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Thermoluminescence under an exponential heating function: II. Glow-curve deconvolution of experimental glow-curves

G Kitis[1], R Chen[2], V Pagonis[3], E Carinou[4], P Ascounis[4] and V Kamenopoulou[4]

[1] Aristotle University of Thessaloniki, Nuclear Physics Laboratory, 54124-Thessaloniki, Greece
[2] School of Physics and Astronomy, Raymond and Beverly Sackler Faculty of Exact Sciences, Tel-Aviv University, Tel-Aviv 69978, Israel
[3] Physics Department, McDaniel College, Westminster, MD 21157, USA
[4] Greek Atomic Energy Commission. PO Box 60092, Ag. Paraskevi 15310, Greece

E-mail: gkitis@auth.gr

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Abstract
Thermoluminescence (TL) glow-curves measured using an exponential heating function (EHF) in constant temperature hot gas readers, cannot be analysed using the existing single TL glow-peak equations derived assuming a linear heating rate. In the present work single TL glow-peak equations, which were recently derived assuming an EHF, are used to perform a computerized glow-curve deconvolution analysis of experimental glow-curves measured using a stable temperature hot gas reader. Glow-curves of the most commonly used dosimetric material LiF:Mg,Ti were analysed using the first order kinetics glow-peak equations. The glow-curves were analysed for samples that were pre-irradiation annealed at 400 °C for 1 h and 100 °C for 2 h, with and without a post-irradiation annealing at 80 °C for 1 h. TL glow-peak equations of the general order kinetics were used to analyse experimental glow-curves of the dosimetric material $Li_2B_4O_7$: Mn,Si. The results showed that the recently derived TL equations are very efficient for analysing glow-curves measured using stable temperature hot gas readers.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Thermoluminescence (TL) glow-curves measured using constant temperature hot gas readers are characterized by a relatively poor resolution of the individual glow-peaks, making the computerized glow-curve deconvolution (GCD) procedure a difficult task [1]. Previous efforts to analyse the glow-curves obtained by constant temperature hot gas readers [1,2] were not based on single glow-peak models associated with an exponential heating function (EHF). Recently, Kumar *et al* [3] used an EHF model to fit the glow-curve of $CaSO_4$.

In a preceding paper Kitis *et al* [4] presented TL kinetic equations describing single glow-peaks for the first and general order kinetics. These equations were of the form $I(n_0, E, s, \alpha, T)$ for the first order kinetics and of the form $I(n_0, E, s, b, \alpha, T)$ for the general order kinetics, and they were derived for the case of an EHF. In these equations n_0 (cm^{-3}) denotes the initial concentration of trapped electrons, s (s^{-1}) is the frequency factor, E (eV) is the activation energy, b is the kinetic order, α (s^{-1}) is associated with the rate of heating and T is the temperature in kelvin. These kinetic equations were further transformed into more efficient equations of the

general form $I(I_m, E, T_m, b, T)$, which can be used for GCD. These equations contain the maximum TL intensity I_m and the peak maximum temperature T_m, which can be evaluated directly from the experimental glow-curves. The equations which will be used in the GCD analysis of the data in this paper are described here.

When the exponential integral appearing in the TL kinetic equations is approximated by an asymptotic series [5, 6], the $I(I_m, E, T_m, T)$ equation for the first order kinetics is

$$I(T) = I_m \exp\left[-\frac{E(T_m - T)}{kTT_m} + \frac{T_g - T_m}{T_m} \times \left(Z_m - \frac{T}{T_m} \exp\left[-\frac{E(T_m - T)}{kTT_m} \right] Z(T) \right) \right], \quad (1)$$

$$Z(T) = \sum_{n=0}^{N} (-1)^n n! \left(\frac{KT}{E} \right)^n \left(\left(\frac{T_g}{T_g - T} \right)^{n+1} - 1 \right) + EC, \quad (2)$$

where EC [4] is a correction term equal to the one half of the $(N+1)$th term, with the appropriate sign. This term is added in order to minimize the error due to the use of the asymptotic series and Z_m is the value of $Z(T)$ at T_m.

When a convergent series is used to approximate the exponential integral appearing in the TL kinetic equations [5,6], the equations of the form $I(I_m, E, T_m, b, T)$ for the first and general order kinetics are

$$I(T) = I_m \exp\left[-\frac{E(T_m - T)}{kTT_m} - \frac{E(T_g - T_m)}{kT_m^2} \times \exp\left(\frac{E(T_g - T_m)}{kT_m T_g} \right) (Z_1(T_m) - Z_1(T))_{(\mathrm{CSA})} - \frac{T_g - T_m}{T_m} \left(Z_2(T_m) - Z_2(T) \frac{T}{T_m} \times \exp\left(-\frac{E(T_m - T)}{kTT_m} \right) \right) \right] \quad (3)$$

and

$$I(T) = I_m \exp\left(-\frac{E(T_m - T)}{kTT_m} \right) \times \left[1 - \frac{(b-1)(T_g - T_m)}{bT_m} \left(\frac{E}{kT_m} \exp\left(\frac{E(T_g - T_m)}{kT_g T_m} \right) \times (Z_1(T) - Z_1(T_m))_{(\mathrm{CSA})} + \frac{T}{T_m} Z_2(T) \times \exp\left(-\frac{E(T_m - T)}{kTT_m} \right) - Z_2(T_m) \right) \right]^{b/(b-1)}, \quad (4)$$

where

$$Z_1(T)_{\mathrm{CSA}} = \gamma + \ln|G(T)| + \sum_{n=1}^{50} \frac{G(T)^n}{n \cdot n!}, \quad (5)$$

$$Z_2(T) = \sum_{n=0}^{N} (-1)^n n! \left(\frac{KT}{E} \right)^n + EC, \quad (6)$$

$$G(T) = \frac{E}{kT} \frac{T - T_g}{T_g}, \quad (7)$$

where $\gamma = 0.577\,215\,6649$ is the Euler constant and $Z_1(T_m)$, $Z_2(T_m)$ are the values of $Z_1(T)$ and $Z_2(T)$ at T_m. The correction term $E \cdot C$ has the same meaning as in equation (2).

1.1. Experimental procedure

Experimental glow-curves of the dosimetric materials LiF : Mg,Ti and $Li_2B_4O_7$: Mn,Si were measured using the automatic stable temperature hot gas TLD reader of Rados Technology Oy (Finland). The LiF : Mg,Ti chips were annealed at 400 °C for 1 h and at 100 °C for 2 h, whereas the $Li_2B_4O_7$: Mn,Si chips were annealed at 300 °C for 30 min. Both materials were post-irradiation annealed at 80 °C for 1 h. A test dose of 5 mGy was delivered using the Rados automatic TLD irradiator with the built-in calibrated $^{90}Sr/^{90}Y$ beta ray source.

The GCD analysis is performed using the MINUIT program, which is used to find the minimum value of a multi-parameter function and to analyse the shape of a function around its minimum. The principal application of MINUIT is for statistical analysis, for work on the chi-square or log-like functions and to compute the values of the best fit parameters and their uncertainties, including correlations between the parameters. It is especially suited to solve difficult problems [7]. The initial values for the fitting parameters I_m and T_m are evaluated from the experimental glow-curves, whereas the guessing values for the fitting parameter E(eV) are taken from the literature [11–15].

Goodness of fit was tested using the figure of merit (FOM) of Balian and Eddy [8] , given by

$$\mathrm{FOM} = \sum_i \frac{|Y_{\mathrm{Exper}} - Y_{\mathrm{Fit}}|}{A}, \quad (8)$$

where Y_{Exper} is the experimental glow-curve, Y_{fit} is the fitted glow-curve and A is the area of the fitted glow-curve.

2. Computerized GCD

2.1. Reference glow-curve (RGC)

Before applying the GCD analysis to the experimental results, it is necessary to estimate the efficiency of the single glow-peak expressions proposed by Kitis *et al* [4]. A reference glow-curve (RGC) was derived using the software package Mathematica in which the exponential integral is a built-in function and no approximation is required. The RGC consists of three first order kinetics glow-peaks with activation energies $E = 1.1$ eV, 1.25 eV, 1.4 eV, frequency factors $s = 10^{12}\,\mathrm{s}^{-1}$, $5 \times 10^{12}\,\mathrm{s}^{-1}$, $10^{13}\,\mathrm{s}^{-1}$ and the initial concentrations of trapped electrons equal to $n_0 = 10^{13}\,\mathrm{cm}^{-3}$, $10^{13}\,\mathrm{cm}^{-3}$ and $2 \times 10^{13}\,\mathrm{cm}^{-3}$, respectively. The T_g was 600 K and $\alpha = 0.29\,\mathrm{s}^{-1}$. The GCD procedure was applied twice to the RGC. In the first case, the expression given by equation (3) was used as a model for all the three glow-peaks. The resulting FOM value was 8.9×10^{-6}. In the second case a combination of equations (1) and (3) was used. Which of these two equations is used depends on the value of the argument of the exponential integral (see [4] for details). When the values of the argument are $|z| > 10$ only then equation (1) is used, whereas when the values of the argument $|z|$ are between 0 and 10, equation (3) is employed instead. In this case the FOM value is further improved to 4×10^{-6} relative to the first case where only equation (3) is employed. It must be noted, however, that the initial values of the trapping parameters were

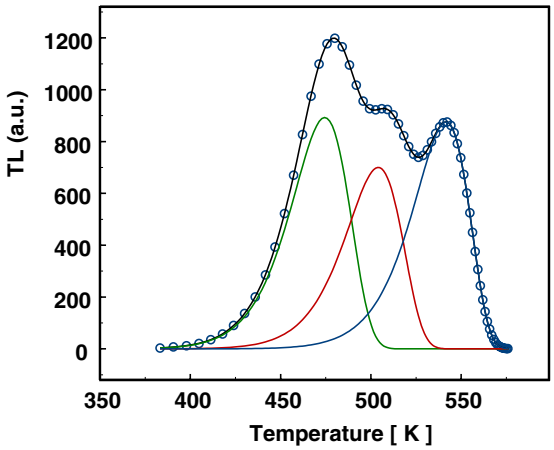

Figure 1. GCD of a synthetic glow-curve. Open circles correspond to the RGC and the solid lines to the fitted curves for the individual glow-peaks. FOM $= 4 \times 10^{-6}(4 \times 10^{-4}\%)$.

successfully reproduced by the fitting procedure in both the cases. The results of the GCD analysis are shown in figure 1. In conclusion, the single TL glow-peak expressions given by Kitis *et al* [4] are extremely efficient for the deconvolution of the complex glow-curves consisting of several overlapping single glow-peaks.

2.2. *LiF : Mg,Ti*

The experimental glow-curves measured using a stable temperature hot gas reader are not recorded as a function of the temperature T of the sample but are measured as a function of the heating time t. The temperature has to be evaluated using [4, 5]

$$T(t) = T_g - (T_g - T_0)\mathrm{e}^{-\alpha t}, \tag{9}$$

where T_g is the hot gas temperature, T_0 is the temperature at time $t = 0$, and α in s^{-1} given by the expression

$$\alpha = \frac{\delta A}{mc_p}, \tag{10}$$

where δ is the heat transfer efficiency, A is the heated area, m is the mass of the sample and c_p is the heat capacity.

Parameter α must be evaluated from equation (10). However, since the dosemeters used are sintered pellets, the values of the parameters in equation (10) are unknown, so that α cannot be evaluated. Therefore, an effective value of α has to be evaluated experimentally for each material. The first approach is to find the possible region of the values of α for each dosimeter. Solving equation (9) for α, one obtains

$$\alpha = -\frac{1}{t}\ln\left[\frac{T_g - T}{T_g - T_0}\right]. \tag{11}$$

The time corresponding to the maximum of the glow-peak is $t = t_m$ and one has to find the corresponding temperature T_m in order to evaluate parameter α. For example, in the case of the glow-curve of LiF : Mg,Ti measured with $T_g = 300\,^{\circ}\mathrm{C}$, the time t_m of glow-peak 5 appears at a time value of 7 s. The value of $t_m = 7$ s is the only information available, since values of T_m (and of any T) are not available for a stable temperature hot gas reader. However, values of T_m are available from glow-curves measured using a constant heating rate β. In the case of EHF the heating rate is equal to $\beta(T) = \alpha(T_g - T)$, which at the maximum is equal to $\beta(T_m) = \alpha(T_g - T_m)$. Using the condition at the maximum TL intensity [4], which holds for any heating function (including, of course, linear and exponential) [6], it is obvious that when $\beta = \beta(T_m)$, the glow-peaks obtained using either a linear or an EHF have the same values of T_m, as the glow-curves measured using constant heating rates are between 8 and $30\,^{\circ}\mathrm{C\,s}^{-1}$ [9, 10]. Therefore, it is allowed to take T_m values of peak 5 from the glow-curves received using constant heating rates between 8 and $30\,^{\circ}\mathrm{C\,s}^{-1}$ [9, 10] and to insert them in equation (11). Using this method, values of α between 0.25 and $0.35\,\mathrm{s}^{-1}$ are evaluated. However, this is a rather broad range of values, so an additional procedure has to be applied in order to find a more representative value of α. Samples of LiF : Mg,Ti annealed at $400\,^{\circ}\mathrm{C}$ for 1 h and post-irradiation annealed at $80\,^{\circ}\mathrm{C}$ for 1 h were readout using a hot gas temperature of $300\,^{\circ}\mathrm{C}$. The resulting glow-curves, which contain mainly glow-peaks 4 and 5, were analysed into their individual glow-peaks using equation (3). The GCD procedure was applied on the same glow-curve for different values of α. The criterion adopted for the selection of the most representative α was as follows. The most representative value of α is the value which gives the 'correct values' of activation energies for the glow-peaks 4 and 5. These 'correct values' are obtained from the GLOCANIN intercomparison programme

Table 1. Evaluation of the most representative value of the constant α from a GCD procedure, using EHF algorithms (equation (3)).

	EHF algorithm		
α	E_4	E_5	FOM
0.2	1.07	1.498	0.8
0.25	1.19	1.79	0.9
0.28	1.47	1.923	0.84
0.29	1.44	2.106	0.61
0.3	1.68	2.065	0.63
0.31	1.69	2.2	0.82
0.35	1.9	2.8	2.5

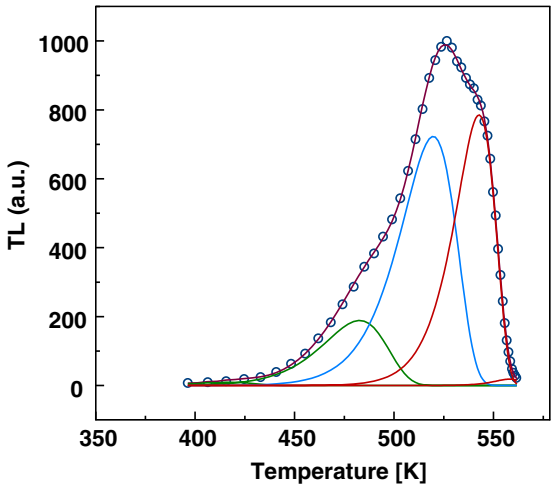

Figure 2. GCD of LiF : Mg,Ti using equation (3), which uses a convergent series approximation for the exponential integral. Open circles are the experimental points and solid lines the fit to the individual glow-peaks. FOM $= 0.61\%$.

of Bos *et al* [11], and they are 1.4–1.5 eV for glow-peak 4 and 2–2.15 eV for glow-peak 5. The possible existence of temperature lag effects in the stable temperature hot gas reader are not taken into consideration here because the goal of the present work is to test the applicability of the newly obtained algorithms. On the other hand, the presence of temperature lag effects does not seriously affect the 'correct values' of the activation energies when they are obtained by the peak shape and curve fitting methods. As shown previously by Piters and Bos [12] and Kitis and Tuyn [13], the relative error in the activation energy $\Delta E/E$ is of the order of $\Delta T/T_m$, with ΔT representing the temperature lag.

The results of this method are shown in table 1. The very low FOM values obtained indicates an excellent fitting in all the cases. The 'correct values' of the activation energies are obtained for values of α between 0.28 and 0.31 s^{-1}. Finally, a value of $\alpha = 0.29\,s^{-1}$ is adopted, since it gives the lowest FOM value. A glow-curve analysed with $\alpha = 0.29\,s^{-1}$ is shown in figure 2.

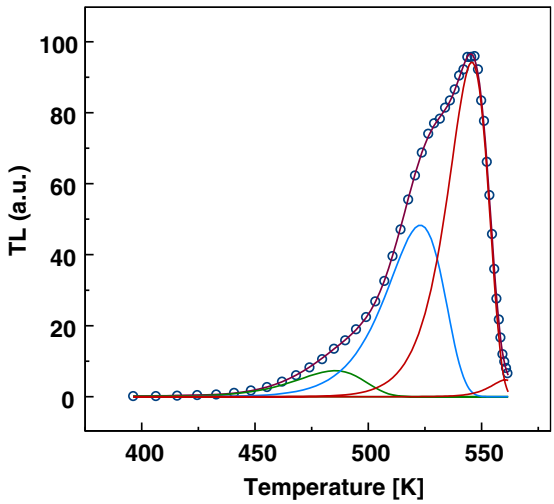

Figure 3. Deconvolution of LiF : Mg,Ti glow-curve resulting from that of figure 2 normalized over the heating rate function given by $\alpha(T_g - T)$, using equation (3), which uses a convergent series approximation for the exponential integral. Open circles indicate experimental points and solid lines the fit to the individual glow-peaks. FOM = 0.62%.

In the case of glow-curves measured using a linear heating rate, the TL intensity is measured as counts per unit time and is subsequently normalized by dividing by the constant heating rate to yield counts per unit temperature. In the case of a glow-curve measured using an EHF, the TL is measured per unit time which, however, corresponds to a variable temperature interval. The normalization in this case must be performed by dividing by the heating rate function, which is given by $\alpha(T_g - T)$. By performing this normalization the normalized glow-curve of figure 3 is obtained, which has a shape very similar to that obtained using a linear heating function. This glow-curve can also be analysed by a GCD analysis using the same equation (3) as for the data of figure 2, with exactly the same E values but with a slightly higher value of T_m than in figure 2. The result shows that the normalization over the heating rate function does not alter the geometrical characteristics of the glow-peak.

The left-hand side of figure 4 shows the analysis of a glow-curve of LiF : Mg,Ti, which was measured without the post-irradiation annealing, so that it contains all glow-peaks 1–5 in this material. Glow-peak 1 in this material is known to be a double peak. The analysis of this glow-curve was performed as follows [4]. When the absolute value of the argument $|z|$ of the exponential integral appearing in TL kinetics equations (which in the present work is given by function $G(T)$ of equation (7)) is greater than 10 then equation (1) is used. On the other hand, when $|z|$ is between 10 and 0, equation (3) is used instead. The right-hand side of figure 4 was normalized by dividing by the heating rate function $\alpha(T_g - T)$ and then a GCD analysis was performed using the same procedure as for the data shown on the left-hand side of figure 4.

A very interesting and important test of the single TL glow-peak expressions under an EHF is to examine what happens when temperature T approaches the temperature of the hot gas T_g, i.e. when $T \to T_g$. This case can be examined experimentally by setting the T_g equal to 250 °C, which is

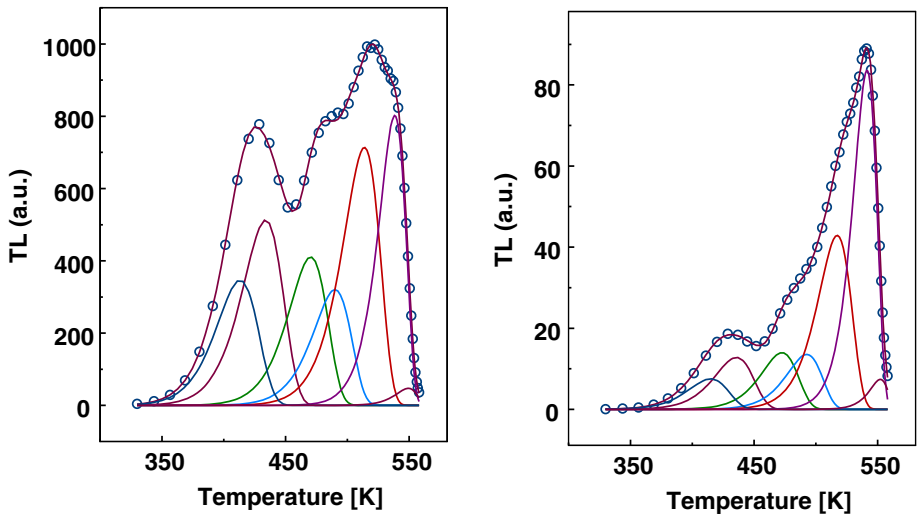

Figure 4. Deconvolution of LiF : Mg,Ti glow-curve in which all the glow-peaks 1–5 are present. Left-hand side: 'As received', FOM = 0.73% and the right-hand side: the same data normalized over the heating rate function given by $\alpha(T_g - T)$. FOM = 0.76%.

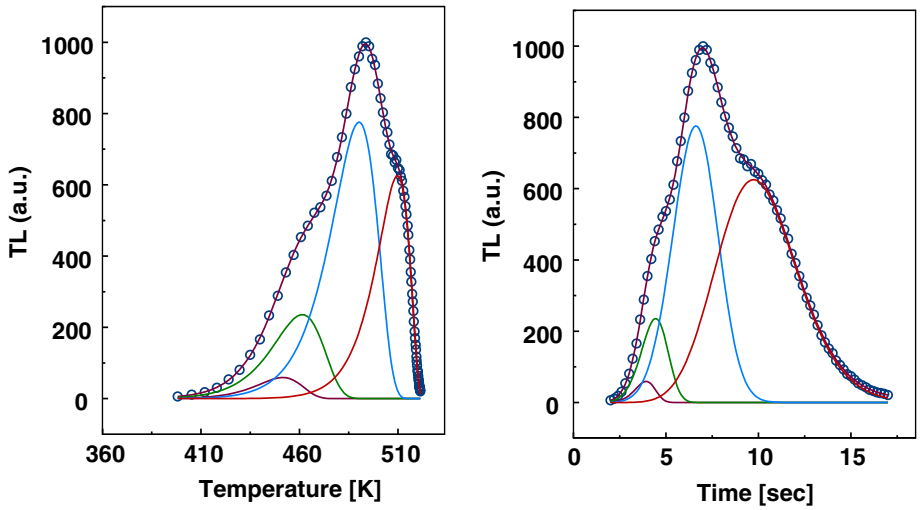

Figure 5. Deconvolution of LiF:Mg,Ti glow-curve measured using a hot gas temperature of 250 °C, which is close to the peak maximum temperature of the glow-peak 5, up to a readout time of 16 s. Left-hand side: in the temperature scale, FOM = 0.7% and the right-hand side: in the time scale.

very close to the peak maximum temperature of glow-peak 5 and by setting the readout time to 25 s. The resulting glow-curve is shown in figure 5. When the temperature T is much smaller than T_g, the glow-curve is recorded in the usual way. However, when the temperature T approaches T_g within 3 K or less, the heating rate $\alpha(T_g - T)$ becomes less than 1 K s^{-1} and as the readout continues the heating rate approaches zero very slowly, and glow-peak 5 exhibits, in fact, an isothermal-like decay. This case is shown on the left-hand side of figure 5. The deconvolution of this glow-curve is performed using equation (3). Since at the end of the glow-curve the temperature increases very slowly, there is a high density of experimental points at the end of the glow-curve when it is drawn on a temperature scale. The details of the fitting at the end of the glow-curve become clear only when the plot is drawn on a time scale, as shown on the right-hand side of figure 5. The region of the glow-curve between 10 and 16 s corresponds to a very slow increase in temperature from 237 (510 K) to 247 °C (520 K), which includes 60 experimental points. Note that the temperature region up to 237 °C, which contains glow-peaks 1a, 1b, 2, 3, 4 and a part of glow-peak 5, includes 100 experimental points.

The GCD of LiF : Mg,Ti described in the previous paragraph was performed up to a readout time of 16 s, although the total readout time was 25 s because the fit fails after 15 s. The reason for this failure is that the readout up to 250 °C influences a higher temperature glow-peak, such as peak 5a or peak 6. For this reason, the fitting of the glow-curve above 16 s, which takes place within a temperature range of less than 5 K and includes 90 experimental points, needs to include one more component. The final fit is shown in figure 6, where the Y-axis is shown on a log scale in order to see the details at long readout times. The last fit proves how powerful equation (3) is.

2.3. $Li_2B_4O_7$: Mn,Si

The glow-curve of $Li_2B_4O_7$: Mn,Si contains a single broad glow-peak, which can be used to test the general order kinetics

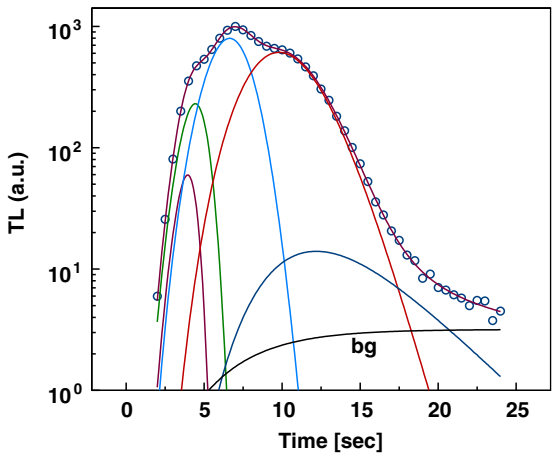

Figure 6. Deconvolution of LiF:Mg,Ti glow-curve received using a hot gas temperature of 250 °C, which is close to the peak maximum temperature of glow-peak 5 , up to a readout time of 25 s. The Y-axis is shown on a logarithmic scale in order to see the details at long readout times. FOM = 0.74%.

equation (4). The stable hot gas temperature is 300 °C. For this condition the corresponding value of t_m obtained from the experimental glow-curve is 4.2 s. The respective peak maximum temperature given by the manufacturer's manual (RADOS) is 220 °C. By substituting these values in equation (11) a value for α of 0.29 s^{-1} is found.

The glow-curve of this material measured using a linear heating rate was analysed by Kitis *et al* [15]. As a first step, a GCD is performed aiming for the best possible fit with the lowest possible number of individual glow-peaks. This case is shown on the left-hand side figure 7. The glow-curve is obtained using a hot gas temperature of 300 °C. The fit was achieved using three components for the main dosimetric peak, and the FOM value obtained for the fit was 1.6%. The first, second and third component in the deconvolution process gave activation energies of 0.69 eV, 0.72 eV and 1.6 eV, respectively. Although the aim of the present work is to test the derived equations and not to extract exact kinetic parameters, it is

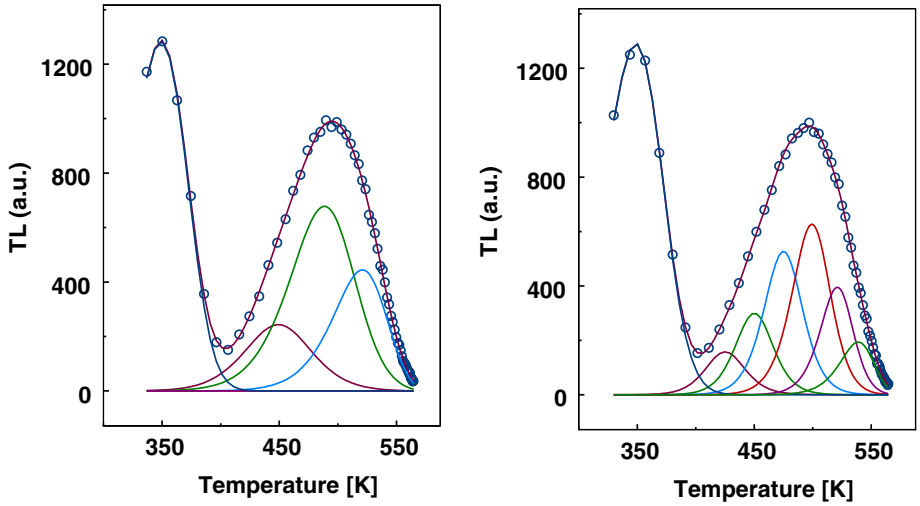

Figure 7. Deconvolution of the glow-curve of $Li_2B_4O_7$: Mn,Si, measured with a hot gas temperature of 300 °C, using the general order kinetics glow-peaks. Left-hand side: first step of the deconvolution, FOM = 1.6% and the right-hand side: second step of deconvolution, FOM = 1.54%.

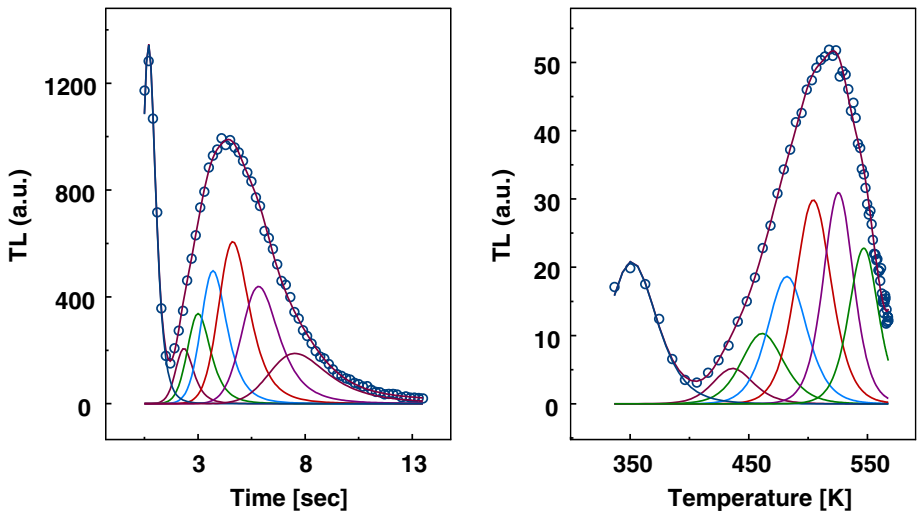

Figure 8. Deconvolution of the glow-curve of $Li_2B_4O_7$: Mn,Si, measured with a hot gas temperature of 300 °C, using the general order kinetics glow-peaks. Left-hand side: in the time scale and right-hand side: normalized over the heating rate function given by $\alpha(T_g - T)$.

necessary to obtain some more reliable values of the kinetic parameters and therefore a more sophisticated deconvolution is performed. Kitis *et al* [15] applied the T_m–T_{stop} and initial rise method to this material and found that the main dosimetric glow-peak is very complex and that the activation energy as a function of temperature across the main peak varies from 1.0 to 1.6 eV. The right-hand side of figure 7 shows the results of this deconvolution analysis. The E values found for each of the six glow-peaks from left to right are, 1.2, 1.4, 1.5, 1.6, 1.9 and 1.83 eV. The respective kinetic order b in all the cases is 2, i.e all peaks are of the second order. Figure 8 shows the same data as that on the right-hand side of figure 7 on a time scale, as well as after it is normalized by the heating rate function. The results look very similar to those obtained using a linear heating rate. From the latter results it is obvious that the gas temperature of 300 °C, which is used in routine readout of this material is rather low, since it does not erase the whole glow-peak and leaves a residual TL signal which can accumulate over successive readouts.

3. Conclusions

In stable temperature hot gas readers the sample is heated according to an EHF. In order to analyse complex glow-curves in hot gas readers into their individual glow-peaks, expressions describing a single glow-peak under an EHF are needed. Such expressions are those given by equations (1), (3) and (4). These expressions were used for the glow-curve deconvolution of the complex glow-curves of LiF : Mg,Ti and $Li_2B_4O_7$: Mn,Si.

The GCD of LiF:Mg,Ti was performed on samples which were readout (i) without post-irradiation annealing and (ii) with post-irradiation annealing, in order to remove the lower temperature glow-peaks. The FOM values obtained in all the cases were well below 1%, which indicates a very good fit. The values obtained for the trapping parameters of glow-peaks 4 and 5 are in excellent agreement with the previously published reference values [11].

In the case of $Li_2B_4O_7$: Mn,Si the GCD procedure was performed in two steps. In the first step the GCD

was performed by aiming for the best possible fit with the lowest possible number of individual glow-peaks. In the second step the GCD analysis was performed using values of activation energies obtained from the initial rise and T_{stop}–T_m methods [15].

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