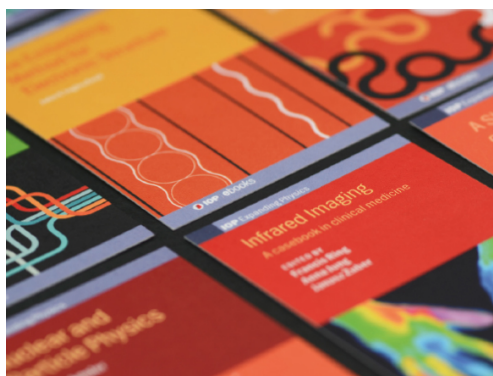


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Luminescence of F_2 and F_3^+ centres in LiF crystals irradiated with 12 MeV ^{12}C ions

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Abstract. Dependences of the nanohardness and photoluminescence of F_2 and F_3^+ centers on the depth in LiF crystals irradiated with 12 MeV ^{12}C ions to fluences 10^{10} - 10^{15} ions/cm² were studied using laser scanning confocal microscopy, luminescent spectroscopy, and the nanoindentation method. The nanohardness measurements showed a significant hardening effect at the end of the ion run with the dominant contribution of defects formed by the mechanism of elastic collisions. The observed attenuation of the luminescence intensity at high fluences is associated with the intense nucleation of dislocations as traps for aggregate color centers.

1. Introduction

Ion modification of materials has proved to be an effective tool for the formation and modification of electrical, optical, mechanical and chemical properties of a wide range of materials. Investigation of the change in the properties of materials in the relationship is a complex task and LiF crystals play a role in solving such problems, due to the simple structure and good study of radiation damage.

In this paper, we present results on the study of the bulk luminescence of F_2 and F_3^+ centers, the luminescence of these centers along the ion path, nanohardness on the frontal surface, and the surface of the transverse cleavage in LiF crystals irradiated with 12 MeV carbon ions.

2. Experimental equipment

Irradiation of samples of LiF crystals (SOI, St. Petersburg and ISU, Irkutsk) was carried out on a DC-60 cyclotron at a beam current density of 10 nA/cm², for which the sample heating during irradiation can be neglected [1, 2]. The fluence varied from 10^{12} to 10^{15} ion/cm². Photoluminescence of the F_2 and F_3^+ centers along the ion pathway was measured with a confocal scanning microscope (LCSM). Agilent Nano G200 (USA) – the system for indenting was used to study nanohardness along the



profile depth, characterizing the evolution of nanoscale defects along the ion path, it makes it possible to make standard measurements of CSM (Continuous Stiffness Measurement) in the regime of constant recording of the applied force and displacement of the point of the indenter.

3. Results and discussion

Absorption spectra for LiF crystals irradiated with 12 MeV ^{12}C ions are shown in figure 1. Analysis of the spectra shows that for large values of the fluences, the absorption in the F and F_n regions of the centers decreases. The concentration of F centers was determined by the Smakul-Dexter formula $n_F = 9.48 \cdot 10^{15} \cdot D_{opt}(F)$, where $D_{opt}(F)$ – optical density at the maximum of the absorption band of F centers, and volume concentration $N_F(\text{cm}^{-3}) = n_F/R$. To compare the relative concentrations of F_n and F centers, we used integral absorption (A_{F_n} and A_F , which were determined by formulas

$$A_F = \int_{4.13}^{5.90} D(\varepsilon) d\varepsilon \quad \text{and} \quad A_{F_n} = \int_{1.77}^{4.13} D(\varepsilon) d\varepsilon$$

Figure 2 shows the dependences of N_F , A_F and A_{F_n} for LiF crystals irradiated by ^{12}C ions with energy of 12MeV. The decay of N_F , A_F and A_{F_n} can be explained by the creation of larger aggregates with increasing absorbed energy. We note that the N_F decay occurs at an absorption energy several times lower than for A_F . This is explained by the fact that at large absorbed energies the area under the F-center curve widens due to the formation of defects from trace elements (mainly associated with magnesium).

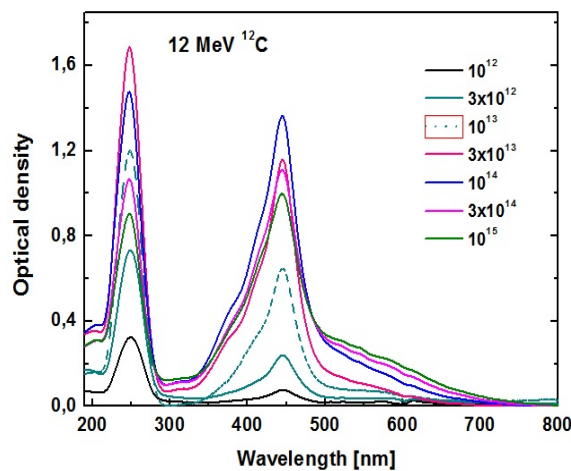


Figure 1. Absorption spectra of LiF crystals irradiated by ^{12}C ions with energy of 12MeV to various fluences.

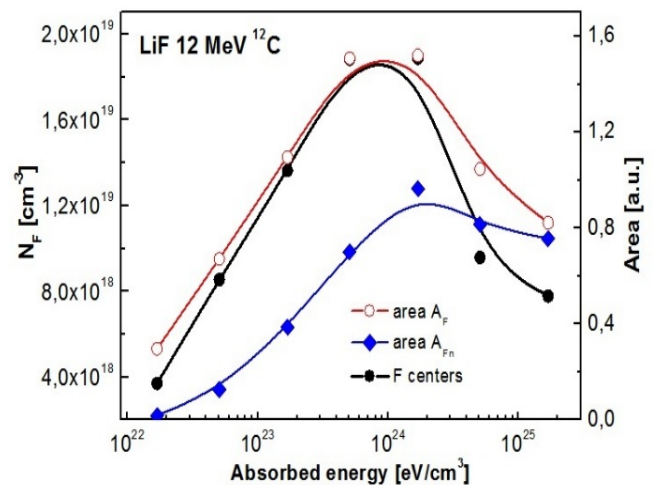


Figure 2. Dependence of concentration of F centers N_F (cm^{-3}) and integral absorption of A_F and A_{F_n} for F and F_n centers as a function of the absorbed energy density E_{abs} (eV/cm^3) in LiF crystals irradiated with 12 MeV ^{12}C ions.

The absorption bands of F_2 and F_3^+ centers (the main F_n centers) overlap greatly, making it difficult to study. For the first time detailed studies of F_2 and F_3^+ centers were carried out by the scientist Nahum [2]. In the future, studies (Skuratov, [4, 5]) established that these colour centres are formed by the reactions: $F + V_{a^+} \rightarrow F_2^+$, $F_2^+ + e \rightarrow F_2$, $F_2 + V_{a^+} \rightarrow F_3^+$. The presence of F_2 and F_3^+ centers can be observed by their luminescence (figure 3). It should be noted that at low optical densities, the concentration of F_2 and F_3^+ centers is proportional to the luminescence intensity. However, this is impeded by a

decrease in the luminescence of both F_2 and F_3^+ centers with increasing fluence, as shown in figures 3 and 4. Quenching of the luminescence of these color centers was observed by Montereali upon irradiation with electrons [6] and Skuratov under irradiation with fast ions [4, 5]. Quenching of luminescence [4, 5] in LiF crystals is associated with radiation-induced mechanical stress, which changes the potential and activation energy of F_2 and F_3^+ centers.

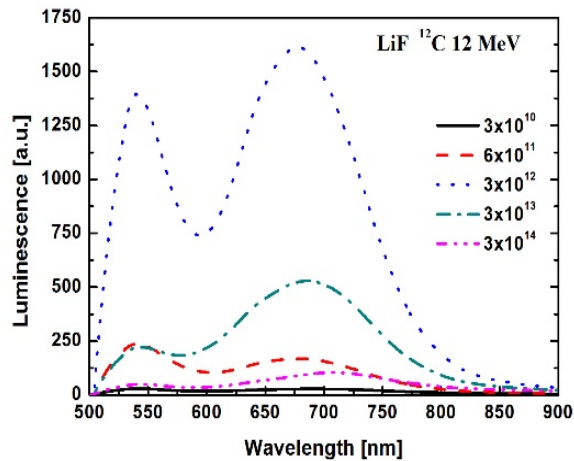


Figure 3. Photoluminescence spectra for F_2 (670 nm) and F_3^+ centers (530 nm) under excitation with 473 nm light for LiF crystals irradiated with 12 MeV ^{12}C ions with different fluences.

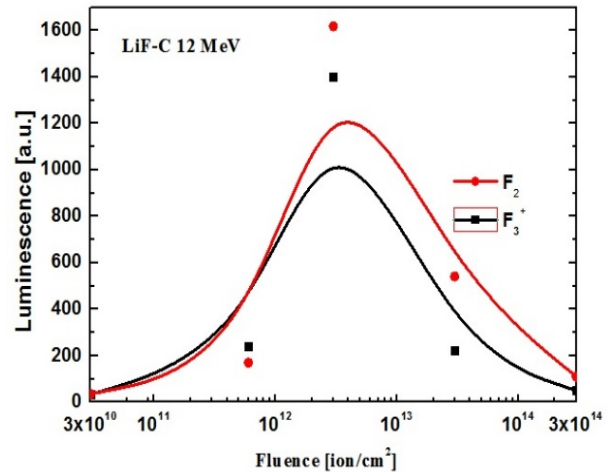


Figure 4. The luminescence intensity for LiF crystals irradiated with ions of ^{12}C with energy of 12 MeV depending on fluence (density of absorbed energy).

The growth of the luminescence of F_2 and F_3^+ centers begins with a fluence of 3×10^{10} ion/cm² for 12 MeV ^{12}C ions, reaches a maximum at 6×10^{12} ion/cm² for F_2 centers, and a maximum for F_3^+ centers is shifted toward lower fluences. At low fluences, F_3^+ centers dominate, but since the fluence is 3×10^{12} ion/cm², F_2 centers dominate. If in the stage of small fluences the processes associated with the capture of anion vacancies that are components of charged Frenkel pairs dominate, then the overlap of tracks contributes to the increase in the fluence, leading to the creation of F_2 centers by the reaction: $F + F \rightarrow F_2$. Starting with fluences $\Phi > 6 \times 10^{11}$ ion/cm², when tracks start overlapping, detectable hardening and the appearance of dislocations are observed (figure 5). The luminescence of the F_2 and F_3^+ centers is sensitive to disturbances in the surrounding structure, and the F_3^+ centers, being charged, are more sensitive, which explains the shift of the intensity maximum towards lower fluences. A general decrease in the complex F_n centers also contributes to this effect.

Nanoindentation tests on the frontal surface oriented normally to the ion beam show a significant hardening effect in LiF crystals when irradiated with 12 MeV carbon ions. The effect of hardening the frontal surface is present with fluence above 6×10^{11} ion/cm², increasing with the fluence and reaching saturation at fluences above 6×10^{13} ion/cm². Nanohardness measurements were made on the surface profile in order to characterize the damage along the ion path. The results presented in figure 5 show the evolution of hardness on the transverse cleavage of samples irradiated with 12 MeV ^{12}C ions to various fluences. Calculated depth profiles for electron energy losses (dotted line) and for nuclear losses (solid line) are presented for comparison. At fluences 5×10^{10} and 10^{11} ion/cm², hardening was not detected. At fluences of 3×10^{11} and 6×10^{11} ion/cm², hardening on the profile surface appeared only in the Bragg maximum region. At high fluences, the hardening effect increases, and its variations along the ion range follow the changes in electron energy losses of the ion, calculated using the SRIM code. This trend is clearly visible in figure 5.

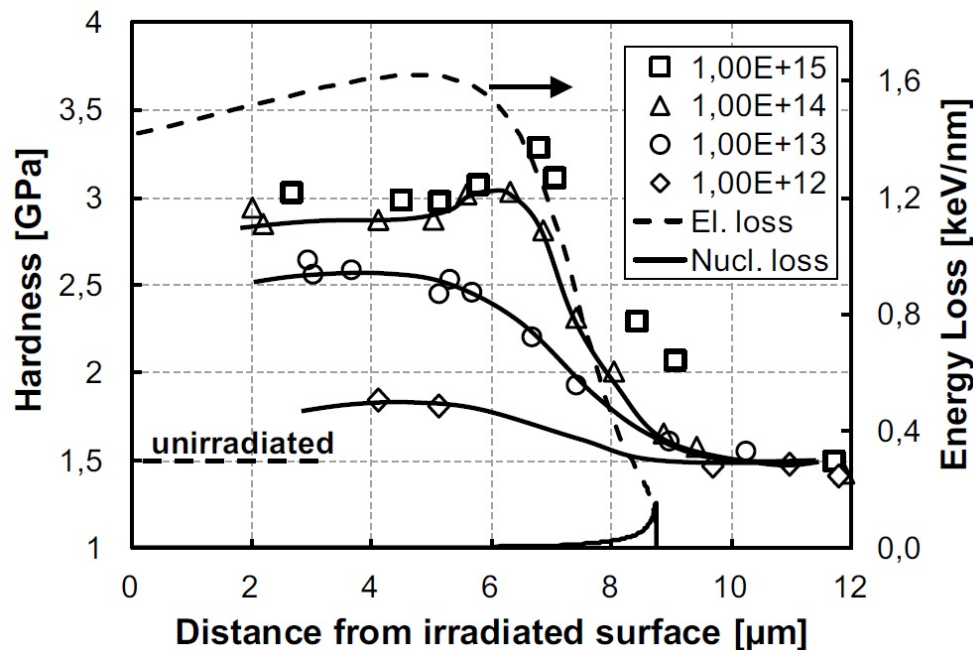


Figure 5. Nanohardness on the surface profile versus depth for LiF irradiated by 12 MeV ^{12}C ions under different fluences [7].

In general, the results of nanoindentation characterize the evolution of lesions on the frontal and profile surfaces. The results show a significant hardening at the end of the ion range, despite the fact that the value of the calculated electron losses decreases to the lowest values. At high fluences, the area of maximum hardening expands to the end of the ion range, where the nuclear energy losses shift to the maximum (figure 5) [7]. We can conclude that irradiation with light ions ^{12}C is capable of creating severe damage and hardening by means of the elastic collision mechanism. The effect is manifested in a narrow zone close to the end of the ion run.

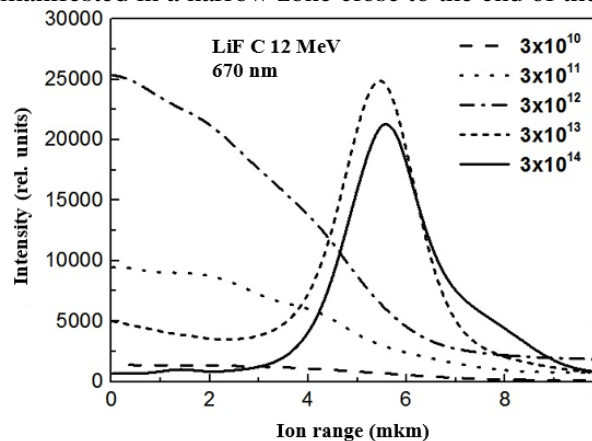


Figure 6. Luminescence of F_2 centers (670 nm) along the ion range as a function of depth and fluence for LiF irradiated with 12 MeV C ions.

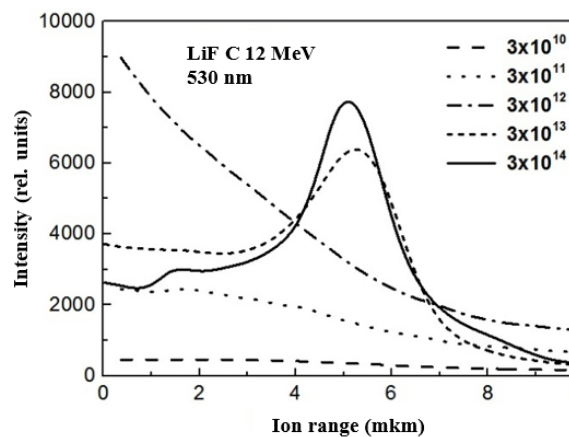


Figure 7. Luminescence of F_3^+ centers (530 nm) along the ion range as a function of depth and fluence for LiF irradiated with 12 MeV C ions.

The luminescence intensity of F_2 and F_3^+ (figures 6 and 7) centers along the ion range grows at the surface from a fluence of 3×10^{10} ion/cm² to 3×10^{12} ion/cm², with a decrease in intensity as ions move inward, that is, these centers are created as a result of the mechanism of electronic energy losses. The

observed attenuation of the luminescence intensity at high fluences is associated with the intense nucleation of dislocations as traps for aggregate color centers. This is also associated with the process of hardening. The luminescence quenching begins at 3×10^{13} ion/cm², and a luminescence burst of both centers is observed at a depth of 6 nm, which corresponds to a sharp decrease in the electron energy losses and to an increase in losses due to elastic collisions, creating strong lesions creating a large number of anion vacancies. The creation of anion vacancies is more preferable than cation vacancies due to the more probable interaction of a positively charged carbon ion. Quenching of the luminescence of aggregate electron color centers occurs as a negative fluorine ion.

Acknowledgments

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