Optical, Scintillation and Dosimeter Properties of MgO Translucent Ceramic Doped with Cr³⁺

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Abstract

We have investigated the photoluminescence (PL), scintillation and thermally-stimulated luminescence (TSL) dosimeter properties of MgO translucent ceramic doped with Cr³⁺ ion (0.001, 0.01 and 0.1 %). The ceramic samples were synthesized by a Spark Plasma Sintering (SPS) technique. The broad and sharp emission peaks appeared around 600-850 nm in all the samples. The PL decay time constants of all the samples were a few ms which were on the typical order of Cr³⁺ doped phosphors. As with the PL, the peak resulted from Cr³⁺ ion was detected in the scintillation spectra. The TSL glow curves showed the main peak around 140 °C. The TSL response was confirmed to be linear to the irradiation dose over the dose range from 0.1 to 1000 mGy.

Keywords: translucent ceramic, MgO, Cr, scintillator, dosimeter, TSL

1. Introduction

Accurate measurements of radiation dose, energy and spatial distribution have been demanded, for example, in medical [1], security [2] and also for personal dose monitoring [3-5] applications. Required dosimeter properties depend on applications, but typically suitable sensitivity, dose linearity, energy response and low fading are considered. In addition, if one is to measure a radiation dose absorbed in human body, it is desirable that the effective atomic number (Z_{eff}) of the dosimeter 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 calibration for energy dependence is required ideally. Therefore, for such dosimeter applications, it is preferred for the detector materials to consist of light elements. Dosimeters using an inorganic phosphor are mainly classified in to three types: thermally stimulated luminescence (TSL), optically stimulated luminescence (OSL) and radiophotoluminescence (RPL) dosimeters. Examples of these dosimeter materials used in practice are: Ti and Mg doped LiF ceramics [3], C-doped Al_2O_3 crystalline powder [4] and Ag-doped phosphate glass [5], respectively.

Magnesium oxide (MgO) is a wide band-gap insulator ($E_g = 7.8$ eV) with a rock-salt crystal structure (fcc) under ambient pressure, and the Mg ions occupy the octahedral sites within the anion close-packed structure [6, 7]. In the 1970s, undoped MgO was found to show dosimeter properties. When it was irradiated by X-ray, γ -ray and UV, two TSL glow peaks were found around 90-100 and 140 °C [8-10]. Moreover, it was reported that the emission wavelength appeared at 400 and 710 nm, and the latter is due to contamination of Cr ion. On the other hand, similar studies have been performed for MgO powder doped with isoelectronic impurities and transition metal ions. It was revealed that Cr^{3+} , Mn^{2+} and Ni^{2+} act as emission centers while Fe^{3+} , Co^{2+} and Cu^{2+} act as quenchers in γ -ray induced TSL [11,12].

In addition to the dosimeter properties, the luminescence of undoped MgO has been studied in many material forms including powders, films, bulk single crystals and ceramics [13-22]. In MgO crystals, it has been demonstrated that there are two primary oxygen vacancies, namely F^+ and F color centers, which capture one and two electrons, respectively. Although absorption bands of the F^+ and F color centers are almost the same energy, ~5 eV (~250 nm), they show different photoluminescence (PL) emission bands with the peaks at ~2.3 eV (~500 nm) and ~3.2 eV (~400 nm), respectively [18]. In recent years, with advancement of ceramic fabrication techniques, MgO transparent ceramic was developed [23-27]. It shows phosphorescence (or called long afterglow or persistent luminescence) emission at the wavelength of 390 nm due to the F^+ center acting as a luminescent center [27]. The phosphorescence, in other words, is a form of TSL at room temperature; therefore, the MgO transparent ceramic is potentially applicable for TSL dosimeter if the TSL peak temperature can be somehow increased in order to store the dose signal for a long term without fading.

In this study, we have synthesized a MgO translucent ceramic doped with Cr^{3+} ion (0.001, 0.01 and 0.1 %) by spark plasma sintering (SPS) and studied the dosimeter properties against X-rays. Since the contamination of Cr in MgO raw powder is unavoidable by purification techniques nowadays,

investigations of influences of Cr doping in MgO are important. Further, we have characterized the optical and scintillation properties. Recently, it was pointed out that scintillation and dosimeter are complementarily related in some material systems [28, 29] so investigations of both the dosimeter and scintillation properties are important to understand the luminescence phenomena induced by radiations. In general, SPS is performed in a highly reductive atmosphere; therefore, oxygen vacancies are effectively created, and the dosimeter properties expected to be enhanced.

2. Experiment

MgO transparent ceramic samples were synthesized by an SPS method using Sinter Land LabX-100. Here, a reagent grade of MgO (99.99 %) and Cr₂O₃ (99.99 %) powder was homogeneously mixed. The total mass of the mixture was 0.5 g. The mixture was then loaded in a graphite die and sandwiched by two graphite punches. The sintering temperature was increased from 600 °C to 1500 °C at the rate of 17 °C /min and held for 60 min while applying the pressure of 80 MPa. After the synthesis, wide surfaces of the ceramic sample were polished. In the course of study, the following measurements were carried out for all the prepared samples.

The in-line transmittance was evaluated by using JASCO V670 spectrometer in the spectral range from 190 to 2700 nm with 1 nm step. The PL emission spectrum and the PL decay lifetime monitoring at 410 nm were measured with 280 nm excitation using Hamamatsu Quantaaurus- τ (C11367-04, Hamamatsu) and a short-cut filter (LU0300, Asahi Spectra). **The 280 nm excitation is the shortest wavelength of LED equipped to Hamamatsu Quantaaurus- τ .** In this condition, some leakage from the excitation source would affect the shorter wavelength ranges in the PL spectrum. The X-ray induced scintillation spectrum was measured using our lab-constructed setup. The sample was excited using an X-ray generator in which the applied tube voltage and current were 40 kV and 5.2 mA, respectively. The scintillation emission was guided to either of the following two spectrometers: Andor CCD-based spectrometer (CCD, DU920-BU2NC, Andor; grating, SR163i-UV, Andor) or Ocean Photonics CCD-based spectrometer (QE65Pro). The details of the setup was described previously [30]. The former spectrometer was used for measuring a spectrum in the UV and visible range while the latter was used for the NIR wavelengths. Further, the X-ray induced afterglow was measured using an afterglow characterization system equipped with a pulsed X-ray tube [31], which was designed and constructed by us, and it became commercially available from Hamamatsu as a custom-ordered instrument. An afterglow level was calculated using the formula of one of the famous companies for scintillation detectors in security applications [32]. **After samples were annealed at 1000 °C for 20 minutes, and then left in the dark for a while, the afterglow was measured.** The afterglow is TSL around room temperature and has a significant relation with dosimeter properties.

In order to investigate the properties of MgO ceramic as a TSL dosimetric detector, we have measured

a TSL glow curve using Nanogray TL-2000 [33] after X-ray irradiations with various doses from 0.1 mGy to 1000 mGy. The heating rate used for all the TSL measurements was fixed to 1 °C/s, and the sample was heated from 50 to 300 °C to measure the glow curve. Further, TSL spectrum was measured using an Andor CCD-based spectrometer as mentioned above 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

The synthesized MgO:Cr³⁺ translucent ceramic samples are illustrated in Fig. 1. The top image shows the samples under room light while the bottom image shows those under UV (302 nm) light. As seen in the figure, these samples are visually transparent. It looks like that the PL emission becomes feeble with higher concentration of Cr added.

3.2 Optical properties

Fig. 2 shows the in-line transmittance spectra of MgO samples. For all the samples, at 220 nm and shorter wavelengths, the transmittance is close to zero. These wavelengths are longer than the wavelength of the bandgap of MgO (~7.8 eV=159 nm) [6]. The 0.1 % Cr-doped sample show absorption bands in the visible region. The mechanism responsible for these bands is explained in the literature in terms of crystal field splitting [34, 35]. The octahedral crystal field around the Cr³⁺ ion splits the ⁴F level into a ground state of singlet ⁴A₂ and excited states of triplet ⁴T₂ and ⁴T₁. Also, due to the crystal field, splitting of the free-ion excited state of ²G gives rise to level ²E. The broad absorption bands are due to transitions from ⁴A₂ to the ⁴T₂ (absorption around 665 nm) and ⁴T₁ (absorption around 455 nm) levels.

Fig. 3 represents the PL spectra of the MgO ceramic doped with Cr³⁺ (0.001, 0.01 and 0.1 %) samples under 280 nm excitation. The emission bands appeared around 350 and 410 nm, and the former peak is due to the excitation leakage. Moreover, the broad and sharp emission peaks appeared around 600-850 nm in all the samples. Since emission wavelength agrees with that of trivalent Cr ion in MgO [36-39], this emission should be due to the electron transitions of Cr³⁺ ion. The sharp peaks around 600-850 nm mainly originates from ²E → ⁴A₂ transitions in Cr³⁺ ion placed at Mg²⁺ substitutional site with octahedral symmetry. Another broad peak around 600-850 nm is due to ⁴T₂ → ⁴A₂ transitions in Cr³⁺ ion if the ⁴T₂ level shifts below the ²E as expected for Cr³⁺-Mg vacancy pairs aligned with a MgO[1 1 0] direction [36]. From 0.001 to 0.01 %, the peak intensity around 600-850 nm increased. However, from 0.01 to 0.1 %, the peak intensity around 600-850 nm decreased. The decrease of the peak intensity may be caused by the concentration quenching. The peak at 410 nm is attributed to oxygen vacancies with one electrons (F⁺ center) which was observed in the undoped MgO [18, 27].

The PL decay time constants of the MgO:Cr³⁺ translucent ceramics are shown in Fig. 4. The inset shows the measured PL decay curves. Here, the excitation wavelength is 280 nm and the monitoring

emission wavelength is 750 nm. The decay curves of all the samples are well approximated by a sum of two exponential functions. The obtained faster decay time components of 0.001, 0.01 and 0.1 % Cr-doped samples resulted 870, 355 and 228 μ s, respectively. Possibly, the obtained faster components would be ascribed to $^4T_2 \rightarrow ^4A_2$ transitions of Cr^{3+} ion, and the excitation pulse may also affect. The obtained slower decay time components of 0.001, 0.01 and 0.1 % Cr-doped samples resulted 3.78, 2.02 and 0.95 ms, respectively. These slower components are due to Cr^{3+} emission because the observed values are consistent with typical decay times of $^2E \rightarrow ^4A_2$ transitions [40, 41], and shortening of decay time with Cr^{3+} concentration would be due to concentration quenching.

3. 3 Scintillation properties

Fig. 5 shows the scintillation spectra of MgO translucent ceramic samples doped with Cr^{3+} (0.001, 0.01 and 0.1 %). All the samples showed an emission peak around 600-850 nm due to Cr^{3+} . The emission intensity showed a similar behavior of PL spectra against the Cr concentration. Unlike PL, no F^+ center emission around 410 nm was detected or it was embedded in the tail part of the 350 nm emission. Although the origin of the 350 nm emission is still unclear because it was not detected in typical PL studies, this peak was only observed in OSL spectrum [42]. We think that the origin would relate to **surface (grain boundary) defects** [43], but further investigation will be required.

In Fig. 6, the afterglow levels are shown. The inset shows the afterglow profiles. The obtained afterglow levels of 0.001, 0.01 and 0.1 % Cr-doped samples are 0.17, 0.12 and 0.11 %, respectively. The afterglow levels were in inverse proportion to the Cr concentration.

3. 4 Dosimeter properties

Fig. 7 shows TSL glow curves measured after the samples were irradiated with an X-ray dose of 1 Gy. All the samples showed glow peaks around 85 and 140 °C. These peaks are typical in MgO and is consistent with the previous works reported in the literatures [8-12]. In addition, the more amount of dopant added, the lesser TSL intensity, and the TSL peak temperature slightly shifted to lower temperature. These results were consistent with afterglow results that afterglow level was intense in lower Cr concentration. The activation energy and the frequency factor were derived using the initial rise method [44] on the assumption of first order kinetic [45], and the estimated value was summarized in Table 1. As Cr concentration increase, the activation energy was lower, in comparison with that of undoped MgO in Ref [8] which was evaluated with the same manner. This fact indicates that the trap depth shallowed by Cr-doping. Additionally, the frequency factor values were in inverse proportion to the Cr concentration, and it showed that Cr-doping suppressed the thermal ionization probability from F-centers in MgO. From this analysis, if more pure MgO powder which is not contaminated with Cr is available, the TSL properties of MgO would be improved.

Fig. 8 depicts the TSL spectra around 140 °C. The emission bands were detected around 340 and 600-

850 nm. The TSL peak at 140 °C was by the F^+ center and Cr^{3+} ion. However, it should be noted that emission from the 600-850 nm bands was not accumulated in TSL glow curves (Fig. 7) since the thermal radiation cut filter had a transmittance up to 520 nm was equipped in the instrument. As a result, F^+ center and Cr^{3+} emissions had a same dopant concentration dependence (Fig. 8), and whether Cr^{3+} emission was included in TSL glow curves or not did not affect the discussion.

Fig. 9 shows the relation between the TSL intensity and irradiated X-ray dose, namely the dose response curve. By using the dose response curve, we can calibrate the TSL response to the corresponding dose value in practical uses. The TSL signal used here is the integrated peak intensity at 140 °C in Fig. 7. From our experimental results, the 0.001, 0.01 and 0.1 % Cr-doped MgO transparent ceramics have a linear response against the incident X-ray dose over the dose range of 0.1–1000 mGy. The linear response was confirmed the tested range and calculated coefficient of determinations of 0.001, 0.01 and 0.1 % Cr-doped MgO are 0.9759, 0.9815 and 0.9649, respectively. It is an equivalent sensitivity with commercial personal dosimeters and the highest sensitivity for MgO among the earlier reported works we could find [46-48]. Dosimeter having a linear response is advantageous because it enables us to easily and accurately calibrate the dose.

4. Conclusions

We have synthesized MgO translucent ceramics doped with Cr^{3+} ion by an SPS technique. All the samples were visually transparent. Subsequently, we have studied the optical, scintillation and TSL properties. In the PL and scintillation emissions, the optimum Cr^{3+} concentration turned out to be around 0.01%. The TSL glow curves of all the samples had the main peak around 140 °C. Using this glow peak, the MgO: Cr^{3+} samples had a dose detection ability at least over the range 0.1–1000 mGy Furthermore, the dose response was linear over the dose range tested. Among the present samples, lower Cr concentration was preferable for TSL dosimeter.

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Fig. 1 MgO ceramic doped with Cr³⁺ (0.001, 0.01 and 0.1 %) samples under room light (top) and UV (302 nm) light (bottom).

Fig. 2 Transmittance of MgO transparent ceramic samples doped with Cr³⁺ (0.001, 0.01 and 0.1 %).

Fig. 3 PL emission spectra of the MgO transparent ceramic samples doped with Cr³⁺ (0.001, 0.01 and 0.1 %) under 280 nm excitation.

Fig. 4 PL decay times of the MgO transparent ceramic samples doped with Cr³⁺ (0.001, 0.01 and 0.1 %). The monitoring PL emission wavelength is 750 nm during the excitation at 280 nm. The inset shows the PL decay profiles.

Fig. 5 X-ray induced scintillation spectra of the MgO transparent ceramics doped with Cr³⁺ (0.001, 0.01 and 0.1 %). The inset expands the 300-700 nm wavelength region.

Fig. 6 Afterglow levels of MgO transparent ceramics doped with Cr³⁺ (0.001, 0.01 and 0.1 %). The inset shows the afterglow profiles.

Fig. 7 TSL glow curves of MgO transparent ceramics doped with Cr³⁺ (0.001, 0.01 and 0.1 %) samples. The inset expands from 0 to 0.4 TSL intensity for Cr³⁺ 0.1 % sample.

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Samples	Peak Temperature (°C)	Activation energy (eV)	Frequency factor (s ⁻¹)
Ref [8]	144	1.03	—
0.001% Cr	139	0.29	8.02×10^9
0.01% Cr	130	0.21	2.43×10^7
0.1% Cr	130	0.15	1.62×10^4

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