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Scintillation and dosimeter properties of Ca-doped MgO transparent ceramics

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ABSTRACT

We have synthesized 0.001, 0.01 and 0.1% Ca-doped MgO transparent ceramics by the Spark Plasma Sintering (SPS) method, and then we investigated the optical, scintillation and dosimeter properties. In scintillation spectra, some emission bands appeared around 340, 400, 440, 600 and 750 nm. After X-ray irradiation, all the Ca-doped samples showed TSL with a glow peak ~140 °C, and the 0.01% Ca-doped sample showed an additional broad peak ~250 °C. The TSL spectrum consists of a broad peak over 300–500 nm and some peaks around 600 and 750 nm. The TSL response was confirmed to linearly increase with irradiation dose over a dose range from 0.1 to 100 mGy.

1. Introduction

Phosphor materials have been widely used to measure ionizing radiations, and they are typically used in two different types of radiation detectors: scintillation detectors and dosimeters. Both types of phosphor materials absorb incident radiation energy and generate a large number of electrons and holes. Next, scintillator materials immediately emit a numerous number of low energy photons (typically in ultraviolet and/or visible ranges) via charge recombination at emission center and direct excitation of emission center by secondary electrons. Scintillators are used in various fields such as security [1], medical imaging [2], high energy physics [3] and well-logging [4]. In contrast, dosimeter materials store and accumulate generated electrons in a form of trapped charges at localized centers such as lattice defects. The stored duration depends on the application but typically several weeks. These charges can be de-trapped by thermal or optical stimulation and then recombine at emission centers to emit light. The emission by thermal stimulation is so-called thermally-stimulated luminescence (TSL) [5] while that by optical stimulation is called optically-stimulated luminescence (OSL) [6]. These TSL and OSL are often applied for personal dosimetry [7,8]. In various applications as dosimeters, sensitivity to radiation dose, response linearity to dose, energy independence and low fading of storage signal are typically considered. In addition, especially when we are supposed to measure radiation dose absorbed in human body, it is preferred that the effective atomic number (Z_{eff}) of dosimeter material is close to that of biological tissue (Z_{eff} = 7.51). Ideally, using such a tissue equivalent dosimeter, it does not require mathematical processes to calibrate energy dependence, so light elements are preferred as the chemical components of dosimeter materials.

MgO is a common insulator material with a wide band gap (E_g = 7.8 eV) and has received much attention for the fact that it is a simple oxide system with a rock salt (fcc) structure [9] and that the structural defects give unique optical properties. Luminescence properties of undoped MgO have been reported in different material forms including powder, single crystal, films and ceramics [10–18]. In MgO, it is known that electrons trapped at oxygen vacancies form F and F^+ centers which act as luminescent centers [19]. The F-center is a neutral charged oxygen vacancy which captures two electrons, and the F^+-center is a positively charged center with only one electron captured. Although these centers have a common absorption band at ~250 nm, the F and F^+ centers indicate broad photoluminescence bands of different wavelengths at ~500 nm and ~400 nm, respectively [11,14,19].

Dosimeter properties of MgO were reported in the 1970s, and it is known to show two TSL glow peaks around 90–100 and 140 °C when MgO powder was irradiated by X-rays, γ-rays and UV light [20]. MgO is a very interesting material from a viewpoint of biological equivalence, and it has been actively studied for dosimeter applications [21–23]. It is also interesting as it can be made in a ceramic form with optical transparency [24–27], and it was reported to show a broad and long-time phosphorescence emission peaking around 390 nm due to the F^+ center at room temperature [27]. Phosphorescence is in fact a form of TSL since it is the emission by thermal stimulation at room temperature.

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In this context, MgO transparent ceramics potentially have a capability to store charges and may thus be used as TSL dosimeters if the TSL glow peak temperature is somewhat increased. In our group, recently, dosimeter properties of MgO transparent ceramics have been actively studied, and it demonstrated a wide dynamic range of TSL from 0.1 mGy to 1 Gy of X-rays [28–32]. Furthermore, we have attempted to enhance the dosimeter properties by doping with transition metals [29,30], rare-earth [31] and C [32]. Following these studies, we draw an attention to doping with Ca^{2+} ion because Ca and Mg are in the same alkaline earth group, but the radius of Ca^{2+} ion (9.9 nm) is larger than that of Mg^{2+} ion (6.6 nm). Therefore, it would be possible to generate additional lattice defects and improve the dosimeter properties by doping MgO with Ca ion. Although the catalytic, physical and optical properties of Ca-doped MgO (Ca:MgO) nanocrystals [33], powders [34], and transparent ceramics [35] were reported earlier, the scintillation and dosimeter properties could not be found.

In this study, we have synthesized 0.001, 0.01 and 0.1% Ca:MgO transparent ceramics by the spark plasma sintering (SPS) method and investigated their dosimeter properties. Moreover, we have characterized the scintillation properties since dosimeter and scintillation properties are known to be complementarily related in some material systems which can be explained by a simple energy conservation law between the two properties [36,37]. So, it is important to study both scintillation and dosimeter properties in order to understand inclusively the luminescence mechanisms induced by ionizing radiations, and the development of scintillators and dosimeters will be promoted.

2. Experiment

Ca:MgO transparent ceramics were synthesized by the SPS method using a Sinter Land LabX-100. MgO (99.99%) and CaO (99.99%) powders of a reagent grade were uniformly mixed. The mixture (0.5 g) was then loaded into a graphite die and held between two graphite punches. During the sintering, the temperature was increased from 600 °C to 1500 °C at a heating rate of 17 °C/min and then kept for 60 min while applying a pressure of 80 MPa. The wide surfaces of the obtained ceramic samples were mechanically polished. A typical size of the samples was approximately 10 mm in diameter and 1.6 mm in thickness, and they are shown in Fig. 1. The top image shows the samples under room light while the bottom one illustrates those under UV (254 nm) light. As seen in the top image, the obtained samples are visually transparent, but with increasing the concentration of Ca, the transparency slightly decreases.

An in-line transmittance spectrum was measured by using JASCO V670 spectrometer in a spectral range of 190–2700 nm with 1 nm interval.

X-ray induced scintillation spectrum was measured by using our laboratory-constructed set-up [38]. The X-ray source was an X-ray tube equipped with a tungsten anode target and beryllium window (XRB80P &N200X4550, Spellman). The X-ray tube was supplied with a bias voltage of 40 kV and tube current of 2.5 mA during the measurement. We used two spectrometers depending on the spectral range of measurement. For the UV and visible ranges, an assembly of Shamrock SR163 monochromator and Andor DU-420-BU2 CCD was used. The CCD was cooled down to 188 K to reduce the thermal noise. For the visible and near-infrared (NIR) ranges, Ocean Optics QEPro was used.

By using Nanogray TL-2000 [39], a TSL glow curve was measured. The instrument was equipped with a photomultiplier tube (PMT; H7827-001, Hamamatsu), and the temperature range of measurement was from 50 to 490 °C. A sample was irradiated with varying doses of X-rays from 0.1 to 100 mGy before readout. In our setup, 0.1 mGy was the lowest dose of delivery. The heating rate used here was fixed to 1 °C/s. The X-ray dose was calibrated using an air-filled ionization chamber (TN30013, PTW). TSL spectrum was measured by heating an irradiated sample on an electric heater (SCR-SHQ-A, Sakaguchi E.H Voc) at a constant temperature while measuring the spectrum by using the same Andor CCD-based spectrometer as above.

All the above characterizations were conducted at room temperature unless stated.

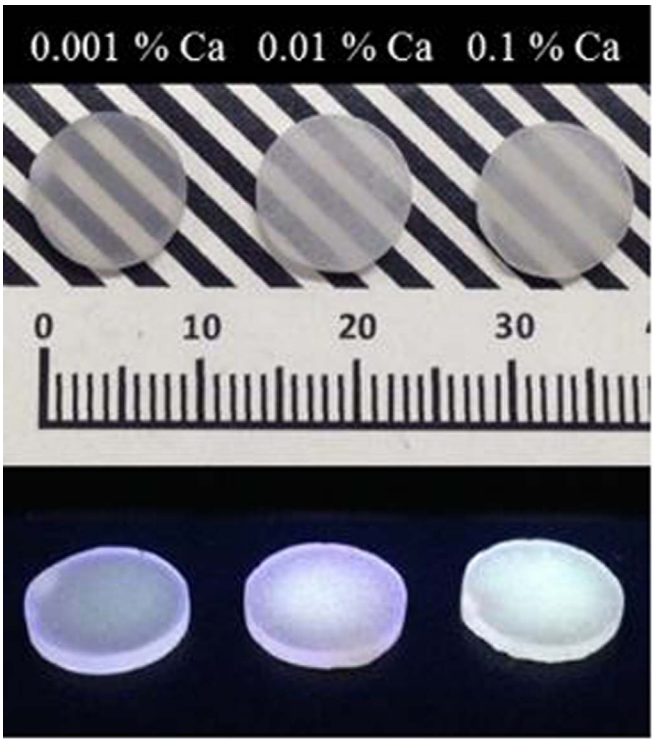

Fig. 1. MgO transparent ceramics doped with Ca^{2+} (0.001, 0.01 and 0.1%) under room light (top) and UV (254 nm) light (bottom).

3. Results and discussion

3.1. Optical properties

Fig. 2 shows in-line transmittance spectra of obtained Ca:MgO transparent ceramics. The transmittance of these samples was close to zero at wavelengths shorter than 190 nm. This spectral range is longer than the wavelength corresponding to the band gap energy of MgO (~7.8 eV = 159 nm) [9,40]. The transmittance decreased with increasing the Ca concentration, and the slope of transmittance curve

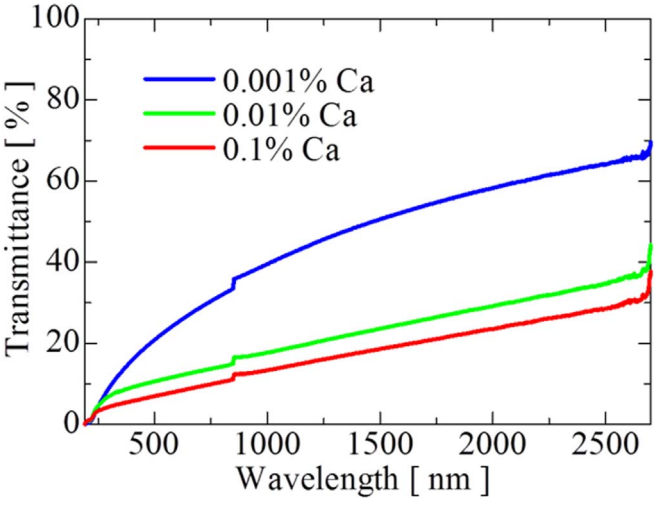

Fig. 2. Transmittance spectra of the MgO transparent ceramics doped with Ca^{2+} (0.001, 0.01 and 0.1%).

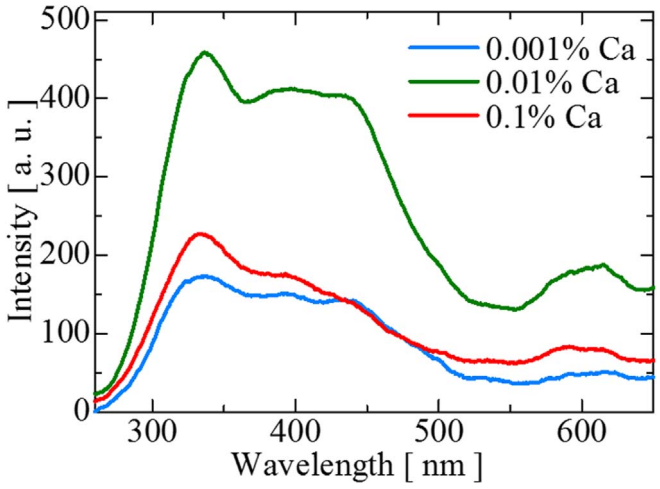

Fig. 3. Scintillation spectra of MgO transparent ceramics doped with Ca^{2+} (0.001, 0.01 and 0.1%) in the UV–vis range.

observed over a wide spectral range is a typical feature of Mie scattering.

3.2. Scintillation properties

Figs. 3 and 4 show X-ray induced scintillation spectra of Ca:MgO transparent ceramics in the UV–Vis and NIR ranges, respectively. All the samples showed some emission bands peaking around 340, 400, 440, 600 and 750 nm. These emission origins are attributed to surface (grain boundary) defects [41], F^+ center [11,14,27], some defects [42–46] and interactions of impurities (Cr^{3+}, Mn^{2+} and Fe^{3+} ions) [14,30,47–49], respectively. The emission appeared around 600–750 nm was interpreted as a mixture of some luminescence processes, and possible origins ever reported are as follows. Most likely, the origin of the emission band around ~700 nm is a combination two processes: Cr^{2+} + hole → Cr^{3+} + $h\nu$ and Fe^{2+} + Cr^{3+} → Fe^{3+} + Cr^{2+}. The emission bands around ~652.5 and ~649 nm are known as a complex impurity-vacancy defect, or Mn^{2+}-F^+ (or F) center [50]. Moreover, the emission band around 760 nm was reported to be due to Mn^{2+} [30]. The emission bands around 610 and 730 nm was derived from the following emission processes: Fe^{2+} + hole → Fe^{3+} + $h\nu$ (610 nm) and $V_{OH^-}Fe^{3+}$ + e^- → $V_{OH^-}{}^{-}Fe^{2+}$ + $h\nu$ (730 nm) [48]. In this regard, however, there are various interpretations of the mechanisms of emissions appeared across 600–750 nm, and the mechanisms

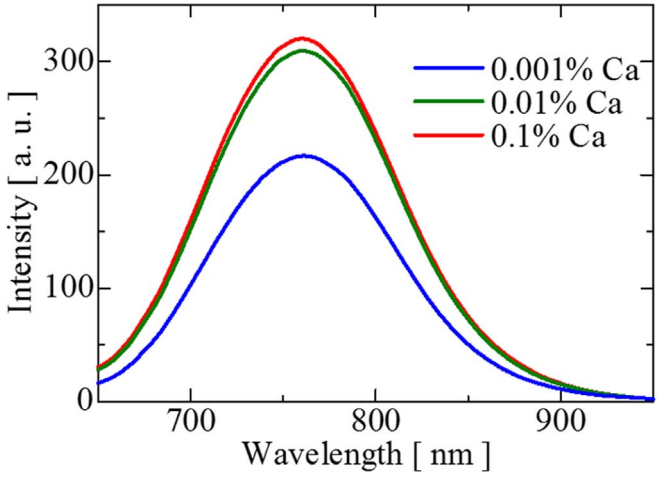

Fig. 4. X-ray induced scintillation spectra of MgO transparent ceramics doped with Ca^{2+} (0.001, 0.01 and 0.1%) in the vis-NIR range.

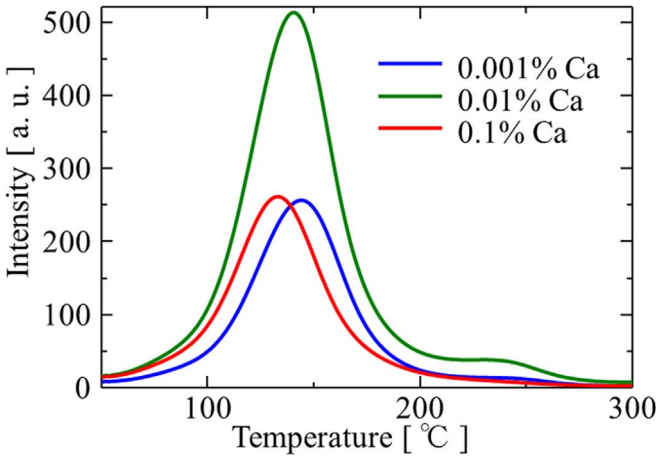

Fig. 5. TSL glow curves of the MgO transparent ceramics doped with Ca^{2+} (0.001, 0.01 and 0.1%) after 100 mGy X-ray irradiation.

have been still under investigations. In the previous reports of MgO transparent ceramics in our group [28–32], the 440 nm emission was not observed. The origin of this emission band was interpreted as point defects generated by deformation of MgO [45,46], and this emission was reported in undoped MgO bulk single crystals, nanoparticle and macropowder [42–45] as well as bulk Ca-doped MgO single crystal [46]. We interpret that such point defects cannot be generated effectively by SPS but Ca-doping.

3.3. Dosimeter properties

TSL glow curves of Ca:MgO samples were measured after the samples were irradiated with X-rays of 100 mGy. The obtained glow curves are shown in Fig. 5. All the samples showed a glow peak around 140 °C. With increasing the concentration of Ca, the glow peak around 140 °C shifted to lower temperature, and the shift like this was also shown in, for example, Cr- [29] and Y-doped [31] MgO transparent ceramics. The 0.01% Ca-doped sample also showed a noticeable broad glow peak around 250 °C. These peaks are typical for MgO transparent ceramics [28–32]. The TSL intensity of 0.01% Ca:MgO was the highest among all the samples.

Figs. 6 and 7 show TSL spectra of Ca:MgO samples measured around 140 °C in the UV–visible and NIR ranges, respectively. Broad and weak emissions appeared over the spectral range of 300–500 nm, and some emissions appeared from 600 to 750 nm. The TSL intensity of 0.01% Ca-

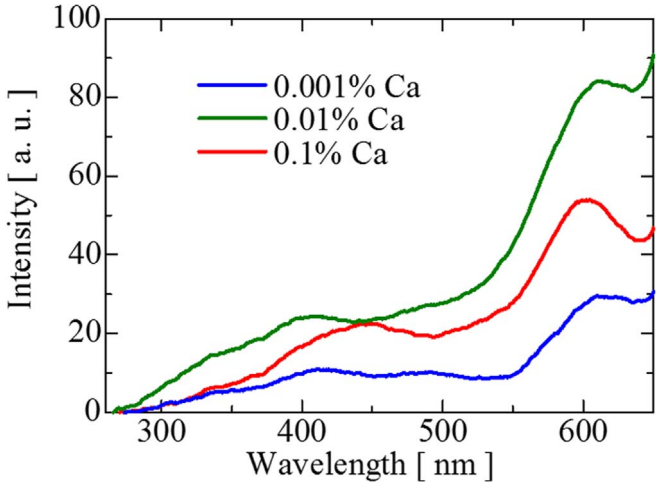

Fig. 6. TSL spectra of the MgO transparent ceramics doped with Ca^{2+} (0.001, 0.01 and 0.1%) in the UV–vis range.

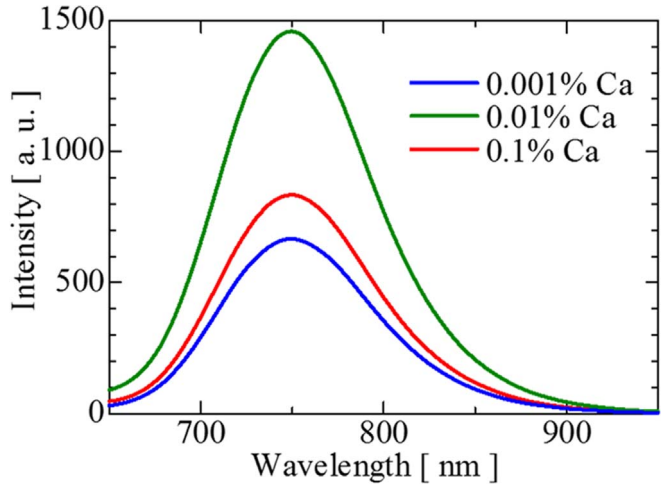

Fig. 7. TSL spectra of the MgO transparent ceramics doped with Ca^{2+} (0.001, 0.01 and 0.1%) in the vis-NIR range.

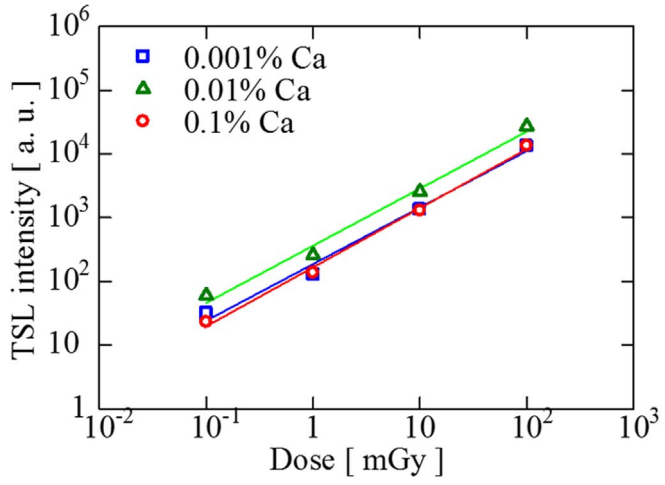

Fig. 8. Dose response curves of the MgO transparent ceramics doped with Ca^{2+} (0.001, 0.01 and 0.1%). Response values are integrated signals of a glow peak observed at 140 °C.

doped sample was the highest among the samples tested. The weak and broad emissions from 300 to 800 nm seem to consist of emissions peaking around 340, 400, 440, 500, 600 and 750 nm, and the emission origins are considered to be due to surface defects, F^+ center, some defects, F center and the interaction of impurities (Cr^{3+}, Mn^{2+} and Fe^{3+}), respectively, according to the literature [11,14,19,42–46,48,49]. Comparing between scintillation and TSL, the emission intensity at 750 nm is not correlated with the doping concentration in the same way; however, it is not necessarily the same because the luminescence processes are different between scintillation and TSL as mentioned in the introduction part. In terms of the TSL intensity, 0.01% is the optimum concentration of doping to enhance the emission at 750 nm. The TSL intensity depends on the number of trapping site, emission efficiency and optical transmittance of host. By Ca-doping, the number of trapping site is considered to be increased due to the mismatch of ionic radii of Mg^{2+} and Ca^{2+}, and the number of emission centers should also increase since the emission centers of MgO are mainly defect centers. On the other hand, transmittance monotonically decreased with Ca concentration (see Fig. 2). Thus, the TSL intensity was the highest for the 0.01% Ca-doped sample as a result of these physical properties balanced out.

In Fig. 8, dose response curves of Ca:MgO transparent ceramics are shown. The dose response curve represents a relationship between TSL intensity and delivered X-ray dose. Here, the TSL signal is an integrated signal of the glow peak at 140 °C. It was confirmed that all the samples exhibited the response correlated linearly with the incident X-ray dose across the dose range from 0.1 to 100 mGy. For higher doses, the signal was too strong to be measured by the instrument. Each dose response curve can be approximated by a power function, which is summarized in Table 1. From the coefficients of determination, the response linearity was confirmed to be better for the samples with higher concentrations of Ca. To our knowledge, among the MgO ceramics reported, the present MgO samples doped with Ca showed the highest TSL sensitivity [23,51,52], and this sensitivity is comparable with commercial personal dosimeters [53]. It is worth mentioning here that the TSL reader used in this research does not have high sensitivity to the emissions around 600 and 750 nm as the detector equipped was a PMT. With a use of photodetectors such as APD, much higher detection sensitivity would be achieved.

Table 1
Approximations for dose responses and coefficient of determinations.

Samples	Approximated equations	Coefficients of determination (R^2)
0.001% Ca	$y = 187.07x^{0.888}$	0.9881
0.01% Ca	$y = 363.33x^{0.897}$	0.9893
0.1% Ca	$y = 166.79x^{0.928}$	0.9962

4. Conclusions

We have synthesized Ca-doped MgO transparent ceramics by the SPS method. Further, we have investigated their optical, scintillation and dosimeter properties. In scintillation and TSL, 0.01% Ca-doped samples showed the highest intensity among the present samples. The TSL glow curve of all the Ca-doped samples showed a glow peak around 140 °C. The TSL dose response curve of the glow peak around 140 °C showed good linearity over a dose range from 0.1 to 100 mGy.

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