100> and <110> crystallographic directions in a special cryostat at low temperatures (85 K) under conditions of high technical vacuum (10<sup>-6</sup>) Torr [29].

Scanning of the X-ray luminescence spectra (XRL) of the crystals was held in the range of  $1.8 \div 6.0$  eV using a high-aperture MSD-2 monochromator and a H 8259 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. With this mode of operation of the X-ray machine, structural radiation defects are not created in crystals, for example, F-centers, which have a maximum absorption spectrum at 2.3 eV, distorting the emission spectra of crystals due to reabsorption.

### **Experimental results**

The experiments were conducted in successive impacts of local (Na), thermoelastic and uniaxial deformation on the XRL spectra of the 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 quenching temperature, which are realized by a sharp cooling of the crystals calcined in a quartz ampoule to various high temperatures –  $170^{\circ}C$ ,  $320^{\circ}C$ ,  $470^{\circ}C$ ,  $520^{\circ}C$  and  $700^{\circ}C$ . The quenching 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

quenched 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 unquenched 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.



Figure 1. X-ray luminescence spectrum of KCl-Na crystals measured at room temperature before quenching.

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 [30]. As a result of the formation of aggregates in KCI-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 KCI-Na crystal, regardless of the sodium concentration (Figure 1).

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 ( $170 \div 700^{\circ}C$ ) in order to obtain a stable intense signal characterizing the placement of single sodium ions in the cationic lattice sites.

Figure 2 presents the X-ray luminescence spectra at room temperature of KCl-Na crystals (50 ppm), quenched by calcination to high temperatures (curve  $2 - \text{ at } 400^{\circ}C$ , curve  $3 - \text{ at } 520^{\circ}C$ , curve  $4 - \text{ at } 700^{\circ}C$ ) followed by cooling on a quartz substrate in air up to room temperature.



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

Dependence of the intensity of the luminescence band with a maximum at 2.8 eV on the quenching temperature –  $I \sim f(t)$  of KCl-Na crystals (50 ppm) after quenching 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). ( $\varepsilon = 0.8 \div 0.9\%$ ).

It follows from Figure 2 that the intensity of the luminescence band with a maximum at 2.8 eV begins to grow with an increase in the quenching temperature (Table 1): at 400°C 44.8 times than before quenching (compare curves 2 and 1), at 520°C 296.6 times than before quenching (compare curves 3 and 1) and at 700°C by 511.5 times than before quenching (compare curves 4 and 1). At high quenching 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 formation in the field of paired sodium ions.

Each thermal quenching was carried out on a fresh piece of KCl-Na crystals grown on zone-purified KCl raw materials. Comparison of the intensities of the XRL spectra of KCl-Na and KCl crystals was carried out under strictly identical conditions of quenching and registration at room temperature with a constant operation of the RUP-120 X-ray unit with a W anticathode (3 mA and 100 kV).

ratio $(I_{2.8}/I_0, I_{3.1}/I_0)$ depending on the quenching temperature					
Number of	<i>I</i> - intensity		Ratio		Quenching
spectrum	I <sub>2.8</sub>	I <sub>3.1</sub>	$I_{2.8}/I_0$	$I_{3.1}/I_0$	temperature, °C
1	330	290	1.5	1.3	27
2	9860	3190	44.8	14.5	400
3	65250	12965	296.6	58.9	520
4	112520	52865	511.5	240.3	700

Table 1.

Luminescence intensities with maxima at 2.8 eV ( $I_{2.8}$ ) and 3.1 eV ( $I_{3.1}$ ) in KCl-Na, as well as the background signal ( $I_0 = 220$  impulses) for KCl and their ratio ( $I_{2.8}/I_0$ ,  $I_{3.1}/I_0$ ) depending on the quenching temperature.

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

The inset in Figure 2 shows the complete contour of the dependence of the intensity of the luminescence band with a maximum at 2.8 eV on the quenching temperature –  $I \sim f(t)$ , constructed on the basis of direct experimental data on the registration of X-ray luminescence spectra (curves 2–4 of Figure 2) of quenched KCl-Na crystals. Points (1-8) correspond to the maximum intensity of the luminescence band at 2.8 eV of quenched KCl-Na crystals (50 ppm).

It can be seen from the full contour of the dependence  $I \sim f(t)$  that, starting from 400°C of quenching (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 quenching temperatures ( $600^{\circ}C \div 700^{\circ}C$ ), apparently, the deformation in the crystal lattice increases due to an increase in the thermoelasticity of the material, and the formation 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 quenching temperature range of  $500^{\circ}C \div 700^{\circ}C$  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 quenched 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  $3.05 \div 3.2$  eV and  $2.6 \div 2.8$  eV in the X-ray luminescence spectrum of the zone-purified KCl crystal were registered by us (curve 1 in Figure 3). The latter seem to be associated with uncontrolled defects or impurities. Also the radiative relaxation of anionic excitons localized in the field of anionic vacancies and divacancies is known [31–33], having maxima at 2.7 eV and  $3.1 \div 3.4$  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 radiative relaxation of excitons in their environment.

From the comparison of curves 1 and 2 in Figure 3, it can be seen that in the X-ray luminescence 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 XRL spectrum of KCl crystal. With an increase in the sodium 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 3). Now, the ratio with the XRL spectrum of KCl crystal is 25 times. Curve 3 in Figure 3 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 3).



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

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 3) are more intense than in KCl crystal (curve 1 in Figure 1) by more than 55 times and 35 times, respectively.

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 4). 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.



Figure 4. Dependence of the intensity of the X-ray luminescence spectra with maxima at 2.8 eV (1) and 3.1 eV (2) of KCl-Na crystals on the sodium concentration at room temperature.

It also follows from Figure 4 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 radiative annihilation of the exciton-like formation.

It is known that the accumulation of impurities in the form of aggregates is thermally dissociated by quenching crystals by calcination to high temperatures, at least not lower than half of the AHC melting point [34]. However, the radiation with a maximum at 3.1 eV, which is interpreted by the radiative relaxation of an exciton-like formation in the field of paired sodium ions, after quenching 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 formation of NaCl phase, the melting point of which being higher (801°C) than the main KCl

#### lattice (770°*C*).

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 hightemperature (300 K) ignition of the intensity of exciton-like formation 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 formation 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 formation is located at two anionic nodes of the lattice, which has the structure  $X_2^-$  of a molecule oriented along the <110> crystallographic direction [1, 3, 4].

This circumstance creates a unique opportunity for a targeted impact on the core of an exciton-like formation 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 5) 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 5), and with <110> - 3.5 times.

These differences are logically compatible with the AHC deformation theory [34] 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 formation.

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

Figure 5 (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 of the 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 formation.

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 (2*a*) in a crystal KCl [2, 4]. Where *a* is the lattice constant of KCl 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 radiative relaxation of an exciton-like formation in the field of single and paired sodium ions, respectively.



Figure 5. X-ray luminescence spectra of KCl-Na crystals (100 ppm) at 300 K in the absence of deformation (1), with deformation ( $\epsilon = 0.9\%$ ) along crystallographic directions <100> (2) and <110> (3).

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 –  $16 \div 20a$ ,  $8 \div 10a$  and  $5 \div 7a$ . 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 5). 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 quenching (500-600°*C*), an increase in the concentration of Na ions (for example, 1000 ppm), as well as under the influence of uniaxial deformation along the crystallographic directions (<100> and <110>).

Lowering the 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 formation increases, the radiative relaxation of which occurs in the field of local, thermoelastic and uniaxial deformation with intense luminescence.

# Acknowledgments

This research work has been funded within the framework of the grant funding project of the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan (IRN AP08855672).

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