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1994 J. Phys.: Condens. Matter 6 2357

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Relaxation of excitons in wide-gap CsCl crystals

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Received 13 July 1993, in final form 18 October 1993

Abstract. Optical characteristics of free and self-trapped excitons are investigated in simple cubic CsCl crystals of high purity. The reflection, photoluminescence, cathodoluminescence and x-ray luminescence spectra, as well as the excitation spectra for intrinsic, impurity and near-defect emissions and spectra of the anion Frenkel defect creation by radiation are measured in CsCl crystals at 5–80 K in the spectral region of 2 to 10 eV. A weak emission at 8.3 eV and π -emission of self-trapped excitons (2.82 eV) are detected in CsCl at 5 K. The energy gap is estimated as $E_g = 8.4$ eV at 5 K. Some peculiarities of the radiative and non-radiative decay of excitons in sc CsCl in comparison with those of fcc KCl and RbCl crystals are discussed.

1. Introduction

Excitonic, electron–hole and ionic processes have been studied in detail in many wide-gap crystals with the face-centred-cubic structure B1 [1–4]. Seventeen out of twenty alkali halides (AHs) have such a structure, whereas crystals of CsCl, CsBr and CsI have the simple-cubic (SC)-lattice structure B2 under standard conditions and have not as yet been sufficiently investigated (especially CsCl).

For the growth of CsCl crystal from the melt it is necessary to pass through the region of the phase transition B1 \rightarrow B2 (\simeq 740 K). At this temperature the volume of a crystal decreases by 17 per cent and the coordination number changes from 6 to 8 [5]. The presence of the phase transition impedes the growth of SC CsCl crystals of high perfection and thus their availability for investigation.

The CsCl crystal is a rare system where the physical cation radius (0.188 nm) is larger than the anion radius (0.171 nm) [6]. Satpathy has calculated the electronic band structure of SC CsCl, taking into account relativistic effects [7]. Absorption spectra for CsCl thin films [8], reflection spectra for CsCl single crystals [9], and evaporation layers [10] and spectra for electron energy losses in CsCl [11] have been measured. Self-trapped holes (V_K centres) in CsCl have been examined in [12]. Formation energies of anion and cation vacancies, as well as Frenkel defects (FDs), have been calculated in SC [13]. Cross-luminescence has been detected in CsCl and CsBr crystals [14–16].

On the other hand, characteristics of free excitons (FEs) and self-trapped excitons (STEs), and their radiative and non-radiative decay have not been investigated. There are no experimental data on interstitial halogen atoms and ions, which in FCC AHs are called H and I centres.

High-purity SC CsCl crystals and CsCl crystals doped with Na^+ , Rb^+ , Ba^{2+} , In^+ , Tl^+ , Br^- and I^- have recently been grown in our laboratory [17]. Low-temperature energy

transfer by excitons to luminescence impurity ions has been investigated in these doped CsCl crystals [18].

The aim of the present study is to detect the intrinsic luminescence of free and self-trapped excitons and to analyse the possibility of non-radiative STE decay with the formation of anion Frenkel defects in a high-purity CsCl crystal. Starting with the measurement of the reflection spectrum of CsCl at 5 K we tried to investigate a complicated process for FE relaxation into the self-trapped state and the subsequent radiative or non-radiative decay of STES.

2. Experimental details

A single crystal of CsCl was grown by the Stockbarger method from CsCl salt, which was preliminarily dried and refined from Br^- and I^- impurity ions by a CsCl melt treatment in Cl_2 gas flow, and additionally refined by manyfold (40 and 60 zone) recrystallization from the melt [17]. The contents of two-valent metals and OH^- in our best samples of CsCl was less than 10^{-2} ppm, Na^+ and I^- less than 1 ppm and the contents of Br^- was on the level of a few ppm.

The experimental arrangement for the photoluminescence investigation at 5 to 300 K is described in [17, 19]. The crystals were placed in a helium cryostat with MgF_2 windows and exposed to a deuterium lamp (MgF_2 window) through a vacuum monochromator (concave grating with $R = 0.5$ m and dispersion 0.132 nm mm^{-1}). The luminescence (2 to 6 eV) was recorded through a MDR-23 monochromator (dispersion 0.13 nm mm^{-1}) by a photomultiplier operating in the photon counting mode. Necessary corrections were made in the emission spectra. Excitation spectra were measured at equal quantum intensities of the excitation falling onto the crystal. Cathodoluminescence of CsCl was investigated at 5 to 300 K by means of a double-vacuum monochromator, employing a double-Johnson-Onaka system (dispersion 0.167 nm mm^{-1}). The luminescence was excited by an electron beam (6 keV, $0.1\text{--}150 \mu\text{A cm}^{-2}$) and detected by a solar-blind photomultiplier in the photon counting regime.

3. Experimental results

3.1. Reflection spectra

The reflection spectrum is an important characteristic of a crystal, which contains information on unrelaxed electronic excitations created by photons. One of us (IK) measured the reflection spectrum of an unpurified CsCl crystal at 10 K, using synchrotron radiation (DESY, Hamburg). The experimental arrangement was similar to that described in [16]. A non-oriented CsCl sample was exposed to synchrotron radiation through a grating monochromator ($R = 2$ m, $1200 \text{ lines mm}^{-1}$). Reflectance measurements were made at the incidence angle of 11° . The reflection spectrum is presented in figure 1. We obtained an analogous reflection spectrum at 8 K in the spectral region 7.5–8.5 eV for the (100) plane of a high-purity CsCl crystal.

The positions of maxima in the absorption spectrum of a CsCl thin film at 10 K [8], in the reflection spectrum of a CsCl evaporation layer at 22 K [10], and in our reflection spectra of unpurified CsCl at 10 K and a high-purity CsCl crystal at 8 K are collected in table 1.

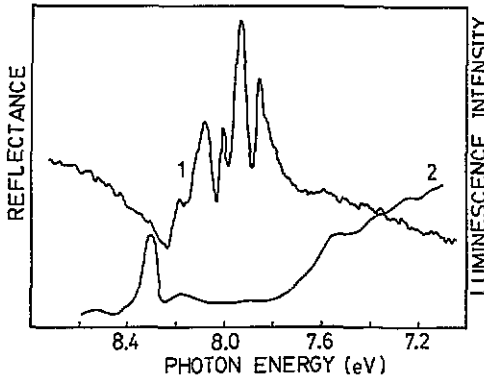


Figure 1. The reflection spectrum of a CsCl crystal measured at 10 K with a resolution of 0.1 nm (curve 1). The emission spectrum measured on the excitation of a CsCl crystal by a 6 keV electron beam at 10 K (curve 2).

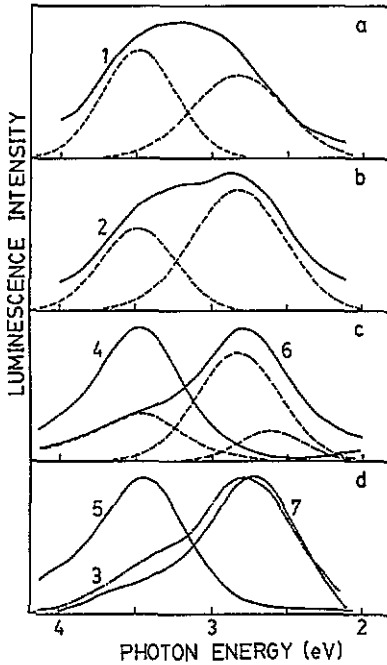


Figure 2. The luminescence spectra of a CsCl crystal on excitation by photons of 8.00 ± 0.03 (curves 1 to 3), 7.40 ± 0.03 (curves 4 and 5) and 8.95 ± 0.09 eV (curves 6 and 7). The spectra are measured at 5 K (curves 1, 4 and 6), 50 K (curve 2) and 80 K (curves 3, 5 and 7). Broken curves show the main components (Gaussians) of the corresponding emissions.

The two peaks at 7.85 and 8.08 eV are interpreted in [10] as the spin-orbit splitting of p^5s excitons and the peaks at 7.93, 8.01 and 8.19 eV correspond to the formation of p^5d excitons. There is a deep minimum at 8.23 eV in the reflection spectrum of CsCl. According to Creuzburg [11], the peak at this energy in the spectrum of electron energy losses corresponds to the formation of longitudinal excitons in CsCl.

Table 1. Positions of the absorption (E^A) and reflection (E^R) maxima (in eV).

E^A	7.84	7.94	8.01	8.08	8.20	[8]	10
E^R	7.85	7.93	8.01	8.08	8.19	[10]	22
E^R	7.860	7.936	8.008	8.086	8.184		10
E^R	7.855	7.935	8.005	8.085	8.187		8
p^5s		p^5d	p^5d	p^5s	p^5d	[10]	T (K)

Our investigations of the creation spectra of self-trapped holes in CsCl crystals (see section 3.4) show that the value for the energy gap is $E_g = 8.35$ eV at 80 K and $E_g = 8.40$ eV at 5 K. The binding energy for p^5s excitons at 5 K in CsCl $E_B = E_g - E^R = 0.5$ eV, is significantly lower than in NaCl, KCl and RbCl (0.78, 0.92 and 0.96 eV, respectively).

3.2. Low-temperature cathodoluminescence

We made an attempt to detect the edge emission due to the annihilation of FEs on the excitation of a CsCl crystal by a 6 keV electron beam at 10 K. We have earlier revealed the emission of FEs in a number of wide-gap ionic crystals (NaI, KI, RbI, CsI, NaBr, CsBr, BeO, MgO, etc) [20, 21].

For the first time we have succeeded in detecting in chlorides a weak narrow emission at 8.3 eV with the apparatus bandwidth (≈ 0.05 eV), and also a weak continuous emission, which ranges in CsCl ($h\nu < 7.7$ eV) from the exciton absorption band to lower energies (see curve 2 in figure 1). The intensity of the narrow emission is 2.5×10^3 times lower than the peak intensity of the simultaneously measured cross-luminescence at 5.2 eV. The 8.3 eV emission was rapidly quenched by irradiating the crystal by an electron beam, creating radiation defects. Irradiation of CsCl at 10 K by an electron beam leads to the formation of longitudinal excitons with energies of 8.2 to 8.3 eV. With the participation of phonons these excitons can populate the upper polaritonic branch. In this way we can interpret a weak emission band in CsCl at 8.3 eV. The broad emission at $h\nu < 7.7$ eV can be ascribed to a hot luminescence of STE (see, for example, [21, 22]).

3.3. Low-temperature photoluminescence

Figure 2 represents luminescence spectra of a high-purity CsCl crystal at 5, 50 and 80 K in the case of excitation by photons of 8.00 ± 0.03 , 8.95 ± 0.06 and 7.40 ± 0.03 eV, which selectively create excitons, separated electrons and holes, or near-impurity-localized electronic excitations. The main components of these emissions approximated by Gaussians are shown by broken curves. Some components responsible for 10 per cent of the total emission are not shown in the figure.

A short-wavelength emission component (with maximum $E^I = 3.48$ eV and half-width $\delta^I = 0.58$ eV) can be effectively excited out of the region of crystal intrinsic absorption by 7.4 eV photons. Curve 2 in figure 3 shows the excitation spectrum of 3.60 ± 0.03 eV emission. The quantum efficiency of this emission in the region of $h\nu > 7.80$ eV is two orders of magnitude lower than in the region of impurity and defect absorption ($h\nu < 7.7$ eV). On the contrary, the emission component $E^I = 2.82$ eV ($\delta^I = 0.70$ eV) can be effectively excited on selective exciton formation and in the region of band-to-band transitions (see figures 2 and 3). The intensity of the 3.5 eV emission is sharply increased in the CsCl crystal not purified from the Br^- impurity ions as well as in thermally quenched or deformed crystals (see below).

We interpret the 2.82 eV emission as STE luminescence in the CsCl crystal. The quantum efficiency of this emission on the optical generation of excitons by 7.8 to 8.3 eV photons

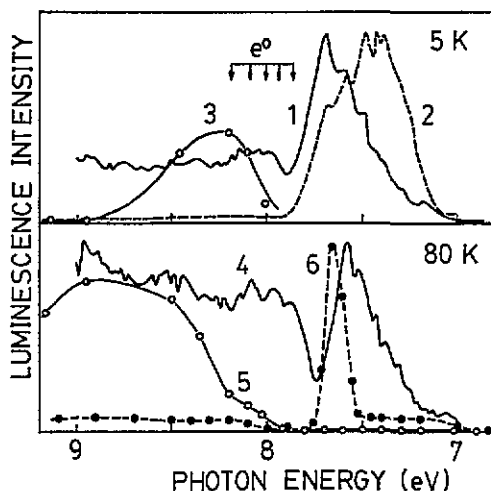


Figure 3. The excitation spectra of the 2.60 ± 0.04 (curves 1 and 4) and 3.60 ± 0.04 eV (curve 2) emission of a CsCl crystal. The creation spectra of anion vacancies (curve 3), self-trapped holes (the TSL peak at 185 K, curve 5), and BrCl^- molecules (the TSL peak at 245 K, curve 6) by VUV radiation in a CsCl crystal. The spectra are measured at 5 K (curves 1 to 3) and 80 K (curves 4 to 6).

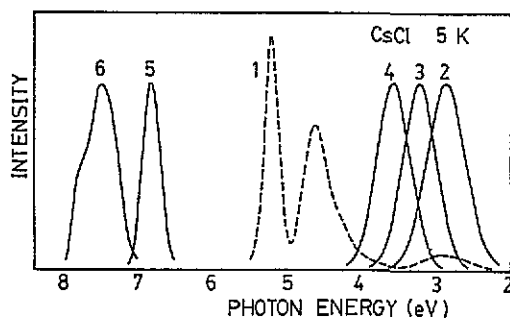


Figure 4. The luminescence spectra of a CsCl crystal at 5 K: curve 1, crossluminescence; curve 2, STE luminescence; luminescence of halogen ions surrounding an anion vacancy (curve 3); or divacancy (curve 4). The excitation spectra for luminescence of 3.2 (curve 5) and 3.55 eV (curve 6) at 5 K.

at 5 K is about 0.05. The duration of STE emission is estimated to be 10^{-6} – 10^{-3} s, using excitation by impulse x-rays. The 2.82 eV emission can be excited in the total volume of CsCl on the excitation of the crystal by laser emission in the regime of two-photon absorption ($3.85 \times 2 = 7.9$ eV).

The intensity of impurity-defect emission of ≈ 3.5 eV at 5 K is higher than that of STE luminescence, even in a high-purity CsCl crystal. According to [18] the run of FE before self-trapping in CsCl at 5 K is about 350 lattice constants and the excitons interact with defects and impurity centres. The integral intensity ratio of the 2.8 and 3.5 eV emission bands increases twice (see figure 2(a) and (b)) after heating the crystal from 5 to 80 K. A previous investigation of ours has shown that the run of FEs before self-trapping at 80 K

is about twice as small as that at 5 K [18]. So, the energy states of FEs and STEs are separated in CsCl (as well as in iodides and bromides [20, 21]) by an activation barrier. The excitations of STE emission occurs mainly in the energy region with $h\nu > 7/7$ eV, i.e. in the region of intrinsic absorption of a crystal (curve 4 in figure 3).

The 2.82 eV emission is dominant even at 5 K in the case of excitation by 8.95 eV photons, creating separated electrons and holes. The run of holes before self-trapping in CsCl, as well as other AHS, is smaller than the run of FEs. A long-lived emission component ($E^I = 2.60$ eV, $\delta^I = 0.52$ eV) arises on the excitation of CsCl at 5 K in the region of band-to-band transitions (figure 2(c)). Our experimental data show that this emission is connected with the radiative electron tunnelling transfer between spatially close F and V_K centres.

3.4. Creation spectra of self-trapped holes

Irradiation of a CsCl crystal at 5 K by x-rays or VUV radiation leads to the appearance of long-duration tunnelling luminescence of F, V_K pairs as well as of the peaks of thermally stimulated luminescence (TSL) at 17, 19, 23, 30, 38, 68, 85, 110, 160, 185, 245, 370 and 450 K (at a heating rate of 0.05 K s^{-1}). Our data on the annealing of a radiation-induced absorption band of V_K centres ($E^K = 3.25$ eV) show that the TSL peak at 185 K is connected with the hopping motion of V_K centres, i.e. Cl_2^- molecules situated at two anion sites. The intensity of the TSL peak at 245 K increases sharply in CsCl doped with Br^- impurity ions. This peak is connected with the dissociation of $BrCl^-$ molecules.

Figure 3 (curves 5 and 6) shows the creation spectra of TSL peaks at 185 and 245 K in high-purity CsCl and CsCl-Br crystals at 80 K. The efficiency of self-trapped hole creation is high in the region of band-to-band transitions ($h\nu \geq 8.35$ eV) and is by two orders of magnitude lower in the region of optical formation of p^5s and p^5d excitons (7.75–8.25 eV). The photons with $h\nu < 7.75$ eV do not create V_K centres. This last result is in good agreement with the relativistic calculation of the electronic band structure of CsCl, that predicts a direct band gap at the Γ point [7]. The TSL peak at 245 K in CsCl-Br can be effectively created in a narrow energy region of 7.6 to 7.7 eV, due to the partial ionization of chlorine ions situated near impurity bromide ions. As a result, $BrCl^-$ molecules arise.

3.5. Luminescence of crystals with defects

Curve 1 in figure 4 shows a luminescence spectrum of high-purity CsCl measured on crystal excitation by x-rays at 5 K. Crystal excitation by photons of 6 to 13 eV does not lead to the appearance of luminescence with maxima at 4.6 and 5.2 eV. According to [16], the excitation threshold of these emission bands coincides with the beginning of photo-ionization of $5pCs^+$. Emission arises due to the radiative recombination of electrons from the halide valence band with holes from the core cation band, created during irradiation of the crystal (so-called cross-luminescence).

Intensive 3.2 eV luminescence stimulated by photons of 6.8 eV arises in a CsCl crystal after x-irradiation at 5 K (curve 3 in figure 4). Taking into account the analogous effect in FCC AHS [19, 22], we interpret this experimental result as the radiational creation of anion vacancies and interstitial chlorine ions (α , I pairs) in the CsCl crystal. The α absorption band (curve 5 in figure 4) is connected to the absorption of halide ions near an anion vacancy and is situated in CsCl in the same spectral region as in other chlorides ($E^K = 6.8$ eV, $\delta^K = 0.28$ eV). The thermal annealing of the main part of α centres in CsCl occurs due to the recombination of mobile interstitial ions with anion vacancies and is accompanied by TSL peaks at 17 and 19 K.

X-irradiation of the CsCl crystal at 5 K leads to the appearance of a radiation-induced absorption band at 1.95 to 2.25 eV, i.e. a F absorption band. Thermal annealing of the F absorption band occurs at 20 to 40 K and is accompanied by TSL peaks at 23, 30 and 38 K. The annealing process is connected with the hopping diffusion of interstitial halide atoms (H centres) and their recombination with F centres.

The formation of non-equilibrium pairs of anion and cation vacancies (divacancies) after the fast thermal quenching of a crystal from near-melting temperature to 77 K has been detected in KCl and KBr [22]. We carried out an analogous experiment. A high-purity CsCl crystal was slowly heated up to 820 K, quickly cooled down to liquid nitrogen temperature and then replaced into a helium cryostat and cooled down to 5 K. Thermal quenching led to the increase of the 3.55 eV emission ($\delta^I = 0.50$ eV) intensity (curve 4 in figure 4) several-fold. The excitation spectrum of this emission characterizes the absorption of the halide ions surrounding a divacancy, which is shown in figure 4 (curve 6).

3.6. Spectra of Frenkel defect creation by VUV radiation

There is no doubt that α , I and F,H pairs of FDs can be created in a CsCl crystal at 5 K, not only on x-irradiation, but also on optical generation of the excitons by 7.8 to 8.3 eV photons. We have investigated the process of radiational formation of FDs in CsCl by means of photo- or thermally stimulated luminescence methods. The CsCl crystal was irradiated at 5 K by an equal number of photons of various energies. The intensity of the 6.8 eV photon-stimulated α -luminescence (3.2 eV) was taken as the number of α ,I pairs. Curve 3 in figure 3 presents the creation spectrum of α ,I pairs measured by the procedure mentioned above. The annealing of α ,I pairs occurs in CsCl in the region of 15 to 20 K, and is accompanied by TSL peaks at 17 and 19 K. Similar to the KCl crystal [19,3], the creation of α , I pairs in CsCl occurs mainly on the non-radiative decay of excitons, and is at least an order of magnitude lower on the non-radiative recombination of electrons with relaxed V_K centres.

The spectrum of F,H pair creation by VUV radiation at 5 K has been measured in [17]. The efficiency of F,H-pair formation is high in the wide region of intrinsic absorption (7.8 to 10 eV), where VUV radiation selectively creates excitons and separated electrons and holes.

4. Discussion of experimental results

Models of some of the defects mentioned above are schematically shown in figure 5. The physical radii for anions and cations after Schannon [6] are used for the construction of the (110) plane of the CsCl crystal. It is obvious without any theoretical calculation that the orientation of self-trapped holes along $\langle 100 \rangle$ directions is energetically more profitable than along $\langle 110 \rangle$. This speculation is confirmed by the ESR investigations of V_K centres in the CsCl crystal [12].

There can be three types of STE in AHS [4,23]:

(i) STE_{on} , an on-centre STE can be described as an electron and Cl_2^- molecule symmetrically situated at two anion sites, i.e. the V_K +electron model;

(ii) STE_{off}^w , a weak off-centre STE corresponds to a slight shift of the electron and the hole component of the exciton relative to each other and to anion sites;

(iii) STE_{off}^s , a strong off-centre STE corresponds to the displacement of an exciton hole component into a one-anion site with one electron being localized in another.

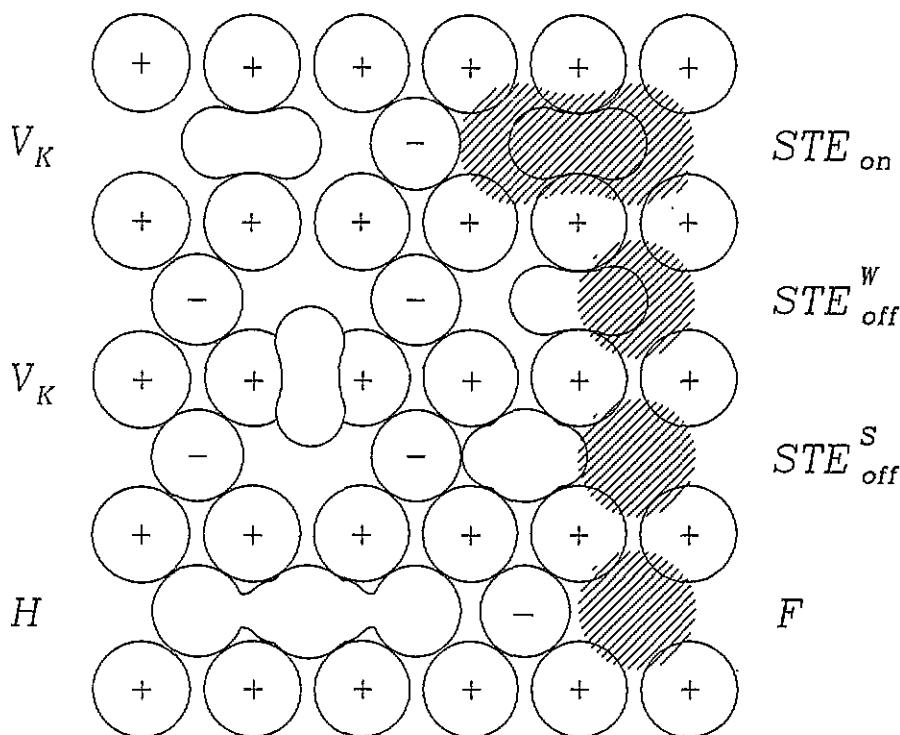


Figure 5. Models of self-trapped holes (V_K centres), H and F centres and three types of self-trapped excitons (STEs). The physical radii for Cs^+ and Cl^- [6] are used for the construction of the (110) plane of the CsCl crystal. The area of spreading of the electron component of the centre is shaded.

The third type of STE is particularly profitable for the exciton decay, with the formation of spatially separated F and H centres (see figure 5).

Values for the relative Stokes shift ($\mu = (E^k - E^l)/E^k$) for π -emission of the three STE configurations mentioned above are significantly different. NaI and NaBr crystals are systems with the STE_{on} configuration, with $\mu \simeq 0.25$ – 0.35 for π - and σ -emissions of STEs. Both σ - and π -emissions of STEs are observed in KI and RbI crystals. The STE_{off}^W configuration is typical of these crystals with larger values for μ of π -emissions (0.43 and 0.48 for KI and RbI, respectively). The σ -emission is missing in KCl and RbCl crystals, with large values for μ of π -emissions (0.71 and 0.73). The case of the STE_{off}^S configuration is realized in KCl and RbCl.

The π -emission of STEs in SC CsCl can be characterized by the value $\mu = 0.64$ ($E^k = 7.85$ eV and $E^l = 2.82$ eV) and can be attributed to STE_{off}^S . Similar to KCl and RbCl, the quantum yield of π -emission in CsCl is less than 0.05 at 5 K. Up to now we have failed to detect the σ -emission of STEs in CsCl.

In our opinion the non-radiative transitions in CsCl occur already during the vibrational relaxation of STEs. This relaxation process is slow. In CsCl the frequency of intramolecular vibrations for a hole component of STEs ($\simeq 240$ cm^{-1}) is higher than the threshold frequency of lattice vibrations (164 cm^{-1}), i.e. STEs have local vibrational modes.

There are two types of non-radiative transitions on STE decay in solids. The excess of vibrational energy can be transformed into small displacements of a large number of ions. That means energy conversion into heat. The second non-radiative channel of STE decay is

connected with energy transformation into large displacements of single ions that result in the creation of FDs [3].

The non-radiative decay of STEs in CsCl is mainly connected with the formation of FDs. In CsCl and KCl crystals we have compared the efficiencies of α -centre creation on the decay of the excitons created by 8.0 or 7.75 eV photons at 5 K, respectively. The efficiencies of anion vacancy creation differ from each other in CsCl and KCl by less than 25 per cent. Approximately 5 per cent of optically formed excitons decay with the creation of α ,I pairs in the KCl crystal at 5 K [3]. The decay of STEs in KCl leads to the creation of F,H pairs, part of which transform into α ,I pairs after tunnelling recharge [3, 19]. We suppose that the same scheme of STE decay is realized in the CsCl crystal as well. STE decay with F,H-pair formation occurs in CsCl mainly during a slow vibrational relaxation, i.e. before the equilibrium of STEs with the surrounding lattice is reached.

The investigation of the creation spectra of FDs by VUV radiation shows that even the optical generation of excitons with $n = 1$ leads to the effective creation of F,H and α ,I pairs in CsCl at 5 K. The crowdion configuration of the H centre in CsCl (see figure 5) should facilitate an effective spatial separation of F and H centres, even at 5 K.

5. Conclusion

We have managed to follow the complicated many-stage process of exciton relaxation in the SC CsCl crystal. Free excitons are transformed into a self-trapped state through an activation barrier. During slow vibrational relaxation part of the STEs decay non-radiatively, with the formation of F centres and mobile H crowdions. After vibrational relaxation on a small part of the STEs causes a typical π -emission. Anion vacancies and interstitial halogen ions arise due to the tunnelling recharge of a part of the primary F,H pairs.

Acknowledgment

One of us (AL) would like to point out that this work was partly supported by a Soros Foundation Grant E7, awarded by the American Physical Society.

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