

## Scintillation properties of In-doped NaI transparent ceramics

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### ABSTRACT

In-doped NaI transparent ceramic scintillators were synthesized by the spark plasma sintering method, and In-doped NaI single crystal was grown by the Bridgman–Stockbarger method. The diffuse transmittance at the emission wavelength was ~70% for the transparent ceramics and ~90% for the single crystal. In the scintillation spectra under X-ray irradiation, emission peaks were observed at around 320, 460, and 510 nm in both of the transparent ceramic and single crystal samples. The peaks at 460 and 510 nm are due to  $^3P_1 \rightarrow ^1S_0$  transitions of In<sup>+</sup>. The decay time constants of the transparent ceramics and the single crystal were 1.8  $\mu$ s. The X-ray induced afterglow level of the 0.1% In-doped NaI transparent ceramic and the single crystal was 170 and 777 ppm, respectively. In pulse height analysis, 662 keV photoabsorption peaks were clearly observed in all the samples when irradiated with  $^{137}\text{Cs}$   $\gamma$ -rays. The light yield of the 0.1% In-doped NaI transparent ceramic was 19,000 ph/MeV, and that of the 0.1% In-doped NaI single crystal was 12,000 ph/MeV. The transparent ceramics showed better scintillation properties than the single crystal.

### 1. Introduction

Scintillators are a type of phosphor that rapidly converts a single ionizing radiation quantum such as X- and  $\gamma$ -rays into several thousands to tens of thousands of visible photons via a large scale of quantum cutting. To meet the growing demand for ionizing radiation detectors, various scintillators with different chemical compositions, including oxides, sulphides, and halides, have been developed. In terms of material forms, single crystals have been widely employed as scintillators due to their high transparency, which is crucial for efficiently delivering scintillation photons to a photodetector without a significant loss. However, recent advances in sintering technology have made it possible to synthesize ceramics with exceptionally high transparency, and successful examples of transparent ceramics have been found in laser field [1]. This technology was imported to scintillator field in late 1990's, and this breakthrough has opened up new possibilities for their application in scintillators [2].

Transparent ceramic scintillators have numerous advantages over conventional single crystal scintillators, such as low synthesis temperature which connects to low fabrication cost, high degree of freedom in composition which can sometimes avoid the phase transition problem of single crystals, and reduction of lattice defects that adversely affect luminescence properties. In the field of transparent ceramic scintillators,

extensive research has been conducted on garnet oxides [3–6], pyrochlore [7], spinel oxides [8], sesquioxide [9,10], Lu<sub>2</sub>SiO<sub>5</sub> [11], ZnO [12], Bi<sub>3</sub>Ge<sub>4</sub>O<sub>12</sub> [13], BaF<sub>2</sub> [14,15], and CaF<sub>2</sub> [16]. Among them, some transparent ceramic scintillators such as Ce-doped Gd<sub>3</sub>Al<sub>2</sub>Ga<sub>3</sub>O<sub>12</sub> [17], Pr-doped Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> [18], and Ce-doped Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> [3] have exhibited superior scintillation properties compared to single crystals. Consequently, transparent ceramic scintillators have the potential to exceed the performance of their single crystal counterparts.

In this study, we fabricated NaI doped with indium (In) as a new transparent ceramic scintillator by the Spark Plasma Sintering (SPS) method and evaluated their optical and scintillation properties. In-doped NaI single crystals were also prepared by the Bridgman–Stockbarger method and compared with transparent ceramics. Tl-doped NaI single crystals are relatively inexpensive and have a high light yield (LY: ~38,000 ph/MeV) [19], and many studies have been conducted to investigate their properties [20–25]. However, there is limited research on dopants other than Tl<sup>+</sup>. Therefore, we focused on In<sup>+</sup> which has a similar electron configuration and ionic radius to Tl<sup>+</sup>, as an alternative dopant of Tl<sup>+</sup>. Both Tl and In are classified as ns<sup>2</sup>-type luminescent centers, and their emission is based on spin-allowed transitions ( $^3P_1 \rightarrow ^1S_0$ ) [26]. Therefore, we expected that In-doped NaI would exhibit high light yield as same as Tl-doped NaI. In fact, some have reported that the LY of In-doped CsI single crystals reached 34,700 ph/MeV [27]. In

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addition to In-doped CsI single crystals, there have been several reports on photoluminescence (PL) and scintillation based on  $^3P_1 \rightarrow ^1S_0$  transition of  $In^+$  in In-doped KBr, KCl, RbBr, and CsCl [28–30]. To our knowledge, there are no studies on the PL and scintillation properties of In-doped NaI transparent ceramics and single crystals, and there remains a room for investigation.

## 2. Experimental procedure

### 2.1. Sample preparations

In-doped NaI transparent ceramics with various In concentrations (0.05, 0.1, 0.2, and 0.5 mol%) were prepared by the SPS method. The raw powders were NaI (99.999%, Sigma-Aldrich) and InI<sub>3</sub> (99.99%, Kojundo Chemical). These powders were mixed in a glove box with a humidity of less than 20% in N<sub>2</sub> atmosphere. The mixed powders were then dried at 400 °C for 3 h using a small electric furnace under vacuum, then removed from the furnace and sintered by an SPS furnace (LABOX-110, Sinterland). The sintering was carried out under a vacuum of less than 10 Pa, and the sintering procedure is outlined below. Initially, the temperature was raised at a rate of 10 °C/min until reaching the target temperature of 450 °C. Simultaneously, the pressure was increased at a rate of 1 MPa/min until reaching the target pressure of 45 MPa. Subsequently, the temperature and pressure were held at 450 °C and 45 MPa for 15 min, respectively.

On the other hand, for the comparison, the 0.1% In-doped NaI single crystal was grown by the conventional vertical Bridgman–Stockbarger method using the above same raw powders. The single crystal was prepared in the same way in the previous experimental procedures [31]. Nominal concentration of 0.1% In was selected by the best values from previous studies of Tl-doped NaI and In-doped CsI [32,33]. Afterward, the surfaces of the transparent ceramic and single crystal samples were polished using 3,000 grit sandpaper.

### 2.2. Optical and scintillation properties

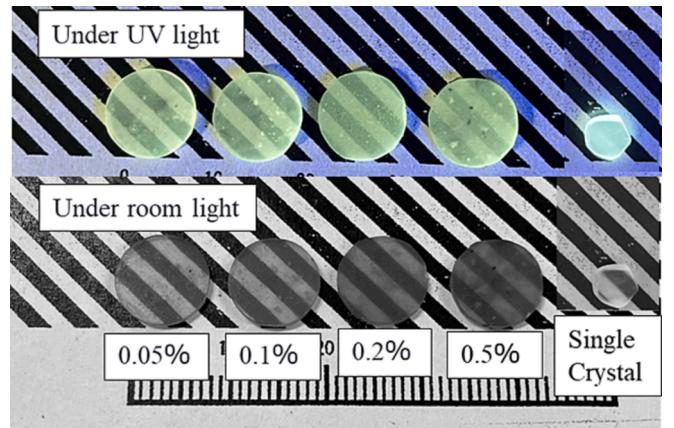
Diffuse transmittance was measured using a spectrophotometer (SolidSpec-3700, Shimadzu). PL excitation/emission maps and PL quantum yields (QYs) were measured using Quantaurus-QY (C11347, Hamamatsu). Based on the results of PL excitation/emission maps, PL decay curves were measured using Quantaur- $\tau$  (C11367, Hamamatsu).

Under X-rays exposure, Scintillation spectra, scintillation decay curves, and afterglow profiles were measured using our original setup [17,34]. Pulse height spectra were measured using  $\gamma$ -rays from <sup>137</sup>Cs, and the LYs were calculated using a Pr-doped Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (Pr:LuAG) single crystal (8,400 ph/MeV) as a reference sample produced by Furukawa. The LY of reference was measured by the Si avalanche photodiode and <sup>55</sup>Fe method.

## 3. Results and discussion

### 3.1. Samples

**Fig. 1** shows the 0.05, 0.1, 0.2, and 0.5% In-doped NaI transparent ceramics and the 0.1% In-doped NaI single crystal. The weights of the 1 mm thickness of transparent ceramics and single crystal were 0.24 and 0.04 g, respectively. All the samples were polished using sandpaper, and thicknesses of all the samples were standardized to 1 mm. The transparent ceramic and single crystal samples were visibly transparent, and the black diagonal lines in the background could be seen through the samples. However, the transparent ceramic samples were slightly black and cloudy, which would be due to carbon contamination during the sintering process. They also showed yellow-green luminescence under UV light.

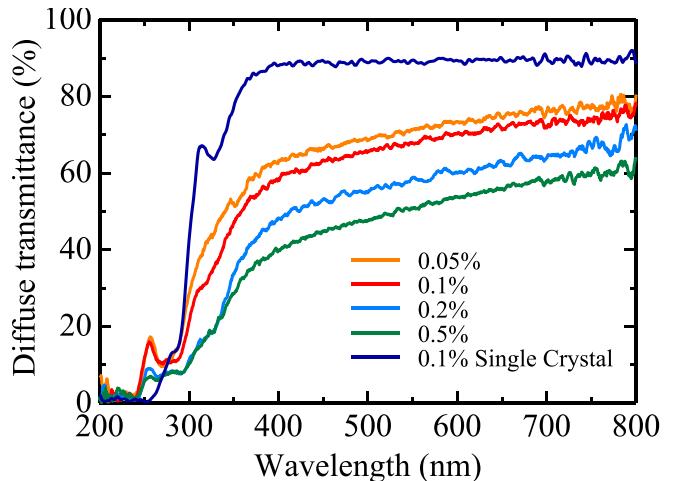


**Fig. 1.** Appearance of the 0.05, 0.1, 0.2, and 0.5% In-doped NaI transparent ceramic and the 0.1% In-doped NaI single crystal samples. The upper panel shows the samples under UV light.

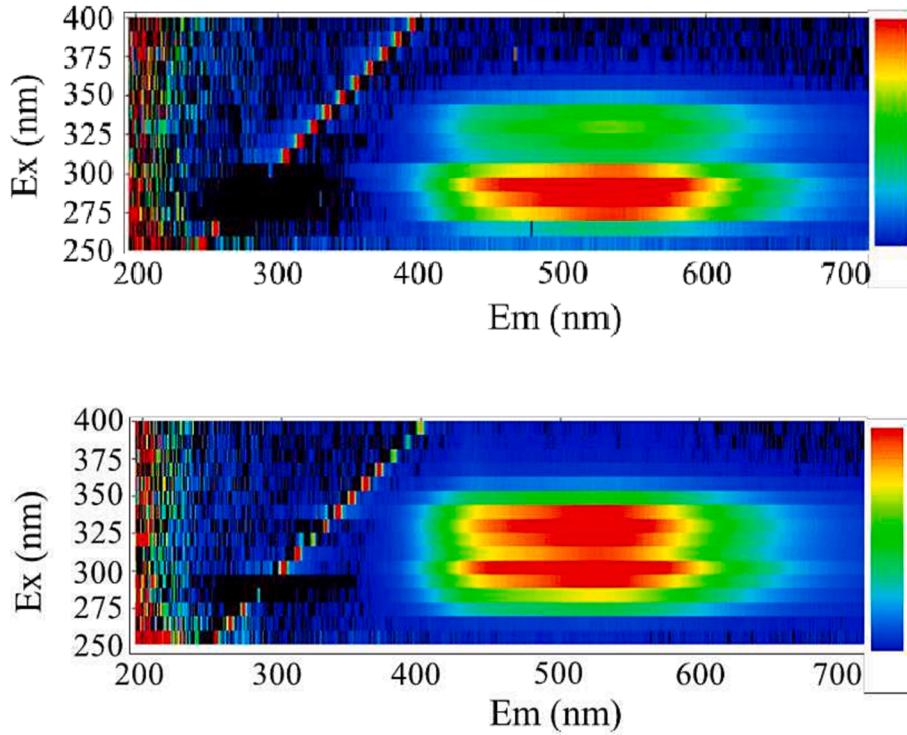
### 3.2. Optical properties

**Fig. 2** shows the diffuse transmittance spectra of the In-doped NaI samples. Absorption bands were observed at around 290 and 320 nm for the transparent ceramic and single crystal samples. These absorptions are caused by  $^1S_0 \rightarrow ^3P_2$  and  $^1S_0 \rightarrow ^3P_1$ , commonly observed in alkali halides such as In-doped NaCl and KBr [35,36]. The absorption at wavelengths shorter than 250 nm is considered to be due to the band gap. Although we could not find any experimental report on the band gap of the undoped NaI, it was estimated to be ~5 eV (~250 nm) by simulation [37], which is in general agreement with the results of this experiment. This absorption has also been reported in undoped NaI transparent ceramic and single crystal [31]. At the wavelength longer than 400 nm, the transmittance of ~90% was achieved, and this value was close to the theoretical limit. Alternatively, that of transparent ceramics was ~70% in In 0.05% one, and the reduction of transmittance was caused by the carbon contamination (**Fig. 1**).

PL excitation/emission maps of all In-doped NaI transparent ceramics and single crystal samples are measured. Here, the PL excitation/emission maps of the 0.1% In-doped NaI samples are shown in **Fig. 3** as a representative since no clear difference was observed in the PL excitation/emission maps of the transparent ceramics with different In concentrations. A main emission band was observed at around 510 nm for all transparent ceramic and single crystal samples. This wavelength is



**Fig. 2.** Diffuse transmittance spectra of the In-doped NaI transparent ceramics and single crystal from 200 to 800 nm.



**Fig. 3.** PL excitation/emission maps of the 0.1% In-doped NaI transparent ceramic (upper part) and single crystal (lower part). Horizontal and vertical axes are emission and excitation wavelength, respectively.

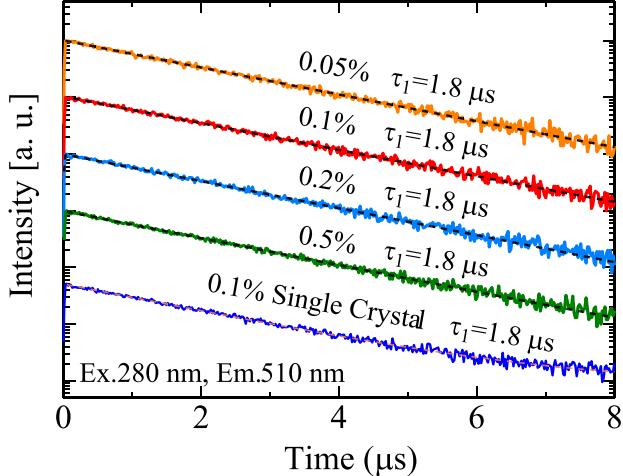
consistent with the emission wavelength of In-doped CsI single crystals due to the  $^3P_1 \rightarrow ^1S_0$  transition [38]. Excitation wavelengths for the emission at 510 nm were 290 and 330 nm, which are consistent with the absorption wavelengths on the diffuse transmittance spectra shown in Fig. 2. In addition, the difference in the spectral shape of the PL excitation between the transparent ceramics and the single crystal sample would be attributed to self-absorption. The QY of the 0.05%, 0.1%, 0.2%, and 0.5% In-doped transparent ceramic samples under 290 nm excitation was 31, 32, 28, and 27 %, respectively. In contrast, the QY of the 0.1% In-doped NaI single crystal sample was 39%.

The PL decay curves of the transparent ceramic and single crystal samples are shown in Fig. 4. The PL decay curves were monitored at a wavelength of 510 nm under 280 nm excitation. The decay curves of all the samples could be approximated by an exponential decay function.

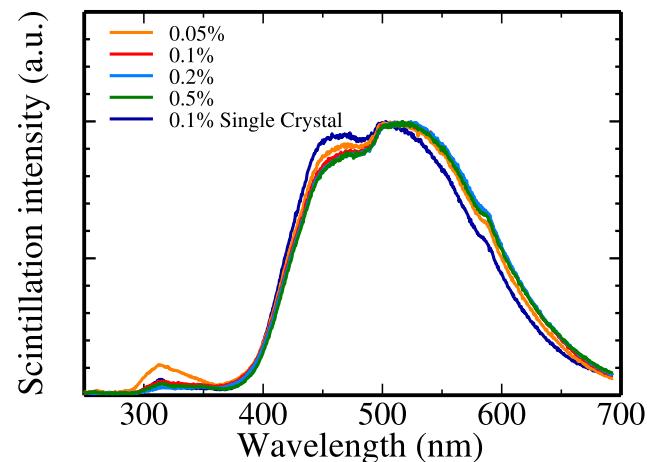
The decay time constants for the transparent ceramics and single crystal were all 1.8  $\mu$ s, and the values did not vary with In concentrations. The tendency was consistent with PL QY which was ~30% regardless of In concentration. These are almost consistent with the previously reported decay time constant of the  $^3P_1 \rightarrow ^1S_0$  transition of In-doped CsI single crystal (1.9  $\mu$ s) [33]. Judging from these analogical optical and photoluminescence properties with other In-doped alkali-halide crystals and some previous reports on Tl or In-doped alkali-halides, In would substitute the Na site [35].

### 3.3 . Scintillation properties

Fig. 5 shows the scintillation spectra of the In-doped NaI transparent ceramics and single crystal under X-ray irradiation. For all the samples,



**Fig. 4.** PL decay curves of the In-doped NaI transparent ceramics and single crystal.



**Fig. 5.** X-ray-induced scintillation spectra of the In-doped NaI transparent ceramics and single crystal. The scintillation intensities were normalized at wavelength with the highest intensity.

scintillation peaks were observed at around 320, 460, and 510 nm. The peaks at 460 and 510 nm are due to the  $^3P_1 \rightarrow ^1S_0$  transitions of In<sup>+</sup>. The difference between the PL and the scintillation spectra is the presence of the peak at 320 nm. This emission is also observed in undoped NaI single crystals and transparent ceramics [31], originating from self-trapped excitons (STE) [39]. The intensity of this peak decreased with increasing In concentrations. This was a typical tendency of the intrinsic emissions like STE.

Fig. 6 shows the scintillation decay curves of In-doped NaI transparent ceramic and single crystal samples under X-ray irradiation. The decay curves were approximated by a single exponential decay function. The decay time constant for the 0.05, 0.1, 0.2, and 0.5% In-doped NaI transparent ceramics was all 1.8  $\mu$ s which was equal to the PL decay time constant. The scintillation decay time constant for the In-doped NaI single crystal was 1.9  $\mu$ s. Considering the PL characteristics and scintillation spectra, these decay time constants would be attributed to the  $^3P_1 \rightarrow ^1S_0$  transition in In<sup>+</sup> [40].

Fig. 7 shows afterglow curves of the In-doped NaI transparent ceramics and single crystal. The afterglow levels (AL) were determined by the intensity at 20 ms after X-ray irradiation for 2 ms. Here, AL is defined as  $AL [\text{ppm}] = (I_2 - I_0) / (I_1 - I_0) \times 10^6$ , where  $I_0$  is the background signal intensity,  $I_1$  is the average signal intensity under X-ray irradiation, and  $I_2$  is the signal intensity at 20 ms after X-ray irradiation. The measured ALs are listed in Table 1 with some other properties. The AL of transparent ceramics was 106, 170, 193, and 293 for the 0.05%, 0.1%, 0.2%, and 0.5% samples, respectively. On the other hand, the AL of the single crystal sample was 777 ppm. The ALs of the In-doped NaI transparent ceramics were relatively low compared to the reported one of Tl-doped CsI single crystals (300 ppm) measured with the same setup [41]. The reason for the increased afterglow levels with increasing the In concentrations is the higher concentration of point defects. These defects lead to a greater number of shallow trap levels. This phenomenon has been reported for In-doped CsI single crystals [40]. In this previous study, the relationship between charge carrier traps and afterglow levels was investigated using the thermally stimulated luminescence method, and it was concluded that afterglow levels depend on the activator concentration.

Compared with the In-doped NaI single crystal sample, the transparent ceramics had lower ALs. This suggests that the transparent ceramic samples have fewer shallow trapping levels formed by defects. The main reason for this difference is due to the different fabrication methods between single crystals and ceramics. In this study, the transparent ceramics were fabricated using the SPS method, while the single crystal sample was prepared by the Bridgman–Stockbarger method. In

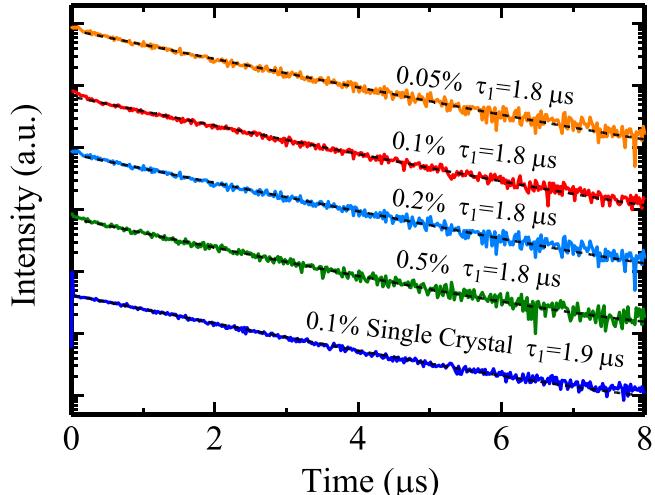


Fig. 6. X-ray-induced scintillation decay curves of the In-doped NaI transparent ceramics and single crystal.

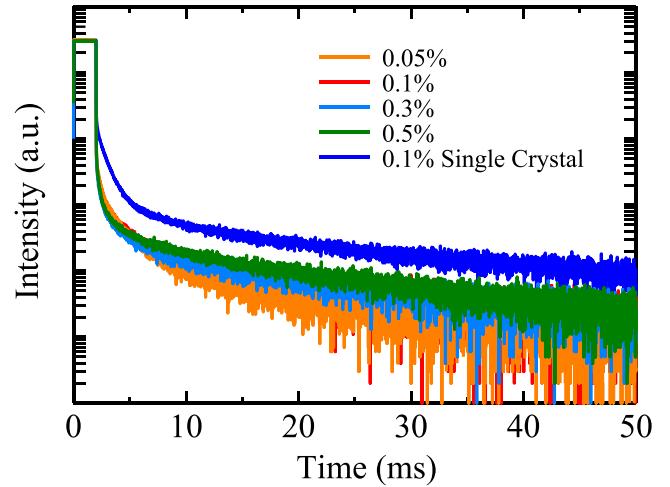


Fig. 7. X-ray-induced afterglow curves of the In-doped NaI transparent ceramics and single crystal.

Table 1

PL QYs, ALs, LYs, and energy resolution of the In-doped NaI transparent ceramics (0.05, 0.1, 0.2, and 0.5%) and single crystal.

Sample	QY (%)	AL (ppm)	LY (ph/ MeV)	Energy resolution (%) at 662 keV
0.05 % In	31	106	15,000	28
0.1 % In	32	170	19,000	27
0.2 % In	28	193	16,000	27
0.5 % In	27	293	15,000	33
0.1 % Single Crystal	39	777	12,000	29

the Bridgman–Stockbarger method, single crystal samples are grown by heating above the melting point and then slowly cooling. In contrast, in the SPS method, sintering occurs by solid-state reactions that proceed below the melting point, which may reduce lattice defects such as iodine.

Fig. 8 shows pulse height spectra of the In-doped NaI transparent ceramic and single crystal samples. In this experiment,  $^{137}\text{Cs}$  was used as the  $\gamma$ -ray source, and Pr:LuAG was used as the reference sample. The photoabsorption peak of NaI transparent ceramics doped with 0.05, 0.1, 0.2, and 0.5% In was observed at 388, 489, 435, and 389 channel,

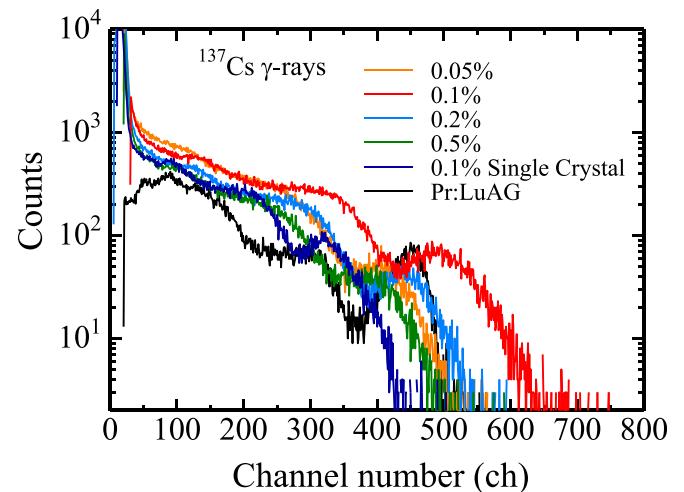


Fig. 8. Pulse height spectra of the In-doped NaI transparent ceramics and single crystal under  $\gamma$ -ray irradiation from  $^{137}\text{Cs}$ . The spectrum of Pr:LuAG is also illustrated as a reference.

respectively. On the other hand, the photoabsorption peak of the 0.1% In-doped NaI single crystal was observed at 320. The emission wavelength of the In-doped NaI transparent ceramic and single crystal sample was the same (510 nm), and the quantum efficiency (QE) of PMT at 510 nm was 25%. The emission wavelength of the reference sample (Pr: LuAG) was 320 nm, and the QE of PMT at 320 nm was 30%. Taking into consideration about peak channel number and QE, the LY of the 0.05, 0.1, 0.2, and 0.5% In-doped NaI transparent ceramics was estimated to be 15,000, 19,000, 16,000, and 15,000 ph/MeV, respectively. On the other hand, the LY of the 0.1% In-doped NaI single crystal was 12,000 ph/MeV. The energy resolutions of 0.05, 0.1, 0.2, and 0.5% In-doped NaI transparent ceramics at 1 mm thickness were 28, 27, 27, and 33%, respectively. On the other hand, the energy resolution of the In-doped NaI single crystal was 29%. Although the LYs of In-doped NaI were lower than commercial Tl-doped NaI single crystal (38,000 photons/ MeV) [42], the In-doped NaI transparent ceramics showed higher LYs than that of the single crystal. The LY of In-doped CsI single crystals has been reported as 27,000 ph/MeV [40]. Consequently, the LY of the In-doped NaI samples was found to be lower than that of In-doped CsI. In general, Indium exists in two valence states, monovalent ( $In^+$ ) and trivalent ( $In^{3+}$ );  $In^+$  can contributes to luminescence while  $In^{3+}$  does not [27]. Since the raw powder used in this study was  $InI_3$ , it is assumed that trivalent ions ( $In^{3+}$ ) that can not contribute to luminescence were present in the sample. In the future, the LY of In-doped NaI could be greatly improved by using raw powders with high  $In^+$  ion content, such as  $InI$ .

#### 4. Conclusion

We successfully fabricated In-doped NaI transparent ceramics and single crystals for the first time. All the samples showed PL and scintillation due to the  $^3P_1 \rightarrow ^1S_0$  transition of  $In^+$ . The decay time constant was approximately 1.8  $\mu s$ . Although the single crystal showed the highest PL QY among all the samples, the LYs of the transparent ceramics were larger than the single crystal. Compared with the same In concentration samples (0.1%), the LY of the 0.1% In-doped NaI transparent ceramic was 19,000 ph/MeV, while that of the 0.1% In-doped NaI single crystal was 12,000 ph/MeV. The ALs of the transparent ceramic were smaller than the single crystal, which would bring about the higher LY of the transparent ceramic.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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