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Evaluation of TL and OSL responses of CaF_2:Tm for electron beam processing dosimetry

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ABSTRACT

The thermoluminescence (TL) and infrared stimulated luminescence (IRSL) responses of in-house produced CaF_2:Tm dosimeters are investigated in this work, envisaging their application in electron beam (EB) radiation processing. The irradiations were performed at an industrial EB accelerator (1.5 MeV) covering a dose rate range of 2–8 kGy/s and dose up to 10 kGy. In general, the TL glow curves display four peaks, termed as peaks 2, 3, 4, and 5, corresponding to temperatures at ~150, 200, 240, and 300 °C, respectively. The intensity of the low-temperature peaks (2 and 3) grows with the dose, while the others remain constant (saturated). Nevertheless, an evident dose effect on the glow curves manifests in decreased peak3/peak2 ratio with increasing doses.

The CW-IRSL curves exhibit similar patterns with an initial signal increase, followed by an exponential decay. Instead of the normal monotonic decays, these peak-shaped curves might be due to the charge capture competition between empty shallow traps and recombination centers. Both TL and IRSL intensities increase linearly with doses up to 6 kGy, and for higher doses, they become sub-linear with a saturation trend around 10 kGy. Another common feature of TL/IRSL response is its dose rate dependence, being more sensitive at higher dose rates. Despite being dose-rate dependent, the CaF_2:Tm dosimeters might be suitable for EB processing dosimetry. However, for their use as routine dosimeters, relevant dosimetric characteristics, such as fading and response reproducibility, have to be investigated. Work in this direction is underway.

1. Introduction

Thulium-doped calcium fluoride (CaF_2:Tm), commercially known as TLD-300, is a thermoluminescent (TL) dosimeter widely used for electromagnetic radiation, neutrons, and charged particles (Lucas and Kapsar, 1977; Wang et al., 1989; Bos et al., 1995; Massillon et al., 2008; Muñoz et al., 2015; Rojas-López et al., 2019). In general, the glow curves induced by these radiations have a similar structure with two well-resolved peaks whose intensity is different depending on the radiation linear energy transfer (LET). This property has allowed these dosimeters to be used in mixed radiation fields composed of light and heavy charged particles from accelerators (Hoffmann and Prediger, 1983; Marczewska et al., 2001; Hajek et al., 2008; Muñoz et al., 2015).

Due to the wide applications range of CaF_2, several routes for its fabrication are available in the literature, such as precipitation of $CaCl_2$ + NaF, followed by sintering (Dhopte et al., 1992), sintering with rare-earth dopants (Fukuda, 2002), Sol-gel (Zhou et al., 2007), chemical reactions under nitrogen atmosphere (Nandiyanto et al., 2011). However, most of them are time-consuming, use hazardous materials (such as HF), and need oxygen-free atmospheres, or crystal-growing infrastructure, restricting its widespread fabrication.

Some years ago, our group synthesized CaF_2:Tm via a liquid-phase solution (Vasconcelos et al., 2014), adapted from (Nandiyanto et al., 2011), and in continuity, via solution combustion synthesis (SCS) technique based on the oxidation-reduction reaction between nitrate and fuel at relatively low temperatures (500 °C) (Vasconcelos et al., 2016).

Recently, the optically stimulated luminescence (OSL) response of the CaF_2:Tm dosimeters, produced via SCS technique, was investigated with blue (BSL) and infrared stimulation (IRSL) for beta doses over 2.5 Gy using a $^{90}Sr/Y^{90}$ radioactive source (Asfora et al., 2016). These results point to the feasibility of using CaF_2:Tm as OSL dosimeters, as predicted by (Nanto et al., 2015).

Furthermore, the TL and OSL dosimetry properties of CaF_2:Tm and

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Table 1
Doses received by the pellets in one pass through the irradiation zone at 6/min speed over a dose-rate range of 2–8 kGy/s.

Beam Current (mA)	Dose rate (kGy/s)	Dose per pass (kGy)
0.5	2	0.5
1.0	4	1.0
1.5	6	1.5
2.0	8	2.0

its in-house production may favor using these dosimeters in the electron beam (EB) processing dosimetry (Martins and Silva, 2014). In this field, dosimetry plays an important role in characterizing EB facilities, establishing the process, and routine monitoring (IAEA - TECDOC 1386, 2004). All these activities have been carried out by calorimeters, alanine/EPR dosimeters, cellulose triacetate (CTA) and, radiochromic films (ICRU Report 80, 2008; ISO/ASTM 51649, 2015). Within this context, this work aims to evaluate the TL and IRSL response of CaF_2:Tm pellets for routine monitoring EB processing applications with a few tents of kGy like, for example, biological samples, food irradiation, sprout inhibiting and, parasite control. (Cleland, 2006; Calvo et al., 2012).

2. Materials and methods

Pellets of CaF_2: Tm, produced via the SCS route, with a Tm concentration of 0.2% mol, an average mass of (48.3 ± 1.1) mg, 6 mm in diameter, and 2 mm in thickness, were used in this work. A detailed description of the CaF_2:Tm pellets production and their dosimetric TL properties for gamma rays from ^{60}Co can be found in our previous paper (Vasconcelos et al., 2016). Fifty samples from the same batch, with TL/OSL sensitivity variation lower than 6%, were selected to carry out all measurements.

Irradiations of the CaF_2:Tm pellets were performed in free air with a 1.5 MeV electron beam from the accelerator DC 1500/25/04 - JOB 188 at the Instituto de Pesquisas Energéticas e Nucleares (IPEN). The reference dose of this facility, previously calibrated using alanine standard reference dosimeters with traceability to the National Physical Laboratory, is expressed in absorbed dose to water. The relevant features of the EB irradiator are: scan width of 1.02 m, beam length of 2.54 cm, and surface dose uniformity better than 6.2% (k = 2) in the center of the scan width, hereafter referred to as reference position (Kuntz et al., 2015).

At each step of irradiation, four pellets were placed in a PMMA dosimeter holder previously fixed on the conveyor tray at the reference position and conducted at a speed of 6 m/min through the irradiation zone. The dose rate range of 2–8 kGy/s covered in this work was achieved by varying the beam current from 0.5 up to 2.0 mA. The doses attainable in one single pass of the dosimeter through the beam length, at different dose-rates, are presented in Table 1.

Before either TL or OSL readings, all pellets were preheated at 100 °C for 15 min to empty shallow traps and unstable peaks at low temperatures. Shortly after that, both TL and IRSL measurements were carried out by a Riso TL/OSL reader, model DA-20, using the optical filter Hoya U-340. The TL signal was integrated from 50 °C up to 350 °C at a heating rate of 2 °C/s. The IRSL readings were performed by the continuous-wave optically stimulated luminescence (CW-OSL) technique with

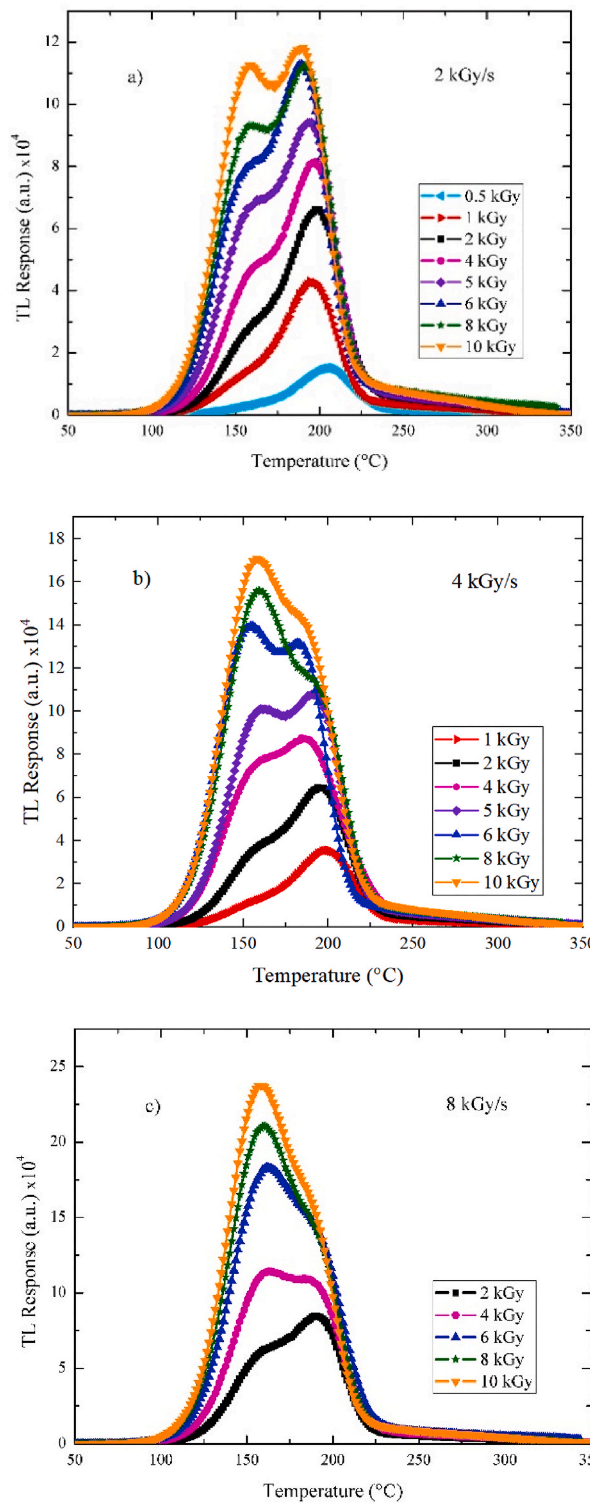

Fig. 1. TL glow curves of CaF_2:Tm irradiated to doses up to 10 kGy and different dose rates: a) 2 kGy/s, b) 4 kGy/s, c) 8 kGy/s.

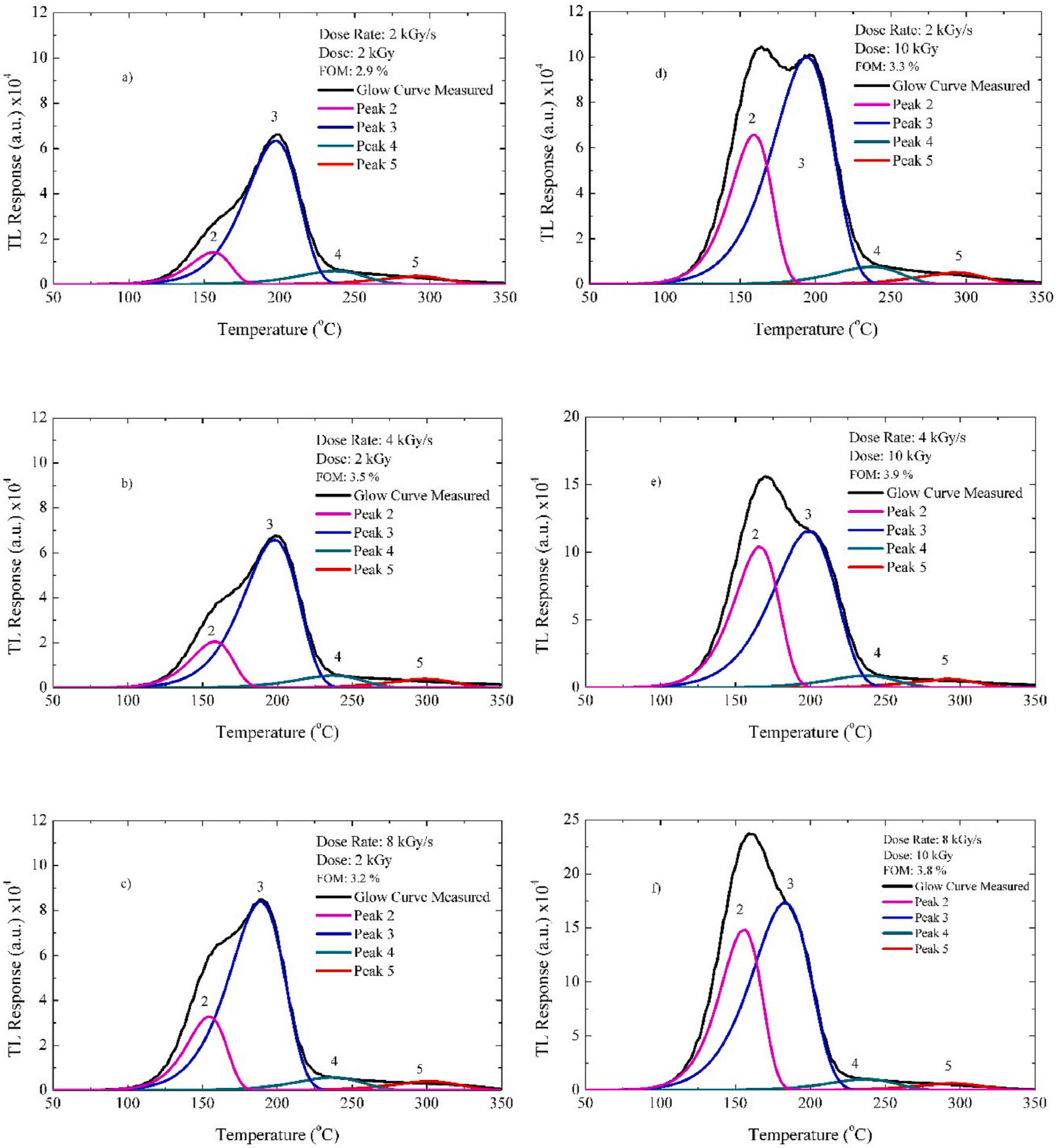

Fig. 2. Peak fitting of TL glow curves of CaF_2:Tm irradiated to 2 kGy and 10 kGy at dose rates ranging from 2 kGy/s to 8 kGy/s. Fig. 2a, b, and 2c corresponding to 2 kGy and doses rates of 2, 4 and 8 kGy/s, respectively. Those gathered at 10 kGy are referred to as Fig. 2d (2 kGy/s), Fig. 2e (4 kGy/s), and Fig. 2f (8 kGy/s).

thermal assistance at 150 °C. The pellets were stimulated by a set of light-emitting diodes (LED) with peak emission at 850 nm and optical power of 240 mW/cm^2 during 240 s. Residual TL glow curves were carried out after IRSL stimulation times, under the same TL reading conditions mentioned above.

3. Results and discussion

3.1. TL response

Fig. 1 presents the TL glow curves obtained over doses up to 10 kGy and dose rates of 2, 4, and 8 kGy/s. Each point on the plots is given by

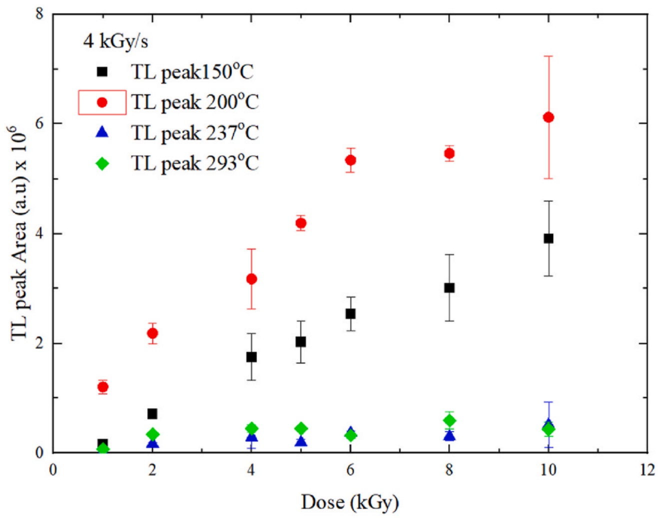

Fig. 3. The intensity of each peak as a function of dose over a range of 10 kGy at 4 kGy/s. The glow curves corresponding to 2 kGy and 10 kGy are shown in Fig. 2b and e.

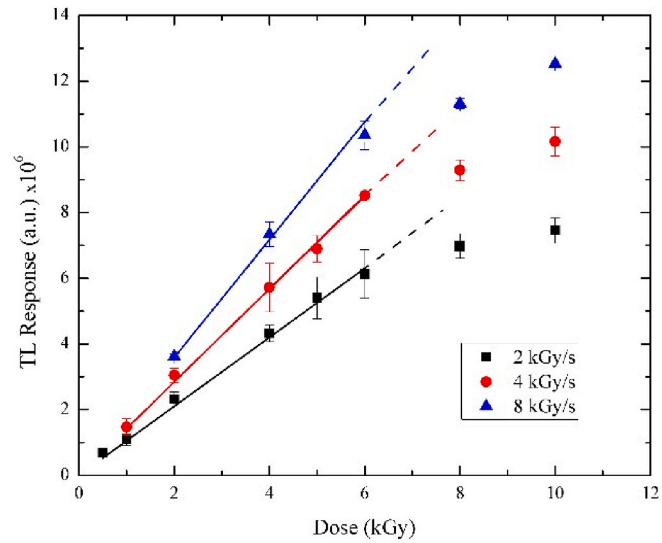

Fig. 5. TL dose-response of CaF_2:Tm irradiated up to 10 kGy at dose rates between 2 and 8 kGy/s. The operational dose rate sets the minimum achievable dose. The dashed lines indicate the linear behavior.

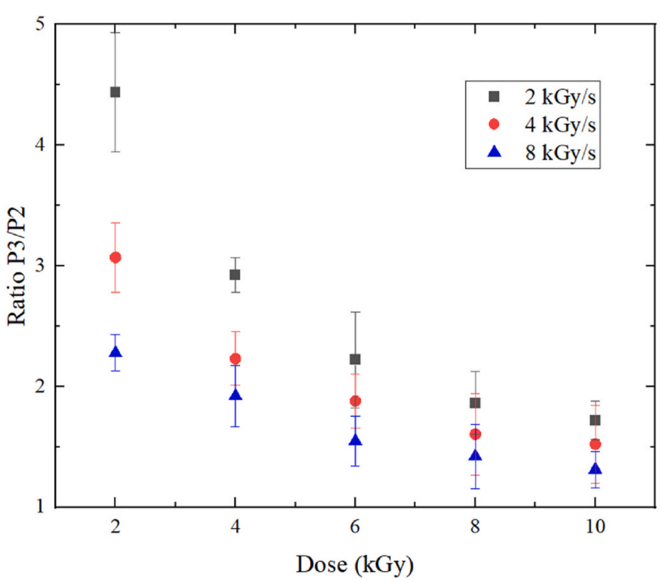

Fig. 4. Peak area ratio (P_3/P_2) as a function of the dose up to 10 kGy and dose rates between 2 and 8 kGy/s.

the average of two readings with the corresponding standard deviation. In general, the glow curves display similar patterns with two peaks, at almost 150 °C and 200 °C, whose relative intensity changes with increasing accumulated doses.

The glow curves were decomposed into individual TL peaks using the GlowFit software version 1.1 (Puchalska and Bilski, 2006), with first-order kinetics. The best-fitting was achieved with four peaks at 150, 200, 240, and 300 °C, with corresponding activation energies of 1.05, 1.14, 1.22, and 0.5 eV. With this procedure, the number of independent variables was reduced from 12 to 4, therefore obtaining a more physically meaningful result. The Figure of Merit (FOM) used as a measure of the “goodness-of-fit” was calculated by the GlowFit software and was found to be between 2.9% and 3.9%.

Fig. 2a–f shows the glow curves of pellets irradiated to 2 and 10 kGy with a dose-rate range of 2–8 kGy/s. These TL curves and the peaks labeled as 2, 3, 4, and 5 do not match those assessed with the commercial CaF_2:Tm (TLD-300) available in the literature. It is likely to occur due to the differences between the basic chemical purities, dopant concentrations, and preparation methods.

In all glow curves, where the peaks are labeled to help the reader, the intensity of the low-temperature peaks (2 and 3) grows with increasing dose while those of high temperatures remain saturated (4 and 5). It is quantitatively exhibited in Fig. 3 that shows each peak intensity versus dose over a range of 10 kGy at 4 kGy/s. Identical behavior is observed at dose rates of 2 and 8 kGy/s.

The relative contribution of peaks 2 and 3 to the glow curves was also investigated by plotting the peak area ratio (P_3/P_2) as a function of the dose at different dose rates (Fig. 4). For higher doses and dose-rates, the results reveal a decrease in the sensitivity of peak 3 while the one corresponding to peak 2 increases. Consequently, the ratio (P_3/P_2) decreases with increasing doses and dose-rates. The remarkable dose-rate dependence of the glow curves is also evidenced in Fig. 4.

It is important to note that the increase in the intensity of peak 2 with higher doses was not observed in the TL glow curve of CaF_2:Tm, produced by wet chemical routes, irradiated to 10 kGy with ^{60}Co gamma rays (Vasconcelos et al., 2014). Based on this result, it appears that the visible changes in the intensity of peaks 2 and 3 might be associated with changes in the traps induced in the CaF_2:Tm when subjected to high electron fluence rates (or dose rates) and accumulated doses herein achieved.

In Fig. 5, the TL intensities (total area under the glow peaks) of CaF_2:Tm, irradiated with dose rates between 2 and 8 kGy/s, are plotted as a function of the dose within the range of 0.5–10 kGy. Regardless the dose

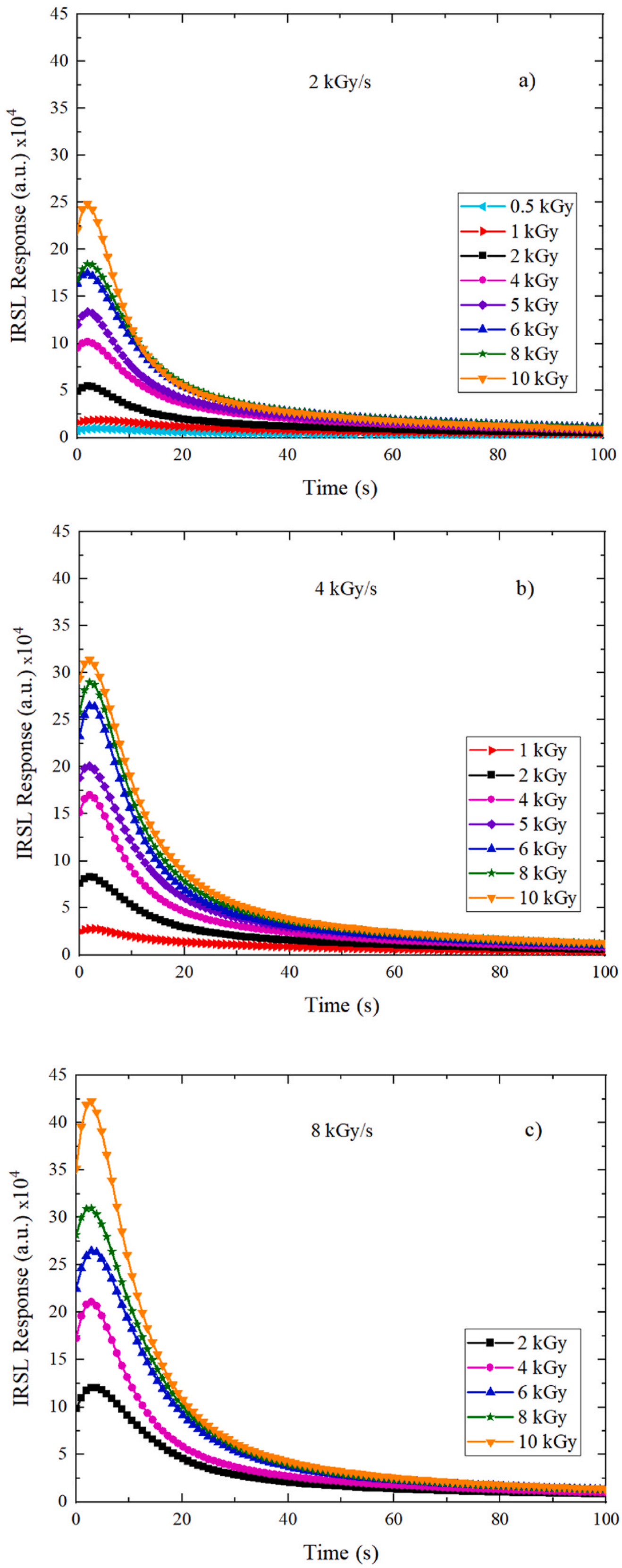

Fig. 6. CW-IRSL curves of CaF_2:Tm irradiated at different doses at dose rates of a) 2 kGy/s, b) 4 kGy/s, c) 8 kGy/s. The IRSL readings were performed with thermal assistance at 150 °C.

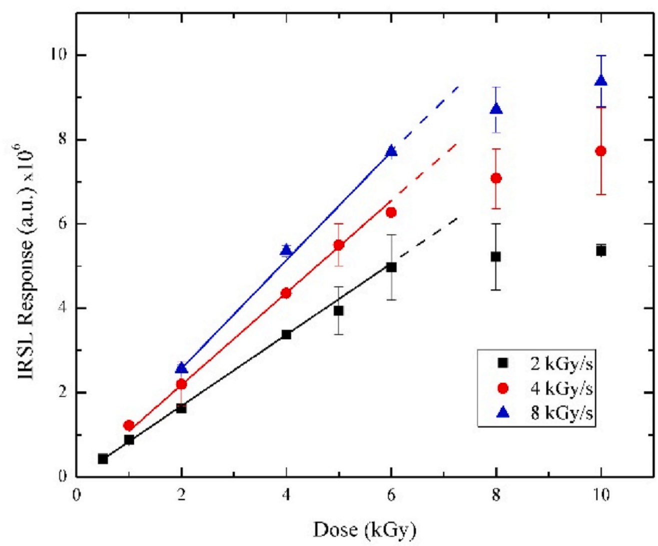

Fig. 7. IRSL response of CaF_2:Tm irradiated up to 10 kGy at dose rates between 2 and 8 kGy/s. The operational dose rate sets the minimum achievable dose. The dashed lines indicate the linear behavior.

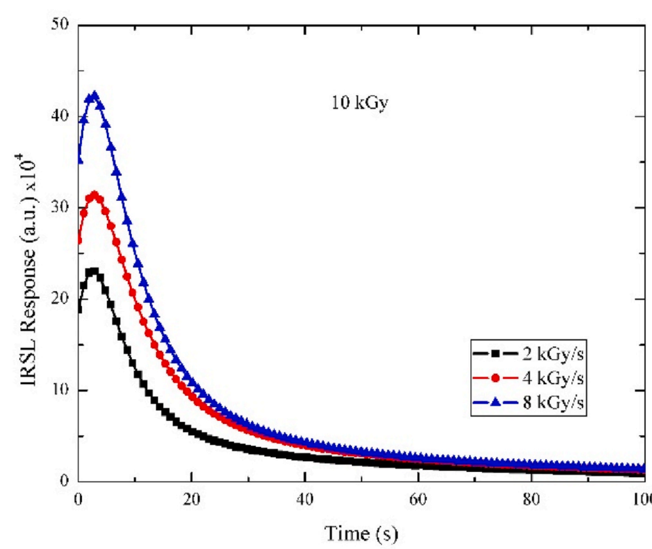

Fig. 8. CW-IRSL curves of CaF_2:Tm irradiated to 10 kGy at different dose rates covering the range from 2 kGy/s up to 8 kGy/s.

rate, the TL responses are linear for doses up to 6 kGy and, subsequently, are sub-linear with a saturation trend around 10 kGy. Nevertheless, the TL response is dependent on the dose rate, being more sensitive with an increasing dose rate. Numerically, the TL sensitivity (slope of the linear region of the TL response) at 8 kGy/s is about 25% and 65% higher than those reached at 4 kG/s and 2 kGy/s, respectively.

Consequently, the TL response is dose rate-specific, which is not a great disadvantage in the radiation processing field where, except for calorimeters (primary standard) and alanine (reference standard), many routine dosimeters (radiochromic films, cellulose triacetate, electronic devices) are dose-rate dependent and strongly influenced by environmental factors (temperature, humidity, light, etc.). For this reason, the calibration of these dosimeters must be performed at a specific facility

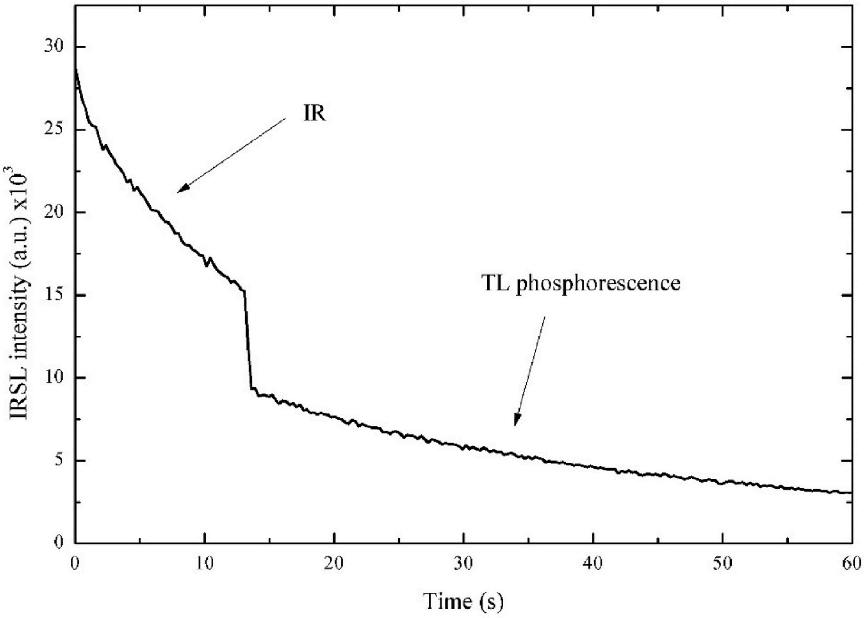

Fig. 9. Influence of the phosphorescent TL signal on the thermally-assisted IRSL readings (LEDs on). Without optical stimulation (LEDs off), the signal amplitude decreases and is due to the TL emission. The CaF_2:Tm pellet was irradiated to 1 Gy and hold at 150 °C.

under the same conditions as those of eventual use, as post-calibration corrections are often unfeasible (ICRU Report 80, 2008).

3.2. IRSL response

Fig. 6 shows the CW-IRSL curves of CaF_2:Tm pellets irradiated to different doses up to 10 kGy at 2 kGy/s (a), 4 kGy/s (b) and 8 kGy/s (c). All of them show similar patterns with an initial signal increase, followed by an exponential decay. From the available literature (McKeever et al., 1997; McKeever, 2001; Denis et al., 2011; Yukihara et al., 2014) the origin of these peak-shaped CW-IRSL curves, instead of the normal monotonic decays, might be due to the charge capture competition between empty shallow traps and recombination centers. In our results, the peak profile becomes more prominent with increasing doses, which appears to show the presence of shallow traps.

The integration of all IRSL signals within 100 s is presented in Fig. 7 as a function of the dose achieved at different dose rates. In general, the IRSL response grows linearly with increasing doses up to approximately 6 kGy, regardless of the dose rate. From this dose, it becomes sub-linear, reaching saturation at almost 10 kGy. Similarly to that found for the TL response, the dose rate dependence of the IRSL signals characterized by the growth of their optical intensity with increasing dose rates is clear, even at a constant dose.

Experimentally, this dose rate dependence is easily confirmed by the IRSL signals, corresponding to a dose of 10 kGy accumulated at 2, 4, and 8 kGy/s, depicted in Fig. 8. However, theoretically, this behavior cannot be explained without knowing the underlying physical mechanisms responsible for the dose rate dependence of IRSL/TL response of CaF_2:Tm. Further studies are needed to explain these results.

Another issue herein addressed refers to the contribution of the phosphorescent TL signal to the thermally-assisted IRSL readings. The TL glow curves (Figs. 1 and 2) show that holding the sample at 150 °C is likely to produce a non-negligible phosphorescence. This hypothesis was checked by measuring the photomultiplier output signal with and without optical stimulation, switching on/off the LEDs. In Fig. 9, the results obtained with a pellet irradiated to 1 Gy evidence that the phosphorescent TL emission is superimposed on the infra-red stimulation.

After almost 15 s, with the LED turned off, the signal amplitude decreases significantly and is exclusively due to the TL emission. The contribution of the phosphorescent TL signal is roughly calculated as about 50% of the total signal.

These preliminary results point to the need to investigate the temperature and other experimental parameters that affect the thermally-assisted IRSL response of CaF_2:Tm. Work in this direction is underway.

3.3. Residual TL

The residual TL readout was carried out shortly after the CaF_2:Tm pellets stimulation at 850 nm, the optical power of 240 mW/cm^2, and 240 s. The residual TL glow curves of all pellets irradiated to different doses and dose rates are assessed under the same previous TL readings conditions. In all of them, peak 2 is decreased by direct thermoluminescence due to the thermal assistance (150 °C) of the optical signal readout, which prevents the assessment of any correlation between the traps of this peak and the IRSL response. Fig. 10 shows the residual TL glow curves of pellets irradiated to 10 kGy at dose rates of 2 kGy/s (a), 4 kGy/s (b), and 8 kGy/s (c) plotted together with the corresponding TL glow curves for comparative purposes. It is noticeable that the residual TL of the dosimetric peak 3, is almost the same (10%), with an agreement of 2% at different dose rates at any dose rate. Also, the high-temperature peaks (4 and 5) are hard to bleach and, therefore, their

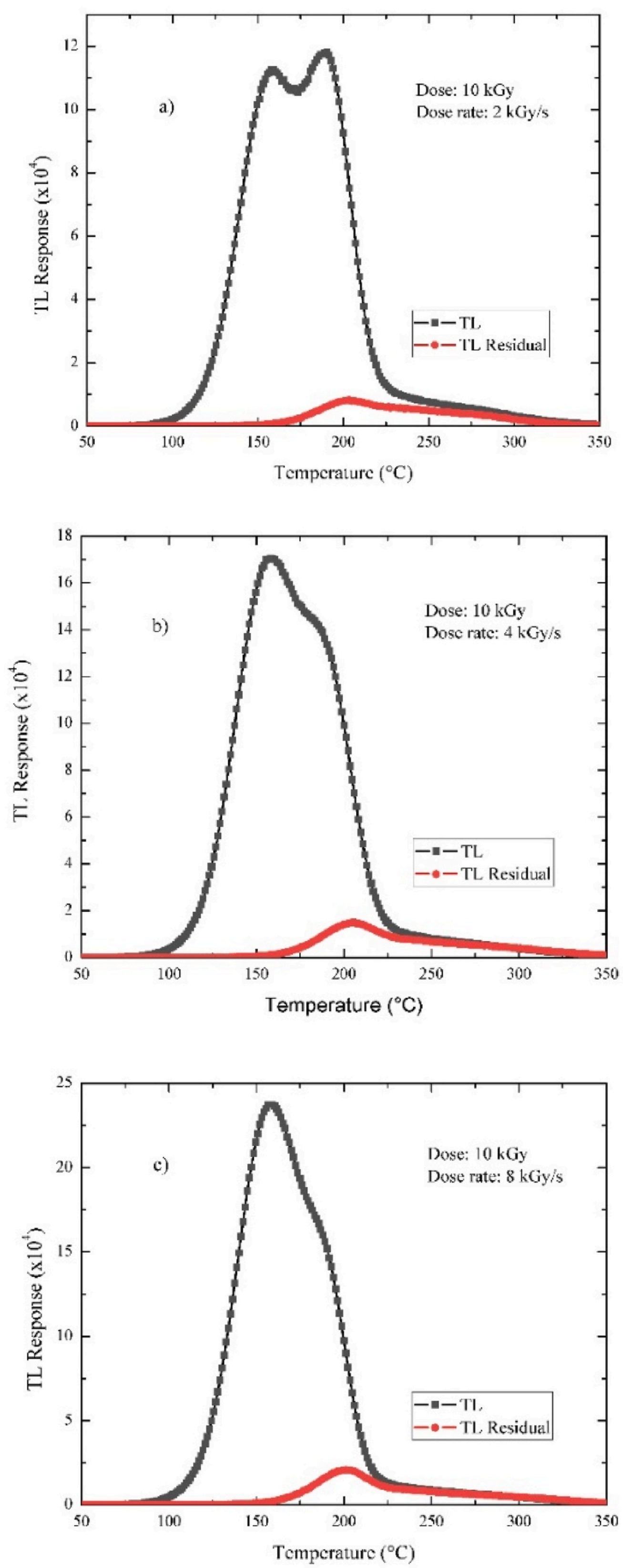

Fig. 10. Residual TL glow curves of pellets irradiated to 10 kGy at different dose rates: a) 2 kGy/s, b) 4 kGy/s, c) 8 kGy/s. The corresponding TL glow curves are plotted together for comparison.

contribution to the TL glow curve remains constant in the Residual TL.

4. Conclusions

The TL and IRSL responses of in-house produced CaF_2:Tm dosimeters were investigated in this work, envisaging their electron beam (EB) radiation processing application. The irradiations have been performed at an industrial EB accelerator (1.5 MeV) covering a dose rate range of 2–8 kGy/s and dose up to 10 kGy. In general, the TL glow curves display four peaks, termed as peaks 2, 3, 4, and 5, corresponding to temperatures at ~150, 200, 240, and 300 °C, respectively. The intensity of the low-temperature peaks (2 and 3) grows with a dose, while the others remain constant (saturated). However, an evident dose effect on the glow curves manifests in decreased peak3/peak2 ratio with increasing doses.

The CW-IRSL curves exhibited similar patterns with an initial signal increase, followed by an exponential decay. Instead of the normal monotonic decays, these peak-shaped curves might be due to the charge capture competition between empty shallow traps and recombination centers. In our results, the peak profile becomes more prominent with increasing doses, which appears to show the presence of shallow traps.

Regarding the TL and IRSL intensities, both increase linearly with doses up to 6 kGy, and for higher doses, they become sub-linear with a saturation trend around 10 kGy. Another common feature of TL/IRSL response is its remarkable dose rate dependence, being more sensitive with higher dose rates. Despite being dose-rate dependent, the CaF_2:Tm dosimeters might be suitable for EB processing dosimetry. However, for their use as routine dosimeters, relevant dosimetric characteristics, such as fading and response reproducibility, have to be investigated. Work in this direction is underway.

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.

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