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## Effect of thermic annealing on gamma-induced color centers and activator luminescence in $\text{Lu}_2\text{SiO}_5:\text{Ce}$ scintillator crystals

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Cerium doped lutetium orthosilicate  $\text{Lu}_2\text{SiO}_5:\text{Ce}$  is used as a scintillation detector for nuclear radiation. However, after  $^{60}\text{Co}$ -gamma-irradiation to a high dose  $5 \cdot 10^6$  Gy at 310 K the yield of gamma-luminescence (GL) decreases by 25%. For revealing reasons and mechanism of such degradation, we studied isochronous thermal anneal of the induced optical centers and recovery of the activator GL in the temperature range from 373 to 773 K in air. The irradiation was shown to generate absorption bands at 238 nm ( $F$ -center), 263 ( $\text{Ce}^{3+}/\text{Ce}^{4+}$ ) and 293 nm ( $\text{Ce}^{3+}/F^+$ ) and also broad band at 422 nm, which is supposed to relate to  $\text{Ce}^{4+} + \text{V}_\text{O}$ -defect center and yellow color, causing the decrease in GL bands 399 and 420 nm (Ce1-center) and 450 nm (Ce2-center). At temperatures  $> 573$  K  $F$ -centers and  $\text{Ce}^{4+} + \text{V}_\text{O}$  complexes anneal and the GL recovers up to the origin level, that is due to migration of an interstitial oxygen to an anion vacancy followed by healing the radiation defect.

**Keywords:**  $\text{Lu}_2\text{SiO}_5:\text{Ce}$ , gamma-irradiation, color centers, thermal annealing, gamma-induced luminescence.

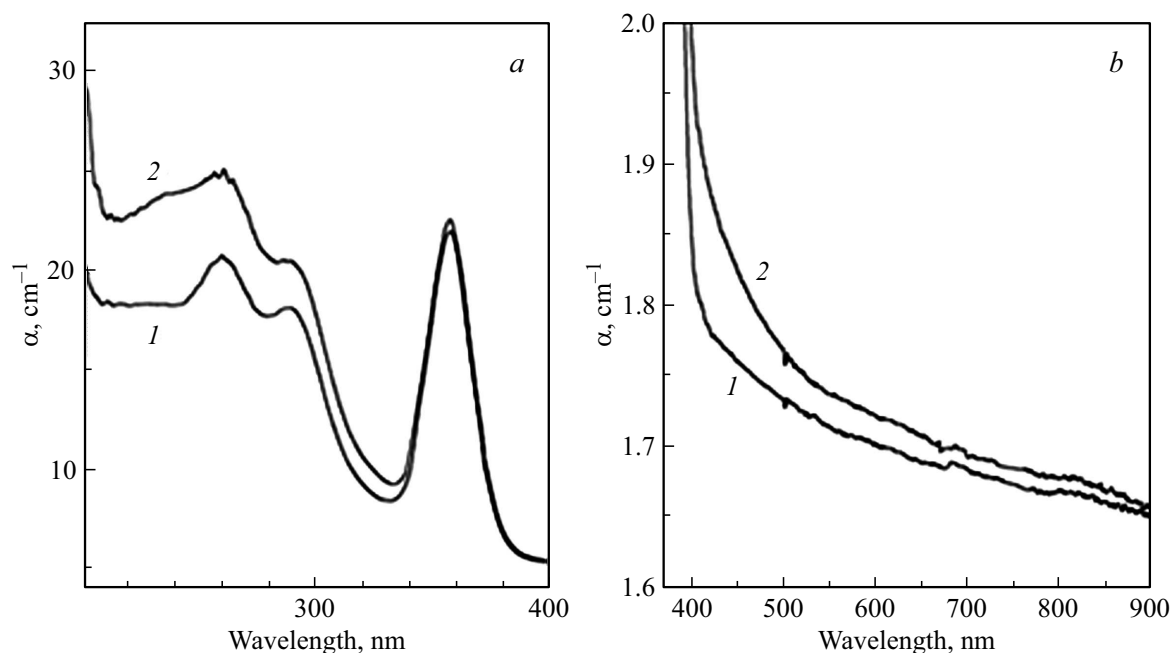
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### Introduction

Cerium-doped lutetium oxyorthosilicate,  $\text{Lu}_2\text{SiO}_5:\text{Ce}$  (LSO:Ce), is used as a scintillator in nuclear medical devices and is promising for nuclear physics and space research [1–3]. LSO:Ce crystal has a high density of  $7.4 \text{ g/cm}^3$ , and therefore a small volume of nuclear particle deceleration; the decay time is about 40 ns, therefore it is a fast-response crystal; light output in the visible range (420 nm) is more than 25,000 ph/MeV, so it has high efficiency; the crystal is excellent for use in various 2D–3D detector devices operating even at room temperature [1–3]. To ensure stable operation under conditions of exposure to nuclear radiation, LSO:Ce crystals should have high optical stability in terms of transmittance and light output. Therefore, studies were carried out on the effect of radiation damage of the LSO:Ce structure on the optical spectra [1–10]. Changes in the absorption and luminescence spectra of LSO:Ce crystals after irradiation with  $^{60}\text{Co}$  gamma-quanta in the dose range of  $70 - 5 \cdot 10^7$  Gy have been studied and it has been shown that the 238 nm band is associated with the  $F^+$ -center (an oxygen vacancy that has captured one electron), and the 263 nm and 293 nm bands are associated with  $\text{Ce}^{3+}/\text{Ce}^{4+}$  and  $\text{Ce}^{3+}/F$  gamma-induced centers ( $F$  is an oxygen vacancy that has captured two electrons), respectively [4]. Based on a detailed structural analysis of the initial crystal [5] an assumption is made that the formation of color centers in the UV region of the spectrum at doses of  $> 5 \cdot 10^4$  Gy and their competition in the capture of charge carriers result in a decrease in

the intensity of 399 nm and 420 nm (Ce1-centers) and 450 nm (Ce2-centers) gamma-luminescence (GL) bands. Studying optical transmission in the visible region of the spectrum from 350 to 700 nm in LSO:Ce with a thickness of 10 mm after gamma-irradiation with a dose of  $10^8$  Rad, the authors of [6] observed a decrease in transmittance in the region of the 420 nm luminescence band. The authors of [7,8] discovered yellow color of some crystals grown in the atmosphere of  $\text{N}_2$ , in the transmission spectra of which (unlike colorless samples) an absorption band with a maximum at 430 nm is detected. After exposure to a gamma-dose of  $2.4 \cdot 10^4$  Rad, some initially colorless samples acquire the same absorption band [7]. The induced absorption band of 430 nm is almost completely removed by thermal annealing in a ( $\text{N}_2$ ) gas flow at  $300^\circ\text{C}$  (573 K) for one day (exact hours are not specified) [6] or by annealing in the air at the same temperature for a time shorter than the 20 h annealing in nitrogen, which is associated with a gamma-induced defect [7]. The authors of [6] believe that the capture centers in the trap may be relatively close to the conduction band, which is formed by the gamma radiation, therefore the thermal annealing restores optical transmission.

The yellow color of the LSO:Ce crystal acquired in the process of Czochralski growth is attributed to lattice defects around/near the  $\text{Ce}^{4+}$  ions [8]. There are different opinions about the yellow color. Analysis of these data shows that the induced absorption band in the region of 420–430 nm is overlapped by scintillation in a wide band of 420 nm, therefore, it can affect the radiation sensitivity of the LSO:Ce



**Figure 1.** Absorption spectra of activated LSO:Ce crystals in (a) UV (with a thickness of 1 mm) and (b) visible regions (3 mm): initial unirradiated (curves 1) and after irradiation with a dose of  $5 \cdot 10^6$  Gy (curves 2).

scintillation crystal. So, clarifying the nature of absorption bands in the 430 nm region and in the UV region of the spectrum, as well as studying their thermal stability can help in finding out the cause of the decrease in the activator luminescence yield after a  $> 5 \cdot 10^4$  Gy dose of gamma-quanta in LSO:Ce [4].

Bearing this in mind, the goal of this study is a comprehensive investigation of the thermal annealing effect on optical absorption and GL in LSO:Ce crystals with thicknesses of 1 mm and 3 mm exposed to a  $5 \cdot 10^6$  Gy gamma-irradiation dose (for the analysis in the UV and visible regions), when there is a noticeable decrease in the activator luminescence yield.

## Object and methods of the study

The samples of LSO:Ce<sup>3+</sup> single crystals (Ce concentration is 0.25 at.%) were grown by the Czochralski method in the nitrogen (N<sub>2</sub>) atmosphere containing 3000 ppm of oxygen (O<sub>2</sub>) (Scintillation Materials Research Center, Department of Materials Science and Engineering, University of Tennessee, Knoxville, USA). The samples were colorless.

For optical studies,  $7 \times 3 \times 1$  mm wafers were cut from one  $15 \times 15 \times 3$  mm boule; two faces ( $7 \times 3$  mm) of the wafers were optically polished.  $7 \times 3 \times 3$  mm samples were already polished.

To limit the effect of the atmosphere on the surface, the samples were wrapped in aluminum foil and irradiated at the Institute of Nuclear Physics of the Academy of Sciences of the Republic of Uzbekistan in a dry channel of the source with <sup>60</sup>Co gamma-quanta with energies of 1.17 MeV and

1.33 MeV at a dose rate of 1.1 Gy/s to a dose of  $5 \cdot 10^6$  Gy at a temperature of 310 K. To prevent possible photobleaching when exposed to daylight, the irradiated samples were stored in aluminum foil from the beginning of irradiation until installation in the closed measuring cell of the spectral device.

Thermal isochronous annealing of LSO:Ce<sup>3+</sup>, irradiated with a gamma dose of  $5 \cdot 10^6$  Gy, was carried out in a muffle furnace with a heating rate of 10 grad/min in successive steps at 373 K, 473 K, 573 K, 673 K, and 773 K in the air. After holding at each temperature for 40 min, the sample was cooled down to room temperature in the switched off furnace.

Optical absorption (OA) spectra before and after irradiation in LSO:Ce<sup>3+</sup> samples with thicknesses of 1 mm and 3 mm were measured at a temperature of 305 K in a SF-56 spectrophotometer (LOMO) in the spectral region of 190–1100 nm. The time interval between the end of irradiation and the start of OA spectra measurements was 10 min.

GL spectra of  $7 \times 3 \times 3$  mm LSO:Ce<sup>3+</sup> were recorded in a measuring cell in the same dry channel of the gamma source at a power of 1.1 Gy/s (during scanning of the GL spectrum in the 200–600 nm spectral region the sample receives a dose of 330 Gy) at a temperature of 310 K with the luminescence being output using polished aluminum tubes and a mirror to the entrance slit of the MDR-12 diffraction monochromator with FEU-39 photomultipliers protected from high-power gamma radiation. To determine the background signal, measurements of the luminescence spectrum are first carried out in an empty cell; then the

background is subtracted from the GL spectrum of the sample.

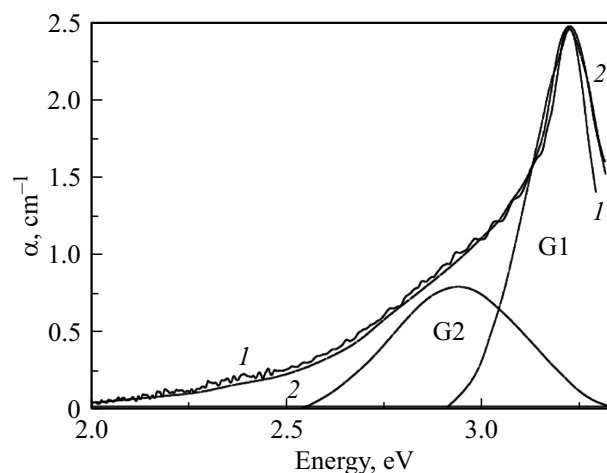
## Results and discussion

In the UV region of the optical spectrum of unirradiated 1 mm thick  $\text{LSO}:\text{Ce}$  samples, partially overlapped absorption bands at 238 nm, 263 nm, and 293 nm are observed, as well as an intense band at 360 nm (Fig. 1, *a*, curve 1), similar to the spectra previously published in [4,9–11] and associated with ( $4f \rightarrow 5d$ ) transitions of the  $\text{Ce}^{3+}$  ion [9–11]. After the gamma-irradiation with a dose of  $5 \cdot 10^6$  Gy, a strong increase in the absorption coefficient  $\alpha$  ( $\text{cm}^{-1}$ ) takes place at wavelengths of 238, 263 and 293 nm, although the separate 360 nm band of the activator  $\text{Ce}^{3+}$ -center remains almost unchanged (Fig. 1, *a*, curve 2).

Fig. 1, *b* shows spectra of unirradiated samples (curve 1) and samples irradiated with a dose of  $5 \cdot 10^6$  Gy (curve 2) with a thickness of 3 mm, where changes in the absorption coefficient  $\alpha$  ( $\text{cm}^{-1}$ ) are more distinct in the  $> 370$  nm region, which covers the scintillation at 420 nm in  $\text{LSO}:\text{Ce}$  crystals [6,7]. After irradiation with a dose of  $5 \cdot 10^6$  Gy, a yellow color appears, an increase in the OA coefficient is observed in the region of 390–550 nm (curve 2) in comparison with the original sample (curve 1). The resulting spectra are similar to the optical transmission spectra upon gamma-irradiation with doses of  $2.4 \cdot 10^4$  Rad ( $2.4 \cdot 10^2$  Gy) [7],  $10^8$  Rad ( $10^6$  Gy) in  $\text{LSO}:\text{Ce}$  [6],  $10^6$  Rad ( $10^4$  Gy) in  $\text{Ce}:\text{Sc}:\text{LPS}$  [12] and in the  $\text{LYSO}:\text{Ce}$  crystal grown by the Czochralski method [13]. Then, we decomposed into Gaussians the differential OA spectra of the initial sample and the sample irradiated with a dose of  $5 \cdot 10^6$  Gy. In the visible region, two bands are identified with maxima at 3.23 eV — 383 nm (G1) and 2.93 eV — 422 nm (G2) (Fig. 2). The obtained result is consistent with the result of decomposition into Gaussians of the differential spectra of the initial sample and the sample gamma-irradiated with a dose of  $2.4 \cdot 10^2$  Gy [7], where two absorption bands, 390 nm and 430 nm, are also distinguished. So, after the exposure to gamma-irradiation with a high dose of  $5 \cdot 10^6$  Gy, the induced wide region of 370–550 nm (Fig. 1, *b*, curve 2) consists of 383 nm and 422 nm OA bands.

It is known that under the elastic action of neutrons [14–16] or protons [17] on nominally pure oxide crystals of  $\text{Y}_3\text{Al}_5\text{O}_{12}$  [14] and  $\text{Lu}_3\text{Al}_5\text{O}_{12}$  [15] with cubic structure,  $\text{Y}_2\text{SiO}_5$  (YSO) [16] and  $\text{Lu}_2\text{SiO}_5$  with  $C2/c$  monoclinic structure with two nonequivalent metal sites [17] oxygen atoms are displaced from anion sites with the formation of paired structural defects (intrinsic defects).

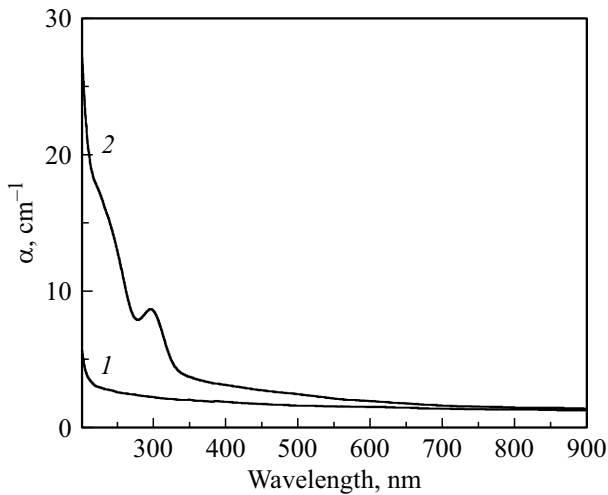
In the OA spectra of a nominally pure YSO crystal after irradiation in a reactor with a fission-neutron fluence of  $10^{17} \text{ cm}^{-2}$ , a wide band is observed in the UV region in the range of 210–340 nm and a more distinct band with a maximum at 300 nm (Fig. 3, curve 2) similar to the results of [16]. In the visible region of the OA spectrum,



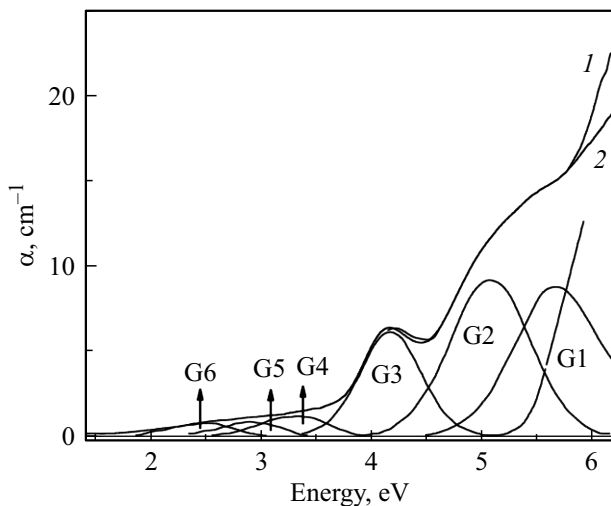
**Figure 2.** Difference spectrum between the absorption spectra of the initial  $\text{LSO}:\text{Ce}$  crystal (Fig. 1, *b*) and the same crystal irradiated with a dose of  $5 \cdot 10^6$  Gy (curve 1), Gaussian components of absorption bands, curves 3.23 eV (G1) and 2.93 eV (G2) and their sum (curve 2).

a wide band is observed in the range of 350–700 nm (Fig. 3, curve 2), which is similar to the spectrum of  $\text{LSO}:\text{Ce}$  after gamma-irradiation with a dose of  $5 \cdot 10^6$  Gy (Fig. 1, *b*, curve 2), but 2 times more intense. Based on the decomposition into Gaussians after irradiation with protons [17] we also decomposed into Gaussians the differential OA spectrum of the initial sample and the sample irradiated with a neutron fluence of  $10^{17} \text{ cm}^{-2}$ . In the UV and visible regions, six bands are distinguished with maxima at 5.63 eV — 220 nm (curve 3), 5.08 eV — 242 nm (curve 4), 4.18 eV — 296 nm (curve 5), 3.25 eV — 380 nm (curve 6), 2.9 eV — 426 nm (curve 7), and 2.4 eV — 516 nm (curve 8) (Fig. 4). This result also confirms our earlier assumptions made in [4] that the 238 nm band is associated with the  $F$ -center, and the 293 nm OA band associated with ( $4f - 5d$ ) transitions of the  $\text{Ce}^{3+}$  ion is superimposed on the  $F^+$ -center band. So after irradiation with a neutron fluence of  $10^{17} \text{ cm}^{-2}$ , the OA bands of 3.25 eV (380 nm) and 2.9 eV (426 nm) observed in the nominally pure YSO do not depend on the presence of the Ce activator ion, and therefore are possibly associated with structural defects in the oxygen sublattice.

In [18], theoretical calculations of the lattice parameters of undoped and doped  $\text{LSO}:\text{Ce}$  (substitutions in  $\text{Ce}_{\text{Lu}1}$  and  $\text{Ce}_{\text{Lu}2}$  cation sites) were carried out, which showed that the bond lengths of the  $\text{Ce}_{\text{Lu}1}$  site with 6 nearest oxygen atoms are increased by 0.125–0.177 Å, and those of the  $\text{Ce}_{\text{Lu}2}$  are increased by 0.126–0.169 Å compared to undoped LSO at Lu sites. These bond elongations are qualitatively consistent with the difference of  $\sim 0.16$  Å between the ionic radii of  $\text{Ce}^{3+}$  and  $\text{Lu}^{3+}$  in the same coordination [18,19]. In such structurally stressed impurity substitution sites in doped  $\text{LSO}:\text{Ce}$ , the formation of defects under irradiation with gamma-quanta can be energetically



**Figure 3.** Absorption spectra of a YSO crystal (thickness 1 mm): initial crystal (curve 1) and crystal after irradiation with a neutron fluence of  $10^{17} \text{ cm}^{-2}$  (curve 2).



**Figure 4.** Difference spectrum between the absorption spectra of the YSO crystal shown in Fig. 2: initial crystal and crystal after irradiation with a neutron fluence of  $10^{17} \text{ cm}^{-2}$  (curve 1), Gaussian components of the absorption bands of the curve 1 — 5.63 (G1), 5.08 (G2), 4.18 (G3), 3.25 (G4), 2.9 (G5), and 2.4 eV (G6); their sum — curve 2.

easier. Therefore, we believe that, along with the OA bands in the UV region at 238 nm ( $F$ -center) and 293 nm ( $\text{Ce}^{3+}/F^+$ -centers) (Fig. 1, *a*, curve 2), the 430 nm band is associated with oxygen vacancy defects ( $V_O$ ) around the Ce impurity. That is, the Ce activator in LSO may contribute to the formation of yellow color centers for the 390 nm and 430 nm OA bands when irradiated with gamma-quanta with a dose of  $\geq 10^5 \text{ Gy}$ . In the following text we use a simplified denomination of these centers as  $\text{Ce}^{4+}+V_O$ -center, according to [8]. In our opinion, this is due to the increase in the thermal luminescence (TL) peak at 540 K up to the dose of  $5 \cdot 10^7 \text{ Gy}$  [4].

The decrease in the intensity of the GL band of the activator  $\text{Ce}^{3+}$ -center, the appearance of an additional TL peak at 375 K and the increase in the TL peak at 540 K with higher gamma-irradiation doses ( $> 5 \cdot 10^4 \text{ Gy}$ ) are presumably attributed to the color centers induced by gamma-quanta [4].

Then, the effect of thermal annealing on the gamma-induced color centers associated with defects of  $F$  (238 nm),  $\text{Ce}^{3+}/\text{Ce}^{4+}$  (263 nm),  $\text{Ce}^{3+}/F^+$  (293 nm) and  $\text{Ce}^{4+}$  (430 nm), as well as on the activator luminescence of LSO:Ce.

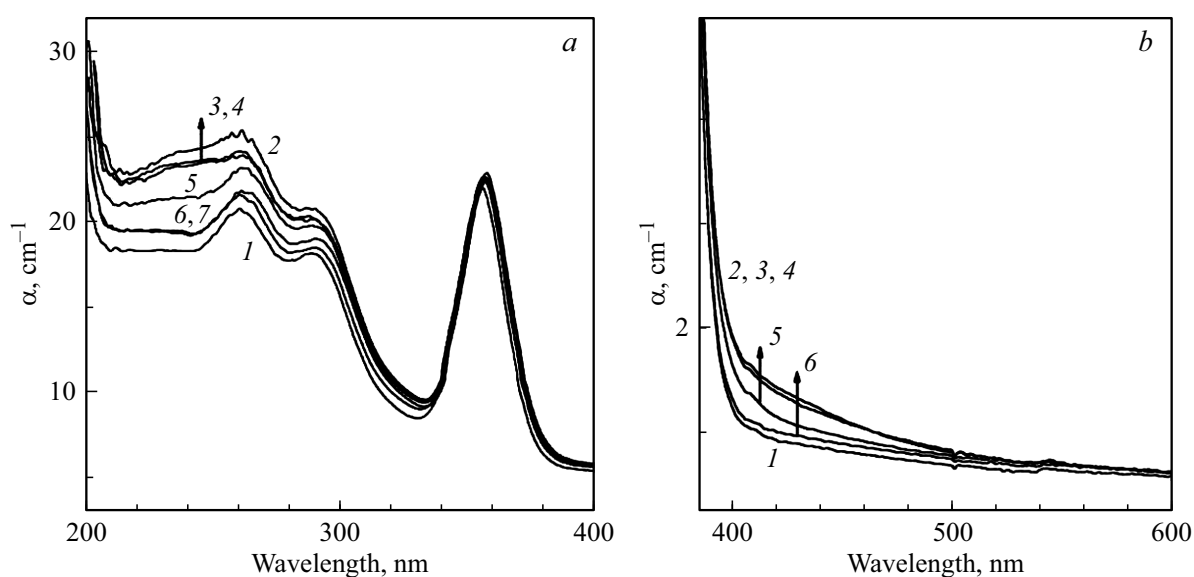
Fig. 5, *a, b* shows the comparative OA spectra of the initial sample (curve 1), the gamma-irradiated sample with a dose of  $5 \cdot 10^6 \text{ Gy}$  (curve 2) and the sample after thermal annealing at 373 K and 473 K (curve 3), 573 K (curve 4), 673 K and 773 K (curve 5). As can be seen from Fig. 5, *a*, a significant decrease in the absorption coefficients of the 238 nm, 263 nm, and 293 nm OA bands is observed after annealing at a temperature of 573 K (curve 5), and after annealing at 673 K and 773 K, the absorption coefficient of the 238 nm band ( $F$ -center) decreases almost down to the level of the initial sample (curves 6, 7). It should be noted that the absorption coefficient of the 360 nm OA band of the activator  $\text{Ce}^{3+}$ -center in the studied annealing temperature range of up to 773 K remains almost unchanged (curves 1–7).

Changes in OA coefficients of the visible 430 nm band with increasing annealing temperature from 373 K to 773 K (Fig. 5, *b*, curves 3–5) are the same as those in the UV region (Fig. 5, *a*, curves 3–6). It should be noted that at an annealing temperature of 573 K, optical centers associated with  $F$  (238 nm),  $\text{Ce}^{3+}/\text{Ce}^{4+}$  (263 nm),  $\text{Ce}^{3+}/F^+$  (293 nm), and  $\text{Ce}^{4+}+V_O$  (430 nm) defects (curve 5) are not completely annealed. That is, the thermal release of captured electrons does not occur from all traps, which appear in TL curves in the region from 300 K to 600 K [4,7,10]. The complete thermal annealing of these color centers occurs at the annealing temperature of  $\geq 673 \text{ K}$  (Fig. 5, *a, b*, curves 5 and 6).

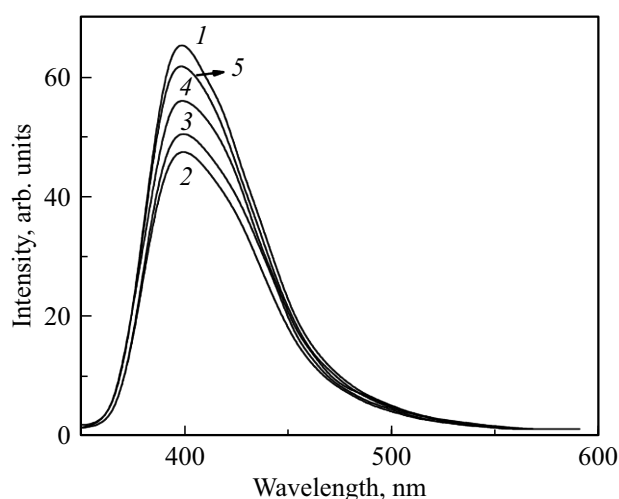
Then, we continued to study the effect of thermal annealing on the GL yield of the  $\text{Ce}^{3+}$  activator luminescence with maxima at 399 nm and 420 nm associated with Ce1-centers (coordinated by seven oxygen ions); 450 nm — with Ce2-centers (coordinated by six oxygen ions), caused by the  $d-f$  transition of  $\text{Ce}^{3+}$  ions [20].

Fig. 6 shows the results of GL (during the GL spectrum scanning, a dose of 300 Gy is accumulated) at 310 K for the initial sample (curve 1) and for the gamma-irradiated sample with a dose of  $5 \cdot 10^6 \text{ Gy}$  (curve 2), as well as for the sample after annealing at 373 K and 473 K (curves 3, 4), 573 K (curve 5) and 673 K and 773 K (curves 6, 7). The GL yield in 399 nm, 420 nm, and 450 nm bands after the irradiation with a critical dose of  $5 \cdot 10^6 \text{ Gy}$  (curve 2) decreased by  $\approx 25\%$  of the GL yield of the initial sample (curve 1).

After the thermal annealing in the temperature range of 373–773 K, an increase is observed in the intensity



**Figure 5.** Absorption spectra of activated LSO:Ce crystals at different scales of  $\alpha$  coefficient: (a) UV region (thickness 1 mm) and (b) visible region (thickness 3 mm); initial crystal (curves 1), crystal after gamma-irradiation with a dose of  $5 \cdot 10^6$  Gy (curves 2), crystal after annealing at temperatures of 373 K (curves 3), 473 K (curves 4), 573 K (curves 5), 673 K (curves 6), and 773 K (curves 7).



**Figure 6.** Gamma-luminescence spectra of activated LSO:Ce crystal (thickness 3 mm): initial crystal (curve 1), crystal after gamma-irradiation with a dose of  $5 \cdot 10^6$  Gy (2) and crystal after annealing at temperatures of 373 K, 473 K (curve 3), 573 K (4) and 673 K, 773 K (5).

of the GL bands at 399 nm, 420 nm and 450 nm of the  $\text{Ce}^{3+}$ -center (curve 4) to the unirradiated level (curve 1). Thus, the restoration of the initial GL yield of the  $\text{Ce}^{3+}$  activator luminescence (Ce1 and Ce2 centers) in a sample irradiated with a critical dose with an increase in its annealing temperature to 773 K (Fig. 5, curves 3–5) is associated with the annealing of competing gamma-induced defective color centers of  $F^+$  (238 nm),  $F$  (293 nm), and  $\text{Ce}^{4+}+\text{V}_\text{O}$  (430 nm) (see OA spectra in Fig. 5, a, b).

## Conclusion

A comparative study is conducted of the optical properties (absorption and luminescence) of lutetium orthosilicate crystals activated by cerium ions (LSO:Ce) before and after gamma irradiation ( $^{60}\text{Co}$ ) with a dose of  $5 \cdot 10^6$  Gy at 310 K, as well as after thermal annealing of gamma-induced optical centers in the temperature range from 373 to 773 K in air for 40 min.

It has been shown that after LSO:Ce irradiation with a dose of  $5 \cdot 10^6$  Gy, in the OA spectrum, along with the 238 nm ( $F$ ), 263 nm ( $\text{Ce}^{3+}/\text{Ce}^{4+}$ ), and 293 nm ( $\text{Ce}^{3+}/F^+$ ), an absorption band of 422 nm is also recorded, which is presumably associated with a defective  $\text{Ce}^{4+}+\text{V}_\text{O}$ -center and is responsible for the observed yellow color of the irradiated samples.

We believe that, taking into account the TL curves in the temperature range from 300 to 600 K recorded in [4,7,10], the decreases in the concentrations of  $F$ ,  $\text{Ce}^{3+}/\text{Ce}^{4+}$ , and  $\text{Ce}^{3+}/F^+$  UV centers observed in the OA spectra after thermal annealing at temperatures up to 573 K of the LSO:Ce crystal gamma-irradiated with a dose of  $5 \cdot 10^6$  Gy may be associated with the thermal release of electrons not only from UV centers but also from  $\text{Ce}^{4+}+\text{V}_\text{O}$  (422 nm) color centers, which radiatively recombine with the hole  $\text{Ce}^{4+}$ -center in the spectral region of 390–450 nm. Therefore, we can not rule out the process of partial reabsorption of this luminescence by  $\text{Ce}^{3+}$  color centers in the 422 nm absorption band ( $\text{Ce}^{4+}+\text{V}_\text{O}$ ), which is observed at irradiation doses of  $\geq 5 \cdot 10^4$  Gy and is not completely annealed at temperatures up to 573 K.

At isochronous annealing temperatures of  $> 573$  K, gamma-induced interstitial oxygen diffuses to the nearest

anion vacancy and recombines (Frenkel pair recovery in the anion sublattice). Consequently, this process leads to the annealing of  $F$ ,  $Ce^{3+}/Ce^{4+}$ , and  $Ce^{3+}/F^+$  UV centers, as well as  $Ce^{4+}+V_O$  centers and the yellow color. All these processes lead to the recovery of GL yield of 399 nm, 420 nm, and 450 nm bands of the  $Ce^{3+}$ -center to the level of the initial state.

It is possible to get rid of gamma-induced color centers in LSO:Ce scintillators, noticeable after irradiation doses of  $> 5 \cdot 10^4$  Gy up to a critical dose of  $5 \cdot 10^6$  Gy, with a loss of 25% of the yield, which lead to a decrease in the yield of activator luminescence [4], if thermal annealing is carried out at  $> 573$  K for 60 min in the air. Uniform annealing in the scintillator volume is ensured by an increase in its duration with increasing volume; for example, a 20 mm thick crystal was annealed for one day at  $300^\circ\text{C}$ ,  $360^\circ\text{C}$ , and  $460^\circ\text{C}$  [6,7]. In the above-mentioned dose range of  $5 \cdot 10^4 - 5 \cdot 10^6$  Gy, it is recommended to introduce a correction for a decrease in GL yield from 0.10 to 0.25 according to the dose [4].

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### Conflict of interest

The authors declare that they have no conflict of interest.

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