

A COMPARISON OF THE LUMINESCENCE AND SCINTILLATION PROPERTIES OF $\text{Lu}_{2.5}\text{Y}_{0.5}\text{Al}_5\text{O}_{12}:\text{Ce}$ SINGLE CRYSTAL AND TRANSLUCENT CERAMIC

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Abstract

This research studied the comparative luminescence and scintillation properties of Ce-doped $\text{Lu}_{2.5}\text{Y}_{0.5}\text{Al}_5\text{O}_{12}$ single crystal and translucent ceramic. The translucent ceramic and single crystal samples were fabricated by spark plasma sintering (SPS) furnace and the floating zone (FZ) furnace, respectively. The microstructure of both samples was investigated by scanning electron microscopy (SEM) technique. The transmittance of single crystal showed 77% in visible light range with the absorption at 350 nm (4f-5d₂) and 450 nm (4f-5d₁), while the translucent ceramic exhibited approximately 8% in visible light range with absorption is 350 nm (4f-5d₂) and 490 nm (4f-5d₁). The photoluminescence (PL) emission band was 518 nm (Ce^{3+} : 5d-4f transition) for single crystal and 548 nm (Ce^{3+} : 5d-4f transition) of translucent ceramic. The radioluminescence spectra presented a broad emission band 490-700 nm for both samples. At 662 keV γ -rays, the scintillation light yield value of single crystal and translucent ceramic was 6,700 and 4,900 photons/MeV, respectively, via comparison with Ce-doped Gd_2SiO_5 (GSO:Ce). The PL decay curves were fitted by single exponential function with a fast decay time constant of 51 ns for single crystal and 60 ns for translucent ceramic.

Keywords: Crystal; Light yield; Scintillator; Translucent ceramic

Introduction

Scintillator is a luminescence material which can change high energy photons to UV or visible light (Yanagida *et al.*, 2023). The often-utilized scintillators are glass (Akatsuka *et al.*, 2021), plastic (Koshimizu *et al.*, 2019), ceramic and single crystal

(Kantuptim *et al.*, 2022) as single crystal scintillator being the most popular. Scintillator materials are used in the security check, non-destructive analysis, high energy physics and medical fields such as positron emission tomography (PET), single photon

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emission computed tomography (SPECT) scan (Blasse and Grabmaier, 1994; Ronda, 2008). The important properties for a scintillator material are high light output and a short decay time (Eijk, 2003). At present, researchers around the world still have a common mission to improve the mentioned properties of scintillator material. Therefore, this study intends to develop ceramic scintillator, with a fast fabrication time, low fabrication temperature, good mechanical properties and low product cost. Generally, single crystal scintillator is a stable structure with high light yield, especially commercially available $\text{Y}_3\text{Al}_5\text{O}_{12}$ (YAG:Ce) with light yield about 28,000 ph/MeV (Nikl and Yoshikawa, 2015) and Ce^{3+} doped $\text{Lu}_3\text{Al}_5\text{O}_{12}$ (LuAG:Ce) with light yield about 26,000 ph/MeV (Dujardin *et al.*, 2010). However, single crystal has a complex fabricate and high product cost. By these reason, we are interested in studying translucent ceramic comparison with single crystal to improve ceramic scintillator, which has similar properties to single crystal.

This work is utilizing superb characteristic between lutetium (Lu_2O_3) and $\text{Y}_3\text{Al}_5\text{O}_{12}$ (YAG) garnet as doped Ce^{3+} in order to produce a great scintillator material. Lutetium is discussed to be an outstanding competition for rare earth activator ions owing to it has a good physical properties for scintillator field such as high phase stability and low thermal expansion (Tang *et al.*, 2021) especially increasing Z_{eff} of sample that is effect on light output (Wang *et al.*, 2013) as well. Thus, translucent ceramic and single crystal $\text{Lu}_{2.5}\text{Y}_{0.5}\text{Al}_5\text{O}_{12}:\text{Ce}$ have the potential to present high light yield value under the γ -rays. In addition, the translucent ceramic and single crystal samples are fabricated by spark plasma sintering (SPS) furnace and the floating zone (FZ) furnace, respectively.

Materials and methods

Chemical powders of Al_2O_3 , Y_2O_3 , Lu_2O_3 , and CeO_2 with 99.99 % purity were used as starting materials for fabrication of ceramic and crystal scintillator. The starting chemical powders were weighed according to the composition of $\text{Lu}_{2.50}\text{Y}_{0.50}\text{Al}_{5.0}\text{O}_{12}$ (LuYAG) with Ce 1.0 mol% in respect of Lu and Y combined. All of the powered were mixed in the agate mortar for each sample.

For the single crystal sample, the mixed powder was packed in the rubber mole for rod shaping by isostatic pressure. The compacted powder rods were sintered at 1200°C for 10 h. After sintering process, the sintered rod was single-crystal growth by dual halogen lamps floating-zone

furnace (Canon Machinery FZD0192) with the pulling down rate of 4 mm/h. Each single-crystal rod was cut and polished for suitable measurement conditions.

For the translucent ceramic sample, the 0.5 g of the mixture was loaded in a graphite die and held between two graphite punches. A sintering condition consists of two steps by using spark plasma sintering (SPS) furnace (Sinter Land LabX-100) in vacuum atmosphere. First, the temperature was increased from room temperature to 600°C in 5 min with pressure of 6.5 MPa. Next, the temperature was elevated from 600 to 1500 °C at a rate of 10°C/min and kept at 1500°C for 20 min while applying a pressure of 100 MPa. After sintering, the flat surfaces of the sample pallet were polished to remove residual carbon from the graphite die.

X-ray diffraction (XRD) pattern was measured in the 2θ range of 10° - 80° using SHIMADZU XRD-6100 X-ray diffractometer with the Cu-K α X-ray source to determine the phase composition of the translucent ceramic and single crystal sample. The images of these samples were investigated by using SEM JSM-IT300 (JEOL, Japan) at 20 kV with secondary electron (SE) mode. Transmittance spectra were tested by with Shimadzu SolidSpec-3700 spectrometer. Photoluminescence (PL) spectra were measured using Shimadzu RF-5301PC Spectro fluorophotometer. Radioluminescence (RL) spectra were recorded using the X-ray-induced scintillation spectra are evaluated by using our in-house developed instruments (Yanagida *et al.*, 2013a). The sample is attached to the fiber optic that leads to the CCD spectrometer (DU-420-BU2, Andor). In this measurement, the total X-ray dose is 1 Gy and the observation wavelength is 200-700 nm.

Light yield (LY) measurements were done under the excitation of 662 keV γ rays (^{137}Cs source) with the photomultiplier tube (Hamamatsu Photonics R7600-200), pre-amplifier (ORTEC 113), shaping amplifier (ORTEC 572), and a multichannel analyzer (MCA, Amptec Pocket MCA 8000A). The signal from the MCA are fed into a personal computer for analysis (Yanagida *et al.*, 2013b). In addition to the setup, scintillation light yield of the sample in this study was calculated in comparison with the Ce-doped Gd_2SiO_5 (GSO:Ce) reference sample. Lastly, photoluminescence decay time was measured by using an Edinburgh 199S spectrofluorometer equipped with a coaxial hydrogen-filled flashlamp. Figure 1 shows the photograph of translucent ceramic and single crystal samples.

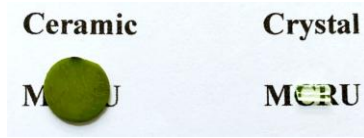


Figure 1. Photograph of translucent ceramic and single crystal samples

Results and Discussion

Structural analysis

Figure 2 shows the XRD pattern of translucent ceramic and single crystal of $\text{Lu}_{2.5}\text{Y}_{0.5}\text{Al}_5\text{O}_{12}:\text{Ce}$. Both sample patterns match with a reference pattern of cubic $\text{Y}_3\text{Al}_5\text{O}_{12}$ (PDF#63-0291), which confirm translucent ceramic and single crystal samples has been successfully fabricated with the single phases of YAG. In addition, the ionic radii of Lu^{3+} (0.98 Å) are similar size to Y^{3+} (1.02 Å). Thus, Lu^{3+} is substituted Y^{3+} at dodecahedral sites (Ueda and Tanabe, 2019).

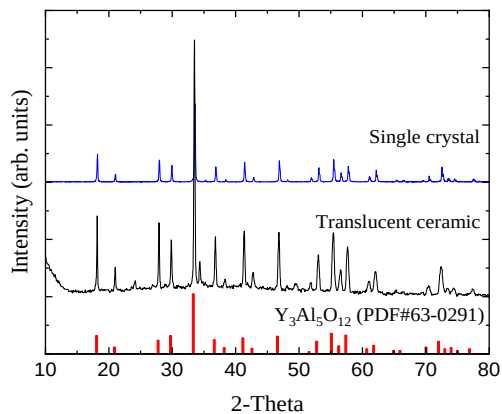
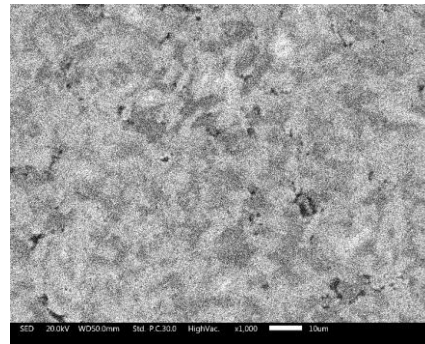


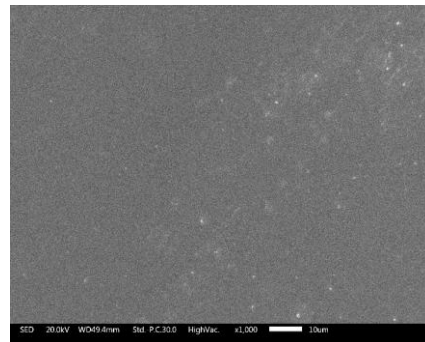
Figure 2. XRD pattern of LuYAG:Ce translucent ceramic and single crystal comparison with reference pattern of garnet structure of YAG.

Figure 3 presents the microstructure of translucent ceramic and single crystal was investigated by scanning electron microscopy (SEM) technique at $\times 1,000$ magnification. The SEM image exhibited microstructure of translucent ceramic and single crystal so clearly which single crystal had no grain boundary. On the other hand, translucent ceramic sample showed clear grain boundaries as polycrystalline characteristics. The grains are separated by grain boundaries and generally have random crystallographic orientations. The magnitude of the grains may differ

from nanometers to millimeters (Wang and Duan, 2018). Furthermore, the grain boundaries are effect on luminescence efficiency because grain boundaries region exist of abundant defects (trap) that possibly delay electrons in transport process of scintillation mechanism (Dhanaraj *et al.*, 2010; Wallace and McKenna, 2014; Yu *et al.*, 2023). Finally, electrons are trapped at defects and lead to energy losses due to nonradiative recombinations (Dhanaraj *et al.*, 2010).



(a) Translucent ceramic



(b) Single crystal

Figure 3. Microstructure of LuYAG:Ce a) translucent ceramic and b) single crystal was investigated by scanning electron microscopy (SEM) technique at $\times 1,000$ magnification, 20kV, SE imaging

Optical and photoluminescence characteristics

Figure 4 presents the transmittance spectra of LuYAG:Ce translucent ceramic and single crystal samples. The transmittance of single crystal showed 77% in the visible light region and absorption at 350 nm ($4f-5d_2$), 450 nm ($4f-5d_1$) in while translucent ceramic exhibited approximately 8% in the visible region (low transmittance) as absorption is 350 nm ($4f-5d_2$), 490 nm ($4f-5d_1$). The Ce^{4+} charge transfer (CT) absorption is about 250 nm (Kamada *et al.*, 2015; Lucchini *et al.*, 2016) that can observe in both samples.

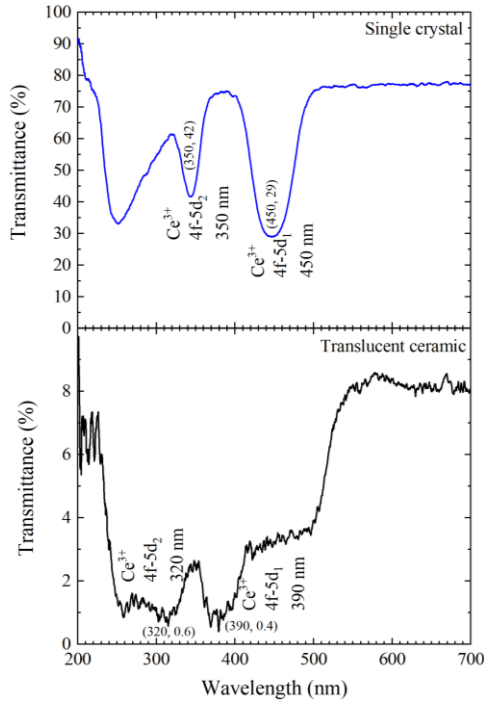


Figure 4. Transmittance spectra of LuYAG:Ce translucent ceramic and single crystal samples

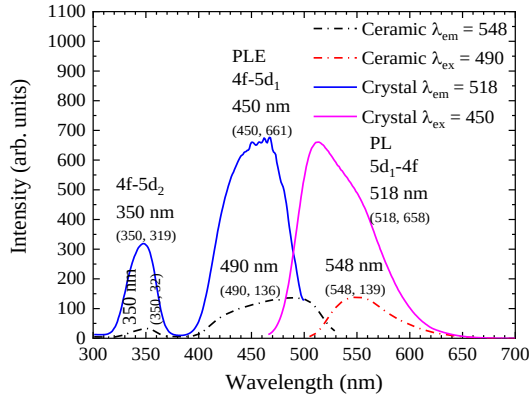


Figure 5. Photoluminescence PL and PLE spectra of LuYAG:Ce translucent ceramic and single crystal samples

The photoluminescence PL and PLE spectra of translucent ceramic and single crystal are shown in Figure 5. The PL spectra of translucent ceramic exhibit emission peak at 548 nm from the Ce^{3+} : $5d_1$ - $4f$ transition (510-650 nm) under excitation at 490 nm as the Ce^{3+} : $4f$ - $5d_1$ absorption (Chen *et al.*, 2019; Maa *et al.*, 2019). However, the PL band of single crystal is between 470-650 nm as Ce^{3+} : $5d_1$ - $4f$ transition, which shows a peak at 518 nm from

excitation at 450 nm (Ce^{3+} : $4f$ - $5d_1$) (Nikl *et al.*, 2013). Two outstanding bands in the PLE spectrum of both samples posit region 320 - 380 nm and 380 - 500 nm belong to Ce^{3+} : $4f$ - $5d_2$ and $4f$ - $5d_1$ transitions, respectively.

Figure 6 presents photoluminescence decay (PL decay) curve of LuYAG:Ce translucent ceramic and single crystal samples. The decay curves of both samples are approximated by a single-exponential function: $I(t) = \sum A_i \exp(-t/\tau_i) + \text{background}$. A_i is an initial intensity of the τ_i decay component. Both samples were excited at 330 nm and emitted at 548 nm (translucent ceramic), 518 nm (single crystal). The decay time constants of LuYAG:Ce translucent ceramic and single crystal are 60 and 51 ns, respectively, which these values are typical for the Ce^{3+} : $5d_1$ - $4f$ transition (Lyu and Hamilton, 1991; Nakauchi *et al.*, 2023). Both translucent ceramic and single crystal display fast decay time, which is a desired property for the scintillator material.

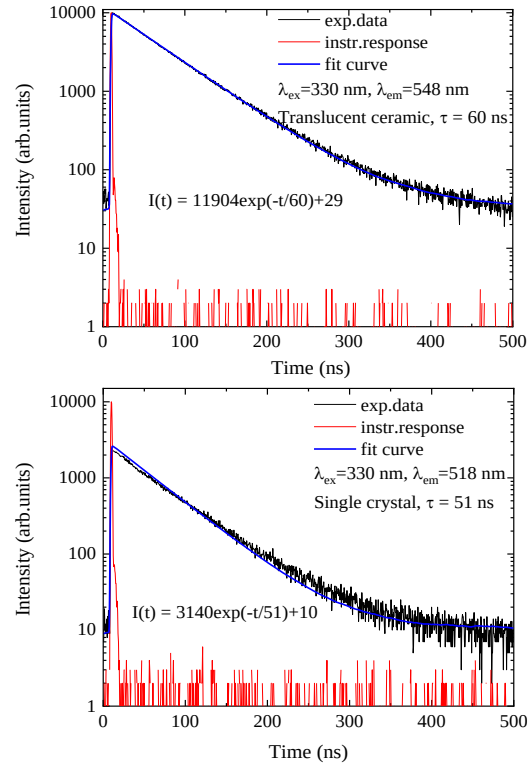


Figure 6. PL decay curve of LuYAG:Ce translucent ceramic and single crystal samples

Scintillation characteristics

Radioluminescence (RL) spectra of LuYAG:Ce translucent ceramic and single crystal samples were tested under the same conditions are

presented in Figure 7. The emission band under excitation via X-ray exhibited between 500-690 nm (peak at 548 nm) from Ce^{3+} : $5d_1-4f$ transition for translucent ceramic while single crystal presented peak at 518 nm and emission band was 480-690 nm belong to Ce^{3+} : $5d_1-4f$ as well. The integral scintillation efficiency (defined as integrated intensity of RL spectrum) of single crystal is about 5 times of translucent ceramic.

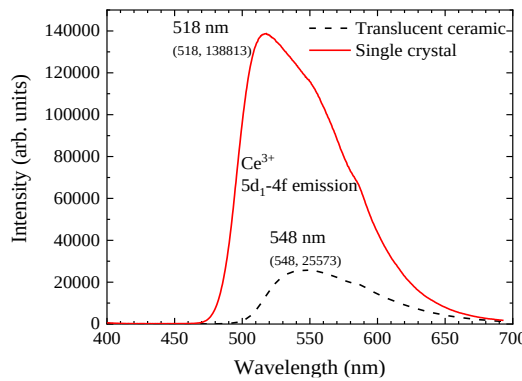


Figure 7. RL spectra of LuYAG:Ce translucent ceramic and single crystal samples

The pulse height spectra of 662 keV γ rays from a ^{137}Cs source measured with LuYAG:Ce samples and reference GSO:Ce crystal are exhibited in Fig 8. The Light yield (LY) of LuYAG:Ce ceramic and crystal was assigned by comparison with the GSO:Ce (LY = 7,000 ph/MeV) reference which the shaping time constant was determined at 2.0 μs for both samples as high voltage (HV) was 700 and 600 kV for translucent ceramic and single crystal, respectively. The LY_γ and energy resolution (%R) values are displayed in Table 1. The translucent ceramic scintillator exhibited LY_γ value lower than the single crystal scintillator. One of the possible reasons was rather poor optical transmittance (8%) in a visible region that related with RL of translucent ceramic was less than single crystal as well. Moreover, microstructure of translucent ceramic has many grain boundaries which consist of numerous defects or traps (Trofimov *et al.*, 2021) (as shown in Figure 8), so it affects the LY value (Kumar *et al.*, 2017).

LY_γ value of translucent ceramic and single crystal in this work may not high when compared with scintillator in the garnet family including YAG:Ce at 20,300 ph/MeV (Moszyński *et al.*, 1994) and other Ce activated scintillator such as GSO:Ce at around 10,000 ph/MeV (Itoh *et al.*, 2007). However, when comparing the samples in

this study with glass scintillator, it is very explicit their LY value more than glass scintillator. The synthesis glass scintillator $50\text{SiO}_2 - 20\text{Al}_2\text{O}_3 - 15\text{BaF}_2 - 15\text{Gd}_2\text{O}_3$ doped with CeO_2 (Chewpraditkul *et al.*, 2019) presented a LY_γ value 2050 ph/MeV under excitation by γ -ray that is less than our work.

Table 1. The LY_γ and energy resolution ($\Delta E/E$) values of LuYAG:Ce samples and comparison with reference GSO:Ce crystal

Vehicle type	Lateral shift (meters)		
	TW	3W	Car
Curve 1	2.88	2.93	2.09
Curve 2	0.96	0.98	0.83
Curve 3	1.05	1.09	0.88
Curve 4	1.38	1.49	0.81

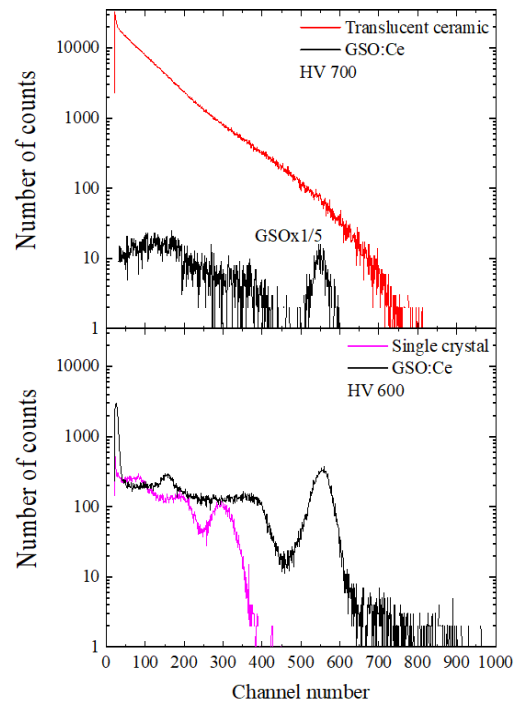


Figure 8. Pulse height spectra of 662 keV γ rays measured with LuYAG:Ce samples and comparison with reference GSO:Ce crystal

Conclusions

The LuYAG:Ce translucent ceramic was fabricated by using spark plasma sintering (SPS) technique in vacuum atmosphere. On the other hand, single crystal of the same material was grown by floating-zone method with the pulling down rate of 4 mm/h.

SEM image technique showed microstructure of samples that were single crystal and polycrystalline (many grain boundaries) belong to single crystal and translucent ceramic scintillator, respectively. The grain boundaries are effect on luminescence efficiency due to grain boundaries area contain of various defects (trap) that probably delay electrons in the transport process of scintillation mechanism. The transmittance (%T) of single crystal exhibited 77% in the visible light range while translucent ceramic was 8%. The photoluminescence (PL) emission band presented 518 nm (Ce^{3+} : $5d_1-4f$ transition) for single crystal and 548 nm (Ce^{3+} : $5d_1-4f$ transition) of translucent ceramic.

The integral scintillation efficiency of single crystal was about 5 times of translucent ceramic. The emission band of samples when was excited by X-ray source which exhibited between 500-690 nm (peak at 548 nm) from Ce^{3+} : $5d_1-4f$ transition belong to translucent ceramic while single crystal presented peak at 518 nm and emission band was 480-690 nm of Ce^{3+} : $5d_1-4f$ as well. The pulse height spectra with the 662keV γ -rays presented LY values of 4,900 and 6,700 ph/MeV for translucent ceramic and single crystal, respectively. The decay time response for both samples presented fast decay in tens of nanoseconds. An increasing of LY value for translucent ceramic is improving the fabrication technique to enhance its transparency.

Lastly, the LuYAG:Ce translucent ceramic and single crystal samples still display good properties for scintillator materials, so the both samples can be applied for radiation detector and non-destructive testing in the engineering field.

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