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PAPER

Thermal annealing of radiation damages produced by swift ^{14}N and ^{16}O ions in LiF crystals

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Abstract

Annealing of color centers was studied in lithium fluoride crystals, irradiated with 23-MeV nitrogen and 28-MeV oxygen ions. Basing on the optical absorption spectroscopy and reaction-rate modelling, a new interpretation of the annealing kinetics at the practically important temperatures below 500 K is suggested. Proposed model explains simultaneous decrease of the F and F_2/F_3^+ peaks as a result of migration of the F centers and formation of larger aggregates, and does not include additional assumptions about impurities and cation vacancies. It specifies the migration energy of the F centers in the ground state to be about 1.3 eV, that corresponds to earlier studies.

1. Introduction

Lithium fluoride is a very popular object of basic and applied researches due to its relatively simple structure, which however provides a rich variety of properties, among those radiation defects—the color centers are of particular interest. Similarly to the other alkali halides, LiF is very sensitive for ionizing irradiation, that creates defects in the anion sublattice via decay of the electronic excitations [1]. Note, that in lithium fluoride the color centers cannot be obtained with the additive coloration technique [2].

Investigations of the radiation damage with swift ions are aimed to understanding of both peculiarities of the material modification and interaction of swift projectiles with wide-band dielectrics in general [3–12]. Swift ions, depositing most of their energy to the electronic subsystem of target materials, effectively create primary Frenkel pairs in the fluorine sublattice of the lithium fluoride crystals. Further kinetics of the defects depends on the irradiation conditions, in particularly on the temperature [13]. At room temperature constituents of the Frenkel pairs can be actively separated due to diffusion, and form stable color centers [14–16]. Thus, the anion vacancies capture electrons, forming stable F centers and their aggregates F_n. Meanwhile the single fluorine interstitials (H centers) are highly mobile at room temperature, and those, avoided recombination, form aggregates or complex centers with impurities and cation vacancies [17]. Due to the strong ion binding of the lithium fluoride even intensive ion irradiation does not cause amorphization, although XRD can reveal nanostructuring of the crystals [18].

Our study is focused on the thermal stability of the color centers, formed in lithium fluoride with energetic nitrogen and oxygen ions. Accumulation of the stable defects and their annealing behavior is important for functional properties of optical materials and dosimetry application [19–22]. Results of the study can be valid for other alkali-halide crystals, having similar kinetics of the point defects [5]. However they should be applied with a caution to halides of alkali-earth metals due to the considerably higher defect formation energy in the crystals, which provide high radiation resistance of the materials [10, 23–26].

UV–vis absorption spectroscopy, employed in our research, is a widely used and reliable method for investigation of color centers. Supported with results of other techniques, it allows to attribute the absorption peaks to the certain color centers [26–28]. Thus, modification of the absorption spectra because of the heating

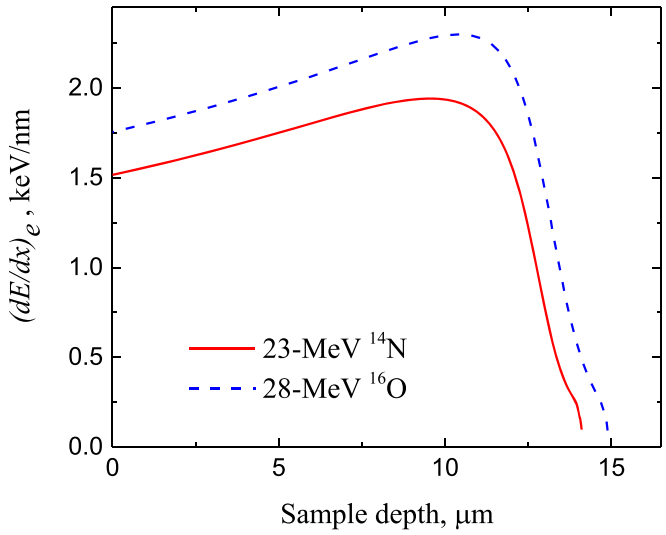

Figure 1. Electronic energy losses of the nitrogen and oxygen ions in lithium fluoride according to SRIM [29].

Table 1. Color centers in lithium fluoride at room temperature [31].

Center	Absorption maximum	
	(nm)	(eV)
F	248	5.00
F_2	444	2.79
F_2^+	685	1.81
F_3	317, 377	3.91, 3.29
F_3^+	448	2.77
F_3^-	950	1.31
F_4	518, 540	2.39, 2.30

allows to estimate changes of the defects' concentrations. However, the microscopic interpretation of their kinetics in some cases remains unclear. In particular, the main attention in our study is paid to the practically important temperature range of 400–500 K.

2. Experimental procedure and results

Lithium fluoride crystals were grown in vacuum from the pre-purified charge in JSC 'Research and Production Corporation S I Vavilova' (S I Vavilov State Optical Institute, Saint-Peterburg, Russia). Our XRD measurements confirm perfect crystallinity of the samples. Irradiations with 23-MeV ^{14}N and 28-MeV ^{16}O ions were carried out at DC-60 accelerator (Astana, Kazakhstan) at room temperature. According to the SRIM code [29] the stopping ranges R of the ions are 14.13 and 14.91 microns correspondingly, which are less than sample thicknesses. The electronic energy losses alongside the ion trajectories are plotted in the figure 1, the nuclear ones are negligible till the very end of the ion paths. As the stopping power of the both projectiles remains fair below 10 keV nm^{-1}, one should not expect the track core formation [30].

Optical absorption spectra of the irradiated samples were measured in the range of 1.5–6.5 eV by means of the spectrophotometer SF-2000 (Russia). The spectra reveal absorption peaks of various color centers, which are listed in the table 1. Without thermal annealing or optical excitation (bleaching) these centers are known to be stable at room temperature [31, 32]. Their behavior during thermal annealing was studied by heating an irradiated sample in the muffle furnace in atmosphere up to increasingly higher temperatures (T) and holding there for a certain time Δt (15 minutes for the samples irradiated with ^{14}N, and 10 min for ^{16}O). The temperature was controlled by the chromel-alumel thermocouple, mounted close to the sample holder. The heating rate was 5 K per second, so the heating times were noticeable less than Δt. Then the sample was cooled down the room temperature, and the corresponding absorption spectrum was measured.

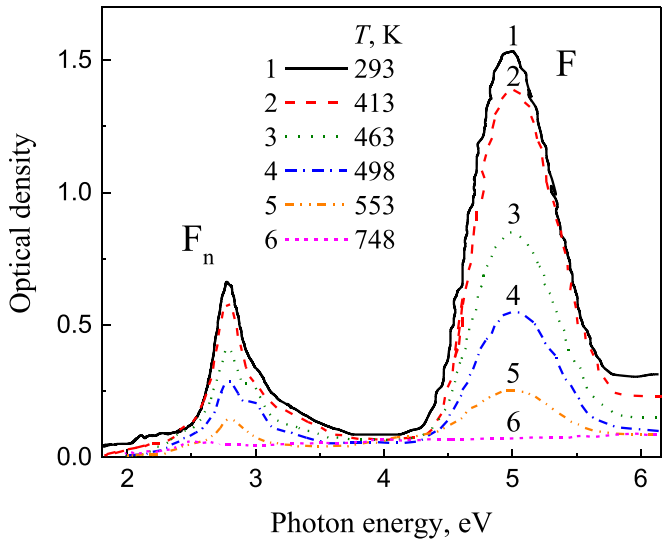

Figure 2. Absorption spectra of LiF crystals after irradiation with 4×10^{12} 23-MeV ^{14}N ions per cm^2 and subsequent annealing.

Absorption spectra of the lithium fluoride crystals after irradiation and subsequent annealing steps are shown in the figures 2 and 3. All spectra until annealing above 550 K contain F peak with maximum at 5 eV, which is slightly distorted, presumably due to cation defects [33–35]. Various F_n centers (see the table 1) form the absorption band in the range 1.77–4.13 eV [31]. Further aggregation of the F centers ($n > 4$) leads to formation of nF aggregates—precursors of Li colloids with the absorption maximum at 2.79 eV [31, 32, 36].

In the figure 2 one can see the absorption spectra of LiF crystals, irradiated with 23-MeV nitrogen ions. The F peak is dominating since the projectile energy and applied fluence of 4×10^{12} ions cm^{-2} are not high enough to provide active aggregation process [27, 28]. Similarly in the figure 3(a) the spectrum of the samples after irradiation with 28-MeV oxygen ions demonstrates higher F peak for the fluence of 1×10^{13} ions cm^{-2}. Contrarily in the figure 3(b), corresponding to the fluence of 1×10^{15} ions cm^{-2}, the F_n band is dominating.

The main contribution to the F_n band is given by F_2 and F_3^+ centers, possessing very close absorption peaks (table 1). They can be better distinguished by luminescent spectroscopy [37–39], including the ion-beam luminescent measurements [40, 41]. However, one can see that F_3^+ peak is decreasing faster than F_2 with the heating treatment, especially for high fluence (figure 3(b)).

3. Analysis and discussion

Optical density at the F-peak absorption maximum OD_F allows to evaluate the surface concentration of the F centers according to the Smakula-Dexter formula [42, 43], which at room temperature gives [3]:

$$n_F[\text{cm}^{-2}] = 9.48 \times 10^{15} \times OD_F \tag{1}$$

The average volume concentration of the F centers in the irradiated layer can be estimated as n_F/R, and the average distance between them correspondingly as

$$d = (n_F/R)^{-\frac{1}{3}} \tag{2}$$

For the irradiated samples before annealing with spectra shown in the figures 2 and 3 the equation (2) evaluates d of 3.8–4.8 nm, that means 13–17 interatomic (F-F ion) distances. Note, that the actual distribution is not uniform, and close to the ion trajectories the color centers can be separated by just a few interatomic distances [44].

From the other hand, the number of diffusional hops at a constant temperature is

$$M = \left(\Delta t \ \ \nu \exp\left(-\frac{E_F}{k_B T}\right)\right)^{\frac{1}{2}} \tag{3}$$

where ν is the attempt frequency factor about 10^{13} s^{-1} [45], E_F—the activation energy of the F centers, and k_B—the Boltzmann constant.

Absorption spectra in the figures 2 and 3 demonstrate that above 400 K both F and F_n peaks are starting to decrease. In this paper we suggest, that the early stage of annealing can be described as a result of the F centers'

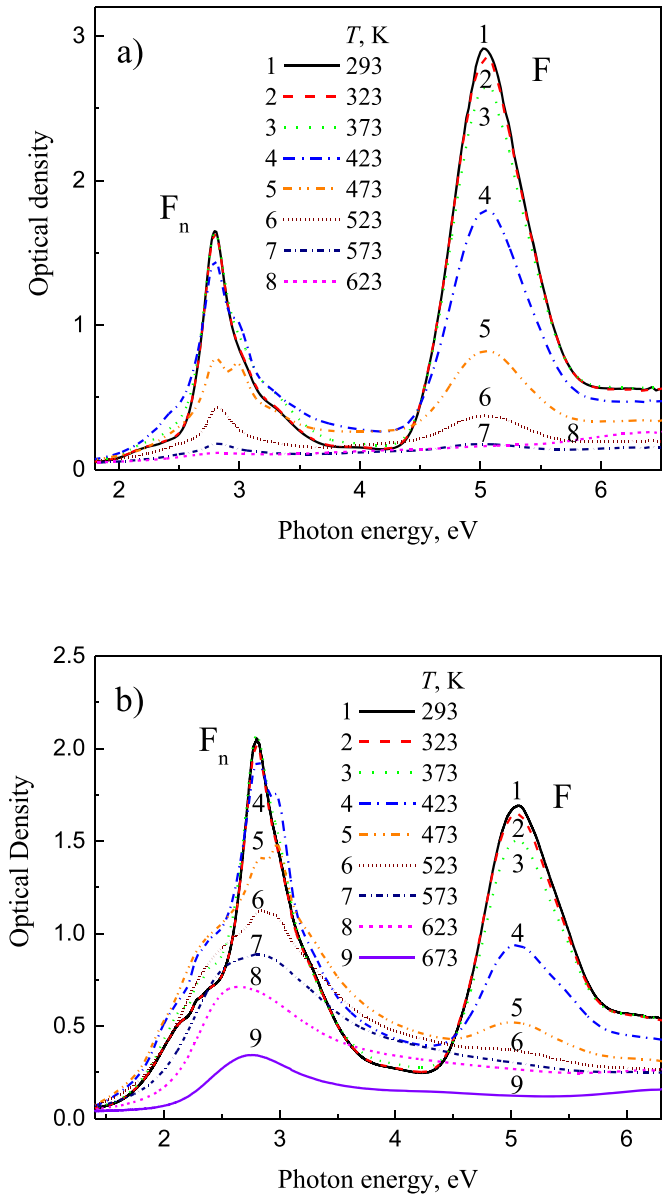

Figure 3. Absorption spectra of LiF crystals, irradiated with 23-MeV ^{16}O ions and stepwise annealed. Fluences of 1×10^{13} (a) and 1×10^{15} ions cm^{-2} (b) were applied.

diffusion, assuming their migration energy in the ground state of about 1.10–1.40 eV (figure 4), which seems quite plausible. We suppose the following reactions, comprising the mobile F centers:

$$F + F \rightarrow F_2, \quad F + F_n \rightarrow F_{n+1}, \quad F + H_n \rightarrow H_{n-1} \tag{4}$$

The following equations describe kinetics of the F centers and their aggregates [9, 46]:

$$\frac{\partial f_1}{\partial t} = -D_H f_1 \left(3r_F(1) f_1 + \sum_{n=1}^{\infty} r_F(n) f_n + \sum_{n=1}^{\infty} r_H(n) h_n \right) + D_H h_1 (r_F(2) f_2 - r_F(1) f_1)$$

$$\frac{\partial f_2}{\partial t} = D_F f_1 (2r_F(1) f_1 - r_F(2) f_2) + D_H h_1 (r_F(3) f_3 - r_F(2) f_2)$$

$$\frac{\partial f_n}{\partial t} = D_F f_1 (r_F(n-1) f_{n-1} - r_F(n) f_n) + D_H h_1 (r_F(n+1) f_{n+1} - r_F(n) f_n), \quad n \geqslant 3 \tag{5}$$

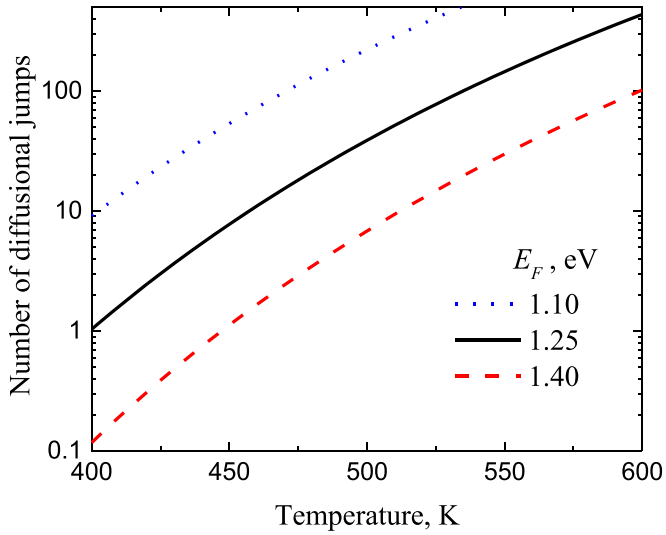

Figure 4. Number of diffusional jumps of F centers during the annealing step $\Delta t = 600$ s versus temperature for different migration energies E_F according to the equation (3).

where f_1, f_2, f_n are the distribution function for F, F_2 and F_n centers correspondingly. For $n > 4$ we assume nF aggregates.

Reaction $F + H_2 \rightarrow H$ releases mobile interstitials, and those further interactions should be considered. Thus, for distributions of H centers (h_1) and their aggregates (h_2, h_n) we have:

$$\begin{aligned}
\frac{\partial h_1}{\partial t} &= -D_H h_1 \left(3 r_H(1) h_1 + \sum_{n=1}^{\infty} r_H(n) h_n + \sum_{n=1}^{\infty} r_F(n) f_n \right) \\
&\quad + D_F f_1 (r_H(2) h_2 - r_H(1) h_1) \\
\frac{\partial h_2}{\partial t} &= D_H h_1 (2 r_H(1) h_1 - r_H(2) h_2) \\
&\quad + D_F f_1 (r_H(3) h_3 - r_H(2) h_2) \\
\frac{\partial h_n}{\partial t} &= D_H h_1 (r_H(n-1) h_{n-1} - r_H(n) h_n) \\
&\quad + D_F f_1 (r_H(n+1) h_{n+1} - r_H(n) h_n), \quad n \geqslant 3
\end{aligned} \tag{6}$$

Here D_F and D_H are the diffusion coefficients, r_F and r_H are the reaction factors, describing interaction of the corresponding aggregates with mobile defects. We assume [46]

$$r_F(n) = n^{\frac{2}{3}}, \quad r_H(n) = n^{\frac{2}{3}} \tag{7}$$

as a simple model of the reaction rates, proportional to the aggregate surface areas.

Initial distribution of the color centers can be taken from the modelling of irradiation at room temperature [46], then equations (5), (6) should be solved to find their concentration after a given time of annealing, which can be expressed as the number of diffusional hops M according to the equation (3). Typical results of the calculations are presented in the figure 5.

Below 500 K we do not take into account possible evaporation of the single F centers from F_n centers and smaller nF aggregates, that would result in the Ostwald ripening, leading to formation of larger nF aggregates and metal colloids [5, 32, 47, 48].

The modelling confirms our suggestion that the reactions (4), caused by mobility of the single F centers, provide decrease of both F and F_2 centers in favor to larger aggregates. In earlier investigations such simultaneous decrease of both absorption peaks was attributed to decay of the interstitial aggregates [45], release of the H centers and their subsequent recombination with F and F_n centers. Alternatively, decay of complexes, composed of the anion vacancy and a trace element (e.g. oxygen) or cation vacancy, and appearance of the mobile anion vacancies was proposed in [31]. Anyway, the F centers were supposed to be immobile until 500 K to explain the effect formerly. But assuming the reactions (4) those mechanisms become unnecessary. The H centers, despite their high mobility, seemingly do not initiate the annealing reactions below 500 K due to strong binding within their aggregates.

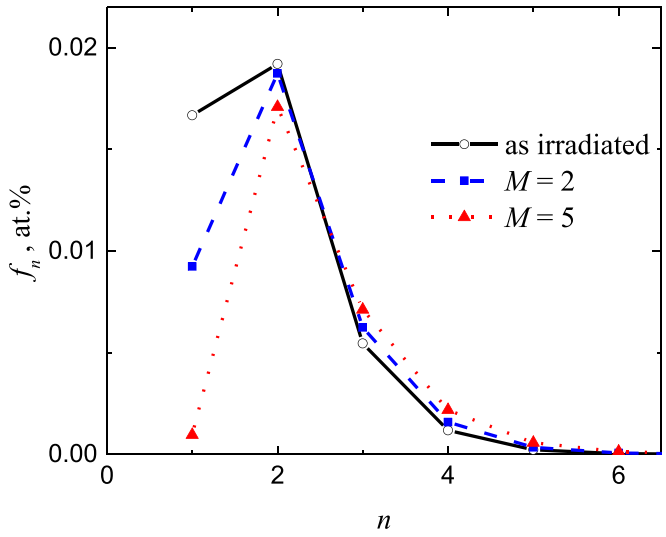

Figure 5. Calculated concentrations of the F centers and their aggregates (F_n, nF) as irradiated and after annealing, providing M hopping distances for the single F centers.

Additional decrease of the F_2/F_3^+ peak can be associated with reactions of the F_3^+ centers, providing new color centers, whereas appearance of the new charged F_3^+ centers is very unlikely during annealing.

4. Conclusion

Optical absorption spectra of the lithium fluoride crystals, irradiated at room temperature with 23-MeV nitrogen and 28-MeV oxygen ions, comprise of the F and F_n peaks, and both are decreasing during the thermal annealing, starting from about 400 K. The new interpretation of the effect is suggested.

We assume that below 500 K the annealing process is originated by mobility of the single F centers, their mutual interactions, and reactions with other color centers. Proposed model does not assume additional mechanisms, like release of the anion vacancies [31], for this annealing stage. Reaction-rate kinetic modelling of the color centers during annealing confirms this interpretation and gives migration energy of the F centers about 1.3 eV, that is consistent with the earlier data [24]. It also explains faster decrease of the F_3^+ (2.77 eV) peak relative to the F_2 (2.79 eV).

Annealing at the temperatures above 500 K demonstrates further decrease of the absorption peaks and should include decay of the complex color centers and defect aggregates, that will be considered elsewhere.

Acknowledgments

Authors deeply miss Prof. Dr Kurt Schwartz—our patient teacher, dear colleague, and kind friend, passed away last year. He was a real expert in the field of radiation defects in ionic crystals, and we try our best to follow his dedication to science. M V S desperately sorrows over his dear son, friend, and co-author [9] Anton Sorokin, who has suddenly passed away at the age of 22.

This work has been carried out using computing resources of the federal collective usage center Complex for Simulation and Data Processing for Mega-science Facilities at NRC 'Kurchatov Institute', http://ckp.nrcki.ru/.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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