

PHOTOLUMINESCENCE AND SCINTILLATION PROPERTIES OF CE-DOPED $\text{Lu}_{0.5}\text{Gd}_{0.5}\text{AlO}_3$ SINGLE CRYSTALS GROWN BY FLOATING ZONE METHOD

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Abstract

Undoped and Ce-doped $\text{Lu}_{0.5}\text{Gd}_{0.5}\text{AlO}_3$ (LuGdAP:Ce) single crystals were grown by the floating zone method. The coloration of the samples faded by annealing under a reducing atmosphere, and the characterizations of the samples after annealing were evaluated. The characterizations included X-ray diffraction, photoluminescence (PL), and scintillation properties. In PL properties, the 0.3% Ce-doped sample exhibited a broad emission at 350–500 nm under 290 nm excitation, and the emission would be ascribed to 5d-4f transitions of Ce^{3+} ions. The undoped sample showed emission at 650–750 nm under excitation at 320 nm, and the emission would be due to charged oxygen vacancies. In scintillation properties, all the Ce-doped samples showed a broad emission at 360–500 nm, which came from 5d-4f transitions of Ce^{3+} ions. According to pulse height spectra under γ -ray irradiation from ^{137}Cs , the scintillation light yield (*LY*) of the 0.3% Ce-doped was 1,610 ph/MeV, and the energy resolution was 54.4%. The 0.3% Ce-doped samples showed the lowest afterglow level at 20 ms after X-ray irradiation for 2 ms among all the Ce-doped samples. Therefore, the optimal Ce-concentration was 0.3% in terms of the *LY* and afterglow.

Keywords: Scintillator; Ce^{3+} ; Perovskite; Floating-zone; Radiation detection

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Introduction

Scintillators were luminescent materials that converted high-energy ionizing radiation into lower-energy photons (Yanagida, 2018). For this function, scintillators were used in many areas, for example, γ -ray (gamma-ray) detection (Fantidis *et al.*, 2025), radiography for medical purposes (Welsh, 2007), and international security (Ellis *et al.*, 2016). The luminescence property came from the interaction between the scintillators and radiation. Within the material, numerous electron-hole pairs were generated, transported, and then, when electrons recombined with holes, the energy was released in the form of photons, which had lower energy than the ionizing radiation. Therefore, the light was released over a wide wavelength range (UV-IR), depending on the energy of the emitted photons. This mechanism was called the scintillation mechanism and could happen instantly. However, the properties of scintillators could have been changed depending on the use case. For example, to detect γ -rays, the effective atomic number (Z_{eff}) needed to have a high value. And the emission range needed to fit within the range of common photodetectors (generally 300-600 nm). Ce-doped LuAlO₃ (LuAP:Ce) single crystal became a topical interest in the mid-1990s due to its potential to become low in density and Z_{eff} but have high light yield (LY) and fast decay time (Lempicki *et al.*, 1995; Lempicki & Glodo, 1998). Unfortunately, LuAP did not produce results that matched expectations, and extremely unstable growth was another problem that had to be mentioned. Ce-doped GdAlO₃ (GdAP:Ce), on the other hand, even though it had more stability than LuAP, suffered from reverse energy transfer mechanisms of Ce³⁺ (5d¹) to Gd³⁺ (6P_{3/2}, 5/2, 7/2) (Dieke *et al.*, 1961), resulting in poor scintillation performance. However, research on multi-component perovskite materials such as (Gd, Y)AP:Ce in the form of thin-film growth with the liquid phase epitaxy (LPE) method (Riva *et al.*, 2016) showed that Gd³⁺ could improve performance in terms of decay time by transferring energy from Ce³⁺ to nearby Gd³⁺ cation sites (Rathaiah *et al.*, 2019), resulting in decreased primary component (Ce³⁺) decay time, along with a slow component decay time from Gd³⁺, generally around 60-250 ns. In addition, one research reported Lu_xGd_{1-x}AP:Ce to be a breakthrough single-crystal multi-component perovskite, by varying the Gd-to-Lu ratio and growing with the Czochralski (CZ) method. The research showed high LY approaching 21,000 ph/MeV, decreased afterglow to millisecond time scale, and high Z_{eff} (from calculation) close to

LuAP:Ce ($Z_{\text{eff}} = 64.9$), which was suitable for bulk scintillators that required heavy, fast, and high LY (Pokorný *et al.*, 2021).

In this paper, we provided the results of measurements on LuGdAP:Ce single crystals with various concentrations of Ce from 0 to 0.5%, grown by the floating zone (FZ) method. The results were mainly divided into three groups: Physical properties (X-Ray Diffraction; XRD), PL properties (PL maps of emission and excitation, PL decay time), and Scintillation properties (spectra, decay time, LY , energy resolution, and Afterglow after irradiation). These measurement results were used to define the optimum point of Ce concentration in LuGdAP:Ce single crystals, which was the main objective of this study.

Materials and Methods

LuGdAP:Ce single crystals were grown by the FZ method. The used raw powders were Lu₂O₃ (Nippon Yttrium, 99.99%), Gd₂O₃ (Furuuchi Chemical, 99.99%), Al₂O₃ (Teimei Chemicals, 99.99%), and CeO₂ (Furuuchi Chemical, 99.99%). The amount of material was prepared in the stoichiometric ratio, with the goal for each sample rod being 5 grams. The samples had different amounts of Ce dopant percentages, for a total of 5 samples, including undoped, 0.05, 0.1, 0.3, and 0.5%. All the materials were mixed by using an agate mortar. Then, each of the mixed powders was formed into a cylindrical rod shape around 5 mm in diameter using hydrostatic pressure. After that, all the rods were sintered in an electric furnace on alumina crucibles, with leftover powder of the same composition acting as a barrier between the rod and the crucible. All the rods were sintered at 1400°C under an air atmosphere, with the heat profile of the sintering process being heated up until reaching 1400°C within 7 hours (heat rate: 200°C per hour), holding the temperature for 8 hours, then cooling down for 9 hours and 20 minutes. After that, the sintered rods were placed into the FZ furnace (Canon Machinery Inc., FZD0192) to grow the crystal, using a rotation speed of about 10 rpm with a pulling down rate of 5 mm/h. In addition, the growth condition was performed at atmospheric pressure with an air atmosphere. The grown crystals are shown in Figure 1. Next, the grown crystals were crushed by a piler to obtain large pieces of sample before being polished down to a flat surface of about 1.00 ± 0.05 mm using a polisher (Buehler, MetaServ 250). The obtained samples were

annealed under vacuum using carbon powder at 800°C for 72 hours.

To confirm that there is no impurity within grown crystals, powder X-ray diffraction (XRD) patterns were obtained by using diffractometers (Rigaku, Miniflex600). PL properties such as PL emission, excitation spectra, and quantum yield (QY) will be measured with Quantaaurus-QY (Hamamatsu, C11347) using an excitation source from 250-400 nm. In addition, PL decay curves were measured by Quantaaurus- τ (Hamamatsu, C11367) using a 280 nm LED as an excitation source in the Ce-doped sample. In the undoped sample, which shows long-range emission, we used an Xe lamp with a 315 nm band-pass filter (BPF315) for the excitation wavelength. On the detection side, a 650 nm short-cut filter (SCF650) was applied to block wavelengths below 650 nm before being monitored by the detector.

Next was scintillation properties, for the scintillator spectrum that was obtained by using an X-ray tube (Spellman, XRB80P&N200X4550) operating at 60 kV and 1.2 mA as the X-ray source. The samples were connected to an optical fiber, covered with Polytetrafluoroethylene (Teflon) to reflect scintillation photons back to the optical fiber, and wrapped with a black Polyvinyl sheet to ensure the sample was in a blackout environment. The samples were then exposed and emitted photons due to X-rays. Photons were transferred through the optical fiber to a monochromator (Andor Technology, SR-163) and CCD spectrometer (Andor Technology, DU-420-BU2), where the CCD was actively cooled by a Peltier module to reduce thermal noise during measurement (Yanagida *et al.*, 2013). For scintillator decay time, an X-ray pulse was generated by using pulses of photons from an LED or laser diode controlled by a digital delay generator (Stanford Research System, DG645) and a light pulser (Hamamatsu, C10196). The photons were converted to electrons when they hit a multi-alkali photocathode within the X-ray tube (Hamamatsu, N5084), and produced a pulse of X-rays under a bias voltage of 30 kV. The sample was then excited by the X-ray, and scintillator photons were detected using the Time-Correlated Single-Photon Counting (TCSPC) technique with a PMT (Hamamatsu, R7400P-06), which was supplied with 950 V by the supplier (Hamamatsu, C9525) and recorded (Hamamatsu, C5594) into a PC at a time range of 1 μ s per cycle (Yanagida *et al.*, 2014). Lastly, LY and energy resolution of the scintillator were measured via pulse-height analysis. The sample was attached to the PMT using optical grease (Oken, BC-630) and covered with two layers of Teflon tape, and scintillation photon detected by the PMT

(Hamamatsu, R7600-U200) with 600 V bias voltage by the supplier (ORTEC, 556), and an irradiated γ -ray source by ^{137}Cs with 662 keV on top of the sample holder. The signal from the PMT went into a pre-amplifier (ORTEC, 113) and was shaped by a shaping amplifier (ORTEC, 570). The settings needed to be adjusted as shown in Table 1. The signal then went into the Multi-Channel Analyzer (MCA, Amptek, Pocket MCA 8000A) to convert the data from analog to digital and was recorded on a PC (Yanagida *et al.*, 2013). However, due to Gd's long decay time and hardware limits, LY of the undoped sample could not be measured.

The LY and energy resolution were determined from Gaussian fitting of the pulse-height spectra. The LY of each sample was estimated by comparison with 0.5% Ce-doped Gd_2SiO_5 (GSO:Ce) reference sample, which has a known LY of 8,000 ph/MeV measured using a silicon photodiode (Si-PD). The sample LY was calculated as described by (1)

$$LY_s = LY_r \times \frac{PC_s}{PC_r} \times \frac{Gain_r}{Gain_s} \times \frac{QE_r}{QE_s} \quad (1)$$

where LY_r is the light yield of the reference scintillator (8,000 ph/MeV), $PC_{s,r}$ are the full-energy peak channels of the sample and reference, respectively, and $QE_{s,r}$ are the PMT quantum efficiencies at the corresponding emission wavelengths of the sample and reference.

Table 1. Setting used in shaping amplifier for different Ce-doped concentration of LuGdAP:Ce, and GSO:Ce reference sample to obtain optimal LY

Sample name	Gain	Shaping time	Bias voltage
0.05% Ce	500	2 μ s	600 V
0.1% Ce	500		
0.3% Ce	200		
0.5% Ce	500		
0.5% GSO:Ce	100		

Afterglow was measured using a setup similar to the X-ray induced scintillation decay time, with a slight change in the LED pulse duration to 2 ms. The measurements were recorded (Hamamatsu, M9003-01) with a time range of 52.2 ms (Yanagida *et al.*, 2014). The result of the afterglow can be described by (2)

$$\frac{I_2 - I_0}{I_1 - I_0} \times 10^6 \quad (2)$$

where I_0 is the average intensity of the background level, I_1 is the average intensity of the

X-ray pulse, and I_2 is the average intensity of the afterglow (Fukushima *et al.*, 2019).

Results and Discussion

Physical properties

A photograph of the LuGdAP:Ce grown crystals in Figure 1 showed a significant change in color with different amounts of Ce dopant. The crystal was then crushed and polished down to samples with a thickness of around 0.97-1.05 mm, subjected to different annealing times, temperatures, and various light environments, as shown in Figure 2. The photograph clearly indicated that annealing the samples at 800°C tended to improve their transparency and reduce their brownness, indicating the excess amount of O₂ within the crystal (Brenes *et al.*, 2018).

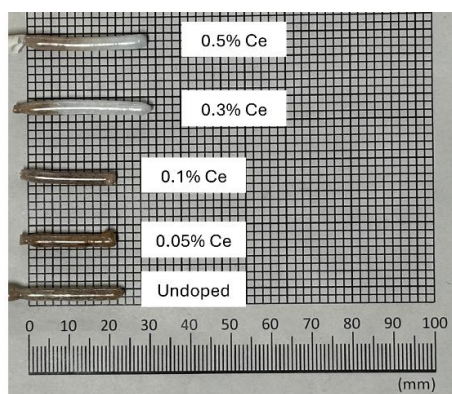


Figure 1. Photograph of LuGdAP:Ce crystals grown by the FZ furnace

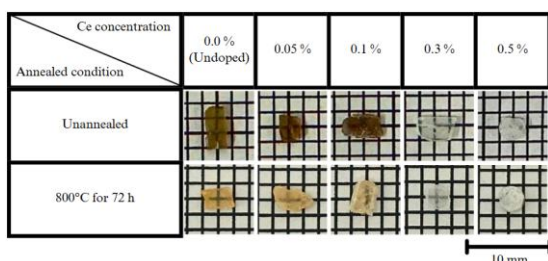


Figure 2. Photograph of Lagace samples of unannealed and annealed at 800°C for 72 hours

Figure 3 displays the powder XRD patterns of LuGdAP:Ce with Ce concentrations ranging from 0 to 0.5%. All the samples exhibited similar patterns

to reference one, and the peaks were consistent with the reference pattern of perovskite GdAlO₃ (Crystallography Open Database, COD 2014561). This indicates that the samples did not contain any impurity phases.

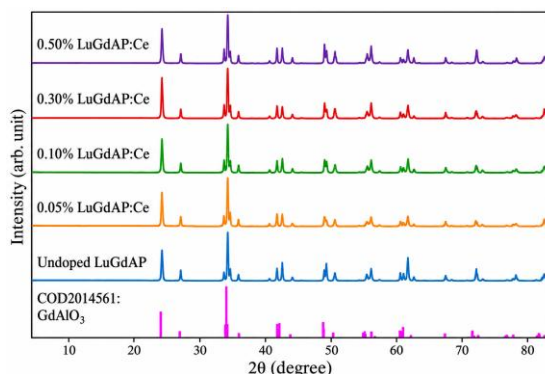


Figure 3. The XRD patterns of LuGdAP:Ce powder are shown, with a COD 2014561 reference

Photoluminescence properties

PL emission maps of undoped and Ce-doped LuGdAP are shown in Figure 4. The undoped samples showed the emission e at 650-750 nm when excited under 320 nm, which possibly came from aggregates of charged oxygen vacancies emission (Zorenko *et al.*, 2018). Moreover, the 0.3% Ce-doped sample exhibited emission wavelength at 350-500 nm when excited by 290 nm, and the QY was about 1%.

Additionally, the PL decay curves of the undoped and Ce-doped samples are shown in Figure 5. The monitoring and excitation were 400 nm and 280 nm in the Ce-doped samples. The PL decay curves were approximated by a sum of two exponential functions, and these decay time constants were typical values of the 5d-4f transition Ce³⁺ (Dujardin *et al.*, 1997; Takagi & Fukazawa, 1983). In addition, the fast decay times decreased with the increase of Ce concentration, which may have been due to the effect of Ce concentration quenching (Setlur *et al.*, 2013). In the undoped sample, the monitoring and excitation wavelengths were 315 nm and 700 nm, and the PL decay curve consisted of an exponential function except for instrumental response functions (IRF). The PL decay time was estimated to be 2.06 ms.

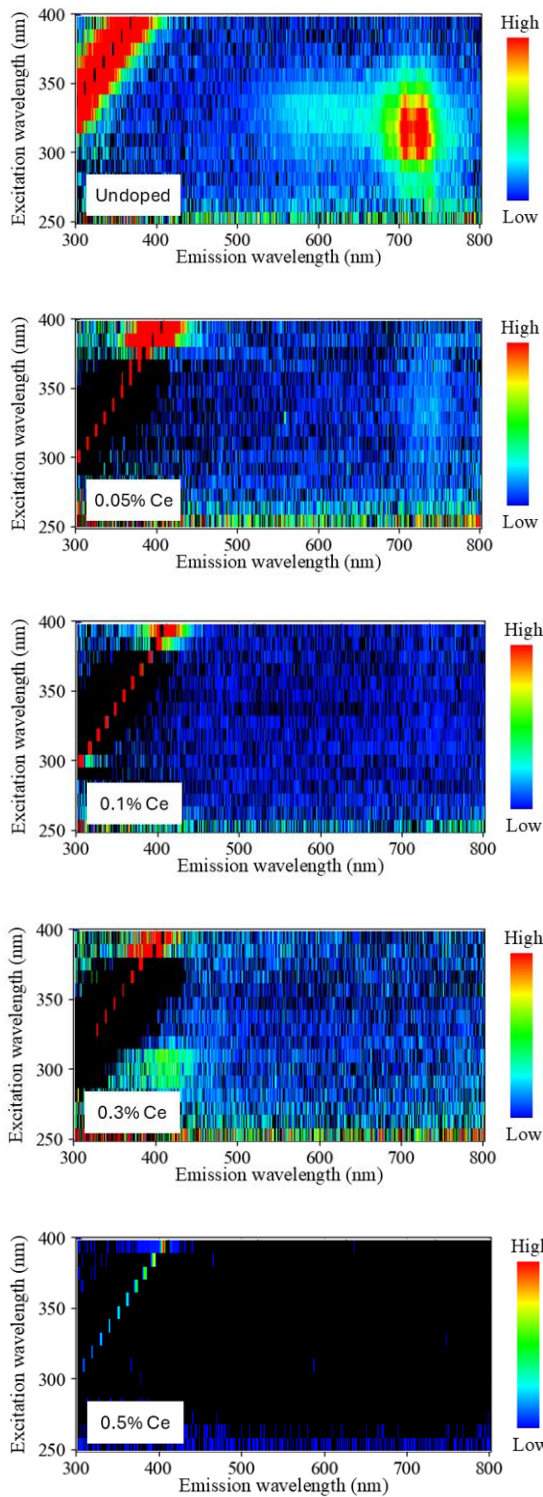


Figure 4. PL emission maps of annealed LuGdAP:Ce. The horizontal axis represents the emission wavelength, and the vertical axis represents the excitation wavelength

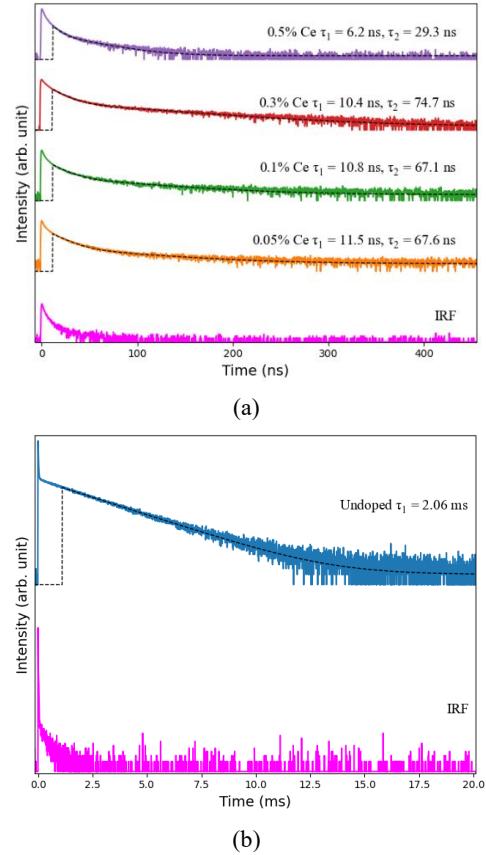


Figure 5. PL decay time profiles of annealed LuGdAP:Ce (A) and the undoped LuGdAP (B)

Scintillation properties

Figure 6 represents the X-ray-induced scintillation spectra of LuGdAP:Ce single crystal samples. The undoped samples showed emission at 315 nm originating from 4f-4f transitions of Gd^{3+} (Peijzel *et al.*, 2004) and at 590 nm from aggregates of charged oxygen vacancies (Zorenko *et al.*, 2018). On the other hand, the Ce-doped samples did not show Gd emission, and the emission from vacancies tended to decrease as the Ce concentration increased. However, all Ce-doped samples showed emissions in the 360-500 nm range, which originated from the 5d-4f transition of Ce^{3+} . Additionally, the intensity of the spectra cannot indicate which samples are the best in terms of scintillation performance, as intensity can be affected by various factors such as thickness, self-absorption, and sample size. The comparison of the intensity between each sample was observed in the upcoming discussion in the pulse height measurement results.

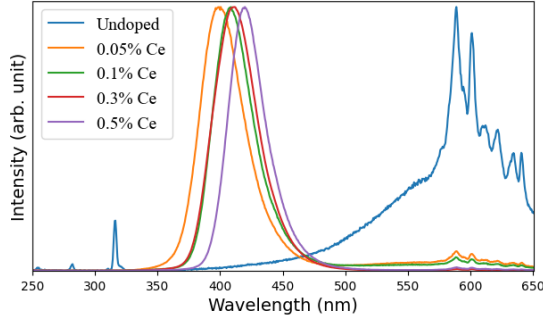


Figure 6. X-ray induced scintillation spectra of LuGdAP doped with different concentrations of Ce samples annealed at 800°C for 72 hours

Figure 7 indicates the decay time of the samples when induced by scintillation decay time with X-ray, which agrees with the trend observed in the PL decay time. The decay time of the fast component was 17.4, 13.6, 18.4, and 6.76 ns for the 0.05%, 0.1%, 0.3%, and 0.5% Ce-doped LuGdAP samples, respectively. Overall, the X-ray-induced decay time of the 0.5% sample also showed the effect of Ce concentration quenching, like PL decay time. The decay time of X-ray induced had a higher value than PL because scintillation photons are more than photons generated by PL.

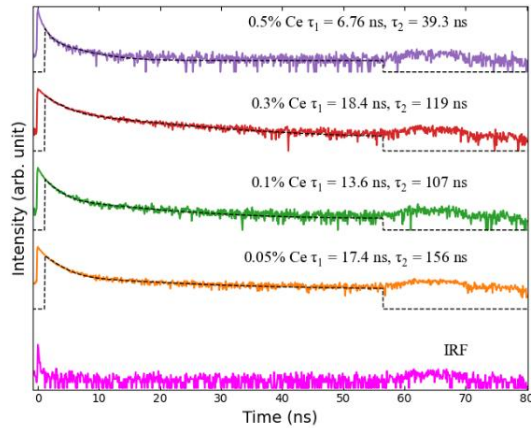


Figure 7. X-ray-induced scintillation decay time profiles of LuGdAP:Ce samples

Pulse height spectra under ^{137}Cs γ -ray irradiation are shown in Figure 8. In only the 0.3% Ce-doped sample, the structure like a photoabsorption peak was observed. The LY was calculated by comparing with the peak channel of reference (GSO:Ce), and the 0.3% Ce-doped sample showed 1,610 ph/MeV and an energy

resolution of 54.4% under 662 keV γ -ray irradiation. The LY was low compared with CZ growth LuGdAP:Ce with 21,000 ph/MeV and an energy resolution of 10% at 662 keV (Pokorný *et al.*, 2021) or the commercial scintillator such as Ce-doped $\text{Gd}_3(\text{Ga},\text{Al})_5\text{O}_{12}$ (GAGG:Ce) with 28000 ph/MeV and an energy resolution of 9.43% at 662keV (Zhu *et al.*, 2020).

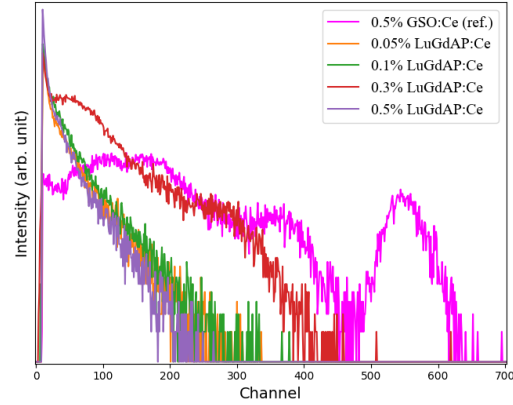


Figure 8. Pulse-height spectra of 0.05 – 0.5% Ce-doped LuGdAP samples and Ce-doped GSO reference under the ^{137}Cs (662keV) γ -ray irradiation

Afterglow curves after X-ray irradiation for 2 ms are shown in Figure 9. The afterglow levels at 20 ms of the 0.05, 0.1, 0.3, 0.5 % Ce-doped were 128, 101, 32, 113 ppm, respectively. According to the previous report (Nakauchi *et al.*, 2020), the afterglow level of CsI:Tl, which was a commercial scintillator for X-ray detection, was ~ 300 ppm, and all the samples showed lower afterglow than that of CsI:Tl. The 0.3% Ce-doped sample had the lowest afterglow level among all the Ce-doped samples. Generally, afterglow was thermoluminescence at room temperature, and defect-driven luminescence capability, such as thermoluminescence and optically stimulated luminescence, was reported to be inversely correlated with scintillation (Yanagida, 2016). In this case, an inverse correlation between scintillation and afterglow was observed. Additionally, the result agreed with the assumption that the 0.3% sample exhibited the lowest afterglow value. Therefore, the presence of trap sites within the samples is one of the factors responsible for the lower LY than expected compared with CZ growth LuGdAP:Ce. According to the relationship $LY \propto S \times QY$, where S is the energy transportation efficiency, and QY is the quantum yield. The result of low afterglow suggests that S was good.

Therefore, the low LY is mainly attributed to poor QY .

To improve LY in the future, the crystal was grown under a reducing atmosphere, and more caution was taken in monitoring the crystal growth, aimed at reducing defects and Ce^{4+} within the structure. This led to improvements in LY and energy resolution (Jia *et al.*, 2020) with shorter decay time.

Conclusions

The undoped and Ce-doped LuGdAP single crystals were prepared by the FZ method. The XRD pattern from powder diffractometry indicates that the material is a single-phase perovskite without impurity phases. In terms of PL properties, the undoped sample showed emission of 650-750 nm with 320 nm excitation, which originates from charged oxygen vacancies. Also, on 0.3% Ce-doped sample showed the emission in the 350-500 nm range, originating from the 5d-4f transition of Ce^{3+} , with a PL QY up to 1% when excited by 290 nm. The PL decay time constant was slightly above 10 ns. The scintillation spectra aligned with the PL spectra, 0.3% Ce-doped sample showed emissions around 360–500 nm, which is consistent with Ce^{3+} emission. The decay time of X-ray-induced scintillation also followed a similar trend to the PL, with decay times in the 10-20 ns range. Under 662 keV γ -ray irradiation, only the 0.3% Ce-doped sample showed an absorption peak, with a LY of 1,610 ph/MeV and an energy resolution of 54.4%. The Scintillation properties of all samples investigated in this work are summarized in Table 2.

Table 2. Scintillation properties of LuGdAP:Ce samples

Sample name	LY (ph/MeV)	Energy resolution	X-ray-induced decay time (ns)
Undoped	-	-	-
0.05% Ce	-	-	17.4
0.1% Ce	-	-	13.6
0.3% Ce	1,610	54.4	18.4
0.5% Ce	-	-	6.76

Despite attempts to reduce the excess amount of oxygen content through annealing, which showed improvements in emission intensity, other properties, such as PL QY (only 1% for the 0.3% Ce concentration) and scintillation properties (like LY) remained low. Additionally, the energy resolution was worse than commercials (9.43% at 662keV for

GAGG:Ce). The low LY was further supported by the afterglow data, which indicated that the 0.3% Ce sample exhibited the lowest afterglow. Therefore, the poor performance may be attributed to the chosen growth technique and conditions, which were conducted in an air atmosphere, leading to oxidation and degradation of the scintillation properties. In conclusion, while the FZ growth method for LuGdAP crystals holds potential, growth crystal under a reducing atmosphere and applying new techniques to obtain better crystal quality may improve its performance for scintillation applications.

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Author Contributions

Conceptualization: K.M., P.K.; Methodology: K.M., K.I., A.F.; Validation: T.K., D.N., N.K.; Formal Analysis: T.T.; Investigation: T.T., K.M., K.I.; Data Curation: T.T.; Writing - Original Draft: T.T.; Writing - Review & Editing: P.K., T.Y.; Supervision: T.Y.; Project Administration: P.K.; Funding Acquisition: P.K., N.K., T.Y. All authors have read and agreed to the published version of the manuscript.

Ethics Statement

Ethical approval was not required for this study because it did not involve human participants, animals, identifiable personal data, or materials requiring institutional ethical review.

Declaration of Generative AI Use

During the preparation of this manuscript, the authors used ChatGPT to assist with language fluency. The tool was used solely for grammar checking, language editing, and improving the clarity and readability of the manuscript. All AI-assisted outputs were critically reviewed, verified,

and revised by the authors, who accept full responsibility for the content of this manuscript.

Conflict of Interest

The authors declare that they have no conflicts of interest related to this work.

Data Availability Statement

The datasets generated and/or analyzed during the current study are available from the corresponding author upon reasonable request.

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