



# Radiation effects in $\text{Gd}_3(\text{Al,Ga})_5\text{O}_{12}:\text{Ce}^{3+}$ single crystals induced by swift heavy ions

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## ABSTRACT

Radiation effects in cerium doped  $\text{Gd}_3(\text{Al,Ga})_5\text{O}_{12}$  (or GGAG) single crystals irradiated by swift heavy ions with fluences ranging from  $6 \cdot 10^{10}$  to  $2 \cdot 10^{12}$  ions/cm<sup>2</sup> have been studied. A stable strong induced absorption observed in the spectral range 200–350 nm correlates with the irradiation fluence. It is suggested that several centers are responsible for this induced absorption in GGAG single crystals and their possible origin (F-type centers and V-centers or holes trapped near cation vacancies) is proposed and discussed. The swift heavy ions irradiation strongly modifies the luminescence properties of GGAG, namely, the excitation spectra of the  $\text{Ce}^{3+}$  emission, which have been measured over a wide spectral range including vacuum ultraviolet diapason. In particular, it was shown that the formation of the stable radiation defects under swift heavy ions irradiation leads to the effective  $\text{Ce}^{4+} \rightarrow \text{Ce}^{3+}$  transformation in the  $\text{Mg}^{2+}$  co-doped GGAG single crystals. The reasons leading to the alteration in the luminescence properties of irradiated GGAG single crystals are elucidated and discussed.

## 1. Introduction

Cerium-doped  $\text{Gd}_3(\text{Al,Ga})_5\text{O}_{12}$  (or GGAG) is one of the promising scintillator materials for practical applications in high-energy physics and medicine. The interest in this compound is explained by an incredibly high scintillation light yield (58,000 photons/MeV) [1], high density (6.63 g/cm<sup>3</sup>), and relatively fast decay time (90 ns) [2]. However, the most essential drawback of this compound is a long afterglow of  $\text{Ce}^{3+}$  emission, which is explained by the influence of shallow traps which re-trap charge carriers during their transfer to the  $\text{Ce}^{3+}$  ions. Two approaches are considered how to improve the luminescence properties of GGAG: cation substitution or co-doping. As examples of cation substitutions  $\text{Gd}^{3+}$  replacement by  $\text{Lu}^{3+}$  and/or  $\text{Y}^{3+}$  [3] or  $\text{Ga}^{3+}$  substitution by  $\text{Sc}^{3+}$  [4] should be mentioned. In respect of the co-doping, it should be noted that cerium-doped GGAG is usually co-doped by divalent ions ( $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Ba}^{2+}$ ), which accelerate  $\text{Ce}^{3+}$  emission decay [5,6]. In addition, the co-doping of GGAG by tetravalent ( $\text{Zr}^{4+}$ ) [7,8] and

monovalent ( $\text{Li}^+$ ) [9] ions has been recently reported too.

Along with luminescent properties, the radiation resistance of the scintillator plays an important role in practical applications because scintillators as ionizing radiation detectors are naturally subjected to radiation influence. Considering that GGAG is a relatively new compound, its radiation resistance against ionizing radiation is still poorly known. In order to fill this knowledge gap, we have studied in current research the GGAG:Ce co-doped by  $\text{Mg}^{2+}$  and irradiated by swift heavy ions. Such irradiation is capable to produce radiation defects similar to those generated by neutrons i.e., it is a good alternative to neutron irradiation, which needs a significantly longer time for samples' relaxation after neutron treatment [10–14]. Therefore, it is suggested that the irradiation by swift heavy ions produces stable radiation defects in GGAG crystal lattice which subsequently are studied by means of optical and luminescence spectroscopy. The influence of radiation defects on optical and luminescence properties of irradiated samples has been demonstrated and the origin of the induced radiation defects is discussed

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## 2. Experimental

The  $\text{Gd}_3\text{Ga}_3\text{Al}_2\text{O}_{12}$  (GGAG) single crystals have been grown by the Czochralski method in OJSC “Fomos-Materials” (Moscow, Russia). The details of growth procedure of doped and co-doped GGAG single crystals have been reported in Ref. [15]. The crystals have been cut in small thin pieces and polished for optical experiments. The cerium concentration is 3 at%, while the concentration of magnesium is 350 ppm.

The crystals were irradiated with 156 MeV Xe ions to fluences from  $6.6 \cdot 10^{10}$  to  $2 \cdot 10^{12} \text{ cm}^{-2}$  at the IC-100 at FLNR JINR (Dubna, Russia). Ion beam homogeneity over the irradiated specimen surface was controlled using beam scanning in the horizontal and vertical directions and was better than 10%. Average Xe ion fluxes were less than  $10^8 \text{ cm}^{-2}\text{s}^{-1}$  in order to avoid any significant heating of the specimens. To widen the ion energy range, Al degraders of varying thicknesses have been used. Using SRIM calculations the penetration depth of the Xe ions into GGAG is estimated as 9.88  $\mu\text{m}$ .

The radiation-induced defects in the GGAG:Ce,Mg single crystals have been studied by means of optical spectroscopy (spectrophotometer Cary 7000). The irradiated crystals have also been studied utilizing luminescence spectroscopy (spectrometer FLS 1000 from Edinburgh Instruments). These experiments have been carried out at room temperature (RT). In addition, the samples have been studied under synchrotron radiation excitations in vacuum ultraviolet spectral range. These experiments have been carried out at Superlumi setup recently installed on the P66 beamline of PETRA III storage ring at DESY synchrotron facility (Hamburg, Germany). Some details of this experimental setup can be found elsewhere [16]. The excitation and reflection spectra were measured in 330–100 nm excitation range at low temperature (12 K). These spectra have been corrected in respect of the sodium salicylate signal.

## 3. Results and discussion

Transmission spectra of the virgin and irradiated GGAG:Ce,Mg single crystals measured in 200–800 nm spectral range are shown in Fig. 1 (a), while spectra details in the 200–350 nm region are depicted in Fig. 1 (b). The transmission spectra for all samples reveal well-recognized absorption bands in cerium-doped GGAG. The absorption bands due to 4f–5d transitions within a  $\text{Ce}^{3+}$  ion are peaked at 440 nm ( $4f-5d_1$ ) and 343 nm ( $4f_1-5d_2$ ). In addition, two series of  $\text{Gd}^{3+}$  sharp lines due to f-f transitions can be resolved (Fig. 1 (b)).

Comparing the transmission spectra of the initial and irradiated samples, one can see a gradual deterioration of transparency in the spectral region of 200–350 nm with an increase in the radiation fluence. In order to analyze the induced absorption we have transformed the transmission spectra into absorption spectra (Fig. 2a) with the subsequent plotting of the difference of optical densities for virgin and

irradiated GGAG:Ce,Mg single crystals in Fig. 2b (i.e. the spectra of radiation-induced optical absorption). This picture (Fig. 2b) clearly shows that the induced absorption is highly dependent on the irradiation fluences – if irradiation fluence increases, the induced absorption becomes more intense. The main induced absorption band in the region 210–250 nm (Fig. 2) reaches the saturated value of optical density of 4.0 if irradiation fluence is higher than  $6 \cdot 10^{11} \text{ ions/cm}^2$ . This saturation of optical density is explained by the obvious technical/experimental limit of the spectrometer utilized. In addition to the main induced absorption band, there is also less intensive induced absorption in the spectral range 250–340 nm, which also increases under higher irradiation fluences.

Obviously, the broad induced absorption observed in Fig. 2 is the result of the overlapping of several absorption bands, which belong to several types of radiation defect centers in GGAG. The identification of these centers by means of EPR is strongly limited because of  $\text{Gd}^{3+}$  ion in the lattice, which has a strong EPR signal hiding all other possible EPR signals. Therefore, at the current stage we can only make a qualitative analysis by comparing the induced absorption in our GGAG single crystals with those observed in other garnets, for instance in  $\text{Y}_3\text{Al}_5\text{O}_{12}$  (or YAG) and in  $\text{Gd}_3\text{Ga}_5\text{O}_{12}$  (or GGG). In these garnets, it is well established that the main radiation defects are F-type centers, which are, in fact, oxygen vacancies with two or one trapped electrons. It is known that the F-center (two electrons trapped by oxygen vacancy) in YAG has absorption bands at 240 nm [17–19] and 195 nm [18,19], while the  $\text{F}^+$ -center (an electron trapped by a vacancy) reveals absorption bands at 230 nm and 370 nm [19,20]. In the case of the GGG compound, the literature data exhibit that  $\text{F}^+$ -center has absorption energies at about 300 nm [21–26], which is the most high-energy absorption band in GGG single crystals. Therefore, the induced absorption spectra observed in GGAG (Fig. 2) have more similarities with the absorption spectra of F-type centers in YAG rather than in GGG.

If we make a comparative analysis of the absorption bands of radiation defects in GGAG and YAG, we can trace some analogy keeping in mind that lattice parameters for these compounds are 12.6 Å and 11.9 Å respectively. These parameters are very close to each other. Therefore, if we determine the absorption energy of the  $\text{F}^+$ -center using the *Mollwo-Ivey* law [19], we can predict the similar absorption values for YAG and GGAG. The *Mollwo-Ivey* law is not applicable for the determination of absorption energies of F-centers, nevertheless, we suppose that their absorption energies in YAG and GGAG will be also similar. Therefore, we suggest that the induced absorption at 200–250 nm in Fig. 2 is the result of the overlapping of several close absorption bands belonging to the F and  $\text{F}^+$  in GGAG.

In addition to the intensive absorption in the 200–250 nm spectral range, there is less intensive absorption at 250–350 nm. Usually, less intensive absorption can be explained by the absorption of hole-type centers. Absorption of hole centers as a rule is weaker than that of the electron centers (F-type centers) because of much lower oscillator strength value for the transitions in hole centers. It is known that the cation vacancy  $V_{\text{Ga}}$  in GGG crystals has an absorption band peaking at

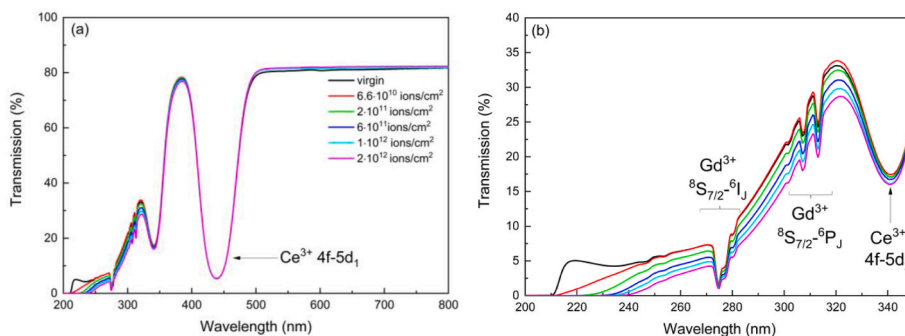
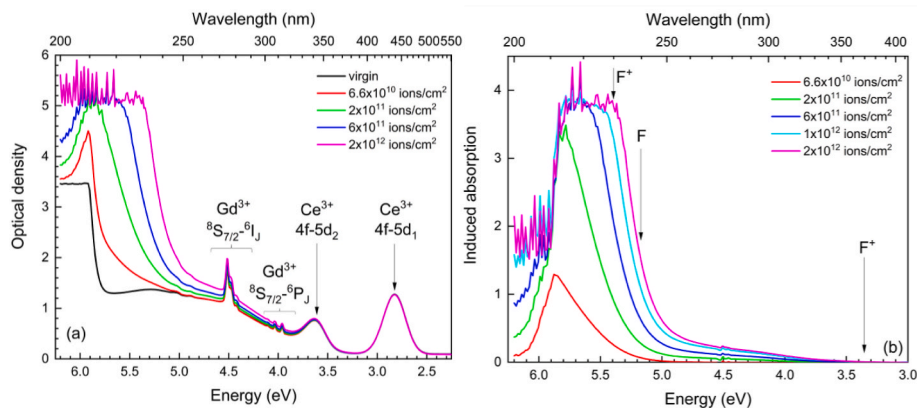


Fig. 1. Transmission spectra of virgin and irradiated GGAG:Ce,Mg single crystals under different fluences (a). The fluences (in  $\text{ions/cm}^2$ ) are shown in the legends. The high-energy spectral region is shown in detail in (b).

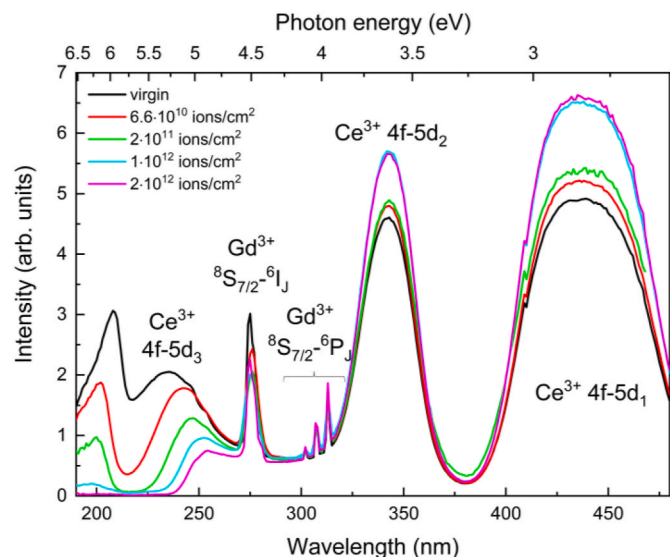


**Fig. 2.** Absorption spectra of virgin and irradiated GGAG:Ce,Mg single crystals under different fluences (a). Difference spectra are given in (b): optical densities of a virgin sample are subtracted from those for the irradiated GGAG:Ce,Mg crystals at different fluences (in ions/cm<sup>2</sup>). The arrows indicate the spectral positions of absorption bands of F-type centers in a YAG single crystal (b).

270 nm [21,22]. Moreover,  $V_{Ga}$  in garnet structure can be a site of a bound hole  $O^-$  center localization. In GGG and YAG, the absorption of  $O^-$  center localized at a cation sublattice defect lies in the region of 310–340 nm [21,27]. In other complex oxides,  $O^-$  center near a cation vacancy is also well known and reveals absorption in a wide spectral range [28–30].

Taking into account that GGAG:Ce,Mg is a scintillator material, the influence of radiation damage on the luminescence properties of this compound is vital. The typical yellow-green luminescence band due to the 5d-4f radiation transitions in  $Ce^{3+}$  has been observed in all samples studied. The shape of the  $Ce^{3+}$  emission band, which is a goal of practical applications, remains unchanged after irradiations independently of the irradiation fluences. Fig. 3 exhibits the comparison of the excitation spectra of  $Ce^{3+}$  emission in virgin and irradiated crystals. The excitation spectra have been measured at room temperature by means of the FLS-100 spectrometer. The crystals have the same shape and have been measured at identical light geometry. Thus, the comparison of the excitation spectra depicted in Fig. 3 is done without spectra normalization.

The excitation spectrum observed in the non-irradiated sample reveals three bands which are explained as intrinsic (intra-center) excitation of a  $Ce^{3+}$  ion. The excitation bands at about 440 nm and 340 nm



**Fig. 3.** The excitation spectra of  $Ce^{3+}$  emission monitored at 550 nm at room temperature in virgin and irradiated GGAG:Ce,Mg single crystals under different fluences (the fluences in ions/cm<sup>2</sup> are shown in legend).

are resulting from the 4f-5d<sub>1</sub> and 4f-5d<sub>2</sub> transitions, respectively. These excitation bands spectrally coincide with the corresponding absorption bands presented in Fig. 1. The third excitation band is due to 4f-5d<sub>3</sub> transitions and is located in the spectral range 220–270 nm. The sharp  $Gd^{3+}$  lines in the excitation spectra are, in principle, the same as the corresponding lines in the absorption spectra in Fig. 1.

Analyzing the excitation spectra in Fig. 3 for the irradiated crystals one can see that the intensity of the 4f-5d<sub>1</sub> and 4f-5d<sub>2</sub> excitation bands becomes higher if the irradiation fluence is increasing up to  $1 \cdot 10^{12}$  ions/cm<sup>2</sup> with subsequent saturation at higher fluence ( $2 \cdot 10^{12}$  ions/cm<sup>2</sup>). The rise in the intensity of the 4f-5d<sub>1</sub> and 4f-5d<sub>2</sub> excitation bands is explained by the increase of the concentration of  $Ce^{3+}$  ions in irradiated GGAG. The GGAG single crystals were intentionally co-doped by  $Mg^{2+}$  ions in order to convert some amount of  $Ce^{3+}$  ions into the  $Ce^{4+}$  charge state. It means the as-grown (virgin) crystals contain some amount of  $Ce^{4+}$  ions to compensate for the lack of positive charge induced by  $Mg^{2+}$ . Under swift heavy ion irradiation, the radiation defects in the crystal lattice are created. As we discussed above, one of the most probable radiation defects in GGAG lattice is the  $F^+$ -center. Direct collision of a swift heavy ion causes the displacement of an oxygen ion from its regular site into an interstice and forming an oxygen vacancy. The oxygen vacancy is positively charged in respect of the regular lattice and two electrons are needed to compensate for this positive charge. If the oxygen vacancy captures only one electron, the  $F^+$ -center is formed. The  $F^+$ -center is a stable radiation defect detected in many binary oxides and complex oxides [19,31]. The creation of neutral F-type centers and complementary interstitials will be most probable due to the absence of Coulomb attraction between them. However,  $F^+$  center neighboring  $Mg^{2+}$  ion compensates a lack of positive charge instead of recharging of  $Ce^{3+}$  into  $Ce^{4+}$ .

The explanation of the transformation of  $Ce^{4+}$  into  $Ce^{3+}$  considered above is, of course, speculative and needs to be proven experimentally. Moreover, this model is intricate because it implies recharging of an interstitial oxygen. Thus, quite possible, that under swift heavy ion irradiation a defect in cation sublattice can also be formed and such defect is a favorable center for a hole localization. In this case an electron can be captured by  $Ce^{4+}$  forming a stable  $Ce^{3+}$  center nearby. We admit that this model, as well as the model described above, are speculative and further studies are needed. However, at the current stage we would like to fix the experimental fact: the concentration of  $Ce^{3+}$  centers in irradiated GGAG:Ce,Mg crystals increases. It is worth noting that the recharging of cerium ions, in principle, is possible under ionizing irradiation. However, under X-ray,  $\gamma$ -ray and low-energy particle irradiations the stable conversion of  $Ce^{4+}$  to  $Ce^{3+}$  has not been reported in the literature so far.

In contrast to the excitation bands considered before, the

dependence of the 4f-5d<sub>3</sub> excitation band on the irradiation fluence is the opposite – the transition intensity decreases with increasing irradiation fluence. In addition, this excitation band has a modified shape if compared with the corresponding band for the virgin crystal. The fact is that the 4f-5d<sub>3</sub> excitation band overlaps with another strong induced absorption band depicted in Fig. 2. Thus, induced absorption re-absorbs the excitation intended for the Ce<sup>3+</sup> and suppresses the intensity of the 4f-5d<sub>3</sub> excitation.

Another important excitation range in Fig. 3 is the excitonic region. The excitonic band at about 200 nm degrades strongly in the highly irradiated crystals. However, this band's position is close to the spectral limit of the FLS 100 spectrometer. Therefore, the same crystals have been examined on the Superlumi setup, which allows to extend photo-excitation to the vacuum ultraviolet (VUV) spectral range. Moreover, the excitation spectra have been measured at low temperature and the results are summarized in Fig. 4.

The excitation spectra in Fig. 4 at low excitation energies are, in principle, the same as the spectra measured at room temperature and shown in Fig. 3. The excitation of the Ce<sup>3+</sup> luminescence after energy transfer from the host lattice starts at energies higher than 6 eV or 200 nm. The excitation intensity increases if the excitation energy is higher than 11 eV due to the so-called multiplication of electronic excitation processes [32–42]. The VUV excitation spectra for co-doped GGAG single crystals have been recently reported and discussed in Refs. [7,8]. However, in the current study the influence of radiation damages on the excitation spectra in VUV range is important for us. Fig. 4 exhibits a strong degradation/suppression of the excitation spectra in irradiated crystals and this degradation is strongly dependent on the irradiation fluence.

In our opinion, the degradation of the excitation spectra in VUV is due to surface loss processes when generated charge carriers are effectively trapped at the surface and/or imperfections instead of their transfer to the luminescence center – a Ce<sup>3+</sup> ion. Swift heavy ions have a penetration depth of about 10 μm, while the penetration depth of the VUV photons in the fundamental absorption region is several tens of nanometers. Therefore, the excitation spectra shown in Fig. 4 are measured in a highly damaged area close to the crystal surface.

Swift heavy ions irradiation modifies optical properties of the irradiated crystals in the UV-VUV spectral range. Fig. 5 shows the comparison of the reflection spectra in the UV-VUV spectral range for irradiated crystals. Taking into account that the reflection spectra have been measured from the polished surfaces, the structure of the reflection

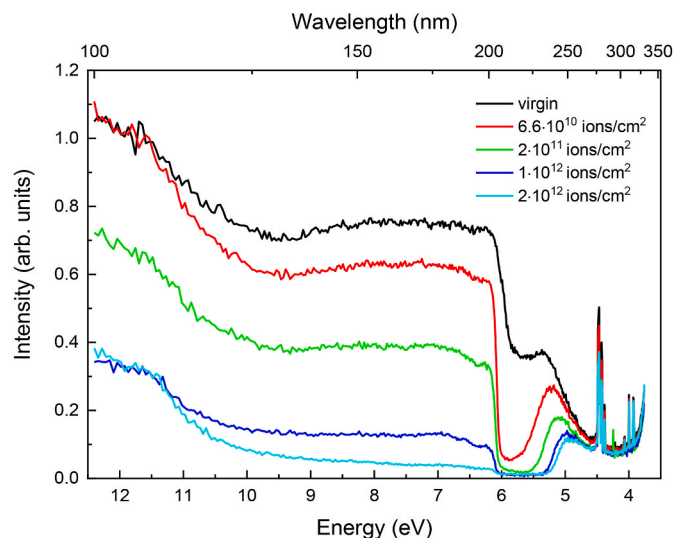


Fig. 4. The excitation spectra of Ce<sup>3+</sup> emission monitored at 550 nm and 12 K in virgin and irradiated GGAG:Ce,Mg single crystals under different fluences (the fluences in ions/cm<sup>2</sup> are shown in legend).

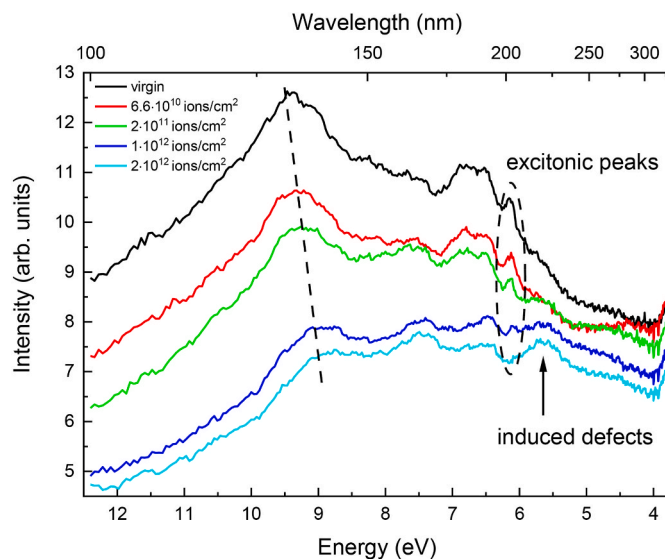


Fig. 5. The reflection spectra at 12 K in virgin and irradiated GGAG:Ce,Mg single crystals under different fluences (the fluences in ions/cm<sup>2</sup> are shown in legend). The dashed area indicates the excitonic peaks.

spectra is smoothed and the reflection peaks are not well pronounced. Nevertheless, a reflection of the excitonic peak is detected (Fig. 5 dashed area). This excitonic peak is diminishing in the highly irradiated crystals and completely absent in the most irradiated sample. The reflection peak at 220 nm (5.6 eV) is better pronounced in the reflection spectra for highly irradiated samples. This reflection peak correlates with the induced absorption peak considered in Fig. 2. All other reflection peaks degrade with the rise of the irradiation fluence. In particular, the relative degradation of the most intensive reflection peak at 9.0–9.5 eV in respect of others is obvious. In addition, there is a clear red shift of this peak at higher irradiation fluences. Considering that this reflection peak is located in VUV spectral range, high-energy excitations that exceed the energy of band-to-band transitions are involved. Therefore, we suggest that swift heavy ion irradiation modifies the electronic structure in addition to the creation of point defects in GGAG single crystals.

#### 4. Conclusions

The pioneering results on radiation damage in GGAG:Ce,Mg single crystals induced by swift heavy ions have been obtained. Optical and luminescence properties of the irradiated crystals have been studied. The main results can be summarized as follows:

1. Induced optical absorption have been observed in the irradiated GGAG:Ce,Mg single crystal in 200–350 nm spectral range. It is suggested that the F-type centers (F and F<sup>+</sup>) are responsible for the intensive induced absorption at 210–250 nm, while V-type hole centers cause the induced absorption band in 250–340 nm diapason.
2. The luminescence excitation spectra clearly demonstrate the increased concentration of Ce<sup>3+</sup> centers in the irradiated GGAG:Ce, Mg single crystals. Two possible models explaining the transformation of Ce<sup>4+</sup> dominating in the virgin sample into Ce<sup>3+</sup> centers in the irradiated crystals have been proposed.
3. The radiation damage induced by swift heavy ion irradiation of the GGAG:Ce,Mg single crystals results in the significant damage of the crystal surface and, as a consequence, causes the strong suppression of Ce<sup>3+</sup> luminescence excitation in the VUV spectral range.

#### Author agreement statement

We the undersigned declare that this manuscript is original, has not



been published before and is not currently being considered for publication elsewhere.

We confirm that the manuscript has been read and approved by all named authors and that there are no other persons who satisfied the criteria for authorship but are not listed. We further confirm that the order of authors listed in the manuscript has been approved by all of us.

We understand that the Corresponding Author is the sole contact for the Editorial process.

He/she is responsible for communicating with the other authors about progress, submissions of revisions and final approval of proofs.

#### CRediT authorship contribution statement

**V. Pankratova:** Formal analysis, Investigation, Writing – original draft, Writing – review & editing. **V.A. Skuratov:** Conceptualization, Methodology, Investigation. **O.A. Buzanov:** Investigation, Resources. **A.A. Mololkin:** Investigation, Resources. **A.P. Kozlova:** Formal analysis, Investigation. **A. Kotlov:** Investigation, Methodology. **A.I. Popov:** Conceptualization, Funding acquisition, Writing – review & editing. **V. Pankratov:** Conceptualization, Methodology, Investigation, Project administration, Funding acquisition, Writing – review & editing, Funding acquisition.

#### 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.

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