



Full Length Article

Excitation-dependent luminescence behavior of solvothermally synthesized $\text{LaF}_3\text{:Ce}^{3+}$ nanophosphors

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ABSTRACT

Ce^{3+} -doped LaF_3 nanophosphors synthesized by a solvothermal method are promising candidates for X-ray-induced photodynamic therapy owing to their efficient ultraviolet emission and biocompatibility. While the synthesis conditions of $\text{LaF}_3\text{:Ce}^{3+}$ nanoparticles have been optimized in previous studies, the excitation-dependent luminescence processes remain insufficiently understood. In this work, the luminescence mechanisms of solvothermally synthesized $\text{LaF}_3\text{:Ce}^{3+}$ nanophosphors were investigated using vacuum-ultraviolet (70 nm), ultraviolet (255 nm), and X-ray excitation.

Photoluminescence measurements revealed characteristic Ce^{3+} emission bands centered at approximately 290 and 305 nm, corresponding to the 5d–4f transitions. Distinct differences in the relative emission intensities were observed depending on the excitation source. Under direct excitation at 255 nm, the emission associated with the $^2\text{F}_{7/2}$ terminal state was dominant, whereas host-lattice excitation at 70 nm preferentially enhanced the $^2\text{F}_{5/2}$ -related emission. These results indicate that the relaxation pathway of excited carriers strongly depends on the excitation mechanism.

Temperature-dependent measurements performed between 8 K and 300 K showed that the discrete features observed in the excitation spectra were preserved over the entire temperature range, suggesting that their origin is not related to low-temperature effects. Furthermore, X-ray-excited luminescence exhibited spectral characteristics similar to those obtained under direct Ce^{3+} excitation, suggesting that secondary electronic processes generated by X-ray irradiation may contribute to the excitation of Ce^{3+} luminescent centers.

The present results demonstrate that the emission characteristics of $\text{LaF}_3\text{:Ce}^{3+}$ nanophosphors are strongly governed by excitation pathways and associated energy-transfer processes. These findings provide fundamental insight into the design of highly efficient fluoride-based scintillating nanophosphors for future X-ray-induced photodynamic therapy applications.

1. Introduction

Rare-earth-doped fluoride nanocrystals have attracted considerable interest owing to their low phonon energies, high luminescence efficiency, excellent chemical stability, and low biological toxicity [1]. In particular, LaF_3 is a promising host material because of its wide band gap and its ability to accommodate lanthanide ions without significant lattice distortion [1,2]. Owing to their efficient ultraviolet emission under high-energy excitation, Ce^{3+} -activated fluoride nanophosphors have also been considered as potential scintillating materials for

X-ray-induced photodynamic therapy (X-PDT), in which scintillators convert penetrating X-rays into ultraviolet or visible photons capable of activating photosensitizers [3,4].

Ce^{3+} is one of the most important activator ions for ultraviolet-emitting phosphors and scintillators. Its luminescence originates from the parity-allowed 5d–4f transition, resulting in high radiative probability and short decay times compared with most rare-earth ions [5,6]. Because the 5d orbitals are strongly influenced by the local crystal field, the emission wavelength and excitation characteristics of Ce^{3+} are highly dependent on the host lattice environment [7,8]. Furthermore,

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spin-orbit coupling splits the 4f ground state into the $^2F_{5/2}$ and $^2F_{7/2}$ levels, giving rise to characteristic doublet emission bands in Ce^{3+} -activated phosphors [6].

The optical properties of Ce^{3+} -activated phosphors have been extensively investigated. Berends et al. summarized the luminescence behavior of Ce^{3+} materials from conventional phosphors to nanoscale systems and emphasized the importance of local structure and surface effects on luminescence processes [9]. In addition, Jacobsohn et al. reported that surface Ce^{3+} ions significantly affect the luminescence properties of LaF_3 nanoparticles, suggesting that nanoscale effects play an important role in determining their optical behavior [4].

In our previous work, solvothermally synthesized $LaF_3:Ce^{3+}$ nanoparticles were systematically optimized for biomedical applications. The effects of solvent species, stabilizers, and Ce^{3+} concentration on luminescence intensity, morphology, and colloidal stability were investigated, and PVP-assisted synthesis was found to provide favorable luminescence properties and stability [10].

Despite these advances, the excitation-dependent luminescence processes of $LaF_3:Ce^{3+}$ nanoparticles remain insufficiently understood. Previous studies have mainly focused on synthesis optimization and emission efficiency, whereas the influence of excitation pathways on energy-transfer and relaxation processes has rarely been discussed. Since direct Ce^{3+} excitation, host-lattice excitation, and X-ray excitation are expected to produce different carrier relaxation routes, a detailed investigation is necessary for a fundamental understanding of $LaF_3:Ce^{3+}$ luminescence. Similar excitation-dependent luminescence behavior has also been reported in other Ce^{3+} -activated phosphors [9,11].

Therefore, the purpose of this study is to clarify the excitation-dependent luminescence behavior of solvothermally synthesized $LaF_3:Ce^{3+}$ nanophosphors. Particular attention is devoted to the comparison of direct Ce^{3+} excitation, host-lattice excitation, and X-ray excitation. Furthermore, temperature-dependent photoluminescence measurements are performed to elucidate the origin of the characteristic excitation features observed in the excitation spectra. Based on these results, the possible excitation and relaxation processes of $LaF_3:Ce^{3+}$ nanophosphors are discussed.

2. Experimental

2.1. Sample preparation

$LaF_3:Ce^{3+}$ nanophosphors were synthesized by a solvothermal method. Lanthanum chloride ($LaCl_3$), cerium chloride ($CeCl_3$), and ammonium fluoride (NH_4F) were used as precursor materials. Ethanol was employed as the reaction medium based on the optimized synthesis conditions reported in our previous study. The Ce^{3+} concentration and PVP content were fixed at 5 mol% and 0.75 g, respectively, according to the optimized synthesis conditions reported in our previous work, which provided the highest luminescence intensity and favorable colloidal stability. A mixed solution containing $LaCl_3$ and $CeCl_3$ was prepared in ethanol and stirred for 15 min. PVP was subsequently added and the solution was stirred for an additional 10 min. An aqueous NH_4F solution was then introduced under continuous stirring. The resulting suspension was transferred into a Teflon-lined stainless-steel autoclave and maintained at 200 °C for 4 h. After natural cooling to room temperature, the precipitate was collected by centrifugation, washed several times with ethanol and deionized water, and finally dried at room temperature.

The synthesis conditions were identical to those optimized in our previous work in order to focus on the excitation-dependent luminescence behavior of the obtained nanoparticles.

2.2. Structural characterization

The crystal structure of the synthesized samples was investigated by powder X-ray diffraction (XRD) using $CuK\alpha$ radiation ($\lambda = 0.15405$ nm) with a Rigaku RINT-2200 diffractometer. The crystallite size was

estimated from the full width at half maximum of the (111) diffraction peak using the Scherrer equation.

2.3. Morphological observation

The morphology of the synthesized nanoparticles was examined by scanning transmission electron microscopy (STEM) using a JEOL JEM-2100F microscope.

The particle size and morphology were evaluated to confirm that the obtained phosphors were suitable for biomedical applications requiring nanoscale dimensions.

2.4. Photoluminescence measurements

Photoluminescence (PL) and photoluminescence excitation (PLE) measurements were carried out at the Ultraviolet Synchrotron Orbital Radiation Facility (UVSOR-III), Institute for Molecular Science, Japan.

Emission and excitation spectra were measured using Beamline BL3B. Measurements were performed at both 8 K and 300 K to investigate the temperature dependence of the excitation and luminescence processes. Low-temperature measurements were conducted using liquid helium cooling.

For excitation-dependent studies, the samples were excited at 255 nm, corresponding to direct excitation of Ce^{3+} ions, and at 70 nm, corresponding to host-lattice excitation of LaF_3 . The resulting emission spectra were compared to clarify differences in energy-transfer processes.

2.5. Radioluminescence measurements

Radioluminescence (RL), i.e., luminescence emitted during X-ray irradiation, was measured using a Hitachi Power Solutions HSY-1015 system. A tungsten X-ray source was employed with an operating power of 160 kV and 3.0 mA.

The luminescence spectra obtained under X-ray irradiation were compared with those obtained under ultraviolet and vacuum-ultraviolet excitation. Particular attention was devoted to the relative intensities of the emission bands associated with transitions from the excited 5d state of Ce^{3+} to the split ground states ($^2F_{5/2}$ and $^2F_{7/2}$).

2.6. Investigation of excitation-dependent luminescence behavior

To clarify the luminescence mechanism of $LaF_3:Ce^{3+}$ nanophosphors, excitation spectra and emission spectra obtained under direct Ce^{3+} excitation, host-lattice excitation, and X-ray excitation were systematically compared. Furthermore, temperature-dependent measurements were performed to determine whether the discrete features observed in the excitation spectra originated from thermal effects or from intrinsic electronic processes.

The excitation and relaxation pathways were subsequently discussed based on the observed spectral characteristics.

The relative contribution of the two emission bands was evaluated using the integrated band area ratio ($A^{2F_{7/2}}/A^{2F_{5/2}}$) obtained from spectral deconvolution.

3. Results and discussion

3.1. Crystal structure and crystallinity

Fig. 1 shows the XRD pattern of the synthesized $LaF_3:Ce^{3+}$ nanophosphor. All diffraction peaks were assigned to the hexagonal LaF_3 phase, and no impurity phases were detected [10].

Broad diffraction peaks were observed, confirming the nanocrystalline nature of the sample. The crystallite size estimated from the (111) diffraction was approximately 30 nm, which is comparable to previously reported $LaF_3:Ce^{3+}$ nanophosphors prepared by solvothermal synthesis

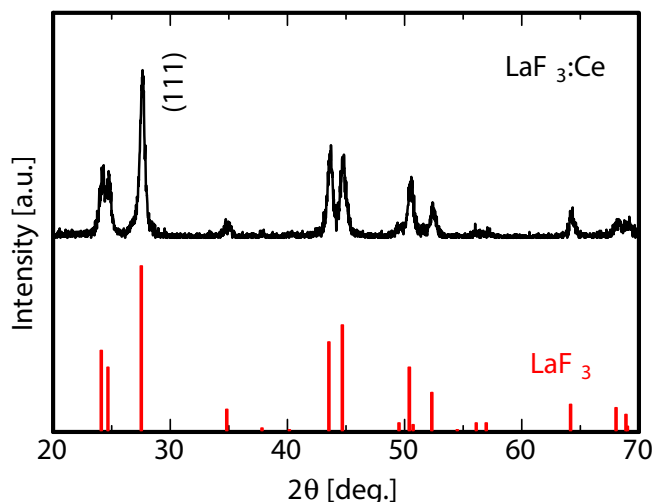


Fig. 1. XRD pattern of the $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphor prepared by the solvothermal method.

[3,10].

These results indicate that the sample possesses the expected crystal structure and crystallinity. Consequently, the excitation-dependent luminescence behavior discussed below can be analyzed without considering structural changes in the host lattice.

3.2. Temperature dependence of excitation spectra

Fig. 2 shows the photoluminescence emission and excitation spectra of $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors measured at 8 K and 300 K. Several discrete

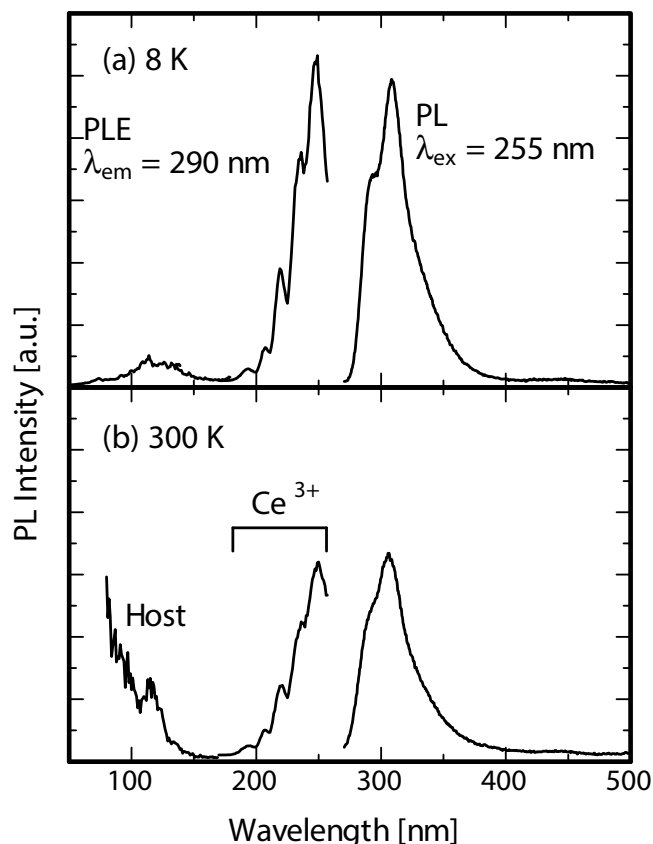


Fig. 2. Photoluminescence and excitation spectra of $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors measured at (a) 8 K and (b) 300 K.

features were observed in the wavelength region between approximately 200 and 250 nm.

In general, Ce^{3+} -activated phosphors exhibit broad excitation bands originating from allowed 4f–5d transitions. Such behavior has been reported for Ce^{3+} -activated phosphors including $\text{YAG}:\text{Ce}^{3+}$ and $\text{SrS}:\text{Ce}^{3+}$ [9,11].

To investigate whether the observed structures are associated with temperature effects, excitation spectra were measured at both low temperature and room temperature. As shown in Fig. 2, the spectral profiles remained essentially unchanged. The discrete structures observed at 8 K were also present at 300 K.

These observations indicate that the excitation features are not primarily related to thermal broadening or temperature-dependent changes in the Ce^{3+} electronic levels. Since the energies of the 5d states of Ce^{3+} are strongly influenced by the local crystal environment, the observed structures may be associated with intrinsic electronic states of the $\text{LaF}_3:\text{Ce}^{3+}$ system. Possible origins include crystal-field-related states arising from the splitting of the Ce^{3+} 5d levels as well as surface-related Ce^{3+} centers, which are known to affect the optical properties of $\text{LaF}_3:\text{Ce}^{3+}$ nanoparticles [4,7,8]. However, the present results are insufficient to determine their exact origin. Additional spectroscopic investigations will be necessary to clarify whether these features are related to crystal-field splitting, surface Ce^{3+} sites, or other electronic processes [4,7,8].

In addition to the persistence of the discrete excitation features, a change in the relative intensity of the host-related excitation band was observed between 8 K and 300 K. In particular, the host excitation component became more pronounced at room temperature. This behavior may indicate a temperature dependence of the energy transfer process between the LaF_3 host lattice and Ce^{3+} luminescent centers. However, no significant temperature dependence was observed for the positions of the discrete excitation structures themselves, suggesting that thermal activation effects are unlikely to be the dominant factor governing their appearance. The present experimental results do not allow a definitive conclusion regarding the origin of this effect. Further spectroscopic studies will be required to clarify the relationship between temperature and host-mediated excitation processes.

Additional measurements were performed by monitoring the 305 nm emission band. The resulting excitation spectrum was essentially identical to that obtained for the 290 nm emission band within experimental uncertainty. In particular, no significant difference was observed in the position of the major excitation bands. These results suggest that the excitation-dependent changes in the emission spectra are unlikely to originate from differences in the excitation spectra of the individual emission bands.

3.3. Comparison of direct Ce^{3+} excitation and host-lattice excitation

Fig. 3 compares the emission spectra obtained under excitation at 255 nm and 70 nm. Two main emission bands were observed at approximately 290 and 305 nm. These bands are attributed to the allowed 5d–4f transitions of Ce^{3+} ions terminating at the spin–orbit split $^2\text{F}_{5/2}$ and $^2\text{F}_{7/2}$ ground states, respectively [6–8]. For ease of comparison, the emission spectra shown in Fig. 3 were normalized to their respective maximum intensities. Therefore, the discussion below focuses on differences in spectral shape and relative emission-band contributions rather than absolute luminescence intensity.

Under excitation at 255 nm, the emission band near 305 nm was relatively stronger than the band near 290 nm. In contrast, excitation at 70 nm resulted in an increase in the relative intensity of the 290 nm emission band.

The excitation wavelength of 255 nm lies within the Ce^{3+} absorption region and therefore mainly corresponds to direct excitation of Ce^{3+} ions [4,6]. On the other hand, excitation at 70 nm is located near the host excitation region of LaF_3 and is expected to involve energy transfer from the host lattice to Ce^{3+} luminescent centers [4,10].

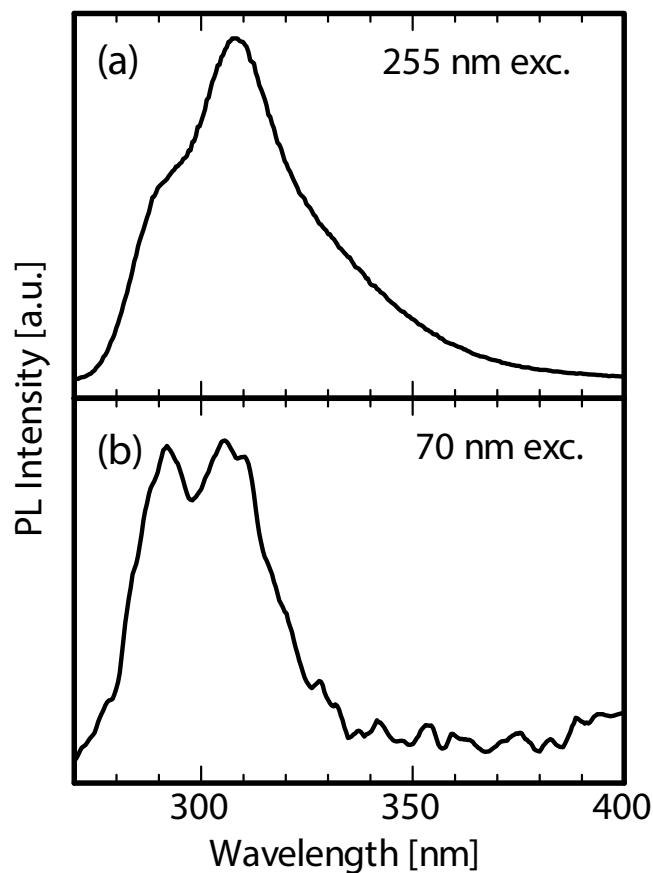


Fig. 3. Comparison of the emission spectra of $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors obtained under excitation at (a) 255 nm and (b) 70 nm.

The emission bands observed under 70 nm excitation appeared in the same spectral region as those obtained under direct Ce^{3+} excitation and X-ray excitation. In addition, the overall spectral shape was essentially preserved. Therefore, the observed luminescence is attributed primarily to Ce^{3+} luminescent centers rather than to defect-related emission.

The difference in the relative intensities of the two emission bands suggests that the relaxation process following host excitation differs from that following direct excitation of Ce^{3+} ions. However, the present experimental results do not allow a definitive identification of the microscopic mechanism responsible for the change in the emission-band intensity ratio. Further studies, including time-resolved spectroscopy, will be necessary to clarify the excitation-dependent relaxation processes.

Similar excitation-dependent luminescence behavior has been reported for various Ce^{3+} -activated phosphors, where the excitation pathway influences carrier relaxation and energy transfer processes [9, 11].

Although the present data clearly demonstrate excitation-dependent spectral variations, the detailed mechanism responsible for the redistribution of emission intensities remains unclear. Therefore, the observed phenomenon should be regarded as evidence that different excitation pathways produce different relaxation processes in $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors.

3.4. Radioluminescence

Fig. 4(a) compares the normalized emission spectra obtained under 255 nm, 70 nm, and X-ray excitation. The radioluminescence (RL) spectrum exhibited emission bands in the same wavelength region as those observed under ultraviolet excitation, confirming that the luminescence originates from Ce^{3+} ions [6,10]. The RL spectrum exhibited a

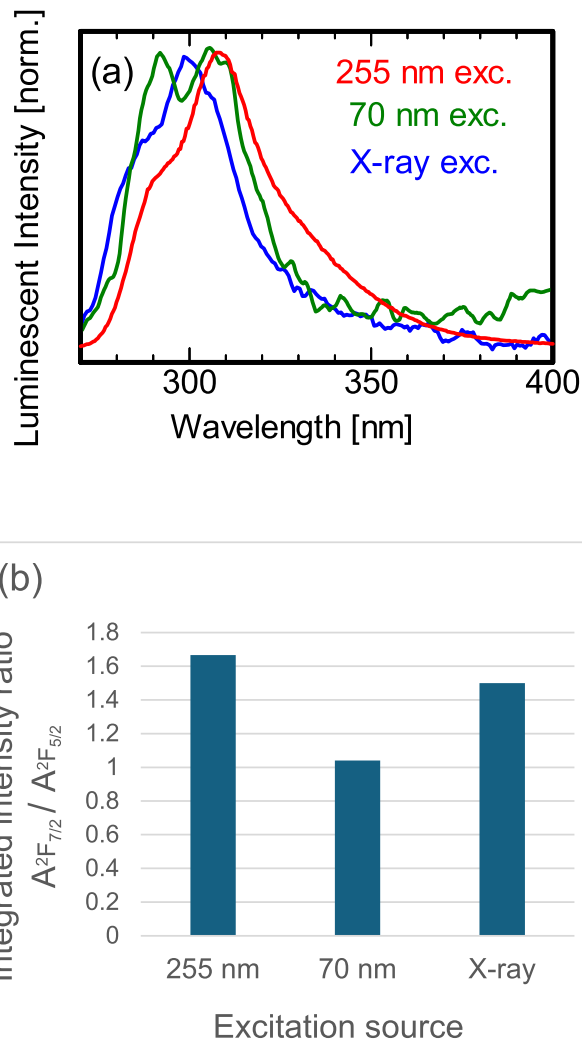


Fig. 4. (a) Normalized emission spectra of $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors under 255 nm, 70 nm, and X-ray excitation. (b) Relative integrated intensity ratio ($A^{2F_{7/2}}/A^{2F_{5/2}}$) of the emission bands associated with the $^2F_{5/2}$ (around 290 nm) and $^2F_{7/2}$ (around 305 nm) transitions under different excitation conditions.

spectral profile more similar to that obtained under direct Ce^{3+} excitation than to that observed under host-lattice excitation. Although the overall spectral profiles are similar, a small difference was observed in the long-wavelength tail. However, the relative contributions of the two emission bands remained different from those observed under host excitation at 70 nm.

To quantitatively compare the excitation-dependent changes in the emission spectra, the emission bands were deconvoluted and the integrated band-area ratio ($A^{2F_{7/2}}/A^{2F_{5/2}}$) was calculated. Fig. 4(b) shows the integrated intensity ratio ($A^{2F_{7/2}}/A^{2F_{5/2}}$) of the 305 nm and 290 nm emission bands under 255 nm, 70 nm, and X-ray excitation. Interestingly, the RL spectrum was more similar to the spectrum obtained under 255 nm excitation than to that obtained under 70 nm excitation. In particular, the emission band at approximately 305 nm remained dominant. The integrated intensity ratio ($A^{2F_{7/2}}/A^{2F_{5/2}}$) was approximately 1.5 under both 255 nm and X-ray excitation, whereas a value close to unity was obtained under 70 nm excitation.

This result suggests that the excitation processes involved under X-ray irradiation differ from those associated with ordinary host-lattice excitation. One possible explanation is that secondary electrons generated through X-ray absorption contribute significantly to the excitation

of Ce^{3+} luminescent centers. Another possibility is that multiple excitation channels coexist under X-ray irradiation, including both host-mediated and direct excitation processes.

At present, the available experimental results do not allow a definitive identification of the dominant excitation pathway. Nevertheless, the similarity between the UV-excited and X-ray-excited spectra indicates that excitation routes different from conventional host excitation likely contribute to the RL process.

A slight shift of the apparent emission maximum was also observed depending on the excitation source. This behavior may partly originate from changes in the relative contributions of the emission bands associated with the $^2\text{F}_{5/2}$ and $^2\text{F}_{7/2}$ transitions, as demonstrated by the excitation-dependent integrated intensity ratio. In addition, contributions from Ce^{3+} centers located in different local environments cannot be excluded. Further spectroscopic investigations will be necessary to clarify the origin of this wavelength shift.

3.5. Discussion of excitation-dependent luminescence processes

Based on the experimental observations, a possible schematic model of the excitation-dependent luminescence process is proposed.

Fig. 5 shows a proposed schematic illustration of the excitation and relaxation processes in $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors.

When excited at 255 nm, in Fig. 5(a), photons are predominantly absorbed by Ce^{3+} ions through the 4f–5d transition. The excited electrons subsequently relax radiatively to the split 4f ground states, producing the characteristic ultraviolet emission bands observed in the present study [6–8].

In contrast, excitation at 70 nm in Fig. 5(b) primarily excites the LaF_3 host lattice because the excitation energy is close to the host band-gap region [4,10]. Energy must therefore be transferred from the host lattice to the Ce^{3+} ions before radiative relaxation occurs. The excitation-dependent variation of the integrated band-area ratio further suggests that this host-mediated process differs from direct Ce^{3+} excitation. Under X-ray irradiation in Fig. 5(c), ionization processes and the generation of energetic secondary electrons are expected to occur. The similarity between the RL spectrum and the spectrum obtained under direct UV excitation suggests that these secondary processes may play an important role in the excitation of Ce^{3+} ions. However, further

experiments, such as time-resolved spectroscopy, will be necessary to clarify the detailed excitation mechanism.

Overall, the present results demonstrate that the luminescence characteristics of $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors are strongly dependent on the excitation pathway. In particular, direct excitation, host-lattice excitation, and X-ray excitation generate different emission characteristics, indicating the presence of distinct relaxation processes. A better understanding of these excitation-dependent phenomena will be important for the optimization of Ce^{3+} -activated fluoride nanophosphors for scintillation and X-ray-induced photodynamic therapy applications. Nevertheless, the detailed origin of the discrete excitation structures and the microscopic mechanism responsible for the emission-band redistribution remain subjects for future investigation.

4. Conclusion

The excitation-dependent luminescence behavior of solvothermally synthesized $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors was investigated using ultraviolet, vacuum-ultraviolet, and X-ray excitation.

Distinct excitation features were observed in the excitation spectra between 200 and 250 nm. Temperature-dependent measurements performed at 8 K and 300 K demonstrated that these structures were preserved over the entire temperature range, indicating that they are not primarily associated with thermal broadening or low-temperature effects.

The luminescence spectra exhibited characteristic Ce^{3+} emission bands originating from the 5d–4f transitions. Significant changes in the relative integrated band-area ratio ($A^2\text{F}_{7/2}/A^2\text{F}_{5/2}$) were observed depending on the excitation source. The radioluminescence spectrum showed a spectral profile similar to that obtained under direct Ce^{3+} excitation, whereas host-lattice excitation resulted in a distinctly different emission-band ratio.

These observations suggest that different excitation pathways produce different relaxation and energy-transfer processes in $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors. Although the detailed origin of the discrete excitation structures and the microscopic mechanism responsible for the excitation-dependent emission behavior remain to be clarified, the present findings provide useful insight into excitation-dependent luminescence processes in Ce^{3+} -activated fluoride nanophosphors.

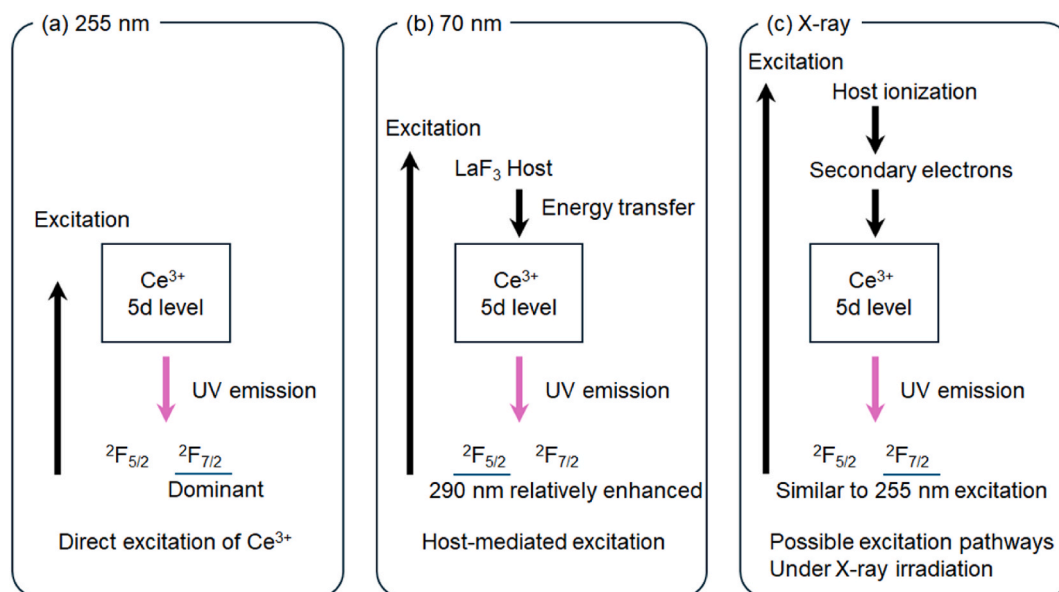


Fig. 5. Proposed schematic illustration of excitation and relaxation processes in $\text{LaF}_3:\text{Ce}^{3+}$ nanophosphors. (a) Direct UV excitation (255 nm) mainly excites Ce^{3+} ions through the 4f–5d transition, whereas (b) VUV excitation (70 nm) predominantly excites the LaF_3 host lattice followed by energy transfer to Ce^{3+} ions. (c) Under X-ray irradiation, secondary electronic processes are suggested to contribute to the excitation of Ce^{3+} luminescent centers. The different excitation pathways are considered to be responsible for the observed changes in the integrated intensity ratio.

CRediT authorship contribution statement

Hiroko Kominami: Writing – review & editing, Writing – original draft, Validation, Supervision, Project administration, Investigation, Funding acquisition, Conceptualization. **Ryoya Ishihara:** Investigation. **Tomoki Sadamori:** Investigation. **Kazuhiko Hara:** Supervision. **Katsuyuki Takagi:** Investigation. **Toru Aoki:** Supervision.

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Data availability

Data will be made available on request.

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