Performance characterisation of a real-time fiber dosimetry system using radiophotoluminescent glasses

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We report the first demonstration of a real-time fiber-optic-coupled dosimetry system using Ag-activated phosphate glasses based on radiophotoluminescent (RPL) phenomena. The performance characterisation using real-time fiber RPL glass dosimetry is compared with that of real-time fiber plastic scintillation dosimetry. The inevitable build-up phenomena occurring in Ag-activated RPL glasses both during and after X-ray exposure were measured. The real-time RPL curves for the X-ray exposure region and the build-up region are analytically evaluated and fitted to the second-order polynomial functions and the sum of three exponentials, respectively. In addition, some resolved problems of real-time fiber RPL glass dosimetry are discussed. © 2018 The Japan Society of Applied Physics

1. Introduction

Ag-activated phosphate glass is the most widely known radiophotoluminescent (RPL) material1–5 and can be used in not only personal, environmental, and clinical dosimeters but also two- and three-dimensional (2D and 3D) dose imaging detectors,6–8 fluorescence nuclear track detectors (FNTDs),9 and the visualisation of micropatterns in Ag-activated glass written using a focused MeV light ion beam.30 Accumulating passive RPL glass dosimeters are based on radiation-induced, optically active, Ag-related atomic-scale color centers; therefore, the ultimate intrinsic spatial resolution of the dosimeters is several nanometres.5

Various types and shapes of Ag-activated RPL glass dosimeters have been developed for various applications. Among them, a newly developed RPL glass dosimeter (Chiyoda Technol Dose Ace) is miniaturized and has a cylindrical shape with four different models in combination with/without identification (ID) and with/without an energy compensator filter. The models are GD-301, GD-302M (with ID), GD-351 (with a filter), and GD-352M (with ID and filter).9 The GD-302M and GD-352M have a length of 12 mm and a diameter of 1.5 mm, while GD-301 and GD-351 have a length of 8.5 mm and a diameter of 1.5 mm. The GD-301 and GD-302M without Sn filters are used to measure the dose of high-energy photons, such as in radiotherapy, while the GD-351 and GD-352M with a Sn filter in the capsule can be used for measuring the dose of low-energy photons, such as in diagnostic radiography.3–6 RPL glass dosimeters have been recognized as possessing desirable properties, including a high spatial resolution, long-term stability against fading, a linear response across a wide dose range, uniformity/batch homogeneity, high sensitivity, non-destructive readout capability, and good reproducibility, for in vivo dosimetry systems.1,3,5,10,11

Various optical fiber real-time dosimetry systems have also been demonstrated, providing both accumulated absorbed dose and dose rate using optically stimulated luminescence (OSL) and scintillation and radioluminescence (RL), respectively. To facilitate real-time dosimetry using OSL and RL, Al2O3:C,12–14 BeO,15,16 KBr:Eu,17,18 RhMgF3:Eu,19 SiO2:Ge fiber,20 SiO2:Cu fiber,13,21 SrS:Ce, Sm,22 SiO2:Ag,23 and LiMgPO4:Eu,Sm,R24 have been developed as potentially useful medical dosimetry materials. In addition, as real-time fiber dosimetry using scintillation and RL, various types of plastic scintillators25–28 and CsI(Tl) crystals29 have been extensively studied for the clinical demands of real-time in vivo patient dosimetry applications.

In this work, instead of OSL and scintillator dosimeters, a commercially available Ag-activated phosphate glass based on RPL phenomena is first used in real-time dose dosimetry (GD-302M). First, the transient state of orange and blue RPL dosimetry for three measurement sequences. At the same time, an inevitable build-up phenomenon occurring in Ag-activated RPL glasses during and after X-ray exposure is studied. Third, the accumulated real-time RPL curves for the X-ray exposure and the build-up regions are fitted to the second-order polynomial functions and the sum of three exponentials, respectively. Finally, the capabilities of Ag-activated glasses in real-time RPL dosimetry are discussed.© 2018 The Japan Society of Applied Physics

2. Experimental procedure

2.1 Samples

In this study, the GD-302M dosimeter without a Sn filter in the capsule was used. The weight composition of the material was the same as that of the FD-7 in GD-450 (Asahi Techno Glass), namely, 31.55% P, 51.16% O, 6.12% Al, 11.00% Na, and 0.17% Ag. The mass density, photon effective atomic number, and refractive index of this material are 2.61 g/cm³, 12.04, and 1.52, respectively. In general, RPL glass dosimetry requires a 30 min preheating at 70–100 °C to accelerate the build-up1–3 (i.e., the RPL center concentration, especially attributed to Ag2+ centers, increases slowly as a function of elapsed time both during and after irradiation). Moreover, a 30 min annealing at 400 °C is necessary to erase the stable Ag0 and Ag2+ color centers for reuse.

2.2 X-rays and calibration for absorbed doses

Soft-X-ray irradiances were performed using an X-ray unit
(RTW MCB 65C-0, 2× and Spellman MNX50P50) with a molybdenum (Mo) anode target with the Be window operating at a tube voltage of 40 kV and a current ranging from 1.0 to 0.2 mA. The Ag-activated glasses were placed approximately 20 cm from the tube, and the absorbed doses delivered to the samples were up to 100 Gy at room temperature. The irradiated X-ray beam diameter was 5 mm. A radiochromic film (Ashland RTQA2) was used for the alignment and position verification of the X-ray radiation field.

A calibration for the absorbed dose and dose rate was performed to use a plastic scintillator (Saint-Gobain BC-490) with an optical fiber dosimeter (Acro Bio MIDSOF). Further details of the system are described elsewhere. The calibration coefficient of the scintillation dosimeter was determined using combinations of a Ramtec 1500B electrometer (Toyo Medic) and an RC6M ionization chamber dosimeter (Radcal) for the X-ray unit under the same conditions in this study. The calibration coefficient of variation (CV) was within 0.89% for the scintillation dosimetry in the effective energy range of 15 to 17 keV. Following the process, the real-time RPL dosimetry system was also calibrated with scintillation dosimetry.

### 2.3 RPL real-time dosimetry system and time-resolved optical spectra

The heart of the real-time RPL dosimetry system consists of a dichroic mirror, a miniaturized GD-302M rod dosimeter, a silica optical fiber, and a UV laser. The pulse duration of the excitation UV laser was less than 5 ns (FWHM) at a repetition rate of 1 kHz for a pulse energy of 1 µJ at the front position of the glass dosimeter. For RPL signal detection, a combination of a photonic multichannel analyser (Hamamatsu Photonics PMA-12) and a delay/pulse generator (Stanford Research Systems DG535) is used, as shown in Fig. 1.

The dichroic mirror is capable of reflecting wavelengths from 325 to 404 nm and transmitting wavelengths from 415 to 850 nm (Edmund Optics 86-330). Reflected light from a high-repetition-rate Q-switched laser (Spectra Physics Explorer One) at 349 nm was coupled with the core of a 600 µm silica optical fiber of 2 m length with a numerical aperture (NA) of 0.22. The RPL signal emitted by the UV light is carried back from the dosimeter via the same silica fiber and transmitted through the dichroic mirror. The blue and orange RPL signals are collected by a fiber adapter (Hamamatsu Photonics A9607) and guided towards the multichannel analyser via a 0.2-NA fiber probe. Such an arrangement provides useful measurements of both the real-time dose operation and time-resolved optical spectra.

In addition, a variable attenuator (Sigmakoki VBS-50S03-1-355) and a mechanical shutter (Sigmakoki SSH-S and SSH-C2B) were also used to adjust the light intensity of a laser and to control remotely the turn on/off of laser light, respectively. Computer-assisted software developed in the laboratory was used to precisely control the real-time RPL dosimetry system, including the excitation laser, OMA-12, DG535, and the mechanical shutter.

### 3. Results and discussion

#### 3.1 Time-resolved analysis: Gate time dependence

The time-resolved RPL spectra were measured in the wavelength range from 400 to 850 nm for a variable gate time from 10 ns (minimum temporal resolution) to 500 µs using a PMA-12 analyser equipped with an image intensifier (II), as shown in Fig. 2. Before measuring, each GD-302M glass rod dosimeter was heated to suppress the build-up kinetics after X-ray irradiation with an absorbed dose of 10 Gy. The exposure time for excitation light was fixed at 500 ms in this study; therefore, each spectrum is representative of the accumulation results of 500 pulses because of the 1 ms period of the excitation laser.

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**Fig. 1.** (Color online) Schematic view of the real-time fiber RPL glass dosimetry system.

**Fig. 2.** (Color online) A set of time-resolved RPL spectra of X-ray-irradiated Ag-activated glass after UV laser irradiation at 349 nm for different gate times. Shown in the inset are the accumulated RPL intensity as a function of gate time.
A set of broadband spectra that peaked at 450 and 650 nm were observed. The former band (the so-called “blue RPL” signal) was attributed to the electron-trapped Ag\(^0\) centers, whereas the latter band (the so-called “orange RPL” signal) was attributed to the hole-trapped Ag\(^{2+}\) centers.\(^{1,4,32}\) Such blue and orange RPL signals were simultaneously emitted at an excitation wavelength of 349 nm. The inset shows the integrated RPL intensity as a function of the gate time. Note that the vertical axis shows the integrated RPL area intensity in the wavelength range of 400–500 nm for the blue RPL and in the range of 600–700 nm for the orange RPL. Each curve reflects the luminescence decay time of the blue RPL with a fast lifetime of 4.5 ns and the orange RPL with a slow lifetime of 2.3 \(\mu\)s.\(^{4}\) The intensity of the blue RPL rapidly reached a saturation level of approximately 30 ns, whereas that of the orange RPL progressively reached a saturation level of approximately 20 \(\mu\)s.

3.2 Time-resolved analysis: Exposure time dependence

Figure 3 shows the integrated orange RPL area intensity in the wavelength range of 600–700 nm (see the inset) as a function of the exposure time from 20 to 2000 ms for excitation light. The gate time was fixed at 2 \(\mu\)s; therefore, only the orange RPL signal was detected. Before measuring, each GD-302M glass rod dosimeter was also heated after X-ray irradiation with a dose of 10 Gy. The relationship between the accumulated RPL intensity and exposure time was almost linear, with an \(R^2\) of 0.999 for the GD-302M.

3.3 Minimum measurable dose

The low-dose measurement ability of a dosimeter is usually of great significance in a variety of fields. To confirm the minimum measurable dose of the real-time RPL dosimeter, the GD-302M glass rod dosimeter was subjected to \(^{137}\)Cs gamma ray (662 keV) irradiation performed at the Oarai Research Center, Chiyoda Technol Corporation, at room temperature and given doses of 1, 100, and 1000 mGy, as shown in Fig. 4. An acrylic phantom was placed 50 cm from a \(^{137}\)Cs source, which is the location of the GD-302M glass rod dosimeter. The vertical axis shows the integrated orange RPL intensity in the wavelength range of 600–700 nm. The result suggests that the minimum measurable dose was approximately several tens of mGy in the case of our constructed RPL real-time dosimetry system. In addition, note that the RPL peak intensity ratio of blue peaking at 450 nm to orange peaking at 650 nm luminescence after X-ray irradiation in Fig. 2 and gamma ray irradiation in Fig. 4 was significantly different from each other. Such behaviors were also observed under various types of radiation, including alpha particles,\(^{33}\) heavy charged particles (HCPs),\(^{34}\) and fs laser pulses\(^{35}\) at room temperature.

3.4 Timing chart for real-time RPL dosimetry

The timing chart for a real-time RPL measurement is represented schematically in Fig. 5. The mechanical shutter inserted in the excitation laser beam path is turned on and off at regular intervals in this study, with the on period being 0.5 s and the off period being 1.5 \(\text{s}\) in duration (hereafter, 0.5 s on/1.5 on). During the on period of 0.5 s, the dosimeter was excited by 500 pulse trains with a period of 1 ms at a wavelength of 349 nm. As a result, the absorbed doses can be obtained by integrating over the entire orange RPL areas between 600 and 700 nm. During the off period of the laser, in the case of the real-time scintillation dosimetry system, the RL signal can also be detected, which is generally proportional to the dose rate.\(^{25–28}\) However, in the case of the real-time RPL glass dosimetry system, the RL signal during the off period of the laser could not be detected owing to a much lower intensity level.
3.5 Experimental results of real-time RPL dosimetry

Figure 6(a) shows the relationship of the integrated orange RPL signal including pre-dose (PD) and X-ray exposure versus elapsed time measured using real-time RPL glass dosimetry, and Fig. 6(b) shows the absorbed dose (using scintillation) and dose rate (using RL) versus elapsed time under the same soft X-ray exposure. In the case of real-time scintillator dosimetry, there are two types of luminescent processes, namely, scintillation and RL, occurring in the scintillators: the scintillation signal occurring during optical excitation either during or after X-ray exposure is proportional to the absorbed dose and has been mostly used to measure radiation dose in real-time measurements. On the other hand, the RL signal occurring during X-ray exposure can provide information on the dose rate of the radiation field. In this work, such two types of luminescent phenomena are clarified as follows: the former is related to the stimulated recombination of electron–hole pairs created within the dosimeter and trapping centers either during or after exposure, while the latter is the spontaneous luminescence and is related to the prompt recombination of radiation-induced electron–hole pairs within the dosimeter during exposure.

In the case of the RPL dosimeter, similar processes are also observed, i.e., RPL and RL, either during or after exposure. If the dosimeter is stimulated during exposure, the prompt RL signal has to be taken into account and subtracted from the RPL signal. In this case, both RPL and RL during exposure will be observed simultaneously, but these two contributions could not separate owing to a much lower intensity of the RL signal. Therefore, the dose rate of real-time RPL dosimetry is estimated to use the light output of the plastic scintillation dosimeter for comparison.

The RPL signal was accumulated for 500 ms, while the plastic scintillation dosimetry collected signals for a period of 1 s. Both the integrated RPL intensity in real-time RPL dosimetry and absorbed dose in real-time scintillation dosimetry increased almost linearly with elapsed time for 1 h of X-ray irradiation. In the case of scintillation dosimetry, the total absorbed doses up to 87.6 Gy and the average dose rate of 1.46 ± 0.015 Gy min⁻¹ were measured and evaluated.

After X-ray irradiation with a total dose of 87.6 Gy, the integrated RPL intensity was also recorded in the sequence 0.5 s on/59.5 s off (i.e., every 1 min) for 5 h, as shown in Fig. 7. The RPL signal intensity gradually increased with elapsed time. This phenomenon is representative of a characteristic RPL build-up curve of Ag-activated phosphate glass, i.e., after X-ray irradiation, some holes are captured by the PO₄ tetrahedron at the beginning of the migration, and then the holes produce stable Ag²⁺ centers due to interactions with Ag⁺ ions over time. Several researchers have reported on the build-up kinetics including the Ag concentration, base composition, and fading. Such a build-up curve of the orange RPL attributed to the trapped hole Ag²⁺ centers was fitted to the sum of three exponentials with the form \( A_i \exp(-t/i) + C \) plus a constant, in which \( i \) indicates the component (i = 1–3). The fitted curve indicates that if RPL glass dosimetry was operated without preheating, the RPL signal intensity would not completely stabilize for a long time after irradiation. Using the fitting equations, it will take at least approximately 20 h to reach a saturation level of approximately 1.1 × 10⁶ counts at a room temperature of 22°C. On the other hand, the blue RPL peaking at 450 nm attributed to the trapped electron Ag⁰ centers can form and grow quickly at the beginning stage after X-ray irradiation, although the intensity of the blue RPL signal is much lower than that of the orange RPL signal, as shown in Fig. 2. This finding is currently being explored.
enhance the rapid growth of the RPL centers using a conventional technique, the glass dosimeter was subjected to preheating at 70 °C for 30 min for comparison. As a result, the integrated RPL intensity after the preheating increased approximately twofold, as shown on the right-hand side of the figure.

Figure 8(a) shows the accumulated orange RPL signal using real-time RPL glass dosimetry, and Fig. 8(b) shows the absorbed dose and dose rate using scintillation and RPL signals, respectively, under the same X-ray irradiation. As shown in Fig. 8(b), the absorbed dose was accumulated for 1 h at a dose rate of 1.46 Gy min$^{-1}$, and then the shutter was off for 1 h, maintaining a dose of 87.6 Gy, and this process was repeated. The same sequence was also applied to the RPL glass dosimetry, as shown in Fig. 8(a). In the case of the RPL dosimetry, the orange RPL intensity decreased exponentially despite the second X-ray exposure with a dose of 87.6 Gy. In general, for a typical glass size (8.5 × 8.5 × 1.5 mm$^3$) and type of reader, the RPL peaks at about 30–50 Gy and decreases with further radiation. However, with a small dosimeter such as a glass rod, where the readout geometry describes a UV path length of only 1 mm, less fluorescence suppression is observed and the response may be linear up to 10$^3$ Gy.

As described in Ref. 9, in the case of GD-302M glass rod dosimetry, the dose ranges are divided into two categories, low-dose linearity range (10 μGy–10 Gy) and high-dose linearity range (1–500 Gy). A commercially available reader (Dose Ace FGD-1000) automatically distinguishes the dose range according to a different readout magazine. In the case of high-dose measurements, the RPL signal emitted by a UV laser is collected via a 600-µm-diameter pinhole in the capsule at a depth of approximately 1 mm below the front surface of the GD-302M glass dosimeter.

In our constructed real-time RPL glass dosimetry system, a UV excitation beam at 349 nm was focused at the same depth of nearly 1 mm below the front surface of the dosimeter and then the RPL signal, which is proportional to the absorbed dose, was collected with a 600-µm-core-diameter silica fiber. As a result, it was possible to measure a high-dose exceeding 100 Gy using a combination of the GD-302M rod glass dosimeter and a silica 600 µm optical fiber.

Figures 9(a) and 9(b) show the real-time RPL signal and the corresponding scintillation signal as a function of elapsed time, respectively, after the following sequence: 3 min off/15 min on at a 1.46 Gy min$^{-1}$ dose rate, 10 min off/15 min on at a 0.86 Gy min$^{-1}$ dose rate, 10 min off/15 min on at a 0.28 Gy min$^{-1}$ dose rate, and 3 min off. The RPL signal of the build-up region is not depicted in Fig. 9(a). Different dose rates from 1.46, 0.86, and 0.28 Gy min$^{-1}$, in decreasing order, were obtained to change the tube current of the X-ray source from 1.0, 0.6, and 0.2 mA, respectively. The results indicate that the total absorbed doses are proportional to the product of the dose rate and elapsed time, as shown in Fig. 9(b). In the case of the corresponding real-time RPL dosimetry, the absorbed dose at the first step at the 1.46 Gy min$^{-1}$ dose rate
Gy min doses, dose rate, and background signal were 42.2 Gy, 1.41 \text{ Gy min}^{-1} \text{ dose rate}, and third step (0.28 Gy min^{-1} dose rate), the absorbed doses decreased exponentially, as shown in Fig. 8(a). These results suggest that the absorbed doses cannot follow a quick response to on/off switching operations accurately.

Figure 10(a) shows the accumulated orange RPL intensity as a function of elapsed time. Three successive processes, namely, X-ray exposure containing the PD, build-up, and fading of the real-time RPL glass dosimeter, are performed to investigate the degrees of these effects. Each process was measured as follows: the PD (for 5 min) and X-ray exposure (for 30 min) regions were in 2 s intervals (0.5 s on/1.5 s off), the build-up (for 30 min) region was in 60 s intervals (0.5 on/59.5 s off), and the fading (for 30 min) region was in 2 s intervals (0.5 s on/1.5 s off). The corresponding data for the same processes were collected using real-time scintillation dosimetry, as shown in Fig. 10(b). The total absorbed doses, dose rate, and background signal were 42.2 Gy, 1.41 Gy min^{-1}, and 2 mGy min^{-1}, respectively.

The experimental real-time data during X-ray exposure were fitted to second-order polynomial functions, as shown in Fig. 10(c). In this case, the parameter values are as follows: intercept = 6.02 × 10^4, \( a = 2261.4 \), and \( b = 17.2 \) \( (R^2 = 0.996) \).

After X-ray irradiation, as described in Fig. 7, the build-up curve of the orange RPL was fitted to the sum of three exponentials of the form \( A \exp(-c \times t) + D \times t \), where \( a \) and \( b \) are constants. In this case, the parameter values are as follows: \( a = 5.87 \times 10^6 \), \( d = -0.068 \) \( (R^2 = 0.979) \). These results indicate that the build-up and fading effects simultaneously occur both during and after X-ray irradiation, and as a result, the RPL response is superimposed by these factors.

From the above-mentioned discussion, it is considered that the marked reduction in the RPL intensity during the second X-ray exposure region for high dose X-ray irradiation up to 100 Gy, as shown in Fig. 8, may not be the saturation effect of the Ag^{2+} color centers but the fading effect due to the high-repetition rate ns-excitation-laser pulses.

As Ag-activated phosphate glasses are used as real-time RPL dosimetry, such build-up behavior cannot be avoided. If other RPL materials, such as LiF,\(^{41,42}\) \( \text{Al}_2\text{O}_3:\text{C,Mg},^{43,44}\) \( \text{CsBr}:\text{Sm},^{45} \) and \( \text{BaF}_2-\text{Al}_2\text{O}_3-\text{B}_2\text{O}_3: \text{Sm} \) glass ceramics\(^4^{6}\), with much lower build-up effects were used, it would be possible to realize accurate real-time RPL dosimetry.

4. Conclusions

The data obtained in this study led to the following conclusions.

1. A real-time fiber dosimetry system using Ag-activated phosphate glasses based on RPL phenomena was demonstrated for the first time.

2. The performance characterisation, such as the build-up and fading effects, of the real-time RPL glass dosimetry, was compared with that of a real-time plastic scintillation dosimetry.

3. The experimental real-time results suggested that the build-up and fading effects are superimposed both during and after X-ray irradiation, and these effects represent major difficulties in applying RPL glasses to real-time RPL dosimetry.

4. To realize a reliable real-time RPL dosimetry, a quick response to on/off switching operation, measurable dose rate, and simple RPL response without build-up are necessary.

5. Although the real-time RPL curve for the build-up region is fitted to the sum of three exponentials, the physical meaning of each component is now being elucidated.

6. From the viewpoint of materials, the main factors affecting the build-up, such as the optimisation of Ag concentrations, chemical composition in glasses, and the use of other RPL materials without build-up, must be considered.
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