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Full Length Article

Preparation and study of the main dosimetric properties by TL of sintered lithium silicate pellets

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ABSTRACT

This work presents the structural characteristics and the dosimetric properties under ionizing radiation of the lithium silicate (LSO) (LSO) phosphor. The structure of the synthesized material was determined by X-ray diffraction (XRD) method and Rietveld refinement method. The dosimetric properties of LSOLSO in the form of pellets were studied by thermoluminescence (TL) under the effect of different doses of γ-irradiation. The phosphor exhibited a TL emission curve with four TL peaks centered at 100, 182, 250 and 290 °C, with a light emission band centered at 385 nm. The TL dose-dependent γ-radiation dose response of the TL peak at 182 °C was linear in the low-dose region, from the order of mGy to 50 Gy. In addition, the phosphor exhibits lower fading, good reproducibility, and sensitivity of the order of commercial TLD-100 dosimeters.

1. Introduction

Radiation quantification or dosimetry can be performed in several ways, generally based on the physical effects caused by radiation on a given material. One way of detecting radiation is by using thermoluminescent materials. These materials are capable of emitting light when heated provided they are previously exposed to ionizing radiation.

Thermoluminescence dosimetry is used in a wide range of doses, including in vivo personal dosimetry, due to its numerous advantages that allow the evaluation of absorbed dose in critical organs and in difficult geometries. In addition, thermoluminescent detectors are generally composed of a single material and are small in size, which makes the TL reading of the detector independent of the angular distribution of the radiation [1,2].

The glow curve and the TL emission spectrum are the main characteristics of a thermoluminescent material for TL dosimetry (TLD) applications. It is preferable for a TL material to have a simple emission curve with few and separate peaks [3]. In addition, the intensity of a given TL peak should exhibit a linear behavior with radiation dose over a wide range. However, this is not always possible, and in many cases, it is used in a certain dose range in which the dosimeter response is linear [4]. Another important characteristic of a TLD material is the fading of its thermoluminescent signal due to storage time. Since TLDs are passive dosimeters, it is necessary that their fading be minimal [5].

Silicates are an attractive class of materials among inorganic phosphors, due to a wide range of applications and certain properties, such as chemical stability, transparency to visible light, physicochemical stability at high temperatures, among others. A very important characteristic of silicates is the sensitivity to ionizing radiation for a certain energy range [6–8].

Silicates with a cation in their crystal structure, such as pure and doped lithium silicate synthesized by different methods [9–11], exhibit certain properties like luminescence in the visible region [12], physicochemical stability at high temperatures [13] and radiation stability [14].

Abramenkovs et al. [15] studied the effect of gamma and neutron radiation on lithium silicate pellets doped with different concentrations of Cr^{3+} and Fe^{3+} ions. They observed that the TL of this material is affected by the addition of these ions, reducing its TL intensity by five times. In addition, they observed that the maxima of the TL peaks

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change for low temperature regions with increasing concentration of the dopants, affecting the thermal stability of the defects responsible for the TL light emission.

Singh et al. [16] synthesized Ce^{3+} doped lithium silicate using the combustion synthesis method and through correlation studies between TL and electron paramagnetic resonance technique identified the radiation-induced defect centers responsible for the TL emission of this material. They observed two intense TL peaks at approximately 110 and 250 °C and four low intensity peaks at 160 , 190 , 320 and 370 °C. Also, they show that Ti^{3+} ion participates as a recombination center giving rise to the peak at 110 and 250 °C.

Barve et al. [17] produced lithium silicate samples by a simple co-precipitation method with commonly available chemicals and showed that Cu-doped lithium silicate exhibits significant properties in its optically stimulated luminescence (OSL) response for application in dosimetry. In addition, Barve et al. [17] and Nur et al. [18] indicate that lithium silicate is a promising material for applications in the field of radiation dosimetry because it has a low Z, with $Z_{eff} = 10.5$ and 10.93 for Li_2SiO_3 and Li_4SiO_4, respectively.

In a recent study, Aynaya-Cahui et al. [19] show that pure lithium silicate synthesized by the solid-state reaction method possesses high sensitivity in its TL response for a wide range of doses. Furthermore, they identify the defect centers induced in the lithium silicate system by γ-irradiation responsible for the TL emission. However, there is no published information on the dosimetric characteristics of this material for its application in TLD. Therefore, there is a need to analyze the dosimetric properties of this material in order to be considered as a TL dosimeter.

In the present work, we have synthesized lithium silicate by solid-state reaction sintering method and studied the dosimetric properties in terms of its TL response such as: linear response to γ-exposure, fading behavior, sensitivity to γ-exposure, minimum detection dose, repeatability, and its thermoluminescence emission characteristics. Furthermore, we verified whether the phosphor synthesized by the solid-state reaction technique exhibits luminescent response by optical stimulation.

2. Materials and methods

In this work, lithium silicate (LSO) was prepared following the high-temperature solid-state reaction synthesis. Lithium carbonate (Li_2CO_3, Aldrich Chemicals, 99.0%) and silicon dioxide (SiO_2, Aldrich Chemicals, 99.0%) were mixed in the molar ratio of Li/Si = 2, according to the Li_2SiO_3 chemical formula. The mixture of these precursors was thoroughly homogenized in a mill using alumina spheres for 4 h and then transferred to alumina crucibles for calcination. The temperature profile of the calcination of the mixture followed an automatic process in a furnace trademarked Nabertherm. The temperature profile started from room temperature rising up to 900 °C with 5 °C/min of heating rate [19]. The temperature was maintained at 900 °C for 10 h and then the furnace was turned off and allowed to cool to room temperature. Fig. 1 (a) shows the phosphor synthesized by the solid-state reaction method.

Powder X-ray diffraction (XRD) analysis on the synthetized sample was used for phase identification and to study LSO microstructural characteristics. The XRD analysis were performed in the Rigaku model miniFlex 600 spectrometer, which uses the Bragg-Brentano geometry. Previous to the analysis, the powder LSO sample was grounded using an agate mortar to decrease particle sizes in order to reduce the particle statistics error [20]. The configuration of XRD acquisition used the Cu-Kα radiation in the 2θ range between 15° and 63° with a step width of 0.005° and a counting time of 0.15 s per step. Both, phase identification and Rietveld refinement were carried out using the software X'pert HighScore Plus version 3.0.

In order to evaluate the dosimetric properties of LSO, pellets with sufficient mechanical strength were fabricated from the fine powder of LSO by pressing with a pressure of 11 ton/cm^2 at room temperature followed by a sintering process at 800 °C for 4 h. These pellets are shown in Fig. 1(b). The pellets have 6.0 mm in diameter, 1.0 mm thick, and have a mass of 50 mg each. After producing a number of 100 pellets, they were irradiated with ^{60}Co γ-radiation. Samples having a sensitivity within a 5.5% standard deviation were selected for experimental use. The selection was performed with the reproducibility and heterogeneity test of a number of pellets. The selection process involved seven cycles of annealing at 500 °C for 30 min, followed by irradiation and TL measurements.

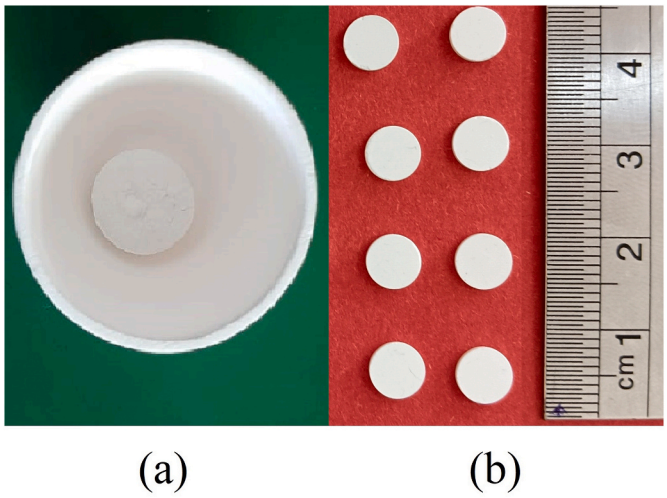

Fig. 1. (a) LSO phosphor obtained by the solid-state reaction method, and (b) LSO pellets of 6.0.0 mm in diameter and 1.0.0 mm thick.

Irradiation of the LSO pellets for TL measurements was performed with a ^{60}Co γ-source facility, model Gammatron with a dose rate of 97.3 mGy/min at a distance of 10 cm from the source. The γ-irradiation was performed at room temperature and under electronic equilibrium conditions. On the other hand, to investigate the OSL emission behavior of LSO phosphor with radiation dose, the phosphor were irradiated with a $^{90}Sr/^{90}Y$ β-source at a dose rate of 81.6 mGy/s from the Risø TL/OSL reader.

TL measurements were performed with a Harshaw TL reader, model 3500, with heating temperature capability up to 400 °C. This instrument is equipped with a Hamamatsu bialkali photomultiplier tube (PMT), model ETE 9125B, and a Schott KG1 optical filter with a transmission band between 330 and 690 nm for light detection. TL glow curve measurements were carried out with a linear heating rate of 4 °C/s and with a constant flow of nitrogen. Before TL measurements, the LSO pellets were preheated at 100 °C for 10 s in the same TL reader tray, in order to empty the shallow traps responsible for the unstable luminescence at low temperature. TL measurements were performed immediately after this procedure.

OSL measurements were carried out at room temperature using an automated Risø TL/OSL reader, model DA-20. The OSL signal was stimulated in a continuous-wave (CW) mode using a wavelength of 470 nm and with a power of 80 mW/cm^2. The OSL signal was detected for 40 s by a bialkali PMT through a 7.5 mm thick Hoya U-340 optical filter with a transmission band between 250 and 390 nm.

The 3D TL emission spectrum was obtained by directly coupling a monochromator in front of the PMT of the Risø TL/OSL reader and the same experimental parameters for TL glow curve measurements were used without the filter. The scanning was performed from 200 to 880 nm, with a step of 20 nm.

3. Results and discussion

3.1. X-ray diffraction

The XRD pattern of the LSO phosphor is shown in Fig. 2. A qualitative

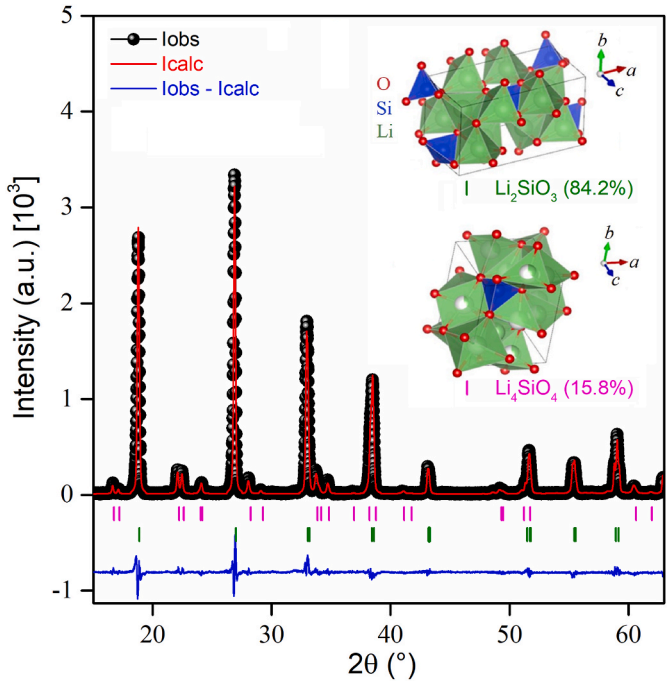

Fig. 2. X-ray diffraction patterns of LSO phosphor (black balls). Qualitative phase identification shows that the LSO phosphor is lithium metasilicates (Li_2SiO_3), and lithium orthosilicates (Li_4SiO_4). Rietveld refinement results of the XRD pattern of the LSO phosphor (red line), and the difference between observed and experimental intensity (blue line).

phase analysis indicates that the samples correspond to lithium metasilicate (Li_2SiO_3) (JCPDS card number, 01-029-0828) with orthorhombic structure and space group $Cmc2_1$. Due to the presence of low-intensity diffraction peaks in the diffractogram, the crystal structure of the synthesized LSO phosphor was analyzed by Rietveld refinement of XRD data. PANalytical X'Pert HighScore Plus software was used for the refinement and to determine the percentage of the crystalline phases in the sample. The results showed that the LSO phosphor crystallized 84.2% in the orthorhombic phase (lithium metasilicate - Li_2SiO_3) with space group $Cmc2_1$ and 15.8% in the monoclinic phase (lithium orthosilicate - Li_4SiO_4) with space group $P2_1m$. The orthorhombic and monoclinic phase crystal structure of the unit cell of Li_2SiO_3 and Li_4SiO_4, respectively, generated with the VESTA program from the lattice parameters obtained from the Rietveld refinement is shown in the inset of Fig. 2. This structural analysis demonstrates that the synthesis method and the ratio of the chemical reagents used are suitable to produce LSOLSO phosphor.

3.2. Thermoluminescence

3.2.1. TL glow curve

LSO pellets with TL peaks at 182 °C between ±5.5% of the relative standard deviation were selected to study their TL response to radiation dose. Fig. 3 shows TL glow curves of LSO pellets exposed to γ-radiation of ^{60}Co for doses ranging from 2 mGy to 50 Gy. Each TL glow curve in Fig. 3 is the average of the responses of the five pellets with a standard deviation of less than 5.5% in all cases evaluated for the peak at 182 °C. For this evaluation, the set of five pellets was thermally treated at 500 °C for 30 min aiming to free trapped electrons in the LSO phosphor avoiding undesirable contributions to the TL signal. In general, the TL emission of LSO pellets presents four local maximums at about 100, 182, 250 and 290 °C, and the maximum intensity is displayed by the 182 °C TL peak. It is observed that the first maximum at 100 °C is totally faded after 1 h. Therefore, a preheating treatment at 100 °C for 10 s was applied to completely eliminate the unstable peak at 100 °C. The

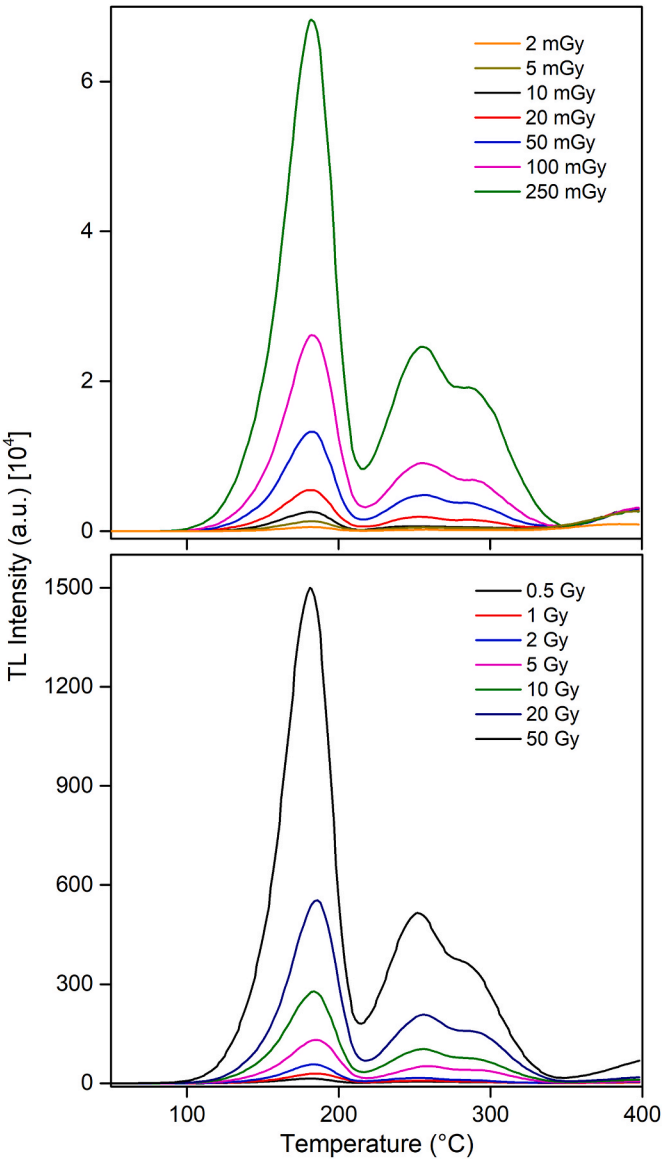

Fig. 3. TL glow curves of LSO pellets irradiated by γ-radiation using a ^{60}Co source. All TL glow curves were obtained with a preheating treatment at 100 °C for 10 s. Measurements were performed on the TL Harshaw 3500 reader.

position of the remaining three peaks remains at the same temperature as the γ-dose increases without changing the shape of the glow curve, but the relative heights of the three peaks change as a function of γ-radiation.

In a recent paper, Aynaya-Cahui et al. [19] show the presence of TL peaks at temperatures above 300 °C for lithium silicate powder samples. However, as we can observe in Fig. 3, these TL peaks above 300 °C have a very low intensity in the pellets sample sintered at 800 °C for 4 h. Therefore, this result shows that the defect centers responsible for the TL peaks above 300 °C are affected by the sintering temperature of the pellets.

3.2.2. TL emission spectrum

Fig. 4 shows the 3D TL emission spectrum of LSO phosphor irradiated with β-radiation at room temperature with a dose of 34.21 Gy. The shape and position of the maximum TL peaks are similar to the TL glow curve obtained with γ-radiation. A band was found in the range 265–550 nm centered at 385 nm. This result indicates that three electron trapping centers and one recombination center are involved during the TL

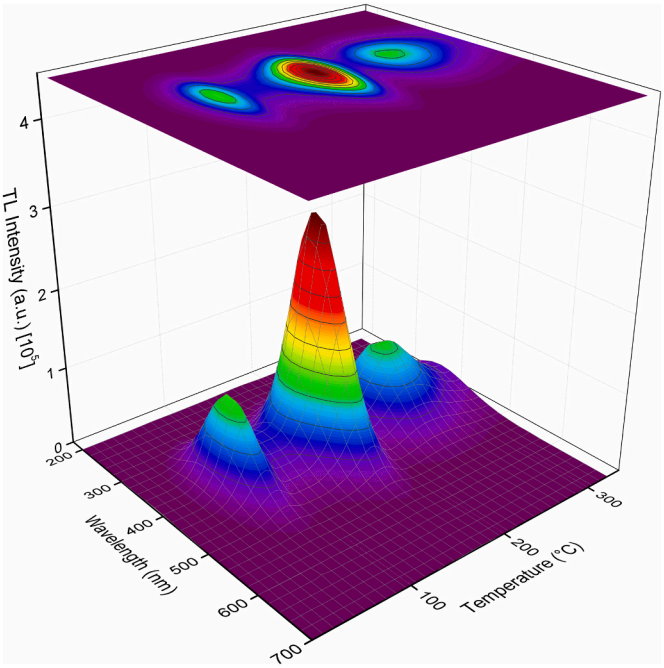

Fig. 4. TL emission spectra of the LSO phosphor measured for a dose of 34.21 Gy. 3D representation (bottom) and contour plot (top). Measurements performed on the Risø TL/OSL reader.

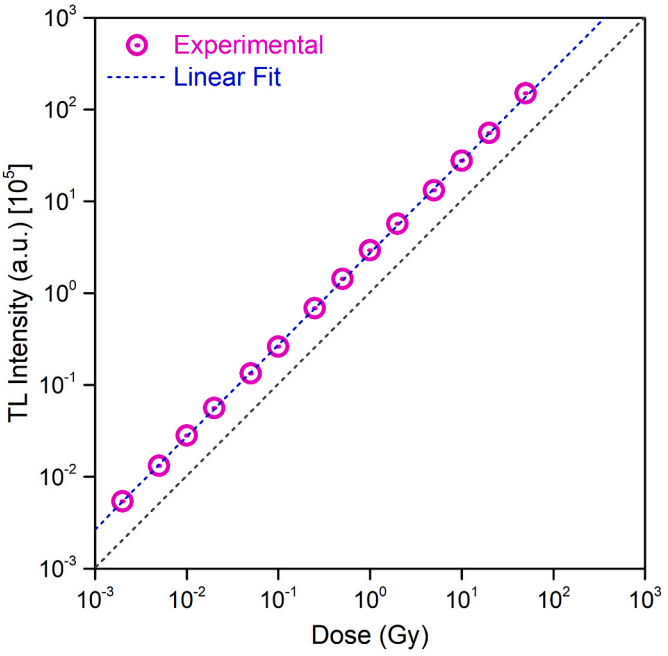

Fig. 5. TL intensity of the maximum at 182 °C for γ-radiation dose from 2 mGy to 50 Gy. Both axes are presented in logarithmic scale for a better representation of the data. The dashed black lines indicate linearity and the dashed blue line is the fit from the experimental results using a linear equation. Measurements were performed on the Harshaw TL 3500 reader.

emission process. This recombination center is due to the presence of defect centers associated to Ti^{3+} ions as observed by Aynaya-Cahui et al. [19] and Singh et al. [16] in lithium silicate samples. The TL spectrum shows that the first TL peak at 100 °C is slightly offset in position relative to the TL peak presented in Fig. 3. This displacement is due to the fact that the TL spectrum was recorded immediately after irradiation.

3.2.3. Linearity

Thermoluminescent materials with linear behavior of TL intensity versus absorbed dose in the required dose region are considered suitable for applications in radiation dosimetry [5]. The linearity analysis of the TLD response is performed with the calibration curve. The dose-response curve can be constructed using the areas under the emission TL curves as a function of the deposited doses or using the maximum peak TL intensities as a function of the deposited doses if the peak is well-defined. For each absorbed dose, there should be a corresponding maximum peak area or peak intensity value. These two parameters can be referred to as the TL response of the dosimeter. In this work, since the TL emission curve of the LSO pellets present a well-defined peak at around 182 °C, it was chosen to represent the dose-response curve by the ratio between the maximum peak intensity and the absorbed dose. The linearity study was performed by analyzing the graphs of the calibration curves with logarithmic axes and decades of the same dimensions. In this way, we can observe graphically whether the curve has a linear (slope = 45°), supra-linear (>45°) or sub-linear (<45°) behavior. The behavior of the TL intensity peak at around 182 °C was analyzed for exposed doses of γ-radiation (^{60}Co) up to 50 Gy. It is clearly observed in Fig. 5 that the linear fit (blue-spaced line) and the linearity curve (black-spaced line) are parallel, indicating that the LSO pellets present a linear response in the analyzed dose range.

3.2.4. Minimum detection dose

The minimum detection dose (MDD) depends on a combination of the TL sensitivity of the dosimetric material and the detection system using optimal photomultiplier tubes for the quantification of the emitted TL light. Fig. 6 presents the determination of the MDD, defined as three times the standard deviation ($3\sigma_{TL}$) of the zero-dose reading of the

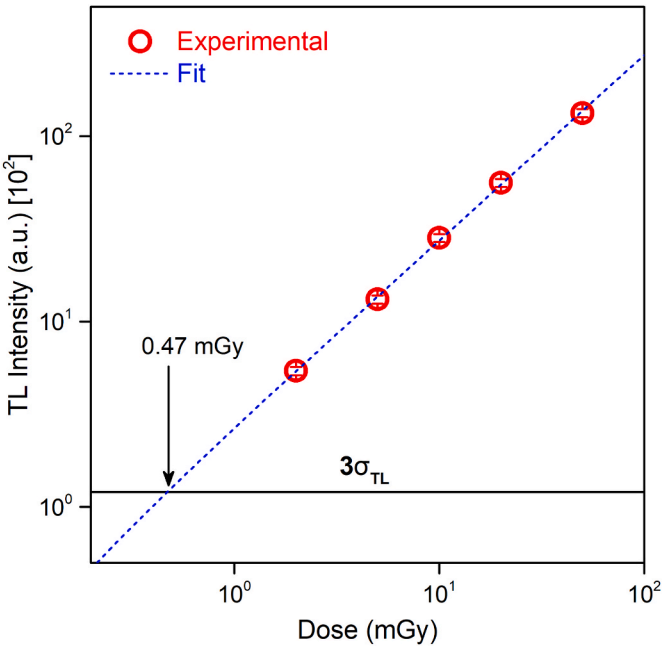

Fig. 6. TL intensity of the maximum at 182 °C as a function of γ-radiation dose from 2 to 50 mGy, and minimum detectable dose (MDD) for LSO pellets. The dashed blue line is the fit from the experimental results using a linear equation. Measurements were performed on the TL Harshaw 3500 reader.

dosimeters [21]. An MDD of 0.45 mGy was obtained for LSO pellets using the Harshaw 3500 TL reader.

3.2.5. Sensitivity

Fig. 7 presents a comparison of the TL sensitivity of the LSO pellets and commercially available TLD-100 chips, irradiated with the same

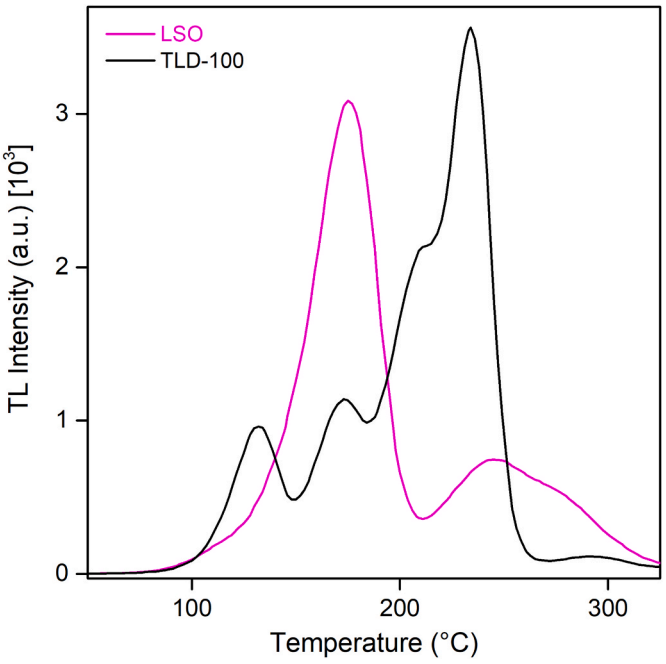

Fig. 7. TL sensitivity comparison of LSO pellets with TLD-100 dosimeter irradiated with a γ-dose of 1 Gy. TL intensity is normalized to the mass of each dosimeter. Measurements were performed on the TL Harshaw 3500 reader.

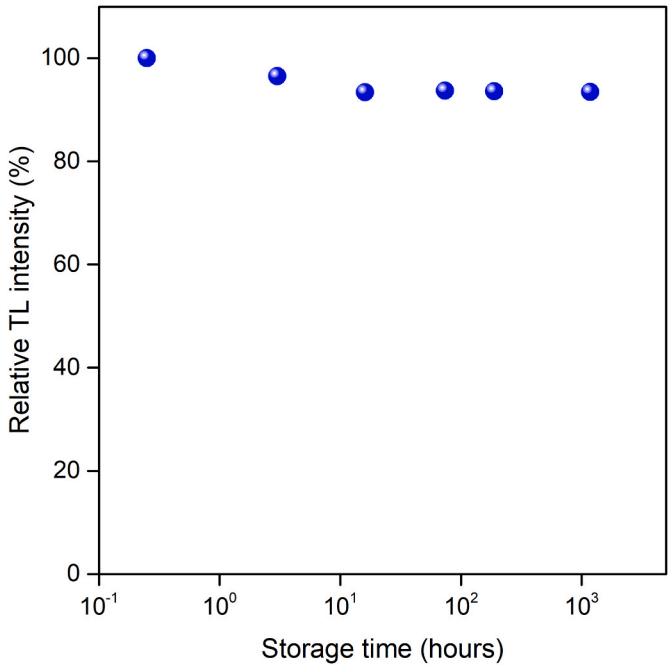

Fig. 8. Fading of the main TL peak at 182 °C of LSO pellets, storage in dark at room temperature. Measurements were performed on the TL Harshaw 3500 reader.

dose of γ-radiation (1 Gy). For comparison purposes, the TL intensity of both samples in Fig. 7 was normalized by the mean value of the mass of a group of five chips. It can be seen that the maximum intensity of the TL peak at 182 °C of the present sample has approximately 0.87 times the intensity of the main dosimetric peak of TLD-100. Since the maximum peak for the LSO appears to have its major contribution from a single TL curve, this characteristic would be an important advantage with respect to the maximum of the TLD-100 at 234 °C because it is clearly affected by other TL curves. In addition, the annealing procedure before and after the TL reading of this phosphor is very simple compared to the complicated two-stage annealing procedure followed for the TL-100 to maintain the sensitivity and shape of the glow curve for reuse.

3.2.6. *Fading*

The basis of the TL models is the excitation of electrons in the valence band due to exposure to a radiation field. The exited electrons change their state to energy levels in the conduction band where they are trapped by trapping centers [22]. The maintenance of electrons in their trapped states depends on the environmental condition and the condition of storage of the dosimeters. The quality of storage of the absorbed radiation energy by the TL material by the trapped electrons and their stability with respect to time is named fading. A material with fading of less than 20% per month up to a storage temperature of 50 °C is considered suitable for dosimetry applications [23]. On the other hand, the Commission of the European Communities [5] recommends that a fading of less than 5% at room temperature during the time of use of the material is more suitable for application in radiation dosimetry. However, some commercial dosimeters such as TLD-200 (CaF_2:Dy) and TLD-400 (CaF_2:Mn) with 16% fading in weeks and 15% fading in 3 weeks, respectively, are widely used for absorbed dose quantification [24].

Fig. 8 presents a fading evaluation of a set of eight LSO pellets that were packed in a PMMA (polymethyl methacrylate) container and irradiated to 1 Gy of γ-radiation of ^{60}Co. The TL responses of the set of pellets used for this study have a relative standard deviation of 5.7%. Each point in Fig. 8 corresponds to the TL intensity at 182 °C for one LSO pellet. The results show no decay in the TL at 182 °C for a time evaluation of up to approximately 50 days. Fig. 8 shows the fading pattern up to a time of 50 days from the sample removal, indicating that ~7.0% of the TL signal fades in the first 24 h and then stabilizes. Therefore, the LSO pellets prepared in this work may be suitable for application in γ-radiation dosimetry.

3.2.7. *Reproducibility*

Reproducibility after several annealing-irradiation-readout cycles of a detector array is crucial for its potential use in dosimetry [5]. If the TL response or sensitivity of the material does not change with annealing, repeated irradiation, and readout, then it is considered as a good material for TL dosimetry. To verify this property, reproducibility tests of LSO pellets were performed. The LSO pellets were exposed to 1.0 Gy γ-rays from the ^{60}Co source and the TL glow curve was recorded, and then rapidly cooled to room temperature. The same samples were irradiated a second time with the same dose and their TL response was read. Seven annealing-irradiation-readout cycles were performed on the same samples with the same TL reading parameters. Fig. 9 shows the TL intensity behavior of the TL peak at 182 °C of one of the pellets. The standard deviation of the mean TL values of the seven cycles was estimated to be 3.2%. In all LSO pellets, no significant changes in the sensitivity of the dosimetric TL peak were observed. These results show that the LSO pellet exhibits very good reproducibility of the peak at 182 °C, and can be used in radiation dosimetry using the TL technique.

3.3. *Optically stimulated luminescence*

Optically stimulated luminescence (OSL) is a technique that is gradually replacing TL in dosimetry. The fact that it does not require a heating system, which requires considerable energy and relatively longer times of TL experiments, gives OSL very relevant advantages compared to the TL technique. Taking into account these advantages of the OSL technique in relation to TL, the synthesized LSO phosphor was investigated whether the material has the necessary response to be used as dosimeters by OSL. The LSO phosphor was irradiated with a ^{90}Sr/^{90}Y source belonging to the Risø TL/OSL reader itself, with doses between 0.3 and 50 Gy. As can be seen in Fig. 10, the OSL curve of the LSO

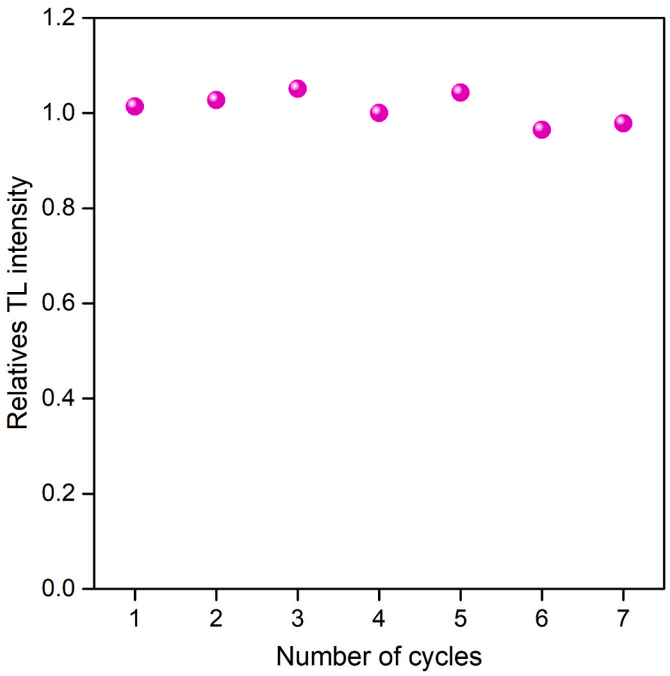

Fig. 9. Reproducibility of the TL signal from LSO pellets over seven repeated cycles of annealing-irradiation-readout. Measurements were performed on the TL Harshaw 3500 reader.

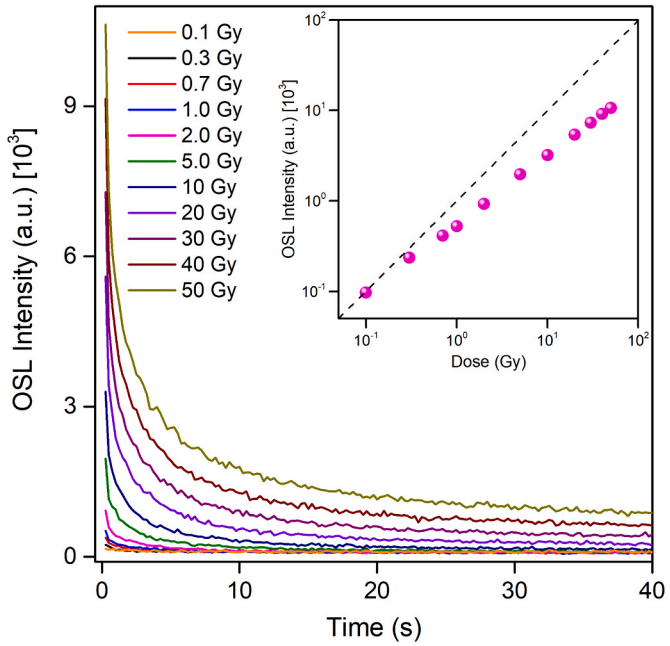

Fig. 10. OSL decay curves (under blue-light stimulation) of LSO phosphor in powder form irradiated with different β-radiation doses of 0.3 Gy up to 50 Gy. Inset: OSL intensity behavior as a function of β-radiation dose; the dashed lines (black) indicate linearity. Measurements were performed on the Risø TL/OSL reader.

phosphor shows an exponential decay as the optically active traps are emptied, indicating that the traps responsible for the OSL decay of LSO phosphor have a high shock section for photoionization to blue LEDs, i.e., the traps responsible for the emissions have a high sensitivity to the stimulus source. The inset of Fig. 10 shows that the OSL intensity increases with increasing β-dose. It is observed that the behavior of the OSL signal with respect to dose is sub-linear over the range of the applied dose. OSL intensities were determined using the first point of the OSL signal. This result implies that the synthesized material with low Z_{eff} (10.5) may have a potential application in dosimetry, but a detailed study is needed to confirm its applicability in dosimetry by analyzing its dosimetric characteristics.

4. Conclusions

LSO phosphor has been successfully synthesized using the solid-state reaction method. The results of powder XRD and Rietveld refinement indicate that the phosphor structure is constituted of two crystalline phases, 84.2% of lithium metasilicate and 15.8% of lithium orthosilicate. The TL glow curve consists of four peaks at 100, 182, 250, and 290 °C. The TL emission spectrum showed that the phosphor LSO emits thermoluminescent light at 385 nm. This would imply that in the synthesized material there is a single recombination center involved in the TL process. The TL response of phosphor LSO irradiated at different γ-doses from 2 mGy to 50 Gy has been measured. It is found that the main dosimetric peak at 182 °C possesses a linear behavior. This peak presented a 7.0% fading of the TL intensity during the first 24 h, being totally constant for periods longer than this time. The TL sensitivity of LSO phosphor was 0.85 times higher than that of commercial dosimeter TLD-100. The minimum detectable dose (MDD) was found to be 0.47 mGy with 3σ background. Reuse studies showed that the phosphor could be reused for several cycles without any change in its TL response. Due to these promising dosimetric properties, LSO pellets produced through the compaction and sintering process at 800 °C are a dosimetric material that has the potential to be used as a solid-state dosimeter in personal and medical radiation measurement.

Credit authors statement

Nilo F. Cano: Conceptualization, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing. **Sandra C. Aynaya-Cahui**: Formal analysis, Investigation, Methodology. **Zaida V. Vilca**: Data curation, Investigation. **René R. Rojas**: Investigation, Methodology, Visualization. **T.K. Gundu Rao**: Formal analysis, Investigation, Validation, Writing – review & editing. **Noemi B. Silva-Carrera**: Investigation. **Alejandro H. Lopez-Gonzales**: Investigation. **Henry S. Javier-Ccallata**: Investigation. **Jorge S. Ayala-Arenas**: Funding acquisition, Project administration, Resources, Validation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The data that has been used is confidential.

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