Dopant concentration dependence of radiation-induced positive hysteresis of Ce:GSO and Ce:GSOZ

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Abstract

Positive hysteresis and radiation tolerance to high-dose radiation exposure were investigated for Ce 0.5, 1, and 1.5%-doped Gd₂SiO₅ (GSO) and for Zr co-doped GSO with the same Ce concentrations (GSOZ). When they were irradiated by 200-800 Gy ⁶⁰Co in 200 Gy steps, all Ce-doped GSO samples exhibited light yield enhancement (positive hysteresis). On the other hand, the light yield of GSOZ decreased greatly. Ce 0.5%-doped GSO showed the highest positive hysteresis, with ~ 20% light yield enhancement. When the Ce concentration was increased, the positive hysteresis became weaker.

Keywords: Scintillation detector; Single crystal; Gamma-ray; Positive hysteresis; GSO; GSO:Ce, GSO:Ce Zr codoped

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1. Introduction

Scintillators, which convert high-energy ionizing radiation with typically keV-MeV energy to thousands of visible photons (Yanagida, 2013a), have been widely used as the main devices of solid state radiation detectors in many fields such as nuclear medicine (Yanagida et al., 2010), security (Totsuka et al., 2011), well-logging (Yanagida et al., 2013b), astrophysics (Kawaharada et al., 2004), and particle physics (Yamaoka et al., 2005). At present, some bright oxide scintillators such as Ce-doped Lu_2SiO_5 (Cutler et al., 2009), $(\text{Lu},\text{Y})_2\text{SiO}_5$ (Cutler et al., 2009), Gd_2SiO_5 (Takagi et al., 1983), YAlO_3 (Zorenko et al., 2007), LuAlO_3 (Wojtowicz 1998), $\text{Lu}_3\text{Al}_5\text{O}_{12}$ (Yanagida et al., 2011), $(\text{Lu},\text{Gd})_3(\text{Ga},\text{Al})_5\text{O}_{12}$ (Kamada et al., 2011), and $\text{Y}_3\text{Al}_5\text{O}_{12}$ (Takahashi et al., 2006) have been developed. Among them, Ce-doped Gd_2SiO_5 (Ce:GSO) exhibits interesting characteristics of positive hysteresis (Tanaka et al., 2002; Kawade et al., 2011). Positive hysteresis is a phenomenon in which the light yield of the scintillator is increased after exposure to ionizing radiation. In addition to Ce-doped GSO, Tl-doped CsI is also known to exhibit positive hysteresis (Grescovich et al., 1992).

Ce:GSO is mainly used for high-energy physics applications (Ito et al., 2007; Kokubun et al., 2004; Kawade et al., 2011) because it has relatively high stopping power for γ -rays, a medium level light yield (~ 10000 ph/MeV), medium energy resolution ($\sim 7\%$ at 662 keV), no intrinsic radio activity (unlike ^{176}Lu), and a fast decay time (30-80 ns). Furthermore, scintillation applications, dosimeter properties such as optically stimulated luminescence (Hazelton et al., 2010) and thermally stimulated luminescence (Cooke et al., 2001) have been reported for Ce:GSO. In Ce:GSO, Zr co-doping (GSOZ) was found to improve scintillation properties (Shimura et al., 2006). GSOZ showed a maximum light output approximately 20% larger than that of conventional Ce:GSO. This improvement can be explained by the fact that Ce^{4+} in the crystal may be changed to Ce^{3+} due to charge compensation caused by dopant Zr^{4+} in a Gd^{3+} site. This conversion decreases the light absorption in the crystal (Shimura et al., 2006). Although Ce:GSO is known to show positive hysteresis, Ce:GSOZ has not been

evaluated for this property. In addition, no investigation of the Ce concentration dependence of Ce:GSO positive hysteresis has been reported.

In the present work, we examine the role of Ce concentration on the positive hysteresis and radiation tolerance of Ce 0.5, 1, and 1.5%-doped GSO and GSOZ fabricated by Hitachi Chemical. The physical dimensions of each sample was the same, $5 \times 5 \times 5 \text{ mm}^3$.

2. Experimental procedures

First, basic scintillation properties such as X-ray-induced radioluminescence spectra and scintillation decay time profiles were evaluated at the Kyushu Institute of Technology. X-ray-induced emission spectra were measured using a DU420A-BU2 Charge Coupled Device (CCD) camera equipped with an SR163 monochromator, made by Andor Technology. The CCD was cooled to 193 K using a Peltier module. The excitation source was an X-ray generator supplied with 80 kV and 1 mA. The scintillation emission was fed to the monochromator through a 2 m optical fiber to avoid X-rays hitting the CCD directly. A detailed explanation of the radioluminescence setup was previously published (Yanagida et al., 2013c). The phosphorescence spectrum after ^{60}Co γ -ray exposure was investigated using the same CCD. Scintillation decay time profiles were evaluated using our original setup, a pulse X-ray streak camera system also described previously (Yanagida et al., 2010; Yanagida et al., 2013d). The mean X-ray energy of this instrument was $\sim 30 \text{ keV}$.

Because high-dose radiation exposure induces the degradation of optical transmittance by creating a lattice defect, optical transmittance was measured using a V670 spectrometer (JASCO) before irradiation. Photoluminescence (PL) quantum yield (QY) was also evaluated using a Hamamatsu Quantaaurus-QY. The absolute QY was calculated by the following equation, $QY = N_{\text{emit}}/N_{\text{absorb}}$, where N_{emit} and N_{absorb} are the numbers of emission and absorption photons, respectively. These properties were also evaluated after 800 Gy γ -ray

exposure. Because our laboratory and the irradiation facility were distant, these basic optical properties were evaluated one day after irradiation.

In the irradiation facility of Nagoya University, 800 km distant from the Kyushu Institute of Technology, ^{60}Co was used to irradiate samples in steps of 200 Gy. At each step, we evaluated the pulse height spectrum using a ^{137}Cs source. We mounted each crystal on the PMT (R7600-200, Hamamatsu) with silicon grease (OKEN 6262A). When γ -rays from ^{137}Cs were detected, the signals were fed into a preamplifier (ORTEC 113), a shaping-amplifier (ORTEC 572) with 2 μs shaping time, a multichannel analyzer (hereafter MCA, Kromek K102), and finally to a personal computer. We evaluated the pulse height spectra of six sample pieces after 0, 200, 400, 600, and 800 Gy irradiation. The time interval between the ^{60}Co exposure and pulse height spectrum measurement was typically a few minutes, the time required to walk from the irradiation room to the measurement room in the same building. All experiments were carried out at room temperature.

3. Experimental results and discussion

The X-ray-induced radioluminescence spectra of Ce:GSO and Ce:GSOZ are shown in Fig. 1. Emission peaks due to Ce^{3+} 5d-4f transition were clearly observed from 400 to 500 nm. In this integrated type evaluation, performed before exposure, Ce 0.5%-doped GSOZ exhibited the highest emission intensity. Fig. 2 illustrates the X-ray-induced scintillation decay time profiles. As the Ce concentration increased, the decay time became faster in both Ce:GSO and Ce:GSOZ scintillators. The main decay time components were in the time range of 55-80 ns. These scintillation properties were typical for Ce:GSO and Ce:GSOZ.

The measurements of optical transmittance before and after γ -ray irradiation are shown in Fig. 3. In these panels, before (0 Gy) and after (800 Gy) exposure are compared for each sample. In near-infrared wavelengths, no difference was observed, and the typical

transmittance was approximately 80%. In contrast, transmittance approximately 400 nm degraded after the exposure; this degradation was especially large in low Ce concentration samples. In Ce 1.5%-doped GSO and GSOZ, radiation damage was not observed to affect transmittance. It was confirmed that the transmittance of low Ce concentration samples degraded easily with irradiation. Consistent with the transmittance data, low Ce concentration samples were visibly brownish after irradiation. The degradation of the transmittance by approximately 400 nm is related to the oxygen vacancies (Kobayashi and Ishii, 1991) created by the γ -ray irradiation used in this study. Also supporting this interpretation, the experimental results showed that thermal annealing in an oxygen atmosphere restored the transmittance of Ce-doped LSO by approximately 400 nm (M. Kobayashi et al., 1993). The effect of radiation damage on transmittance was not as high as in other famous scintillators such as Ce:LaCl₃ (Pei et al., 2007) and Ce:LaBr₃ (Drozdowski et al., 2008). In addition, it must be noted that the positive hysteresis and light yield degradation of samples persisted after our return to the Kyushu Institute of Technology.

The absolute PL *QY* of all scintillators before and after 800 Gy exposure is presented in Fig. 4. Before irradiation, all samples showed 60-80% *QY*; these values are typical for commercially available inorganic luminescent materials. In heavily Ce-doped samples, *QY* became worse due to concentration quenching. When samples were irradiated with 800 Gy γ -rays, the *QY* of all scintillators decreased. Interestingly, the relative degradation of the *QY* of Ce:GSOZ was worse than that of Ce:GSO. In particular, Ce 1%-doped GSOZ exhibited the worst degradation following 800 Gy exposure. Conversely, Ce 1.5%-doped GSO and GSOZ were relatively resistant to the effects of exposure, in contrast to the low Ce concentration samples. These results were consistent with the transmittance measurements before and after 800 Gy exposure.

One day after the exposure, because our laboratory (Kyushu Institute of Technology) and the irradiation facility (Nagoya University) are not located close together, we evaluated the phosphorescence spectrum. Fig. 5 shows the phosphorescence spectrum of Ce 1%-doped GSOZ. The spectrum was weak but shaped similar to that of the X-ray-induced radioluminescence spectrum. In the other samples, we could not detect phosphorescence. Phosphorescence is a thermally stimulated luminescence around room temperature; therefore, the phosphorescence of the other samples had already decayed before we returned to the laboratory. Only the most damaged sample (the sample with the lowest QY) exhibited detectable phosphorescence one day after exposure.

Fig. 6 shows the ^{137}Cs γ -ray pulse height spectra of Ce 0.5%-doped GSO before and after irradiation with 800 Gy. As previously observed (Tanaka et al., 2002; Kawade et al., 2011), the light yield was increased by positive hysteresis. Consistent with our results, previous studies reported 20-30% enhancement of light yield in Ce 0.5%-doped GSO. Fig. 7 summarizes the observed photopeak positions of the 662 keV energy line of ^{137}Cs , depending on the irradiation dose and Ce concentrations. Although all Ce:GSOZ samples showed degradation of the light yield, the Ce:GSO samples exhibited positive hysteresis. Positive hysteresis weakened with increasing Ce concentration in Ce:GSO. Ce 1%-doped GSOZ had the worst radiation tolerance; this was consistent with the result that this sample alone exhibited detectable phosphorescence one day after irradiation (Fig. 5). Generally, phosphorescence is caused by radiation-induced defects, and the creation of defects causes the degradation of luminescence properties.

The energy resolution at 662 keV is summarized in Fig. 8. Though all samples showed a good energy resolution of $\sim 8\%$ before irradiation, the energy resolution was degraded to 10-14% after irradiation. Similar to the light yield degradation, the energy resolution degradation

was small in Ce singly doped GSO scintillators. In spite of the light yield enhancement, the energy resolution of the Ce:GSOs decreased.

In scintillators, there is an empirical relation between the number of generated electron-hole pairs n_{e-h} and the band gap E_g , which may be expressed as $n_{e-h} = BE_\gamma/\beta E_g$, where E_γ is the energy deposited by γ -quanta (Dorenbos, 2000). The factor B relates the phonon energy loss during the ionization and is expressed as $B = 1/(1 + 0.65K)$, where K is defined as the ratio of energy lost by optical phonons to the energy lost for an ionization event (Mikhailik and Kraus, 2011). Thus, the light yield (LY) is simply expressed as $LY = 10^6 SQB/\beta E_g$ [ph/MeV], where S is the energy transport efficiency from the host to the emission center, Q is the luminescence quantum efficiency at the emission center, and β is an empirical constant close to 2.5 (Dorenbos, 2000) in famous (bright) scintillators and is theoretically 2.3 (Robbins, 1980; Lempicki et al., 1993). In the above formula, Q is equal to QY in the present work. Based on the experimental results, which showed that the scintillation light yield increased in spite of the degradation of QY ($= Q$), we concluded that the remaining parameters, the product of energy migration efficiency from the host to emission centers and optical phonon energy loss, BS , were enhanced in Ce:GSO by γ -ray irradiation. Though the enhancement of defects due to ^{60}Co γ -ray irradiation would affect parameter B , the effect is considered to be low based on a generally accepted notion of basic solid state physics. Thus, the energy transport efficiency S is the dominant factor in positive hysteresis. From this result, S , which is mainly related to the energy dissipation processes of δ -rays, is shown to be key to the design of more efficient scintillators. S can be tuned by the introduction of some types of defects. High-dose γ -ray irradiation may create some defect-related bands, and such intermediate bands would enhance the energy transport efficiency. To examine this hypothesis, Synchrotron measurements immediately after the high dose irradiation would be ideal.

4 Conclusion

Ce 0.5, 1, and 1.5%-doped GSO and GSOZ crystal scintillators were irradiated by 200-800 Gy ^{60}Co γ -rays. Although Ce:GSOZ was degraded by irradiation, the light yield of Ce:GSO was enhanced by positive hysteresis. With regards to transmittance, GSO and GSOZ highly doped by Ce are better. Ce:GSO has better light yield than Ce:GSOZ in high-dose applications.

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Figure Captions

Figure 1 X-ray-induced radioluminescence spectra of Ce:GSO and Ce:GSOZ.

Figure 2 Scintillation decay time profiles of Ce:GSO and Ce:GSOZ.

Figure 3 Transmittance of Ce:GSO and Ce:GSOZ samples before (black) and after (red) 800 Gy ^{60}Co γ -ray irradiation.

Figure 4 PL QY before (circle) and after (square) 800 Gy exposure.

Figure 5 Phosphorescence spectrum of Ce 1%-doped GSOZ.

Figure 6 ^{137}Cs γ -ray pulse height spectra of Ce 0.5%-doped GSO before and after 800 Gy irradiation.

Figure 7 Photopeak position of 662 keV energy line of ^{137}Cs plotted against dose in the range 0-800 Gy.

Figure 8 Energy resolution of 662 keV energy line of ^{137}Cs plotted against dose in the range 0-800 Gy.

Figure3

Figure5

Figure6

Figure7

Figure8