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Radiation Defect Formation in Strontium and Calcium Fluoride Crystals Doped by Divalent Cadmium and Zinc Ions

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Abstract—Ionization radiation is shown to reduce impurity ions to the univalent state in strontium and calcium fluoride crystals doped by divalent cadmium and zinc ions. In this case, a univalent ion is surrounded by eight equivalent fluorine ions and exhibits cubic symmetry O_h. At room temperature, the symmetry of the center is revealed to be sequentially lowered to C_{3v} and then to C_{2v} owing to the addition to the nearest environment of the impurity univalent ion of one or two anion vacancies, respectively, which are intrinsic defects not forming in undoped strontium and calcium fluoride crystals. Stable intrinsic defects are assumed to form through the separation of anion vacancy–interstitial fluorine ion pairs in the electric field induced by the reduced impurity ions. This electric field lowers the energy barrier to thermal separation of charged intrinsic defects.

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1. INTRODUCTION

By now, the processes of electron excitation decay creating intrinsic radiation defects in the anion sublattice of alkali-halide crystals have been well studied. The main channel of radiationless decay of a relaxed exciton in the crystals is the formation of a neutral defect pair, namely, an anion vacancy trapping an electron and an interstitial halogen atom (*F* and *H* centers, respectively). A pair of charged defects, such as an anion vacancy and an interstitial haloid ion (α and *I* centers, respectively), is formed significantly less effectively. The creation of stable intrinsic defects involves two stages, namely, the formation of a pair of nearest neighbor defects and the subsequent increase in the distance between these individual defects to a fairly large value to prevent their mutual recombination [1, 2].

The processes of radiation defect formation in alkali-earth fluoride crystals are less well understood. It has been revealed that all sufficiently pure alkali-earth fluoride crystals are radiation-resistant at room temperature. At 77 K, the radiation coloration of calcium and strontium fluoride crystals creates defects in amounts that at least two to three orders of magnitude less than those in most of alkali halides, with calcium fluoride crystals being the most radiation-resistant [3, 4].

The formation of radiation defects in these crystals, as well as in alkali halides, is related to radiationless decay of triplet excitons e^0. According to optically detected magnetic resonance data, the exciton structure in these crystals is close to the $F + H$ configuration [5, 6], unlike the $V_k + e$ configuration in alkali halides [7, 8]. Despite the fact that a radiation-induced pair of closely spaced defects in alkali-earth fluorides consists of an *F* and an *H* center, stable V_k (self-trapped holes) and *F* centers observed experimentally and also interstitial fluorine ions are formed at a certain stage of the defect separation. In this case, the interstitial fluorine ions manifest themselves as disturbances of *F* centers [2].

The efficiency of radiation defect formation in undoped BaF_2 crystals at 77 K exceeds that in calcium fluoride crystals by at least one to two orders of magnitude. The radiation coloration of barium fluoride crystals at 77 K causes the formation of intense absorption bands with maxima at 3.4 and 2.3 eV. In [9], using electron paramagnetic resonance (EPR), thermoluminescence, optical absorption, and x-ray luminescence, it was shown that these bands are associated with the absorption of V_k and *F* centers, respectively. In this case, stable V_k and *F* centers are formed in two stages as follows. At the first stage, an interstitial fluorine ion and an anion vacancy separate (perhaps with a preliminary tunneling charge exchange of the $F + H$ pair)

$$e^0 \longrightarrow e^0(F\text{–}H) \longrightarrow \alpha\text{–}I(F_i^-).$$

At the second stage, the subsequent capture of the electron by the anion vacancy results in the formation of an *F* center, hole self-localization, and the formation of a V_k center:

$$\alpha\text{–}I(F_i^-) + e^- + e^+ \longrightarrow F + I(F_i^-) + V_k.$$

Thus, unlike the alkali-halide crystals, in which stable neutral radiation defects are formed, stable radiation defects in alkali-earth fluorides are charged. This feature of alkali-earth fluorides necessitates the inclusion of the effect of electric fields on the processes of formation and separation of a pair of charged defects (α–I pair), which is not required in the case of neutral defects (at least, in the case where there is no need to involve tunneling processes [10]). An electric field decreases the potential-barrier energy, which can favor the thermally stimulated separation of oppositely charged defects in accordance with the mechanism proposed by Frenkel [11] and is likely to prevent the mutual recombination of the oppositely charged defects.

The sources of electric fields in ionic crystals can be charged intrinsic or impurity centers, dislocations, and other structure disturbances, and also optical lattice vibrations [12, 13].

We should note one more feature of alkali-earth fluorides that is also likely to manifest itself in the radiation defect formation. Unlike alkali-halide crystals, whose ionic conduction is mainly associated with the cation sublattice, alkali-earth fluorides are superionic conductors whose electrical conduction is associated with pairs of anti-Frenkel defects, namely, an anion vacancy and an interstitial fluorine ion (α–I centers). These pairs are the main intrinsic defects not induced by radiation in alkali-earth fluoride crystals [3].

It is generally believed that an impurity influences the accumulation of intrinsic defects in alkali-halide crystals not at the first stage of the decay of a self-trapped exciton into intrinsic defects but rather at the second stage of interaction of the interstitial halogen atoms with each other or with the impurity, which results in their thermal stability and in an increased concentration of thermally stable intrinsic defects [1].

A different situation occurs with alkali-earth fluorides. The doping of alkali-earth fluorides (mainly calcium fluoride crystals) by a number of trivalent rare-earth ions (La, Ce, Gd, Tb, Lu) and yttrium, which have low third ionization potentials and, thus, are not reduced upon radiation coloration, leads to the formation of photochromic centers. These centers consist of both impurity centers and intrinsic radiation defects (anion vacancies) with one or two electrons [3, 14].

In this work, we show that the radiation-induced formation of intrinsic defects in strontium and calcium fluoride crystals doped by divalent cadmium and zinc ions is associated with the charge state of the impurity, which is charged with respect to the lattice upon its reduction. The electric field of a charged defect lowers the energy barrier to thermal separation of charged intrinsic defects (α–I centers). As a result, defects are formed consisting of both reduced impurity ions and intrinsic radiation defects (anion vacancies).

2. EXPERIMENTAL TECHNIQUE

Strontium and calcium fluoride crystals were grown from the melt using the Bridgman–Stockbarger method in a graphite crucible in vacuum or an inert atmosphere. Since a cadmium fluoride impurity is usually used in growing fluoride crystals to prevent the formation of oxygen impurities and is released during growing, we had to perform the growth under melt seal to prevent the volatilization of cadmium or zinc impurities from the melt. The impurity concentration was determined using atomic-emission analysis.

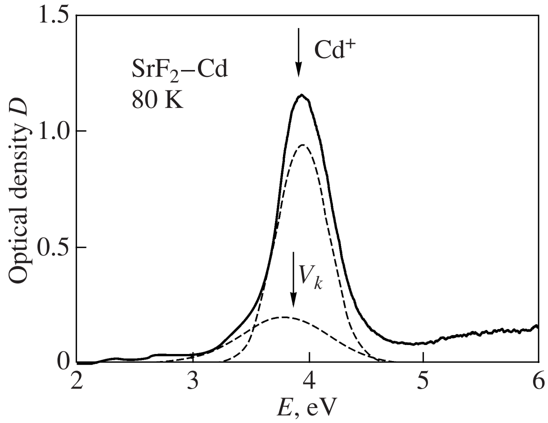

Fig. 1. Absorption spectrum of SrF$_2$–0.016 wt % Cd crystals irradiated with x rays at 80 K. The dashed lines show the decomposition of the spectrum into Gaussians.

The EPR spectra were measured on an RÉ-1306 radiospectrometer using a Dewar vessel of quartz glass for measurements at the liquid-nitrogen boiling temperature. For coloration of crystals, a BKhV-9 palladium-anode x-ray tube operating in the limiting mode at 50 kV and 50 mA was used. The coloration for EPR measurements was performed in a foamed-plastic Dewar vessel for not more than 1 h. For this coloration mode, radiation defects were not detected in undoped calcium and strontium fluoride crystals using the EPR method or optical absorption or luminescence spectra. The absorption spectra were measured using a Specord UV/VIS spectrophotometer in the range 200–800 nm. The luminescence spectra were measured using an FÉU-106 photomultiplier and an MDR-2 monochromator. The spectra were excited by a DDS-30 deuterium lamp using an MDR-12 monochromator.

3. EXPERIMENTAL RESULTS

3.1. SrF$_2$–Cd

Nonirradiated SrF$_2$–Cd crystals have no absorption bands in the visible spectral range and are transparent in a wide range of wavelengths. Only the fundamental absorption is shifted to longer wavelengths as the cadmium concentration increases [15]. X-ray irradiation of SrF$_2$–Cd crystals at 80 K produces Cd$^+$ centers with an absorption-band maximum at 3.95 eV [16] and V_k centers with a maximum at 3.8 eV [17] (Fig. 1). In this case, the absorption bands of the electron and hole centers are poorly separated. The EPR data show that the

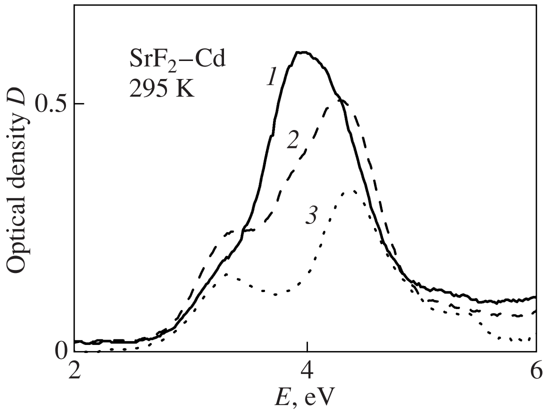

Fig. 2. Absorption spectrum of SrF_2–Cd crystals irradiated with x rays at 295 K for (*1*) 1 min and (*2, 3*) exposed then to this temperature for (*2*) 2 and (*3*) 30 min.

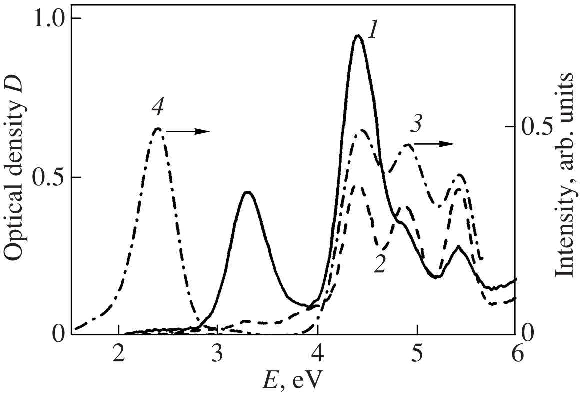

Fig. 3. (*1*, *2*) Absorption spectra at 80 K and (*3*) excitation and (*4*) luminescence spectra at 295 K of SrF_2–Cd crystals irradiated with x rays at 295 K and (*1*) held at this temperature to complete destruction of cubic Cd^+ centers and (*2–4*) bleached then by white light at 295 K.

Cd^+ ion is positioned in the cube center and surrounded by eight equivalent fluorine ions; i.e., its position has cubic symmetry O_h. These results are close to the previously obtained data [18]. With this symmetry, the Cd^+ *p* state is not split and transforms according to the irreducible representation t_{1u}. This is likely to be a relatively simple case where the univalent cadmium ion is an effective deep electron trap and a hole is stabilized at 77 K as a V_k center. At sufficiently high temperatures, a hole moves to the electron center owing to the Coulomb attraction and their mutual recombination occurs. In this case, the interaction cross section between oppositely charged defects is two to three orders of magnitude larger than that between neutral defects. However, heating of SrF_2–Cd crystals irradiated at 77 K to room temperature does not destroy completely the cadmium centers reduced to the univalent state. Some reasons for this behavior of holes are considered in [19].

Heating to room temperature leads to transformation of the cubic Cd^+ centers into cadmium centers of lower symmetry with two absorption bands having maxima at 3.30 and 4.39 eV and an intensity ratio of 1 : 2, respectively (Fig. 2). The room-temperature x-ray coloration gives the same result. The Cd^+-center absorption band splitting is related to the lowering of the symmetry of the center, which is likely due to addition of a perturbing defect (anion vacancy, as will be seen below). In this case, the symmetry of the Cd^+ center is lowered to C_{3v} and the excited *p* level is split into two states transforming according to the irreducible representations a_2 and *e*, with the intensity ratio in the absorption spectra being 1 : 2, respectively. The addition of an anion vacancy makes the defect electrically neutral, and the concentration of these centers is a maximum at room temperature.

The optical bleaching of the Cd^+ centers with the C_{3v} symmetry at room temperature in both absorption bands leads to the formation of Cd^+ centers with even lower symmetry having three absorption bands of approximately the same intensity (curve *2* in Fig. 3).

A small amount of these centers with three absorption bands is formed immediately after the room-temperature x-ray coloration. The photoexcitation in the absorption bands of these centers results in luminescence with a maximum at 2.35 eV (527 nm; green glow). Cooling to 77 K shifts the luminescence band to longer wavelengths with a maximum at 2.1 eV (590 nm). The luminescence band is asymmetric; there is a long tail on the low-energy side. The excitation spectra exhibit three bands of almost the same intensity; i.e., the degeneracy of the *p* state is completely removed (Fig. 3). Cooling to 77 K has almost no effect on the position of the excitation bands but decreases their half-widths, and the resolution of the spectrum improves. Addition of one more anion vacancy lowers the symmetry of the Cd^+ center to C_{2v}, with the excited state transforming according to the irreducible representations a_1, b_1, and b_2, which must give in experiment three absorption bands of almost equal intensities if the center is sufficiently localized. It is this situation that is observed experimentally.

3.2. SrF_2–Zn

Very similar results are obtained for strontium fluoride crystals doped by divalent zinc ions. The x-ray irradiation of SrF_2–Zn crystals at 80 K causes the formation of Zn^+ centers with a maximum at 4.35 eV and V_k centers with a maximum at 3.8 eV. The absorption band of a zinc ion reduced to the univalent state is shifted to larger energies as compared to the Cd^+-ion absorption, which permits a fairly reliable separation of the absorption band of hole V_k centers (Fig. 4).

Heating of the crystals to room temperature results in the transformation of the cubic Zn^+ centers into zinc centers with lower symmetry C_{3v} having two absorption bands with maxima at 3.35 and 4.55 eV and an

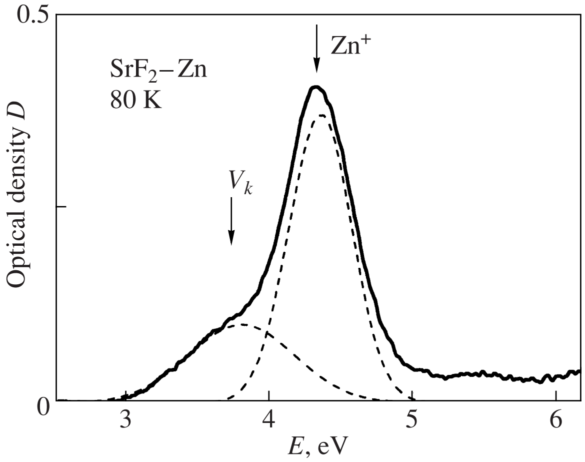

Fig. 4. Absorption spectrum of SrF_2–Zn crystals irradiated with x rays at 80 K. The dashed lines show the decomposition of the spectrum into Gaussians.

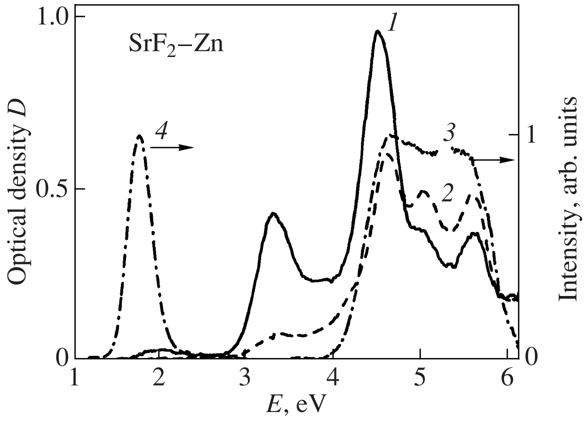

Fig. 5. (*1*, *2*) Absorption spectra at 80 K and (*3*) excitation and (*4*) luminescence spectra at 295 K of SrF_2–Zn crystals (*1*) irradiated with x rays at 295 K and (*2*–*4*) bleached then by white light at 295 K.

intensity ratio of 1 : 2, respectively. The room-temperature x-ray coloration causes at once the formation of Zn^+ centers having a double absorption band. These centers are partially transformed into Zn^+ centers with even lower symmetry C_{2v} (Fig. 5) after optical bleaching at 300 K. The room-temperature excitation in the absorption bands of the latter centers causes luminescence with a maximum at 1.8 eV (690 nm; red glow) (Fig. 5). In this case, the excitation spectra have three bands (which are less clearly defined than those in the cadmium-containing crystals) of almost equal intensities; i.e., the degeneracy of the *p* state is completely removed. Luminescence is strongly quenched (by more than an order of magnitude) and becomes more complex at 77 K.

The fact that the processes of thermal and optical transformation into centers with lower symmetry have an efficiency of less than 100% indicates that these processes are limited by external conditions (perturbing defects) rather than by the internal thermal instability of the center.

3.3. CaF_2–Cd and CaF_2–Zn

For the calcium fluoride crystals doped with divalent zinc or cadmium ions, we obtained results similar to those presented above for the strontium fluoride crystals (Tables 1–3). However, there are some specific features. Unlike the strontium fluoride crystals doped by zinc or cadmium, the glow in the calcium fluoride crystals doped by cadmium is observed upon photoexcitation in the absorption bands of the cadmium centers not only with symmetry C_{2v} but also with symmetry C_{3v}. The luminescence in the latter centers occurs upon the excitation in both absorption bands (Fig. 6), despite that the excitation to the higher energy absorption band with a maximum at 4.53 eV destroys these centers with the formation of reduced cadmium centers with symmetry C_{2v}. The photoexcitation in the absorption spectra of the $Cd^+(C_{3v})$ centers causes red glow with a maximum at 1.7 eV and a half-width of 0.34 eV at 295 K. As the temperature decreases, the red-glow intensity decreases significantly, whereas the green-glow intensity increases somewhat.

Table 1. Positions *E* and half-widths Δ*H* (in electronvolts) of the absorption, excitation, and luminescence bands of reduced impurity centers with symmetry C_{2v}

Crystal	Impurity center	Absorption and excitation bands						Luminescence bands		*T*, K
		E_1	ΔH_1	E_2	ΔH_2	E_3	ΔH_3	*E*	Δ*H*	
CaF_2	$Cd^+(C_{2v})$	4.4	0.32	5.04	0.32	5.53	0.32	2.35	0.26	77
SrF_2		4.39	0.48	4.86	0.48	5.39	0.46	2.1		295
		4.38	0.32	4.85	0.36	5.4	0.34	2.35		77
CaF_2	$Zn^+(C_{2v})$	4.75	0.32	5.24	0.34	5.79	0.32	2.36	0.32	16.8
SrF_2		4.65	0.42	5.11	0.44	5.63	0.44			77
								1.8		295

Table 2. Positions E and half-widths ΔH (in electronvolts) of the absorption, excitation, and luminescence bands of reduced impurity centers with symmetry C_{3v}

Crystal	Impurity center	Absorption and excitation bands				Luminescence bands		T, K
		E_1	ΔH_1	E_2	ΔH_2	E	ΔH	
CaF_2	$Cd^+(C_{3v})$	3.65	0.64	4.53	0.62	1.7	0.34	295
SrF_2		3.3	0.64	4.39	0.6			295
		3.28	0.4	4.39	0.4			77
CaF_2	$Zn^+(C_{3v})$	3.6	0.7	4.67	0.7			295
SrF_2		3.5	0.76	4.56	0.76			295
		3.35	0.48	4.55	0.48			77

4. PRELIMINARY RESULTS

Three types of centers were revealed in the strontium and calcium fluoride crystals doped by divalent cadmium and zinc ions. These centers are associated with the impurity ions reduced to the univalent state and differing in the symmetry of the nearest environment. Thermal annealing and photoinduced bleaching of these crystals at room temperature lead to a subsequent lowering of the symmetry of the center (from cubic O_h to C_{3v} and further to C_{2v}), which is indicated by the splitting of the p state of the impurity univalent ion. In the case of cubic symmetry O_h, the univalent impurity ion is in the center of a cube with eight equivalent fluorine ions located at its corners. It would appear reasonable that the lowering of the symmetry of the center is due to the replacement of one or two fluorine ions located in the $\langle 100 \rangle$ plane by additional defects. We show in Section 5 that the defects lowering the symmetry of the nearest environment of a univalent impurity ion are anion vacancies. In this case, the centers having symmetries O_h and C_{2v} are charged with respect to the regular lattice, whereas the centers with environment symmetry C_{3v} are neutral with respect to the lattice. Experimental data show that, after prolonged exposure to room temperature, the concentration of the latter centers significantly exceeds that of the former two centers (Figs. 3, 6), which also does not contradict the model proposed. The structure of the center with symmetry C_{3v} is similar to that of photochromic centers (at least, ionized photochromic centers) in calcium fluoride crystals doped by a number of trivalent rare-earth ions [3, 14]. However, there is a substantial difference; namely, the photochromic centers are formed in crystals containing rare-earth ions that have a low third ionization potential and, therefore, are shallow electron traps [20]. A trivalent impurity ion with a high third ionization potential is reduced to the divalent state upon radiation coloration [20]. The divalent cadmium and zinc ions are deep electron traps; because of this, the electron density is mainly concentrated at the univalent impurity center and does not extend to the anion vacancy. The experimental data on these centers are listed in Tables 1–3.

Naturally, there is a question as to the processes leading to the formation of anion vacancies, i.e., intrinsic defects that are not formed in the undoped crystals. This problem is considered in the following section.

Table 3. Positions E and half-widths ΔH (in electronvolts) of the absorption bands of reduced impurity centers with symmetry O_h

Crystal	Impurity center	Absorption		T, K
		E_1	ΔH_1	
CaF_2	$Cd^+(O_h)$	3.8	0.6	295
		3.82	0.5	77
SrF_2		3.95	0.6	295
		3.95	0.52	77
BaF_2 [16]		4.08	0.64	295
		4.1	0.54	77
CaF_2	$Zn^+(O_h)$	4.26	0.5	77
SrF_2		4.3	0.8	295
		4.35	0.52	77

5. FORMATION OF INTRINSIC DEFECTS IN THE CRYSTALS

In the crystals with low impurity contents ranging approximately from 0.001 to 0.010 wt %, apart from the impurity ions reduced to the univalent state, x-ray coloration at 80 K produces perturbed F centers and additional V_k centers (Fig. 7). At room temperature, such coloration creates only perturbed F centers and reduced impurity centers. The absorption of perturbed F centers consists of two bands, which indicates the presence of a perturbing defect in the nearest neighborhood. The p state in this case transforms according to the irreducible representations a and e, similarly to the situation in the photochromic centers in calcium fluoride crystals (at least, in ionized photochromic centers) [14]. The perturbed F centers are easily bleached by visible light at 77 K, which causes only a partial

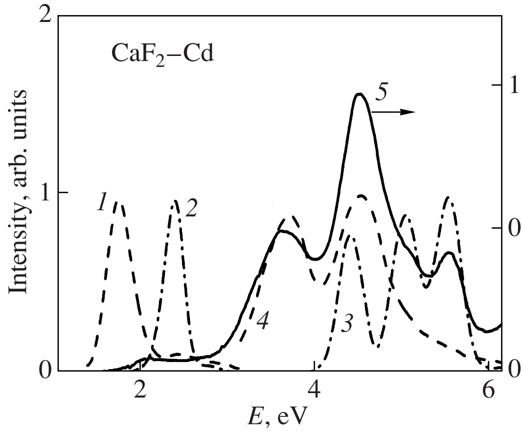

Fig. 6. (*1*, *2*) Luminescence, (*3*, *4*) excitation, and (*5*) absorption spectra of CaF$_2$–Cd crystals irradiated with x rays at 295 K and bleached then by light with a photon energy of 4.5 eV at room temperature. The luminescence spectra were measured under excitation by light with a photon energy of (*1*) 3.5 eV at 295 K and (*2*) 5.4 eV at 77 K. The excitation spectra were measured for luminescence at (*3*) 2.4 eV at 77 K and (*4*) 1.6 eV at 295 K.

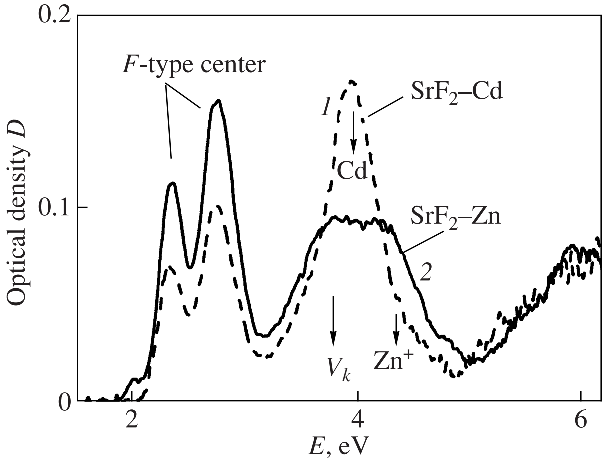

Fig. 7. Absorption spectra of (*1*) CaF$_2$–0.008 wt % Cd and (*2*) SrF$_2$–0.003 wt % Zn crystals irradiated with x rays at 80 K.

destruction of the V_k centers. The optical bleaching of these centers at room temperature increases the concentration of impurity ions reduced to the univalent state with symmetry C_{3v}, which confirms the proposed structure of the defect involving an anion vacancy.

The formation of an F–V_k pair indicates that, analogously to barium fluoride crystals [9], in the strontium and calcium fluoride crystals doped by cadmium and zinc ions, a pair of charged intrinsic defects, namely, an interstitial fluorine ion and an anion vacancy, is separated: $e^0 \longrightarrow e^0(F\text{–}H) \longrightarrow \alpha\text{–}I$. The subsequent electron capture by the anion vacancy results in the F-center formation, and the self-localization of the hole results in a V_k center. The existence of a perturbing defect in the nearest neighborhood of the F centers is indicated by the following two facts: (1) these centers are formed at both room and low temperatures and (2) the splitting of the absorption band of the perturbed F centers is close in value to that of the photochromic centers [14].

The position of the absorption bands, the ratio between the intensities of the split bands, and the magnitude of the level splitting of perturbed F centers are the same in the crystals doped by cadmium and zinc (Fig. 7). In this case, at the beginning of radiation coloration, impurity ions (cadmium or zinc) reduced to the univalent state are formed and then perturbed F centers begin to increase in number.

It would appear reasonable that the perturbing defect is an interstitial halogen ion. Basically, all radiation-induced F centers in alkali-earth fluoride crystals undergo a perturbation from an additional defect. It is commonly believed that this defect is an interstitial fluorine ion [2, 20].

The reason for the separation of a pair of charged intrinsic defects is, in our opinion, that the Cd$^+$ or Zn$^+$ impurity ions reduced to the univalent state are charged with respect to the regular lattice and produce an electric field [12]. The electric field lowers the energy barrier to separation of the charged defects [10, 11] and/or prevents their mutual recombination, which is likely to cause the formation of stable pairs of charged intrinsic defects (interstitial fluorine ion and anion vacancy) in the doped strontium and calcium fluoride crystals.

The formation of intrinsic centers in strontium and calcium fluoride crystals doped by cadmium or zinc ions is unambiguously determined by the impurity concentration. The formation of perturbed F centers and V_k centers requires two electron–hole pairs, and the concentration of these centers achieves ~0.01 wt %. For example, in SrF$_2$ crystals with a cadmium concentration of 0.016 wt %, only a small amount of perturbed F centers is formed (Fig. 1). To form an α–I pair, one electron–hole pair is necessary, and the concentration of α–I pairs achieves ~0.1 wt %. We controlled the appearance of α–I pairs by the formation of Cd$^+$ centers with symmetry lower than cubic one, which include, in our opinion, anion vacancies. Only cubic reduced cadmium or zinc impurity ion centers are formed with concentrations higher than those indicated above.

6. CONCLUSIONS

Irradiation of CaF$_2$–Cd, CaF$_2$–Zn, SrF$_2$–Cd, and SrF$_2$–Zn crystals with ionizing radiation leads to the reduction of the impurity ions to the univalent state. In this case, a hole remains in the valence band and is self-trapped at relatively low temperatures. As a result, a pair of oppositely charged defects is created. Such defects are randomly distributed and create local electric fields. In some cases, the electric field is sufficiently high to separate intrinsic radiation-induced charged defects (an anion vacancy and an interstitial halogen ion) forming during the decay of electron excitations (relaxed excitons). In this case, intrinsic defects are formed in the immediate vicinity of the impurity centers inducing the local electric field and not in the nearest environment, since thermal activation is required to form reduced impurity centers of lower symmetry. It can be assumed that the processes of forming radiation-induced defects that cause the formation of photochromic centers in alkali-earth fluoride crystals doped by a number of trivalent rare-earth ions are similar in physical nature to the processes considered in this work. The photochromic centers are formed in crystals doped by trivalent rare-earth ions that are not reduced upon radiation coloration and, therefore, produce local electric fields stimulating the formation of intrinsic defects (anion vacancies).

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