Dosimeter Properties of MgO Transparent Ceramic Doped with C

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Abstract

We have investigated the photoluminescence (PL), scintillation and thermally-stimulated luminescence (TSL) dosimeter properties of MgO ceramic doped with C ions (0.001, 0.01 and 0.1 %). The samples were synthesized by a Spark Plasma Sintering (SPS) technique. The PL emission peaks appeared around 400 and 750 nm in all the samples. The PL decay time constants at 400 nm were ~10 and ~100 ns which were on the typical order of F^+ center in the undoped MgO. The scintillation emission peaks were detected at 330, 400 and 750 nm under X-ray irradiation. The TSL glow curves showed the ~250 °C peak in 0.1 % C-doped sample. The TSL response was confirmed to be linear to the irradiation dose over the dose range from 0.1 to 1000 mGy. As a result, the sensitivity of MgO was improved by C-doping.

Keywords: transparent ceramic, MgO, C, dosimeter, TSL

1. Introduction

In ionizing radiation detectors, luminescence materials have been used to visualize invisible radiations by visible photons. Among such luminescence materials, scintillators which convert a single high energy ionizing radiation to thousands of ultraviolet-visible photons immediately (Yanagida, 2013a) is the most famous type of the solid state radiation detector and has a wide range of applications such as medical (Yanagida et al., 2010), security (Totsuka et al., 2011). On the other hand, another luminescence type detector, dosimeter stores the absorbed energy for several weeks and release by optical (optically stimulated luminescence, OSL) or thermal (thermally stimulated luminescence, TSL) stimulation. The main application of OSL or TSL device is a personal dosimetry (Mayhugh et al., 1970; McKeever, 2011). Required dosimeter properties depend on applications, but typically suitable sensitivity, dose linearity, energy response and low fading are considered. In addition, 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 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 especially in the human radiation monitor, it is preferred for the detector materials with light elements.

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 (Klein and Hurlbut, 1999; Roessler and Waller, 1967). The luminescence properties of undoped MgO have been studied in many material forms including powders, films, bulk single crystals and ceramics (Chen et al., 1969; Jeffries et al., 1982; Kappers et al., 1970; Martinez-Boubeta et al., 2011; Rosenblatt et al., 1989; Soma et al., 2015; Summers et al., 1983; Uchino et al., 2009; Uenaka and Uchino, 2011; Yanagisawa and Huzimura, 1984). In MgO crystals, Rosenblatt et al. have demonstrated that there are two primary oxygen vacancies, namely F and F^+ color centers, which capture two and one electrons, respectively. Although absorption bands of the F and F^+ color centers are almost at the same wavelength of ~ 250 nm, they show different photoluminescence (PL) bands with the peaks at ~ 500 nm and ~ 400 nm, respectively (Rosenblatt et al., 1989).

From the view point of MgO for radiation detector uses, 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 (Nanto et al., 1975; Sibley et al., 1969; Takeuchi et al., 1975). Furthermore, in the later decades, MgO have been intensively studied for dosimeter applications due to the bioequivalency (Dolgov et al., 2002; Las and Stoebe, 1984; Sathymoorthy and Luthra, 1978; Solomon, 2009; Ziniker et al., 1973). Recently, with advancement of ceramic fabrication techniques, MgO transparent ceramic was developed (Chaim et al., 2004, 2010; Chen et al., 2008; Fang et al., 2004; Wakahara et al., 2013). It shows phosphorescence (or so called long afterglow or persistent luminescence) emission at the wavelength of

390 nm due to the F^+ center acting as a luminescent center (Wakahara et al., 2013). The phosphorescence, in other words, is a form of TSL around 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 order to develop efficient TSL device, we have studied transparent ceramic MgO materials (Kato et al., 2016a, 2016b).

In this study, we have synthesized a MgO transparent ceramic doped with C ions (0.001, 0.01 and 0.1 %) by spark plasma sintering (SPS) and studied the dosimeter properties against X-rays. The C-doping is inspired by C-doped Al_2O_3 crystalline powder used for a personal dosimeter (McKeever, 2011). 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 (Yanagida et al., 2014b, 2016) so investigations of both the dosimeter and scintillation properties are important to understand the luminescence phenomena induced by ionizing 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 %) powder and a carbon powder were homogeneously mixed. A carbon powder was made from shattered graphite die 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 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, the 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 a JASCO V670 spectrometer in the spectral range from 190 to 2700 nm with 1 nm interval. The PL emission spectrum was measured with a 250 nm excitation using FP-8600 (C002361454, JASCO) and a short-cut filter (LU0300, Asahi Spectra). The PL decay lifetime monitoring at 400 nm was measured with 280 nm excitation using Hamamatsu Quantaurus- τ (C11367-04, Hamamatsu). 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 Ocean Photonics CCD-based spectrometer (QEPro). The details of the setup was described previously (Yanagida et al., 2013c). Further, the scintillation lifetime by X-ray irradiation was measured using an afterglow characterization system equipped with a pulsed X-ray tube (Yanagida et al., 2014a). The system is commercially available from Hamamatsu as a custom-ordered instrument. The applied voltage to the pulsed

X-ray source was 30 kV, and the system offers the timing resolution of ~1 ns.

In order to investigate the properties of MgO ceramic as a TSL dosimeter device, we have measured a TSL glow curve using a Nanogray TL-2000 (Yanagida et al., 2013b) 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 490 °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 doped with C (0.001, 0.01 and 0.1 %) samples are illustrated in Fig. 1. The top photograph shows the samples under the room light while the bottom one shows those under UV (254 nm) light. These samples are visually transparent except for 0.1% C-doped sample. It looks like that the 0.001% C-doped sample emits yellowish green color and the 0.01% C-doped sample emits blue color.

3.2 Optical properties

Fig. 2 shows the in-line transmittance spectra of MgO samples. For the 0.001 and 0.01 % C-doped samples, the transmittance is close to zero at 220 nm. These wavelengths are longer than the wavelength of the bandgap of MgO (~7.8 eV=159 nm) (Roessler and Waller, 1967). No particular absorption bands were observed, and the transmittance became worse by dense C-doping. In addition, transmittance grows with an increasing wavelength from 0 up to 50% in the spectral range 450-2500 nm, and this is a typical tendency of Mie scattering. When the doping concentration increased, the number of scatterers also increased.

Fig. 3 represents the PL spectra of the MgO doped with C (0.001, 0.01 and 0.1 %) samples under 250 nm excitation. The emission peaks appeared around 400 and 750 nm, and the origins of these peaks were assigned in previous reports (McKeever et al., 1995; Rosenblatt et al., 1989; Takeuchi et al., 1975; Uenaka and Uchino, 2011); therefore, we think that 400 nm peak may be due to F⁺ center and 600-900 nm peak may be caused by Cr³⁺ ions and Fe³⁺ impurity. It is known that Cr and Fe impurity are typically contaminated when we buy MgO raw powder. Among present samples, the peak intensities of 0.01% C-doped sample was the highest since the observed photoluminescence intensity depended on the transparency and a number of defects (F⁺ centers) due to a charge imbalance of C⁴⁺ and Mg²⁺.

The PL decay profiles of the MgO doped with C (0.001, 0.01 and 0.1 %) are shown in Fig. 4. Here, the excitation wavelength is 280 nm and the monitoring emission wavelength is 400 nm. The decay curves are well approximated by a sum of three exponential functions. The obtained faster decay time components of 0.001, 0.01 and 0.1 % C-doped samples were 2.8, 2.0 and 3.5 ns, respectively. This would be due to the instrumental response because the timing resolution of the instrument is comparable (around 1 ns).

Although we tried to deconvolute the instrumental response, some residual signals were still present since they showed the phosphorescence and it was technically difficult to fully deconvolute in such phosphorescent materials. The obtained second faster decay time components of 0.001, 0.01 and 0.1 % C-doped samples resulted 14.7, 8.4 and 15.4 ns, respectively. The obtained slower decay time components of 0.001, 0.01 and 0.1 % C-doped samples were equal to 161, 143 and 153 ns, respectively. These constants are consistent with the typical ones of F^+ center emissions in MgO (Kato et al., 2016a).

3. 3 Scintillation properties

Fig. 5 depicts the scintillation spectra of MgO transparent ceramic samples doped with C (0.001, 0.01 and 0.1 %). All the samples showed a broad emission peaks around 400 and 750 nm. These peak wavelengths are consistent with the ones seen in the PL spectra under 250 nm excitation; therefore, the origins of 400 nm would be attributed to the F^+ center and 750 nm peak may be due to unexpected impurity Cr^{3+} and Fe^{3+} ions.

Moreover, the emission peak was also observed at 330 nm and it is said that its origin related to surface (grain boundary) defect (Gu et al., 2008). As with the PL spectra, the peak intensities of 0.01% C-doped sample is the strongest among all the samples. Compared with the scintillation spectrum of the undoped MgO transparent ceramic reported by Kato et al. (2016a), 330 and 400 nm peak intensities increased while 750 nm peak intensity decreased with C-doping. The addition of carbon can generate F^+ centers by reducing reaction, and it should also generate some structural defects (as indicated by significant decrease of transmittance). The F^+ centers effectively act as electron trapping centers while the defect centers may act as radiative recombination center and/or charge trapping center. Once electrons and holes are generated by X-rays, with a large number of these centers, it is difficult for charges to make it to a small number of Cr^{3+} ions to emit whereas these are much more probably to be captured or recombine at these centers. Therefore, for these reasons, we think that the emission intensity by Cr^{3+} is suppressed by an addition of carbon.

The X-ray induced scintillation decay time profiles are shown in Fig. 6. It can be decomposed into three exponential functions. The primary decay time components 0.001, 0.01 and 0.1 % C-doped samples were 1.3, 1.0 and 0.9 μ s, respectively. The secondary decay time components 0.001, 0.01 and 0.1 % C-doped samples were 3.4, 3.2 and 3.5 μ s, respectively. The tertiary decay time components 0.001, 0.01 and 0.1 % C-doped samples were 24.2, 18.5 and 17.7 μ s, respectively. We inferred that these components would be due to F^+ center and surface defect. The decay lifetimes are much slower than those of PL, and we could not observe significant fast signal when we tested the fast timing range. This difference is often observed in the relation between PL and scintillation decay time (Kato et al., 2016a).

3. 4 Dosimeter properties

Fig. 7 shows TSL glow curves measured after the samples were irradiated with an X-ray dose of 100 mGy. All samples showed glow peaks around 140, ~250 and 390 °C. In addition, the 0.1 % C-doped sample showed glow peaks around 340 °C. Among these four peaks, three peaks at 140, ~250 and 340 °C are typical in MgO transparent ceramic and is consistent with the previous our work about the undoped MgO (Kato et al., 2016a). It is implied that C-doping generated the new trapping center because the glow curve structure changed remarkably with increasing the dopant amount. The activation energy and the frequency factor were derived using the initial rise method (McKeever, 1985) on the assumption of first order kinetic (Fasoli et al., 2010). The estimated values are summarized in Table 1. As C concentration increases, the activation energy and the frequency factor for 140 °C peak was higher, in comparison with that of undoped MgO. This fact indicates that the trap depth becomes deeper by C-doping, and the TL occurs by common thermal ionization processes. Throughout these experiments, it can be said that C-doping would improve the TSL properties of MgO.

Fig. 8 depicts the TSL spectra around 140 °C. The emission bands were detected around 330, 400 and 750 nm. Likewise in the scintillation spectra, 330 and 400 nm peak intensities increased by C-doping in comparison with the undoped MgO transparent ceramic (Kato et al., 2016a). In addition, the peak intensities around 750 nm are inversely proportional to dopant amount, by contrast, the TSL peak intensities at 340 nm of 0.01% C-doped sample is the strongest among all the samples. It should be noted that emission from the 750 nm bands was not accumulated in TSL glow curves (Fig. 7) since the thermal radiation cut filter was equipped in the instrument, and it was a typical detector setup in practical applications. Therefore if we can prepare more suited readout system, the detection limit can be improved.

Fig. 9 indicates 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 signals used here are the integrated peak intensities at 140, 260, 340 and 390 °C of 0.1 % C-doped sample and the integrated peak intensities at 140, 240 and 390 °C of 0.001 the and 0.01 % C-doped samples in Fig. 7. The dose range was tested from 0.1 to 1000 mGy. The ~250 and 390 °C peaks have no linear response to the tested dose range. The 140 °C peaks of all the samples have a linear response to the dose range of 0.1–100 mGy. Since the 140 °C peak was quite luminous, we could not evaluate higher dose due to the upper detection limit of the instrument. Compared with dose response curve at 140 °C of the undoped MgO (Kato et al., 2016a), the TSL intensities of C-doped samples increased. Hence, it can be said that the dosimeter property is improved by C-doping. On the other hand, the ~250 and 340 °C peak of 0.1 % C-doped sample has a linear response to the dose range of 0.1-1000 and 1–1000 mGy, respectively. It is an equivalent sensitivity with commercial personal dosimeters and the highest sensitivity for MgO among the earlier reported works we could find (Boss et al., 2006; Kvatchadze et al., 2007; Soliman, 2009). Dosimeter having a linear response is advantageous because it enables us to easily and accurately calibrate the dose. As a result, the ~250 °C peak of 0.1 % C-doped sample has the highest-performance dosimeter properties since it has both dose linearity and the lowest fading among these peaks.

4. Conclusions

We have synthesized MgO transparent ceramics doped with C ions by an SPS technique. 0.001 and 0.01 % C-doped samples were visually transparent. Subsequently, we have studied the optical, scintillation and TSL dosimeter properties. The TSL glow curves of all the samples had the main peak around 140 °C. Furthermore, TSL peaks were also detected around ~250 and 390 °C in all samples and the 0.1 % C-doped sample showed additional glow peaks around 340 °C. Using these glow peak, dose response curves were measured. As a result, the peak at ~250 °C of 0.1 % C-doped sample showed the most high-performance throughout this work.

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Fig. 1 MgO ceramic doped with C (0.001, 0.01 and 0.1 %) samples under room light (top) and UV (254 nm) light (bottom).

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

Fig. 3 PL emission spectra of the MgO samples doped with C (0.001, 0.01 and 0.1 %) under 250 nm excitation.

Fig. 4 PL decay profiles of 0.001 (top), 0.01 (middle) and 0.1 (bottom) % C-doped samples. The monitoring PL emission wavelength is 400 nm during the excitation at 280 nm.

Fig. 5 X-ray induced scintillation spectra of the MgO transparent ceramics doped with C (0.001, 0.01 and 0.1 %).

Fig. 6 Scintillation decay profiles of 0.001 (top), 0.01 (middle) and 0.1 (bottom) % C-doped samples.

Fig. 7 TSL glow curves of MgO transparent ceramics doped with C (0.001, 0.01 and 0.1 %) samples. The inset expands 150-490 °C regions.

Fig. 8 TSL spectra measured at 140 °C for the undoped and C-doped samples.

Fig. 9 Dose response curves of the MgO transparent ceramic samples doped with C (0.001, 0.01 and 0.1 %). The TSL signal used top figure is the peak intensity of 0.1 % C-doped sample observed at ~250 and 340 °C. The TSL signal used bottom figure is the peak intensity of undoped and C-doped samples observed at 140 °C.

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Dopant amount (%)	Peak temperature (°C)	Activation energy (eV)	Frequency factor (s⁻¹)
undoped	140	0.44	1.9×10^{15}
0.001	142	0.54	4.7×10^{18}
0.01	137	0.57	4.0×10^{20}
0.1	140	0.47	1.5×10^{16}
0.1	250	0.81	1.7×10^{15}
0.1	340	0.95	4.8×10^{13}

Table. 1 The activation energy and the frequency factor of undoped and C-doped MgO.