

DOI: 10.5862/JPM.230.10

UDC: 535.37

C.B. Palan, N.S. Bajaj, S.K. Omanwar

Sant Gadge Baba Amravati University, Amravati, India

ELEMENTARY RESULTS ON THE DOSIMETRIC PROPERTIES OF SrSO₄:Eu²⁺ PHOSPHOR

A polycrystalline sample of SrSO₄:Eu²⁺ phosphor has been successfully synthesized using the co-precipitation method and studied for its luminescence properties. The phosphor showed rather high optical stimulated luminescence (OSL) sensitivity which was about 75% of that for the commercially available α-Al₂O₃:C phosphor (TLD-500). The continuous wave (CW)-OSL curve exhibited three components having photoionization cross-sections of 1.78·10⁻¹⁷, 7.70·10⁻¹⁷ and 17.69·10⁻¹⁷ cm² respectively. The thermal luminescence (TL) sensitivity was about 100 times higher than that for TLD-500. The kinetic parameters for TL curve such as activation energy and frequency factor were calculated using peak shape treatment. OSL components were determined from CW and linear modulated (LM)-OSL data. The minimum detectable dose was found to be 11.6 mGy with 3σ of background. Also reusability studies showed that it was possible to reuse the phosphor for 10 cycles without change in the OSL output. In the TL mode the dose-response was nearly linear in the range of measurement (20 – 400 mGy), and fading was 40% in 72 h. Photoluminescence spectra of SrSO₄:Eu²⁺ exhibited emission in the near UV region when excited with an UV source at 254, 315, and 323 nm.

CO-PRECIPIATION, TLD-500, CW-OSL, PHOTOIONIZATION CROSS-SECTION.

1. Introduction

Solid-state luminescent dosimetry is based on radiation energy storage in dosimetric materials in the form of lattice defects and captured charge carriers. The stored energy can be released through the light from luminescence centers. Energy release is stimulated either by heating (thermally stimulated luminescence (TSL/TL)) or by irradiating with light quanta of proper energy (optically stimulated luminescence (OSL)). TL-based dosimeters are widely used in radiation dose monitoring but in comparison to TL dosimetry technique OSL has been becoming popular in radiation dosimetry applications [1 – 4]. OSL has been first used in archaeological dating and later proposed for personnel monitoring and environmental monitoring of radiation with the development of Al₂O₃:C [5].

Different stimulation techniques have been followed by OSL measurements that offer different signal-to-noise ratio. A few of them are CW-OSL, P-OSL, LM-OSL, TA-OSL and NL-OSL, amongst which CW-OSL is the most

preferred and popular choice of stimulation mode, because in CW-OSL, the luminescence is recorded very fast and looks like a decay curve, the background count rate or net background is nearly constant and signal-to-noise ratio is high [6].

Over the last several decades sulfate hosts doped with rare earth materials have been widely used in radiation dosimetry, and also these materials show good luminescent properties. Many researchers have reported on these materials with different synthesis methods and studies for different luminescence properties [7–9].

In this paper we are reporting TL and OSL properties (under beta irradiation) of Eu-doped SrSO₄ phosphor synthesised by using co-precipitation.

2. Experimental details

SrSO₄ phosphor activated with Eu was prepared by the co-precipitation method described in our earlier works [10]. The stoichiometry of the reaction was maintained

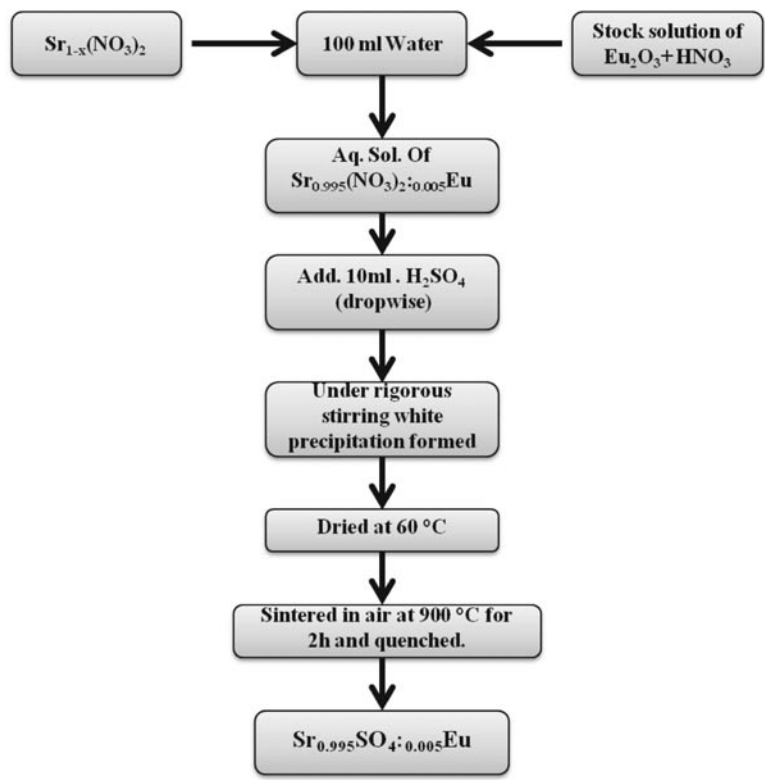


Fig. 1. Flow chart of $\text{Sr}_{0.995}\text{SO}_4:0.005\text{Eu}^{2+}$ synthesized by the co-precipitation method

by the formula $\text{Sr}_{1-x}\text{SO}_4:{}_x\text{Eu}^{2+}$. The nitrate precursor of strontium was dissolved in 100 ml of double-distilled water with drop-wise addition of the stock solution prepared for Eu_2O_3 . The solution was prepared in a glass beaker under stirring to form a homogeneous aqueous solution, and it was confirmed that the precursor was dissolved in distilled water. 10 ml of the H_2SO_4 solution were added drop by drop into the mixed aqueous solution of $\text{Sr}_{1-x}(\text{NO}_3)_2:{}_x\text{Eu}$ under rigorous stirring at room temperature and white precipitation formed. After that, the SrSO_4 precipitate was centrifuged and rinsed several times by distilled water to remove the excess residual salts. The precipitate was dried at 60°C for 2 h by optical heating. The dried sample was annealed at 900°C for 1 h to get a white crystalline powder of $\text{SrSO}_4:\text{Eu}^{2+}$. The complete process involved in the reaction is presented as a flow chart in Fig. 1.

3. Results and discussion

The structure of the as-prepared samples were analyzed by a Rikagu Miniflex X-ray

diffractometer, using monochromatic $\text{CuK}_{\alpha 1}$ ($\lambda = 1.5405 \text{ \AA}$) radiation in the 2θ range of $10^\circ - 60^\circ$. Photoluminescence was studied by means of a Hitachi F-7000 fluorescence spectrophotometer. Emission and excitation spectra were recorded using a spectral slit of 2.5 nm for each window. For studying the TL and the OSL response, all the samples were irradiated using a $^{90}\text{Sr} / ^{90}\text{Y}$ beta source with the dose rate of 20 mGy per minute. All OSL measurements were carried out using an automatic Risø TL/OSL-DA-15 reader system which capable of accommodating up to 48 disks. Blue-light diodes emitting at 470 nm (LEDs with $\text{FWHM} = 20 \text{ nm}$) were arranged in four clusters, each containing seven individual LEDs. The total power from 28 LEDs at the sample position was 80 mW/cm^2 . A green long pass filter (GG-420) was incorporated in front of each blue LED cluster to minimize the amount of directly scattered blue light from reaching the detector system. The standard photomultiplier used in the Risø TL/OSL luminescence reader was a bialkali EMI 9235QA, which has an

extended UV response with maximum detection efficiency between 300 and 400 nm. To prevent scattered stimulation light from reaching the photomultiplier, the Risø reader was equipped with a 7.5 mm Hoya U-340 detection filter, which has a peak transmission around 340 nm (FWHM ~80 nm).

X-ray diffraction pattern. The structure of $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor was orthorhombic, with the space group $Pbnm$ (62), and with the lattice parameters of $a = 6.86$, $b = 8.35$, $c = 5.34$ Å and $\alpha = \beta = \gamma = 90^\circ$. In order to determine the phase purity, chemical nature of the phosphor, X-ray diffraction (XRD) analysis was carried out. Fig. 2, *a* shows the XRD pattern of $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor along with the standard XRD pattern (International Centre for Diffraction Data (ICDD) Card No. 01-075-6773). The XRD pattern showed the formation of pure SrSO_4 phase. The addition of the dopant (Eu) did not seem to have any effect on the XRD pattern which suggested that the dopant was incorporated in the lattice.

Thermally stimulated luminescence. The $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor was studied for its TL/OSL defects. Fig. 3 shows the typical TL glow curve of $\text{SrSO}_4:\text{Eu}^{2+}$ and its comparison with the commercial $\alpha\text{-Al}_2\text{O}_3:\text{C}$ (TLD-500) irradiated to the same dose. The $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor was found to be 100 times more sensitive than that of the commercially available $\alpha\text{-Al}_2\text{O}_3:\text{C}$

(both materials were in powdered form). By using a convenient peak-shape method, kinetic parameters such as activation energy and frequency factor of glow curve were calculated [11 – 15]. Fig. 4 presents the TL glow curve of the $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor deconvoluted using Origin software. The glow peak does not contain any satellites in the temperature range from 150 to 320°C. The geometrical factor, μ_g , calculated for the given peak, confirms the second-order kinetic nature of the curve. Thus, by second-order approximations, the activation energy was found to be 1.496 eV while the frequency factor was found to be $8.41 \cdot 10^{13} \text{ s}^{-1}$ for the glow peak at 246 °C. Fig. 5, *a* shows the dose response of the phosphor in the range from 20 to 400 mGy, which has been found in linear nature. The linear correlation coefficient of linear fitting was found to be 0.998. Fig. 5, *b* shows the fading response up to 5 days. It is clear from the graph that about 40 % fading occurs at the end of 72 h while after this period the intensity remains constant.

Optically stimulated luminescence (OSL). The sample was studied for its OSL response using blue LED stimulation (470 nm). Fig. 6 shows the optically stimulated (continuous wave) luminescence (CW-OSL) curves of $\text{SrSO}_4:\text{Eu}^{2+}$ for 20 mGy beta-dose. The third-order exponential decay curves of $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor can be seen. The goodness of the

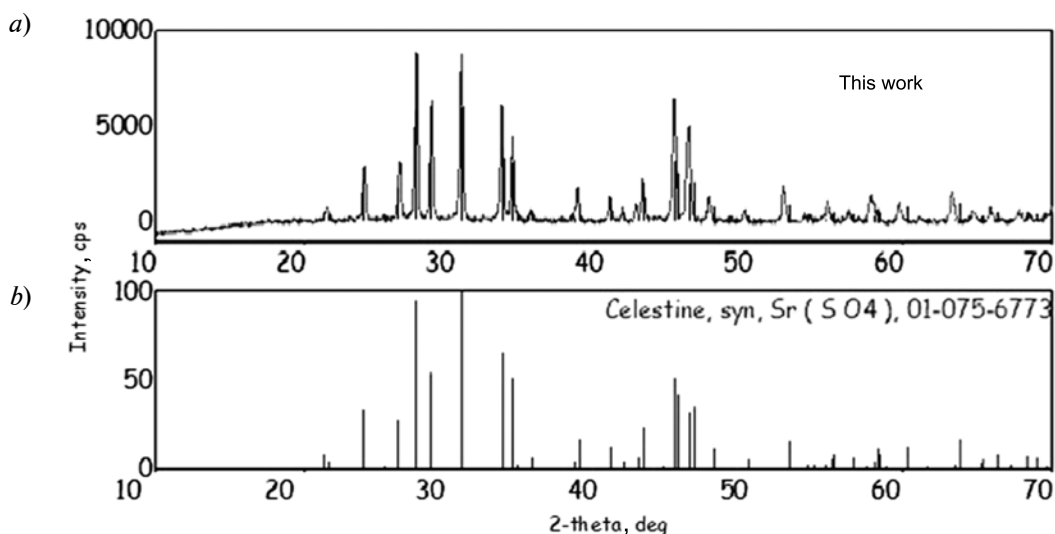


Fig. 2. X-Ray diffraction (XRD) pattern of SrSO_4 prepared by co-precipitation method (*a*) and standard data from ICDD file (*b*)

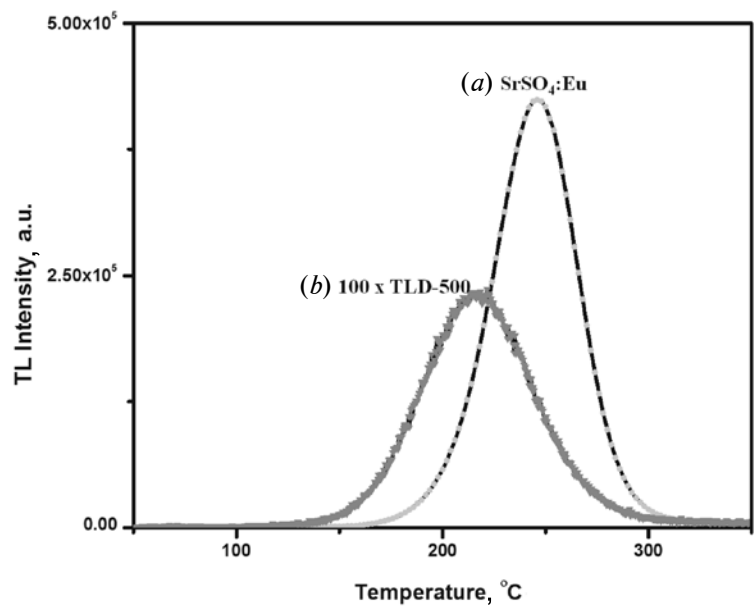


Fig. 3. The comparison of TL intensity for the SrSO₄:Eu²⁺ phosphor (a) with that for TLD-500 (b)

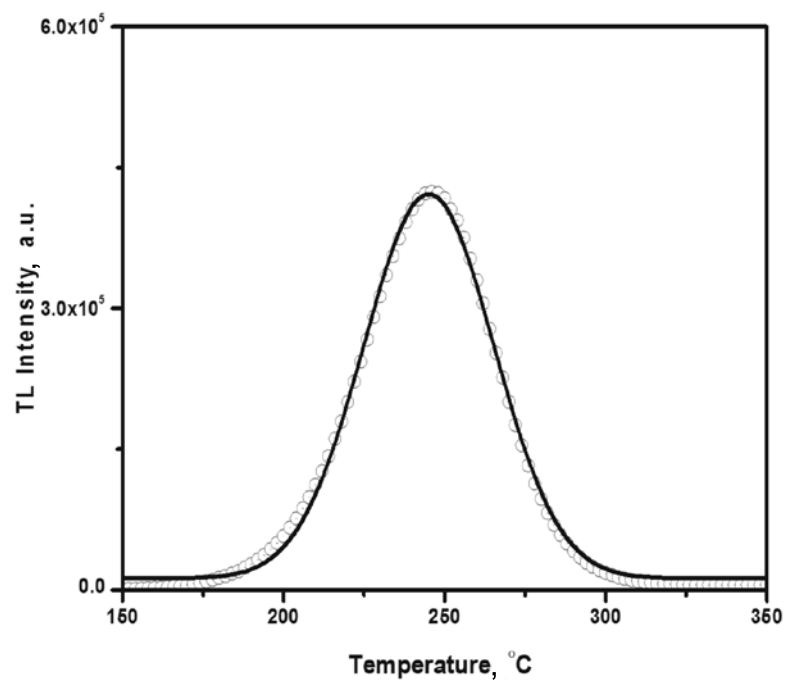


Fig. 4. The deconvolution curve of SrSO₄:Eu²⁺ phosphor

fit was determined by calculating the figure of merit (FOM), and the obtained value of FOM was 0.415% which also confirmed a very good agreement between theoretical and experimental fitting. The OSL components from the CW-OSL decay curve were evaluated

using a third-order equation for the CW-OSL intensity given by the authors of Ref. [16]. The third-order exponential fit to the decay curve showed the presence of three components with photoionization cross-sections of $1.78 \cdot 10^{-17}$, $7.70 \cdot 10^{-17}$ and $17.69 \cdot 10^{-17}$ cm², respectively.

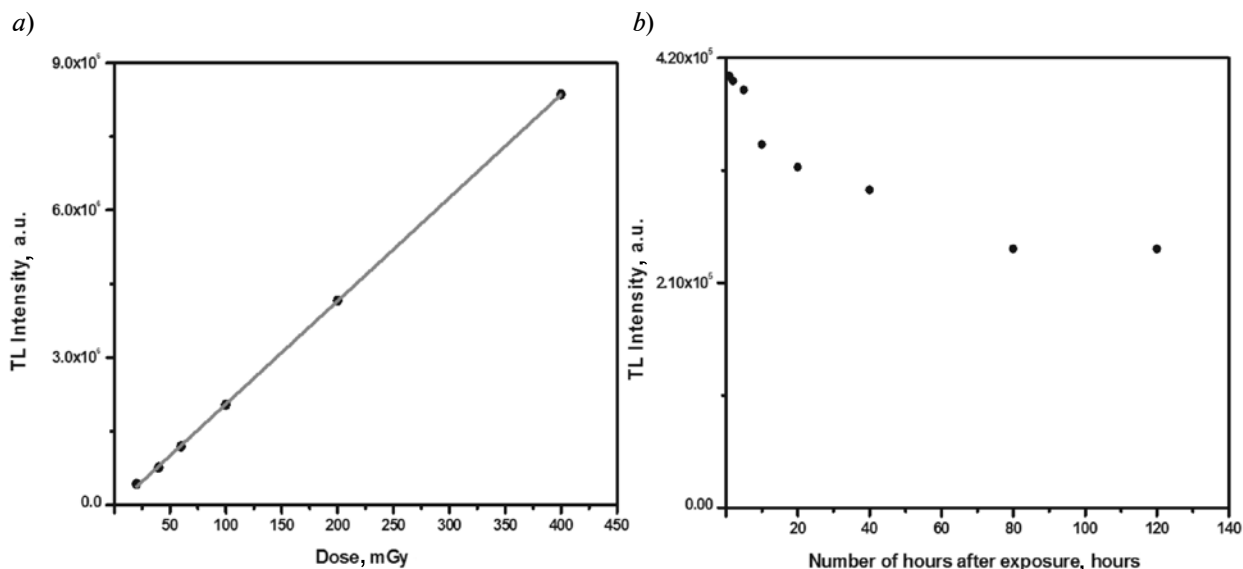


Fig. 5. The dose response of $\text{SrSO}_4:\text{Eu}^{2+}$ sample in the range from 20 to 400 mGy (a) and the fading effect on TL intensity (b)

The OSL sensitivities were compared using two different methods. By the first method, the OSL counts during the first second were compared, whereas the second method was used to take the total area under the OSL curve [17]. Fig. 7 shows the OSL sensitivity of the

$\text{SrSO}_4:\text{Eu}^{2+}$ phosphor compared with that of the commercially available $\alpha\text{-Al}_2\text{O}_3:\text{C}$. The OSL sensitivity of the $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor was found to be 75% of that of the $\text{Al}_2\text{O}_3:\text{C}$ phosphor by the first method (OSL counts during the first second).

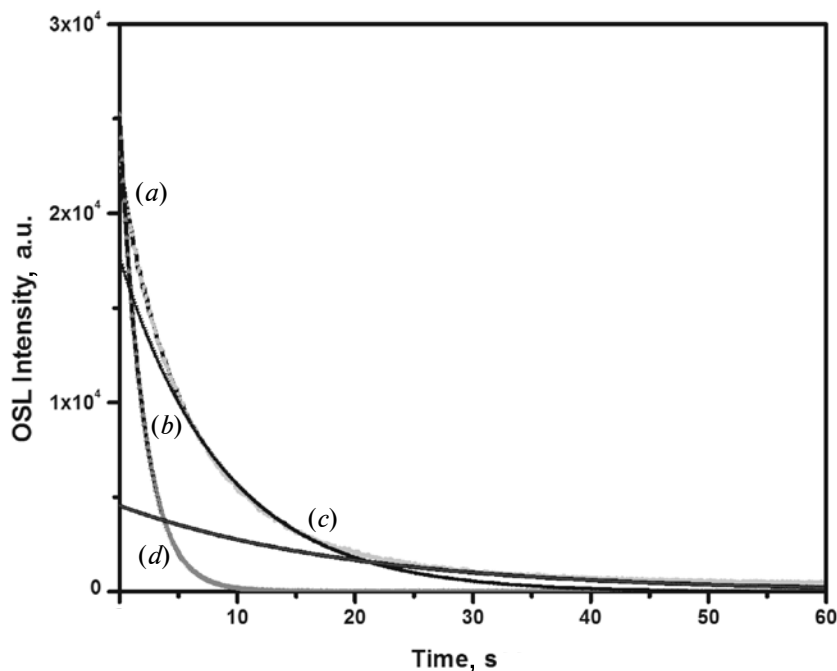


Fig. 6. CW-OSL response of $\text{SrSO}_4:\text{Eu}^{2+}$ for 20mGy beta dose; the third-order exponential fit to the decay curve (a) shows the presence of three components: the first (b), the second (c), the third (d) ones

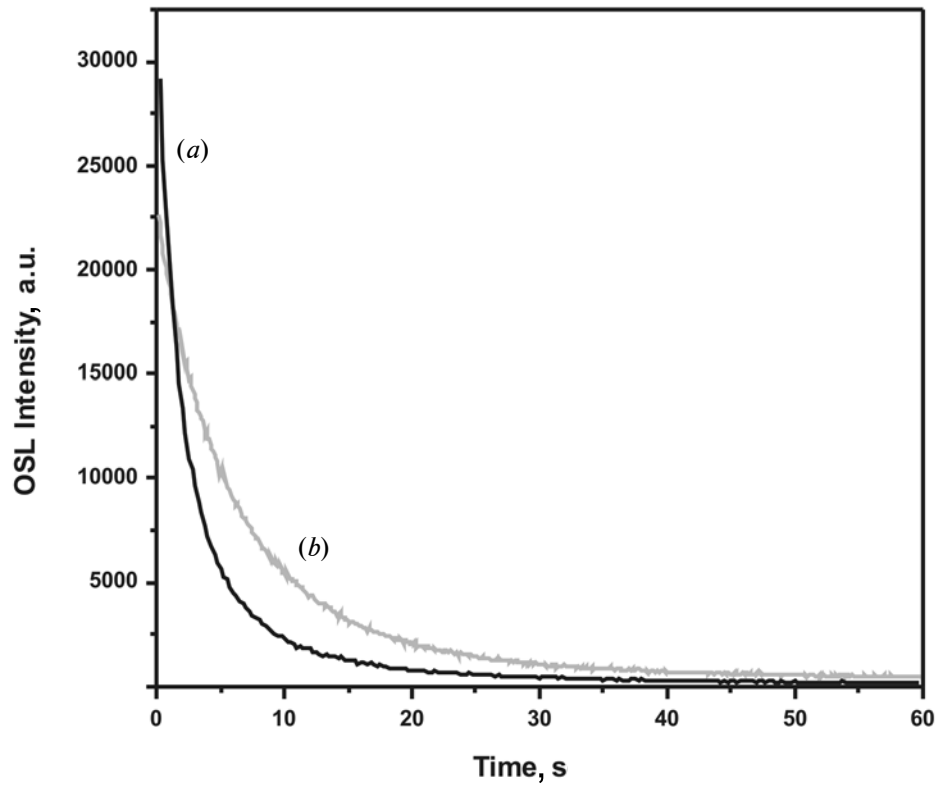


Fig. 7. CW-OSL response of $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor (b) as compared with that of the commercial $\alpha\text{-Al}_2\text{O}_3:\text{C}$ one (a) for 20 mGy beta dose

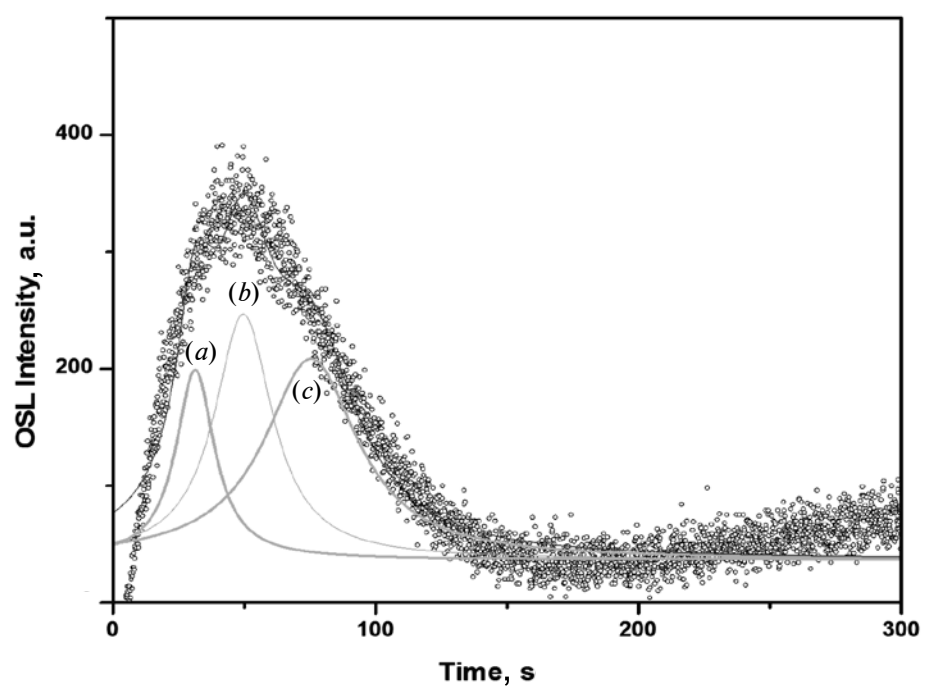


Fig. 8. Deconvolution of the LM-OSL curve for $\text{SrSO}_4:\text{Eu}^{2+}$: the first component (a), the second (b) and the third (c) ones

Table

Kinetic parameters of LM-OSL for the SrSO₄:Eu²⁺ phosphor

Component	μ_g	$\tau = T_m - T_1$	$\delta = T_2 - T_m$	$\omega = T_2 - T_1$
First	0.498	11.30	11.20	22.50
Second	0.502	16.10	16.20	32.30
Third	0.501	26.80	26.90	53.71

Linear modulated optically stimulated luminescence (LM-OSL). The CW-OSL results only exhibit the multiple components but they do not reveal the contribution of traps responsible for the total OSL signal. On the other hand, the application of LM-OSL is expected to separate the different peaks having a distinct spread in the values of photoionization cross section [7]. Fig. 8 presents the LM-OSL response of the SrSO₄:Eu²⁺ phosphor under beta irradiation. The LM-OSL curve is shown to consist of three OSL components similar to those for CW-OSL. The geometrical factor, μ_g , calculated for the given components, confirmed the first-order kinetic nature of the curve components [1]. The calculated results related to the LM-OSL curve are given in Table.

Reusability. This is one of the most

important properties that any dosimetric material should possess. A SrSO₄:Eu²⁺ phosphor disk was exposed to beta radiation of 20 mGy, and the OSL response was measured. Ten such cycles were carried out. The studies showed that it was possible to reuse the phosphor for 10 cycles with no change in the OSL output (Fig. 9).

Minimum detectable dose (MDD). This parameter of the phosphor depends on the standard deviation of the background signal which affects the signal-to-noise ratio. MDD is a function of phosphor sensitivity and instrumentation. The minimum detectable dose was found to be 11.6 mGy corresponding to 3 σ of the background.

Photoluminescence. The combined excitation and emission spectra of the SrSO₄:Eu²⁺ phosphor

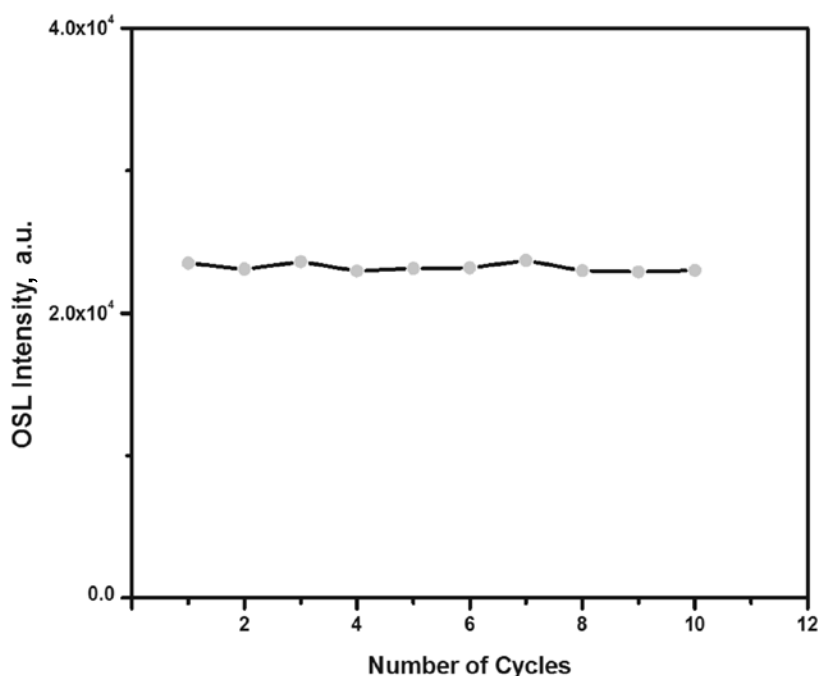


Fig. 9. The result of reusability study of SrSO₄:Eu²⁺

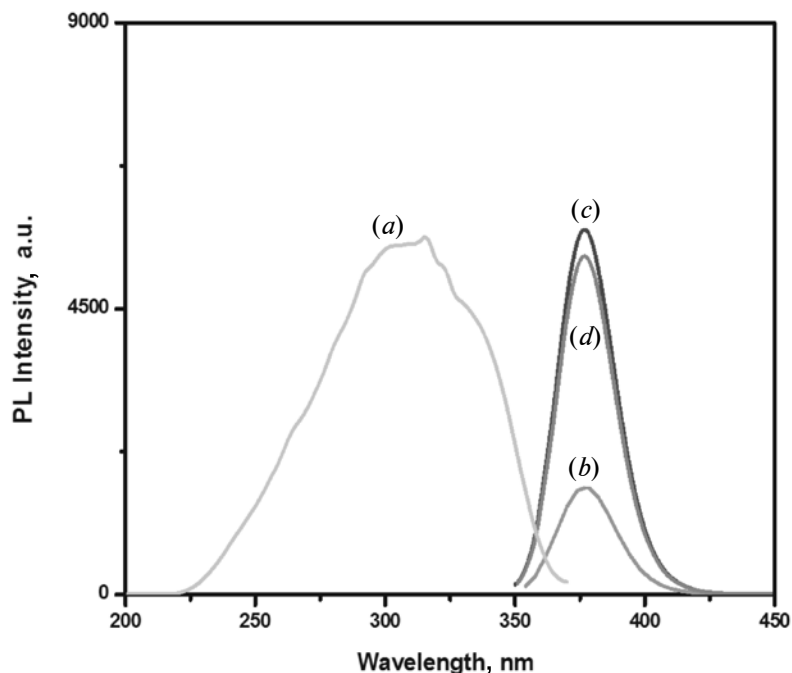


Fig. 10. The combined excitation (a) and emission (the rest) spectra of the $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor. The emission wavelengths (nm): 254 (b), 315 (c) and 323 (d); the excitation at 377 nm

are shown in Fig. 10. The excitation spectra consist of a broad peak around 220 – 360 nm and relatively weak peaks at 254, 315 and 323 nm arising from the transition from $^8S_{7/2}$ state of $4f^7$ configuration to the states belonging to the $4f^65d^1$ one. However, emission was observed at 254, 315 and 323 nm. A narrow peak around 377 nm in the emission spectra was observed for all the excitations. This wavelength corresponds to the transition from the lowest band of the $4f^65d^1$ configuration to the $^8S_{7/2}$ state of the $4f^7$ configuration of the Eu^{2+} ion [18].

4. Conclusions

The co-precipitation method was successfully employed for the preparation of potential TLD and OSLD $\text{SrSO}_4:\text{Eu}^{2+}$ phosphor. The XRD profile of $\text{SrSO}_4:\text{Eu}^{2+}$ was in good agreement with the ICDD file. The comparison of TL and OSL sensitivities showed that the former for $\text{SrSO}_4:\text{Eu}^{2+}$ was 100 times higher than that for TLD-500 and the latter for $\text{SrSO}_4:\text{Eu}^{2+}$ was 75% of the sensitivity for the commercially available $\alpha\text{-Al}_2\text{O}_3:\text{C}$ phosphor (TLD-500). The CW-OSL decay curve was found to consist of three OSL components having photoionization

cross-sections of $1.78 \cdot 10^{-17}$, $7.70 \cdot 10^{-17}$ and $17.69 \cdot 10^{-17} \text{ cm}^2$, respectively. OSL components were determined from the CW and the LM-OSL data. The minimum detectable dose (MDD) was found to be 11.6 mGy with 3σ of background. Also the reusability studies showed that the phosphor could be reused for 10 cycles without any change in the OSL output. In the TL mode phosphor showed the linear dose response. The fading turned out to be up to 40 % at the end of 72 h, and after that the TL intensity became constant.

Although $\text{SrSO}_4:\text{Eu}$ is not a material equivalent to TLD-500 but, due to its high TL and OSL sensitivities and linear dose response, this phosphor can be proposed as a suitable candidate for radiation dosimetry, of course, after further progress in the studies.

Acknowledgements

One of the authors, Chetan B. Palan, is very much thankful to the Head of Radiological Physics and Advisory Division (RPAD), Bhabha Atomic Research Centre, Mumbai-400085, India, for providing the necessary facilities for the analysis of the OSL and TL results.

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THE AUTHORS

PALAN Chetan B.

Sant Gadge Baba Amravati University, Amravati, India
Amravati, Maharashtra 444602, India
Chetanpalan27@gmail.com

BAJAJ Nikhilesh S.

Sant Gadge Baba Amravati University, Amravati, India
Amravati, Maharashtra 444602, India
nsb.0208@yahoo.com

OMANWAR Shreeniwas Kerba

Sant Gadge Baba Amravati University, Amravati, India
Amravati, Maharashtra 444602, India
Omanwar@rediffmail.com

Палан Ч.Б., Баджадж Н.С., Оманвар Ш.К. ОСНОВНЫЕ РЕЗУЛЬТАТЫ ИЗУЧЕНИЯ ДОЗИМЕТРИЧЕСКИХ СВОЙСТВ ЛЮМИНОФОРА SrSO₄:Eu²⁺.

С помощью метода соосаждения был успешно синтезирован поликристаллический образец люминофора SrSO₄: Eu²⁺, и изучены его люминесцентные свойства. Люминофор проявил высокую



люминесцентную чувствительность (для оптически стимулированной люминесценции (OSL)), составляющую примерно 75 % от таковой для коммерчески доступного $\alpha\text{-Al}_2\text{O}_3\text{:C}$ (TLD-500). Кривая OSL с непрерывной оптической стимуляцией (CW-OSL) состоит из трех участков с сечениями ионизации $1,78 \cdot 10^{-17}$, $7,70 \cdot 10^{-17}$ и $17,69 \cdot 10^{-17}$ см^2 соответственно. Чувствительность термостимулированной люминесценции (TL) составила в 100 раз большую величину, чем у TLD-500. Кинетические параметры для кривой термовысвечивания, такие как энергия активации и частотный фактор процесса, были рассчитаны с помощью обработки формы пика. Компоненты кривой OSL определяли как с помощью данных по непрерывной оптической стимуляции люминесценции, так и по спектру линейно-модулированной оптически стимулированной люминесценции. Найдено, что минимальная детектируемая доза люминофора (MDD) равна 11,6 грей, что втрое превышает уровень фона. Исследования повторного применения люминофора показали, что его можно использовать в течение 10 циклов без изменения выхода OSL. В режиме TL зависимость от мощности дозы поглощенного излучения была почти линейной в диапазоне измерений (20 – 400 грей); фединг составил 40 % через 72 часа. Для спектров испускания фотолюминесценции $\text{SrSO}_4\text{:Eu}^{2+}$ характерно свечение в ближней УФ-области на длинах волн 254, 315 и 323 нм при возбуждении УФ-источником.

МЕТОД СООСЖДЕНИЯ, TLD-500, ОПТИЧЕСКИ СТИМУЛИРОВАННАЯ ЛЮМИНЕСЦЕНЦИЯ, СЕЧЕНИЕ ФОТОИОНИЗАЦИИ.

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СВЕДЕНИЯ ОБ АВТОРАХ

ПАЛАН Четан Б. – *M.Sc. (физика), сотрудник кафедры физики университета Сант Гадж Баба Амравати, г. Амравати, Индия.*

Amravati, Maharashtra 444602, India
Chetanpalan27@gmail.com

БАДЖАДЖ Нихилеш Сабхашандра – *Ph.D. (физика), доцент кафедры физики университета Сант Гадж Баба Амравати, г. Амравати, Индия.*

Amravati, Maharashtra 444602, India
nsb.0208@yahoo.com

ОМАНВАР Шринивас Керба – *Ph.D., профессор, заведующий кафедрой физики университета Сант Гадж Баба Амравати, г. Амравати, Индия.*

Amravati, Maharashtra 444602, India
Omanwar@rediffmail.com