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A preliminary study of phototransferred thermoluminescence of alexandrite

A.O. Silva [a], N.M. Trindade [a,*], M.L. Chithambo [b]

[a] Instituto de Física, Universidade de São Paulo, São Paulo, SP, Brazil
[b] Department of Physics and Electronics, Rhodes University, Grahamstown, South Africa

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ABSTRACT

This study reports the phototransferred thermoluminescence (PTTL) of alexandrite. PTTL was induced by 470 nm illumination of samples beta irradiated to 150 Gy. The glow curve of its conventional TL shows five peaks, labelled as I–V. PTTL is observed at peaks I-IV. Time-response profiles of PTTL were analyzed as systems of an acceptor and donors with the number of donors determined by the preheating temperature. The attendant effect of thermal assistance and thermal quenching associated with the optically stimulated luminescence emitted during illumination of sample are reported and analyzed.

1. Introduction

Alexandrite is a greenish rare form of chrysoberyl ($BeAl_2O_4$) [1]. It is pleochroic and attains other hues in artificial light. Its greenish color is attributed to chromium impurities within it. The crystal lattice of alexandrite is distorted by oxygen atoms thereby inducing two distinct aluminum sites. One, labelled Al_1 has inversion symmetry whereas the other, Al_2 adopts mirror symmetry. These sites are thought to be occupied by chromium or iron. In particular, the preferential substitution of Cr^{3+} at the Al_2 site due to longer Al–O bond lengths compared to the Al_1 site is known to induce luminescence in alexandrite [2,3]. There have been several stimulated luminescence studies of alexandrite. Trindade et al. [4] investigated the radioluminescence of alexandrite, discussing the emission in relation to the Cr^{3+}, Mn^{4+} and Fe^{3+} cations whose presence in the material were determined using scanning electron microscopy techniques (SEM) and energy-dispersive X-ray spectroscopy. Others studies suggest that the luminescence and optical properties of alexandrite can be attributed to Cr^{3+}-Cr^{3+} pairs and clusters [5–7]. The thermoluminescence of alexandrite including its dosimetric properties have been reported elsewhere [4,8–10].

This report is concerned with the phototransferred thermoluminescence (PTTL) of alexandrite. PTTL is produced as a result of the optical transfer of electrons from populated electron traps to purposely emptied ones. The number of materials whose PTTL has been looked at is considerable, but one can single out a few. These include various minerals such as quartz [11–14], and synthetic materials like Al_2O_3: C [15–17], Al_2O_3:C, Mg [18,19], Al_2O_3:Cr [20] and BeO [21–23], calcite [24] and CaF_2 [25]. The current report is the first such study of PTTL in alexandrite.

The aim of this work is to report PTTL induced in alexandrite by 470 nm blue light. We discuss its measurement, analysis of PTTL time-response profiles, that is, the dependence of PTTL intensity on duration of illumination and also touch on the influence of stimulation temperature on the optically stimulated luminescence (OSL) emitted during illumination to induce phototransfer.

2. Materials and methods

The alexandrite studied originates from Bahia state, Brazil, and is the same material investigated by Trindade et al. [4,9]. PTTL was measured using a RISØ TL/OSL DA-20 Luminescence Reader. Samples were irradiated using a $^{90}Sr/^{90}Y$ beta source at a nominal dose rate of 0.08 Gy/s. Luminescence was detected using an ET-PDM9107-CP-TTL photomultiplier tube through two Hoya U-340 filters of combined thickness 7.5 mm giving an effective transmission band of 240–390 nm (FWHM).

As a first step in the measurement of PTTL, an irradiated sample was preheated to a specific temperature to deplete electron traps of certain peaks below that temperature. The alexandrite was then illuminated with a set of 470 nm blue LEDs at room temperature to transfer electrons from filled deeper-to the emptied shallower electron traps. The sample was thereafter heated to 500 °C to monitor PTTL. Unless otherwise specified, all measurements were made at a heating rate of 1 °C/s.

* Corresponding author.
E-mail address: neilotrindade@usp.br (N.M. Trindade).

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3. Conventional thermoluminescence

3.1. Glow curve

Fig. 1 shows a glow curve of alexandrite measured after irradiation to 150 Gy. There are at least five peaks at approximately 75 °C (peak I), 120 °C (peak II), 190 °C (peak III), 280 °C (peak IV) and 450 °C (peak V). Previous analysis [4,9] conclude that peak IV is composed of two overlapping components. On the other hand, peak V has not been previously observed and is being reported for the first time here.

3.2. Thermal cleaning

In order to further identify the components of the glow curve, the so-called thermal cleaning procedure [26] was used. The sample was sequentially preheated to different temperatures to remove each peak and thereby reveal any adjacent peak beyond it. The sample was thus heated to 104, 143, 226, 271, 385 and 500 °C in turn after irradiation to 150 Gy.

3.3. Assessment of order of kinetics

3.3.1. Assessment using the dependence of peak position on irradiation dose

The position of each of the five glow peaks was observed to remain within 2 °C of the peak average for each one indicating that the peaks all follow first order kinetics.

3.3.2. T_m-T_{stop}

The T_m-T_{stop} method [26] which monitors the change in the position of a given peak each time the sample is partially heated, was also used to determine the order of kinetics of the peaks. At every step, the sample was irradiated to 150 Gy, preheated to a temperature between 50 and 500 °C and after cooling, heated to 500 °C to measure the full glow curve. The preheating temperatures were incremented in 10 °C intervals. Fig. 2 shows the results. There are five distinct regions representing different glow peaks. The positions of peaks I, II, III and V are essentially independent of T_{stop} which implies that they follow first order kinetics. On the other hand, the position of peak IV increases with $T_{stop.}$ This behavior is consistent with the peak being composed of multiple components as has also been observed experimentally [4,9,10,27]. Its order of kinetics could not be determined on this basis. For purposes of PTTL, we trat it as a single peak as it could not be disaggregated by either thermal cleaning or the T_m-T_{stop} method.

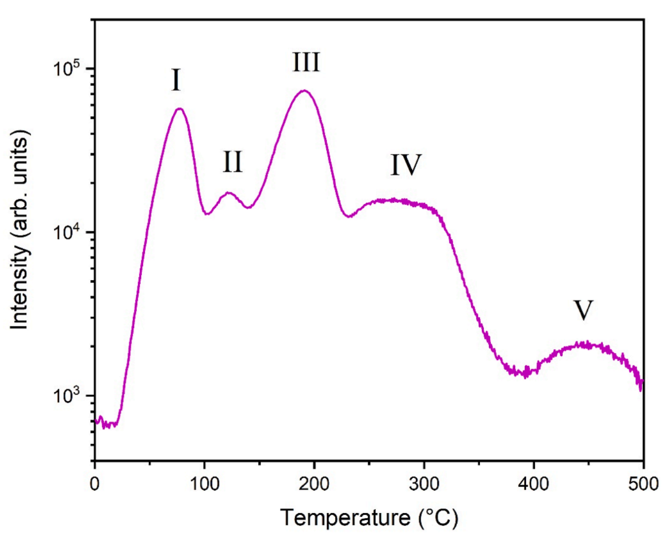

Fig. 1. A glow curve of alexandrite measured at 1 °C/s after beta irradiation to 150 Gy.

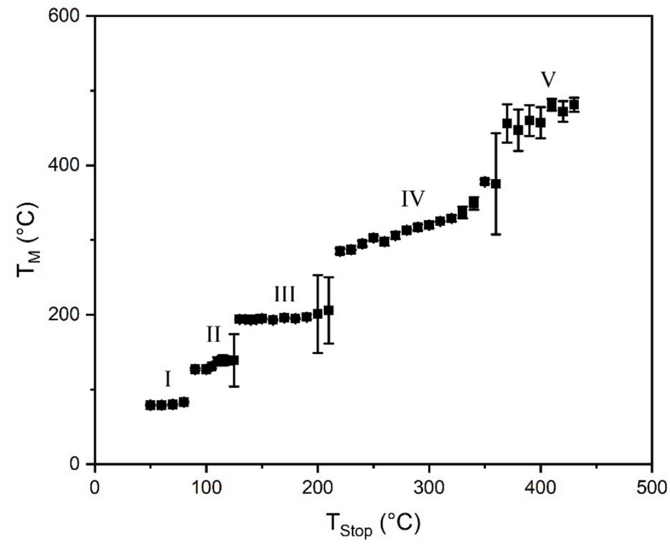

Fig. 2. The dependence of peak position T_m on the preheating temperature $T_{stop.}$ The values of T_{stop} run from 50 to 500 °C in increments of 10 °C. There are five distinct regions associated with each of the five peaks.

4. Preparatory tests for PTTL

4.1. PTTL and preheating temperature

Various test measurements for PTTL were made to determine which glow peaks would appear under phototransfer. This was done by first preheating the sample to the same temperatures as used in the thermal cleaning experiment followed by 470 nm illumination for 100 s and heating to 500 °C to monitor PTTL. The preheating was meant to remove each of the peaks in turn. Fig. 3 shows examples of glow curves measured after various preheats and illumination. Peaks I-III reappear under phototransfer whereas peaks IV and V do not. There was no appreciable PTTL observed after preheating to 385 °C and 500 °C meaning that any contribution from deep traps, if any, is minimal.

4.2. Light induced fading

The aim of this experiment was to identify which peaks would fade

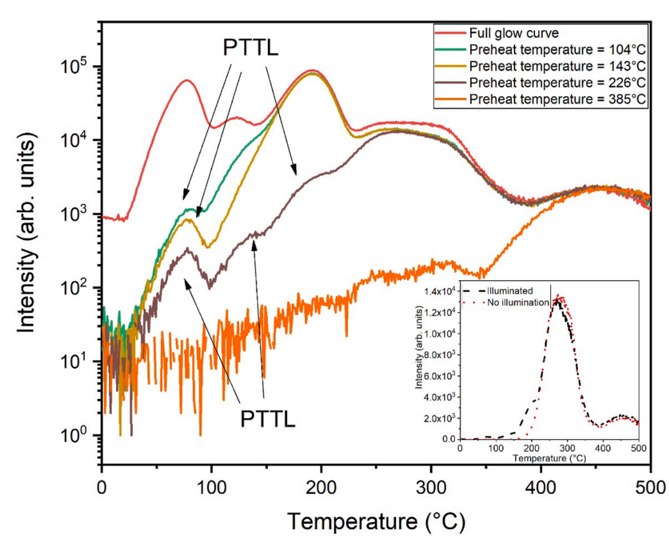

Fig. 3. Glow curves measured after various preheating and illumination. The PTTL produced is indicated. A glow curve measured after preheating to 226 °C but without any illumination is shown for comparison.

under optical stimulation as this may point to their involvement in phototransfer. The sample was irradiated to 150 Gy and illuminated by blue LEDs (470 nm) for periods from 10 to 10000 s before thermoluminescence measurement. We chose blue LEDs based on previous works [8,9], which reported that optically stimulated luminescence (OSL) can be observed with this illumination. Fig. 4 shows selected examples of glow curves corresponding to various illumination times. In general, all TL peaks except peak V decrease in intensity with illumination. Based on this observation, we initially hypothesize that all these peaks, except peak V, may be involved in the phototransfer process.

4.3. Repeatability

During routine PTTL measurements, the same sample is used multiple times. In order to assess the repeatability of peak position and peak intensity, glow curve measurements were made 10 consecutive times on a sample irradiated to 150 Gy each time. The collection of glow curves recorded are shown in Fig. 5. This shows peaks at 77 ± 2 °C (peak I), 122 ± 2 °C (Peak II), 191 ± 1 °C (peak III), 281 ± 6 °C (peak IV) and 452 ± 4 °C (peak V). These peaks are thus reproducible in position. The poorly defined peak V is also fairly reproducible. The coefficient of variation (C.V.) for each peak was calculated based on the intensity, using the standard deviation divided by the average of the ten samples. The average intensity was determined for each peak individually, with the beginning and end of each peak being consistently selected across all ten measurements. The C.V. were 0.7 %, 1.26 %, 2.0 %, 1.6 % and 4.6 %, respectively. Since the coefficients of variation are less than 5 % [28], it is concluded that sample re-use does not lead to any significant changes in the glow curve.

5. Pulse annealing

Pulse annealing is a technique used to qualitatively determine the role of electron traps as acceptors or donors during PTTL [20,22]. In applying the method in this study, the sample was each time irradiated to 150 Gy, preheated to temperatures between 50 and 500 °C at 10 °C intervals, illuminated for 100 s, and heated to 500 °C to record the full glow curve. Fig. 6 shows pulse annealing results for peaks I–V. The main observations are as follows: peak I decreases in intensity with preheating and remains relatively stable near 120 °C beyond which it is reproduced under phototransfer. The peak decreases further thereafter. Peak II is initially stable in intensity before a decrease sets in with preheating. One notices that the change in intensity of peak I follows that of peak II. The intensity of peak III decreases between 150 and 230°C or so with preheating. This change affects peaks I and II which suggests that the electron trap of peak III is a likely donor for PTTL observed at both peaks. Peak IV remains stable until it is removed by preheating. The role of this peak is mostly as a donor for PTTL produced at lower temperatures. The same comments apply for peak V. Table 1 summarizes systems of acceptor and donor in PTTL for the alexandrite sample.

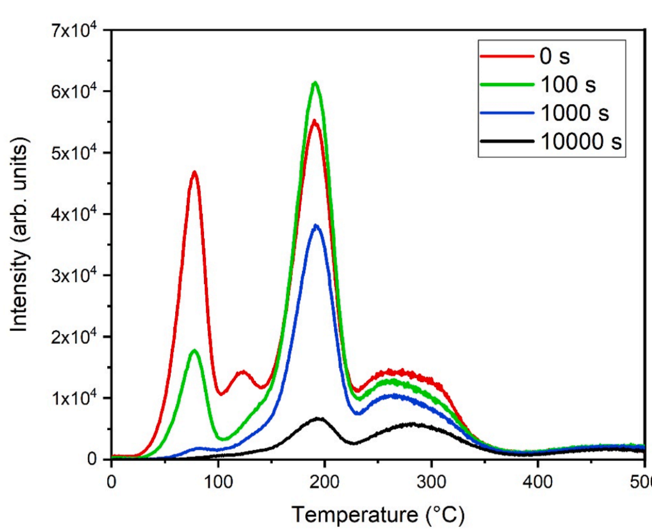

Fig. 4. When the alexandrite is illuminated before use, its TL fades as exemplified for illumination for 100, 1000 and 10000s.

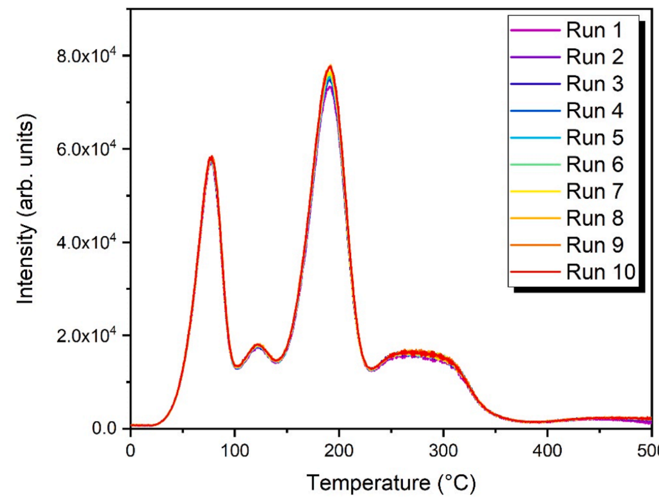

Fig. 5. Glow curve of alexandrite measured 10 consecutive times.

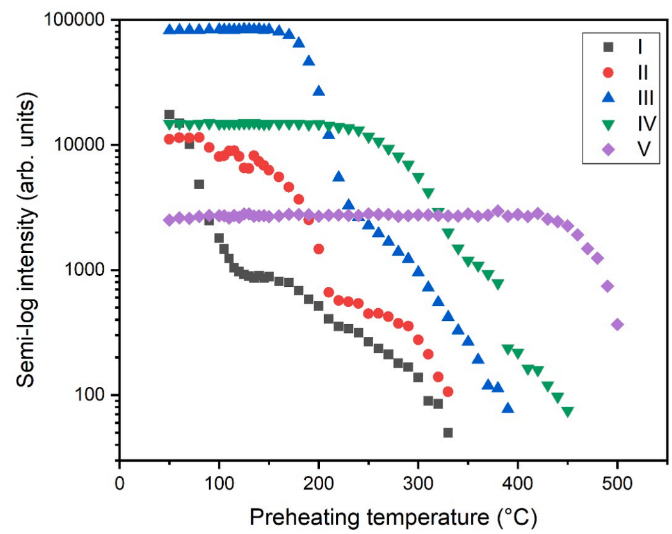

Fig. 6. The dependence of peak intensity on preheating temperature. Glow curves were recorded after illumination each time.

Table 1
A summary of system of acceptors and donors in the PTTL for alexandrite. The acronym 1A2D denotes a system of one acceptor and two donors. Such a system corresponding to peak IV could not be determined. No PTTL is observed at peak V.

Peak label	Preheating temperature (°C)	TL	PTTL	
		T_M (°C)	Donors	System
I	104	77	III, IV	1A2D
II	143	122	III, IV	1A2D
III	226	191	IV	1A2D
IV	385	277		
V	500	448	N/A	

6. PTTL time response profile

The intensity of PTTL peaks I-IV after the preheating to 104, 143, 226 and 385 °C were studied as a function of illumination time. The duration of illumination varied from 5 to 10 000 s.

6.1. PTTL after preheating to 104 °C to remove peak I

Fig. 7(a) shows the time-response profile for peak I obtained after preheating to 104 °C. This preheating removes peak I only. The PTTL briefly rises in intensity in the first 20 s and then decreases thereafter. The PTTL may theoretically be linked to electron traps of peaks II-V as potential donors. However, as deduced from pulse annealing, the main donors for the PTTL correspond to peaks III and IV. This is then a system of one acceptor and two donors (Table 1).

6.2. PTTL after preheating to 226 °C to remove peaks I, II and III

Fig. 7(b) shows the time-response profile for peak I after preheating to 226 °C to remove peaks I– III. The PTTL intensity decreases consistently with illumination time. Possible electrons trap donors for PTTL at peak I correspond to peaks IV and V.

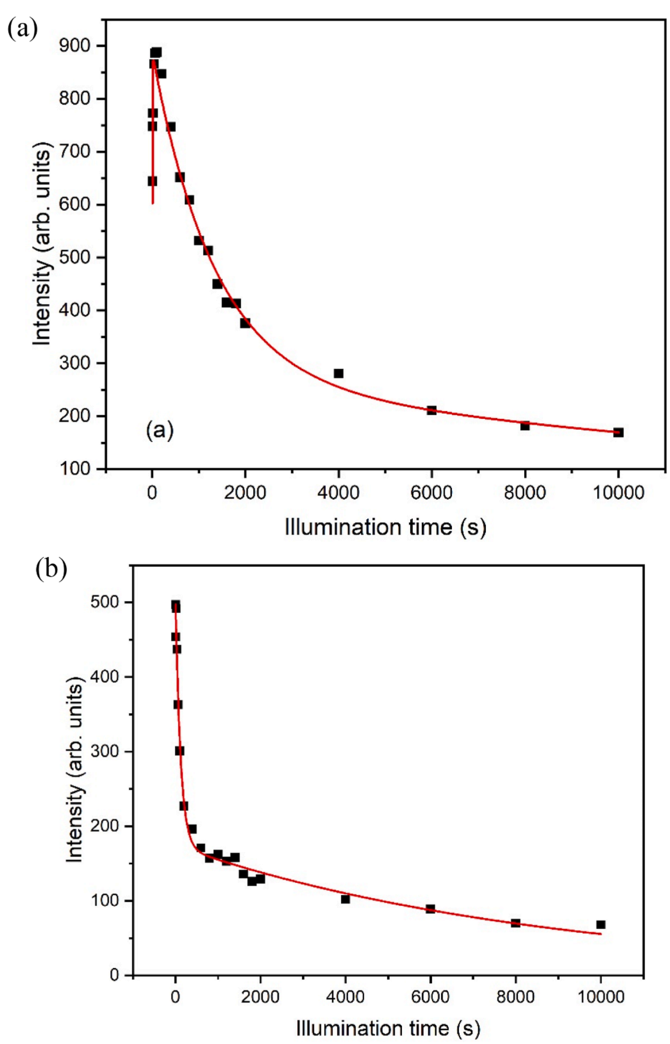

Fig. 7. Time-response profile of PTTL I after preheating to (a) 104 °C to remove peak I (a) to 226 °C to remove peaks I-III (b). The line through data in each graph is the best fit of Eq. (4).

6.3. PTTL after preheating to 385 °C to remove peaks I-IV

Fig. 8(a) shows time-response profiles of peaks III and IV after preheating to 385 °C to remove peaks I-IV. Their intensities increase consistently with illumination. In comparison, the intensity of peak V shown in Fig. 8(b), initially increases and then drops with duration of illumination. The slow change in the profiles of peaks III and IV imply that much longer illuminations are needed to properly define overall behavior. Further, the intensity of peak V, as a supposed donor should have decreased monotonically. The counter-intuitive initial increase is an indication of competition effect where by its electron trap acts both as an acceptor and as a donor. Similar behavior has been seen in tanzanite [29].

7. Brief analysis of PTTL time-response profiles

The phototransfer phenomenon is a process that involves the transfer of electrons to an acceptor from several donors whose number depends on the preheating temperature. To analyze the illumination time profiles, we adopted the method of Chithambo et al. [30], where electron transport in PTTL is described as a system of acceptor and donors using sets of rate equations which are set up on the basis of experimental results. The analytical solutions for each system are applied to the experimental data.

7.1. PTTL after preheating to 104 °C to remove peak I

When the sample is preheated to 104 °C, only the peak I is removed. The peak is reproduced under phototransfer. The peaks that are deduced to contribute most to the PTTL are associated with peaks III and IV. This is a system of one acceptor and two donors. The set of differential equations describing the transfer of charge from donors to the acceptor for this system are:

$$\frac{dN_3}{dt} = -f_3N_3 \tag{1}$$

$$\frac{dN_4}{dt} = -f_4N_4 \tag{2}$$

$$\frac{dN_1}{dt} = -f_1N_1 + af_3N_3 + af_4N_4 \tag{3}$$

where Eqs. (1) and (2) represent the optical stimulation of electrons from donors and Eq. (3) describes charge transport at the acceptor. The first term of Eq. (3) describes the optical loss of electrons from the acceptor at a rate f_1 whereas the other terms give the portion of electrons re-trapped at the acceptor after release from the specified donor. The solution of sets of equations (1)–(3) is given by:

$$N_1 = A\left(e^{-f_3t} - e^{-f_1t}\right) + B\left(e^{-f_4t} - e^{-f_1t}\right) \tag{4}$$

where $A = \frac{a_3f_3N_{3i}}{f_1-f_3}$, $B = \frac{a_4f_4N_{4i}}{f_1-f_4}$. N_{3i} and N_{4i} are initial concentration of electrons at the electron traps III and IV, respectively and the terms a_i are constants of proportionality. The line through data inFig. 7(a) is a fit of Eq. (4) and satisfactorily describes the experimental data.

7.2. PTTL after preheating to 226 °C to remove peaks I, II and III

After preheating to 226 °C to remove peaks I-III, peak I is reproduced under phototransfer. The associated donor peaks were deduced to be IV and V. This is also a system of one acceptor and two donors and the time-dependence of the PTTL at peak I can also be described by Eq. (4). The fit of the latter is shown in Fig. 7(b).

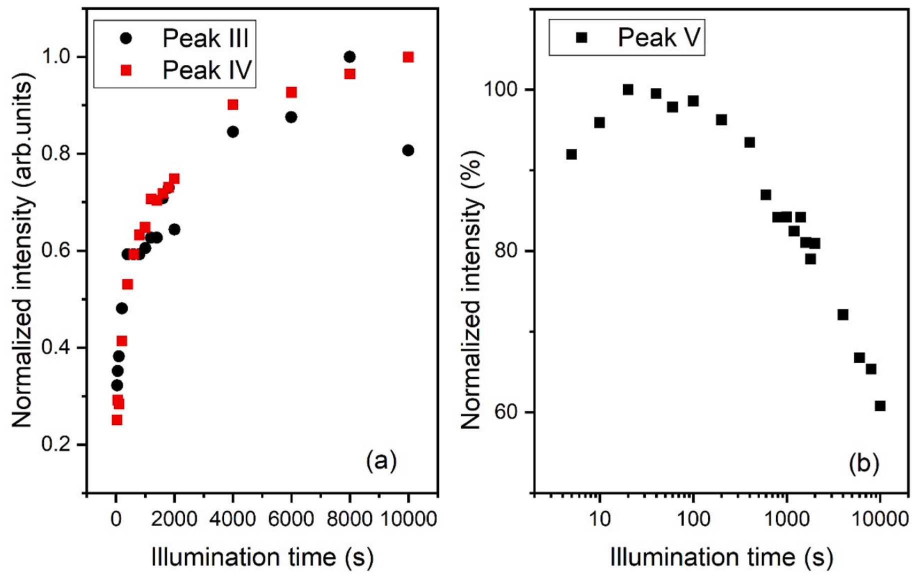

Fig. 8. Time-response profile of PTTL peaks III and IV after preheating to 385 °C (a) the dependence of peak intensity on illumination for peak V (b).

7.3. Measurement after preheating to 385 and 500 °C

As was pointed out earlier, the data of Fig. 8(a), although measured up to 10 000s is not sufficiently well defined. This would require considerably long exposures to achieve this. Qualitatively, the PTTL intensity seems to approach saturation.

8. Influence of thermal quenching and thermal assistance on OSL curves

During sample illumination to induce phototransfer, OSL is emitted. Because the intensity of OSL depends on illumination temperature, this can be used to qualitatively study the influence of thermal quenching and thermal assistance on the OSL. This has been done in previous studies of PTTL [25,31]. To study this, the sample was irradiated to 150 Gy and either illuminated 20 consecutive times at room temperature or illuminated 20 times but with the illumination temperature changed from 20 to 200 °C each time.

Fig. 9 compares the temperature dependence of the OSL measured at room temperature recorded with the temperature changing. The intensity of OSL obtained at higher temperatures exceeds that recorded at room temperature. This is attributed to the effect of thermal assistance although thermal quenching beyond 180°C or so causes the OSL intensity to decrease.

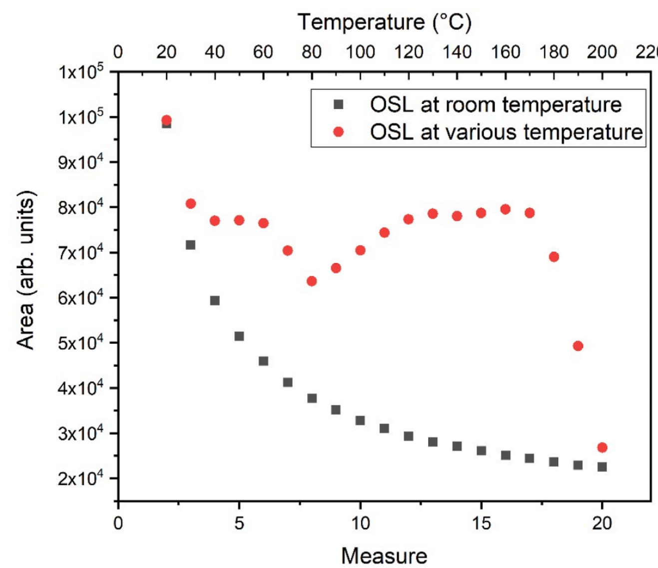

Fig. 9. The change of OSL intensity with re-measurement for experiments made at room temperature (squares) or with the temperature changing from 20 to 200 °C (circles).

9. Conclusions

A preliminary study of the PTTL of alexandrite has been reported. The sample studied shows five glow peaks at 75 °C (peak I), 120 °C (peak II), 190 °C (peak III), 280 °C (peak IV) and 450 °C (peak V). Peaks I-III and V are of first-order kinetics. Peak IV is far too broad for such analysis and is known to consist of at least two closely spaced components. The dependence of PTTL intensity on duration of illumination has been analyzed as systems of acceptor and donors with the number of the latter dependent on the preheating temperature. In qualitative tests, the OSL emitted during sample illumination is shown to be affected by thermal assistance and thermal quenching.

CRediT authorship contribution statement

A.O. Silva: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation. **N.M. Trindade:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources. **M.L. Chithambo:** Writing – review & editing, Writing – original draft, Validation, Resources, Methodology, Formal analysis, Data curation, Conceptualization.

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

Data will be made available on request.

Acknowledgments

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