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Decay kinetics of Ce^{3+} ions under γ and KrF excimer laser excitation in CeF_3 single crystals

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Abstract. Measurements of the decay kinetics of regular Ce^{3+} emission in CeF_3 single crystals have been performed under photo-excitation and γ (^{22}Na radioisotope) excitation. While flashlamp-excited photoluminescence decay is distorted mainly by the influence of the 'near-surface' traps, the multiexponential nature of the scintillation decay is explained using pair interaction of close-lying Ce^{3+} excited ions, which account for the discrete character of their distances. Measurements of the same photoluminescence decay under high-intensity laser excitation show a similar departure from exponentiality as in the case of scintillation decay and support the proposed model.

1. Introduction

The optical properties of CeF_3 single crystals have been studied intensively in the last few years in connection with its fast scintillation characteristics as reported by Anderson [1] and Moses and Derenzo [2]. These characteristics are of interest for the new generation of fast and heavy scintillation detectors needed in high-energy physics, medical and industrial applications [3].

The CeF_3 photoluminescence (PL) including its kinetics has already been well understood. The emission bands at 286 and 305 nm (well resolved at low temperatures [4]) are ascribed to radiative transitions between 5d and the split 4f ground-state levels of Ce^{3+} cations at the regular structural site. The broad 340 nm band is ascribed to the same transition of Ce^{3+} , which is perturbed by a nearby intrinsic defect, most probably an anion vacancy in its first coordination sphere [4]. The typical decay times reported are 17–20 ns for the emission of regular Ce^{3+} cations (Ce_{reg}) and 30–40 ns for perturbed Ce^{3+} sites (Ce_{per}). Both radiative and nonradiative energy transfer processes between Ce_{reg} and Ce_{per} were evidenced from the detailed analysis of the PL decay curves [5]. Also, the influence of the 'near-surface' trap states was invoked to explain the emission features observed in PL [4].

Even faster components with a typical decay time of about 2–5 ns have been reported in several papers under γ or x-ray excitation [6–8]. These cannot be explained by nonradiative energy transfer from Ce_{reg} to the surface states as in PL because, especially under γ excitation, the volume of the crystal is excited. These very short decay times have been attributed to some quenching process associated with excited Ce^{3+} emission centres rather

than to some other independent physical processes other than those observed in PL [9]. The absorption of a γ or x-ray photon in the lattice creates high-density excitation regions along its path. This effect was proposed recently [7] to explain the fast decay by considering mutual interactions between excited Ce^{3+} ions and/or possibly transient defects induced by the interaction of the crystal lattice with the γ -ray or x-ray photon.

It has been estimated that the relatively low scintillation light yield of CeF_3 is mainly due to losses introduced by poor energy transfer efficiency from electron-hole pairs and anion exciton to Ce^{3+} emission centres [8]. However, no qualitative model was proposed to explain the observed distortion of scintillation decay of Ce_{reg} emission centres; moreover, no quantitative estimate of these additional losses, which are introduced in this way in overall scintillation performance, is available.

It is the aim of this paper to explain the observed scintillation decay of Ce_{reg} centres by introducing the interaction between two excited Ce^{3+} ions and to consider their discrete distance distribution in the CeF_3 lattice. We also provide a quantitative estimate of additional nonradiative losses introduced by this process. To support the proposed model, the decay of Ce_{reg} emission was measured under high-power KrF excimer laser excitation in order to obtain a high density of Ce^{3+} excited ions that would be comparable to the density expected locally under γ -ray or x-ray excitation.

2. Experimental details

Three set-ups were used to obtain the experimental results presented. A spectrofluorometer 199S (Edinburgh Instruments) was used to measure emission spectra under steady-state x-ray and γ -ray excitation and a nanosecond hydrogen-filled flashlamp was used for decay kinetics measurements (see [10] for details). γ -ray-excited decays were measured using a ^{22}Na radioisotope and the coincidence method of Bollinger and Thomas [11]. Finally, decay under laser excitation was measured using the LISS-Multilaser, a model 780U excimer laser with a KrF mixture ($E_{\text{max}}^{\text{puls}} = 7$ mJ; $\lambda_{\text{em}} = 249$ nm; $\Delta t = 10$ ns) combined with a closed-cycle refrigerator and monochromator (typically 2 nm dispersion) with Hamamatsu R1398 photomultiplier detection coupled to the Tektronix 2440 digital memory oscilloscope. Emission spectra under the same laser excitation were detected by OMA 1461 EG&G. Photo-excited and x-ray-excited spectra were measured in 45° geometry (emission from the sample surface). The experimental data in each set-up were transferred to personal computers for further evaluation. The emission spectra were corrected for the spectral response of the detection and a deconvolution procedure was used to extract true decay times from the observed decay curves. To deconvolute the γ -excited decays, the instrumental response to the single γ -photon was reconstructed by the sum of several Gaussians from measured overall decay of BaF_2 using published decay time constants (0.88 ns for the fast component and 570 ns for the slow component [12]). The best time resolution used is about 80 ps/channel, and up to 10% error can be expected in the scintillation decay times determined by the procedures described.

CeF_3 samples of the latest generation (oxygen and cation impurity 'free') produced in 1993 year by Optovac, USA, and Preciosa Crytur, Czech Republic, have been used throughout the experiment.

3. Experimental results

The emission spectra of CeF_3 obtained under different excitation and experiment geometry are shown in figure 1, where the UV (curve a), x-ray (curve b) and γ -ray (curve c) excitation

were used. The relative ratio of Ce_{reg} to Ce_{per} emission intensity is strongly modified by the penetration depth L_p ($L_p < 10^{-6}$ m in PL measurement, about 1 mm under x-ray excitation (Mo anticathode; 35 kV) and several centimetres for 511 keV photons from a ^{22}Na source) or by the geometry of the experiment (i.e. the path length of emitted photons in the crystal); the emission is collected from the sample surface for PL and x-ray excitation (45° geometry) or from the side of the sample for γ excitation (90° geometry). In the 90° geometry and/or γ excitation, the short-wavelength side of the CeF_3 spectrum is considerably attenuated because of self-absorption of the CeF_3 lattice (curve c).

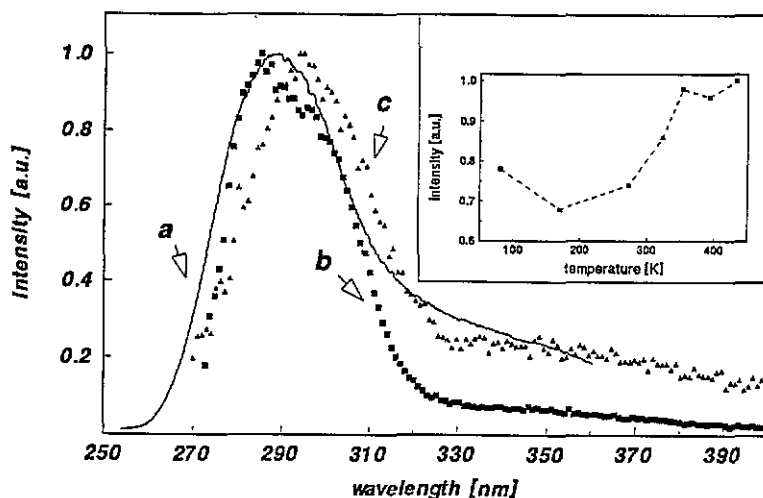


Figure 1. Emission spectra of CeF_3 single crystals at 295 K (a.u., arbitrary units): curve a, KrF-excimer-laser-excited spectrum, $\lambda_{ex} = 249$ nm, sample from Preciosa; curve b, x-ray-excited spectrum, sample from Preciosa; curve c, ^{22}Na -radioisotope (511 keV) excited spectrum, sample from Optovac. The inset shows the temperature dependence of the Ce_{reg} emission intensity (286 and 305 eV peaks; see text for details) under x-ray excitation.

The temperature dependence of x-ray-excited Ce_{reg} emission shown in the inset of figure 1 is obtained using a three-Gaussian approximation of the emission spectra and by taking the intensity proportional to the sum of two Gaussians (286 and 305 nm peaks) appropriate to Ce_{reg} emission (for the details see [5]). A similar increase in intensity is observed between 250 and 400 K as in the case of excitation using synchrotron radiation [7], even though the decrease between 295 and 80 K is more pronounced in [7]. This correlates with a steeper decrease in scintillation decay at 80 K observed in [7] in comparison with our results shown later in figure 3 and is most probably due to the different qualities of the CeF_3 samples used in our experiment and by Pedrini *et al* [7].

The PL decay is given in figure 2. In figure 2(a), a simple single-exponential fit is given (decay time, 16.8 ns), showing distinct disagreement in the initial part of the decay. A much better fit can be obtained using the Forster formula $I(t) = A \exp(-t/\tau_0 - b^*t^{1/2})$, which is generally appropriate when nonradiative energy transfer from observed (donor) centres occurs in other (acceptor) centres via a dipole-dipole interaction.

^{22}Na radioisotope-excited decay of Ce_{reg} emission is given in figure 3. The diverse behaviour in comparison with figure 2 is evident. A three-exponential approximation with decay times $\tau_1 = 1.1-1.3$ ns, $\tau_2 = 6-8$ ns and $\tau_3 \simeq 19$ ns satisfactorily fits the decays

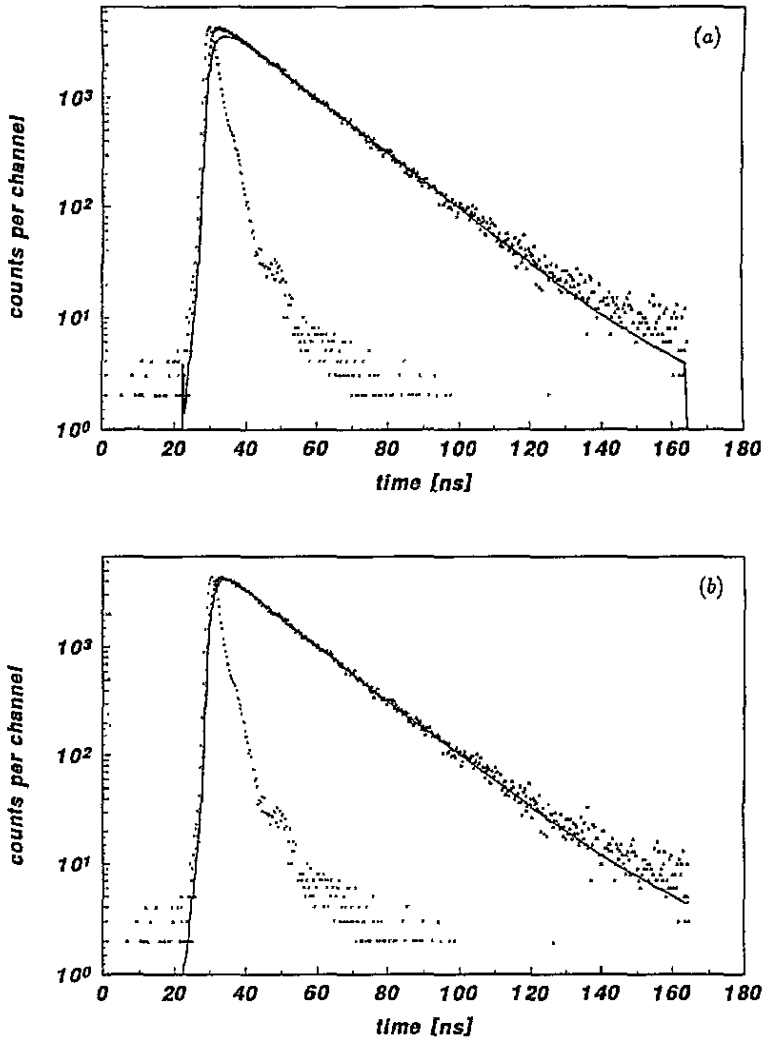


Figure 2. The PL decay ($\lambda_{\text{exc}} = 250 \text{ nm}$, $\lambda_{\text{exc}} = 290 \text{ nm}$) at 295 K. Instrumental response is given by smaller dots. The solid curve is the convolution of the instrumental response with the approximation function defined as (a) a single exponential (16.8 ns decay time) and (b) the Forster formula given in the text ($\tau_0 = 19.5 \text{ ns}$; $b = 1.66 \times 10^{-6}$).

at 80 K and at room temperature for all the samples studied. The relative intensity of the process connected with τ_3 (i.e. process connected with radiative PL decay of Ce^{3+}) increases as the temperature rises (about 45% at 80 K and 55% at 300 K).

4. Discussion

The results observed for the emission spectra and the PL decay kinetics are in general agreement with previously published data. It should be stressed, in addition to the growth technology itself, that several factors are to be taken into account when evaluating material quality from PL measurements. This involves mainly the technology of cutting and polishing

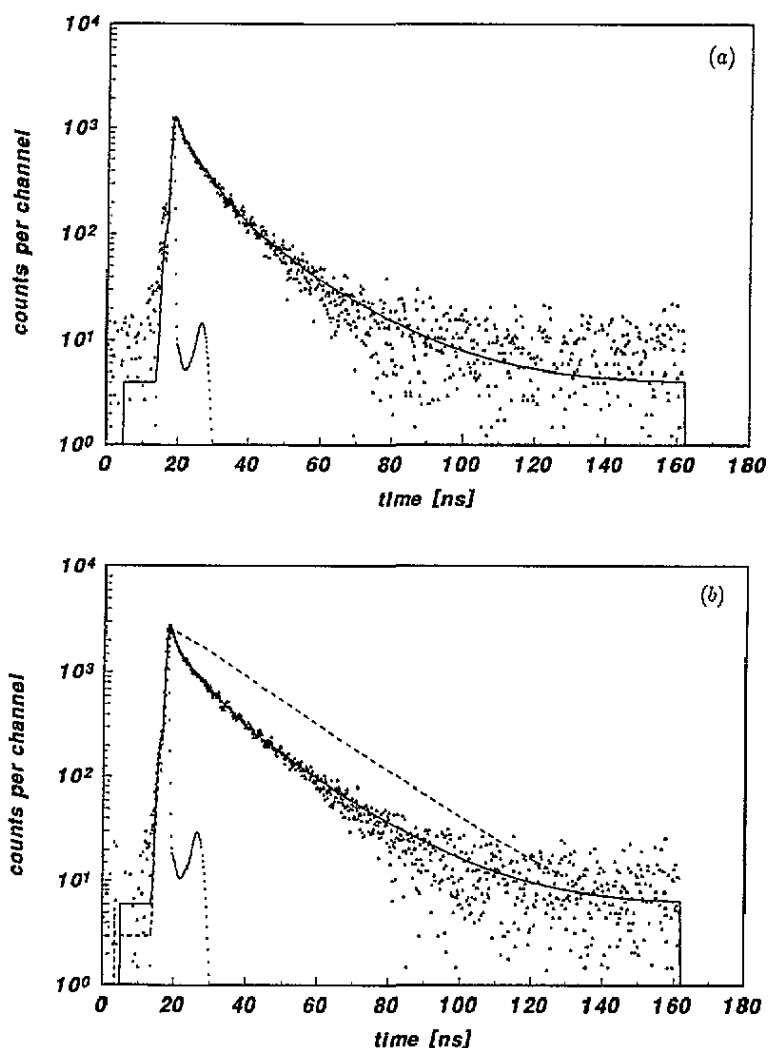


Figure 3. Scintillation decay (^{22}Na excited) of CeF_3 ($\lambda_{em} = 290$ nm). The instrumental response is also indicated by smaller dots; for its construction see section 2. Convolution of instrumental response and sum of three exponentials (pre-exponential factors A_i and decay times τ_i) is indicated by a solid curve with the following parameters: (a) $T = 295$ K; $A_1 = 0.56$, $\tau_1 = 1.3$ ns; $A_2 = 0.45$, $\tau_2 = 6.5$ ns; $A_3 = 0.23$, $\tau_3 = 19$ ns; (b) $T = 80$ K; $A_1 = 0.413$, $\tau_1 = 1.1$ ns; $A_2 = 0.555$, $\tau_2 = 6.0$ ns; $A_3 = 0.17$, $\tau_3 = 19$ ns. The broken line is the convolution of the instrumental response and single exponential (decay time, 19 ns).

the samples, resulting in a certain surface quality, as well as the environment where the samples are stored. Such factors influence the surface layer structure and quality, which may in turn strongly influence the observed PL properties. The fact that Forster kinetics are needed to describe the PL decay at 290 nm, when x-ray-excited spectra indicate a very low intensity of Ce_{per} emission coupled with the remarkably higher content of Ce_{per} emission in PL emission spectrum, provides evidence of the different emission properties of the 'near surface' and bulk parts of the sample.

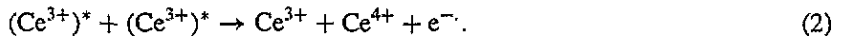
On the contrary, the scintillation decay in figure 3 is not related to the surface quality

since the sample volume is excited. The nearly equal decay times measured at various temperatures and using different samples led us to the idea that the quenching of Ce_{reg} emission is due in this case to interaction with another defect, which is created also by the incident γ photon and situated at discrete distances from the excited Ce_{reg} centre under consideration. Assuming a dipole-dipole interaction between the centres we can deduce for the observed decay time τ

$$\frac{1}{\tau} = \frac{1}{\tau_0} + w \quad w = \frac{1}{\tau_0} \left(\frac{R_0}{R} \right)^6 \quad (1)$$

where τ_0 is the unaffected (radiative) decay time of the Ce_{reg} ion itself, w is the microscopic nonradiative transfer rate to the acceptor centre at the distance R , and R_0 is the critical radius. By taking $\tau_0 = 19$ ns and solving equation (1) for the two shortest decay times observed in the scintillation decays in figure 3 (i.e. τ_1 and τ_2), the best agreement is obtained for $R_0 = 6.5$ Å, $R_1 = 4.2$ Å and $R_2 = 6$ Å. These calculated distances are very close to those between Ce^{3+} ions in the CeF_3 structure. There are 12 NN Ce^{3+} ions at a distance of 4.07–4.36 Å from the central ion, six NNN Ce^{3+} ions at a distance of 5.94–6.04 Å from the central ion, and 20 NNNN Ce^{3+} ions at a distance of 7.11–7.27 Å from the central ion. The value of R_0 is close to that derived for the interaction between C_{reg} and C_{per} at 4.2 K ($R_0 = 7$ Å [5]). Using the derived value of R_0 , we can calculate the decay time value appropriate for the interaction between the central Ce^{3+} ion and those from the NNNN sphere; $\tau = 11$ ns. Because of the noise in the experimental decay curves and the relatively small difference between 11 ns and $\tau_3 = 19$ ns, it is difficult to extract this decay time from the curve in advance, but it is possible to obtain a very good fit of the decays using all four values of the decay times found.

Hence, the observed decay under γ excitation can be explained under the assumption of a dipole-dipole interaction between two excited (Ce^{3+})* ions situated in their regular cationic positions in the CeF_3 structure:



In fact, this interaction between two such (Ce^{3+})* ions results in the loss of two emission photons, as a free electron created in conduction band has a very slight probability of radiative recombination with a Ce^{4+} hole centre [8].

The observed increase in the relative intensity of the process with the longest decay time $\tau_3 = 19$ ns with rising temperature is consistent with the observed increase in the intensity under x-ray excitation (inset of figure 1 and [7]) and can be explained under the assumption that the energy stored in the Ce^{3+} excited state starts to migrate through the cationic sublattice with increasing temperature. The migration may separate nearby excited Ce^{3+} ions and lower the nonradiative losses connected to process (2). Although a relatively complicated calculation is reported in [8] to prove that there is no Ce_{reg} - Ce_{reg} energy transfer within the framework of the resonant dipole-dipole Inokuti-Hirayama model, phonon-assisted energy transfer in the cationic sublattice is probably more appropriate in describing the situation in CeF_3 at sufficiently high (i.e. room) temperatures. Strong support for this concept can be derived from the results obtained for heavily doped $CeF_3: Cd^{2+}$, where a great change in critical radius for Ce_{reg} - Ce_{per} nonradiative energy transfer was found when comparing the values at 4.2 and 300 K [5]. Such a change in critical radius is impossible to explain under the assumption that Ce^{3+} excitations are immobile at all temperatures.

The migration ability of cationic Ce^{3+} exciton can be explained by the competition of two processes:

(1) the tendency of cationic exciton to become self-localized and the speed of this process as discussed by Williams *et al* [13];

(2) the interaction of the 5d excited electron with lattice vibrations [14], which may provide the main contribution for cationic exciton hopping.

A quantitative estimation of the nonradiative losses introduced by this pair interaction between Ce^{3+} excited ions can be deduced from the decay curve in figure 3(a). If these losses are not present, a single-exponential decay with about 19–20 ns decay time (broken line in figure 3(b)) would be observed. By simple comparison of the areas under this hypothetical decay and true experimental decay in figure 3(b) or its three-exponential approximation, we obtain a decreased light output of about 50% due to the pair interaction of excited Ce^{3+} ions in the case of 511 keV exciting photon energy.

It is worthwhile discussing the possible nature of the defect interacting with excited Ce^{3+} ions in more detail. The colour centres and radiation-induced defects in CeF_3 have already been studied in [6, 15, 16]. Several modifications of F centres most probably exist at low temperatures, showing wide absorption bands in the visible region. Other absorption bands in the near UV are probably related to more complicated centres, in which the presence of extrinsic impurities is not excluded.

It follows from our scintillation decay approximations and model proposed that, whatever the nature of the defect, it is situated at discrete distances from the excited Ce^{3+} ion under consideration, which are close to regular Ce^{3+} sites in the CeF_3 structure. It is impossible to find realistic values for R_0 , R_1 and R_2 when considering anion sites. Furthermore, the large magnitude of the effect (i.e. the difference between scintillation and PL decay) rules out any extrinsic impurity or related defect. This is further supported by the fact that the same results were achieved for crystals from two different producers. Hence, the interaction between two excited Ce^{3+} ions near together is the most probable process for explaining the observed differences between PL and scintillation decay.

The model proposed is correct assuming that a maximum of two excited Ce^{3+} ions are present in the volume considered (Ce^{3+} ion and three surrounding Ce^{3+} spheres) of the crystal. This assumption seems reasonable when taking into account the result of Monte Carlo simulations of the creation of the excited region in CeF_3 by the 100 eV photon [17].

To support this model, we measured the decay kinetics of Ce_{reg} emission under intense laser excitation, thereby creating a comparable local density of Ce^{3+} excited ions as expected under γ excitation. In this way, no direct ionization or excitation of deep-core CeF_3 levels occurs and the situation is simplified. The 249 nm line of a pulsed KrF excimer laser (pulse width $\Delta t = 10$ ns) was used for this purpose. The measurements were performed with $E_{pulse} = 50 \mu J$ at the surface of the CeF_3 sample. Changing the spot size from about $0.5 \text{ mm} \times 2 \text{ mm}$ to $2 \text{ mm} \times 8 \text{ mm}$ alters the excitation density roughly about 16 times. An example of the decays is given in figure 4. While at the lowest excitation density (spot $2 \text{ mm} \times 8 \text{ mm}$) the decay is well approximated by a single exponential ($\tau = 16$ –17 ns), in the case of decreasing spot size the decay deviates from the single-exponential course and a two-exponential fit is more appropriate with $\tau_1 = 4$ –6 ns and $\tau_2 = 17$ –18 ns.

We can exclude the influence of pronounced heating of the sample surface by the laser pulse (i.e. increasing the sample surface temperature until the onset of temperature quenching), because the highest excitation density used is still two orders of magnitude lower than that causing observable damage of the surface (most probably just by melting the surface of the sample); we found these effects at about 5 mJ pulse energy and the laser spot size $0.5 \text{ mm} \times 2 \text{ mm}$. Moderate overheating is not excluded, but it is known that it does not decrease the decay time of Ce_{reg} emission until at least 400 K [7], i.e. no thermal quenching occurs. The possible onset of stimulated emission was also tested, which could

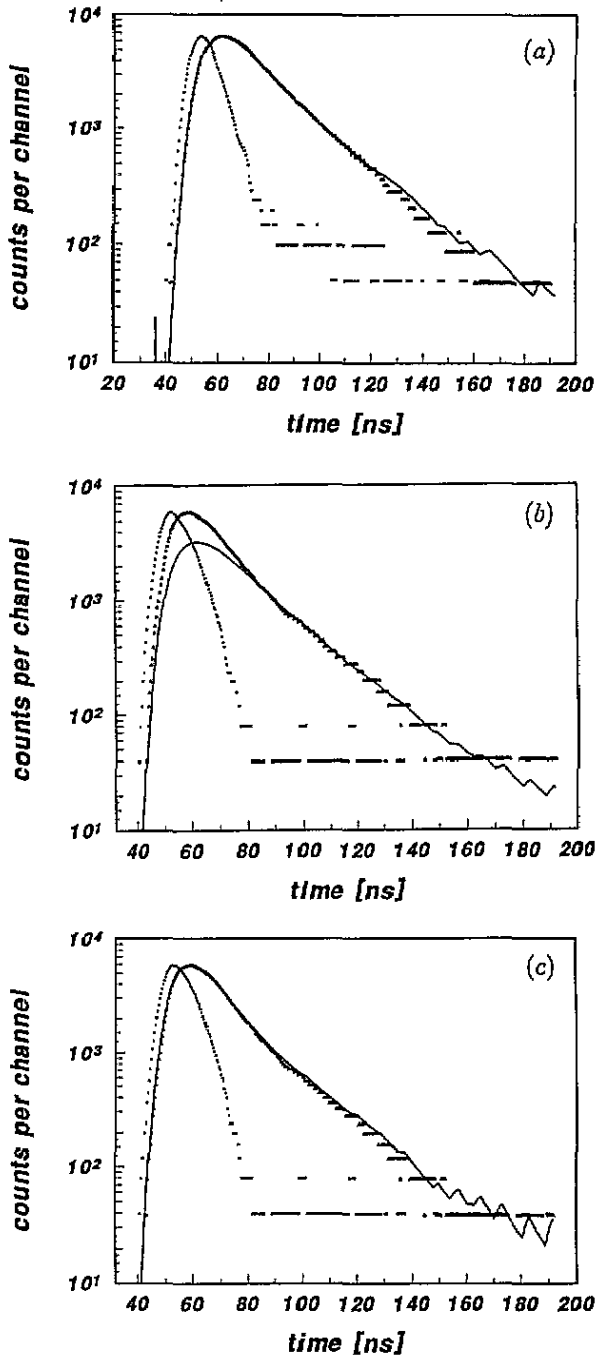


Figure 4. CeF_3 emission decay at 290 nm excited by a KrF excimer laser ($\lambda_{em} = 249$ nm; $E_{puls} = 50 \mu\text{J}$; $\Delta t = 10$ ns) at 295 K. The instrumental response is given by the smaller dots. (a) Laser spot size, 2 mm \times 8 mm; the solid line is the convolution of the instrumental response and a single exponential with a 16.5 ns decay time. (b) Laser spot size, 0.5 mm \times 2 mm; the solid line is the convolution of the instrumental response and a single exponential with a 18 ns decay time. (c) As (b) with a two-exponential fit having the pre-exponential factors A_1 and decay times τ_1 : $A_1 = 0.11$, $\tau_1 = 4$ ns, $A_2 = 0.055$ and $\tau_2 = 18$ ns.

create a decrease in the decay time observed. Namely, the shape of emission spectrum was compared at all excitation densities used, and linearity of emission response with respect to the overall excitation intensity was observed. No changes in emission spectra and no departures from linearity were found.

Furthermore, it is possible to estimate the density of Ce^{3+} excited ions under laser excitation and to compare the percentage of pair-interaction-influenced ions within the proposed model framework and measured decay curve. It can be derived from CeF_3 thin film absorption [18] that about 50% of light at 250 nm is absorbed in a layer about 200 nm thick. Using the known value of the concentration of Ce^{3+} ions in CeF_3 ($1.88 \times 10^{22} \text{ cm}^{-3}$), one can calculate that, in a volume $0.5 \text{ mm} \times 2 \text{ mm} \times 10^{-4} \text{ mm}$, about 3.8×10^{15} Ce^{3+} ions are present and about 3×10^{13} excitation photons are absorbed. Hence, the probability of finding one excited Ce^{3+} ion within three Ce^{3+} spheres mentioned in the text is $P_1 = 39/127 = 0.31$, of finding two excited ions $P_2 \simeq 0.1$, etc. For the same spot size ($0.5 \text{ mm} \times 2 \text{ mm}$) the portion of the decay affected by the interaction between two excited Ce^{3+} ions is given by

$$P_{\text{exp}} = \frac{A_1 \tau_1}{A_1 \tau_1 + A_2 \tau_2} \quad (3)$$

and using the value from the fit of figure 4(c) we obtain $P_{\text{exp}} \sim 0.3$. With respect to the approximate nature and uncertainties in the determination of $P_{1,2}$, the agreement between P_2 and P_{exp} is satisfactory.

To confirm and complete the proposed model, a similar study of $LaF_3:Ce$ (Ce^{3+} concentration of the order of units or a few tens of a per cent) could be performed, in which case the probability of obtaining two $(Ce^{3+})^*$ neighbours would also be modified by the Ce^{3+} concentration.

5. Conclusion

We have derived a model based on the interaction between the couple of excited Ce^{3+} ions situated at their regular cationic positions, which can qualitatively explain the scintillation decay observed for Ce_{reg} emission centres. An estimate of the nonradiative losses introduced by this interaction has been made as well. The results of decay kinetics measurements performed under high-intensity laser excitation provide further support for this interpretation.

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