Structure and luminescence properties of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ synthesized by solid-state reaction method

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Abstract. $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ material used in dosimetry field was synthesized by solid-state reaction method. The scanning electron microscopy image showed that the material exhibits porous surface with a very large surface area. The photoluminescence spectrum confirmed energy transfer process from Gd^{3+} ions to Tb^{3+} ions, and the luminescence of this material was entirely due to Tb ions. The high luminescent intensity at 542 nm was perfectly consistent with the sensitive wavelength range of photomultiplier tubes in the thermoluminescence (TL) reader. The TL-glow curve was very simple with the temperatures of the main peak in the range from 200 to 230°C, and the glow-curve shape was suitable for dosimetry purposes. The TL intensities of $\text{K}_2\text{GdF}_5:10\text{ mol\% Tb}^{3+}$ was higher than that of common $\text{CaSO}_4:\text{Dy}^{3+}$ dosimeters. The estimation on available application of the prepared material in neutron dose measurement was carried out.

Keywords. $\text{GdF}_5:\text{Tb}^{3+}$; $\text{Gd}^{3+}-\text{Tb}^{3+}$ ion pair; energy transfer; neutron dosimetry.

1. Introduction

Currently, the transfer of energy between rare-earth ions has been extremely significant in various applications, including optical devices, monitoring X-ray and recording the radiation dose. The gadolinium host materials have also been investigated for doping the rare-earth ions into the structure, with dopants like Sm^{3+} , Pr^{3+} , Tb^{3+} , Er^{3+} ions, etc. [1–6]. Among them, $\text{Gd}^{3+}-\text{Tb}^{3+}$ ion pairs have special properties, which are widely used in optical applications. For the La^{3+} rare-earth database the emission [7–9] wavelength of Gd^{3+} ion only locates at the short wavelength range (<315 nm), therefore, a phosphor with Gd^{3+} ion can emit in the visible light region which requires the presence of another rare-earth ion paired with Gd^{3+} ion. It is known that the Tb^{3+} ion has strong emission in the visible light region with maximum intensity at 542 nm, so the Tb^{3+} ion has been suitable for this pairing. In addition, the transfer of energy from Gd^{3+} to Tb^{3+} is so efficient that the Gd^{3+} ion is activated as an energy-absorbing centre and the Tb^{3+} ion acts as a luminescence centre [10–13].

The study on new material use as a dosimeter in the nuclear radiation field has been essential, however, only a few materials have been used to measure the dose by the thermoluminescence (TL) method until now [14,15]. Recently, the materials based on fluoride-doped rare-earth ions have been studied, in particular, K_2GdF_5 doped with Dy^{3+} , Pr^{3+} , Sm^{3+} , Tb^{3+} possess remarkable TL properties [16,17].

Due to the thermal neutron absorption of Gd^{3+} ion which is the highest among natural elements (thermal neutron

cross-section = 4.9×10^4 barns), the interaction of the K_2GdF_5 with neutron beam is very strong and can be used as a neutron dosimeter by the TL method. Several studies have shown that the $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ has very high TL intensity and the TL-glow curve shape of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ is simple with a clear dosimetry peak [18–21]. To the best of our knowledge, there is no detailed investigation on optical properties, morphologies and TL of this material. In this paper, we report the structure and luminescence properties of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ samples, which were synthesized by solid-state reaction method. Moreover, the response on thermal neutron beam of prepared material is also presented.

2. Experimental

K_2GdF_5 phosphors doped with terbium ions were synthesized by a solid-state reaction:

where $x = 5, 10, 15, 20\text{ mol\%}$.

The raw materials of KF, GdF_3 and TbF_3 powder with 99.99% pure by grade were purchased from Aldrich Ltd. The mixture of raw materials were thoroughly mixed in an agate mortar for 2 h in a glove-box with argon gas, then the homogeneous mixture was placed in a graphite tube, for calcination in an electric furnace at 620°C for 5 days under N_2 gas. After heating, the product was crushed to micrometre-sized

particles, and then washed with distilled water and ethanol several times to remove excess materials. Then, the powder was dried at 120°C for 30 min, and then annealed at 400°C for 60 min to obtain fine white powder.

The phase identification of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ samples was characterized by an X-ray diffractometer X'Pert from PANalytical with $\text{CuK}\alpha$ (ratio $K_{\alpha 2}/K_{\alpha 1} = 0.5$) radiation. The scans of selected diffraction peaks were carried out in step mode, in the 2θ range from 15 to 70°, step 0.01°, and the measurement temperature was 25°C. The morphology of the powder samples, which were pasted on the carbon tape, were conducted using a scanning electron microscope (SEM) by the MIRA-II Tescan instrument (Czech), operated at 20 kV.

The photoluminescence (PL) measurements were performed on the Horiba JobinYvon FL3-22, resolution of 0.5 nm using the excitation wavelength at 275 nm, and the excitation spectra were also measured on this instrument by a monitor wavelength at 542 nm. The absorption spectra were obtained by using a diffuse reflectance in UV-Vis spectrophotometer V-670 (Jasco, Japan) with integrating sphere ILN-725 (150 mm).

For studying TL properties, the samples were irradiated with various radiation sources, such as: the ^{60}Co gamma source with 0.5 Gy s^{-1} dose rate, $^{90}\text{Sr}/^{90}\text{Y}$ beta with 1.2 Gy min^{-1} dose rate and $^{241}\text{Am}/\text{Be}$ neutron source, the neutron beam (10^7 n s^{-1}) with average energy $E_{\text{avr}} = 4.459 \text{ MeV}$. The TL characteristics were investigated by the Harshaw TLD3500 instrument with the WINREMS program. The heat-treatment range, heating rate were from 50 to 400°C, 2 and 10°C s^{-1} , respectively. The weight of sample on each TL measurement is 20 mg. The TL-glow curves were plotted from data exported by the WINREMS program (Harshaw) for determining the values of the peaks and TL intensities.

3. Results and discussion

3.1 Crystal structure

Figure 1 displays the X-ray diffraction (XRD) spectra of K_2GdF_5 doped with 5, 10, 15 and 20 mol% of Tb^{3+} concentrations. Most of the diffraction peaks are consistent with the orthorhombic structure of K_2GdF_5 and K_2TbF_5 , and the diffraction peaks of this pattern match with the Miller index of K_2GdF_5 by JCPDS no. 77-1924. In addition, XRD pattern has demonstrated that the doping of terbium does not change the structure of K_2GdF_5 host material. In particular, figure 2 shows that $\text{K}_2\text{GdF}_5:10 \text{ mol\% Tb}$ sample has clear peaks with the highest intensity.

Average crystalline size of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ sample doped with 20, 15, 10 and 5 mol% to be 38 ± 2 , 45 ± 1.5 , 74 ± 2 and $42 \pm 1.5 \text{ nm}$, respectively, obtained from XRD patterns using the Scherrer equation were consistent with the respective SEM data. The crystal structure of $\text{K}_2\text{GdF}_5:10 \text{ mol\% Tb}^{3+}$ was simulated by Diamond software. The material has been crystallized in the orthorhombic structure (62) with the space group of Pnm . Figure 3a shows the K^+ cation, which is

Figure 1. XRD pattern of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ with various Tb concentrations.

Figure 2. XRD pattern of K_2GdF_5 doped with 10% Tb^{3+} .

surrounded by eight F^- anions. Each Gd^{3+} cation lies in a D_{2h} symmetry site and is connected to seven F^- anions, forming the GdF_7 polyhedron. The polyhedra are connected by a common edge and form a chain parallel to the c -axis. This one-dimensional structure of the sample has great influence on the energy transfer properties among Gd^{3+} sub-lattices.

The cell parameters of $\text{K}_2\text{GdF}_5:10 \text{ mol\% Tb}^{3+}$ are $a = 10.81 \text{ Å}$, $b = 6.623 \text{ Å}$, $c = 7.389 \text{ Å}$. When doping with Tb^{3+} ion in K_2GdF_5 host lattice, the Gd^{3+} ions will be replaced by the Tb^{3+} ions because the ionic radius of $\text{Tb}^{3+} \sim 109.5 \text{ pm}$ is approximately to $\text{Gd}^{3+} \sim 110.7 \text{ pm}$ (figure 3b). According to Russ. J. Inorg. Chem., 1990 published by the National Institute of Materials Science (Japan), the crystal structure map and the X-ray diffusion schema of K_2GdF_5 and K_2TbF_5 are almost identical, so on replacing Gd^{3+} ions by Tb^{3+} ions, the crystal lattice structure of host material K_2GdF_5 is almost unchanged.

Figure 3. Crystal structure of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$: (a) the coordination polyhedra of Gd^{3+} and F^- in K_2GdF_5 crystal and (b) the replacement of Tb ions in the position of Gd.

3.2 Surface morphology

Surface morphology of the material shows uniform distribution, the structure depends on the grinding mode of the components, proving that in the synthesis process, the material does not melt and does not form big clusters of components. So, it can be concluded that the doping process is very uniform in the material with a correct ratio of components.

The surface morphology of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ powder is presented by SEM images in figure 4, the rough surface of the material structure is due to the distribution of uniform beads at a size of <100 nm. This result was consistent with the data obtained from XRD patterns. Figure 4 shows the change of surface morphology by the Tb^{3+} ion-doped concentrations, in other words, Tb^{3+} ion influences the morphology of the material. The results also indicate that at the concentration of 5 mol% Tb^{3+} , the bead clusters start appearing on the surface, but this appearance is random and overlapping. The density of the particles increases with the increase in doping concentration, as shown in figure 3b–d. However, at 15 mol% doping, it shows a surface with smooth beads covering around the host particles. At 20 mol% doping, the bead distribution is higher, but they are not uniform and appear as thick clusters of beads.

In particular, the sample doped with Tb^{3+} 10 mol% concentration has an optimum surface morphology when the beads are uniform, do not overlap, with high density and appears like a vertical partition on the host particle surface. These surface morphological results are very well correlated with the fluorescence properties. This can be explained by the large porous surface area as well as the uniformity of the vertical partition beads on the 10 mol% doping sample surface.

3.3 PL properties

PL spectrum of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ in the wavelength range of 300–650 nm is measured at excitation wavelength of 275 nm, and it is shown in figure 5a. The fluorescent measurements of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ shows that the emission spectrum consisting main peaks located at 622, 585, 542 and 485 nm corresponding to the $^5\text{D}_4 \rightarrow ^7\text{F}_J$ transitions ($J = 3, 4, 5, 6$) of Tb^{3+} ions. In particular, the highest emission intensity corresponds to the $^5\text{D}_4 \rightarrow ^7\text{F}_5$ transition at 542 nm. Moreover, the K_2GdF_5 sample did not fluoresce in the range of 400–600 nm, and the emission spectrum of K_2TbF_5 sample was similar with the emission spectra of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$. In summary, the PL spectrum also demonstrates that the luminescence centres of the material are purely due to the Tb^{3+} ions.

Figure 5b displays the excitation spectra of K_2GdF_5 : 10 mol% Tb^{3+} , K_2GdF_5 and K_2TbF_5 . The excitation peak with the highest intensity locate at 275 nm, which corresponds to the absorption displacement of Gd^{3+} ion. The other spectral lines of Tb^{3+} ion in the range of 250–300 nm were not observed.

Thus, Gd^{3+} ion clearly has a role in the absorption of excited energy, and the excitation displacement at 275 nm from ground to $^6\text{I}_J$ level, has the highest intensity. In particular, the stimulation transitions at 312 nm corresponding to the displacements from the $^8\text{S}_{7/2}$ base level to the $^6\text{P}_J$ excited levels of ion Gd^{3+} are very high, but conversely the $^6\text{P}_J \rightarrow ^8\text{S}_{7/2}$ emission transitions (312 nm) are not detected in the PL spectrum. Thus, with the existence of the $\text{Gd}^{3+}-\text{Tb}^{3+}$ ion pair, the stimulating energies of the $^6\text{P}_J$, $^6\text{I}_J$ and $^6\text{D}_J$ levels of Gd^{3+} ions can be efficiently transferred to Tb^{3+} ions. These processes enhance the luminescence in the visible range of the phosphor.

Figure 4. SEM images of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ with various Tb^{3+} concentrations.

Figure 5. (a) PL spectra and (b) excitation spectra of $\text{K}_2\text{GdF}_5:10\text{ mol\% Tb}$, K_2GdF_5 and K_2TbF_5 .

The absorption spectrum was obtained by the diffuse reflectance of the $\text{K}_2\text{GdF}_5:10\text{ mol\% Tb}^{3+}$ phosphor, as shown in figure 6a. The spectrum indicates that the absorption peaks at 375 and 485 nm correspond to the energy levels $^5\text{D}_4$ and $^5\text{D}_3$ of Tb^{3+} ion. Thus, the quantum transfer mechanism in the material will follow the rules shown in figure 6b. When the Gd^{3+} ion receives the excited energy, the energy of $^6\text{P}_j$, $^6\text{I}_j$ and

$^6\text{D}_j$ levels will be transmitted to the $^5\text{H}_j$, $^5\text{F}_j$ and $^5\text{K}_j$ levels of the Tb^{3+} ion by T1, T2 and T3 processes, respectively.

Tb^{3+} ion has many energy levels in the range $(25-40) \times 10^3 \text{ cm}^{-1}$, and according to Dieke, this area has 36 levels based on 5D, 5G, 5L, 5H, 5F, 5K states, which are quite close together ($\sim 10^3 \text{ cm}^{-1}$ spacing). So the Tb^{3+} ion quite easily shows non-radiated transfer to the $^5\text{D}_3$ and $^5\text{D}_4$ levels, and

Figure 6. (a) Absorption spectrum of the K₂GdF₅:10 mol% Tb³⁺ phosphor. (b) Schematic of energy levels and mechanisms for the quantum transformation of K₂GdF₅:Tb³⁺.

Figure 7. Glow curves of K₂GdF₅:10 mol% Tb. (a) TL-glow curve, (b) the first PTTL-glow curve, (c) the second PTTL-glow curve and (d) temperature of the single peaks in the TL-glow curve.

then they will transfer to the ground with radiating lines in the visible region shown in figure 5a. The intensity of the transfer from ⁵D₃ to the ground level (range 375–405 nm) is less dependent on the Tb³⁺ concentration and is very weak compared to the transfer from ⁵D₄ level, while the absorption

at the range of 375 nm due to ⁵D₃ is quite high (figure 6a). Thus, it is indicated that there exists two-photon absorption in the wavelength range 375–405 nm, and this mechanism can be described by the ‘abs2’ process in figure 6b. These results are quite consistent with studies on the energy transfer

of Gd–Tb pair in different materials [22–24] and the radiation wavelength range of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ phosphor has advantages in optical measurement applications.

3.4 TL properties

3.4a Characteristic of TL-glow curve: The $\text{K}_2\text{GdF}_5:10\text{mol}\%\text{Tb}^{3+}$ sample was irradiated by ^{60}Co gamma source at 20 Gy dose, then the TL properties were investigated by using a TLD 3500 instrument at a heating rate of 2°C s^{-1} . The TL-glow curve has a dominant peak at 200°C and a low peak at 304°C , as shown in figure 7a. In the down slope of the main peak, there are some low individual peaks covered by the main peak.

To identify these single peaks, the photo-transferred TL (PTTL) method is used. After the TL measurement, all the electrons in the trap corresponding to the main peak have been released, but electrons in the deeper traps have not yet been released. The PTTL measurement was performed with the deuterium (D2) lamp support to irradiate the ultraviolet light on samples and again TL was measured. Figure 7b and c shows PTTL-glow curve of the $\text{K}_2\text{GdF}_5:10\text{mol}\%\text{Tb}$ sample, which was irradiated by ^{60}Co gamma source with 20 Gy dose. After the TL measurement (results shown in figure 7a), the traps corresponding to temperatures below 400°C were erased, but electrons remained in deeper traps were not released. The sample was irradiated by ultraviolet light from a D2 lamp for 10 min, and the TL signal was measured again. The results of this measurement show that the appearance of the old peak at 304°C and a new peak at 234°C is very clear (figure 7b). The peak at 234°C does not appear during the first TL measurement because it is obscured by the very high-intensity peak at 200°C . When the sample was irradiated by the D2 lamp, electrons in deep traps received energy and moved to shallower trap levels, but not enough energy enter the trap level corresponding to the 200°C peak, so, the first PTTL measurement shows the peak which is behind the main peak.

Again, the samples were continued to be irradiated for 10 min by the D2 lamp and the TL signal was measured. The result of the second PTTL measurement (figure 7c) shows the peak still at 304°C , besides the appearance of a new peak at 259°C , while the peak at 234°C is almost disappeared. The TL intensity is very low, indicating that the electron concentrations in the deep traps have been exhausted.

Thus, with the PTTL method and the UV-assisted, the single peaks appeared more clearly without overlapping (figure 7d). The results show that the glow curve of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ includes the main peak at 200°C and other single peaks at 234, 259 and 304°C . The TL intensity of the main peak (dosimetry peak) is very high compared to the lateral peaks, the TL-glow curve has been of simple shape and the radiation dose determination is convenient.

3.4b TL response to beta dose: The beta source investigation results also showed that TL-curve shapes were similar

Figure 8. TL curves of the $\text{K}_2\text{GdF}_5:10\text{mol}\%\text{Tb}^{3+}$ irradiated by $^{90}\text{Sr}/^{90}\text{Y}$ beta source.

to gamma irradiation case. Figure 8 shows the TL response of the $\text{K}_2\text{GdF}_5:10\text{mol}\%\text{Tb}^{3+}$ with the beta radiation and the TL-glow curves were measured from 50 to 350°C with a 10°C s^{-1} heating rate. All the TL curves had the dosimetry peaks at 223°C and the glow curves of various doses were very uniform with the TL intensity proportional to the dose.

3.4c The TL response to neutron dose: The energy transfer issue from Gd^{3+} ion to Tb^{3+} ion is very important for materials used in the field of neutron measurements. Because the gadolinium ion interacts with the neutron, the Gd^{3+} ion moves into an excited state, and then transfers energy to the Tb^{3+} ion for emission. In the TL measurement, the green emission of the Tb^{3+} ion at 542 nm due to $^5\text{D}_4 \rightarrow ^7\text{F}_5$ transition is predominating and this green emission is also well-consistent with the sensitivity range of photomultiplier tube in the TL reader.

K_2GdF_5 doped with 5, 10, 15, 20 mol% Tb^{3+} were irradiated by the $^{241}\text{Am}/\text{Be}$ neutron source (10^7 n s^{-1} activity). All the samples were placed inside a 25 cm diameter polyethylene sphere (the polyethylene sphere is used to thermalize as the neutron beam), distance from the sphere to the $^{241}\text{Am}/\text{Be}$ source was 1 m and irradiation time was 24 h. The TL-glow curves were investigated with heating rate $\beta = 10^\circ\text{C s}^{-1}$, the preheat temperature was 50°C for 10 s, the temperature range for TL measurements was from 50 to 350°C , for comparison purpose, the common dosimeter $\text{CaSO}_4:\text{Dy}^{3+}$ was also investigated together.

Figure 9a presents the TL-glow curves of the $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ and $\text{CaSO}_4:\text{Dy}^{3+}$ samples. The TL intensity of $\text{K}_2\text{GdF}_5:10\text{mol}\%\text{Tb}^{3+}$ is the highest. In particular, with the thermal neutron irradiation, the TL intensity of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ 10 mol% is 10 times higher than that of the common dosimeter $\text{CaSO}_4:\text{Dy}^{3+}$, thus the $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ absorbs significant amount of neutrons.

Figure 9. The TL-glow curves: (a) samples irradiated by thermal neutron and (b) $\text{K}_2\text{GdF}_5:10 \text{ mol\% Tb}^{3+}$ irradiated by fast neutron.

Figure 10. TL-glow curves of $\text{K}_2\text{GdF}_5:10 \text{ mol\% Tb}$, K_2GdF_5 and K_2TbF_5 irradiated by fast neutron.

The results also show that the shapes of curves are very simple, with the main peak temperature at about 223°C (at a heating rate of 10°C s^{-1}). The TL intensity of the main peak is very high and its shape is quite symmetric, corresponding to the second-order kinetic of TL theory. In the TL theory for the dosimeter, the peak temperature must not be too low to avoid the fading phenomena, as well as not too high to avoid being obscured by infrared, and this peak shown in figure 9, which is in the range of $200\text{--}250^\circ\text{C}$ is very suitable for dose measurement. Figure 9b shows the TL-glow curves of the $\text{K}_2\text{GdF}_5:\text{Tb}^{3+} 10 \text{ mol\%}$ irradiated by the various fast neutron doses. The shapes of glow curves are heterogeneous, however, all peaks of glow curves are at the temperature range of $230\text{--}250^\circ\text{C}$ and the TL intensities of the main peak are proportional to the irradiation doses.

I_D compare TL response of the prepared samples, the samples were irradiated with a neutron beam of 8.31 mSv . The

results show that the TL response of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+} 10 \text{ mol\%}$ sample was higher than that of K_2GdF_5 and K_2TbF_5 samples as shown in figure 10. The TL phenomenon is due to the electron traps and recombination centres in the forbidden bands of the electronic band structure and the formation of these traps and centres requires the doping of different ions. Thus, pure K_2GdF_5 and K_2TbF_5 crystals will have a very weak TL. In addition, the PL spectrum of K_2GdF_5 indicates that the luminescence of Gd^{3+} ion was not observed in visible light, so the TL intensity of K_2GdF_5 was very weak. The TL-glow curves of K_2GdF_5 and K_2TbF_5 have no peak in the temperature range required for the dosimetry (about $200\text{--}300^\circ\text{C}$), therefore, K_2GdF_5 and K_2TbF_5 cannot be used as a TL dosimeter. The study results reinforce the neutron dosing characteristics of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$, with the Gd^{3+} ion absorbing the energy of the neutron and the Tb^{3+} ion acts as the luminescence centre.

In the case of irradiation by thermal neutron radiation, the results in figure 9a show that the TL intensity of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+} 10 \text{ mol\%}$ is much higher than that of the common dosimeter $\text{CaSO}_4:\text{Dy}^{3+}$. Therefore, the $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ can be applied as a dosimeter to analyse and measure the nuclear radiation doses.

4. Conclusion

The $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ samples synthesized by the solid-state reaction method had a porous surface and a high contact area due to the formation of grooves on the surface. With this special surface feature, the material can be potential for absorbing maximum energy as well as for high luminescence intensity. In addition, the doping with Tb^{3+} rare-earth ion also enhances emission intensity via the supreme efficiency of energy transfer from Gd to Tb ions, and the effects of these surfaces and ion-doped has been shown in PL measurements. In particular, this study has shown the excellent TL properties of $\text{K}_2\text{GdF}_5:\text{Tb}^{3+}$ related to nuclear radiation, especially

with neutron. The results of the TL investigation indicate that the material has a linear dose response and high sensitivity to nuclear irradiation. Furthermore, the TL-glow curve shape is suitable for applications in the field of dosimetry. Thus, with the structure and luminescence properties investigated in this study, the $\text{K}_2\text{GdF}_5\text{:Tb}^{3+}$ 10 mol% will not only satisfy expectations in the optical field, but also presents a feasible idea for a new dosimeter to measure mixed radiation dose.

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