On low-temperature luminescence quenching in $Gd_3(Ga,Al)_5O_{12}$:Ce crystals

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ABSTRACT

Temperature dependences of the photoluminescence and X-ray excited luminescence intensity and thermally stimulated luminescence glow curves are measured in the 4.2–300 K temperature range for the undoped and Ce^{3+} - doped $Gd_3(Ga,Al)_5O_{12}$ crystals. The conclusion is made that no low-temperature quenching of the Ce^{3+} - related photoluminescence takes place. In both the undoped and the Ce^{3+} - doped crystals, temperature de-pendences of the X-ray excited recombination luminescence intensity correlate with the position and shape of thermally stimulated luminescence glow curve peaks of the hole origin. Low-temperature quenching of the X-ray excited luminescence in these crystals is explained by the fact that at low temperatures, free holes are trapped at oxygen ions while electrons are trapped at various intrinsic defects. In Ce^{3+} - doped $Gd_3(Ga,Al)_5O_{12}$ crystals, thermally stimulated release of the trapped holes and electrons and their subsequent recombination at Ce^{3+} ions result in the enhancement of the Ce^{3+} - related electron recombination luminescence with the increasing temperature in the 10–180 K range.

1. Introduction

Single crystals of $Gd_3(Ga,Al)_5O_{12}$:Ce are intensively studied as promising scintillator materials for medical imaging because of their extremely high light yield (58000 ph/MeV), excellent energy resolution (4.2%@662 keV), relatively high density (6.63 g/cm^3), fast scintillation response, high radiation stability, and high radiation hardness [1,2]). Recently, extremely high light yield of 74000 ± 7400 ph/MeV and energy resolution of 7.8% have been reported for $Gd_3Ga_3Al_2O_{12}$:Ce single crystals under ^{137}Cs (γ-ray, 662 keV) excitation [3]. In the recent years, $Gd_3(Ga,Al)_5O_{12}$:Ce garnets have been intensively investigated by many groups (see, e.g. Refs. [1–15], and references therein). However, physical processes taking place in scintillation mechanism of these materials still need more detailed study.

In Gd - rich Ce^{3+} - doped materials, the gadolinium sublattice is known to play an important role in the energy transfer processes from the host crystal lattice to Ce^{3+} ions owing to an effective migration of the excitation energy along Gd^{3+} ions terminated by the $Gd^{3+} \rightarrow Ce^{3+}$ energy transfer. In Ref. [8], the energy migration along the Gd sub-lattice and the $Gd^{3+} \rightarrow Ce^{3+}$ energy transfer processes were studied for $Gd_3(Ga,Al)_5O_{12}$:Ce crystals. The investigation of the spectra and decay kinetics of the Gd^{3+} - and Ce^{3+} - related photoluminescence under excitation in the $^8S_{7/2} \rightarrow {}^6I_J$ and $^8S_{7/2} \rightarrow {}^6P_J$ absorption bands of Gd^{3+} allowed the authors to make the conclusion on the presence of the nonradiative (resonance) energy transfer between the host lattice Gd^{3+} ions and impurity Ce^{3+} ions. The reduction of the $^6P_{7/2} \rightarrow {}^8S_{7/2}$ emis-sion of Gd^{3+} with the increasing temperature accompanied with the enhancement of the $5d_1 \rightarrow 4f$ emission of Ce^{3+} was explained in Ref. [8] by the $Gd^{3+} \rightarrow Ce^{3+}$ energy transfer.

In Ref. [12], the Ce^{3+} - related radioluminescence and photo-luminescence were studied at 80–570 K for many Ce^{3+} - doped multi-component garnets of different composition. The decrease of the lu-minescence intensity with the decreasing temperature (the so called low-temperature luminescence quenching) was concluded to correlate well with the position, width, and intensity of the thermally stimulated luminescence (TSL) glow curve peak. Therefore, the luminescence quenching upon cooling was explained in Ref. [12] by the localization of charge carriers at traps at low temperatures. For $Gd_3Ga_3Al_2O_{12}$:Ce single crystals, this effect was earlier observed in Ref. [5], where the correlation between the temperature dependence of the radi-oluminescence yield in the 10–150 K temperature range and the posi-tion of the dominating TSL glow curve peak around 75 K is clearly evident. In Ref. [12], no luminescence reduction was observed in Gd - free multicomponent garnets despite the presence of intense low-tem-perature TSL peaks also in these materials. This fact could point to the connection of the observed feature with the $Gd^{3+} \rightarrow Ce^{3+}$ energy

transfer. However, earlier (see, e.g. Refs. [16,17], and references therein), the low-temperature quenching of the luminescence under gamma-, cathode-, and X-ray excitation was reported for $Y_3Al_5O_{12}$:Ce. In Ref. [16], this effect was explained by the presence in the crystal lattice of many shallow traps which compete with the emitting Ce^{3+} centers in free charge carriers capture.

The aim of this work was to investigate in more detail the processes, responsible for the temperature dependence of the Ce^{3+} - related photoluminescence and X-ray excited luminescence intensity in the 4.2–300 K temperature range, and to obtain an additional information on the mechanism of the low-temperature quenching of the Ce^{3+} - related electron recombination luminescence in Ce^{3+} - doped multi-component garnets based on $Gd_3(Ga,Al)_5O_{12}$ host.

Therefore, we have studied:

(1) the correlation between the temperature dependence of the photo-luminescence intensity - I(T) in the $Gd_3(Ga,Al)_5O_{12}$:Ce crystal and the intensity of the TSL peak optically created under selective photoexcitation in the Gd^{3+} - and Ce^{3+} - related absorption bands. For that, the temperature dependences were measured at different excitation/irradiation conditions, where strongly different con-centrations of free traps or optically created electron and hole centers were achieved in the course or before the I(T) measure-ments.

(2) the correlation between the temperature dependence of the X-ray excited luminescence intensity in the undoped and Ce^{3+} - doped $Gd_3(Ga,Al)_5O_{12}$ crystals and thermal stability of intrinsic electron and hole centers. For that, the TSL glow curves were measured for the X-ray irradiated $Gd_3(Ga,Al)_5O_{12}$:Ce crystal (where the TSL peaks of the electron origin should dominate) and undoped $Gd_3(Ga,Al)_5O_{12}$ crystal (where, according to Ref. [18], the TSL peaks arising from the recombination of thermally released holes with electron centers should dominate over the TSL peaks of an electron origin).

2. Experimental details

In this work, the $Gd_3Ga_{2.83}Al_{2.17}O_{12}$:Ce (GAGG:Ce) and $Gd_3Ga_3Al_2O_{12}$ (GAGG) single crystals grown in Ref. [13] using the Czochralski technique, were investigated. The oxides Gd_2O_3, Ga_2O_3 and Al_2O_3 were of the purity 5 N (99.999%), and the CeO_2 dopant was of the purity 99.95%. The composition of the crystal was determined with the use of an electron probe microanalysis. The Ce concentration inside GAGG:Ce (0.035 at.%) was defined as [the number of Ce atoms per total number of atoms in a GAGG molecule (equal to 20)] x 100%.

Photoluminescence spectra, temperature dependences of the pho-toluminescence intensity – I(T), and TSL glow curves of the UV - irra-diated crystals were measured in the 4.2–300 K temperature range with the use of the computer-controlled setup, consisting of the LOT-ORIEL xenon lamp (150 W), two monochromators (MDR-3 and ORIEL CS130 1/8 m), Hamamatsu H8259 photon counting system, and an immersion helium cryostat. Besides the TSL intensity measurement as a function of crystal temperature, the TSL glow curves were also obtained from the temperature dependences of the photoluminescence intensity as a dif-ference between the emission intensities measured at each temperature with the switched-on and switched-off excitation. In the latter case, this allows us to compare quantitatively the intensities of the photo-luminescence and the thermoluminescence in each experiment. The crystal heating rate was 0.2 K/sec.

The X-ray excited luminescence spectra were measured under irra-diation of the crystal with the X-ray tube (40 kV, 15 mA) for 10 min to achieve the intensity saturation. The crystal was located in the close-cycle refrigerator. The luminescence was detected using Andor Shamrock B-303i spectrograph coupled with Andor DU-401A-BV CCD camera. In addition, Hamamatsu H2859 photomultiplier tube was used to gain extra sensitivity. Temperature dependences of the X-ray excited

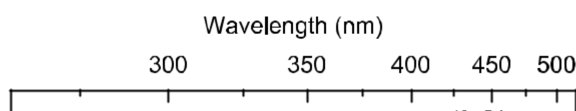

Fig. 1. Absorption spectrum of the GAGG:Ce crystal measured at 295 K.

luminescence intensity and the TSL glow curves of the preliminarily X-ray irradiated samples were measured in the 9–300 K temperature range with the heating rate of 0.2 K/s.

3. Experimental results and discussion

3.1. Temperature dependences of the photoluminescence intensity

In the X-ray irradiated at 8 K GAGG:Ce crystals, the most intense complex TSL glow curve peak is located around 75 K [5,6,9,11]. Similar peak has been observed in Ref. [19] after UV irradiation of GAGG:Ce at 4.2 K in the Gd^{3+} - related $^8S_{7/2} \rightarrow {}^6I_J$ and $^8S_{7/2} \rightarrow {}^6P_J$ or Ce^{3+} - related 4f → $5d_2$ absorption bands shown in Fig. 1. This TSL peak (presented in Fig. 2) appears due to the recombination of the electrons, thermally released from optically created intrinsic electron centers, with the hole Ce^{4+} centers. To clarify the influence of the optically created electron centers responsible for this TSL glow curve peak on the temperature dependence of the Ce^{3+} - related photoluminescence intensity, the I(T) dependences are measured under different excitation conditions:

(i) under excitation in the Gd^{3+} - related $^8S_{7/2} \rightarrow {}^6I_J$ absorption band (4.55 eV), where the $Gd^{3+} \rightarrow Ce^{3+}$ energy transfer takes place

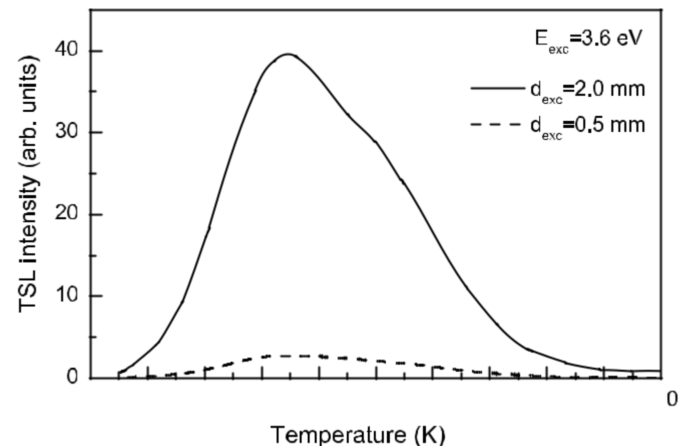

Fig. 2. TSL glow curves measured after irradiation of the GAGG:Ce crystal at 4.2 K in the 4f → $5d_2$ absorption band of Ce^{3+} centers (3.6 eV) with d_{exc} = 2 mm (solid line) and d_{exc} = 0.5 mm (dashed line).

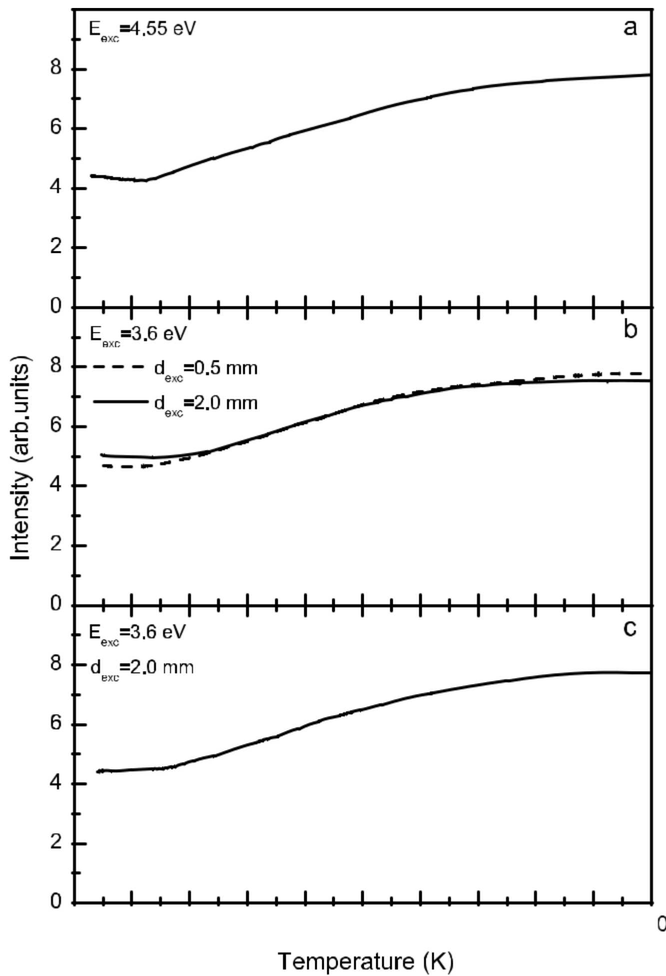

Fig. 3. Temperature dependences of the Ce^{3+} - related photoluminescence in-tensity measured under excitation: (a) in the Gd^{3+} - related $^8S_{7/2} \rightarrow {}^6I_J$ (4.55 eV) absorption band, (b) in the Ce^{3+} - related 4f → $5d_2$ (3.6 eV) ab-sorption band with d_{exc} = 2 mm (solid line) and d_{exc} = 0.5 mm (dashed line),
(c) in the Ce^{3+} - related 4f → $5d_2$ (3.6 eV) absorption band after preliminary irradiation of the sample with E_{irr} = 3.6 eV at 4.2 K for 1 h with d_{exc} = 2 mm.

from the excited ^6P_J state of Gd^{3+} into the $5d_2$ excited level of the neighboring Ce^{3+} ion located inside the conduction band (for more details, see Ref. [19]) (Fig. 3a);

(ii) under direct excitation in the 4f → $5d_2$ (3.6 eV) absorption band of Ce^{3+}, where no $Gd^{3+} \rightarrow Ce^{3+}$ energy transfer can take place (Fig. 3b);

(iii) under excitation in the 4f → $5d_2$ band of Ce^{3+}, but after an intense and prolongated (for 1 h) preliminary irradiation of the crystal in the 4f – $5d_2$ band at 4.2 K. At these conditions, all the traps re-sponsible for the TSL peak become filled up to the saturation be-fore the I(T) measurement (Fig. 3c).

The measurements in Fig. 3b are made under the excitation condi-tions (with the monochromator slit d_{exc} = 2 mm), where the strong TSL peak appears in the course of the I(T) measurement (Fig. 2, solid line), and under the conditions (d_{exc} = 0.5 mm), where the TSL intensity is smaller by about an order of magnitude (Fig. 2, dashed line). Despite the large difference in the TSL intensity, both temperature dependences of the Ce^{3+} - related luminescence intensity presented in Fig. 3b are close. They are also close to the I(T) dependence presented in Fig. 3c and measured under conditions where the concentration of empty electron traps has been minimized by the preliminary strong irradiation of the sample. The TSL intensity in this case is by an order of magnitude larger as compared with Fig. 2, solid line.

The comparison of the data presented in Fig. 3 indicates, that the intensity of the TSL peak (optically created in the course of the I(T) measurement – Fig. 3b or before this measurement – Fig. 3c) does not noticeably influence the temperature dependence of the Ce^{3+} photo-luminescence intensity. A gradual increase of the photoluminescence intensity with the increasing temperature is caused by the well-known phenomenon: the increasing probability of the 4f → $5d_2$ absorption transitions due to the thermal population of the higher-energy levels of the 4f ground state (for more details, see, e.g., Refs. [20,21]). It should be emphasized that this effect does not contribute into the I(T) de-pendence presented in Fig. 3a.

These data allow the conclusion that the enhancement of the Ce^{3+} - related photoluminescence, excited in the Gd^{3+} - related absorption bands, with the increasing temperature in the 30–120 K temperature range shown in Fig. 3a can be explained only by the $Gd^{3+} \rightarrow Ce^{3+}$ energy transfer (as suggested in Ref. [8]). Indeed, both the decrease of the Gd^{3+} emission intensity and increase of the Ce^{3+} emission intensity at T > 30 K, observed under excitation in the Gd^{3+} - related $^8S_{7/2} \rightarrow {}^6I_J$ absorption band (4.55 eV), take place with approximately the same activation energies (about 20–30 meV). This is evident from Fig. 4 where both these dependences are presented in the ln I - 1/T co-ordinates.

Additionally, we have measured the I(T) dependences of the Ce^{3+} emission in GAGG:Ce under excitation in the 4f - $5d_1$ absorption band before and after a strong and long irradiation in the 4f - $5d_2$ absorption band at 80 K, where the electron centers responsible for the TSL peak at ≈120 K are effectively created (see, e.g., Ref. [14]). However, the in-crease of the emission intensity around 120 K, caused by the trapping of thermally released electrons by Ce^{4+} ions, does not exceed 4%. This means that thermally stimulated release of electrons from traps cannot result in the considerable enhancement of the Ce^{3+} photoluminescence with the increasing temperature shown in Fig. 3a.

Thus, the data obtained allow us to conclude that no low-

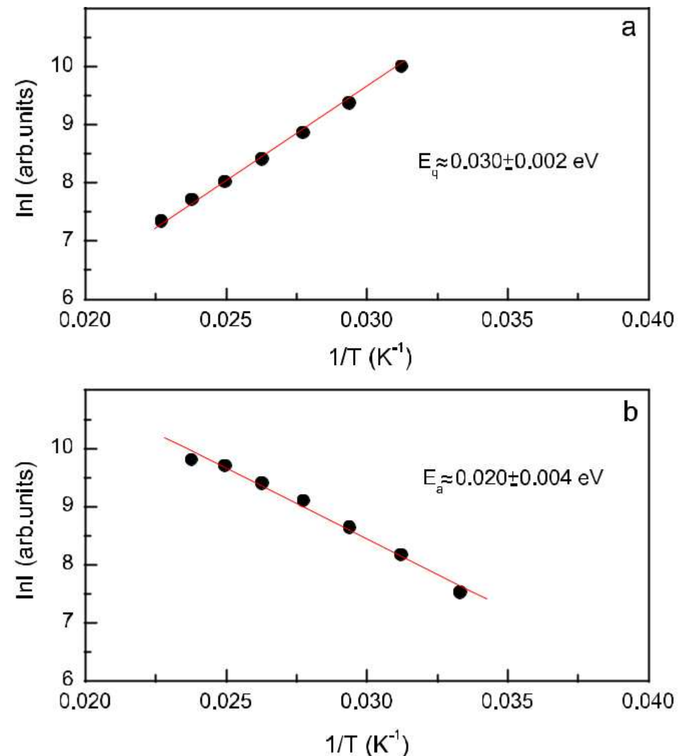

Fig. 4. Temperature dependences of the intensities of (a) the Gd^{3+} - related 3.94 eV emission and (b) the Ce^{3+} - related 2.23 eV emission of the GAGG:Ce crystal, presented in the ln I – 1/T coordinates. E_{irr} = 4.55 eV.

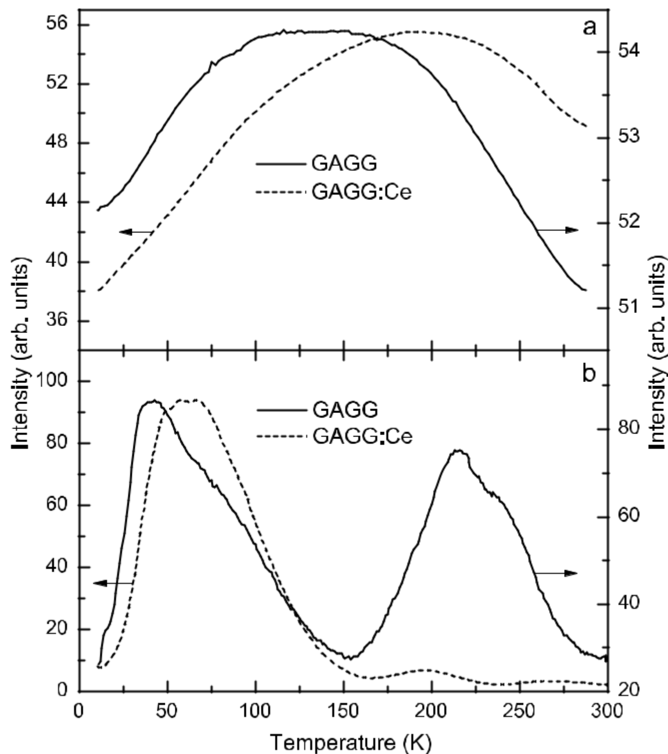

Fig. 5. (a) Normalized temperature dependences of the integrated X-ray excited luminescence intensity measured at the same conditions for the GAGG (solid line) and GAGG:Ce (dashed line) crystals. (b) Normalized TSL glow curves measured at the same conditions after X-ray irradiation at 9 K of the GAGG (solid line) and GAGG:Ce (dashed line) crystals. The heating rate was of 0.2 K/ sec. In GAGG:Ce, the luminescence intensity is by about two orders of magni-tude larger as compared with GAGG.

temperature quenching of the Ce^{3+} - related photoluminescence, which could be caused by the trapping of optically released charge carriers in the host lattice, takes place in GAGG:Ce.

3.2. Temperature dependences of the X-ray excited luminescence intensity

Unlike the photoluminescence, the low-temperature quenching of the radioluminescence, appearing under gamma-, cathode- and X-ray excitation, has been observed in many Ce^{3+} - doped garnets (see, e.g., Refs. [5,12,15–17]). For the undoped GAGG crystal and the GAGG:Ce crystal, temperature dependences of the integrated X-ray excited lu-minescence intensity are presented in Fig. 5a (solid and dashed lines, respectively). For comparison, the TSL glow curves of GAGG and GAGG:Ce, measured with the same heating rate after X-ray irradiation at 9 K, are shown in Fig. 5b (solid and dashed lines, respectively). The luminescence spectra of the same GAGG and GAGG:Ce crystals have been measured in Refs. [9,13].

From the comparison of Fig. 5a and b (dashed lines) it is evident that the reduction of the X-ray excited Ce^{3+} - related luminescence intensity of GAGG:Ce with the decreasing temperature observed at T < 180 K (the low-temperature luminescence quenching) really cor-relates with the position and width of the intense complex TSL glow curve peak located in the same temperature range. Let us consider this effect in more detail.

Under excitation with the ionizing radiation, free electrons and holes are created in the host garnet lattice. At low temperatures, the electrons are mainly trapped at intrinsic crystal lattice defects, like oxygen vacancies, antisite ions, etc. (see e.g., Ref. [22]). According to Ref. [18], the holes in the undoped $Y_3Al_5O_{12}$ and $Lu_3Al_5O_{12}$ garnets are predominantly self-trapped at regular oxygen ions, forming O^- hole centers (self-trapped holes), or trapped at oxygen ions in the vicinity of various defects, like antisite ions, divalent impurity ions, etc., produ-cing more stable O^- - type hole centers. Unfortunately, the EPR in-vestigations, analogous to those carried out in Ref. [18], are not pos-sible in Gd - containing crystals due to a large nuclear spin of Gd. However, it can be suggested that the same hole centers can exist also in the undoped $Gd_3Al_5O_{12}$ crystals. In Ce^{3+} - doped garnets, the free holes are effectively trapped at Ce^{3+} ions producing the hole Ce^{4+} centers.

The recombination of electrons with intrinsic hole centers can be nonradiative or accompanied with the intrinsic luminescence (see, e.g., Refs. [13,17,23]). The Ce^{3+} - related electron recombination lumines-cence appears only as a result of electrons recombination with the hole Ce^{4+} centers. Due to that at low temperatures, the intensity of this luminescence can be noticeably reduced due to the competition in the hole trapping processes between the Ce^{3+} ions and the other, more shallow, intrinsic hole traps.

According to Ref. [18], the self-trapped holes in the undoped $Y_3Al_5O_{12}$ and $Lu_3Al_5O_{12}$ crystals are stable up to about 100 K. Thermal release of the self-trapped holes in these garnets is accompanied with the TSL glow curve peak located at ≈120 K. The corresponding trap depth E_t = 0.25–0.26 eV. In the undoped GAGG crystal, the TSL glow curve peaks around 40 K and 100 K are observed (Fig. 5b, solid line). By analogy with the undoped $Y_3Al_5O_{12}$ and $Lu_3Al_5O_{12}$ garnets studied in Ref. [18], we suggest that both these peaks are of the hole origin and that the 100 K peak appears due to the thermal release of the self-trapped holes in GAGG. The dominating complex low-temperature TSL glow curve peak in the Ce^{3+} - doped GAGG, appearing due to the re-combination of electrons with the hole Ce^{4+} centers, is located in the 10–170 K temperature range (see, e.g. Refs. [5,6,9,11–13,16,19], and Fig. 5b, dashed line). The depths of the corresponding traps E_t vary from 0.02 eV to 0.4 eV [9,13] (from 0.05 eV to 0.24 eV, according to Refs. [5,6]). Thus, the intrinsic electron and hole centers become thermally destroyed in close temperature regions. As the temperature increases, the probability of the electrons and holes trapping in the host crystal lattice decreases while the probability of their recombination at the Ce^{3+} ions increases. This effect results in the increasing intensity of the electron recombination luminescence of Ce^{3+} centers observed in the same temperature range. The intensity decrease at T > 220 K is most probably caused by the thermal quenching of the Ce^{3+} lumines-cence (see, e.g., Refs. [4,13,14]).

It should be noted that the Ce^{3+} - related luminescence intensity presented in Fig. 5a (dashed line) increases with the increasing tem-perature with much smaller activation energies (< 9 meV) as compared with the activation energy of the Gd^{3+} luminescence thermal quenching (≈30 meV, see Fig. 4a). This means that the influence of the $Gd^{3+} \rightarrow Ce^{3+}$ energy transfer on the temperature dependence of the X-ray excited luminescence intensity is negligible.

In case of the undoped GAGG crystal, the increase of the X-ray ex-cited intrinsic luminescence intensity with the increasing temperature up to ≈100 K (Fig. 5a, solid line) can be also caused by the decreasing probability of the free hole self-trapping or trapping at shallow traps. Due to that, the efficiency of the recombination of the free holes with electrons increases. The intensity reduction at higher temperatures (T > 160 K) is not caused by the luminescence thermal quenching as the intrinsic photoluminescence is not quenched up to 500 K [13]. Most probably, the reduction of the intrinsic X-ray excited luminescence around 220 K is caused by an effective trapping of free holes at deeper hole traps responsible for the intense TSL peak located around 220 K (see Fig. 5b, solid line). These data allow us to assume that the radi-oluminescence yield drop at 180–185 K, observed for GAGG:Ce crystals in Ref. [5], is probably caused by the presence of intrinsic hole traps in the investigated crystals which compete with the Ce^{3+} ions in free hole trapping processes.

Thus, in both the undoped and the Ce^{3+} - doped GAGG crystals, the correlation between the temperature dependences of the X-ray excited luminescence intensity and the location of the TSL glow curve peaks of

the hole origin is clearly evident from the comparison of Fig. 5a and b.

4. Conclusions

The investigation of the temperature dependences of the Ce^{3+} - related photoluminescence intensity in the GAGG:Ce crystal measured under different excitation conditions allows the conclusion that no correlation exists between the I(T) dependences and the intensity of the dominating TSL peak located around 70–80 K and optically created before or in the process of the measurements. This result allows us to conclude that no low-temperature quenching of the Ce^{3+} - related photoluminescence, which could be caused by the trapping of the op-tically released charge carriers in the host crystal lattice, takes place in the Ce^{3+} - doped Gd-rich multicomponent garnets. The enhancement of the Ce^{3+} - related photoluminescence with the increasing temperature with the activation energy of about 20 meV is due to the excitation energy migration along the Gd sublattice terminated by the $Gd^{3+} \rightarrow Ce^{3+}$ energy transfer.

Unlike the photoluminescence, the low-temperature radi-oluminescence quenching takes really place. In GAGG:Ce, this effect can be explained by the competition between the processes of the radiative electron-hole recombination at Ce^{3+} ions and the trapping of the free charge carriers, produced by the ionizing radiation, at intrinsic defects, resulting in the creation of the corresponding intrinsic electron and hole centers. Basing on the data [18], we suggest that at low temperatures, the trapping of the free holes at oxygen ions results in the formation of the O^{-} - type hole centers in GAGG:Ce. The free electrons become trapped at different intrinsic defects responsible for the broad compex electron TSL glow curve peak located around 75 K. Thermally stimu-lated release of the self-trapped holes and their effective re-trapping at Ce^{3+} ions results in the formation of the hole Ce^{4+} centers. Thermally stimulated release of the trapped electrons in the same temperature range and their recombination with the hole Ce^{4+} centers explains the enhancement of the Ce^{3+} - related electron recombination lumines-cence with the increasing temperature observed in the 10–180 K range.

We suggest that the temperature dependence of the X-ray excited luminescence intensity in an undoped crystal, which correlates with the position and shape of the TSL glow curve peaks of the hole origin, can be used to estimate the presence and thermal stability of intrinsic hole centers in the corresponding Ce^{3+} - doped crystal.

An effective trapping of electrons and holes at various intrinsic defects, resulting in the suppression of the Ce^{3+} luminescence, reduces the scintillation efficiency. Besides, the tunneling recombination of the holes trapped at oxygen ions and the electrons trapped at intrinsic crystal lattice defects (oxygen vacancies and antisites) is accompanied with the intrinsic emission. In GAGG:Ce, a strong overlap of the broad intrinsic emission band peaking at about 2.4 eV and the Ce^{3+} emission band centered at 2.25 eV results in the appearance of undesirable slow components in the scintillations decay.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influ-ence the work reported in this paper.

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