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Original research article

Comparative study of dosimeter properties of Eu-doped CsBr transparent ceramic and single crystal

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ABSTRACT

We have synthesized Eu-doped CsBr transparent ceramics by spark plasma sintering (SPS) and a single crystal by the vertical Bridgman-Stockbarger method. Subsequently, we have investigated their optical, scintillation and dosimeter properties. In scintillation, both the materials showed a broad emission peaking around 440 nm, and the origin was due to $4f^65d^1$-$4f^7$ transitions of Eu^{2+}. In the optically-stimulated luminescence (OSL) properties, OSL intensity of the ceramic sample was higher than those of the crystal sample. The dosimetric sensitivities were confirmed as low as 0.01 mGy using TSL while 0.1 mGy using OSL for both of the ceramic and single crystal samples.

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1. Introduction

Luminescence materials are often used for ionizing radiation detectors, which are mainly classified into two types. One is scintillators that have a function to convert absorbed energy of ionizing radiation such as X- and γ-rays into low energy photons immediately. Scintillators have been used in various fields, for examples in medicine [1], security [2] and high energy physics [3]. On the other hand, dosimeters with storage phosphors have a function to record the radiation dose. The absorbed energy is stored in carrier trapping centers. The trapped charges can be released by stimulation of heat or light to emit photons. The resultant emission by the stimulation of heat and light are called thermally-stimulated luminescence (TSL) and optically-stimulated luminescence (OSL), respectively. Dosimeters have been utilized in individual radiation monitoring devices [4] and imaging plates (IPs) [5,6].

So far, phosphors utilizing OSL have been proposed for IPs to achieve high resolution in digital radiography [7]. The OSL materials are required to have efficient X-ray absorption, short lifetime (~10 μs), high luminescence output and linear dose dependence [8]. Since the late 1990s, Eu-doped CsBr has attracted much attention as IPs [9–13] because the Eu-doped CsBr has outstanding properties mentioned above. In the CsBr structure, Eu^{2+} replaces Cs^+ ion, and Eu^{2+}-V_{Cs} isolated dipole centers (IDC) are formed in order to compensate the charge imbalance. The Eu^{2+} in the IDC shows an emission peak around 440 nm [14]. The emission can be observed as OSL under He-Ne laser (633 nm) stimulation in practice. Thus, luminescence properties of the Eu-doped CsBr have been studied intensively. However, almost all of the studies were done in a form of bulk single crystal [15], bulk opaque ceramics [16] and film by a vacuum deposition technique [17]. There are only a few reports available on luminescence properties of Eu-doped CsBr transparent ceramics [18]. We have previously reported that

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some X-ray induced luminescence properties are improved in transparent ceramics compared with those of single crystals in some common phosphor materials [19–21]. In particular, dosimeter properties of transparent ceramics synthesized by spark plasma sintering (SPS) are enhanced [22–24] since the SPS was performed in a highly reductive environment, which effectively generates defect centers.

In this paper, we have synthesized Eu-doped CsBr transparent ceramics using spark plasma sintering (SPS) and examined the optical, and dosimeter properties, in comparison with Eu-doped CsBr single crystal prepared by the vertical Bridgman-Stockbarger method. In addition to these properties, we have also investigated the scintillation properties since scintillation and dosimeter properties are complementarily related in some materials [25–27]. So, investigations of both of the properties are important to understand the luminescence phenomena induced by ionizing radiations comprehensively.

2. Experiment

Eu-doped CsBr transparent ceramic was synthesized by the SPS technique using Sinter Land LabX-100 in a vacuum. Raw powder of CsBr (>99.99%, Furutachi Chemical) and $EuCl_3 \cdot 6H_2O$ (>99.9%, Furutachi Chemical) were homogeneously mixed to a molar ratio of 0.994: 0.006. The total mass of the mixture was 1.0 g. The mixture was introduced into a cylindrical graphite die, in which the mixture was held between two graphite punches. During the sintering, the temperature was increased from 20 °C to 450 °C at a rate of 45 °C/min and held for 10 min while applying the pressure of 6 MPa. The temperature was measured using a K-thermocouple attached on the graphite die. After the synthesis, the wide surfaces of the ceramic samples were polished by hand using a sandpaper (3000 grit). On the other hand, Eu-doped CsBr single crystal was grown using the vertical Bridgman-Stockbarger method. The starting compounds were the same as that for the transparent ceramics. The mixture powder was first dried by heating at ∼150 °C for 1 h and ∼536 °C for 2 h in a vacuum. Subsequently, the dried mixture powder was enclosed in a vacuum-sealed quartz ampule, and the sealed ampule was set in a Bridgman furnace (VFK-1800, Crystal Systems Corp.). During the crystal growth, the heater was set to 686 °C, and the ampule was translated downwards at a rate of 1 cm/h. The obtained crystal rod was cut into a rectangle form and then mechanically polished the surfaces by a sandpaper (3000 grit). The sizes of the single crystal and ceramic samples were comparable. The following measurements were carried out by the same manner for each sample.

The in-line transmittance spectra were evaluated by using a spectrometer (V670, JASCO) in the spectral range from 200 to 2700 nm with 1 nm intervals. The PL emission and excitation spectra as well as PL quantum yields were measured using a Quantaurus-QY (C11347, Hamamatsu Photonics). The PL decay curves were evaluated with a Hamamatsu Quantaurus-τ (C11367-04, Hamamatsu). To investigate scintillation properties, X-ray induced scintillation spectra were measured by using our original setup. The radiation source was a conventional X-ray tube operated with a tube voltage and current of 40 kV and 1.2 mA, respectively. The scintillation photons were collected and then guided to a spectrometer (Andor DU-420-BU2 CCD and Shamrock 163 monochromator). The detail of the setup is described elsewhere [28]. Moreover, X-ray induced scintillation decay and afterglow profiles were evaluated by using an afterglow characterization system equipped with a pulse X-ray tube [29]. TSL glow curves were measured using a TSL reader (TL-2000, Nanogray Inc.) [30]. The heating rate was 1 °C/s, and the measurement temperature range was from 50 to 350 °C. To obtain a dose response function, TSL glow curves were measured with different irradiation doses (0.01–3 mGy). The response signal was defined here as an integrated signal from 50 to 350 °C. To measure TSL spectra, the samples were irradiated with X-rays (∼10 Gy) and then heated at a specific temperature on a ceramic heater system (SCR-SHQ-A, Sakaguchi) while measuring the signal using a spectrometer (QEPro, Ocean Optics). OSL spectra were measured after 1 mGy X-ray irradiation by using a spectrofluorometer (FP-8600, JASCO). The stimulation wavelength was 630 nm. In addition, the sample was irradiated by X-rays of a certain dose ranging from 0.01 mGy to 10 mGy and the dose response curves were obtained.

3. Results and discussion

3.1. Sample

Fig. 1 shows a photograph of Eu-doped CsBr single crystal and ceramic samples. The thicknesses were 1.03 and 1.06 mm, respectively. It was confirmed that the stripe patterns on the back of the samples were clearly seen through the samples. Although the crystal sample was colorless, the ceramic sample looked brown. The reason is possibly due to a generation of color centers or the difference of the actual Eu concentrations. In-line transmittance spectra of the samples are indicated in Fig. 2. The transmittance of the ceramic sample showed strong absorption at wavelengths shorter than 400 nm. The origin would be typical absorption due to the $4f^7$-$4f^6 5d^1$ transitions of Eu^{2+} [31]. However, the single crystal sample did not show such strong absorption in the same range in spite of the same nominal Eu concentration.

3.2. Optical properties

Fig. 3 shows PL excitation/emission contour graphs of Eu-doped CsBr transparent ceramic and single crystal samples. The ceramic sample showed a broad emission band peaking around 440 nm under an excitation around 350 nm. The emission origin was the $4f^6 5d^1$-$4f^7$ transitions of Eu^{2+} attributed by the former work [15]. In contract, the single crystal sample presented a broad emission band peaking around 370 nm and sharp emissions peaking at 630 and 700 nm under an excitation

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