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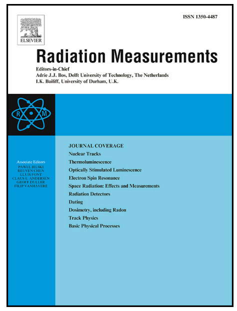

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Phototransferred TL properties of alumina substrates

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Abstract

Alumina substrates, such as those found as surface-mount resistors in mobile phones, are currently the strongest candidate as a surrogate dosimeter material in emergency radiological scenarios using luminescence techniques. However, the rate of fading of the luminescence signal (TL or OSL) imposes a limitation on their longer term use, and also increases the uncertainty in dose assessment. The potential of phototransferred thermoluminescence (PTTL) techniques to access deep traps in alumina substrate samples is reported here. A measurement procedure employing blue (470 nm) illumination was found to produce a PTTL signal with a detection limit of ca 100 mGy, but with a supralinear dose response below 10 Gy. By using a UV source with emission between 307 - 575 nm a linear dose response was obtained within this dose range, although the detection limit was higher (ca 200 mGy), partly arising from the presence of a non-radiation-induced photostimulated TL signal. Pulse annealing experiments indicate that deep traps providing a reservoir of charge are thermally accessible above 500 °C and require annealing to ca 700 °C to thermally clean them. Significantly, using blue illumination, storage experiments performed under dark conditions at room temperature indicate that the loss of charge in the deep traps accessed by the PTTL measurement procedure was less than 30% for storage periods of up to 224 days. Although the physical mechanisms associated with the transfer of charge from the deep traps probed by the PTTL measurements require further clarification, the possibility of significantly reducing the fading observed in conventional TL or OSL measurements introduces a potentially valuable tool in the use of this material for both short and long term dosimetry.

Keywords: Alumina substrate, Phototransferred thermoluminescence, Emergency dosimetry

1. Introduction

The measurement of phototransferred thermoluminescence (PTTL) is a well-established procedure used as a means to access deep traps in luminescent minerals and phosphors. This is achieved by moving a proportion of charge stored in deep traps to previously thermally cleaned shallower traps, typically by using short wavelength optical stimulation. Prompt measurement of the resultant PTTL glow curve enables an indirect measurement of the population of charges in the deep traps. PTTL procedures have been developed for variety of materials, including lithium fluoride (Kharita et al. 1994; Charles 1983), quartz (Bailiff et al., 1977) and aluminium oxide (Akselrod and Gorelova 1993; Colyott et al. 1996; Bulur and Göksu 1999; Polymeris and Kitis 2012; Nyirenda et al. 2016; Chithambo et al. 2017). The primary advantage of using this procedure is to avoid the effects of interfering black-body radiation and thermal quenching where the thermoluminescence (TL) peaks associated

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with the deep traps are measured directly (i.e., ≥ 500 °C). Also, the PTTL may potentially provide a means of circumventing the effects of anomalous fading (Bailiff 1976; McKeever et al. 2017).

Alumina that forms the substrate of surface-mount resistors, which is structurally the polycrystalline analogue of aluminium oxide, has been widely studied as a dosimetry material for use in emergency scenarios (Inrig et al. 2008; Bassinet et al. 2014). Its TL properties are favourable for dosimetry, with a strong TL signal, linearity of dose response and a detection limit of ∼ 10 mGy (Woda et al. 2009; Mesterhzy et al. 2012). However, fading in regions of the TL glow curve where loss at room temperature (RT) is expected to be negligible (300-450 °C, ca 23% loss in 20 days) is a known problem that has been attributed to athermal loss of charge. Although the luminescent characteristics of alumina substrate have been examined in depth (Kouroukla 2015; Ademola and Woda 2017; Bassinet et al. 2010), an investigation of its PTTL characteristics does not appear to have been reported, although observation of an intrinsic photostimulated (PSTL) response stimulated by the UV illumination of alumina resistor substrates that is not related to absorbed dose was recently reported by Ademola and Woda (2017). In this study we applied a PTTL measurement procedure to investigate the presence of deep traps in polycrystalline alumina substrates and to assess whether PTTL provides an advantage in terms of application to longer-term dosimetry.

2. Experimental

All OSL and TL measurements were performed with a Risø model 12 reader (DTU Nutech, Denmark) that incorporates a Sr^{90}/Y^{90} β source irradiator delivering an estimated dose rate to alumina substrates of 0.74 Gy·min^{-1}. A UV (250-350 nm; Schott U-340 filter) and a broad band (360 - 580 nm; Schott BG-39 filter) detection windows were used when measuring blue (470 nm) stimulated OSL and TL. Two illumination sources were employed to transfer charge from deep traps for the PTTL measurements, comprising either the 470 nm LEDs in the Risø reader, delivering a power of 14 mW.cm^{-2} at the sample position, or UV radiation from an unfiltered medium - pressure mercury lamp. The mercury lamp (Hanovia Ltd) delivered a power of ∼ 4 mW·cm^{-2} at the sample position and its emission spectrum contained lines at 307, 364, 403, 434, 544 and 575 nm. The abbreviated terms PTTL[UV] and PTTL[470 nm] are used to distinguish the type of illumination source used to obtain the PTTL. For each set of measurements, five alumina substrate surface-mount resistors of type 1206 (RS pro, ∼ 5.2 mm^2), were placed in a stainless steel cup with the alumina substrate layer facing up, and each test was repeated at least twice to test for reproducibility. The TL peak temperatures were calculated by applying a deconvolution procedure to the measured glow curve data (using Origin 2017) and hence the values obtained reflect the precision of the fitting procedure only. Independent thermocouple measurements of the heater plate temperature indicated that the temperatures displayed by the Viewer software (DTU Nutech) are within 6 °C of the measured value and that, when taking into account thermal lag introduced by the samples and the stainless steel pans, the overall uncertainty in the average resistor temperature is likely to be the order of ± 8 °C. The PTTL (470 nm) measurement procedure (A) followed is summarised in Table 1.

3. PTTL characteristics

3.1. Detection window

Measurements performed using the BG-39 detection filter yielded a PTTL[470 nm] signal about three times stronger (Fig. 1) than with a U-340 filter, indicating that the PTTL emission spectrum is not confined to

Table 1: PTTL measurement procedure A

Step	Measurement
1	TL to T_{stop} 450 °C (procedure A) or 500 °C (procedure B); 5 °C.s^{-1}
2	Repeat step 1 (background); subtraction of the background from the TL glow curve (step 1)
3	Phototransfer illumination, 470 nm LEDs, 120 s at sample temp. of 150 °C; concurrent measurement of OSL
4	PTTL to T_{stop} (500 °C); 0.5 °C.s^{-1}
5	Repeat step 4 (background); subtraction of the background from the PTTL glow curve (step 4)
6	Anneal to 700 °C in a furnace, in air, 20 min

the UV region. This is consistent with radioluminescence (RL) spectra measured with alumina substrates by Kouroukla (2015, p. 132) and Lee et al. (2017) which indicated complex emission that included UV, blue and red bands. It was also observed that the intensity of the shorter wavelength emissions was thermally quenched, giving rise to predominantly red emission at higher temperatures (Kouroukla, 2015, p. 132). However, recording the red TL emission above 400 °C using the BG-39 gave rise to a strong thermal background signal, making the detection of weak signals associated with low dose difficult to resolve above the thermal background signal.

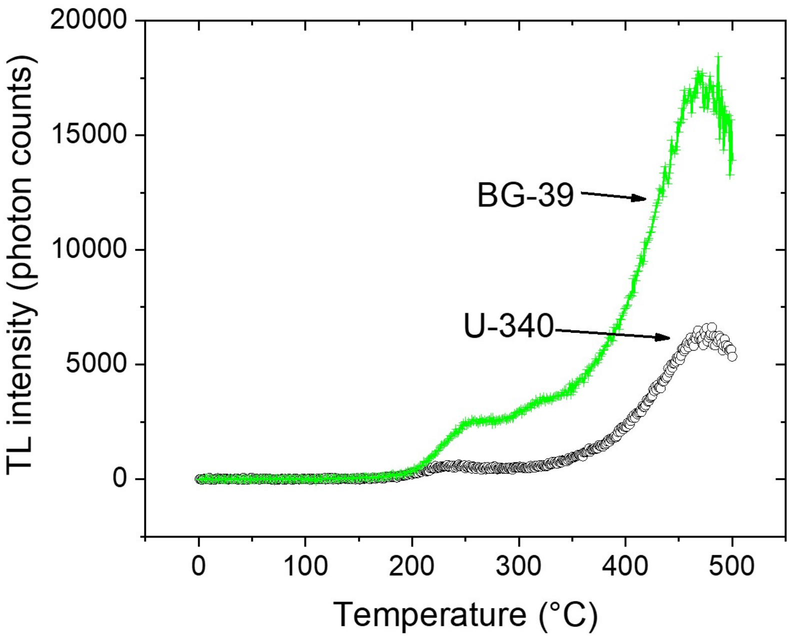

Fig. 1. PTTL[470 nm] glow curves measured with alumina substrate chips following β irradiation (10 Gy). Detection window: U-340 (black curve) and BG-39 (green curve); heating rate 0.5 °C·s^{-1}. The thermal background signal was subtracted.

3.2. Variation of PTTL with sample temperature

The PTTL (470 nm) glow curve was recorded following a 2 min 470 nm exposure (concurrently measuring the OSL) at a sample temperature selected in the range 50-400 °C (Fig. 2). The shape of the PTTL glow curve changes with the sample temperature during illumination and, as the temperature increases, a more efficient transfer of charge to shallower traps gives rise to relatively stronger lower temperature PTTL peaks. Above 200 °C the transfer process competes with thermal bleaching of the PTTL traps during illumination, leading to a progressive decrease of the PTTL signal. A similar measurement procedure was applied by varying the illumination time (30-300 s) for a fixed sample temperature. A plot of the integral of the PTTL glow curve (150-500 °C) vs both sample temperature and duration of illumination, shown in the form of a contour plot in Fig. 3, indicates that the maximum PTTL intensity was obtained for an illumination of 2 mins with a sample temperature of 150 °C, and these measurement conditions were adopted using procedure A (Table 1).

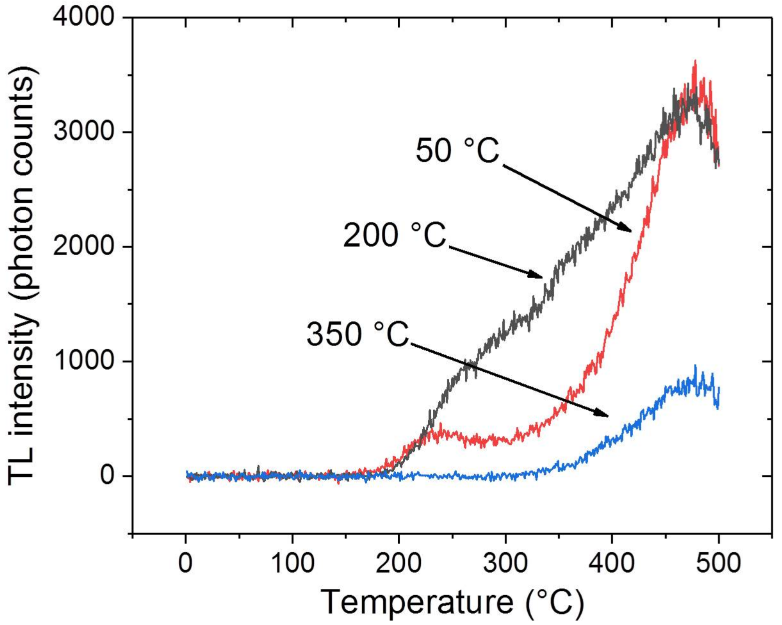

Fig. 2. Phototransferred TL glow curves of alumina substrate measured following a β irradiation (10 Gy), a 2 mins 470 nm illumination at a sample temperature in the range 50-350 °C, three examples of which (50, 200 and 350 °C) are shown. Detection filter: U-340; heating rate 0.5 °C.s^{-1}.

3.3. Dose response

The PTTL (470 nm) glow curve contains several overlapping peaks (Fig. 4a), positioned at 190, 240, 335 and 465 °C. The presence of a native signal was tested by measuring a fresh set of unirradiated resistors, but found to be negligible using 470 nm illumination (Fig. 4a). Fig. 4b shows the growth of the PTTL peaks (integrated TL, 150 -500 °C) with absorbed β dose, measured with the same aliquot, which exhibits a supralinear dose dependence (Fig. 4b) that was also observed for each peak analysed individually. Two preheat temperatures were tested : 450 °C (procedure A) and 500 °C (procedure B), using a heating rate of 0.5 °C.s^{-1}. An additional TL background measured after step 2 in procedure B indicated that some residual TL remained after this step (see Supplementary Material) above 300 °C (Procedure A) or 350 °C (Procedure B). The integration region of the PTTL glow curve was between 150-300 °C to avoid this residual TL. The glow curves measured using procedures A and B contained similar peaks but the signal was weaker using procedure B, although the dose response appeared to be more linear. Interestingly, similar analysis performed using an integration range of 300-500 °C produced similar outcomes.

The reproducibility of the PTTL signal, tested by repeating procedure A five times for a given dose, exhibited similar values of integrated photon counts (within 10 %). Using procedure A, and a quadratic function fitted to the dose response data, the detection limit (calculated as the dose for which the PTTL signal is equal to the

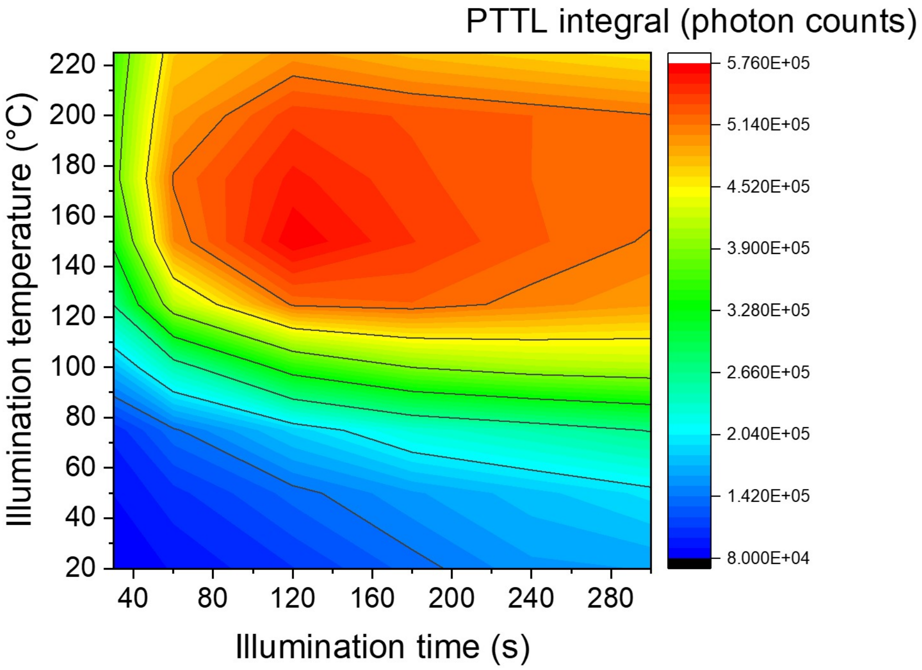

Fig. 3. "Map" of PTTL intensity vs the illumination parameters (duration and temperature). The PTTL intensity corresponds to the integral of the signal recorded between 150 and 500 °C.

background signal plus three times its standard deviation) was estimated to be ∼ 100 mGy. The final annealing step (6) was applied to minimise sensitization effects by thermally cleaning the deep electron traps. If this step is omitted, a non-linear increase in sensitivity is observed over repeated cycles of measurements. During 470 nm illumination at elevated sample temperatures (Step 3), the OSL signal was also recorded (Fig. 5) and its slow evolution and subsequent decay reflect a thermally-assisted process (TA-OSL), similar to that observed with Al_2O_3:C (Polymeris et al., 2010), together a supralinear dependence with dose (not shown).

4. Kinetic parameters of the reservoir traps

A pulse-annealing stage was added to procedure A to examine the thermal stability of the traps that provide a reservoir of charge for the PTTL signal. The samples were heated to a temperature selected in the range 450 to 600 °C before the 470 nm illumination in Step 3 and subsequent recording of the PTTL to 500 °C. During the pulse-annealing sequence, the 480 °C PTTL peak exhibited the strongest reduction (Fig. 6a and 6b) in intensity following a preheat to 600 °C, indicating that charges in deep traps accessed by 470 nm illumination are thermally emptied by annealing in the region 450-600 °C. Similar results were obtained testing a second set of chips. Attempts to determine the thermal activation energies of the traps acting as the reservoir of charge were unsuccessful. An assessment of the Arrhenius plots obtained with data produced from a pulse annealing PTTL (470 nm) measurement procedure indicated that the nature of these traps is complex, likely to comprise a continuum of traps associated with TL peaks between 500 and 600 °C.

5. UV illumination

As the supralinear dose response of the PTTL[470 nm] is a potential disadvantage for absorbed dose determinations below ca 0.5 Gy, optimisation of the PTTL response was explored using shorter wavelength illumination.

In contrast to the PTTL[470 nm] results, fresh unirradiated samples, when illuminated (2 mins) with UV produced a "native" glow curve containing two peaks (Fig. 7a). The native signal measured after UV illumination is interpreted as a non-radiation-induced UV-stimulated signal, which is observed, for example, with Al_2O_3:Si,Ti, and applied in UV dosimetry (referred to as photo-stimulated TL, PSTL; Mehta and Sengupta 1977, 1978). A PSTL contribution, if present, needs to be accounted for when applying PTTL to perform dosimetry measurements with a UV light source. The PTTL (UV) glow curve recorded following a β dose of 10 Gy and a 2 min illumination at RT, contains three main peaks (Fig. 7a) at ca 190, 315 and 460 °C, and the PTTL (470 nm) glow curve is shown for comparison. Under the particular illumination conditions used in these experiments, UV transfers significantly more charge into the PTTL traps compared with 470 nm illumination (the integrated PTTL is $\sim$ 4 times greater for a 10 Gy β dose). By annealing the samples at 900 °C for 30 mins and repeating the PTTL measurement procedure, the glow curve measured was similar in shape and intensity to the "native" PSTL signal. The latter indicates that this annealing procedure effectively thermally cleaned the deep traps associated with the PTTL signal. The dose response curve obtained with UV illumination is linear (Fig. 7b), and the estimated detection limit of 200 mGy is higher than that obtained with 470 nm illumination.

6. Fading

Fading tests were performed by irradiating samples with a β dose of 7.4 Gy, storage in the dark at ambient room temperature for periods ranging from 6 h to 224 days and measuring the PTTL (470 nm) following procedures A and B and using a integration interval of 150-300 °C. The resistors used in the fading experiments were obtained from the same manufacturer (RS Pro, UK), but they were obtained from two reels, referred to as batches 1 and 2. Measurements using four sets of 5 resistors from Batch 1 were initially conducted with single aliquots for storage periods of 0.125, 2 and ca 100 days, and the tests were repeated using aliquots that contained resistors from both batches (1 and 2), for storage periods up to 2 days and for various storage periods between 2 and 244 days. The results obtained from the fading tests shown in Fig. 8 include: a) the integrated PTTL (150-300 °C) recorded using procedures A (open diamonds) and B (open triangles) b) the integrated TL recorded in two temperature regions (filled circles, 150-200 °C and filled squares, 200-300 °C).

It can be seen that using the Batch 1 resistors, the extent of long-term fading over 100 days is less than ca 15%, using either of procedures A and B. Analysing the results using an integration range of 300-500 °C in the PTTL glow curve produced a similar outcome, and suggests that the inclusion of remnant TL in the higher temperature region of the PTTL glow curve does not appear to significantly affect the rate of fading. Although the repeated tests performed with resistors from the combined batches show similar behaviour for storage periods up to 2 days, a greater degree of fading for longer periods of storage is evident in these subsequent tests. Assuming that the measurement procedures were applied correctly, these results provide an alert to the possible variability of fading characteristics between batches of resistors, and this requires more detailed investigation. Nevertheless, the loss observed in these tests at ca 200 days remains some 30% less for PTTL than that obtained with conventional TL, as measured with resistors from the same batches (Fig. 8, filled circles and filled squares), and also obtained in previous studies (Ademola and Woda 2017; Kouroukla 2015).

The fading behaviour of the TA-OSL signal was also found to be similar to that of the PTTL. In her study of the long-term fading of OSL of alumina substrate, Geber-Bergstrand (2017) also found the existence of a long-lived OSL component of the decay curve, where the average period after which the signal halved was 790

± 210 days; she estimated that a 0.5 Gy dose could be measured after a period of 2 years, after applying a fading correction.

7. Discussion

The results of the pulse annealing and phototransfer experiments confirm the presence of deep traps that provide a reservoir of charge probed by the PTTL measurement procedure. If we examine for parallels between the PTTL behaviour of alumina substrates and crystalline Al_2O_3, amongst the earlier work on the latter, Akselrod and Gorelova (1993) proposed three types of traps that, in addition to type I traps producing the main dosimetry TL peak at 190 °C, included deep type II and type III traps associated with TL peaks at 550 °C and ca 900 °C respectively. Colyott et al. (1997) studied three PTTL peaks located at -8, 37 and 177 °C measured following preheat treatments ranging from RT to 900 °C, and they found the PTTL to be most efficiently produced when induced by 300 nm illumination. Later work has linked the PTTL with one or more traps associated with TL peaks above 500 °C (Bulur and Göksu, 1999) and above 600 °C (Chithambo et al., 2017). Although the nature of the traps providing the reservoir of charge for PTTL in Al_2O_3 has not been identified, these studies point to the type II and III traps proposed by Akselrod and Gorelova as providing the most likely reservoirs of charge. The results obtained with our alumina substrate samples using 470 nm illumination indicate that the reservoir of charge may originate mainly from traps of similar depth to the type II traps, and under UV illumination ($\geq$ 300 nm) they are likely to be associated with both type II and type III traps, given that annealing at ca 900 °C was required to erase the PTTL (UV) signal. As found with alumina substrate, sensitization effects were reported in monocrystalline Al_2O_3 by Yukihara et al. (2003), and explained by the presence of deeper traps.

In addition to characterisation of the deep traps, the nature of the traps into which the charge is transferred associated with the PTTL is also of interest. Again, seeking parallels with crystalline aluminium oxide, Chithambo et al. (2017) found that the peaks in the PTTL and TL glow curves were similar, suggesting that similar traps participate in both modes of measurement. We have made broadly similar observations with the alumina substrate tested, although the distribution of charge in the traps participating in the PTTL and TL processes differ (Fig. 9), with the lower temperature peak more prominent in the glow curve. In the higher temperature region, the 314 and 480 °C TL peaks are reproduced in the PTTL[470 nm] and PTTL[UV] glow curves and the 260 °C TL peak is also present in the PTTL[470 nm] glow curve. However, the 197 °C peak observed in the PSTL and the PTTL[470 nm; UV] glow curves differs in position relative to the main TL peak (165 °C).

8. Conclusion

The results obtained indicate that PTTL measurement procedures can be applied to polycrystalline Al_2O_3 substrates for dose determination using blue and shorter wavelength illumination. For the substrates tested, and using blue illumination, the dose response was found to be supralinear, with a detection limit of ca 100 mGy for a resistor surface area of 22.5 mm^2. By employing UV illumination ($\geq$ 300 nm) the dose response obtained was linear, although with a higher detection limit of ca 200 mGy, partly arising from the presence of a photo-stimulated thermoluminescence (PSTL) signal. The trapped charge transferred in the PTTL procedure originates from deep traps and although their depths could not be determined using blue illumination, we conclude that 1) the trapped charge associated with the PTTL signal originates from a range of deep traps that are thermally erased in the region 450-600 °C and 2) UV illumination enables access to charge in deeper traps that are thermally erased by heating to 900 °C. Significantly, we found that in laboratory fading tests in the dark at room temperature the measured loss of PTTL was less than 30 % for storage periods of up to 224 days. This is to be compared with a typical loss of 47 % in 50 days using conventional TL (Ademola and Woda 2017; Kouroukla 2015). The PTTL procedure has sufficient sensitivity for dosimetry measurements following radiological emergencies, although further optimisation of the procedure would be required to address the issue of the trend of the decreasing size of surface mount resistors. Although the physical mechanisms associated with the deep traps in the alumina substrate probed by the PTTL measurements require further clarification, the possibility of reducing the extent of the significant fading observed in conventional TL or OSL measurements introduces a potentially valuable tool in the use of this material for short and long term dosimetry.

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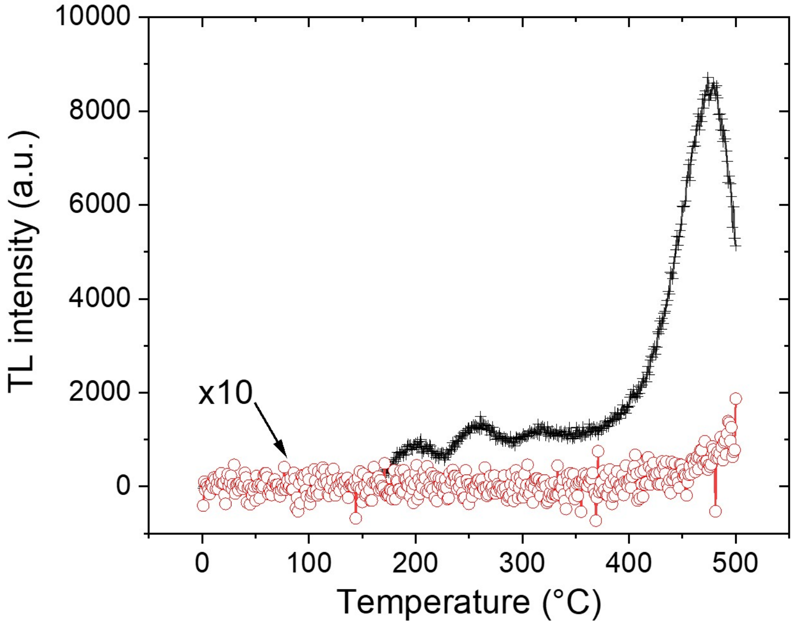

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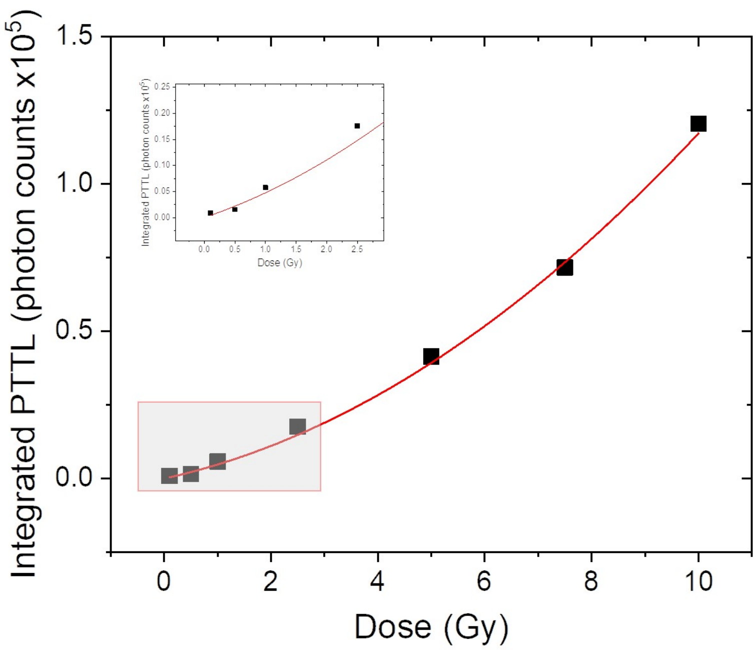


(b)

Fig. 4. a) Native (open circle) and β-induced (10 Gy; black cross) PTTL glow curve of five alumina substrate chips, measured following the PTTL procedure (Table 1). Photon count recorded every second; b) Integral of the PTTL signal (150-300 °C) vs administered β dose, where the solid line represents a quadratic curve fitted to the experimental data. Type A error in counts $\leq$ 1 %. Detection window: U-340; heating rate 0.5 °C.s^{-1}.

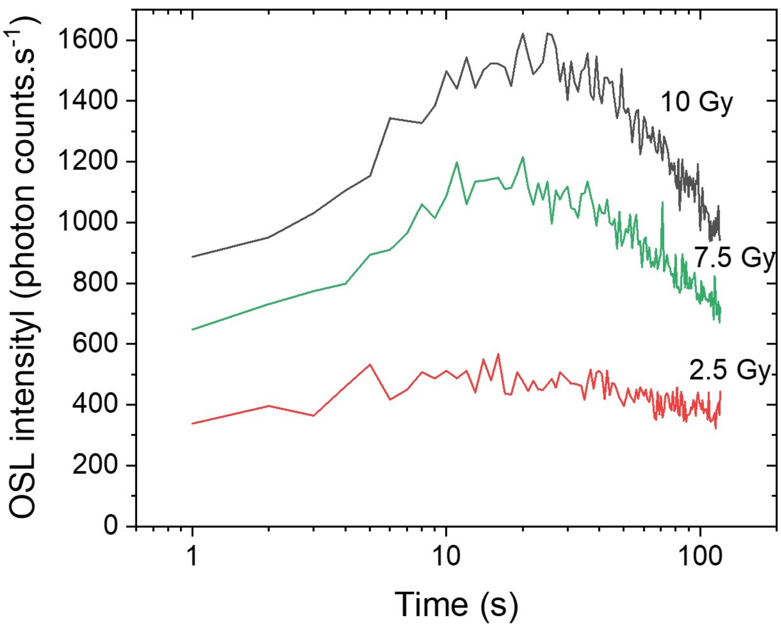
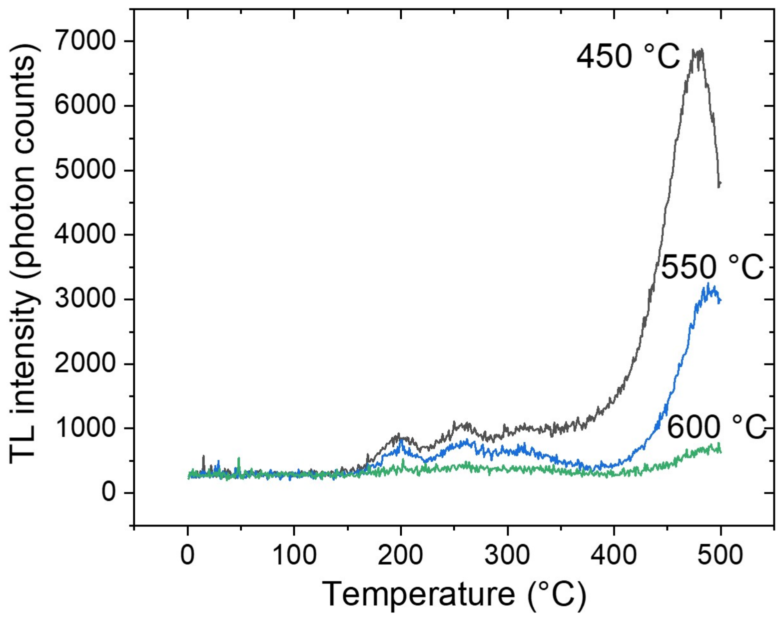


Fig. 5. Blue OSL recorded following β irradiation and 450 °C preheat (Step 3 , Table 1). Detection window : U-340.



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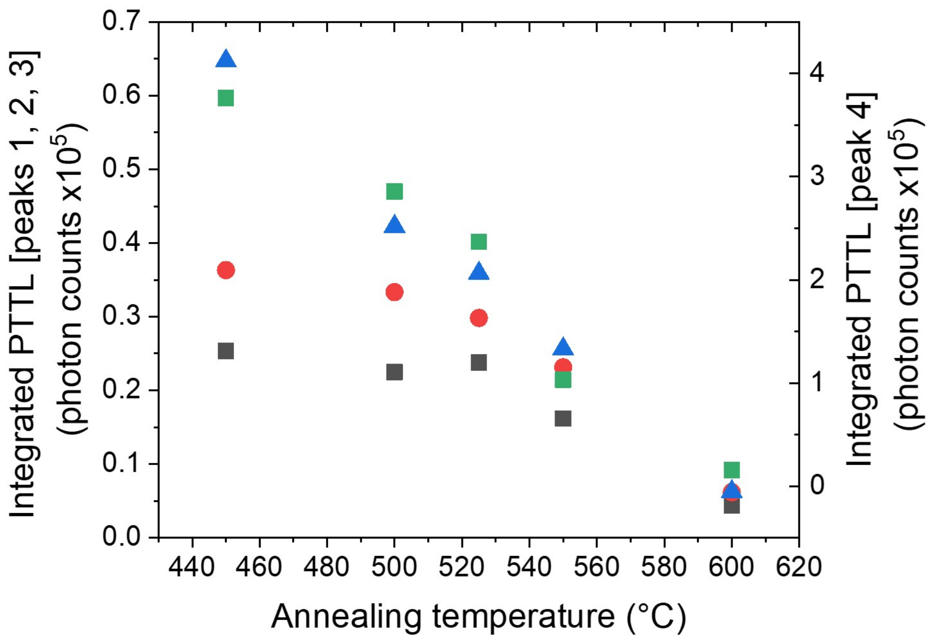
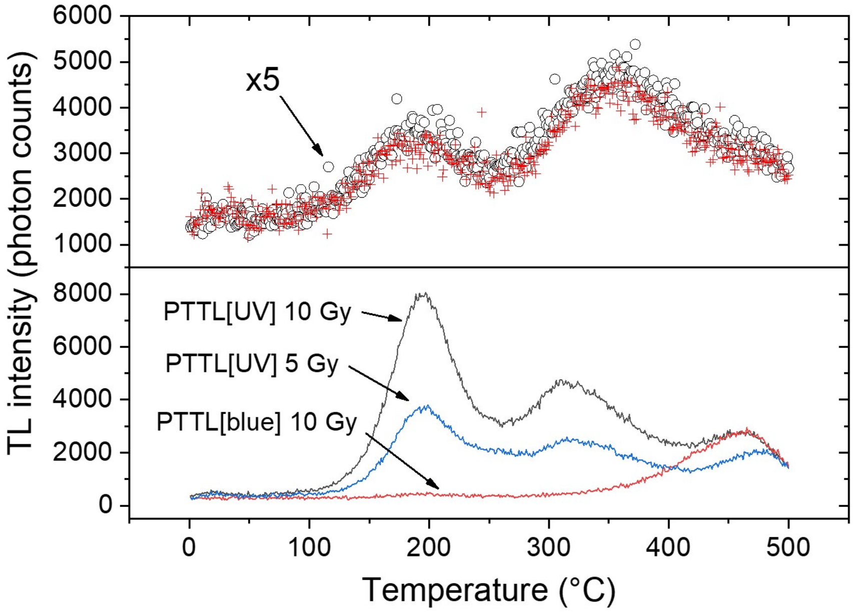


(b)

Fig. 6. PTTL [470 nm] glow curves of alumina substratemeasured following β irradiation (10 Gy) and annealing at the indicated temperatures in the range 450-600 °C . b) PTTL vs annealing temperature: Peak 1 (integrated TL 150-223 °C, black filled squares), Peak 2 (223-284 °C, filled circles), Peak 3 (283-377 °C, blue filled triangles), and peak 4 (377-500 °C, green inverted filled triangles). Detection window: U-340. Heating rate: 0.5 °C·s^{-1}.



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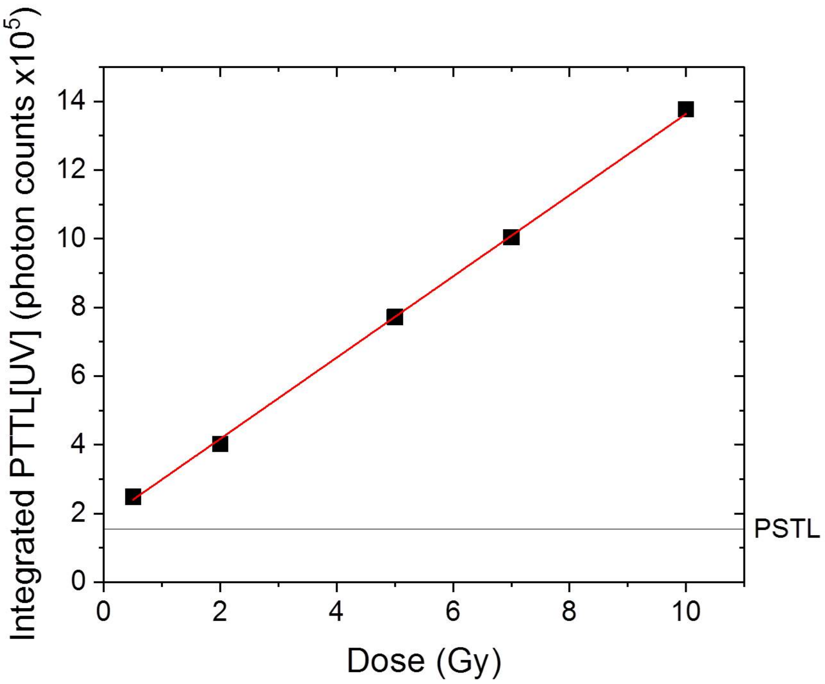

(b)

Fig. 7. a) Upper : native PSTL (black circles) and PSTL measured after a cycle of PTTL[UV] measurements and annealing at 900 °C (red crosses) of alumina substrate following UV illumination 2 minutes at room temperature. Lower : PTTL[UV] following β irradiation 10 Gy and 5 Gy (black and blue lines respectively), and PTTL[470 nm], 10 Gy β dose (red line). All illuminations were carried out at room temperature and the samples were exposed to light for 2 minutes. b) Integral of the PTTL (UV) signal (100-500 °C) vs administered β dose (0.5-10 Gy). The solid line represents a linear curve fitted to the experimental data; the horizontal line indicate the PSTL signal. Type A error in counts $\leq$ 1 %. Detection filter : U-340, heating rate : 0.5 °C.s^{-1}, Risø system.

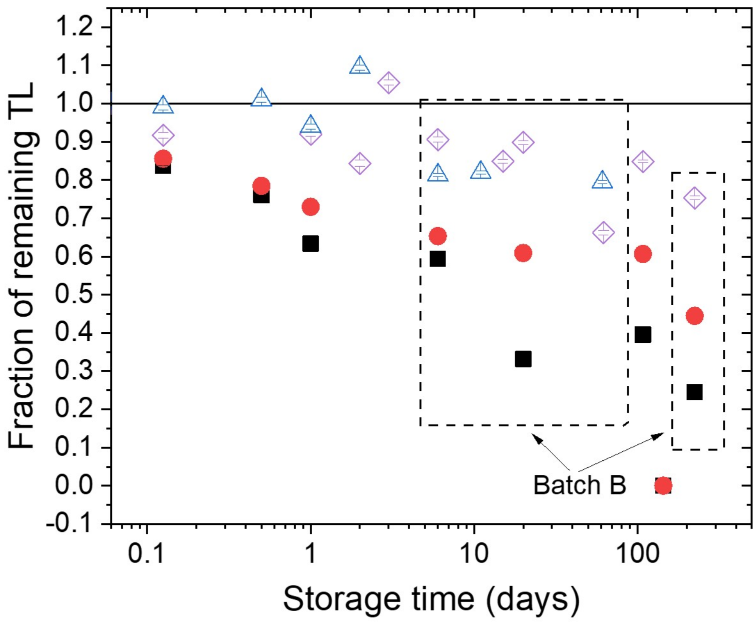

Fig. 8. Remaining PTTL signal (integral, 150-300 °C; purple diamonds: procedure A; blue triangles: procedure B) following storage (0.125- 224 d) in the dark at ambient temperature, compared with the remaining TL signal (integral, 150-200 °C, black squares; integral 200-300 °C, red circles). The horizontal line indicates the absence of fading (y = 1). The box (broken line) groups the results obtained with resistors drawn from combined batches 1 and 2.

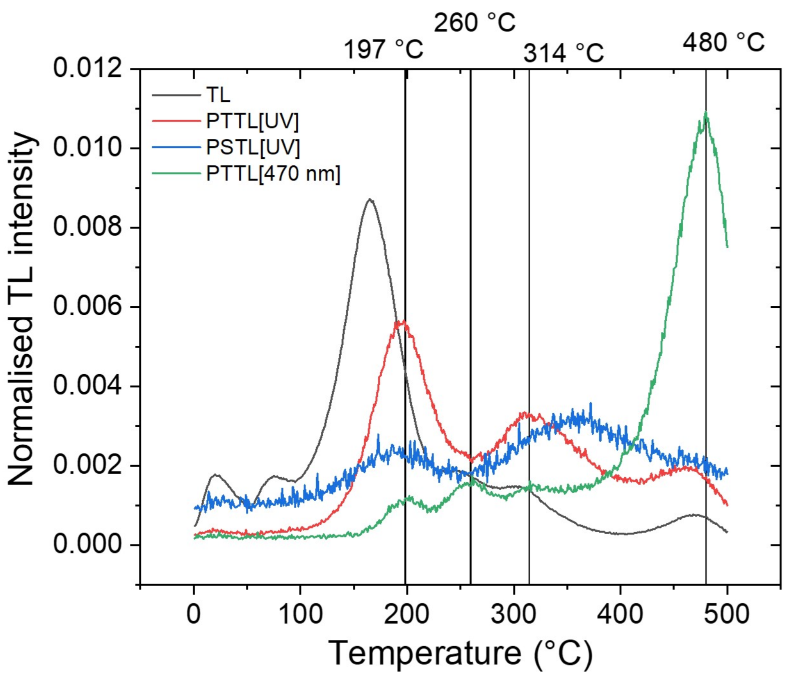

Fig. 9. Alumina substrate glow curves normalised by the number of counts of 1) TL, β dose 10 Gy (black line), 2) PTTL induced by a 2 min UV illumination at room temperature, β dose 10 Gy (red line), 3) photo-stimulate signal induced by a 2 min UV exposure at room temperature (no dose, blue line), and 4) PTTL induced by blue illumination 2 min at 150 °C, β dose 10 Gy (purple line). Some of the peak positions (197, 260, 314, and 480 °C) are indicated by a vertical line. The glow curves were normalised to the total integral of the signal. Risø system, U-340, detection window : U-340, heating rate : 0.5 °C·s^{-1}.

Research Highlights

- Phototransferred TL properties (PTTL) of alumina substrate (surface mount resistors) investigated for emergency dosimetry applications.
- The parameters of the illumination stage (temperature, duration, wavelength) influenced the PTTL emission.
- Supra-linear dose response and detection limit of 100 mGy using blue illumination; linear dose response and detection of 200 mGy if UV is used.
- Reduced rate of fading using PTTL compared to TL.