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Luminescence properties of $\text{Y}_3\text{Al}_5\text{O}_{12}:\text{Ce}$ nanoceramics

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ABSTRACT

Comparative analysis of the luminescent properties of $\text{Y}_3\text{Al}_5\text{O}_{12}:\text{Ce}$ (YAG:Ce) transparent optical ceramics (OC) with those of single crystal (SC) and single crystalline film (SCF) analogues has been performed under excitation by pulsed synchrotron radiation in the fundamental absorption range of YAG host. It has been shown that the properties of YAG:Ce OC are closer to the properties of the SCF counterpart, where Y_{Al} antisite defects are completely absent, rather than to the properties of SC of this garnet with large concentration of Y_{Al} antisite defects. At the same time, the luminescence spectra of YAG:Ce OC show weak emission bands in the 200–470 nm range related to Y_{Al} antisite defects and charged oxygen vacancies (F^+ and F centers). YAG:Ce OC also possesses significantly larger contribution of slow components in the Ce^{3+} luminescence decay under high-energy excitation in comparison with SC and SCF of this garnet due to the involvement of antisite defects, charged oxygen vacancies as well as boundaries of grains in the energy transfer processes from the host to the Ce^{3+} ions.

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1. Introduction

Transparent optical ceramics (OC) of $\text{Y}_3\text{Al}_5\text{O}_{12}$ (YAG) garnet doped with rare-earth ions is now being considered as an alternative to bulk single crystals (SC) of these materials for laser media applications and scintillators [1–5]. Usually OC are obtained from initial row powders with small grain size [1,2] or from sintered YAG-based nanopowders [3,4] previously prepared by sol–gel methods [4–6]. The main differences of the properties of YAG-based OC in comparison with SC can be the relatively lower temperature of preparation of OC (about 1700–1750 °C) [1–6] than that for YAG:Ce SC obtained by melting at high (1970 °C) temperature by the Czochralski or modified Bridgman methods [7]. Therefore, OC can possess, in principle, lower concentration of Y_{Al} antisite defects (AD), which are typical for melt-grown YAG-based SC [7–11]. The mentioned type of defects creates emission centers, which significantly contribute to the intrinsic UV luminescence of YAG SC [12] and is strongly involved in the processes of high-energy excitation of Ce^{3+} luminescence in YAG:Ce SC [7,13–15]. In this respect the behavior of Ce-doped YAG OC can be closer to the properties of YAG:Ce single crystalline films (SCF) obtained by liquid phase epitaxy from melt-solutions at low (900–105 °C) temperatures rather than to the properties of YAG:Ce SC [7,13–15]. However, the typical

high-temperature sintering of OC in the reduction atmosphere (vacuum or Ar) at temperatures below 200–250 °C than that for SC crystallization can also introduce both AD and vacancy-type defects; first of all, oxygen vacancies, which degrade the luminescent and scintillation properties of YAG:Ce OC.

The luminescent properties of YAG:Ce OC in comparison with SC and nanopowder analogues have been recently studied in several works [16–19]. It has been shown that YAG:Ce OC really possesses a low concentration of Y_{Al} antisite defects as emission centers in the UV range [16–18] and trapping centers as well [16]. At the same time, OC shows some disadvantages related to lower light yield (LY) and large amount of slow component in Ce^{3+} emission decay compared to SC [16], and the reasons for such properties of OC are not clear hitherto. Therefore, in this work, we continue detailing the comparative analysis of the intrinsic and the Ce^{3+} -related luminescence of YAG:Ce OC, SC and SCF under excitation by pulsed synchrotron radiation (SR) in the exciton range and the range of interband transitions of the YAG host.

2. Samples' preparation and experimental technique

Highly pure YAG:Ce nanopowder with average grain size of about 30 nm with Ce concentration of 0.5 molar% with respect to yttrium was prepared by a co-precipitation method (see Refs. [5,20] for details) and was used as initial charge for the preparation

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of transparent YAG:Ce OC. The nanopowder was pressed into yellow ceramic tablets. The tablets were hot-press sintered in vacuum $\sim 10^{-3}$ Pa at 1700 °C under pressure of about 300 atm for 8 h. Then they were annealed in air at 1350 °C for 24 h to decrease the content of oxygen vacancies possibly introduced during hot-sintering in a strongly reducing atmosphere. Then the ceramics was polished to a high optical grade. The average grain size in OC was 15–20 μm . The details of OC preparation are described in Ref. [6].

The SCF with a thickness of 55 μm was crystallized from the melt-solution based on $\text{PbO-B}_2\text{O}_3$ fluxes by liquid phase epitaxy methods onto both sides of the YAG substrates (see Ref. [15] for details). The YAG:Ce SC were grown from the melt by the modified Bridgman methods in Ar atmosphere (see Ref. [7] for details). For all the technological experiments, Y_2O_3 oxide of 5 N purity and Al_2O_3 oxide of 4 N purity were used.

The absorption of YAG:Ce OC, SC and SCF was measured using a Perkin–Elmer spectrophotometer at 300 K (RT). The comparative light yield measurements of YAG:Ce OC, SC and SCF were performed using a detector based on FEU-110 PMTs with the maximum sensitivity at 450 nm and a multi-channel single-photon counting system working in a time interval of 0.5 μs under excitation by α -particles of Pu^{239} sources (5.15 MeV) in comparison with the standard YAG:Ce SCF samples with an LY of 16 100 Ph/MeV.

Comparative analysis of the time-resolved intrinsic luminescence of YAG and YAG:Ce OC, SC and SCF has been performed at 10 K under excitation by pulsed (0.127 ns) SR at the Superlumi experimental station (HASYLAB at DESY) with an energy of 3.7–25 eV. The emission and excitation spectra were measured with monochromators ARC and PMT Hamamatsu R6358P, both in the integral regime and in the 1.2–12 and 150–200 ns time intervals (fast and slow components, respectively) in the limits of SR pulse with a repetition time of 200 ns. The decay kinetics of luminescence was measured in the 0–200 ns time range at 10 K. The excitation spectra were corrected for the spectral dependence and intensity of the excitation energy; the emission spectra were not corrected.

3. Experimental results and discussion

The optical quality of samples under study was analyzed by the absorption spectra (Fig. 1). As seen in Fig. 1, the OC has good

transmittance even in the UV range up to 250 nm. The shift of the absorption edge below 250 nm and the worse transmittance of the YAG:Ce OC samples with respect to the SC counterpart is due to some scattering of light at the boundaries of grains. The large absorption in YAG:Ce SCF below 300 nm is caused by Pb^{2+} flux impurity, coming into the SCF from the PbO -based flux at concentrations below 30 ppm [15]. Specifically, the absorption bands peaked at 267 and below 220 nm, related to the $^1\text{S}_0 \rightarrow ^3\text{P}_1$ and $^1\text{P}_1$ transition of Pb^{2+} ions, respectively [20] (not shown in Fig. 1).

The quality of OC, SC and SCF is also characterized by the relative light yield measurements under excitation by α -particles of Pu^{239} sources. The LY of YAG:Ce SCF standard sample (16 100 Ph/MeV), YAG:Ce OC and SC was equal to 100%, 132% and 166%, respectively. Therefore, the YAG:Ce OC sample shows good optical and scintillation performance, compared with the properties of YAG:Ce SC and SCF samples.

The luminescence spectra of YAG:Ce OC, SC and SCF at 8 and 300 K under excitation by SR in the exciton range at 7.28 eV are shown in Fig. 2. Apart from the intensive luminescence in the visible range in the doublet band peaking at 536 and 567 nm at 300 K (523 and 567 nm at 8 K, Fig. 2, curves 1 and 2, respectively), caused by the $5d^1 \rightarrow 4f^2(^2\text{F}_{5/2,7/2})$ transitions of Ce^{3+} ions, the emission spectra of YAG:Ce OC at 300 K consist also of low-intensity intrinsic emission bands in the UV range peaking at 301, 372 and 394 nm. By the analogy with the structure of the intrinsic luminescence of undoped YAG [12] and YAG:Ce SC [7,13–15], the nature of the mentioned UV bands in YAG:Ce OC can be caused by radiation decay of an exciton localized around Y_{Al} AD (LE (AD) centers) [12,21], by recombination luminescence of Y_{Al} AD [12,14] and by the luminescence of F^+ centers localized around ADs [7,22,23], respectively. As can be seen from Fig. 2, the intensity of the intrinsic emission related to Y_{Al} AD centers is at least three times lower in YAG:Ce OC (curve 2) than that in YAG:Ce SC (curve 3). At 8 K, the luminescence of self-trapped excitons (STE) in the band peaking in the vicinity of 260 nm dominates in the spectra of YAG:Ce OC and significantly overlaps with the LE (AD) emission band (Fig. 2, curve 1).

The emission bands formed by Y_{Al} AD and F^+ centers strongly overlap with the Ce^{3+} absorption bands that peaked at 340 and 460 nm (indicated by arrows in Fig. 2). This results in the excitation of Ce^{3+} luminescence in YAG:Ce OC and SC by the slow emission of AD-related centers. Contrary to OC and SC, only Ce^{3+} ions emit in YAG:Ce SCF in the RT range under high-energy

Fig. 1. Absorption spectra of YAG:Ce OC (1), SC (2) and SCF (3) at RT.

Fig. 2. Emission spectra of YAG:Ce OC (1, 2), SC (3) and SCF (4) at 8 K (1) and 300 K (2) under excitation in the range of exciton absorption at 7.28 eV. The part of the emission spectra in the 200–460 nm range is multiplied by a factor of 100.

excitation due to the complete absence of AD in them (Fig. 2, curve 4).

The excitation spectra of the STE and LE (AD) luminescence in YAG:Ce OC are shown in Fig. 3. The STE luminescence in YAG:Ce SC at 8 K is excited in the complex bands located in the exciton range with main maxima at 7.04 and 7.75 eV, respectively (Fig. 3b, curve 1). The low-energy peak at 7.0 eV of STE emission in YAG:Ce SC is noticeably shifted with respect to the excitation maxima at 7.18 eV of the STE emission in YAG SCF [12] as a result of the presence of the excitation bands of LE (AD) luminescence (Fig. 3, curves 2). The fine structure of the excitation band of STE emission (two peaks at 7.04 and 7.75 eV) presumes the existence of two excited levels of STE in the YAG host [12]. Similar doublet structure of the excitation bands is observed in the LE (AD) luminescence in YAG:Ce OC at 300 K (Fig. 2, curves 2). The main excitation maxima of the LE (AD) emission are located at 6.93 and 7.45 eV, when the peak at 7.75 eV is most probably already related to the onset of the interband transitions in the YAG host [12].

The conclusion about the existence of two excited states of excitons in the YAG host is supported by the shape of decay kinetics of STE, LE (AD) and Y_{Al} AD luminescence in YAG:Ce OC (Fig. 4) under excitation by SR in the characteristic points of excitation spectra of the corresponding centers in the exciton range (Fig. 3). The decay kinetics of the luminescence of all the mentioned centers under registration of the emission in the narrow (0–200 ns) time range can be presented as a superposition of two components with significantly different probabilities of the radiative transitions. Specifically, the existence of the fast (in ns range) and main slow (in on range of hundred ns) decay components of STE, LE (AD) and Y_{Al} AD luminescence supports the suggestion [12] about the formation of two excited states of excitons (most probably singlet and triplet states) related to the corresponding centers. The parameters of the three-component fitting of the corresponding decay curves of the STE, LE (AD) and Y_{Al} AD luminescence in YAG:Ce OC are summarized in Table 1.

The excitation spectra of the fast component of Ce^{3+} emission in YAG:Ce OC, SC and SCF (Fig. 5) consist of the band peaking at 7.98 eV related to the onset of the interband transitions in YAG; the dominant bands peaked in the 6.68–6.885 eV range, corresponding to the formation of an exciton bound with the Ce^{3+} ions, and the band peaked at 5.56 and < 3.6 eV, related to the 4f–5d

Fig. 3. Excitation spectra of STE (1) and LE (AD) (2) luminescence in YAG:Ce OC, measured at 260 nm at 8 K (1) and 305 nm at 300 K (2), respectively.

Fig. 4. Decay kinetics of STE (1), LE (AD) (2) and Y_{Al} AD (3) luminescence at 8 K (1) and 300 K (2, 3) in YAG:Ce OC under excitation by SR with an energy of 7.075 eV (1, 2) and 7.28 eV (3), and registration of emission at a wavelength of 260 nm (1), 300 nm (2) and 380 nm (3).

Table 1
Parameters of approximation of the decay curves of STE, LE (AD) and Y_{Al} AD luminescence presented in Fig. 2.

Centers	A_1	τ_1 (ns)	A_2	τ_2 (ns)	A_3	τ_3 (ns)
STE	3083	1.65	133.4	21.5	409	^a
LE (AD)	728	1.4	160.7	22.0	133.4	121
Y_{Al} AD	93	1.6	35	24.9	63.3	140

^a The value τ_3 cannot be correctly evaluated due to narrow (200 ns) time interval for decay kinetic measurements.

Fig. 5. Excitation spectra of fast components of Ce^{3+} luminescence in YAG:Ce OC (1), SC (2) and SCF (3) at 8 K, normalized to the main peak in exciton range. Position of excitation band of Y_{Al} AD in YAG SC is indicated by an arrow.

($^3T_{2g}$ and 2E) transitions of Ce^{3+} ions, respectively [7,13,14]. Two other excitation bands peaking at 5.12 and 4.8 eV are observed in the low-energy side of the excitation spectra of Ce^{3+} emission in YAG:Ce OC. The last bands are also noticeable in the excitation spectra of YAG:Ce SC. The band peaking at 4.74 eV, observed only in the excitation spectra of YAG:Ce SCF, is related to the $^1S_0 \rightarrow ^3P_1$ transitions of the Pb^{2+} flux dopant in the SCF [20].

Due to the presence of the Y_{Al} AD in different concentrations both in YAG:Ce OC and in SC, the excitation spectra of the Ce^{3+} luminescence in them (Fig. 5, curves 1 and 2, respectively) show a noticeable difference in shape in the exciton range in comparison with the ones for SCF (curves 3).

Furthermore, the form of the excitation spectra of YAG:Ce OC looks like the transition form between YAG:Ce SC and SCF. We also note the different positions of the main excitation maxima of Ce^{3+} luminescence in the exciton range corresponding to the energy of formation of excitons bound with Ce^{3+} ions in YAG:Ce SC (6.68 eV), OC (6.78 eV) and SCF (6.885 eV) at 8 K (Fig. 2, curves 1–3, respectively). It is important to note that the position of the excitation bands of Ce^{3+} luminescence in YAG:Ce OC and especially SC (Fig. 5, curves 1 and 2, respectively) is shifted to the position of the excitation bands of Y_{Al} AD luminescence (shown in Fig. 5 by an arrow); i.e. the Y_{Al} AD related centers are involved in the excitation of Ce^{3+} emission in YAG:Ce SC and OC.

Another characteristic feature of the excitation spectra of Ce^{3+} luminescence in YAG:Ce OC and SC in comparison with the ones for SCF is the existence of the excitation bands peaking at 4.8 and 5.12 eV. We presupposed that these bands can correspond to excitation of the emission of F and F^+ centers because the excitation bands of the luminescence of these centers in YAG crystals are located in the same range [23,24]. Thus, the F and F^+ centers can also be notably involved in the excitation of the Ce^{3+} luminescence of both YAG:Ce OC and SC. Namely, the main maxima of the F center luminescence in the YAG host (460 nm) strongly overlap with the Ce^{3+} 4f–5d₁ absorption band that peaked in the same range.

This conclusion is also confirmed by the decay kinetics of Ce^{3+} luminescence under excitation in the interband transition ranges in comparison with the ones for SCF (Fig. 6). Under excitation by SR with an energy of 3.71 eV in the 5d₁ Ce^{3+} absorption bands, the decay times of Ce^{3+} luminescence in YAG:Ce SCF (71 ns), OC (76 ns) and SC (79 ns) are somewhat different (Fig. 5, curves 1–3) mainly due to the difference in the Ce^{3+} content. At the same time, due to the excitation of Ce^{3+} luminescence in YAG:Ce SC by the emission of LE (AD), Y_{Al} , F and F^+ centers [7,13,14] as well as the temporal localization of charge carriers at these centers in the RT range [25], the decay kinetics of the Ce^{3+} luminescence in SC and especially in OC is significantly slower (Fig. 5, curves 2' and 3')

than that in the SCF analogue (Fig. 4, curve 1'). Namely, under high-energy excitation (13.6 eV) the mean decay time of YAG:Ce SCF, SC and OC is equal to 92, 105 and 162 ns, respectively (Fig. 6).

We have also estimated the content of the slow components in the decay of YAG:Ce OC, SC and SCF under high-energy (13.6 eV) excitation, using the ratio between intensity I_s of the decay components, which is slower than the repetition frequency of SR (200 ns), and the decay amplitude I_f of the rising part of the decay curve as $K_s = [I_s/I_f] \cdot 100\%$ (Fig. 6). The content of slow components is significantly larger in YAG:Ce OC (47.6%) than that in SC and SCF (18.8% and 14.6%, respectively) (Fig. 6). The main reason for such a slowing-down of the decay of Ce^{3+} luminescence and an increase of the amount of slow emission components in YAG:Ce OC and SC, sintered/grown at high temperature in reducing atmosphere, is the presence of large content of Y_{Al} AD and oxygen vacancies as emission and trapping centers in comparison with the condition of low-temperature crystallization of SCF from the melt-solution in air. Specifically, due to the more reducing (vacuum) atmosphere for sintering, the content of oxygen vacancies can prevail in YAG:Ce OC, when the concentration of Y_{Al} AD in SC, grown at higher temperature, is significantly larger. Apart from this, the larger content of the slow component in YAG:Ce OC than that in SC can be caused by the presence of pores and boundaries of grains that can play the role of specific “defects” in the energy transfer of charge carriers to Ce^{3+} ions.

4. Conclusion

The comparative analysis of the luminescent properties of YAG:Ce OC in comparison with the SCF and SC analogues allows us to conclude that the behavior of OC is closer to the properties of SCF, where Y_{Al} AD are completely absent [12–15], rather than to the properties of SC of these garnets with large concentration of Y_{Al} AD [7,8]. Specifically, the form of the excitation spectra of the Ce^{3+} emission in the exciton range is similar to that in SCF and the positions of excitation bands related to the creation of the excitons bound with the Ce^{3+} ions in OC and SCF are relatively close in comparison with the case of SC. This reflects the smaller concentration of Y_{Al} AD-related centers in YAG:Ce OC with respect to SC where the role of Y_{Al} AD as emission and trapping centers in the process of excitation of Ce^{3+} luminescence is very significant [13–15].

At the same time, the weak intrinsic UV luminescence of YAG:Ce OC in the 200–450 nm range, as a superposition of the luminescence of excitons, localized around the Y_{Al} AD centers, the recombination luminescence of Y_{Al} AD and the luminescence of F^+ centers, is observed in YAG:Ce OC in the RT range. However, the intensity of emission related to the mentioned centers is lower by a factor of three or greater than that for YAG:Ce SC. YAG:Ce OC possesses also the larger contribution of slow components of the Ce^{3+} luminescence under excitation in the range of interband transitions in comparison with SCF and SC analogues due to the involvement of vacancy-type defects and the boundaries of OC in the delivery of excitation energy from the host to the Ce^{3+} ions.

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Fig. 6. Decay kinetics of Ce^{3+} emission in YAG:Ce SCF (1, 1'), SC (2, 2') and OC (3, 3') at 300 K under excitation by SR with energies of 3.71 eV (1–3) in the Ce^{3+} absorption band and 13.6 eV (1'–3') in the range of interband transitions. Decay time of Ce^{3+} luminescence under 3.71/13.6 eV excitation and content of slow components in Ce^{3+} emission under high-energy excitation (13.6 eV) in YAG:Ce SC and SCF are presented.

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