

PREPARATION OF $\text{Lu}_{2.5}\text{Y}_{0.5}(\text{Al}_{5-x}\text{Ga}_x)\text{O}_{12}:\text{Ce}$ TRANSLUCENT CERAMIC FOR X-RAY DETECTION APPLICATIONS USING SPARK PLASMA SINTERING (SPS)

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Abstract

In this research, translucent ceramics of $\text{Lu}_{2.5}\text{Y}_{0.5}(\text{Al}_{5-x}\text{Ga}_x)\text{O}_{12}$ doped with Ce 1 mol% were fabricated using a spark plasma sintering (SPS) furnace. The microstructure of translucent ceramic was revealed by a scanning electron microscopy (SEM) technique which indicated grain boundaries as polycrystalline characteristics. The absorption spectra showed the absorption lines of Ce^{3+} , with an absorption edge around 480 nm, corresponding to the 4f-5d transitions of the Ce^{3+} ions. The photoluminescence (PL) emission band was presented at approximately 507-535 nm, attributed to the Ce^{3+} : 5d₁-4f transition. In addition, PL emission results also showed a slight blue shift with increasing Ga substitutional content in the garnet structure. The radioluminescence (RL) spectra displayed a broad emission band between 450 and 690 nm under X-ray excitation. The PL decay time constants for all samples were on the order of tens of nanoseconds. The increasing Ga content in these ceramic scintillators leads to improvement of luminescence efficiency, luminescence decay time reduction, which is crucial for the development of novel scintillators for X-ray detection applications.

Keywords: Luminescence; Translucent ceramic; X-ray detection

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Introduction

Ceramic scintillating material is a type of material used in radiation detection and imaging applications. Scintillators are substances that emit light (usually in the visible or ultraviolet spectrum) when excited by ionizing radiation such as X-rays, gamma rays, or particles like alpha and beta particles (Blasse and Grabmaier, 1994; Yanagida, 2018). This light can then be detected by photodetectors, such as photomultiplier tubes or photodiodes, for measure the intensity and energy of the incoming radiation. Ceramic scintillators are typically made of inorganic, crystalline materials that have been sintered or formed into a ceramic structure. These materials are chosen for their ability to efficiently convert incoming radiation into light. Moreover, their durability, stability, and ability to be fabricated into various shapes and sizes are also important properties for good scintillators (Lecoq *et al.*, 1994; Yanagida *et al.*, 2023). Ceramic or polycrystalline scintillators are preferred in some applications over single-crystal scintillators due to their potentially lower cost, easier manufacturing processes, and the ability to create larger and more complex shapes (Ronda, 2007; Mori *et al.*, 2016). Generally, the choice of a particular ceramic scintillator depends on factors such as the type of radiation to be detected, the required efficiency, the emission spectrum, and the application's specific requirements.

In this work, we studied a ceramic scintillator fabricated by a spark plasma sintering (SPS) furnace. The $\text{Lu}_{2.5}\text{Y}_{0.5}(\text{Al}_{5-x}\text{Ga}_x)\text{O}_{12}$ (LuYAG) translucent ceramic scintillator is derived from the $\text{Y}_3\text{Al}_5\text{O}_{12}$ (YAG) garnet structure, with lutetium (Lu) and gallium (Ga) incorporated to rise the ceramic host properties. When doped with selected luminescence centers like Ce^{3+} , the YAG garnet is known for its high light yield, fast decay time, and excellent energy resolution, making it highly suitable for various applications, particularly in the medical field (Koshimizu *et al.*, 2018). In the previous study, the addition of the Lu in YAG structure increases the effective atomic number (Z_{eff}), which enhances scintillation efficiency under the X-rays and γ -rays (Yanagida *et al.*, 2011; Wang *et al.*, 2013), while Ga^{3+} improves the energy band structure. Substituting Ga^{3+} for Al^{3+} modifies the energy band, improving luminescence and scintillation properties. Ga^{3+} ions decrease shallow electron traps by adjusting the conduction band and defect levels (Grigorjeva *et al.*, 2018). As Ga content increases, it lowers the conduction band energy, decreasing the energy gap between the conduction band and trap states, thereby

reducing trap depths. This change minimizes the formation of shallow traps (Ueda *et al.*, 2013; Wu and Ren, 2013; Grigorjeva *et al.*, 2018). Additionally, Ga^{3+} ions, especially in the presence of Ce^{3+} , introduce defect levels that interact with the conduction band, destabilizing electron trapping in shallow states. This disturbance of the lattice by Ga^{3+} ions ultimately restrain shallow trap formation (Ueda *et al.*, 2013; Wu and Ren, 2013; Grigorjeva *et al.*, 2018).

In this research, we investigated the effect of varying gallium content on the photoluminescence and scintillation properties of the samples. Our objective was to raise the efficiency of scintillator materials, with a particular emphasis on improving important properties such as radioluminescence and photoluminescence decay time, which are essential characteristics of scintillators.

Experimental Details

Chemical powders of Al_2O_3 , Y_2O_3 , Lu_2O_3 , Ga_2O_3 and CeO_2 with 99.99 % purity were used as starting materials for fabricating a translucent ceramic scintillator. The starting chemical powders were weighed according to the composition of $\text{Lu}_{2.5}\text{Y}_{0.5}(\text{Al}_{5-x}\text{Ga}_x)\text{O}_{12}$ ($x = 1, 2$, and 3) with 1.00 mol% Ce substitution in respect of Lu and Y site, and mixed in an agate mortar for each sample. The translucent ceramic sample 0.5 g of the mixture was placed in a graphite die and held between two graphite punches. The sintering process involved two steps using Sinter Land LabX-100 spark plasma sintering (SPS) furnace under vacuum. First, the temperature was increased from room temperature to 600°C in 5 min under a pressure of 6.5 MPa. Then, the temperature was raised from 600 to $1,500^\circ\text{C}$ at a rate of $10^\circ\text{C}/\text{min}$ and held at $1,500^\circ\text{C}$ for 20 min under 100 MPa pressure. After sintering, the flat surfaces of the sample were polished to remove any residual carbon from the graphite die.

The translucent ceramic phases were analyzed by X-ray diffraction (XRD) pattern that measured in the 2θ range of $10^\circ - 60^\circ$ by SHIMADZU XRD-6,100 X-ray diffractometer with the $\text{Cu-K}\alpha$ (40 kV/15 mA) X-ray source. The XRD pattern of samples were compared with the standard data PDF#63-0291. The images of sample were investigated by using scanning electron microscope (SEM) JSM-IT300 (JEOL, Japan) at 20 kV with secondary electron (SE) mode. The absorption spectra were measured on Hitachi U2900

spectrophotometer. Photoluminescence (PL) spectra were measured by Shimadzu RF-5301PC Spectrofluorophotometer. The photoluminescence decay time analysis was measured by using an Edinburgh 199S spectrofluorometer equipped with a coaxial hydrogen-filled flashlamp.

The scintillation properties of the polished LuYAG translucent ceramic samples were diagnosed by radioluminescence (RL) spectra by using our original setup including an Spellman Monoblock XRB80N100/CB X-ray generator and an Andor DU-420-BU2 CCD spectrometer. The tube voltage and current of the X-ray generator were set to 40 kV and 1.2 mA, respectively (Yanagida *et al.*, 2013). The sample is attached to the fiber optic that leads to the Andor Du-420-BU2 CCD spectrometer. In this measurement, the total X-ray dose is 1 Gy and the observation wavelength is 200-700 nm.

Results and Discussion

Structural Analysis

Figure 1 shows the XRD patterns for $\text{Lu}_{2.5}\text{Y}_{0.5}(\text{Al}_{5-x}\text{Ga}_x)\text{O}_{12}:\text{Ce}$ ($x = 1, 2, \text{ and } 3$) translucent ceramic samples. For better clarity and visual representation, $x = 1, 2, \text{ and } 3$ were labeled as 1Ga, 2Ga, and 3Ga, respectively. All samples were compared with the cubic garnet structure of $\text{Y}_3\text{Al}_5\text{O}_{12}$ (PDF#63-0291). The XRD analysis indicates a slight shift in diffraction peaks with increasing Ga content. This shift was attributed to the larger lattice size caused by the substitution of Ga^{3+} ions (ionic radius 0.47 Å) for Al^{3+} ions (ionic radius 0.39 Å) at the tetrahedral (4-fold) sites. Meanwhile, Lu^{3+} (ionic radius 0.97 Å) and Y^{3+} (ionic radius 1.02 Å) occupy similar dodecahedral (8-fold) sites (Haynes, 2014; Ueda and Tanabe, 2019).

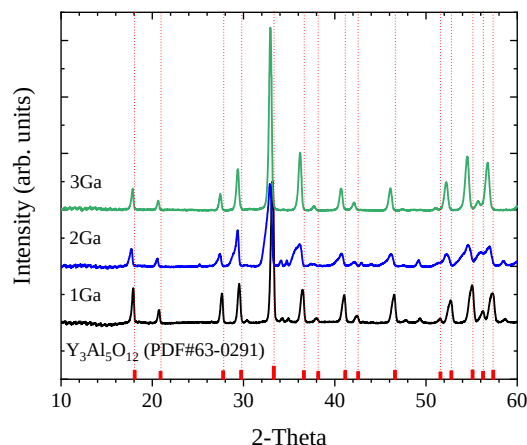


Figure 1. The XRD patterns of the LuYAG:Ce translucent ceramic samples

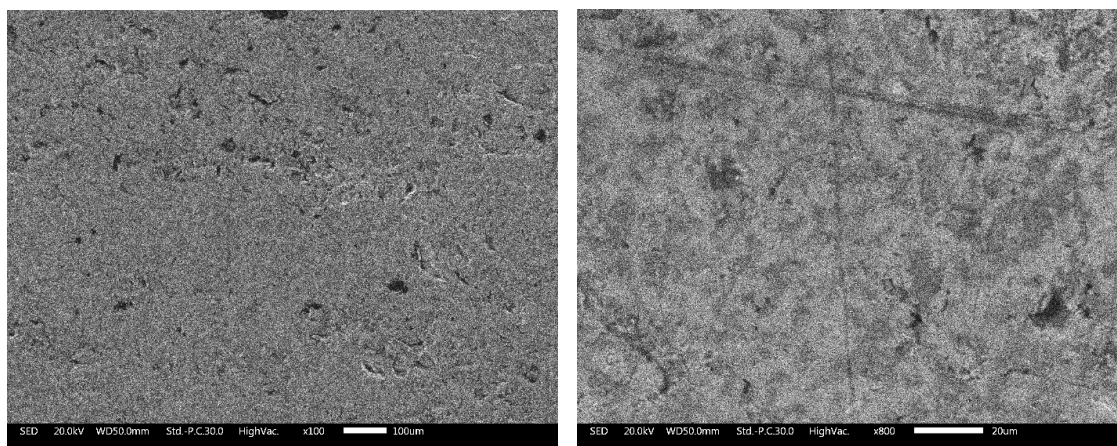


Figure 2. Microstructure of LuYAG:Ce translucent ceramic samples were investigated by scanning electron microscopy (SEM) technique at $\times 100$ and $\times 800$ (1Ga representative sample) magnification, 20kV, SE imaging

Figure 2 displays the microstructure of the 1Ga translucent ceramic as a representative, investigated using SEM at $\times 100$ and $\times 800$ magnifications. The SEM images clearly reveal the microstructure, with the $\times 100$ magnification highlighting numerous porosities on the ceramic surface. At $\times 800$ magnification, the images prominently show the grain boundaries, characteristic of a polycrystalline structure. The grains were separated by these boundaries, and generally exhibit random crystallographic orientations, with sizes ranging from nanometers to millimeters (Wang and Duan, 2018). Moreover, the grain boundaries affect luminescence efficiency, as they contain abundant defects (traps) that can delay electron transport in the scintillation mechanism (Wallace and McKenna, 2014). These trapped electrons lead to energy losses owing to nonradiative recombination at defect sites (Dhanaraj *et al.*, 2010; Yu *et al.*, 2024).

Optical and Photoluminescence Characteristics

Figure 3 shows the absorption spectra of LuYAG:Ce translucent ceramic samples, with an absorption edge around 500 nm. The peaks observed at 420 nm and 340 nm correspond to the $4f-5d_1$ and $4f-5d_2$ absorption transitions of the Ce^{3+} , respectively, (Napierkowski *et al.*, 2023) as indicated by the excitation spectra of Ce^{3+} emission shown in the upcoming PL results.

Figure 4 shows the photoluminescence (PL) and photoluminescence excitation (PLE) spectra of LuYAG:Ce translucent ceramic samples. In the PLE spectrum, two main bands appear: one between 300–380 nm, corresponding to the Ce^{3+} : $4f-5d_2$ transition, and another between 380–500 nm, corresponding to the Ce^{3+} : $4f-5d_1$ transition. Increasing the Ga content caused a blue shift in the $4f-5d_1$ peak, indicating a shift to higher energy, while the $4f-5d_2$ peak shifted to lower energy (red

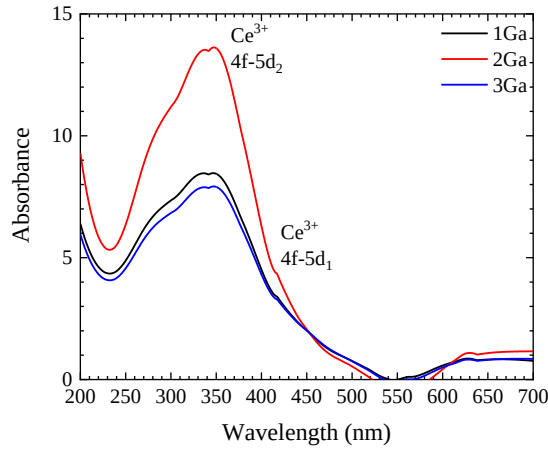


Figure 3. Absorption spectra of the LuYAG:Ce translucent ceramic samples

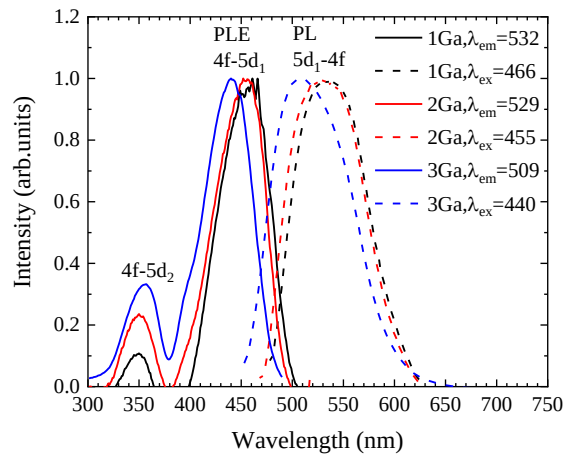


Figure 4. PLE & PL spectra of the LuYAG:Ce translucent ceramic samples

shift), attributed to the decreasing energy gap between the $5d_1$ and $5d_2$ levels (Grigorjeva *et al.*, 2018; Ueda and Tanabe, 2019). This demonstrates that Ga content influences the energy levels of the $5d_{1,2}$ and $4f$ states in LuYAG:Ce, as depicted in Figure 5. The reduction in crystal field splitting (Δ_{12}) of the $5d_1$ and $5d_2$ levels depends on the substitution of the Al^{3+} ion at the octahedral site by the larger Ga^{3+} ion (Dorenbos, 2013; Ueda and Tanabe, 2019). The Δ_{12} values for 1Ga, 2Ga, and 3Ga were 0.89, 0.81, and 0.65 eV, respectively.

The PL emissions of LuYAG:Ce translucent ceramic exhibited an emission band between 450–620 nm, corresponding to the Ce^{3+} : $5d_1$ – $4f$ transition. In addition, both the emission peaks and the $4f$ – $5d_1$ excitation peaks shifted to higher energy (blue shift) as the Ga content increased (Grigorjeva *et al.*, 2018).

The photoluminescence (PL) decay curves of LuYAG:Ce translucent ceramic, excited at 450 nm along with the instrumental response function (IRF) (are presented in Figure 6. The decay curves were fitted using a single exponential decay function: $I(t) = A \exp(-t/\tau) + background$, where A represents the intensity constant, t is the time, and τ is the decay time of the LuYAG:Ce translucent ceramic. The decay times, summarized in Table 1, show a trend of decreasing with increasing Ga content, likely due to thermal ionization of the $5d_1$ level to the conduction band (CB) (Ueda *et al.*, 2011; Ogiegłó *et al.*, 2013; Grigorjeva *et al.*, 2018).

Table 1. PL decay time constants of the LuYAG:Ce translucent ceramic

Samples	τ_1 (ns)
$Lu_{2.5}Y_{0.5}Al_4Ga_1O_{12}:Ce$	51.37
$Lu_{2.5}Y_{0.5}Al_3Ga_2O_{12}:Ce$	47.92
$Lu_{2.5}Y_{0.5}Al_2Ga_3O_{12}:Ce$	34.89

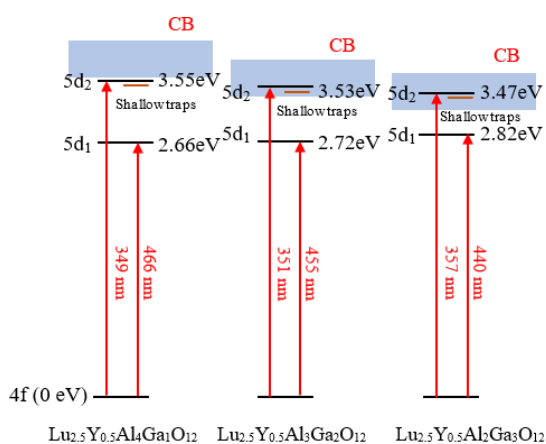


Figure 5. Energy level diagram of Ce^{3+} for LuYAG:Ce translucent ceramic samples

The obtained PL decay time constants correspond to the $5d_1$ – $4f$ transition of Ce^{3+} , and they decrease as the Ga content increases. This decay time is influenced by shallow trapping within the intrinsic ceramic composition, where shallow charge traps slow the movement of electrons and holes toward the luminescence center. Gallium helps mitigate these shallow traps by shifting the bottom of the conduction band downward as the Ga content increases, which affects electron migration (Grigorjeva *et al.*, 2018). Consequently, increasing Ga content reduces the decay time through Ce^{3+} luminescence quenching and thermal ionization of the $5d_1$ excited state (Ueda *et al.*, 2011; Ogiegłó *et al.*, 2013).

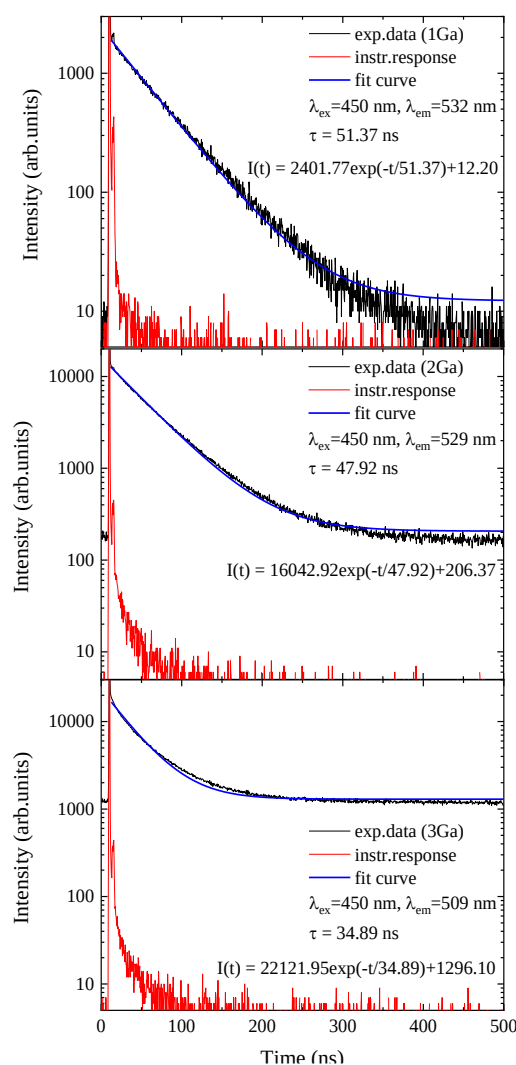


Figure 6. PL decays time of the LuYAG:Ce translucent ceramic samples

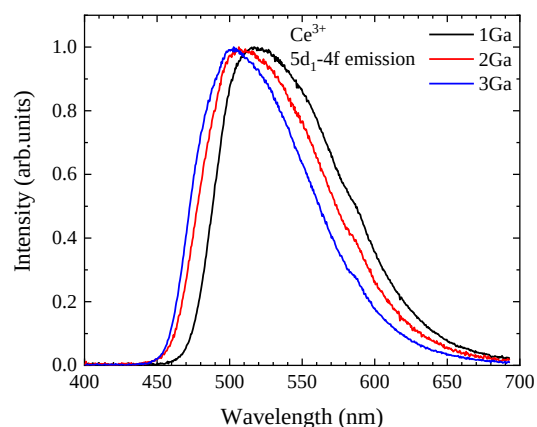


Figure 7. RL spectra of the LuYAG:Ce translucent ceramic samples

Scintillation Characteristics

Figure 7 presents RL spectra of the LuYAG:Ce translucent ceramic samples under the X-ray irradiation. The RL spectra revealed scintillation wavelengths ranging from 450 to 690 nm, attributed to the Ce^{3+} : $5d_1$ -4f transition, similar to the PL spectra shown in Figure 4. Moreover, the scintillation spectra exhibited a slight blue-shifted, which was associated with the elevation of the $5d_1$ energy level as the Ga content increased (Grigorjeva *et al.*, 2018; Ueda and Tanabe, 2019).

Conclusions

The luminescence and scintillation properties of translucent ceramic $\text{Lu}_{2.5}\text{Y}_{0.5}(\text{Al}_{5-x}\text{Ga}_x)\text{O}_{12}$ ($x = 1, 2, 3$) doped with 1 mol% Ce, synthesized using spark plasma sintering (SPS), were investigated for potential X-ray detection applications. The samples were prepared from a combination of lutetium (Lu), yttrium (Y), aluminum (Al), and gallium (Ga), with cerium (Ce) as the dopant. XRD patterns revealed a shift in diffraction peaks with increasing Ga content due to the larger ionic radius of Ga substituting for Al ions. SEM analysis showed distinct grain boundaries typical of polycrystalline materials, along with visible porosities that influence luminescence efficiency. The absorption spectra exhibited characteristic Ce^{3+} absorption bands at 340 nm and 420 nm, corresponding to 4f-5d transitions.

The photoluminescence spectra showed a broad emission band between 450-620 nm, corresponding to the $5d_1$ -4f transitions of Ce^{3+} , with a noticeable blue shift as Ga content increased. Additionally, the photoluminescence decay time

shortened with increasing Ga content, attributed to thermal ionization of the $5d_1$ state into the conduction band. Radioluminescence (RL) measurements under X-ray excitation displayed a broad emission in the 450-690 nm range, closely matching the PL spectra. The RL spectra also exhibited a slight blue shift with increasing Ga content. In conclusion, increasing Ga content in these ceramic scintillators modifies luminescence efficiency, reduces decay time, and improves their suitability for X-ray detection applications.

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