

Dosimetric properties of Al_2O_3 transparent ceramics doped with C

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ABSTRACT

We have investigated scintillation and thermally-stimulated luminescence (TSL) dosimetric properties of Al_2O_3 transparent ceramics doped with different concentrations of C ions (0.001, 0.01 and 0.1%). The samples were synthesized by the Spark Plasma Sintering (SPS) technique. All the samples showed scintillation with emission peaks at 310, 410 and 693 nm under X-ray irradiation. Based on the scintillation decay times measured, the origins of emissions at 310, 410 and 693 nm were attributed to F^+ centers, F centers and Cr^{3+} impurity ions, respectively. The samples show notable TSL with a glow curve consisting of peaks at 30, 100, 165, 230, 330 and 405 °C with much higher intensities than those of undoped Al_2O_3 transparent ceramics. A TSL response was confirmed to be linear to the irradiation dose over a range from 0.1 to 1000 mGy.

1. Introduction

Luminescence materials are utilized as radiation dosimeters for personal and environmental monitoring applications and in imaging plates [1–4]. These materials have a function to store and accumulate absorbed energy of ionizing radiations as a form of trapped electrons and holes. The electrons and holes are stored at localized trapping centers, and they recombine to emit light after de-trapping process by external stimulation. Dosimeters based on luminescence materials are mainly divided into three types by different luminescence mechanisms involved. One is thermally-stimulated luminescence (TSL), which is observed by recombination of electrons and holes de-trapped from trapping centers by heat stimulation. Another is optically-stimulated luminescence (OSL) in which the stimulation is performed by light, instead of heat in TSL. The last one is radio-photoluminescence (RPL) which is a phenomenon which generates photoluminescence (PL) centers via interactions with ionizing radiations. The most common materials used in practice today are Ti and Mg co-doped LiF single crystal [1] for TSL, BeO ceramic [2] for OSL and Ag-doped phosphate glass [3] for RPL.

In general, suitable sensitivity, dose linearity, energy response and low fading are required for dosimetric properties which depend on applications. Moreover, if the aim is to measure a radiation dose absorbed in human body, it is desirable that the effective atomic number (Z_{eff}) of the dosimetric material, in the view point of bioequivalence, is close to that of the soft tissue ($Z_{\text{eff}} = 7.13$). With such a tissue equivalent detector, no mathematical calibrations for energy dependence is required ideally. Therefore, for such dosimeter applications especially

in protection dosimetry, it is preferred that the detector materials consist of light elements. In addition to the dosimeter properties, luminescence materials often show scintillation, which is an immediate luminescence upon radiation exposure, without any external stimulation. Scintillators are used in various different fields such as medicine (e.g. X-ray CT, PET and flat panel detector), security (e.g. luggage inspection system), environmental monitoring and basic science [5–8]. Recently, it was pointed out dosimetric and scintillation properties are complementarily related [9,10]; therefore, it is important to investigate both the properties in order to comprehensively understand luminescence mechanisms induced by ionizing radiations.

Aluminum oxide (Al_2O_3) has been investigated as a phosphor material for use in personal dosimetry due to its chemical stability, thermal stability and low effective atomic number close to the soft tissue. Nowadays, Al_2O_3 doped with carbon ($\text{Al}_2\text{O}_3\text{:C}$) crystals as TSL and OSL dosimeters are well-established in personal dosimetry, and it has been available as a commercial product for almost two decades [11–16]. Carbon ions are included to enhance defect creations for main emission centers in Al_2O_3 . On the other hand, with advancement of ceramic fabrication techniques, it was reported that Al_2O_3 can be fabricated in a form of transparent ceramics [17–23]. However, these studies did not deal with the applications for radiation measurements. Compared with single crystals, ceramic dosimeters are considered to have distinct advantages because a large number of defect centers is included which enhance the dosimetric properties [24,25]. In addition, ceramic materials are mechanically strong, cost effective and flexible to be prepared in a geometric shape of preference in each application.

In this study, we synthesized Al_2O_3 transparent ceramics doped with

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C ions (0.001, 0.01 and 0.1%) by spark plasma sintering (SPS) and investigated the dosimetric properties against X-rays in comparison with an undoped Al_2O_3 transparent ceramic. Further, we also studied their scintillation properties in order to discuss the experimental results comprehensively. In general, SPS is performed in a highly reductive environment; therefore, oxygen vacancies are effectively generated, and radiation response properties of transparent ceramic are expected to be enhanced compared with those of single crystal. Such effects were observed in different material systems and reported elsewhere [24–32].

2. Materials and methods

Al_2O_3 transparent ceramics doped with C ions (0.001, 0.01 and 0.1%) were prepared by the SPS technique using Sinter Land LabX-100. Al_2O_3 (99.99%) powder of reagent grade and a carbon powder were homogeneously mixed. The carbon powder was obtained from grinding graphite component used in SPS by using a ball mill (Premium line P-7, Fritsch). The total mass of the mixture was 0.5 g. The mixture was then loaded in a graphite die and held between two graphite punches. A sintering sequence consists of three stages. First, the temperature was elevated from 25 °C to 600 °C in 5 min. Next, the temperature was slowly increased from 600 °C to 850 °C at a rate of 10 °C/min and held for 10 min while applying a pressure of 70 MPa. Finally, the temperature was further increased from 850 °C to 1300 °C at the rate of 10 °C/min and kept at 1300 °C for 20 min while applying 70 MPa pressure. After the synthesis, the wide surfaces of the obtained sample were mechanically polished with a polishing machine (MetaServ 250, BUEHLER). For comparison purposes, we also prepared an undoped Al_2O_3 transparent ceramic by the same procedures.

Optical in-line transmittance spectra were measured by a spectrometer (V670, JASCO) over a spectral range of 190–2700 nm with 1 nm intervals. In order to identify origins of emission centers, we measured X-ray induced scintillation properties. First, we measured scintillation spectra using our lab-constructed setup. A sample was irradiated by X-rays generated by an X-ray tube in which the applied tube voltage and current were 40 kV and 5.2 mA, respectively. The scintillation emission was guided to a CCD-based spectrometer (Andor DU-420-BU2 or Ocean Optics QEPro depending on the spectral range) to measure the spectrum. Details of the setup was described previously [33]. Second, we evaluated X-ray induced scintillation decay times using an afterglow characterization system equipped with a pulse X-ray tube [34]. The system is commercially available from Hamamatsu as a custom-ordered instrument. The applied voltage to the pulse X-ray source was 30 kV, and the system offers the time resolution of ~ 1 ns.

In order to study TSL properties of the C-doped and undoped Al_2O_3 transparent ceramics, we measured a TSL glow curve using a Nanogray TL-2000 [35] after X-ray irradiations with various doses from 0.1 to 1000 mGy. The heating rate was fixed to 1 °C/s for all the glow curve measurements, and the measurement temperature range was from 50 to 490 °C. Further, a TSL spectrum was measured using the CCD-based spectrometer (QE Pro, Ocean Optics) while the sample was heated by an electric heater (SCR-SHQ-A, Sakaguchi E.H Voc) at a constant temperature.

3. Results and discussion

3.1. Sample

Fig. 1 illustrates synthesized Al_2O_3 transparent ceramics doped with C (0.001, 0.01 and 0.1%) under room light. The thicknesses of the 0.001%, 0.01% and 0.1% C-doped samples were 0.75, 0.80 and 0.70 mm, respectively. These samples are visually transparent, and the black stripe patterns on the back were clearly seen. Fig. 2 shows the in-line transmittance spectra of the Al_2O_3 transparent ceramic samples doped with C (0.001, 0.01 and 0.1%). No particular absorption bands due to unexpected contaminations were confirmed in all the samples.

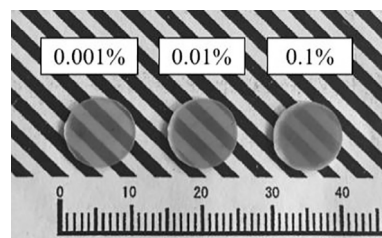


Fig. 1. Synthesized Al_2O_3 transparent ceramics doped with C (0.001, 0.01 and 0.1%) under room light.

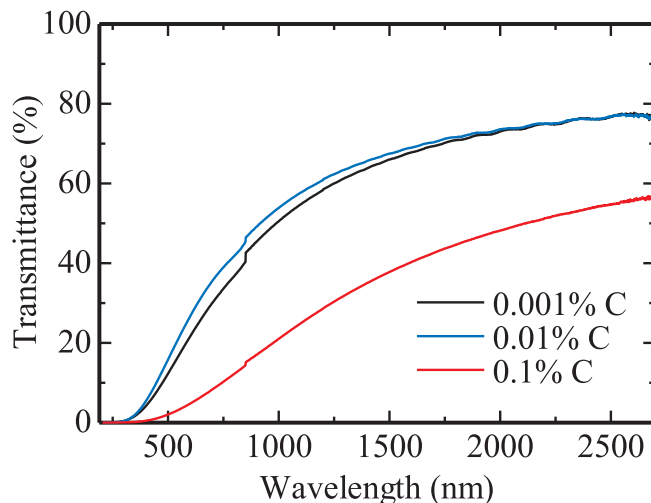


Fig. 2. Transmittance of the Al_2O_3 transparent ceramic samples doped with C (0.001, 0.01 and 0.1%).

The transmittance seems to decrease with increasing the doping concentration while the transmittance increases with an increase of wavelength from 0 to 70% in the spectral range of 300–2700 nm. These characteristics are a typical for Mie scattering. Therefore, we think that the inclusion of carbon causes to increase the number of scattering centers. The significant decrease of transmittance in the 0.1% C-doped sample with respect to the others can be explained as a result of a fraction of carbon powders still remaining in the matrix.

3.2. Scintillation properties

Fig. 3 depicts scintillation spectra of the Al_2O_3 transparent ceramic samples doped with C (0.001, 0.01 and 0.1%). All the samples showed emission peaks around 310, 410 and 693 nm. These peak wavelengths are consistent with the ones reported earlier [36–40]; therefore, the origins of the emissions at 310, 410 and 693 nm are considered to be due to the F^+ centers, F centers and impurity Cr^{3+} ions. These assignments were confirmed by scintillation decay curves shown in Fig. 4. The decay curves measured in the nano-second (ns) range were approximated by a single exponential decay function (indicated in the figure). The derived decay time constants of the 0.001, 0.01 and 0.1% C-doped samples were 4.4, 4.3 and 4.6 ns, respectively. These values agreed with the reported value of the decay constant due to F^+ centers [36,37]. The decay curves measured in the millisecond (ms) range were approximated by double exponential decay functions (indicated in the figure). The obtained decay time constants were 0.9 and 3.9 ms for 0.001% C-doped sample and 0.9 and 4.0 ms for 0.01% C-doped sample and 0.8 and 3.8 ms for 0.1% C-doped sample. The faster decay components obtained reasonably agree with the previously reported value [41] due to Cr^{3+} ion, so we attribute to the same origin in our samples. We assume that the remaining decay constant is possibly due to F centers by the elimination method; however, the obtained values were

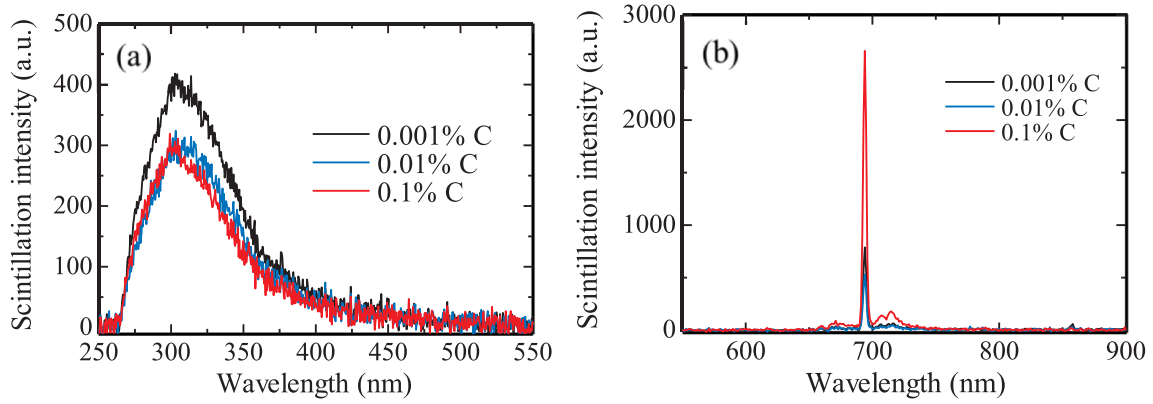


Fig. 3. X-ray induced scintillation spectra of the Al_2O_3 transparent ceramics doped with C (0.001, 0.01 and 0.1%) in the (a) UV and (b) NIR ranges.

not exactly consistent with the previously reported values [36,37]. A possible reason is that the emission intensity is considerably low, according to the observations in the spectra, so the measurement was not as accurate.

3.3. Dosimetric properties

Fig. 5 shows TSL glow curves measured after samples were irradiated with an X-ray dose of 1000 mGy. There was a difference between the glow curve shapes of the undoped and C-doped samples. Moreover, the TSL intensities of C-doped samples were higher than that of undoped sample. From these results, it was implied that C-doping generated additional trapping centers. Fig. 6 shows each TSL glow curve with

glow curve deconvolution (GCD) functions described in [42]. The parameters obtained by the deconvolutions were summarized in Table 1. The free parameters of the GCD functions are the maximum peak temperature (T_m), the maximum peak intensity (I_m) and the activation energy (E). All the glow curves were best-fitted by six GCD functions, and the C-doped samples showed glow peaks about 30, 100, 165, 230, 330 and 405 °C while the undoped sample showed glow peaks about 90, 145, 200, 270, 340 and 450 °C. The GCD function located at the highest temperature was treated as a contribution of black-body radiation by the heater. The peak positions around 30, 165 and 330 °C in C-doped Al_2O_3 are consistent with previous reports [13,43]. Compared with these reports, our samples showed additional glow peaks. These additional peaks can be caused by sintering by using the SPS

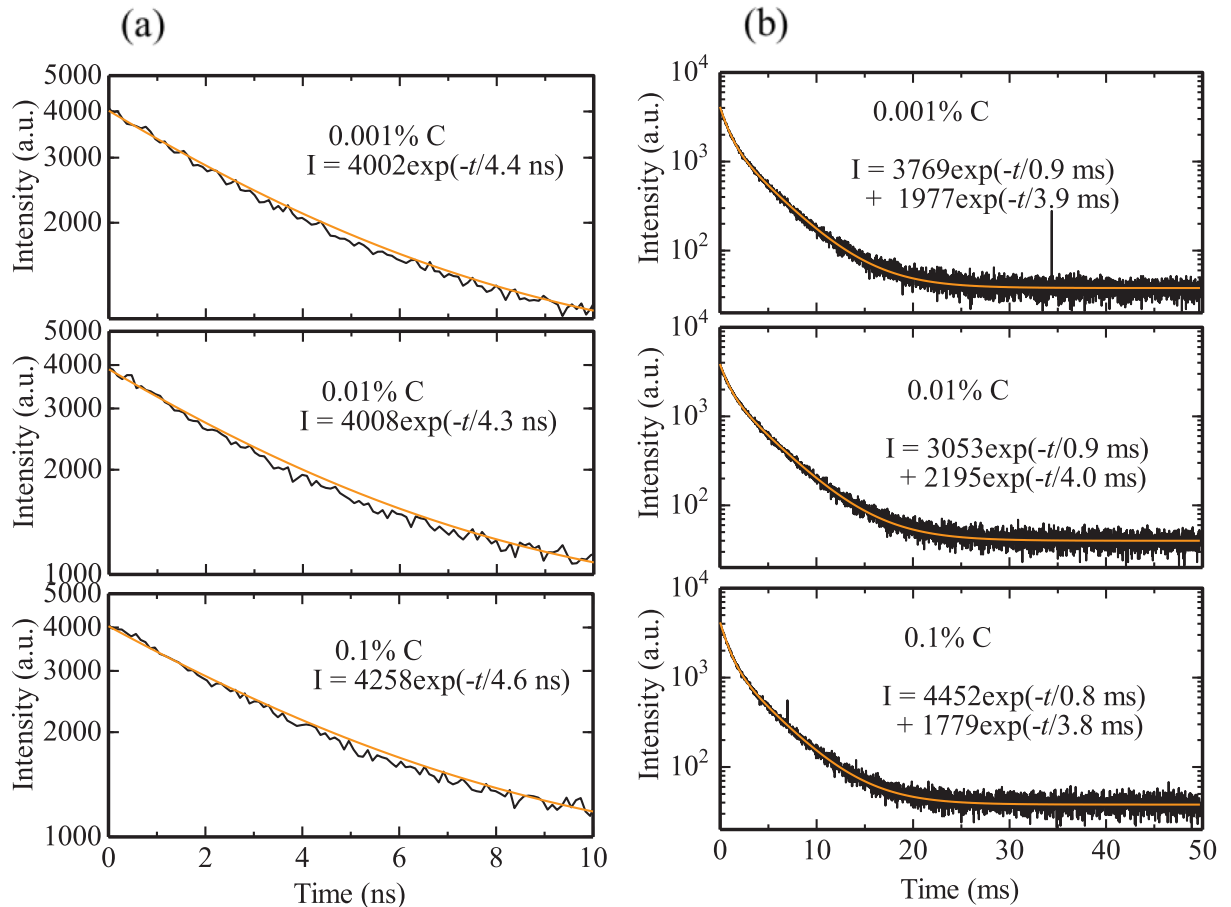


Fig. 4. Scintillation decay profiles of 0.001% (top), 0.01% (middle) and 0.1% (bottom) C-doped samples in the (a) ns and (b) ms ranges.

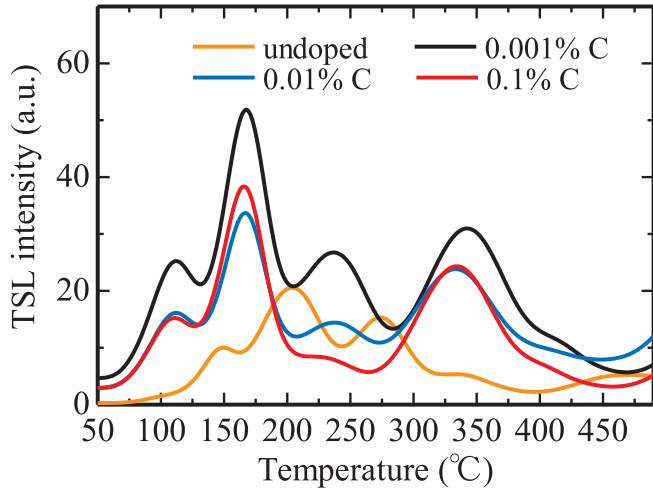


Fig. 5. TSL glow curves of the undoped and C-doped Al_2O_3 transparent ceramics.

method because it is performed in a highly reductive environment. Although it is not clearly stated, the Al_2O_3 samples reported in the literatures [13,43] should have not been prepared by SPS but most likely by the Czochralski technique in air atmosphere as it is typical for $\text{Al}_2\text{O}_3\text{:C}$ crystals synthesized for radiation dosimetry applications. Fig. 7 depicts the TSL spectra measured at 165 °C for the C-doped samples and 200 °C for the undoped sample. The emission bands were detected around 310, 410 and 693 nm. Unlike in the scintillation, the intensities of the 410 nm peak were higher than those of the 310 nm peak. In

Table 1
Parameters of GCD functions.

(a) undoped	Peak1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6
T_m (°C)	93.01	144.01	199.51	271.93	341.14	453.81
I_m (a.u.)	1.25	10.48	28.00	20.35	6.16	4.55
E (eV)	0.14	0.42	0.45	0.68	0.92	0.96
(b) 0.001%	Peak1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6
T_m (°C)	32.15	103.36	164.60	233.71	337.55	412.00
I_m (a.u.)	2.51	28.89	62.79	33.33	37.99	7.31
E (eV)	0.07	0.23	0.42	0.50	0.67	0.98
(c) 0.01%	Peak1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6
T_m (°C)	31.64	102.97	163.90	231.79	327.88	403.91
I_m (a.u.)	1.55	18.25	40.98	16.91	29.19	5.13
E (eV)	0.07	0.22	0.42	0.48	0.65	0.97
(d) 0.1%	Peak1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6
T_m (°C)	30.26	101.48	163.13	224.70	329.86	398.97
I_m (a.u.)	1.44	17.10	47.57	9.64	30.57	4.25
E (eV)	0.07	0.21	0.44	0.50	0.69	1.00

addition, the emission intensities due to F^+ centers (310 nm) and F centers (410 nm) of the C-doped samples were higher than those of the undoped sample.

Fig. 8 indicates relationships between TSL intensity and irradiated X-ray dose, namely dose response curves. By using the dose response

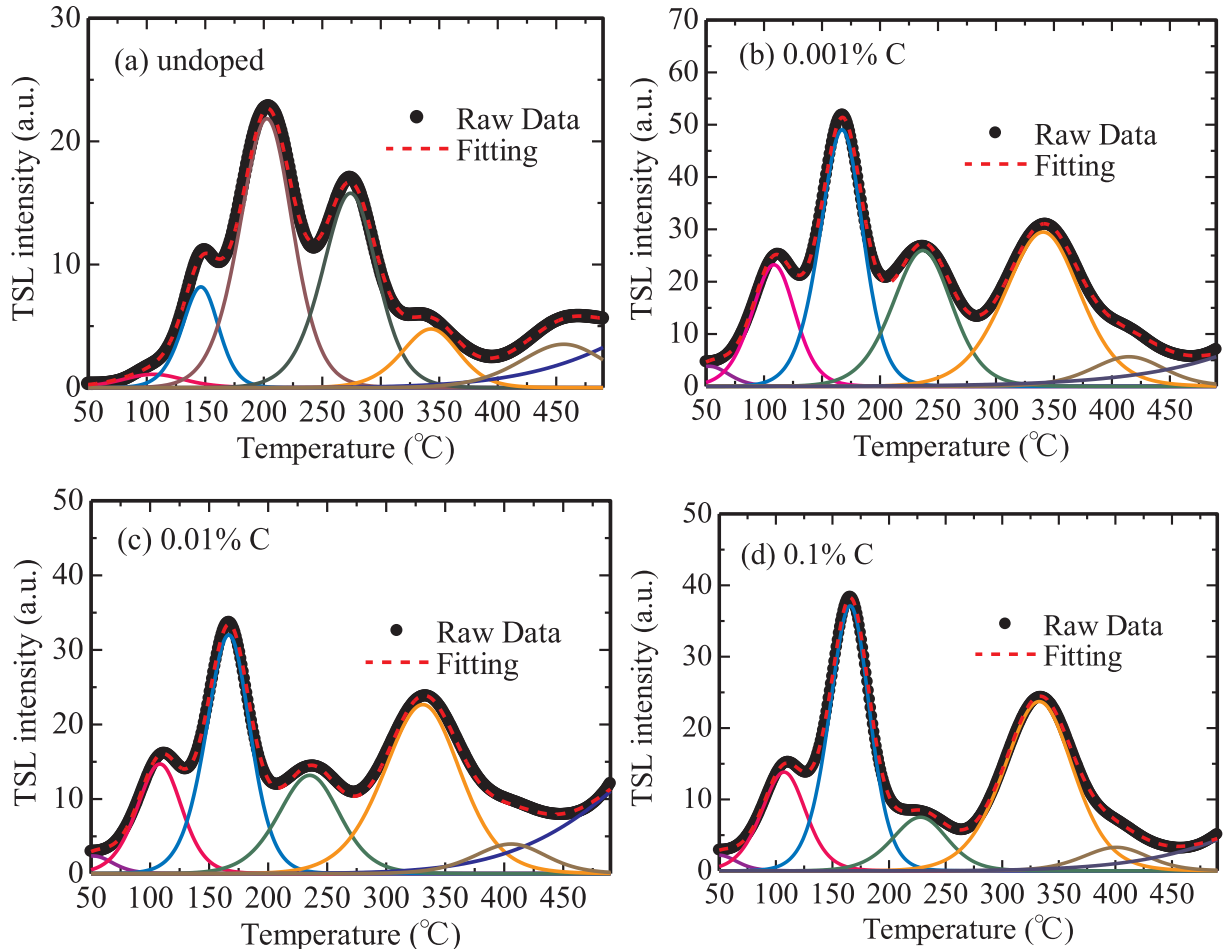


Fig. 6. TSL glow curves with GCD functions of (a) undoped sample as well as (b) 0.001%, (c) 0.01% and (d) 0.1% C-doped samples.

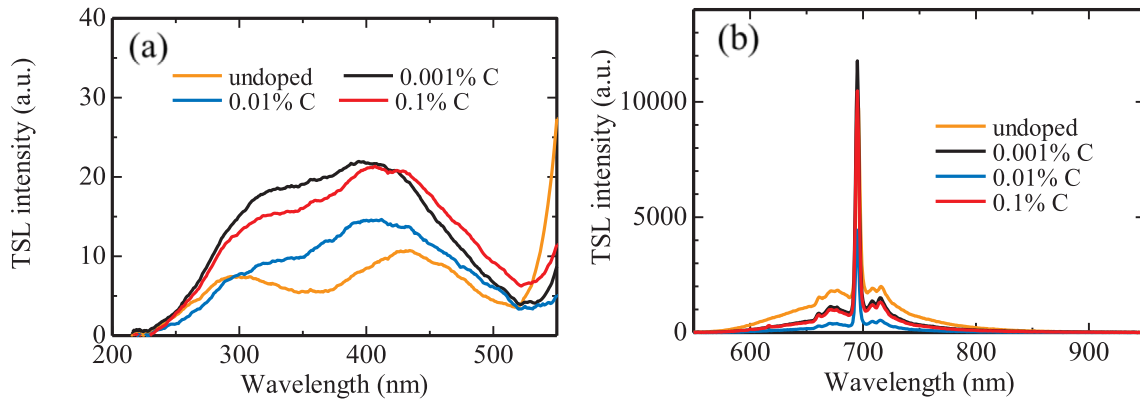


Fig. 7. TSL spectra for the Al_2O_3 transparent ceramics doped with C (0.001, 0.01 and 0.1%) in the (a) UV and (b) NIR ranges. The spectra were measured at 165 °C for the C-doped samples while 200 °C for the undoped sample.

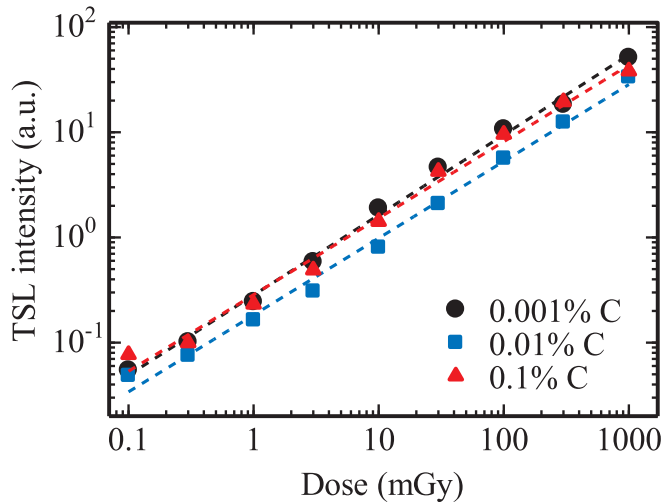


Fig. 8. Dose response curves of the Al_2O_3 transparent ceramics doped with C (0.001, 0.01 and 0.1%). The response was defined as an integrated TSL signal for the glow peak at 165 °C.

curve, we can measure radiation dose from a TSL response. Values of the TSL response used here are integrated peak intensities at 165 °C of the C-doped samples in Fig. 5. The dose range was tested from 0.1 to 1000 mGy. The 165 °C peak of all the samples had a linear response in a dose range of 0.1–1000 mGy. The linearity was confirmed by coefficient of determinations (R^2) derived from a least-square fitting of the experimental data with a power function ($y = a^x + b$), and R^2 of 0.001%, 0.01% and 0.1% C-doped samples were 0.9982, 0.9966 and 0.9955, respectively. It is an equivalent sensitivity with commercial personal dosimeters and the highest sensitivity for C-doped Al_2O_3 including those presented in earlier reports we could find [44,45]. It should be noted that the emission at 693 nm was detected in the TSL glow curve measurements (Fig. 5) due to the thermal-radiation cut filter equipped in the instrument, and it was a typical detector setup in practical applications. Therefore, the sensitivity as a TSL detector should be improved by using a TSL reader optimized for these specific materials.

4. Conclusions

We have synthesized Al_2O_3 transparent ceramics doped with C ions by the SPS technique. Subsequently, we have studied the scintillation and TSL dosimetric properties. From scintillation spectra and decay time constants, we confirmed that the C-doped samples showed emission bands at 310, 410 and 693 nm due to F^+ centers, F centers and impurity Cr^{3+} ions, respectively. The transparent ceramics of Al_2O_3 :C

show notable TSL properties. The glow curve consists of several peaks, in which the peak temperatures were revealed by fitting with GCD functions. As a result, it was found that the glow peak temperatures are different between the C-doped and undoped samples: 30, 100, 165, 230, 330 and 405 °C for the C-doped samples while 90, 145, 200, 270, 340 and 450 °C for the undoped sample. From the difference of the TSL glow curves, it was suggested that C-doping generated additional trapping centers. Furthermore, we demonstrated dose response curves using the glow peak at 165 °C, and we confirmed that the dynamic range was at least over the range of 0.1–1000 mGy. This dose range is equivalent to commercial personal dosimeters and the highest sensitivity among C-doped Al_2O_3 reported.

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