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Effect of 100 MeV swift Si$^{8+}$ ions on structural and thermoluminescence properties of Y$_2$O$_3$:Dy$^{3+}$ nanophosphor

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ABSTRACT
Nanoparticles of Y$_2$O$_3$:Dy$^{3+}$ were prepared by the solution combustion method. The X-ray diffraction pattern of the 900°C annealed sample shows a cubic structure and the average crystallite size was found to be 31.49 nm. The field emission scanning electron microscopy image of the 900°C annealed sample shows well-separated spherical shape particles and the average particle size is found to be in a range 40 nm. Pellets of Y$_2$O$_3$:Dy$^{3+}$ were irradiated with 100 MeV swift Si$^{8+}$ ions for the fluence range of $3 \times 10^{11}$ to $3 \times 10^{13}$ ions cm$^{-2}$. Pristine Y$_2$O$_3$:Dy$^{3+}$ shows seven Raman modes with peaks at 129, 160, 330, 376, 434, 467 and 590 cm$^{-1}$. The intensity of these modes decreases with an increase in ion fluence. A well-resolved thermoluminescence glow with peaks at $\sim$ 414 K ($T_{m1}$) and $\sim$ 614 K ($T_{m2}$) were observed in Si$^{8+}$ ion-irradiated samples. It is found that glow peak intensity at 414 K increases with an increase in the dopant concentration up to 0.6 mol% and then decreases with an increase in dopant concentration. The high-temperature glow peak (614 K) intensity linearly increases with an increase in ion fluence. The broad TL glow curves were deconvoluted using the glow curve deconvoluted method and kinetic parameters were calculated using the general order kinetic equation.

1. Introduction

The Y$_2$O$_3$ material attracted researchers due to its superior properties such as high melting point (2673 K), low thermal conductivity (33 Wm$^{-1}$ K$^{-1}$), high thermal stability, wide transparency range (0.2–8 $\mu$m) with a large band gap of 5.7 eV, high refractive index (1.8) and low phonon energy (380 cm$^{-1}$) ($I$, $2$). Y$_2$O$_3$ is one of the best hosts for rare-earth (RE) ions, because of similarities in chemical properties and ionic radii of RE ions. Among RE ions, dysprosium (Dy$^{3+}$) has attracted much attention due to its unique spectral properties, Y$^{3+}$ and Dy$^{3+}$ are trivalent ions having nearly the same ionic radii. Therefore, the Dy$^{3+}$ ion easily substitutes in Y$^{3+}$ sites, which leads to enhancement of optical properties. It is well known that greenish-yellow light can be obtained by mixing blue and yellow light ($3$, $4$). Various techniques are available for the preparation of nanomaterials such as chemical
Raman spectroscopy is one of the non-destructive material characterization techniques. Raman spectroscopy is able to deliver structural information on swift heavy ion- (SHI) induced lattice disorder and nature of crystallinity and phonon vibrations because it is sensitive to structural disorder that occurs within optical skin depth of a laser beam (8). Thermoluminescence (TL) is a simple technique for investigation of radiation-induced defects. TL and optically stimulated luminescence dosimeters have many advantages over currently used dosimeters in radiotherapy and space dosimetry (9) with heavy charged particles. Heavy ion beams have been used for diagnostic and therapeutic purposes (10). The treatment with highly energetic ion beams has several advantages in comparison with conventional irradiation with photons. The high-energy particles are present in space environment, where the components used in various subsystems of air crew and space craft get exposed to such radiations. There are several ways of measuring the dose of radiation. At present, the dose of radiation is measured largely using thermoluminescent dosimetry (TLD) badges. There are several materials that exhibit TL properties with ion beam irradiation (11, 12). Among these, RE-doped SHI-induced TLD properties have been extensively studied during the past few decades. Dy-doped CaSO4 (13) and CaF2 (14) are used for standard dosimetry applications. Sanyal et al. studied the behavior of thermally treated CaSO4:Dy phosphor. The phosphor revealed linear response of the low-temperature glow peak and could be an efficient dosimetry system for the food commodities (15). However, efforts are still being made to improve dosimetry for measuring high-energy radiations. The objective of the present work is to study SHI-induced structural modifications and TL properties of Y2O3:Dy3+ nanophosphor. TL trapping parameters are evaluated by the glow curve deconvoluted (GCD) method and results are discussed in detail.

2. Material and methods
Dy3+-doped yttrium oxide nanophosphor was synthesized by the solution combustion technique, using yttrium nitrate hexahydrate and dysprosium nitrate pentahydrate as oxidizer and urea as fuel. Yttrium nitrate hexahydrate (Y(NO3)3·6H2O) with 99.8% purity, dysprosium nitrate pentahydrate (Dy(NO3)3·5H2O) with 99.9% purity were procured from Aldrich Chemicals, urea (CH4N2O) with 99.5% purity was procured from Merck Specialties Private Limited. All the chemicals are directly used without any purification. Stoichiometric amount of yttrium nitrate, dysprosium nitrate and urea were dissolved in 50 ml of double distilled water in a cylindrical pyrex dish of 600 ml capacity. The homogeneous mixture was introduced into a muffle furnace maintained at 500 ± 5°C. Initially, the solution boils and undergoes dehydration followed by decomposition with evaporation of a large amount of gases leaving behind the powder. The final product is voluminous and whitish in nature (11). The synthesized powder was annealed at 900°C for 2 h (7, 16). Pellets of Y2O3:Dy3+ samples were prepared based on the procedure discussed elsewhere (16) and pellets irradiated with 100 MeV swift Si8+ ions having 2 pA current using 15 UD Pelletron at
the Inter University Accelerator Centre, New Delhi, India. The irradiation fluences were varied from $3 \times 10^{11}$ to $3 \times 10^{13}$ ions cm$^{-2}$. Pellets were characterized by the X-ray diffraction (XRD) technique using the Bruker D8 diffractometer, with Cu-K$_{\alpha}$ radiation of wavelength 1.5406 Å. Morphology of the synthesized samples was studied by field emission scanning electron microscopy (FE-SEM) [MIRA II LMH from TESCAN]. Raman spectra were recorded in the range 100–1000 cm$^{-1}$ using the Renishaw 1000 Raman spectrometer with an excitation wavelength of 514.5 nm from 0.5 mW of Ar ion laser as the excitation source. TL glow curves were recorded at a heating rate of 5 K s$^{-1}$ using the Harshaw TLD reader (Model 3500). All experiments were performed at room temperature.

3. Results and discussion

3.1. X-ray diffraction

Figure 1 shows the XRD patterns of 900°C annealed pristine and 100 MeV swift Si$^{8+}$ ion-irradiated Dy$^{3+}$ (0.6 mol%) doped Y$_2$O$_3$. XRD patterns are indexed to the cubic Y$_2$O$_3$ phase with the space group Ia$\overline{3}$ according to the standard card (JCPDS No. 88–1040) (16, 17). Further it is observed that ion irradiation does not induce additional peaks and there is no change in peak position. However, peak broadening and decreasing peak intensity are observed after ion irradiation due to loss of crystallinity with an increase in ion fluence. The loss of crystallinity and lattice damage could be interpreted based on the thermal spike model. According to this model, high energy deposited by the ions is diffused to electrons. Then, the electrons transfer their energy to the lattice in solid by electron-atom interaction.

![Figure 1. XRD patterns of pristine and SHI-irradiated Dy$^{3+}$ (0.6 mol%) doped Y$_2$O$_3$.](image_url)
Finally, the energy is diffused into lattice and induces a local increase of lattice temperature in the cylindrical ion path region. This local heating followed by a rapid quenching may lead to the loss of crystallinity and lattice disorder (18).

The crystallite size and lattice strain are estimated using the Williamson–Hall (W–H) method (17, 19). Figure 2 shows a plot of $\beta \cos \theta / \lambda$ versus $4 \sin \theta / \lambda$. We will get a straight line with a slope equal to the value of the lattice strain and the $y$-axis intercept equal to the inverse of the crystallite size and was found to be 29–36 nm. The average crystallite size of pristine and ion-irradiated samples is also calculated using Scherrer’s formula and found to be 20–28 nm. From the XRD data, dislocation density ($\delta$) and the lattice strain are calculated and are found to increase with an increase in ion fluence as shown in Table 1. This is attributed to the ion-induced loss of crystallinity and an increase in lattice disorderness.

Table 1. Structural parameters of pristine and 100 MeV swift Si$^{8+}$-irradiated Dy$^{3+}$-doped Y$_2$O$_3$.

<table>
<thead>
<tr>
<th>Ion fluence (ions cm$^{-2}$)</th>
<th>Crystallite size, $D$ (nm)</th>
<th>Lattice constant, $a$ (Å)</th>
<th>Density, $\rho$ (g cm$^{-3}$)</th>
<th>Dislocation density, $\delta \times 10^{15}$</th>
<th>Interplanar space in at (222) (Å)</th>
<th>Lattice strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pristine</td>
<td>28.10</td>
<td>36.03</td>
<td>10.606</td>
<td>5.028</td>
<td>1.27</td>
<td>3.062</td>
</tr>
<tr>
<td>$3 \times 10^{11}$</td>
<td>26.22</td>
<td>34.05</td>
<td>10.609</td>
<td>5.024</td>
<td>1.45</td>
<td>3.063</td>
</tr>
<tr>
<td>$3 \times 10^{13}$</td>
<td>20.21</td>
<td>29.00</td>
<td>10.609</td>
<td>5.024</td>
<td>2.44</td>
<td>3.063</td>
</tr>
</tbody>
</table>
3.2. FESEM micrograph analysis

FESEM is an important tool for the study of topography and surface morphology of the synthesized nanopowders. It is well known that the combustion synthesized product is highly influenced by fuel to metal–ligand complex formation. Depending on the nature of fuel, fuel to oxidizer ratio, the nature of combustion differs from flaming to non-flaming reactions which involve liberation of enormous amount of gases \(^{(20)}\). Figure 3(a) shows the FE-SEM image of 900°C annealed \(\text{Y}_2\text{O}_3:\text{Dy}^{3+}\) (0.6 mol%) and it is observed that the particles are loosely agglomerated with spherical shape and voids due to the evolution of large amount of gases during the combustion process. And, the average particle size of \(\text{Y}_2\text{O}_3:\text{Dy}^{3+}\) is found to be \(\sim 22\) nm. The composition of the sample is estimated through

![Figure 3](image)

**Figure 3.** (a) FE-SEM image of 900°C annealed \(\text{Y}_2\text{O}_3:\text{Dy}^{3+}\) nanophosphor and (b) EDX of 900°C annealed \(\text{Y}_2\text{O}_3:\text{Dy}^{3+}\) nanophosphor.
the energy-dispersive X-ray spectroscopy (EDX) technique. It is proved to be a powerful tool to obtain the elemental composition ratio as shown in Figure 3(b) of the Y$_2$O$_3$:Dy$^{3+}$ (0.6 mol%) sample. We can see only the element peaks of Y, O and Dy in the sample. The concentrations of Y, O and Dy in the sample are 32.25, 67.47 and 0.28 at%, respectively.

### 3.3. Raman spectroscopy

Raman spectroscopy is a very sensitive non-destructive technique and it provides vibration of molecules within the scattering volume. The representation of optical modes in cubic Y$_2$O$_3$ are given (21) as

$$\Gamma_{\text{opt}} = 4A_g + 4E_g + 14F_g + 5A_{zu} + 5E_u + 16F_u,$$

where $4A_g$, $4E_g$ and $14F_g$ are Raman active, $16F_u$ is IR active and $5A_{zu}$, $5E_u$ are inactive modes. Thus, there are 22 Raman active modes, where $E_g$ and $F_g$ modes are doubly and triply degenerated, respectively. The Raman active modes of cubic Y$_2$O$_3$ can be divided into 15 modes ($3A_g + 3E_g + 9F_g$) coming from the vibration of O ions and 7 modes ($A_g + E_g + 5F_g$) coming from the vibration of Y ions (21). Figure 4 shows the room temperature Raman spectra of pristine and 100 MeV S$_8^{8+}$ ion-irradiated Y$_2$O$_3$:Dy$^{3+}$ (0.6 mol%) samples. The Raman modes observed at 129, 330, 376, 434, 467, 590 and 160 cm$^{-1}$ are attributed to $F_g$ and $F_g + A_g$ vibration modes. The Raman active mode of 376 cm$^{-1}$ shows

![Figure 4](image-url)
high scattering intensity. This peak position is attributed to the Y–O vibration bond. SHI irradiation is expected to cause a decrease in all vibration modes intensity and full width half maxima of 376 cm$^{-1}$ peak increases with an increase in ion fluence. At high fluence ($3 \times 10^{13}$ ions cm$^{-2}$), all Raman modes disappear except the 376 cm$^{-1}$ mode (22). It indicates an increase in lattice damage in the samples and loss of crystalline nature.

3.4. Thermoluminescence

TL glow curves of pure and Dy$^{3+}$- (0.1–1.0 mol%) doped Y$_2$O$_3$ nanophosphor irradiated with 100 MeV Si$^{8+}$ for fluence of $3 \times 10^{11}$ ions cm$^{-2}$ are shown in Figure 5. A well-resolved TL glow with peak at $\sim$ 414 K and another one with peak at $\sim$ 614 K are observed. Whereas in pure samples the lower temperature of the TL glow peak was observed at $\sim$ 403 K besides the 600 K glow peak. The formation of traps at different depths corresponding to these TL glow peaks is the result of different penetrating levels of the SHI. The lower temperature peak (414 K) appears due to electrons being trapped at a shallow level, that is, F-center (9, 22). The higher temperature peak at 614 K might be due to electrons activated from deep traps. The electrons captured by deep traps may go without getting retrapped at intermediate levels to the conduction band and recombine subsequently giving rise to the high-temperature glow peak. It is believed that energetic SHI creates oxygen vacancies and other structural lattice defects in the Y$_2$O$_3$:Dy$^{3+}$ sample. The behaviors of the two peaks are different, because they arise from two different types of defect centers. Glow peak at 414 K is due to electrons trapped at the shallow level, that is, the F-center (9, 22). The TL intensity at 414 K glow peak is found to increase with increasing Dy$^{3+}$ molar concentration up to 0.6 mol% and thereafter decreases with a further increase in Dy$^{3+}$ concentration as can be seen in the inset of Figure 5. This could be due to an increase in the number of luminescence centers with the Dy$^{3+}$ content. Beyond 0.6 mol% the probability of energy transfer from traps to the luminescence centers decreases and hence the intensity. This effect is

![Figure 5](image-url)
known as concentration quenching (17). It indicates that Dy\(^{3+}\) creates more number of F-centers initially. During this process complex defect centers such as F\(_2\)-centers are formed resulting in decreased TL intensity corresponding to F-center. The high-temperature peak at 614 K might be due to deep level electron trap, that is, the F\(_2\)-center (23). The TL intensity at 614 K glow peak is found to decrease with increasing Dy\(^{3+}\) molar concentration. However, TL glow peak temperature at this peak is almost steady.

TL glow curves of 100 MeV swift Si\(^{8+}\) irradiated 0.6 mol% of Dy\(^{3+}\)-doped Y\(_2\)O\(_3\) for various ion fluence in the range 3 \times 10^{11}–3 \times 10^{13} ions cm\(^{-2}\) are shown in Figure 6. It is observed that low temperature TL glow peaks (414 K) intensity decreases with increase in ion fluence while the higher temperature glow peak (614 K) intensity increases linearly up to 1 \times 10^{12} ions cm\(^{-2}\) and then decreases with a further increase of ion fluence as shown in the inset of Figure 6. The saturation effect has been observed in the recorded ion fluence (Figure 3), and it is explained based on the track interaction model (24, 25) and is quite suitable to explain the linear/sub linear/saturation response of nanophosphors in a long period of irradiation. According to this model, SHI could generate a track of length equal to the order of few 10s of nanometers.

At low fluences, electrons escaping their host tracks are intercepted by the nonradioactive competitive centers in the intermediate region between the tracks. The TL signal is proportional to the number of ion tracks. At high fluences the separation between neighboring ion tracks is reduced. Therefore, effective electrons escaping from tracks are more and more and recombine with luminescent centers, resulting in more TL signal. Furthermore, with an increase in ion fluence, the separation between the tracks is reduced resulting in overlapping of tracks causing reduction in TL signal hence resembling saturation. A further increase in ion fluence leads to reduction in the TL signal. In the present work, the saturation effect has not been observed due to low ion fluence (3 \times 10^{11} ions cm\(^{-2}\)). SHI

![Figure 6](image)

**Figure 6.** TL glow curves of SHI-irradiated Y\(_2\)O\(_3\):Dy\(^{3+}\) nanophosphor for different fluences. Inset: Variation of TL intensity as a function of ion fluence.
induces more number of surface defects (oxygen vacancies) and they are responsible for ion tracks. However, the high-temperature glow peak (614 K) is found to increase linearly with an increase of ion fluence, but sub linear/saturation effect has not been observed.

The TL mechanism of the material may be explained based on the knowledge of kinetic parameters (trapping parameters), that is, activation energy ($E$), order of kinetics ($b$) and frequency factor ($s$) associated with the glow peaks. TL of Si$^{8+}$ ion-irradiated Y$_2$O$_3$:Dy$^{3+}$ nanophosphor exhibits broad TL glow due to overlapping of more than two glow peaks. The symmetry factor ($\mu_g$) for this glow was estimated to be $>0.65$. This value is greater than the allowed values for the first (0.42), second (0.52) and the general (values between 0.43 and 0.51) order of kinetics (26). Therefore, the above complex glow curve was deconvoluted using the GCD technique. The deconvoluted TL glow curves are obtained for the best fit of the theoretical curve with the experimental one. In the present work, the theoretical fit matches with experimental data for six deconvoluted peaks. The detailed GCD technique is discussed by Kitis et al. (27). They reported the TL glow curve equation of general order kinetics which is used for glow curve deconvolution

$$I(T) = I_m \cdot b^{b/(b-1)} \cdot \exp \left( \frac{E}{kT} \cdot \frac{T - T_m}{T_m} \right) \times \left[ (b - 1) \cdot (1 - \Delta) \cdot \frac{T^2}{T_m^2} \times \exp \left( \frac{E}{kT} \cdot \frac{T - T_m}{T_m} \right) + Z_m \right] \cdot -b/(b-1),$$

where

$$\Delta = \frac{2kT}{E}; \Delta_m = \frac{2kT_m}{E}; Z_m = 1 + (b - 1) \cdot \Delta_m,$$

where $k$ is Boltzmann’s constant ($8.6 \times 10^{-5}$ eV K$^{-1}$), $\beta$ is the linear heating rate (5 K s$^{-1}$), $I_m$ is the glow peak intensity and $T_m$ is the glow peak temperature. The above equation can be inserted into an Excel spreadsheet. For the fitting procedure, one has to insert initial, arbitrary but meaningful values for the parameters $I_m$, $T_m$, $E$ and $b$ for each single glow peak. When the curve fitting procedure is completed, it gives the net values of $I_m$, $T_m$, $E$ and $b$. In addition, the frequency factor and figure-of-merit (FOM) values are found (28). In the present work, theoretical fit matches with the experimental data for six deconvoluted peaks. The expression for the frequency factor and FOM are given by (17)

$$\frac{\beta E}{kT_m^2} = s \exp \left[ -\frac{E}{kT_m} \left[ 1 + (b - 1) \Delta_m \right] \right],$$

$$\text{FOM} = \frac{\sum |TL_{exp} - TL_{the}|}{\sum TL_{the}}.$$  

Here, $TL_{exp}$ and $TL_{the}$ represent the TL intensity of experimental and theoretical glow curves, respectively. The deconvoluted TL curves of 100 MeV Si$^{8+}$-irradiated undoped and Dy-doped nanocrystalline Y$_2$O$_3$ for a fluence of $3 \times 10^{11}$ ions cm$^{-2}$ are shown in Figure 7 and 8. The FOM for the present curve fitting is found to be 1.80% and 1.94% for undoped and doped ion irradiation samples, respectively. This indicates a good agreement between theoretically generated and experimentally recorded TL glow curves. The deconvoluted experimental glow curves revealed different TL glows with peaks at 404, 433, 488, 533 and 606 K for undoped and peaks at 414, 422, 488, 533 and 606 K for ion-irradiated Dy-doped Y$_2$O$_3$. It is found that the above-deconvoluted glow obeys the general order kinetics.
The activation energies are found to increase and the frequency factor decreases with an increase of TL glow peak temperature. The above trapping parameters are tabulated in Table 2. The activation energy of the Dy-doped $Y_2O_3$ sample shows a higher value when compared with that of the undoped sample. It is due to $Dy^{3+}$ being incorporated in the $Y^{3+}$ lattice modifying the band gap of the material, therefore the doped sample exhibited deeper traps of electron and holes center \( (29) \). The obtained activation energy and
Table 2. Trap parameters of TL glows of 100 MeV swift Si\(^{8+}\)-irradiated undoped and Dy\(^{3+}\)-doped Y\(_2\)O\(_3\).

<table>
<thead>
<tr>
<th>Samples</th>
<th>(T_m) (K)</th>
<th>(E_a) (eV)</th>
<th>(s) (s(^{-1}))</th>
<th>FOM (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Undoped ((3 \times 10^{11}) ions cm(^{-2}))</td>
<td>404</td>
<td>1.15</td>
<td>1.25 (\times 10^{15})</td>
<td></td>
</tr>
<tr>
<td></td>
<td>433</td>
<td>1.8</td>
<td>1.30 (\times 10^{14})</td>
<td></td>
</tr>
<tr>
<td></td>
<td>488</td>
<td>2</td>
<td>1.33 (\times 10^{13})</td>
<td>1.80</td>
</tr>
<tr>
<td></td>
<td>538</td>
<td>2</td>
<td>1.35 (\times 10^{12})</td>
<td></td>
</tr>
<tr>
<td></td>
<td>606</td>
<td>2</td>
<td>1.40 (\times 10^{10})</td>
<td></td>
</tr>
<tr>
<td>Y(_2)O(_3):Dy(^{3+}) ((0.8\text{ mol%})) ((3 \times 10^{11}) ions cm(^{-2}))</td>
<td>414</td>
<td>1.3</td>
<td>1.35 (\times 10^{16})</td>
<td></td>
</tr>
<tr>
<td></td>
<td>442</td>
<td>2</td>
<td>1.40 (\times 10^{15})</td>
<td></td>
</tr>
<tr>
<td></td>
<td>488</td>
<td>2</td>
<td>1.42 (\times 10^{14})</td>
<td>1.94</td>
</tr>
<tr>
<td></td>
<td>533</td>
<td>2</td>
<td>1.44 (\times 10^{12})</td>
<td></td>
</tr>
<tr>
<td></td>
<td>606</td>
<td>2</td>
<td>1.47 (\times 10^{9})</td>
<td></td>
</tr>
</tbody>
</table>

Figure 9. Effect of fading with storage time in Y\(_2\)O\(_3\):Dy\(^{3+}\) nanophosphor.

frequency factor values are much closer to the standard dosimeter samples \((26)\). Hence, this material might be used for high-energy space dosimeter applications.

3.4.1. TL fading effect

To study the effect of fading on TL glow curves of Y\(_2\)O\(_3\):Dy\(^{3+}\), samples were exposed for a test fluence of \(5 \times 10^{11}\) ions cm\(^{-2}\). Figure 9 shows the plot of normalized TL intensity as a function of storage time after ion irradiation \((22, 26)\). Initially, the fading was very fast for the first eight days \((33\%\) and \(21\%\) for 414 and 614 K glow peaks, respectively\) and later the fading became slow. It is found that there is \(46\%\) and \(24\%\) fading over a period of 30 days for 414 and 610 K glow peaks, respectively. These results conclude that high-temperature glow peak \((614\ K)\) is suitable for high-energy space dosimeter studies.

4. Conclusions

Combustion synthesized Dy\(^{3+}\)-doped Y\(_2\)O\(_3\) exhibits cubic phase and an average crystallite size of \(~28\) nm. The FE-SEM image shows well-separated spherical shape particles and the
average particle size is found to be in the range 40 nm. In the present studies, good TL efficiency was observed with an optimum Dy$^{3+}$ dopant concentration of 0.6 mol%. The prominent high-temperature TL glow peak intensity increases linearly with ion fluence up to $3 \times 10^{13}$ ions cm$^{-2}$ in Dy$^{3+}$-doped Y$_2$O$_3$. It is observed that TL glow curves exhibit second-order kinetics since the re-trapping of electron is high and the obtained activation energy and frequency factor are close to the standard dosimeter samples. The high-temperature glow peak (614 K) shows less fading over a period of 30 days. It is suggested that the present sample might be suitable for high-energy space dosimetry application.

**Disclosure statement**

No potential conflict of interest was reported by the authors.

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