Low temperature delayed recombination decay in scintillating garnets

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A B S T R A C T

We study the temperature dependence of delayed recombination decay intensity in a variety of scintillating crystals based on garnet hosts in a wide temperature range 8–500 K. Previous work on several scintillating materials based on silicate hosts showed nonzero constant signal from very low temperatures up to about 120 K. This observation supported a previously suggested hypothesis on low temperature losses of fast scintillation light due to quantum tunneling between the activator and nearby traps. In garnets, the subject of the present article, we observe practically constant signal in the entire monitored temperature range. We thus further test and confirm the quantum tunneling hypothesis in a different class of scintillating materials. We also show that there is no thermal ionization of the activator's excited state in all materials studied, well above room temperature.

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1. Introduction

In applications of scintillators where fast response is required any losses of fast scintillation light are highly undesired and need to be prevented. They are usually associated with thermal effects such as classical thermal quenching of the activator's luminescence or thermal ionization of its excited state. Recently, however, using the delayed recombination decay technique, it has been observed that the losses of fast scintillation light can occur at very low temperatures where none of the above thermal effects apply [1].

Delayed recombination measurement has been developed to study thermal ionization of the luminescence center excited state [2] as an alternative to other techniques, such as photoconductivity using blocking electrodes [3], the microwave resonator technique [4] or thermoluminescence after UV excitation [5–7]. It consists in the observation of slow tails in luminescence decay under UV excitation. When the luminescence center is thermally ionized, charge carriers that do not decay promptly can migrate through conduction or valence bands. After being trapped and released they return to the luminescence center and recombine producing delayed (slow) light.

As addressed above, a nonzero delayed recombination signal was also observed at very low temperatures. These low temperature losses of fast scintillation light have been associated with quantum tunneling from the activator to a nearby trap and back,

as in the scheme in Fig. 1. A similar mechanism of excitation energy or electron transfer is in the usual donor–acceptor situation also referred to as Dexter transfer, or transfer due to the exchange interaction depending on the overlap between the wave functions of the activator and the trap.

The hypothesis was supported both experimentally and theoretically. Experimentally, in a variety of Ce and Pr-doped scintillating silicates, constant delayed recombination decay intensity was measured at least up to about 120 K, a result consistent with the temperature independence of quantum tunneling [8]. Theoretical support was first provided by a simple one-dimensional model [9] that attained several orders of magnitude delay of the activator's luminescence decay due to quantum tunneling. Extending the numerical calculation to two dimensions allowed a finer check of the tunneling hypothesis, and indeed the correct orders of magnitude were again obtained. Furthermore, analytic estimates on a relation between a trap's depth and its distance from the activator have also been made [10] and are expected to allow estimates of significant properties of the traps. Unlike many calculations based on tunneling between traps and recombination centers in various materials [11–17] this approach is independent of any assumptions about "attempt frequency".

In this work we study the temperature dependence of delayed recombination decay intensity in a variety of scintillating Ce^{3+} and Pr^{3+}-doped simple garnets. These materials are considered to have significant potential in the family of fast and high light yield oxide scintillators. For this reason, experimental and theoretical aspects have been addressed by many groups in last two decades

[18–27]. The present work is intended to help understand unexplained features previously observed in a novel, more complex material, multicomponent garnet $Gd_3Ga_3Al_2O_{12}$:Ce^{3+} (GGAG:Ce) [8]. Ultraefficient GGAG:Ce single crystal scintillator was discovered in 2011 [28] and immediately became a hot topic in the scintillator community [28–32]. We now monitor simple garnets in a broad temperature region 8–500 K and observe practically constant signal throughout. These results show that quantum tunneling between activator and nearby trap(s) is at work in another wide class of scintillating materials. Previously observed features in GGAG:Ce delayed decay are discussed in the light of results currently obtained for simple garnets.

2. Samples and experimental details

Pr^{3+}-doped $Lu_3Al_5O_{12}$ (LuAG) and $Y_3Al_5O_{12}$ (YAG) single crystals with Pr concentration in the crystal of 0.19 wt% and 0.16 wt%, respectively, were grown by the Czochralski technique from 5N Lu_2O_3 and 5N Pr_2O_3 raw powders by CRYTUR, Ltd. (Turnov, Czech Republic).

Two LuAG:Ce single crystals were grown by the Czochralski technique in a molybdenum crucible under a reducing atmosphere by CRYTUR, Ltd. (Turnov, Czech Republic). The samples with Ce concentration 0.03, and 0.12 wt% were grown from 5N Lu_2O_3 and 4N Al_2O_3 raw powders.

LuAG:Ce nanopowder was prepared by UV-irradiation of aqueous solutions containing soluble metal salts. The details of preparation can be found in [33]. Ce(III) nitrate hexahydrate was added to the solutions according to the stoichiometry $Lu_{3−x}Ce_xAl_5O_{12}$, where x = 0.5%. Photochemically prepared powder was calcined at 1200 °C.

Single crystalline LuAG:Ce thin film was grown by liquid phase epitaxy from a $BaO–Ba_2O_3–BaF_2$ flux on (1 1 1) oriented LuAG substrate; the growth process is described elsewhere [34,35]. The thickness of the film was 12.3 μm and the Ce concentration 0.28%.

Two single crystals of YAG:Ce were grown by the Czochralski technique from 6N Y_2O_3 and 5N Al_2O_3 raw powders in a molybdenum crucible by CRYTUR, Ltd. (Turnov, Czech Republic). The Ce concentration in these crystals was about 0.32 wt% of CeO_2 in the melt (later designated as low Ce) and 1000 ppm of Ce in the high quality industrial single crystal (later designated as high Ce).

Absorption spectra were measured by the Shimadzu 3101PC spectrometer in the 190–1200 nm range. Photoluminescence emission (PL) and excitation (PLE) spectra were measured by using a custom made 5000M Horiba Jobin Yvon spectrofluorometer. Excitation was realized with a deuterium steady state lamp. Temperature between 8 and 497 K was controlled by a Janis Instruments closed cycle refrigerator. PL delayed recombination decays were excited by a microsecond xenon flashlamp and measured using the multichannel scaling method. The decay data were fit by three-exponential function used the *Spectra-Solve* program package. From the fit three delayed decay times were determined.
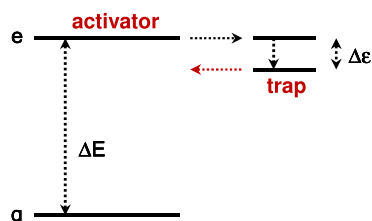

Fig. 1. Simple scheme for a tunneling process between the activator, with ground state *g* and excited state *e*, and the trap located nearby. The energy separation of the ground and excited state of the activator is ΔE while that of the excited state of the activator and the trap is $\Delta\varepsilon$.
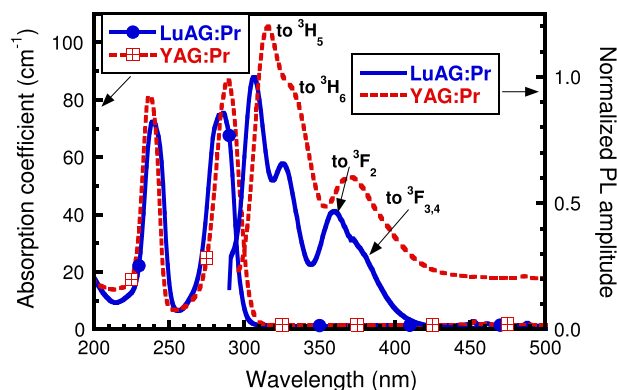

Fig. 2. Absorption spectra at RT and normalized PL spectra at 8 K of LuAG:Pr^{3+} and YAG: Pr^{3+} single crystals, excitation wavelength is 285 nm. Emission spectrum of YAG:Pr^{3+} is vertically shifted by 0.2.

The errors on decay times from the shortest to the longest were estimated from statistical error of the number of counts in each channel to be up to 1%, 2–3% and 4–5%, respectively. Based on these estimates the error on delayed recombination intensities obtained by integration of the decay curves is estimated to be up to 5%.

3. Results and discussion

3.1. Pr^{3+}-doped aluminum garnets

Absorption spectra at room temperature (RT) and PL emission spectra of LuAG:Pr^{3+} and YAG:Pr^{3+} measured at 8 K are displayed in Fig. 2. The lowest absorption band, at about 280 nm, corresponds to a transition from the $^3H_4$ ground state to the lowest $^5d_1$ state of Pr^{3+}, while the band at about 240 nm corresponds to a $^3H_4 \to 5d_2$ transition [36]. The PL emission between 280 and 420 nm contains several bands corresponding to $5d_1 \to 4f$ transitions of Pr^{3+} (final ^3H_x and ^3F_x multiplet 4f states are well resolved as shown in Fig. 2). The same transitions in a YAG host are shifted to lower energies with respect to the LuAG host.

In Fig. 3a we display the delayed recombination intensity of LuAG:Pr^{3+} and YAG:Pr^{3+} measured under UV excitation into the $4f \to 5d$ transition of the Pr ion within the 8–500 K temperature interval. An almost perfectly constant signal is observed in the whole temperature range. Such temperature independence of the delayed recombination intensity manifests two important features. First, it supports the hypothesis of quantum tunneling between the activator and nearby trap(s) as suggested in [1,9]. Second, it also shows no signs of thermally induced ionization of the Pr^{3+} excited state and therefore confirms the known temperature stability of Pr^{3+}-doped aluminum garnets well above RT [2]. Fig. 3b shows an example of delayed recombination decay at 8 K for both Pr^{3+}-doped LuAG and YAG. The decay is approximated by a three-exponential function indicated in the figure. Such complex decay represents a tunneling between an activator and several traps: either traps of a different kind or traps of the same kind distributed at various distances from the activator.

3.2. Ce^{3+}-doped lutetium aluminum garnet

The results for two Ce^{3+}-doped LuAG single crystals are displayed in Figs. 4 and 5. We compare two samples with different concentrations of Ce^{3+} in the crystal, *low* and *high* (cf. Section 2). The Ce concentration difference can be assessed from absorption spectra shown in Fig. 4, namely from the Ce^{3+} absorption band
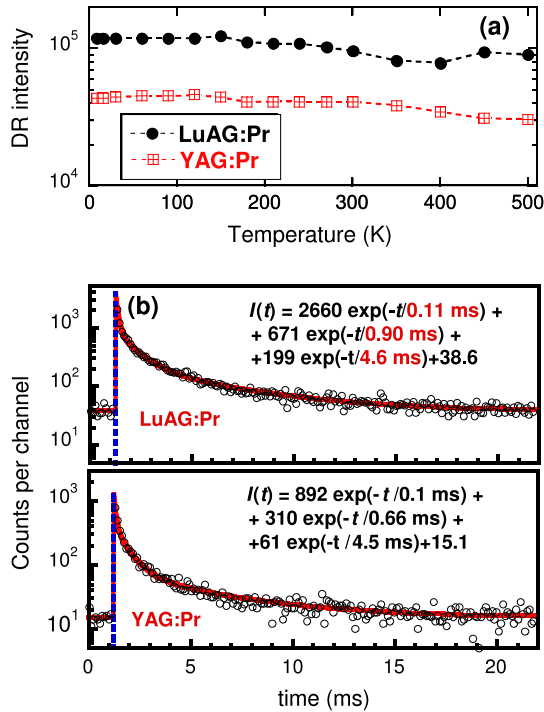
Fig. 3. In (a) delayed recombination intensity of LuAG:Pr^{3+} (λ_{ex} = 285 nm, λ_{em} = 310 nm) and YAG: Pr^{3+} (λ_{ex} = 285 nm, λ_{em} = 325 nm) single crystals as a function of temperature. In (b) delayed recombination decays at 8 K. Empty circles are experimental data, solid line is the fit of function $I(t)$, given in the figure, to the data.
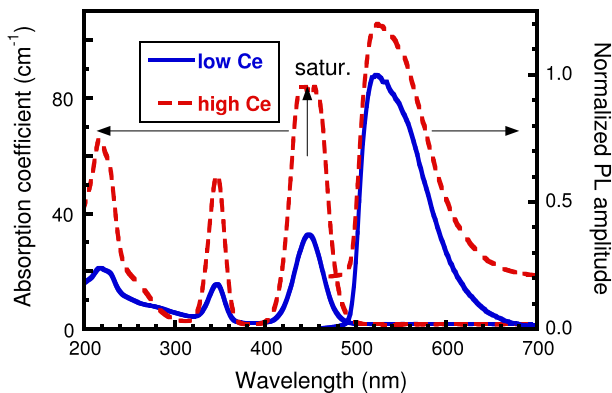

Fig. 5. In (a) delayed recombination intensity of two crystals of LuAG:Ce^{3+} (λ_{ex} = 450 nm, λ_{em} = 530 nm) with the Ce concentration of 0.03 wt% (low Ce) and 0.12 wt% (high Ce) as a function of temperature. In (b) delayed recombination decays at 8 K. Empty circles are experimental data, solid line is the fit of function $I(t)$, given in the figure, to the data.

to the overall delayed recombination signal would drop. In any case, as for the Pr^{3+}-doped LuAG above, there is no indication of thermal ionization of the Ce^{3+} excited state which also gives a confirmation of high thermal stability of Ce-doped LuAG. The delayed recombination decays at 8 K (Fig. 5b) are complex, as above, approximated by a three-exponential curve.

In Figs. 6 and 7 we present data for LuAG:Ce^{3+} in the form of a single crystalline thin film prepared by liquid phase epitaxy and nanopowder prepared by radiation synthesis. Room temperature PLE and PL spectra show two lowest excitation bands of Ce^{3+} and broad emission due to Ce^{3+}, respectively. Correspondingly, delayed recombination intensities as a function of temperature are displayed in Fig. 7b. The intensity of the thin film sample is perfectly constant up to 200 K and as above, shows a similar drop above 200 K. The intensity course of a nanopowder sample presents

Fig. 4. Absorption spectra and normalized PL spectra at RT of LuAG:Ce^{3+} single crystals. Ce concentration is 0.03 and 0.12 wt%, designated as low Ce and high Ce, respectively. PL spectra are excited at 450 nm. PL spectrum of the high Ce sample is vertically shifted by 0.2.

4f → 5d$_2$, peaking at about 350 nm. The lowest Ce^{3+} absorption band 4f → 5d$_1$, peaking at about 450 nm is saturated for the sample with a high concentration of Ce.

In both samples, the delayed recombination intensity under UV excitation of the 4f → 5d$_1$ transition of Ce^{3+} ion is practically constant up to 200 K (Fig. 5a). There is a slight decrease of intensity in the temperature interval 200–500 K. This drop in intensity for Ce^{3+}-doped LuAG compared to Pr^{3+}-doped LuAG above may be due to decreasing amplitude of absorption into 5d$_1$ band in favor of increasing amplitude of absorption into the 5d$_2$ band with increasing temperature [37]. Alternatively, with increasing temperature the activator can get out of resonance with some of the previously (at lower temperatures) participating traps ($\Delta \varepsilon$ is too big– cf. Fig. 1) due to which the total number of traps contributing
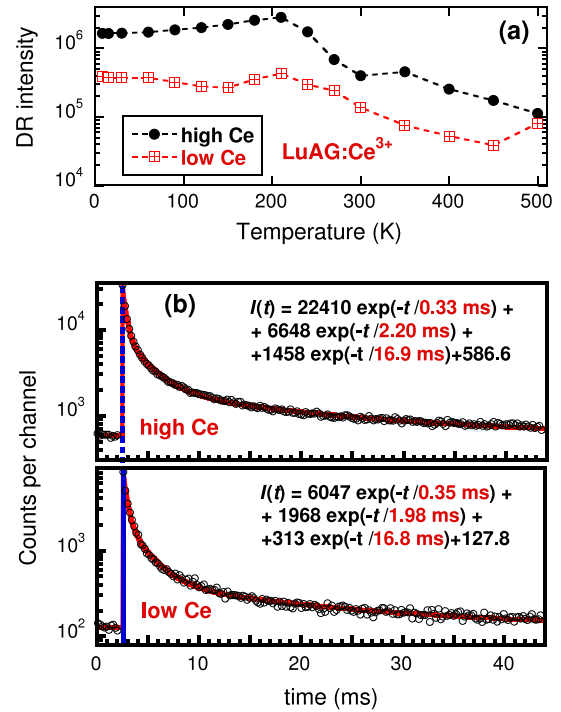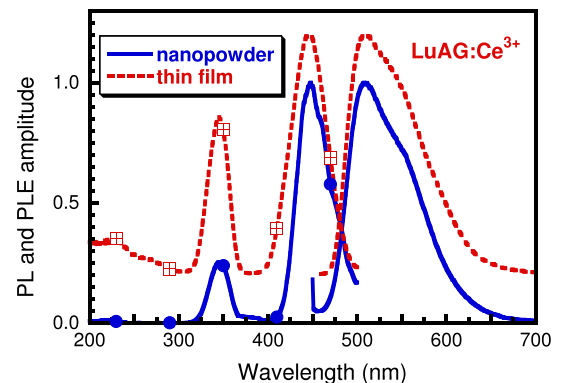

Fig. 6. Normalized PLE and PL spectra of the LuAG:Ce^{3+} compacted nanopowder (λ_{ex} = 430 nm, λ_{em} = 510 nm) calcined at 1400 °C and thin film λ_{ex} = 445 nm, λ_{em} = 510 nm) at RT.

Fig. 8. Absorption spectra and normalized PL spectra at RT of YAG:Ce^{3+} single crystals. The concentration was 0.32 wt% of CeO_2 in the melt and 1000 ppm of Ce^{3+} in the crystal, designated as *low Ce* and *high Ce*, respectively. PL spectra are excited at 430 nm. PL spectrum of the *high Ce* sample is vertically shifted by 0.2.

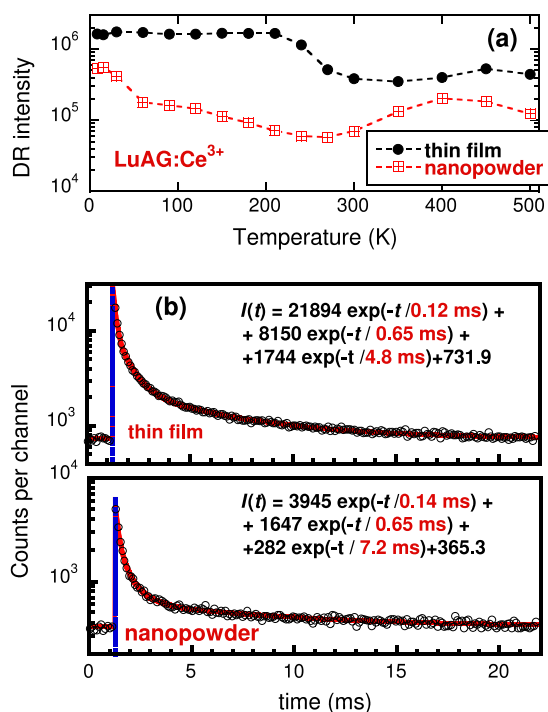**Fig. 7.** In (a) delayed recombination intensity of LuAG:Ce^{3+} thin film (λ_{ex} = 435 nm, λ_{em} = 530 nm) and nanopowder (λ_{ex} = 435 nm, λ_{em} = 525 nm) as a function of temperature. In (b) delayed recombination decays at 8 K. Empty circles are experimental data, solid line is the fit of function $I(t)$, given in the figure, to the data.

somewhat irregular variations. However, intensity changes do not exceed an order of magnitude and may possibly be due to distortions caused by light scattering on the powder grains. On the average, taking into account the possible distortions mentioned earlier one may consider the signal almost constant. The delayed recombination decays at 8 K (Fig. 7b) are again complex, approximated by a triple exponential. The results on this variety of LuAG:Ce samples confirm the plausibility of the quantum tunneling hypothesis.

3.3. Ce^{3+}-doped yttrium aluminum garnet

The results for two Ce^{3+}-doped YAG single crystals are displayed in Figs. 8 and 9. As in the case of a LuAG host, we compare two samples with different concentration of Ce^{3+} in the crystal, low and high. As in LuAG, the Ce concentration difference can again be assessed from the Ce^{3+} absorption band $4f \rightarrow 5d_2$ peaking at about 350 nm (see Fig. 8). The lowest Ce^{3+} absorption band $4f \rightarrow 5d_1$ peaking at about 450 nm is also saturated for the sample with high concentration of Ce.

In both samples the delayed recombination intensity under UV excitation into the $4f \rightarrow 5d_1$ transition of Ce^{3+} ion is practically constant up to 200 K (Fig. 9a). The drop of intensity above 200 K observed in LuAG is found for the YAG host as well. The drop is more significant in the low Ce sample. This may be connected with the quality of the sample. While high Ce crystal is designated by the producer as a *high quality* industrial sample, the quality of the low Ce sample is supposedly lower, which suggests a higher number of traps compared to the high Ce crystal. This could lead to a reduction in participating traps in the tunneling process as the temperature increases when the activator's excited state gets out of resonance with the trap level (as suggested above). Nevertheless, as in LuAG host, no signs of thermally induced ionization are present and thermal stability of YAG:Ce crystals is confirmed up to 500 K. The delayed recombination decays at 8 K (Fig. 9b)
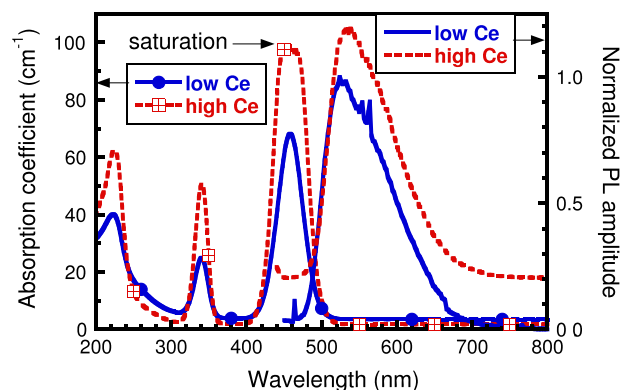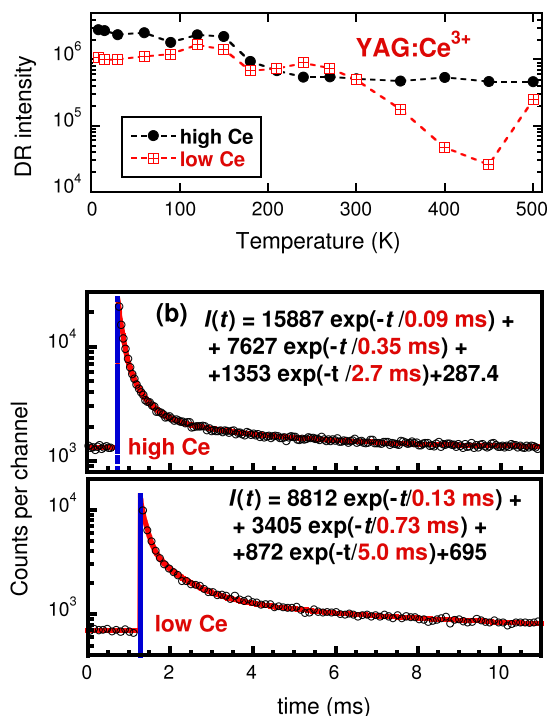

Fig. 9. In (a) delayed recombination intensity of two single crystals of YAG:Ce^{3+} (λ_{ex} = 430 nm, λ_{em} = 535 nm) with the different Ce concentration of 0.32 wt% of CeO_2 in the melt (*low Ce*) and 1000 ppm of Ce^{3+} in the crystal (*high Ce*) as a function of temperature. In (b) delayed recombination decays at 8 K. Empty circles are experimental data, solid line is the fit of function $I(t)$, given in the figure, to the data.

are complex, as in all cases studied, and are approximated by a three-exponential curve. Therefore participation of several traps in the tunneling process is expected.

3.4. GGAG:Ce^{3+} versus simple garnets

The delayed recombination decay intensity of a GGAG:Ce^{3+} crystal was monitored in the 8–230 K temperature range in [8]. For convenience, the results are reproduced in Fig. 10.

Unlike all other silicates studied in the same work, the signal is not constant and there is an intense peak around 100 K. Before hypothesizing on the origin of the peak in [8] we first wanted to test the simple garnets to see whether such a feature might be universal in garnet hosts. None of simple garnets studied in the pres-
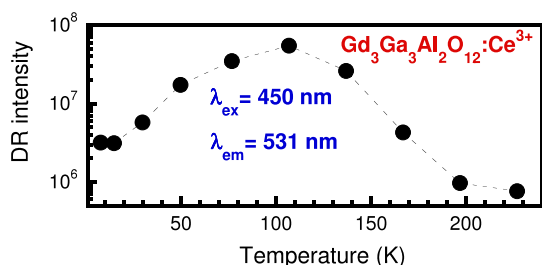
Fig. 10. Delayed recombination decay intensity of GGAG:Ce^{3+} as a function of temperature. Excitation and emission wavelengths are displayed in the figure from [8].

ent work have manifested such a peak. Therefore, the peak is uniquely present in GGAG:Ce and would be associated with particular features of this host. The band structure calculations of this multicomponent garnet host have not yet been reported. However, compared to LuAG and YAG, due to the presence of Gd^{3+} and Ga^{3+} cations, its band gap shrinks and a composition of the bottom of the conduction band profoundly changes [38–40]. These changes would also affect the positioning of the Ce^{3+} excited states together with that of electron traps below the conduction band. Recently it has been shown that the shallow trap levels in GGAG:Ce are quasi-continuously distributed rather than being discrete [41]. Such feature might explain the peak around 100 K in question. With increasing temperature the number of participating kinds of traps (from the distribution) would be increasing up to the point when they start to get out of resonance with the activator's excited state and the signal would start decreasing. Besides the presence of the peak around 100 K, there is a slight drop of the DR signal around 200 K compared to values at the low temperature limit in GGAG:Ce (cf. Fig. 10). In contrast to the peak around 100 K, such a drop was consistently observed in all the studied Ce-doped simple garnets (see above).

4. Conclusion

Quantum tunneling between traps and activator centers had previously been shown to play a decisive role in low temperature delayed recombination [8–10]. In this article we confirmed similar behavior for another wide class of scintillating materials, namely simple garnets. Compared to the silicates previously studied, tunneling in garnets involves deeper trap(s). This follows from the position of the activator's excited state with respect to the conduction band. For example, the 5d$_1$ state of Ce^{3+} in Y$_3$Al$_5$O$_{12}$ is situated about 1.2 eV below the conduction band [42], while that of Pr^{3+} in Lu$_2$Si$_2$O$_7$ is about 0.5 eV [7] and that of Ce^{3+} in Lu$_2$SiO$_5$ is about 0.45 eV [43]. We studied various forms of garnets, in particular Czochralski grown single crystals, LPE thin film and a nanopowder prepared by a radiation synthesis. Different activators, Ce^{3+} or Pr^{3+}, have been used. The change due to the activator's concentration between the low and high level within the same sample batch was checked. For all samples studied we obtained consistent results showing practically constant delayed recombination intensity within the entire temperature range 8–500 K. Such results confirm first, the presence of quantum tunneling between the activator and nearby traps and second, the absence of thermal ionization of the activator's excited state well above room temperature.

Acknowledgements

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