

# Photoluminescence, Scintillation, and Thermally Stimulated Luminescence Properties of Eu-doped Al<sub>2</sub>O<sub>3</sub> Single Crystals

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The photoluminescence (PL), scintillation, and thermally stimulated luminescence (TSL) properties of Eu-doped (0.01, 0.1, and 1%) Al<sub>2</sub>O<sub>3</sub> single crystals grown by the floating zone method were investigated systematically. The PL of the samples showed several sharp emission peaks across 550–750 nm originating from the 4f–4f transitions of Eu<sup>3+</sup> and the 3d–3d transitions of Cr<sup>3+</sup> impurity ions. The scintillation spectrum of the samples showed a broad emission peak at 320 nm due to the F<sup>+</sup> center and an emission peak at 700 nm due to the 3d–3d transitions of Cr<sup>3+</sup> impurity ions. All the samples showed TSL glow peaks at around 200, 320, and 375 °C. The TSL intensity of the 1% Eu-doped sample was the highest among the present three samples, and the TSL response was proportional to the irradiated X-ray dose in the range from 10 to 1000 mGy. The Eu-doped Al<sub>2</sub>O<sub>3</sub> single crystal could be a novel candidate for personal dose monitoring applications.

## 1. Introduction

Radiation-induced phosphors can be classified into two types: scintillators, in which carriers are excited by ionizing radiation irradiation at the luminescent center and immediately emit light, and phosphor-based dosimeters, in which captured carriers are re-excited by external stimulation such as heat or light, leading to emission.<sup>(1,2)</sup> Scintillators are applied in medical imaging equipment such as X-ray computed tomography (CT) and positron emission tomography (PET), security equipment such as baggage screening devices, and environmental monitoring.<sup>(1,3,4)</sup> In contrast, phosphor-based dosimeters are mainly applied in personal dose monitoring.<sup>(5–7)</sup> The luminescence types of phosphor-based dosimeters are classified as follows: thermally stimulated luminescence (TSL), in which carriers are re-excited by thermal stimulation; optically stimulated luminescence (OSL), in which carriers are re-excited by light;

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and radio-photoluminescence (RPL), in which the interaction with ionizing radiation creates new emission centers in the material.<sup>(8–12)</sup>

So far, LiF single crystals doped with Ti and Mg<sup>(13)</sup> and Cu-doped LiB<sub>4</sub>O<sub>7</sub> ceramics<sup>(14)</sup> for TSL, BeO ceramics<sup>(15)</sup> for OSL, and Ag-doped phosphate glass<sup>(16,17)</sup> for RPL have been studied. In particular, personal dosimeters should have an effective atomic number ( $Z_{eff}$ ) close to that of human soft tissue ( $Z_{eff} = 7.51$ ) from the viewpoint of bioequivalence, in addition to high sensitivity dose–response characteristics and low fading.<sup>(18)</sup> To develop novel phosphor-based dosimeters, the TSL, OSL, and RPL properties of many new materials have been investigated.<sup>(19–29)</sup> In particular, Al<sub>2</sub>O<sub>3</sub> has high thermal stability, chemical stability, and low  $Z_{eff}$ ; thus, C-doped Al<sub>2</sub>O<sub>3</sub> is commercially developed as TSL and OSL personal dosimeters.<sup>(7,30)</sup>

In recent years, transparent ceramics, which have more defects than single crystals, have been attracting attention in phosphor-based dosimeter applications.<sup>(31–34)</sup> Moreover, the TSL and OSL properties of Al<sub>2</sub>O<sub>3</sub> transparent ceramics doped with C, Mg, Ti, Ce, and Eu have also been studied.<sup>(35–38)</sup> In contrast, there are few reports on Al<sub>2</sub>O<sub>3</sub> single crystals doped with impurities other than C, leaving room for further research.

Single crystals are expected to have a higher detection efficiency than opaque ceramics because of their high transmittance and the ease of extracting internally generated light to the outside. In addition, the mass production of large-area transparent ceramics is difficult with current technology. In contrast, single crystals are suitable for mass production because large-area single crystals can be fabricated by the pulling method such as the Czochralski method. Therefore, if dosimeters that emit light with high efficiency in single-crystal form are discovered, they could be one of the best candidates for new dosimeter materials compared with opaque and transparent ceramics. Recently, MgAl<sub>2</sub>O<sub>4</sub> and Mg<sub>2</sub>SiO<sub>4</sub> single crystals doped with some dopants have been studied as candidates for new dosimeter materials, realizing defects produced in single-crystal form with high transmittance and high TSL and OSL intensities.<sup>(39–44)</sup> In this study, Eu<sup>3+</sup> is selected as a luminescence center. Eu<sup>3+</sup> ions are often used as luminescence centers because they have intense red emission due to the 4f–4f transition.<sup>(45)</sup> To date, the PL, scintillation, and TSL properties of Eu-doped Al<sub>2</sub>O<sub>3</sub> transparent ceramics have been reported.<sup>(38,46)</sup> In Al<sub>2</sub>O<sub>3</sub>, it is considered necessary to evaluate dosimetric properties not only in the transparent ceramic form but also in the single-crystal form. For the above reasons, Eu-doped Al<sub>2</sub>O<sub>3</sub> single crystals exhibit intense luminescence and good TSL properties.

In this study, Eu-doped Al<sub>2</sub>O<sub>3</sub> single crystals were synthesized by the floating zone (FZ) method, and in addition to their PL and scintillation properties, their TSL response properties as a dosimeter were also investigated.

## 2. Materials and Methods

Eu-doped Al<sub>2</sub>O<sub>3</sub> single crystals were synthesized using an FZ furnace equipped with four xenon lamps. Al<sub>2</sub>O<sub>3</sub> (99.99%) and Eu<sub>2</sub>O<sub>3</sub> (99.99%) were used as starting materials and were mixed uniformly using a mortar and pestle. The nominal doped concentrations of Eu were 0.01, 0.1, and 1%. The X-ray powder diffraction (XRD) patterns were measured in the range of 20–80° (Rigaku, UltimaIV). The PL emission was measured using a CCD-based spectrometer

(Otsuka Electronics, MCPD-9800-2285C). The excitation source was a xenon lamp with a monochromator, and the excitation light was led to the sample through an optical fiber. The X-ray-induced scintillation spectra were measured using laboratory-made setups.<sup>(47)</sup> The applied voltage and tube current during the measurement of scintillation spectra were 40 kV and 1.2 mA, respectively. The TSL glow curves were measured using a TSL reader (TL-2000, Nanogray) after X-ray irradiation. No background processing was conducted for the blackbody radiation signal.

### 3. Results and Discussion

Figure 1 shows a photograph of the Eu-doped  $\text{Al}_2\text{O}_3$  single crystals. The samples were approximately 1 mm thick and colorless. The following measurements were conducted using these samples. The actual Eu concentration inside the crystal was considered to be lower than the nominal concentration. The weights of the 0.01, 0.1, and 1% Eu-doped  $\text{Al}_2\text{O}_3$  single crystals were 0.0592, 0.0211, and 0.0204 g, respectively.

Figure 2 shows the XRD patterns of the Eu-doped  $\text{Al}_2\text{O}_3$  single crystals. The remaining crushed samples were used for XRD measurements. The XRD patterns of the samples matched the reference pattern for  $\text{Al}_2\text{O}_3$  (ICSD. No. 31545). Therefore, the samples are considered to be single-phase  $\text{Al}_2\text{O}_3$ . The coincidence of the valence and coordination numbers of  $\text{Al}^{3+}$  and  $\text{Eu}^{3+}$  suggested that  $\text{Eu}^{3+}$  ions were substituted at the  $\text{Al}^{3+}$  site.

Figure 3 shows the PL excitation and emission spectra of the Eu-doped  $\text{Al}_2\text{O}_3$  single crystals. The Eu-doped samples showed emission peaks at 587, 616, 678, and 714 nm. These emission peaks are consistent with those attributed to the 4f–4f transition of  $\text{Eu}^{3+}$ .<sup>(38,48,49)</sup> The sharp emission peak at 693 nm is considered to be due to the 3d–3d transition of  $\text{Cr}^{3+}$ .<sup>(37,46)</sup> The intensity of the emission peak due to the 4f–4f transition of  $\text{Eu}^{3+}$  weakened as the Eu doping concentration increased. This is the same trend reported previously for Eu-doped  $\text{Al}_2\text{O}_3$

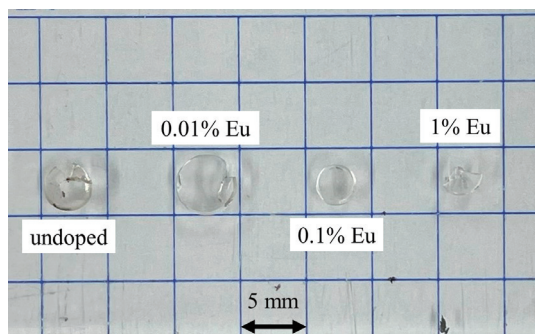


Fig. 1. (Color online) Photograph of Eu-doped  $\text{Al}_2\text{O}_3$  single crystals.

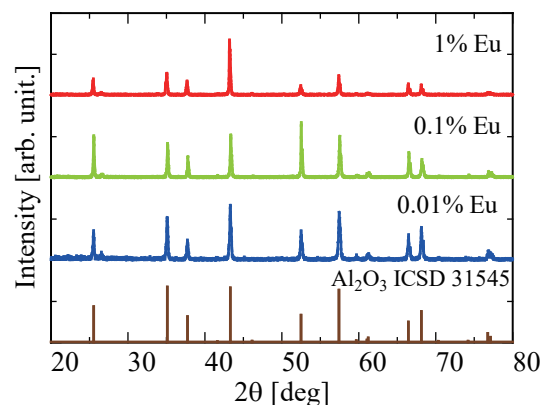


Fig. 2. (Color online) XRD patterns of Eu-doped  $\text{Al}_2\text{O}_3$  single crystals.

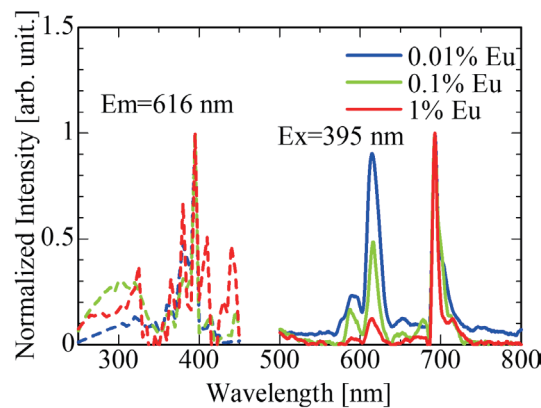


Fig. 3. (Color online) PL emission and excitation spectra of Eu-doped  $\text{Al}_2\text{O}_3$  single crystals.

transparent ceramics. Since the samples in this study were synthesized in air ambient, emission peaks originating from the  $4f-4f$  transition of  $\text{Eu}^{3+}$  were observed while no emission bands originating from the  $5d-4f$  transition of  $\text{Eu}^{2+}$  were observed. Excitation spectra showed sharp peaks at 365, 380, 395, and 415 nm. These peaks are attributed to the  $4f-4f$  transition of  $\text{Eu}^{3+}$ .<sup>(38,48,49)</sup> The broad peak at about 300 nm is attributed to charge transfer transitions between  $\text{Eu}^{3+}$  and  $\text{O}^{2-}$ .

Figure 4 shows X-ray-induced scintillation spectra of Eu-doped  $\text{Al}_2\text{O}_3$  single crystals. A broad scintillation peak was observed at  $\sim 320$  nm. On the basis of previous studies, the scintillation peak at 320 nm is attributed to the  $\text{F}^+$  center.<sup>(35,46,50)</sup> As with PL, the emission near 700 nm is considered to be a peak due to the  $3d-3d$  transition of  $\text{Cr}^{3+}$ . No emission due to the  $4f-4f$  transition of  $\text{Eu}^{3+}$ , which was observed in the PL spectra, was observed. PL directly excites carriers to the luminescent center, and recombination leads to luminescence. In scintillation, carriers are ionized, transferred to the luminescent center via energy transport, and recombined to emit light. The luminescence mechanism differs between PL and scintillation, and in scintillation, the carriers do not always recombine at the desired emission center. Therefore, in Eu-doped  $\text{Al}_2\text{O}_3$  single crystals, the luminescence was attributed to  $\text{Cr}^{3+}$  rather than  $\text{Eu}^{3+}$  ions.

Figure 5 shows the TSL glow curves of Eu-doped  $\text{Al}_2\text{O}_3$  single crystals after 1000 mGy X-ray irradiation. The TSL intensity was normalized by sample weight. All the samples showed TSL glow peaks at  $\sim 200$ ,  $\sim 320$ , and  $\sim 375$  °C. The 1% Eu-doped sample showed the highest TSL intensity among the present samples. Since the ionic radius of  $\text{Eu}^{3+}$  (0.947 Å, 6-coordination) is larger than that of  $\text{Al}^{3+}$  (0.535 Å, 6-coordination), strain is generated, and defects are formed when  $\text{Eu}^{3+}$  ions are substituted at the  $\text{Al}^{3+}$  sites. Therefore, 1% Eu-doped  $\text{Al}_2\text{O}_3$  single crystals have the strongest TSL intensity because the generated number of defects is greater than those of the other samples. Figure 6 shows the fitted curves of the TSL glow curve of the 1% Eu-doped  $\text{Al}_2\text{O}_3$  single crystal. The TSL glow curve was fitted with a general order kinetics in the formula shown below.<sup>(51)</sup> The obtained parameters are listed in Table 1. The 1% Eu-doped sample showed peaks at 150, 187, 221, 320, and 380 °C. The shapes of the TSL glow curves between Eu-doped  $\text{Al}_2\text{O}_3$  single crystals and those in transparent ceramics are different. In a previous study, Eu-

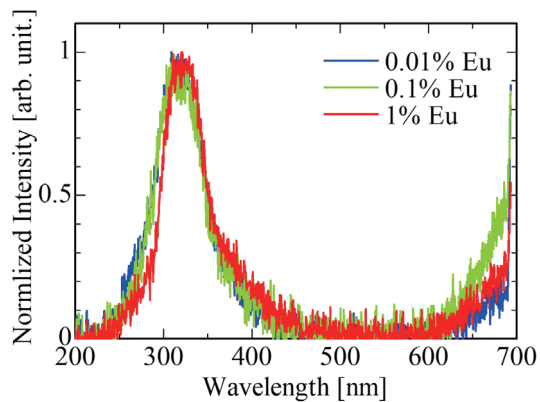


Fig. 4. (Color online) X-ray-induced scintillation spectra of Eu-doped  $\text{Al}_2\text{O}_3$  single crystals.

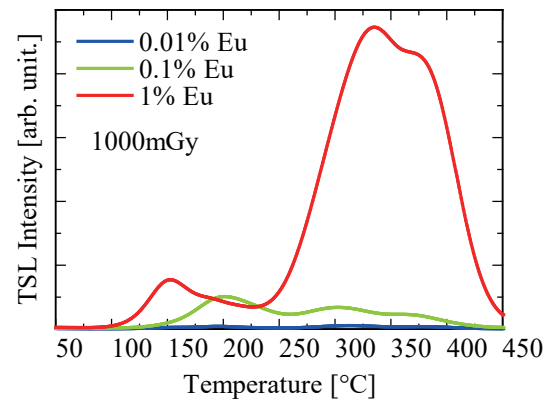


Fig. 5. (Color online) TSL glow curves of Eu-doped  $\text{Al}_2\text{O}_3$  single crystals.

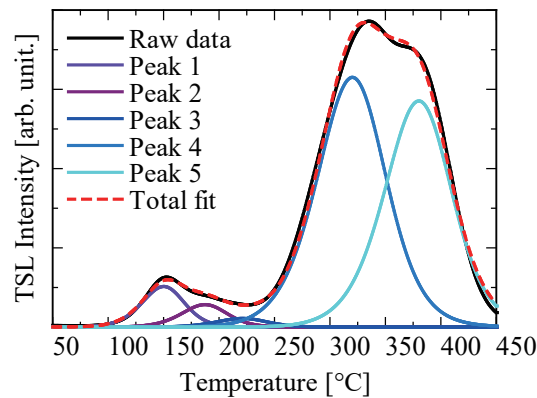


Fig. 6. (Color online) Fitted curves of TSL glow curve of 1% Eu-doped  $\text{Al}_2\text{O}_3$  single crystal.

Table 1

Parameters of fitted glow curves using 1% Eu-doped  $\text{Al}_2\text{O}_3$  single crystal.

	Peak intensity (arb. unit)	Peak temperature (°C)	Trap depth (eV)	Frequency factor ( $\text{s}^{-1}$ )
Peak 1	33	150	1.02	$1.48 \times 10^{11}$
Peak 2	18	187	1.11	$1.38 \times 10^{11}$
1% Eu Peak 3	7	221	1.14	$3.66 \times 10^{10}$
Peak 4	201	320	1.21	$1.21 \times 10^9$
Peak 5	182	380	1.51	$2.93 \times 10^{10}$

doped  $\text{Al}_2\text{O}_3$  transparent ceramics showed the TSL glow peak at  $\sim 100$  °C.<sup>(46)</sup> In contrast, the present Eu-doped  $\text{Al}_2\text{O}_3$  single crystals showed no TSL glow peak at  $\sim 100$  °C. The defects generated depend on the fabrication method. Typically, single crystals have fewer defects than transparent ceramics.

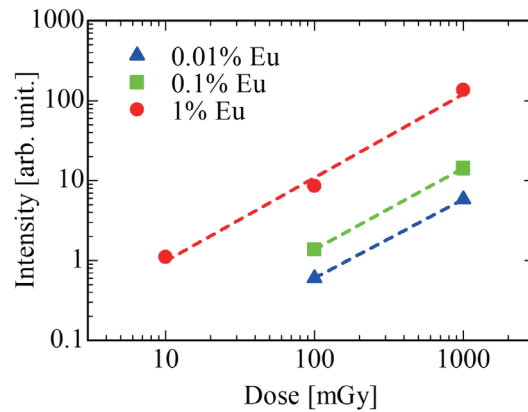


Fig. 7. (Color online) TSL dose response functions of Eu-doped  $\text{Al}_2\text{O}_3$  single crystals.

$$I(T) = I_M b^{\frac{b}{b-1}} \exp\left(\frac{E}{kT} \frac{T - T_M}{T_M}\right) \left[ 1 + (b-1) \frac{2kT_M}{E} + (b-1) \left( 1 - \frac{2kT}{E} \right) \left( \frac{T^2}{T_M^2} \exp\left(\frac{E}{kT} \frac{T - T_M}{T_M}\right) \right) \right]^{\frac{-b}{b-1}} \quad (1)$$

Figure 7 shows the TSL dose response functions of Eu-doped  $\text{Al}_2\text{O}_3$  single crystals. The TSL intensity at each dose represents the maximum intensity at 300 °C. The TSL intensity increased linearly with irradiation dose in the range of 10–1000 mGy for the 1% Eu-doped sample and in the range of 100–1000 mGy for the 0.01 and 0.1% Eu-doped samples. In the previous study of Eu-doped  $\text{Al}_2\text{O}_3$  transparent ceramics, the TSL intensity decreased with increasing Eu doping concentration.<sup>(46)</sup> In contrast, Eu-doped  $\text{Al}_2\text{O}_3$  single crystals showed a trend toward higher TSL intensity with increasing Eu doping concentration. The number of defects may increase with increasing Eu doping concentration, and the TSL intensity depends on the number of defects. Thus, the highly Eu-doped  $\text{Al}_2\text{O}_3$  single crystals exhibited strong TSL intensity. The lower sensitivity limit of the Eu-doped  $\text{Al}_2\text{O}_3$  single crystal was 10 mGy, which is higher than the detection limit of 10–100  $\mu\text{Gy}$  in the personal dose monitoring application. In future works, we would like to improve the TSL intensity by growing  $\text{Al}_2\text{O}_3$  doped with different luminescent centers and annealing in a reducing atmosphere.

#### 4. Conclusions

Eu-doped (0.01, 0.1, and 1%)  $\text{Al}_2\text{O}_3$  single crystals were successfully grown by the FZ method. In terms of PL, the samples showed several sharp emission peaks across 550–750 nm originating from the 4f–4f transitions of  $\text{Eu}^{3+}$ . In terms of scintillation, the samples showed a broad emission peak at 320 nm due to the  $\text{F}^+$  center. Furthermore, the Eu-doped  $\text{Al}_2\text{O}_3$  single crystals showed PL and scintillation due to not only  $\text{Eu}^{3+}$  but also  $\text{Cr}^{3+}$  impurity ions. In terms of TSL, all the samples showed TSL glow peaks at  $\sim 200$ , 320, and 375 °C, and the TSL intensity of the 1% Eu-doped sample was the highest among the present samples. The absence of the TSL peak at  $\sim 100$  °C is suggested to be advantageous for applications such as personal dose monitoring. The TSL response was confirmed to be linearly related to the irradiated X-ray dose

in the range from 10 to 1000 mGy. Therefore, the Eu-doped Al<sub>2</sub>O<sub>3</sub> single crystal can be a novel candidate for personal dose monitoring applications.

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