Optically stimulated luminescence properties of Tl-doped NH$_4$Cl transparent ceramics fabricated by SPS method

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ABSTRACT
We fabricated Tl-doped NH$_4$Cl transparent ceramics using SPS method with varying Tl concentrations (0%, 0.01%, 0.05%, 0.1%, 0.5%, and 1%) and evaluated the properties of photo-luminescence (PL), radioluminescence (RL), and optically stimulated luminescence (OSL). In PL and RL spectra, Tl-doped samples exhibited two emission peaks at around 380 and 530 nm due to the $^3P_1 \rightarrow ^1S_0$ transitions of Tl$^+$ and the off-center self-trapped excitons near Tl$^{2+}$, respectively. In the OSL spectra, Tl-doped samples exhibited an emission peak at around 350 nm due to the $^3P_1 \rightarrow ^1S_0$ transitions of Tl$^{2+}$. In the OSL dose–response function, all the samples showed a good linearity over a range of 10–10,000 mGy.

1. Introduction
Phosphors can work in two models: instant and delayed, and scintillators and storage phosphors utilize the respective models [1]. Scintillators have a function that absorbs the energy of ionizing radiations and then immediately converts them into low-energy photons [2]. Scintillators are generally combined with photoelectric conversion devices such as photomultiplier tubes (PMT) and Si photodiodes and are used as radiation detectors in various fields such as medical imaging techniques [3–5], environmental monitoring [6,7], and security [8,9]. In contrast, storage phosphors can store the absorbed radiation energy and release it as luminescence at any time for users. When storage phosphors are irradiated with ionizing radiation, a number of electrons are excited, and electron–hole pairs are generated. The electron–hole pairs are transferred to trapping centers where they remain until release. After that, when the electrons or holes are re-excited by external stimulations, the re-excited electrons or holes are relaxed and recombined at luminescence centers to emit light. When the stimulation is light, the luminescence is called optically stimulated luminescence (OSL) [10–12]. In contrast, when the stimulation is heated, the luminescence is called thermally stimulated luminescence (TSL) [13,14]. Owing to the advantage that users can read out the luminescence signals at any time, storage phosphors are often used for personal dosimeters and imaging plates [15–19]. When used as personal dosimeters, storage phosphors ideally require the following properties. First, to evaluate the radiation effect on the human body precisely, the effective atomic number ($Z_{\text{eff}}$) should be close to that of soft human body tissues ($Z_{\text{eff}} \sim 7.22$) [20]. Second, to estimate the accurate exposure dose, there should be a good proportionality between the irradiated dose and the luminescence intensity [21]. Third, to underestimate exposure dose, the fading of luminescence signals should be low at a room temperature [22]. However, there are no storage phosphors fulfilling all of these requirements; thus, a number of studies have been continuously carried out to enhance the properties of the storage phosphors [19,23–25].

In recent years, transparent ceramics fabricated by spark plasma sintering (SPS) have attracted much attention as storage phosphors due to larger volume and due to higher concentration of defects than single crystal [26,27]. The larger volume achievable for transparent ceramics leads to more intense luminescence (volumetric effect), and we can detect storage-type luminescence not only from the surface but also from the full volume of materials. Thus, this effect makes a contribution to the luminescence intensity. Ceramics also have more defects acting as trapping centers than single crystals. In particular, by using SPS, materials are sintered in a reducing atmosphere; thus, ceramics fabricated by SPS have more defects than those fabricated by atmospheric sintering [28–34].

In this study, we focused on Tl-doped NH$_4$Cl transparent ceramics as OSL phosphors. The
reasons why we chose this material are as follows. First, $Z_{\text{eff}}$ of NH$_4$Cl is ~14.5, which is close to that of soft human body tissues. Second, Tl-doped ammonium salts such as (NH$_4$)$_2$SiF$_6$ and NH$_4$Br were reported to exhibit a high OSL sensitivity to fast neutrons [35]; thus, we expected that Tl-doped NH$_4$Cl would also exhibit effective OSL properties. In addition, although there have been some reports of optical properties of Tl-doped NH$_4$Cl [36–38], the OSL properties have not been studied yet. For the above reasons, we fabricated Tl-doped NH$_4$Cl transparent ceramics using SPS and evaluated the OSL properties. Moreover, in this work, we evaluated the properties of photoluminescence (PL) and radioluminescence (RL) in order to identify luminescence centers.

2. Materials and methods

2.1. Sample preparation

Tl-doped NH$_4$Cl transparent ceramic samples were fabricated using an SPS system (LABOX-110, Sinter Land). First, NH$_4$Cl powder (>99.5%, Fujifilm Wako Pure Chemical Industries) was uniformly mixed with Tl powder (99.9%, High-Purity Chemicals) by varying the ratio of Tl (0, 0.01, 0.05, 0.1, 0.5, and 1 mol%) to NH$_4$Cl. After that, the obtained mixture was loaded into a cylindrical graphite die and was sintered with the following conditions. The temperature was raised from 20 to 1800°C with a heating rate of 10°C/min and was then kept constant for 20 minutes. In parallel, the pressure was controlled as follows. The pressure was raised to 0.5 MPa in 1 minute and was then kept constant for 26 minutes. The pressure was then raised to 5.5 MPa in 1 minute and was then kept constant for 9 minutes. After the sintering process, the obtained samples were polished with sandpaper, and the thickness of all the samples was equally adjusted.

2.2. Property evaluation

The microstructures of the obtained samples were observed using a scanning electron microscope (SEM, JCM-6000Plus, Japan Electron Optics Laboratory). To evaluate the basic optical properties, in-line transmittance spectra were measured using spectrophotometers (V-670, JASCO). Regarding the PL properties, the emission maps and the quantum yields (QYs) were measured using Quantaurus-QY (C11347, Hamamatsu). In this measurement, the spectral range for excitation and emission wavelengths was 250–400 nm and 300–700 nm, respectively. The PL decay profiles were measured using Quantaurus-t (C11367, Hamamatsu). In this measurement, the monitored wavelength was 530 nm under 280 nm excitation.

The X-ray-induced RL spectra were measured using a setup consisting of an X-ray generator (XRBB0N100, Spellman) and a spectrometer (163 monochromator, Shamrock, and DU-420-BU2 CCD, Andor) [39]. The X-ray-induced RL decay time curves were measured using our setup, illustrated in a previous report [40].

The measurements concerning the OSL properties were carried out using a spectrofluorometer (FP-8600, JASCO). The stimulation and emission spectra were measured after X-ray irradiation (2 Gy). To obtain the dose–response function, OSL decay time profiles were measured by varying the X-ray doses from 1 to 10,000 mGy. Here, the X-ray doses were controlled by altering the tube current of the X-ray generator (0.052–5.2 mA) and the distance between the X-ray source and the samples (0–82 cm).

3. Results and discussion

3.1. Sample characterization

Photographs of the obtained samples are shown in Figure 1. The diameter and thickness of all the samples
were ~10 and ~1 mm, respectively. The appearance was transparent, and the transparency seemed to decrease as Tl concentrations increased. Figure 2 illustrates a SEM image of the surface of the 0.01% Tl-doped NH₄Cl sample as a representative. From this image, the average diameter of the particles was estimated to be ~25 μm.

The in-line transmittance spectra over a range of 190–2700 nm are shown in Figure 3(a), and the enlarged spectra over a range of 190–400 nm are shown in Figure 3(b). The transmittance decreased with increasing Tl concentrations, which can be due to the increase in the light scattered on the surface of grain boundary. Absorption at around 1300, 1600, 2200, and 2700 nm was observed for all the samples, and the one at around 240 nm was observed for Tl-doped samples. The spectrum shape of the longer wavelength is similar to that of the NH₄Cl fine powder in previous reports; therefore, the peaks at around 1300, 1600, 2200, and 2700 nm can be assigned to the combination of N-H stretching and N-H bending (A), the first overtone stretch of N-H bonds (B), the combination of NH₄⁺ asymmetric stretching and NH₄⁺ bending (C), and O-H stretching (D), respectively [41–44]. Compared with the previous report of the Tl-doped NH₄Cl single crystals, the peak at around 240 nm was attributed to the 1S₀ → 3P₁ transitions of Tl⁺ [36–38].

3.2. Photoluminescence properties

Figure 4 shows the PL emission maps of the Tl-doped NH₄Cl samples. Two emission peaks were detected for the Tl-doped samples; one appeared at around 380 nm under the excitation of 250 nm, and the other appeared broadly at around 530 nm under the excitation around 270 nm. The intensity ratio of the emission peak at 530 nm to the one at 380 nm increased with increasing Tl concentrations. The excitation and emission wavelengths of these two peaks agreed with those in previous reports of Tl-doped halide phosphors [45–47]; therefore, the emissions at 380 and 530 nm were attributed to the 3P₁ → 1S₀ transitions of Tl⁺ and the off-center self-trapped excitons (STEs) near Tl⁺, respectively. The Tl-undoped sample exhibited no emission peaks in the emission map. Table 1 shows the PL QYs of the emissions at 300–450 and 450–700 nm. The former QY decreased at higher concentrations of Tl (0.1%, 0.5%, and 1%), which is a typical feature of concentration quenching. On the other hand, the latter QY increased with increasing Tl concentrations. These behaviors were also observed in the other Tl-doped materials [45,47,48].

Figure 5 shows the PL decay time profiles of the Tl-doped NH₄Cl samples where the excitation and the monitored wavelengths are 280 and 530 nm, respectively. Here, the profile of Tl-undoped sample is excluded since the sample exhibited no emission peak in the emission map (Figure 4). All the profiles were approximated by a single exponential decay function. The obtained decay time constants were 1.14–1.18 μs, which were almost constant despite the
different concentrations of Tl. The obtained decay time constants agreed well with those due to the off-center STEs near Tl\(^{+}\) in previous reports of Tl-doped materials having the same crystal structure as NH\(_4\)Cl\([46,48]\); therefore, this emission can originate from the off-center STEs near Tl\(^{+}\).

### 3.3. Radioluminescence properties

Figure 6 shows the RL spectra of Tl-doped NH\(_4\)Cl samples under X-ray irradiation. The 0.05%, 0.1%, 0.5%, and 1% Tl-doped samples had two emission peaks; one was observed at around 380 nm, and the other was observed at around 530 nm. The emission feature was similar to that of PL; therefore, the emission peaks at 380 and 530 nm were attributed to the \(^{3}P_{1} \rightarrow ^{1}S_{0}\) transitions and the off-center STEs near Tl\(^{+}\), respectively. The emission intensity of the 1% Tl-doped sample was the highest of the six. The undoped sample exhibited no emission peak as with PL. In addition, the 0.01% Tl-doped sample also exhibited no

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**Table 1.** PL QYs of Tl-doped NH\(_4\)Cl samples where \(\lambda_{\text{ex}}\) and \(\lambda_{\text{em}}\) are the excitation and emission wavelengths, respectively.

| Tl concentration (%) | 0.01 | 0.05 | 0.1 | 0.5 | 1   |
|----------------------|------|------|-----|-----|-----|
| QY (%)               |      |      |     |     |     |
| \(\lambda_{\text{ex}}: 300-450\) nm | 13.9 | 26.9 | 23.5 | 18.1 | 16.6 |
| \(\lambda_{\text{em}}: 250\) nm          |      |      |     |     |     |
| \(\lambda_{\text{ex}}: 450-700\) nm      | 12.4 | 47.5 | 54.8 | 55.9 | 58.9 |
| \(\lambda_{\text{em}}: 270\) nm          |      |      |     |     |     |

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**Figure 4.** PL emission maps of the Tl-doped NH\(_4\)Cl samples. The vertical and the horizontal axes indicate the excitation and the emission wavelengths, respectively.

**Figure 5.** PL decay time profiles of Tl-doped NH\(_4\)Cl samples. The monitored wavelength is 530 nm under 280 nm excitation.
emission peak in this measurement because the emission intensity was significantly lower. For the 0.5% and 1% Tl-doped samples, the shape of the emission peak at 380 nm was different from that of the other samples. Compared with the PL emission map (Figure 4), this emission partially overlapped with the excitation band at around 280 nm of PL. Hence, this emission of the 0.5% and 0.1% samples would be partially absorbed by the off-center STEs near Tl\(^+\). As with PL, with increasing Tl concentrations, the intensity ratio of the emission peak at 530 nm to the one at 380 nm gradually increased. The reason would be that the optimal concentrations of Tl\(^+\) were different between the emission around 380 and 530 nm.

Figure 7 shows the RL decay time profiles of Tl-doped NH\(_4\)Cl samples under pulsed X-ray irradiation and the instrumental response function (IRF). Here, the profiles of 0% and 0.01% Tl-doped samples are excluded since these samples exhibited no emission peaks in the RL spectra (Figure 6). IRF was approximated by a sum of two exponential decay functions, and the RL decay time profiles of the 0.05%, 0.1%, 0.5%, and 1% Tl-doped samples were approximated by a sum of four exponential decay functions including IRF. The obtained decay time constants are shown in Table 2. Compared with the PL decay (Figure 5) and previous reports, the third and fourth decay components were attributed to the \(^{3}P_{1} \rightarrow ^{1}S_{0}\) transitions of Tl\(^+\) and the off-center STEs near Tl\(^+\), respectively [46,47].

3.4. Optically stimulated luminescence properties

Figure 8 shows the OSL emission and stimulation spectra of Tl-doped NH\(_4\)Cl samples after X-ray irradiation (2 Gy). Hereafter, the OSL properties of the undoped sample are not discussed since the sample exhibited no emission both in PL and RL. The emission peak at around 350 nm was observed by the stimulation at 420 nm for all the samples. The difference in peak positions in PL and OSL spectra could be blamed for the read-out method in each measurement. In the OSL-spectrum measurement, since the OSL spectra were observed by scanning from shorter wavelength using the diffraction grating, the OSL signals would fade during the measurement. Compared with the PL emission maps and RL spectra, this emission can be attributed to

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**Table 2.** RL decay time constants of Tl-doped NH\(_4\)Cl samples under X-ray irradiation and IRF.

|         | \(T_1\) (μs) | \(T_2\) (μs) | \(T_3\) (μs) | \(T_4\) (μs) |
|---------|--------------|--------------|--------------|--------------|
| 0.05%   | 0.0094       | 0.0094       | 0.0094       | 0.0094       |
| 0.1%    | 0.13         | 0.13         | 0.13         | 0.13         |
| 0.5%    | 0.17         | 0.20         | 0.18         | 0.17         |
| 1%      | 1.35         | 1.24         | 1.50         | 1.23         |
processes of releasing carriers from trapping states contributed to OSL of Ti-doped NH₄Cl samples.

**Figure 10** shows the OSL dose–response functions of Ti-doped NH₄Cl samples. All the samples exhibited a linear response over a range of 10–10,000 mGy. According to a previous report, the lowest detectable dose of Ti-doped NH₄Br is ~0.4 mGy calculated by scaling linearly the integrated OSL signal to three times the standard deviation of the background [35], which is lower than that of Ti-doped NH₄Cl in this study. Moreover, the commercial OSL dosimeters (Luminess badge, Nagase Landauer) can detect X- and γ-ray over a range of 0.01 mGy–10 Gy [49], and the lowest detectable dose of the obtained samples in this study was 1000 times higher than that of Luminess badges. Therefore, the dose response of Ti-doped NH₄Cl transparent ceramics should be improved in a future work.

### 4. Conclusion

We fabricated Ti-doped NH₄Cl transparent ceramics using the SPS method with varying Ti concentrations (0, 0.01, 0.05, 0.1, 0.5, and 1 mol%) and evaluated the PL, RL, and OSL properties. In the PL emission maps, Ti-doped samples exhibited two emission peaks at around 380 and 530 nm under the excitation at 250 and 270 nm, which were attributed to the \(^3P_1 \rightarrow ^1S_0\) transitions of Ti\(^{4+}\) and the off-center STEs near Ti\(^{4+}\), respectively, and these emission peaks were also observed in the RL spectra under X-ray irradiation. In the OSL spectra, the emission peak was observed at around 350 nm under the stimulation at 420 nm, and this emission was attributed to the \(^3P_1 \rightarrow ^1S_0\) transitions of Ti\(^{4+}\). The OSL intensity was higher under the stimulation whose wavelength was closer to the emission. The OSL decay time profiles were approximated by a sum of two exponential decay curves, which suggests that there were at least two processes of releasing carriers from trapping states in OSL. In the OSL dose–response function, all the samples exhibited a linearity between over a range of 10–10,000 mGy.

### Acknowledgments

This work was supported by Grant-in-Aid for Scientific Research A (17H01375) and B (18H03468 and 19H03533). The Cooperative Research Project of Research Center for Biomedical Engineering is also acknowledged.

### Disclosure statement

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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