

Mechanisms of radiation defect formation in KCl crystals under the influence of local and plastic deformation

K. Shunkeyev, Z. Aimaganbetova, L. Myasnikova^{*}, A. Maratova, Zh. Ubaev

K. Zhubanov Aktoke Regional University, Moldagulova av. 34, 030000 Aktoke, Kazakhstan

ARTICLE INFO

Keywords:

KCl crystals
Plastic deformation
Radiation defects
Vacancy dipole complexes
Optical absorption spectra

ABSTRACT

This work proposes an original approach to the study of trihalide centers in a KCl matrix in the field of local and plastic deformation. Local lattice deformation is carried out by purposeful doping of the KCl crystal with light homologous cations, as well as bivalent impurities, which create dipole strontium complexes to maintain the electroneutrality of the lattice. Plastic deformation, which creates paired vacancy defects in the lattice, divacancies and vacancy quartets, is realized at room temperature within the degree of relative uniaxial deformation of 3–5%. After subsequent cooling down to 80 K the deformed crystal was additionally irradiated with X-rays. The absorption spectra of the crystals subjected to two types of deformation and additional isodose irradiation by X-rays were recorded in a special cryostat at 80 K. The spectral characteristics and formation efficiency of V-type centers, lithium- and strontium-containing centers in the field of pre-irradiation dipole complexes have been considered.

1. Introduction

The knowledge on radiation processes in functional materials is important for designing devices and equipment that operate in the radiation environment. The creation of radiation damage in materials significantly reduces the operation service life of a certain category of equipment. The features of changes in the physical and chemical properties of materials are determined by both the ambient temperature and the degree of compression of the object. The model objects in the study of these processes are simple in structure alkali halide crystals (AHCs) (see, e.g., [1–6]).

In AHCs, one of the relaxation channels of electronic excitations is their nonradiative annihilation (decay) at low temperatures (4.2 K), leading to the creation of pairs of charged and neutral primary point defects, *α-I* and *F-H* Frenkel pairs: an anion vacancy (the co-called *α* center) and an interstitial halogen ion (*I* center) as well as an interstitial halogen atom (*H* center or a dihalide molecule located at one anion site, $(X_2^-)_a$) and the *F* center (an electron localized in the anion vacancy field) [1–3,5–10]. As the crystal temperature increases, *I* and *H* centers become mobile, in KCl around 30 K and 40 K, respectively (see, e.g., [9,10]). Therefore, the efficiency of radiation defect formation in AHCs at temperatures when the *H* centers are mobile (in KCl, at *T* greater than 40 K) is determined by the efficiency of their stabilization. In AHCs, the

high-temperature stabilization of *H* centers, which are complementary radiation defects to the *F* centers, occurs due to the interaction of two *H* centers resulting in the formation of a trihalide X_3^- center by the following reaction (see, [1–3] and references there in):



As a result of this reaction, a linear trihalide center is formed in the lattice, occupying one cation (*c*) and two anion (*a*) lattice sites [1–2]. Note that this molecule is adjacent to a dipole of alkali (*M*) and halide ion interstitials (marked by *i* subscript). The reaction (1) can be carried out with higher efficiency if the association of the *H* centers occurs near an impurity cation with a smaller radius with respect to the main cation (KCl:Li) or if a divacancy ($v_a^+ v_c^-$ – adjacent anion and cation vacancies) is already present in the sample (for example, induced by applied plastic deformation) and participates in the crystal lattice rearrangement, formation of the $(X_3^-)_{aca}$ center.

In this regard, it is relevant to study the formation efficiency of X_3^- centers in the field of already existing vacancy- and dipole-type defects. The mutual interaction of intrinsic and impurity point defects of the crystal lattice, which leads to the formation of dipole complexes, significantly affects the mechanical and electrical properties of AHCs [9–15]. Therefore, it is very important to study the formation and interaction of dipole complexes of vacancy origin, since they are

^{*} Corresponding author.

E-mail address: myasnikova_ln@mail.ru (L. Myasnikova).

<https://doi.org/10.1016/j.nimb.2021.10.010>

Received 28 June 2021; Received in revised form 6 October 2021; Accepted 13 October 2021

Available online 23 October 2021

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responsible for dielectric losses.

Previously [16–19], the possibilities of the formation of vacancy-containing dipole complexes by plastic deformation as well as via the introduction into the KCl crystal of light cations-homologs (Li) or divalent cation impurities, which cause the appearance of additional cation vacancies for charge compensation, were considered. The recorded thermally stimulated depolarization currents unambiguously proved the existence of vacancy dipole complexes – $v_a^+v_c^-$, $Li_{ic}^+v_c^-$ and $Sr_c^+v_c^-$.

In [19], the results of the Li impurity influence on the radiation formation of Cl_3^- and Br_3^- centers in KCl and KBr crystals in the temperature range of 80–300 K have been demonstrated. It has been shown that the rise in the impurity concentration and irradiation temperature to 300 K increases the efficiency of X_3^- center creation by more than an order of magnitude, compared to pure crystals. According to the authors of [19], this happens because a small-size Li^+ cation moves into an interstice (thus causing the formation of a cation vacancy being the seed for an X_3^- center). Therefore, the Li impurity ion will be marked as Li_{ic}^+ .

It is known that complex trihalide defects are formed by the interaction of two mobile halogen atoms in regular lattice region [1–3,20–22]. In this regard, the registered vacancy dipole complexes should significantly increase the efficiency of radiation defect formation, since on their basis it is very energetically advantageous to form a halogen defect that occupies one cation and two anion regular sites of the crystal lattice. Therefore, the logical continuation of the research is to analyze the defect formation efficiency in the field of vacancy dipole complexes. For this purpose, the optical densities (absorption bands) related to trihalide radiation-induced centers in plastically deformed KCl, KBr and KI crystals were estimated and compared to those in case of the absence of deformation.

In ionic AHCs, for charge neutrality reasons, anion and cation vacancies are formed in equal quantities and often considered as vacancy pairs, divacancies. Most of these vacancy pairs are a part of larger or smaller clusters. This conclusion was made on the basis of the data obtained by measuring the thermal conductivity of plastically deformed LiF crystals [23–25]. It should be noted that the formation of vacancy defects occurs due to the interaction of dislocations created by plastic deformation in X-ray-irradiated AHCs [26–27].

Experimental results on the formation of electronic excitations in the field of vacancy-type defects also indicate that plastic deformation creates paired vacancy defects – divacancies – ($v_a^+v_c^-$) and vacancy quartets – $2(v_a^+v_c^-)$. The possibility of using the results on the deformation-induced changes in thermal conductivity is based on the difference in the efficiency of phonon scattering by single vacancies, pairs of vacancies (divacancies) and their clusters. According to [28–29], single vacancies are the most strongly scattered centers, while their complexes do not change the thermal conductivity at all.

The present paper is a reasonable continuation of our previous studies [19,22,30–32] and is carried out in order to obtain new information about the effect of applied plastic deformation as well as the doping with Li^+ and Sr^{2+} impurity cations on the radiation defect creation in KCl crystals.

On the basis of absorption spectroscopy, we obtained original results on the efficiency of the formation of halogen radiation defects (trihalide centers) in KCl, KCl:Li, and KCl:Sr crystals in the field of pre-irradiation vacancy-dipole defects – ($v_a^+v_c^-$, $2(v_a^+v_c^-)$), formed via plastic deformation (up to 4%) and due to the presence of impurity dipole defects ($Li_{ic}^+v_c^-$, $Sr_c^+v_c^-$).

2. Experimental

The absorption spectra of X-ray irradiated crystals were measured at low temperature (80 K) by the Nicolet Evolution 300 spectrophotometer using a specially designed cryostat [33] equipped with an appropriate beryllium window for X-ray irradiation and two LiF windows optically transparent in the spectral range up to 6.5 eV for recording the photons

that passed through the crystal.

The crystal holder is additionally equipped with quartz shielding, which ensures the purity of the crystal surface. The structureless background absorption spectrum of crystals measured before X-ray irradiation confirms the purity of the crystal surface. The level of technical vacuum in the cryostat is 10^{-3} Pa, which is reached firstly by pumping with a pre-vacuum pump, and then with an oil-free absorption pump.

The crystals were irradiated with X-rays using the RUP-120 device operating in the 5 mA and 120 kV mode (W anti-cathode).

The experiments were carried out in the following sequence: the crystal was plastically deformed ($\varepsilon = 4\text{--}5\%$) by uniaxial compression at room temperature (RT), then cooled down to 80 K and irradiated with X-ray radiation at this temperature; finally, the absorption spectra was recorded.

Highly-pure KCl crystals were grown by means of a sixty-fold zone melting in the laboratory of the Institute of Physics of the University of Tartu [15]. The KCl-Li and KCl-Sr crystals were grown by the Stockbarger method from zone-purified starting materials with the addition of vacuum-dried salts LiCl and $SrCl_2$. The plates with a thickness of 0.5 ± 0.05 mm were used for measuring the absorption spectra of the irradiated crystals.

3. Main results

As already mentioned in the Introduction, the presence of the Li^+ ions in a KCl crystal significantly affects the efficiency of radiation creation of Cl_3^- centers. In this case, an increase in the concentration of Li^+ leads to enhanced by more than an order of magnitude efficiency of the Cl_3^- center creation, in comparison with a pure KCl crystal [20]. However, the joint effect of the presence of Li^+ ions and applied plastic deformation on radiation damage of KCl has not been studied.

3.1. Plastic deformation of KCl crystals

Fig. 1 presents optical absorption spectra measured at 80 K for two KCl crystals, in the absence and after plastic deformation at RT, both exposed to isodose irradiation by X-rays at 80 K. In a plastically deformed and additionally irradiated KCl crystal, the absorption band at 5.3 eV related to $V_2 \equiv (Cl_3^-)_{aca}$ centers [1–3] is clearly seen (curve 2). On the other hand, there are no additional absorption around 6.4 eV characteristic of the more complex $(Cl_3^-)_{aca} K_i^+ Cl_i^-$ center, which contains also an interstitial halide ion (just I center is responsible for absorption band peaked at 6.4 eV). This indicates that $V_2 \equiv (Cl_3^-)_{aca}$ centers are predominantly created by X-rays in a plastically deformed KCl crystal (curve 2). The second arrow indicates the position of the maximum of the possible absorption band related to the interstitial chlorine ions. It should be noted that the crystal was plastically deformed to 3.0 % at RT, while subsequent X-ray irradiation and registration of the absorption of $(Cl_3^-)_{aca}$ centers were carried out at 80 K.

The radiation-induced absorption spectrum is rather similar in a nondeformed sample (Fig. 1, curve 1), but the creation efficiency (concentration) of the $(Cl_3^-)_{aca}$ centers is by more than 3 times lower than in case of the plastically deformed crystal. Note that the $(Cl_3^-)_{aca}$ center includes (is located at) two anion and one cation vacancies.

From these experimental results, it can be assumed that the increase in the efficiency of radiation creation of $V_2 \equiv (Cl_3^-)_{aca}$ and $V_3 \equiv ([Cl_2]_2)_{acac}$ centers is probably due to an increase in the concentration of pre-irradiation divacancies $v_a^+v_c^-$ and vacancy quartets, $2(v_a^+v_c^-)$ in the lattice. The interaction of two interstitial halogen atoms (H centers) in the divacancy field (near $v_a^+v_c^-$) with the formation of the X_3^- center is schematically demonstrated in the right corner of Fig. 1.

Fig. 2 shows the results of the similar experiment for nondeformed/plastically deformed (3.0 %, RT) KCl, but isodose X-ray irradiation of both crystals was performed at RT and the absorption spectra were measured again at 80 K. It is clearly seen that high-temperature irradiation leads to the appearance of an additional absorption band peaked at

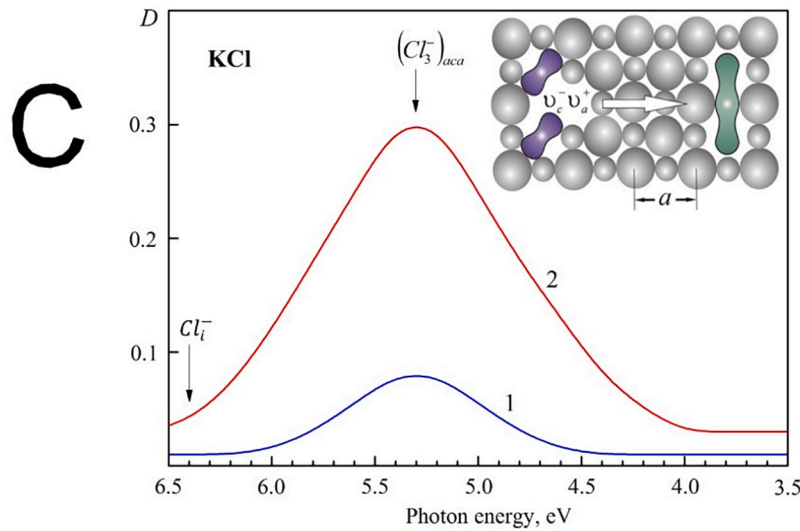


Fig. 1. Absorption spectra measured for two KCl crystals after isodose irradiation by X-rays (90 min) at 80 K. The spectra were measured at 80 K for a nondeformed sample (curve 1) and a plastically deformed one ($\epsilon = 3.0\%$, curve 2). The scheme of interaction of two mobile H centers in the field of a $v_a^+v_c^-$ divacancy resulting in the creation of a $(Cl_3^-)_{aca}$ trihalide center is shown in the right upper corner.

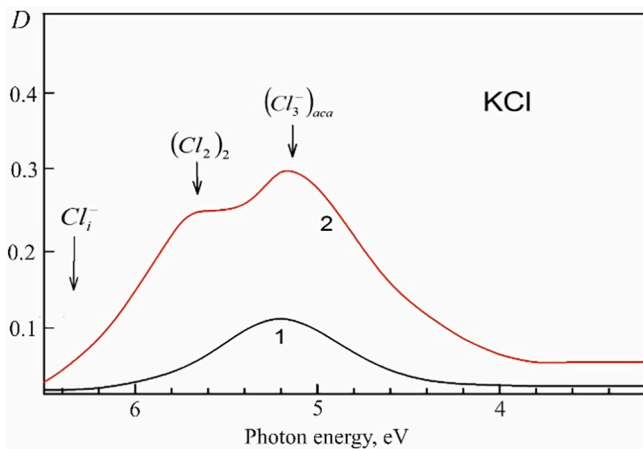


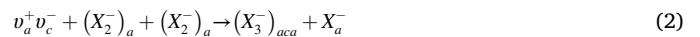
Fig. 2. Absorption spectra measured for two KCl crystals after isodose irradiation by X-rays (90 min) at 300 K. The spectra were measured at 80 K for a nondeformed sample (curve 1) and a plastically deformed one ($\epsilon = 3.0\%$, curve 2).

5.8 eV in the plastically deformed crystal (curve 2). This additional band is connected with more complex radiation defects than single $(Cl_3^-)_{aca}$, the so-called $V_3 \equiv [(Cl_2)_2]_{acac}$ centers [1–3].

According to the dislocation theory [26–27,34], to preserve the electroneutrality of ionic crystals, the interaction between dislocation leads to the formation of divacancies.

In principle, the processes of trihalide defect creation in KBr and KI crystals are similar [22,29–31]. Plastic deformation ($\epsilon = 4\%$) of KBr crystals significantly (almost by 3 times with respect to the nondeformed sample) increases the concentration of X-ray-induced $V_2 \equiv (Br_3^-)_{aca}$ centers responsible for absorption band at 4.6 eV as well as the $V_3 \equiv [(Br_2)_2]_{acac}$ centers (the relevant band maximum at 5.3 eV). In plastically deformed ($\epsilon = 4\%$) KI crystals, the same three-fold concentration increase of radiation-induced (isodose X-ray exposure) $V_2 \equiv (I_3^-)_{aca}$ centers (absorption maximum at 3.8 eV) and $V_3 \equiv [(Br_2)_2]_{acac}$ centers (absorption maximum at 4.5 eV) was detected earlier [20,30–31].

Thus, in KBr, KCl and KI crystals, divacancies $v_a^+v_c^-$ and vacancy quartets $2(v_a^+v_c^-)$, previously created in the sample by applied plastic deformation, contribute to the further formation of anion radiation defects, mainly the $(X_3^-)_{aca}$ centers. In particular, the interaction of two interstitial halogen atoms (an H center is equivalent to a X_2^- dihalide molecule located at one anion site) near a divacancy, schematically shown in Fig. 1, occurs as follows:



Indeed, the effective radiation creation of the $(X_3^-)_{aca}$ centers confirms a high concentration of divacancies in the deformed crystal. On the other hand, the radiation creation of the $[(X_2)_2]_{acac}$ center indicates the presence in the plastically deformed crystal of a vacancy quartet and participation of four mobile H centers in the plastically deformed crystal.

3.2. Local deformation of KCl crystals

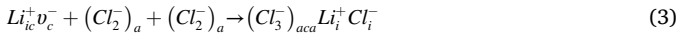
The local deformation is caused by the crystal doping with light cation-homologue, Li resulting in the presence of point lattice defects—Li ions and $Sr_c^+v_c^-$ dipoles in the lattice of the KCl crystal.

In addition to the $v_a^+v_c^-$ divacancies, the $Li_{ic}^+v_c^-$ and $Sr_c^{2+}v_c^-$ charged dipoles, the presence of which causes lowering of local symmetry of the crystal lattice, were also detected in KCl:Li and KCl:Sr crystals, respectively. In this regard, the search for the $(X_3^-)_{aca}$ centers perturbed by an adjacent dipole complex ($Li_{ic}^+v_c^-$ or $Sr_c^+v_c^-$) and the analysis of the efficiency of their creation by X-rays in KCl:Li and KCl:Sr crystals, both nondeformed and exposed to plastic deformation has been continued.

In KCl crystals doped by light homologous cations, the $V_4 \equiv (X_3^-)_{aca}M_{Ai}^+X_i^-$ centers can be formed, where M_{Ai}^+ is an interstitial light impurity cation, in particular, a lithium ion located in interstice of a KCl-Li crystal lattice. It was suggested in [19] that the release of a small-size cation into an interstice is facilitated in AHCs with light impurity cations. Based on a certain absorption band induced during X-ray irradiation and its subsequent thermal annealing, the structure of the corresponding trihalide defect, $(X_3^-)_{aca}$ or $(X_3^-)_{aca}M_{Ai}^+X_i^-$, as well as the mechanism of defect formation can be determined.

Consequently, the formation efficiency of the $(X_3^-)_{aca}$ center in the

field of a homologous small-radius cation has been studied in KCl:Li crystals by the absorption spectroscopy method. According to Fig. 3, the absorption band peaked at 5.1 eV is more intense in a KCl:Li crystal (curve 2) with respect to that in a pure crystal (curve 1). Therefore, the $(Cl_3^-)_{aca}Li_i^+Cl_i^-$ centers tentatively responsible for this X-ray-induced band, are created more efficiently in KCl:Li due to the mutual interaction of mobile interstitial halogen atoms in the field of the $Li_i^+v_c^-$ dipole complex according to the following reaction:

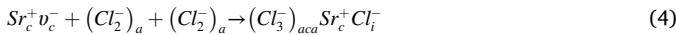


This process is similar to the association of two H centers in regular lattice regions, but is energetically more profitable, since a small-size lithium ion is initially displaced from a regular cation site into an interstice (i.e. along $[111]$ crystallographic direction). In a KCl:Li crystal, the structure of the paramagnetic $H_A(Li)$ center differs sharply from that for the H centers adjacent to other impurity cations. According to the EPR data [35–36], orientation of the $H_A(Li)$ center in KCl:Li is closer to the $[100]$ axis (deviated from $[100]$ by 26°), while the single H center is oriented along $\langle 110 \rangle$ directions [37–39].

When performing the reaction (3), the additional absorption band peaked around 6.4 eV (i.e. typical absorption of the I -type centers in KCl) is expected to appear after X-ray irradiation. Note that the interstitial chlorine ion (I center, Cl_i^-) is a constituent part of the $(Cl_3^-)_{aca}Li_i^+Cl_i^-$ radiation defect (see curve 2 and the defect model in Fig. 3).

Because of the off-center position of lithium ion in the cation lattice site of KCl:Li, it is assumed that dipole defects $Li_i^+v_c^-$ are present already in as-grown (nonirradiated) crystal.

Similarly, a dipole center consisting of a strontium ion Sr^{2+} located at a regular cation site and an adjacent cation vacancy for charge compensation arises in KCl: Sr^{2+} crystals. In this case, the interaction of two mobile interstitial halogen atoms should be energetically more profitable nearby $Sr^{2+}v_c^-$ with respect to that in regular lattice regions. It is worth noting that in KCl:Sr crystals, the association of mobile H centers will occur near an as-grown cation vacancy that facilitates the formation of the so-called $(Cl_3^-)_z = (Cl_3^-)_{aca}Sr_c^+Cl_i^-$ center [1–3] by the following reaction:



The interaction of mobile interstitial halogen atoms in the vicinity of $Sr^{2+}v_c^-$ is more energetically advantageous compared to the undisturbed lattice. During lattice rearrangement according to the reaction (4), a

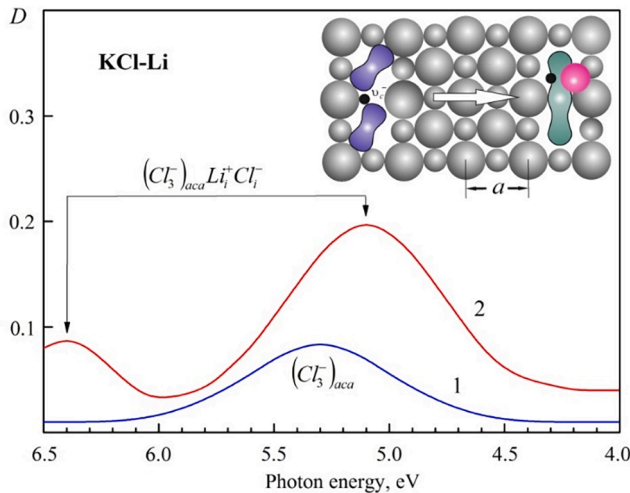


Fig. 3. Absorption spectra measured at 80 K for KCl (curve 1) and KCl:Li crystals (curve 2) after isodose irradiation by X-rays (240 min) at 80 K. In the upper right corner, a scheme of the interaction of two mobile H centers in the field of a $Li_i^+v_c^-$ complex with the formation of a $(Cl_3^-)_{aca}Li_i^+Cl_i^-$ center is shown.

positively charged center $(Cl_3^-)_{aca}Sr_c^+$ and an adjacent negatively charged Cl_i^- interstitial halogen ion should be formed, and just the latter (I center) is responsible for the absorption at 6.4 eV (curve 2, Fig. 4).

It was proved by the EPR method that mobile interstitial halogen atoms could be stabilized nearby strontium-related dipoles thus forming the so-called $H_Z(Sr)$ centers [35–36,38]. In a KCl-Sr crystal irradiated at 80 K, the H_Z center captured at a $Sr_c^{2+}v_c^-$ (oriented along the $[110]$ crystallographic axis) demonstrates a deviation of the $(Cl_2^-)_{ac}$ axis by 14.5° from the $[100]$ direction. These H_Z centers could be thermally destroyed at 230 K and have absorption band with a maximum at 3.86 eV.

Note that according to Fig. 4 (curve 2), two X-ray-induced absorption bands related to the $(Cl_3^-)_{aca}Sr_c^+Cl_i^-$ centers have been detected in a KCl:Sr crystal: firstly, the 5.6 eV band is close to the absorption of Cl_3^- molecules with the maximum at 5.3 eV in a pure KCl (Fig. 4, curve 1) and secondly, the band peaked at 6.4 eV due to the involvement of Cl_i^- . The model of such complex trihalide defect is shown in the right corner of Fig. 4.

The absorption characteristics of different trihalide-type defects, analyzed in the present study are summarized in Table 1.

4. Conclusions

The experimental results presented in this paper allow us to demonstrate the X-ray induced creation of trihalide-molecule-based defects in pure KCl crystals nondeformed or exposed to plastic deformation as well as in KCl crystals doped with small-size Li^+ ions or Sr^{2+} -related dipoles.

In a KCl crystal plastically deformed at room temperature, a subsequent X-ray-irradiation at 80 K leads to the efficient formation of trihalide $(Cl_3^-)_{aca}$ centers responsible for the absorption band at 5.3 eV. The same absorption band, but significantly less intense (by more than 3 times), has been detected after isodose irradiation of a nondeformed KCl crystal. The applied plastic deformation causes the efficient formation of $v_a^+v_c^-$ divacancies, which serve as seeds for further interaction of two mobile radiation-induced H centers (interstitial halide atoms, $(X_2^-)_a$) and, as a result, creation of the $(Cl_3^-)_{aca}$ defects. Additional absorption band with a maximum around 5.8 eV and connected with more complex associations of trihalide molecules appears under irradiation of a plastically deformed KCl crystal by X-rays at room temperature.

The purposeful introduction of impurities (small-size Li^+ ions, displaced from a cation site toward interstice or $Sr_c^{2+}v_c^-$ dipoles) causes lowering of local symmetry of KCl crystal lattice. After X-ray irradiation at 80 K, the efficient creation of $(Cl_3^-)_{aca}Li_i^+Cl_i^-$ and $(Cl_3^-)_{aca}Sr_c^+Cl_i^-$

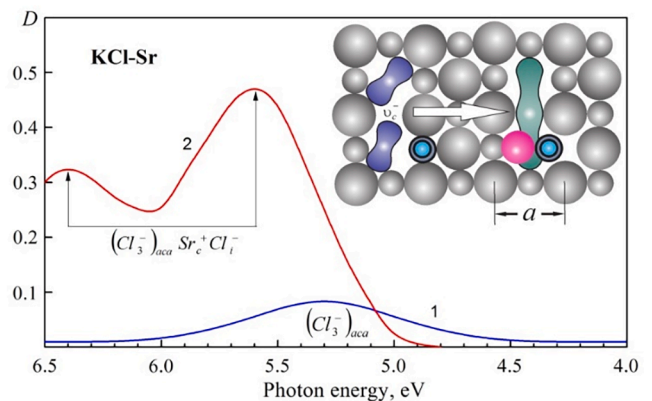


Fig. 4. Absorption spectra measured at 80 K for KCl (curve 1) and KCl:Sr crystals (curve 2) after isodose irradiation by X-rays (180 min) at 80 K. In the upper right corner, a scheme of the interaction of two mobile H centers in the field of a $Sr_c^+Cl_i^-$ complex with the formation of a $(Cl_3^-)_{aca}Sr_c^+Cl_i^-$ center is shown.

Table 1

Absorption characteristics of trihalide-type radiation defects in KCl, KCl:Li and KCl:Sr crystals.

Crystal	The position of the maximum absorption of $(Cl_3)_{acc}$ centers (eV)	The position of the maximum absorption of more complex defects (eV) and relevant defect structure
KCl	5.3	4.9 $(Cl_3)_{acc} K_i^+ Cl_i^-$
KCl:Li	5.3	5.1; 6.4 $(Cl_3)_{acc} Li_i^+ Cl_i^-$
KCl:Sr	5.3	5.6; 6.4 $(Cl_3)_{acc} Sr_i^+ Cl_i^-$

defects have been detected in KCl:Li and KCl:Sr single crystals, respectively. In both cases, in addition to the absorption band typical of a trihalide molecule (peaked at 5.1 eV in KCl:Li and 5.6 eV in KCl:Sr), the second absorption band at 6.4 eV has been detected as well, which reflects the involvement of Cl_i^- interstitial halide ions in both complex radiation defects.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

This research has been funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan (Grant No. AP08855672).

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