

OPTICAL AND SCINTILLATION PROPERTIES OF Mg^{2+} - CODOPED $\text{Gd}_2\text{YAl}_{2.4}\text{Ga}_{2.6}\text{O}_{12}:\text{Ce}$ CERAMIC SCINTILLATOR

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Received: February 01, 2024; Revised: March 26, 2024; Accepted: April 03, 2024

Abstract

Mg^{2+} - codoped $\text{Gd}_2\text{YAl}_{2.4}\text{Ga}_{2.6}\text{O}_{12}:\text{Ce}$ ceramic scintillator was fabricated by sintering in an oxygen atmosphere and hot isostatic pressing. It exhibits a high integral scintillation efficiency of 400 % of $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ crystal scintillator. Under excitation with 662 keV γ rays, it shows a light yield value of 22,200 photons/MeV along with scintillation decay times of 24.5 ns (24%) + 75 ns (76%). The scintillation response under excitation with 5.5 MeV α particles from a ^{238}Pu source was also investigated and discussed.

Keywords: Ce^{3+} ; $\text{Gd}_2\text{YAl}_{2.4}\text{Ga}_{2.6}\text{O}_{12}:\text{Ce}$; luminescence; scintillation; transparent ceramic

Introduction

Great effort to develop efficient scintillators with high light yield (LY) and fast response time is needed for modern medical imaging apparatus, such as single photon emission computed tomography (SPECT) and positron emission tomography (PET). Ce^{3+} -doped $\text{Lu}_3\text{Al}_5\text{O}_{12}$ (LuAG:Ce) and $\text{Y}_3\text{Al}_5\text{O}_{12}$ (YAG:Ce) single crystals are good candidates for these applications (Van Eijk, 2003; Nikl, 2006). However, the best LY value of about 26,000 ph/MeV for LuAG:Ce (Dujardin *et al.*, 2010) and about 28,000 ph/MeV for YAG:Ce (Nikl and

Yoshikawa, 2015) is much lower than the theoretical value of $\sim 60,000$ ph/MeV (Dorenbos, 2010). It is mainly caused by the trapping effect of carriers at antisite Lu_{Al} (Y_{Al}) defect-related shallow traps (Nikl *et al.*, 2005), delaying the energy transport to the Ce^{3+} luminescence centers and the scintillation pulse comprised of a high content of slower component (Nikl *et al.*, 2007; Chewpraditkul *et al.*, 2009). Partial substitution of Al^{3+} with larger Ga^{3+} ions in the aluminum garnet can lower the bottom of the conduction band (CB) to bury shallow

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electron traps (Nikl *et al.*, 2006), while partial substitution of Y^{3+} with larger Gd^{3+} ions downshifts the $\text{Ce}^{3+} 5d_1$ level to prevent thermal ionization in $(\text{Gd},\text{Y})_3\text{Al}_5\text{O}_{12}:\text{Ce}$ (Li *et al.*, 2012; Chen *et al.*, 2015). Consequently, an admixture of Gd^{3+} and Ga^{3+} into aluminum garnet can enhance the LY value of $(\text{Gd},\text{Y})_3(\text{Al},\text{Ga})_5\text{O}_{12}:\text{Ce}$ ceramics (Cherepy *et al.*, 2010) and $(\text{Lu},\text{Gd})_3(\text{Ga},\text{Al})_5\text{O}_{12}:\text{Ce}$ single crystals (Kamada *et al.*, 2011). 4-inch diameter $\text{Gd}_3\text{Al}_2\text{Ga}_3\text{O}_{12}:\text{Ce}$ (GAGG:Ce) single crystal with LY value of 53,000 ph/MeV and scintillation decay time of 76 ns was recently reported (Kochurikhin *et al.*, 2020). A very high LY value of 65,000 ph/MeV with a scintillation decay time of 94 ns was also reported for $\text{Gd}_2\text{YAl}_{2.3}\text{Ga}_{2.7}\text{O}_{12}:\text{Ce}$ single crystal (Wang *et al.*, 2016). $\text{Gd}_2\text{YAl}_2\text{Ga}_3\text{O}_{12}:\text{Ce}$ single crystal with a moderate LY value of 38,000 ph/MeV but a faster scintillation decay time of 57 ns was also reported in our previous study (Chewpraditkul *et al.*, 2018). Acceleration of scintillation decay time with an expense of LY value by Mg^{2+} codoping in GAGG:Ce,Mg (Kamada *et al.*, 2015; Lucchini *et al.*, 2016) and $(\text{Gd},\text{Y})_3(\text{Ga},\text{Al})_5\text{O}_{12}:\text{Ce},\text{Mg}$ (Chewpraditkul *et al.*, 2020; Korzhik *et al.*, 2020) single crystals were investigated. The presence of Ce^{4+} centers is identified by an appearance of its charge transfer (CT) band in the absorption spectrum below 350 nm (Kamada *et al.*, 2015; Lucchini *et al.*, 2016), and also proved by X-ray absorption near edge spectroscopy (Wu *et al.*, 2014; Dantelle *et al.*, 2019). The stabilization of Ce^{4+} centers by divalent ions co-doping in Ce-doped garnet scintillators can provide an additional fast radiative recombination pathway in the scintillation mechanism (Nikl *et al.*, 2014).

Transparent ceramics are alternative scintillators as compared to single crystals due to lower production cost, uniform doping, and lower fabrication temperature (Cherepy *et al.*, 2010; Liu *et al.*, 2014; Fujimoto *et al.*, 2014; Luo *et al.*, 2016; Li *et al.*, 2016). The scintillation properties of $\text{Gd}_2\text{YAl}_2\text{Ga}_3\text{O}_{12}$ (GYAGG:Ce) ceramics were recently investigated (Chen *et al.*, 2017; Chen *et al.*, 2019a; 2019b). Chen *et al.* (2019) reported a high LY value above 40,000 ph/MeV along with two scintillation decay components of 56 ns (66%) + 474 ns (34%) for a GYAGG:Ce (0.2 at.%) ceramic. A moderate LY value of 38,400 ph/MeV with faster scintillation decay times of 34 ns (56%) + 145 ns (44%) was reported for Mg^{2+} - codoped GYAGG:Ce(0.07 at.%) ceramic (Chen *et al.*, 2019).

In this work, the Mg^{2+} codoped $\text{Gd}_2\text{YAl}_{2.4}\text{Ga}_{2.6}\text{O}_{12}:\text{Ce}$ (GYAGG:Ce,Mg) transparent ceramic was fabricated, and its scintillation properties were investigated. In principle, faster scintillation decays could be achieved with

the higher Ce^{3+} concentration, in addition to the Mg^{2+} co-doping mentioned. In this work, the Ce concentration was 0.5 at.%, and Mg^{2+} concentration was 0.1 at.%. The measurements of optical absorption and transmittance spectra, photoluminescence (PL) emission and excitation (PLE) spectra, X-rays excited radioluminescence (RL), LY, and scintillation decay time are presented.

Materials and Methods

Chemical powders of Al_2O_3 , Y_2O_3 , Gd_2O_3 , Ga_2O_3 , CeO_2 , and MgO with 99.99 % purity were used as starting materials for the fabrication of ceramic scintillator. The powders were weighed according to the composition of $\text{Ce}_{0.015}\text{Mg}_{0.003}\text{Gd}_{1.988}\text{Y}_{0.994}\text{Al}_{2.4}\text{Ga}_{2.6}\text{O}_{12}$ (GYAGG:Ce,Mg). The powders were mixed and milled in ethanol for 12 h. After dried at 70°C for 4 h and sieved through a 200-mesh screen, the mixtures were pre-sintered at 800°C for 2 h, dry pressed into Ø 15 mm pellets, and cold isostatically pressed (CIP) at 210 MPa. The obtained pellets were sintered at 1,650°C for 5 h in an oxygen atmosphere to get the transparent ceramics. The ceramic samples were hot isostatically pressed (HIP) under argon gas pressure of 200 MPa at 1,700°C for 2 h to remove the residual closed porosity. Finally, the ceramic samples were annealed in an oxygen atmosphere at 1,350°C for 15 h to remove oxygen vacancies brought during the HIP process. The polished ceramic with a thickness of 2 mm was used for optical and scintillation measurements.

Powder X-ray diffraction (XRD) pattern was recorded 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 ceramic sample. Optical absorption and transmittance spectra were measured with Hitachi U2900 spectrophotometer. PL and PLE spectra were measured using a Shimadzu RF-5301PC Spectro-fluorophotometer. RL spectra were measured using Ocean Optics QE65000 fiber optics spectrometer and under excitation with an X-ray tube (Inel X-ray generator; 2 kW/Cu anode).

LY measurements were done under the excitation of 662 keV γ rays (^{137}Cs source) and 5.50 MeV α particles (^{238}Pu source) using photomultiplier (R6231-100 PMT) as readout; the signal was processed by CANBERRA 2005 preamplifier, and Tennelec TC243 spectroscopy amplifier and Tukan 8k MCA were used to record the pulse height spectra. Scintillation decay time was measured using R7600U-200 PMT as readout, and the scintillation time profile was registered with a TDS3054B oscilloscope. This work BGO

($\text{Bi}_4\text{Ge}_3\text{O}_{12}$) crystal was used reference for RL & LY testing. BGO crystal is a commercial grade from CRYTUR Ltd., Czech Republic, and it has a LY value of about 8,600 ph/MeV.

Results and Discussion

Structural Analysis

Figure 1 presents the XRD pattern of $\text{Gd}_2\text{YAl}_{2.4}\text{Ga}_{2.6}\text{O}_{12}:\text{Ce},\text{Mg}$ (GYAGG:Ce,Mg) ceramic sample, which is well indexed as the cubic garnet structure of $\text{Y}_3\text{Al}_5\text{O}_{12}$ (PDF#63-0291) and without other impurity peaks detected, indicating that the single garnet phase of GYAGG:Ce,Mg ceramic has been successfully fabricated.

A slight shift of the diffraction peaks to the smaller angle is observed for the GYAGG:Ce,Mg with respect to the YAG garnet, which indicates a larger lattice size of the fabricated garnet host. This phenomenon is caused by the larger ionic radii of Gd^{3+} (1.053 Å) than Y^{3+} (1.019 Å) at dodecahedral sites, and Ga^{3+} (0.47 Å) is larger than Al^{3+} (0.39 Å) at tetrahedral sites.

Optical and Photoluminescence Characteristics

Figure 2(a) shows the photographs of the prepared Ce^{3+} -doped ceramic sample, and Figure 2(b) presents the optical absorbance (top) and transmittance (bottom) spectra of GYAGG:Ce,Mg ceramic sample. The absorption band centered at 360 nm and broadband at 400-500 nm belong to the $4f \rightarrow 5d_2$ and $4f \rightarrow 5d_1$ transitions of Ce^{3+} ions in a garnet host, respectively. The Ce^{4+} CT absorption band below 340 nm (Kamada *et al.*, 2015; Lucchini *et al.*, 2016) is clearly observed. Rather, low transmittance, about 35% at the 500-700 nm range, was obtained for the ceramic sample.

The normalized PL and PLE spectra of ceramic are shown in Figure 3. The PL band

between 480-660 nm due to the $\text{Ce}^{3+} 5d_1 \rightarrow 4f$ transition is observed under excitation at 475 nm of $\text{Ce}^{3+} 4f \rightarrow 5d_1$ absorption. Two dominant bands in the PLE spectrum of $\text{Ce}^{3+} 5d_1 \rightarrow 4f$ emission (at 536

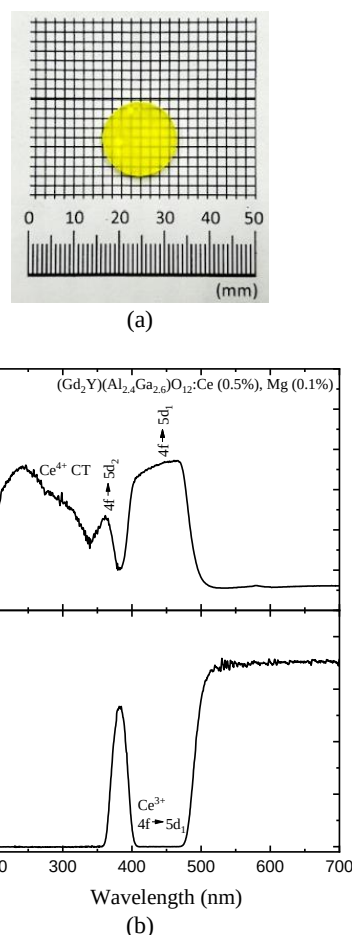


Figure 2. a) Photograph of the prepared ceramic samples and b) Optical absorbance (top) and transmittance (bottom) spectra of GYAGG:Ce,Mg ceramic

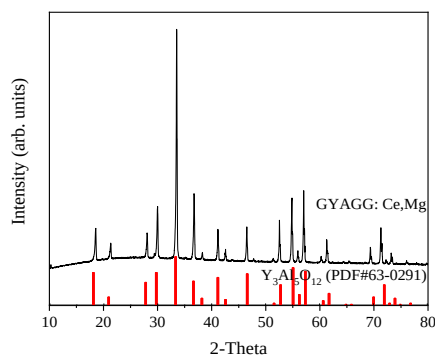


Figure 1. XRD pattern of GYAGG:Ce,Mg ceramic in comparison with the garnet structure of YAG

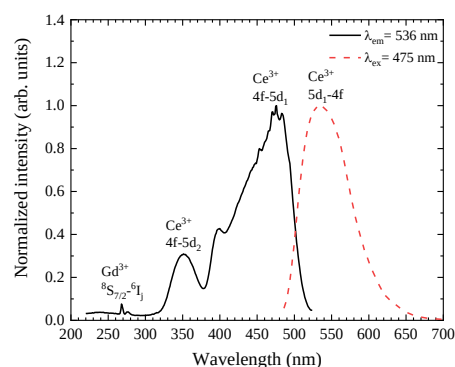


Figure 3. Normalized PL and PLE spectra of GYAGG:Ce,Mg ceramic

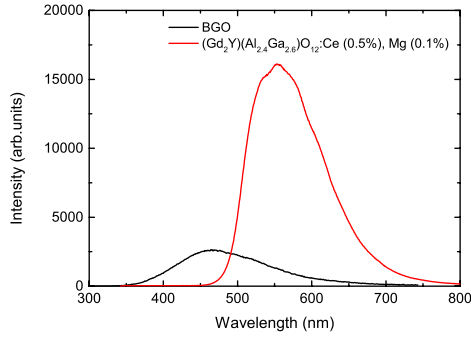


Figure 4. RL spectra of GYAGG:Ce,Mg ceramic and BGO

Table 1. LY and scintillation decay components of GYAGG:Ce,Mg and BGO

Sample	LY (ph/MeV)	τ_i ns (I_i %)
GYAGG: Ce,Mg (This work)	22,200 $\pm$ 1100	24.5(24); 75(76)
GYAGG: Ce,Mg (Chen <i>et al.</i> , 2019)	38,400 $\pm$ 2000	34(56); 145(44)
GYAGG: Ce crystal (Chewpraditkul <i>et al.</i> , 2018)	38,000 $\pm$ 2000	57(80); 600(20)
GYAGG: Ce (Chen <i>et al.</i> , 2019)	40,000 $\pm$ 2100	56(66); 474(34)
BGO	8,600 $\pm$ 440	n.m.

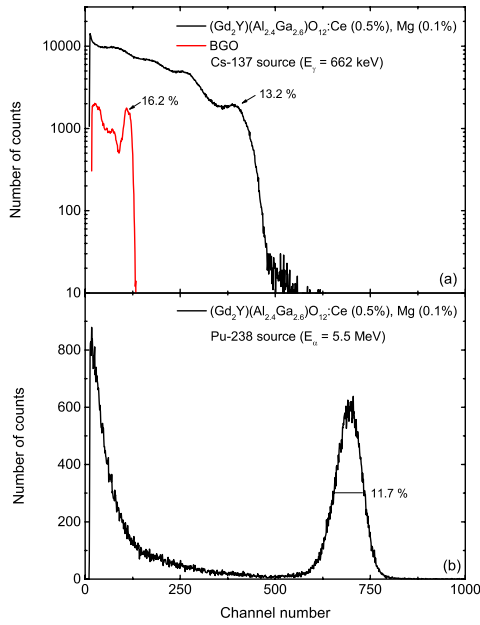


Figure 5. Pulse height spectra of 662 keV γ rays and 5.5 MeV alpha particles measured with GYAGG:Ce,Mg ceramic

nm) located between 320 - 380 nm and 380-510 nm belong to $\text{Ce}^{3+} 4f \rightarrow 5d_2$ and $4f \rightarrow 5d_1$ transitions, respectively. The weak line at 272 nm due to the $\text{Gd}^{3+} {}^8\text{S}_{7/2} \rightarrow {}^6\text{I}_J$ absorption transition is also observed, which indicates the non-efficient energy transfer from Gd^{3+} sublattice to the Ce^{3+} centers in this ceramic (Kucera *et al.*, 2013; Chewpraditkul *et al.*, 2016). This result can be attributed to the non-radiative energy transfer from the $\text{Gd}^{3+} {}^6\text{I}_J$ excited state to the Ce^{4+} CT absorption band which is strongly overlapped as shown in Figure 2.

Scintillation Characteristics

RL spectra of GYAGG:Ce,Mg ceramic, and $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ (BGO) crystal measured under the same

conditions are shown in Figure 4. The integral scintillation efficiency (defined as the integrated intensity of RL spectrum) of GYAGG:Ce,Mg is about 400% of BGO.

The pulse height spectra of 662 keV γ rays from a ^{137}Cs source measured with GYAGG:Ce,Mg and BGO are presented in Figure 5(a). The LY of GYAGG:Ce,Mg was determined by comparison with the BGO (LY = 8,600 ph/MeV) and considering the PMT quantum efficiency of both samples. The LY values are collected in Table 1. The measured pulse height spectrum of 5.5 MeV α particles from a ^{238}Pu source is also displayed in Figure 5(b), and an LY value of 4,600 ph/MeV is obtained under α excitation. Therefore, the LY ratio under excitation with α particles and γ rays (α/γ ratio) for this GYAGG:Ce,Mg ceramic is 0.21. The LY value of a similar composition $\text{Gd}_2\text{YAl}_2\text{Ga}_3\text{O}_{12}:\text{Ce}$ crystal with a thickness of 1 mm was measured in our previous study (Chewpraditkul *et al.*, 2018) is given for comparison. A lower LY value of the studied GYAGG:Ce,Mg ceramic can be due to its rather poor optical transmittance (35%) in the visible (RL) region, as shown in Figures 2 and 4. Non-radiative energy transfer from the $\text{Ce}^{3+} 5d_2$ excited state to the Ce^{4+} CT absorption band is partly responsible for the decrease of LY in this GYAGG:Ce,Mg ceramic. Its LY value is also lower than that of 40,000 ph/MeV measured for the Mg^{2+} -free GYAGG:Ce one with a thickness of 1 mm in Ref. (Chen *et al.*, 2019).

Figure 6 shows the scintillation decay curve of the GYAGG:Ce,Mg ceramic. The decay curve is well approximated by a double-exponential function: $I(t) = \sum A_i \exp(-t/\tau_i)$. A_i is an initial intensity of the τ_i decay component, and its relative intensity (I_i) is equal to $A_i\tau_i / \sum(A_i\tau_i)$. The decay components of GYAGG:Ce,Mg ceramic are given in Table 1. It's worth noting that the studied GYAGG:Ce0.5%,Mg ceramic with higher Ce

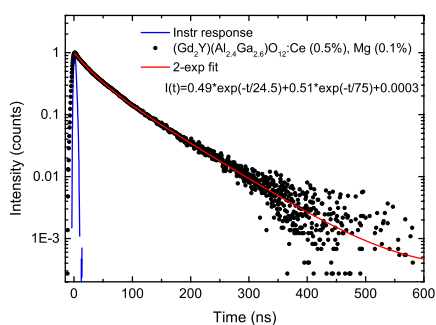


Figure 6. Scintillation decay curve of GYAGG:Ce,Mg ceramic

concentration exhibits faster scintillation response in comparison with the GYAGG:Ce0.07%,Mg ceramic in our previous study (Chen *et al.*, 2019). Moreover, the GYAGG:Ce,Mg ceramic exhibits a much faster scintillation response with respect to the Mg²⁺-free GYAGG:Ce samples. It can be caused by the presence of Ce⁴⁺ centers created by Mg²⁺ ions providing additional fast channels in the scintillation mechanism, mentioned.

Conclusions

The pure garnet phase of Gd₂YAl_{2.4}Ga_{2.6}O₁₂:Ce0.5%,Mg0.1% ceramic scintillator was fabricated by sintering under an oxygen atmosphere and HIP process. It exhibits a high integral scintillation efficiency of about 400% of BGO crystal and an LY value of 22,200 ph/MeV. The scintillation response of this GYAGG:Ce,Mg ceramic is much faster than the Mg-free GYAGG:Ce ceramic and crystal. An enhancement of LY value for this ceramic could be achieved by improving the fabrication technique to increase its transparency and together with an optimization of Mg and Ce concentration. Finally, the transparent GYAGG:Ce,Mg ceramic with high scintillation efficiency and fast response time could be useful for fast medical imaging and industrial applications.

Acknowledgment

This research work was financially supported by the National Research Council of Thailand (No. 164078). Partial support of the Department of Physics and Scientific Research Equipment Center of MCRU and the Department of Physics at KMUTT are gratefully acknowledged.

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