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UNCERTAINTY OF THERMOLUMINESCENCE ANALYSIS: A NEW CRITERION TO ASSESS THE DECONVOLUTION PROCESS

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Abstract

For decades, the deconvolution analysis of the thermoluminescence glow curve has been assessed using the figure of merit (FOM). In the present study, it has been shown that the FOM is not sufficient to assess the deconvolution analysis of TL glow curves. An alternative criterion based on the uncertainty of the deconvolution analysis has been proposed. A comparison between the proposed criterion and FOM was conducted using theoretical simulations and experimental results. It has been shown that the developed criterion can provide detailed information about the fitting quality for each region in the glow curve as well as give an overall assessment of the deconvolution process. The uncertainty of the deconvolution analysis using the general-order kinetics has been estimated for various glow curves. The TL-SDA toolkit has been updated to include the feature of evaluating the uncertainty of the deconvolution process (TLSDA_v2 - File Exchange - MATLAB Central).

Keywords: Evaluation of Uncertainty, Thermoluminescence, Glow Curve Analysis.

1. Introduction

Thermoluminescence (TL) is a phenomenon of luminescence emitted from insulators or semiconductor materials when subjected to thermal stimulation. The TL phenomenon can be explained in the light of the energy band theory of solids as illustrated by Fig. 1.

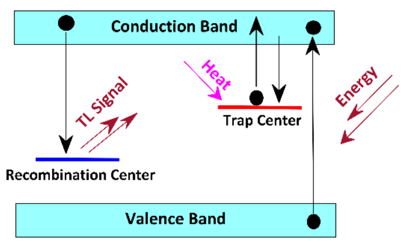

Fig. 1. Energy band gap model showing the electronic transition in a TL material.

The TL emission is a result of a TL material subjected to irradiation process and thermal excitation. During the irradiation, the electrons in the valence band are excited by the radiation energy to the conduction band. The free electrons in the conduction band have the probability to be trapped by a site of crystalline imperfection called trapping state [1]. If the thermal excitation is sufficient, the trapped electrons are released to the conduction band where they

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have a probability to recombine with a hole at some sites, called recombination state [2]. When the trapped electrons are recombined with luminescence centre, TL signals are emitted [3].

The TL emission versus the excitation temperature or time is called a TL glow curve which usually consists of some peaks. Each peak represents a trap type with a defined trap depth called activation energy E. The area under the glow curve is proportional to the concentration of the electrons trapped during the irradiation process [1]. From this point, it was proposed to use TL materials for radiation dosimetric purposes [4].

Prior to using a TL detector, it must be subjected to a calibration process in which the TL responses are calibrated to radiation doses. A linear TL dose-response is preferred in dosimetric applications. However, several factors including the background noise signals, scattering data, and nonlinear signals included in the TL glow curve can affect the accuracy of the TL measurements [5, 6]. Therefore, experimental techniques [7], theoretical approaches and artificial intelligence technology [8] were developed to improve the TL dose measurements. However, the deconvolution analysis of TL glow curves is one of several techniques proposed to separate the dosimetric peak from the redundant signals [9]. Indeed, various dosimetric applications are based on TL technique [10-12]. Furthermore, some application demonstrate analysing the TL spectrum to its individual components [13-17]. On the other hand, the deconvolution analysis was also used to estimate the characteristics of the TL detectors [17-24].

Unfortunately, the deconvolution analysis is tricky because of the great diversity of the TL glow curves [26]. The complex structure glow curve may have different deconvolution analysis solutions [27]. Furthermore, no criteria can clearly identify the optimum solution. However, it is often to assess the deconvolution analysis of TL glow curve using the *Figure of Merit* (FOM) [28] which is defined as:

$$FOM(\%) = \sum_i \frac{|I_i(Experimental)-I(Fit)|}{A} \times 100. \quad (1)$$

The FOM is based on comparing the summation of absolute differences between the experimental results and model estimations normalized to the area under the curve A. A general criterion demonstrates a $FOM < 2.5\ \%$ for satisfactory fitting [28]. While a threshold of $FOM < 5.0\%$ was set by Horowitz and Yossian [9] who concluded that the FOM threshold should consider the number of analyzed glow peaks. In other words, a threshold of 5% for a glow curve deconvolution of multiple peaks is less satisfactory than that for a glow curve deconvolution of a single peak.

The low values of the FOM do not necessarily imply that model could interpretate the data. In fact, the representation of the model to the data is always suspected if information regarding the uncertainties of the model's parameters and their effect on the model output is unavailable [29].

In TL science, there are mainly three models developed to describe the TL glow peak. The first- and second-order kinetics models [30, 31] could describe the TL glow peak as a function of the initial concentration of trapped electrons n_0, the activation energy E, and the frequency factor s. While the general- and mixed-order kinetic models [32, 33] used additional parameters, namely the kinetics order b and mixed-order α, respectively. Later, Kitis *et al.* [34] deduced the first, second-, and general-order kinetics model equations by replacing the parameters n_0 and s with the peak maximum I_m and peak maximum position T_m which can be obtained from the experimental glow curve.

Various software applications were developed to deconvolute the TL glow curves. A list of these software applications was provided by Peng *et al.* [35], who developed a software application to analyze the TL spectrum using various models. Recently, Sadek *et al.* [36] developed the TLSDA toolkit that can run using the MATLAB. The advantage of this application is that it can deconvolute complex structure glow curves without the need to perform

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several trials. Furthermore, there is no need to prior knowledge of the number of TL peaks or the activation energy. Nevertheless, none of these software applications provide an uncertainty for the fitting model used in the deconvolution analysis process. Therefore, the main aims of the present work are:

i. Develop a new criterion to assess the deconvolution analysis of TL glow curve based on the uncertainty of the TL model.
ii. Compare the new criterion with the default FOM.
iii. Release a new version of TLSDA toolkit [36] to include the evaluation of uncertainty of the TL deconvolution analysis process.

2. Methodology of evaluation of uncertainty

The evaluation of uncertainty of the fitting model used in the deconvolution analysis process was performed following the *Joint Committee for Guides in Metrology* (JCGM) guide [37, 38]. The JCGM methodology is based on both Bayesian probabilistic [39] and classical probabilistic methods [40]. Assuming that the error is propagated over the output system, the method evaluates the uncertainty associated with each source of error affecting the output system. Then, these sources of uncertainty are combined into a single value.

In least square problems, the uncertainty of the output model is a combination of the uncertainty components associated with the model's parameters [41, 42]. In TL, the *general order kinetics* (GOK) model equation describes the TL signal assuming a single glow peak by a mathematical representation of 5 parameters as [43]:

$$I(E,s,n_0,b|T) = n_0 s\, e^{-\frac{E}{\mathrm{k}T}} \left[\frac{s(b-1)}{\beta}\, F(T,E) + 1\right]^{-\frac{b}{b-1}} \tag{2}$$

where:

$$F(T,E) = \int_{T_0}^{T} \exp\left(-\frac{E}{\mathrm{k}T}\right)\, dT. \tag{3}$$

It implies that the uncertainty of the model output is a combination of the uncertainty components associated with $n_0, E, s, b,$ and β. The combined standard uncertainty associated can be estimated by [37]:

$$u(I) = \sqrt{\sum_i (v_i^I u_i)^2 + 2\sum_i \sum_j c_i c_j u_i u_j r(i,j)} \tag{4}$$

where v_i is the sensitivity coefficient that represents the impact of the uncertainty component i on the final measured quantity [44, 45], u_i is the associated standard uncertainty, and $r(i,j)$ is the correlation coefficient between the uncertainty components i and j, which is in sometime a crucial factor [46]. The sensitivity coefficient associated with the uncertainty components are evaluated as [37]:

$$v(n_0) = \frac{\partial I}{\partial n_0} = s\, e^{-\frac{E}{\mathrm{k}T}} \left[\frac{s(b-1)}{\beta}\, F(T,E) + 1\right]^{-\frac{b}{b-1}}, \tag{5}$$

$$v(E) = \sigma_2 \left\{\sigma_1 + Tbs\, \left(Ei\left[-\frac{E}{\mathrm{k}T}\right] - Ei\left[-\frac{E}{\mathrm{k}T_0}\right]\right)\right\}, \tag{6}$$

$$v(s) = \frac{n_0 \beta e^{-\frac{E}{\mathrm{k}T}}}{\sigma_1^2} \left(\beta - s\, F(T,E)\right) \left(\frac{\sigma_1}{\beta}\right)^{\frac{-1}{b-1}}, \tag{7}$$

$$v(b) = n_0 s\, e^{-\frac{E}{kT}} \left(\ln\left(\frac{\sigma_1}{\beta}\right) \frac{\left(\frac{1}{b-1} - \frac{b}{(b-1)^2}\right)}{\left(\frac{\sigma_1}{\beta}\right)^{\frac{b}{b-1}}} + \frac{bsF(T,E)}{\beta(b-1)\left(\frac{\sigma_1}{\beta}\right)^{1+\left(\frac{b}{b-1}\right)}} \right), \tag{8}$$

$$\sigma_1 = \beta + s\,F(T,E)(1-b), \sigma_2 = -\frac{\beta n_0 s e^{-\frac{E}{kT}}}{kT\sigma_1\left(\frac{\sigma_1}{\beta}\right)^{\frac{1}{b-1}}}. \tag{9}$$

Ei is the one-argument exponential integral function. In the deconvolution of experimental glow curve, the TL expression deduced as a function of the peak maximum I_m and peak maximum position T_m by Kitis *et al.* [34] is used.

$$I(I_m, T_m, E, b\,|T) = I_m\, e^{-\frac{E}{k}\frac{TT_m}{T+T_m}} \frac{\left(\frac{b}{\sigma_3}\right)^{\frac{b}{b-1}}}{\left(\frac{Ee^{\frac{E}{kT_m}}(b-1)}{kT_m^2\sigma_3}F(T,E)+1\right)^{\frac{b}{b-1}}} \tag{10}$$

where:

$$\sigma_3 = \frac{2kT_m(b-1)}{E} + 1. \tag{11}$$

The effect of each uncertainty component on the output TL signal is illustrated in Fig. 2. It is worth noting that the uncertainty components are a function of the temperature. The uncertainty component associated with the activation energy E is dominant compared to the other sources of uncertainties. Furthermore, the parameter E is correlated with T_m through the peak maximum conditions. This correlation is accounted in the evaluation of uncertainty through the correlation coefficient $r(E, T_m)$.

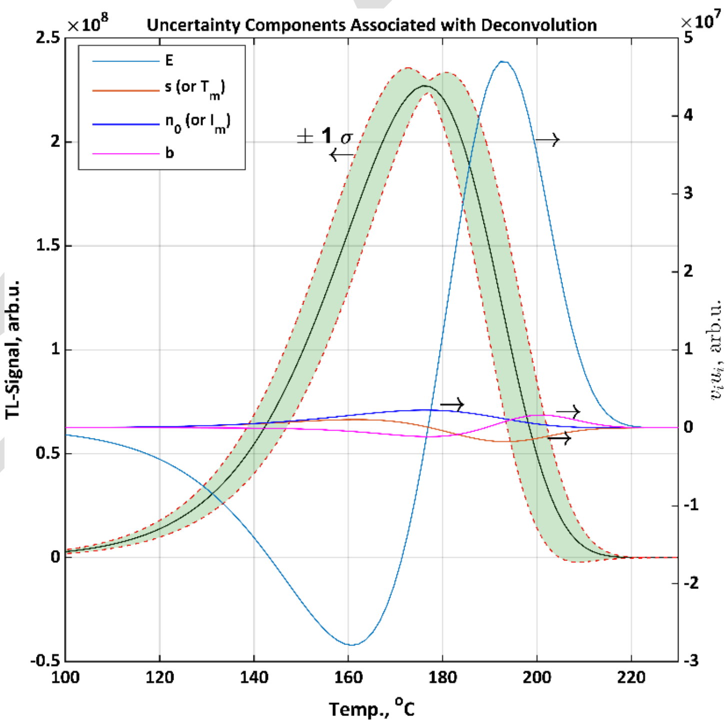

Fig. 2. Effect of each uncertainty component on final TL signal evaluated by deconvolution analysis.

The assessment of the deconvolution process demonstrates to express the combined standard uncertainty evaluated at each channel $\{T_i, I(T_i)\}$ as:

$$\mathbb{u}_{\mathbb{c}}(I), \% = \sum_i \frac{u_c\{I(T_i)\}}{I(T_i)} \times 100 \tag{12}$$

where $u_c\{I(T_i)\}$ is the combined standard uncertainty evaluated at channel i. This standard uncertainty should be investigated over the temperature range of TL glow curve to illustrate the regions of high uncertainty values, and thereby, low model's performance. On the other hand, the parameter $\mathbb{u}_{\mathbb{c}}$ describes the uncertainty of the entire deconvolution analysis process. By investigating the $\mathbb{u}_{\mathbb{c}}$ parameter, a general criterion for satisfactory deconvolution analysis can be established.

3. Factors affecting the uncertainty of deconvolution process

In the present section, the factor affecting the deconvolution analysis of TL glow curve has been investigated throughout theoretical simulations. The glow curve was simulated using the *noninteractive multiple-trap system model* (NMTS) where the electron transitions among the states are described as:

$$\frac{dn_i}{dt} = -n_i s\, e^{-\frac{E_i}{kT}} + n_c(N_i - n_i)A_i, for\ i = 1,2, ..\ \ell, \tag{13}$$

$$\frac{dn_c}{dt} = \sum_{i=1}^{\ell} \left[n_i s\, e^{-\frac{E_i}{kT}} - n_c(N_i - n_i)A_i \right] - n_c m A_m, \tag{14}$$

$$\frac{dm}{dt} = -n_c m A_m \tag{15}$$

where dn_i/dt describes the change in the electron concentrations $n(t)$ in trapped in the trapping states N_i with trapping probability coefficients A_i. The dm/dt describes, on the other hand, the change in concentration of recombination states $m(t)$ of recombination probability coefficient A_m. The dn_c/dt describes the electron transitions among the trapped and recombination states through the conduction band.

3.1. Effect of overlapping between peaks

Critical arguments were proposed that the CGCD cannot yield reliable trap parameters, and the deconvolution analysis cannot reach a global minimum for glow curves of overlapping peaks [47, 48]. Therefore, Kierstead and Levy [49] reported that the CGCD is reliable if the glow peaks are well separated. Unfortunately, the FOM does not provide information about the complexity of the TL spectrum. However, using the uncertainty criterion, information about the complexity of the TL glow curve and the region where the model could not interpretate the data can be provided.

Figure 3 presents a glow curve of four glow peaks simulated using the NMTS model. The trapping parameters were selected in such that two glow peaks overlapping with each other, and the other two peaks are separated. The FOM indicated a satisfactory fitting. The relatively high values of u_c over the temperature indicate the overlapping between peaks. However, the final $\mathbb{u}_c$ (%) may still indicate an acceptable overall deconvolution analysis.

The experimental data of the GLOCANIN project [50] included glow curves of LiF:Mg,Ti detectors subjected to various experimental conditions. It should be noted that in addition to precision, there are several Type-B uncertainty sources affect the TL emission including the TL calibration curve, batch homogeneity, radiation source, TL reader stability, and fading correction. The evaluations of these sources were addressed by Sadek et al [51] and found to be in the level of 4.5% [1σ] for LiF:Mg,Ti detectors and Harshaw 3500 TL system. This level of uncertainty may vary depending on the type of the TL detector used and the TL reader system.

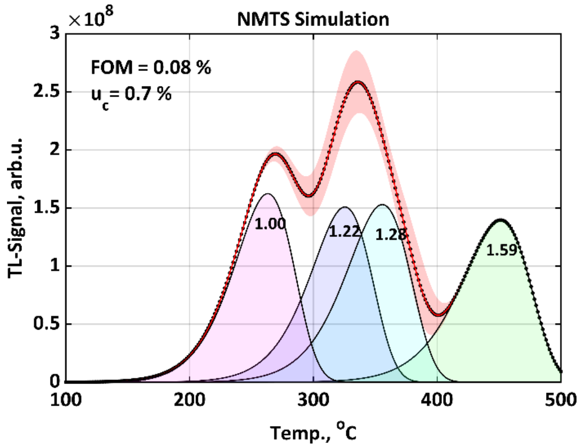

Fig. 3. The deconvolution performed for the glow curve simulated using NMTS model with overlapping peaks. The activation energy obtained by the deconvolution analysis, in unit of eV, is denoted on the maximum of each peak. The quality of the fitting was assessed using default FOM and new $\mathbb{u}_{\mathbb{c}}$ criteria.

For dosimetric applications, where the peak maximum or peak integral estimated from the deconvolution analysis is used, Type-B uncertainty should be considered in the evaluation of the combined standard uncertainty to ensure a reasonable uncertainty assessment. In fact, accounting for Type-B uncertainty enhances the clarity and reliability of uncertainty interpretation with the deconvolution process, providing a comprehensive framework for its implementation in dosimetric applications.

The RefGC#09 represents a glow curve of LiF:Mg,Ti detector irradiated with high Gamma dose levels. However, the deconvolution analysis of the high-temperature glow peaks of this curve is still a challenge because they overlap with each other. The glow curves of LiF:Mg,Ti irradiated by heavy ions are more complicated than the glow curve of LiF:Mg,Ti irradiated with high doses [9, 52]. Therefore, in the present section these glow curves were analyzed, and the analysis quality was estimated. Fig. 4 presents the deconvolution analysis of RefGC#09 and LiF:Mg,Ti alpha irradiation glow curve [27].

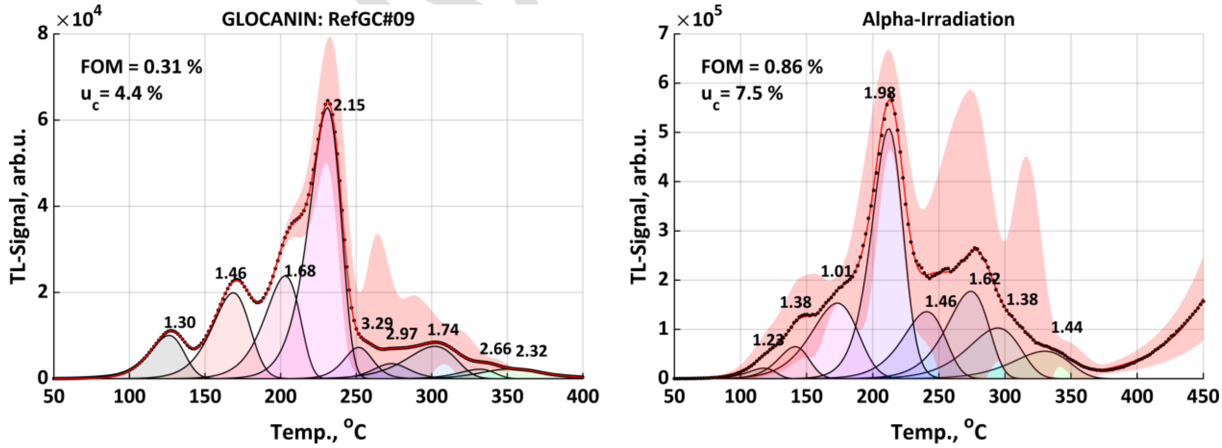

Fig. 4. The deconvolution analysis of the GLOCANIN glow curves #09 and glow-curve of LiF:Mg,Ti irradiated with Alpha particles. The activation energy obtained from the deconvolution analysis, in unit of eV, is denoted on the maximum of each peak. The deconvolution analysis was assessment using the FOM and $\boldsymbol{u_c}$ criteria.

The FOM in both cases of Fig. 4 indicated a satisfactory fit. While high $\mathbb{u}_{\mathbb{c}}$ values were obtained. By investigating the u_c over the temperature range of TL glow curve, one finds that the deconvolution analysis of the high temperature part $T > 250$ ℃ of RefGC#09 suffers from higher uncertainty values. For the Alpha-irradiation case, high uncertainty values were obtained

Metrol. Meas. Syst., Vol. 32 (2025), No. 3
DOI: 10.24425/mms.2025.154675

for the model all over the temperature range. These high uncertainty values are due to the complex structure of the glow curve. It implies that the deconvolution analysis would have many other possible solutions.

The above discussion reveals that the FOM does not provide information about the complexity of TL spectrum. Furthermore, it may provide unrealistic assessment for the deconvolution of overlapping glow peaks. On the other hand, it shows the importance of investigating the model performance over the temperature range of the TL spectrum along with the final uncertainty criterion $\mathbb{u}_{\mathbb{C}}$.

3.1. Effect of data size

The TLD reader systems record the temperature and the corresponding TL intensity over a predefined channel number. In Harshaw 4500 and 3500 TLD reader systems, the temperature and TL intensity are recorded over 200 channels regardless of the temperature profile settings. However, some other TLD reader systems can record the TL glow curve over 1000 channels [2].

Typically, increasing the channels size should improve the fitting quality and increase the reliability of the model's prediction [53]. To investigate the effect of the channels size on the fitting model performance, glow peaks were simulated with different channel sizes using the NMTS model. In each case, the peak was fitted by the GOK expression and the fitting quality parameters were estimated. In TLSDA software [36], the fitting quality was assessed through the FOM, root mean square of error (RMSE), and R − Sqaure, where:

$$R - Square = 1 - \frac{SSE}{SST}, \tag{16}$$

$$SSE = \sum_{i=1}^{Chn}\left(y_i - y(x_i)\right)^2, SST = \sum_{i=1}^{Chn}(y_i - \bar{y})^2, \tag{17}$$

$$RMSE = \sqrt{\frac{SSE}{v}} \tag{18}$$

where Chn is the channel size, and v is the degree of freedom $v = Chn - 1$. Fig. 5 presents the goodness-of-fit parameters of fitting a TL glow peak simulated by the NMTS model with different channel sizes.

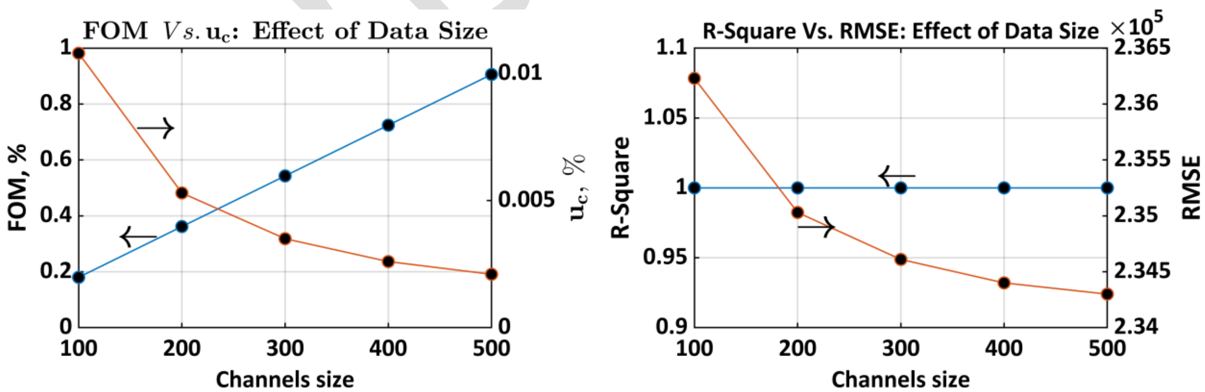

Fig. 5. Comparison between evaluation of goodness of fitting using FOM Vs uncertainty over various channel sizes.

High FOM values were obtained when the channel numbers increased. These high FOM values are attributed to the term of $\sum_i I(experimental) - I(Fit)$ in FOM expression which increases with increasing the channel size. On the other hand, the $\mathbb{u}_{\mathbb{C}}$ criterion showed that the performance of the fitting model was improved as the data size increased. This improvement in the model's performance was also confirmed by the fitting quality parameters. The $RMSE$

parameter is similar to the FOM, except it evaluates the average of the square of the model's error instead of normalizing to the curve area. Therefore, it eliminates the effects of changing the channel size. The $R - Square$ also eliminates the effect of the channel size by the ratio of SSE/SST which takes the average $\bar{y}$ into account.

The Lexsyg Smart TL/OSL reader system can record TL glow curves with different channel sizes by varying the heating rates over the same temperature range. A set of TL glow curves of $GdAlO_3$ detectors exposed to 13.2 Gy beta irradiation were recorded with different channel sizes. The deconvolution analyses of these curves are illustrated in Fig. 6.

It has been observed that at large channel sizes, the uncertainty of the fitted curve is minimum. However, as the channel size decreases, the uncertainty of the fitted curve increases, especially in the temperature region where the glow peaks overlap with each other. On contrast, the FOM increases with increasing the channel size as illustrated in Fig. 7.

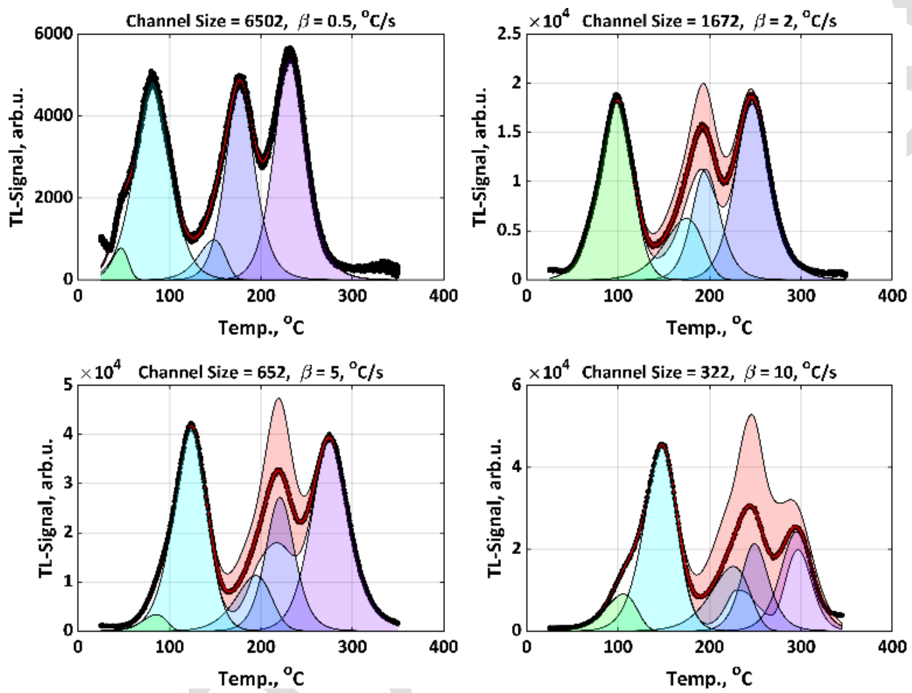

Fig. 6. The deconvolution analysis of a set of glow curves recorded with different channel sizes.

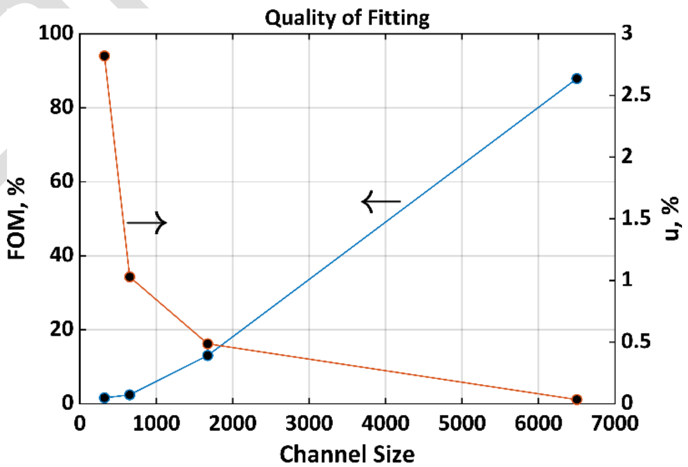

Fig. 7. The FOM and uncertainty of fitting curve over the channel size of the temperature readout.

It has been observed the FOM is very sensitive to the channel size of the temperature readout. In fact, the FOM may reach an unacceptable level because of the large channel size. This illustrates the advantage of using the uncertainty of the fitted curve as a goodness of fitting quality parameter instead of the FOM in these cases. It implies that the FOM may provide unreasonable assessment for the deconvolution analysis of TL spectrums that were recorded with large channel size.

3.2. Effect of scatter data

The effect of scatter data on the evaluation of TL dose-response curve was previously investigated [5, 6]. In these studies, the scatter data was simulated following the Monte-Carlo algorithm [38] where a random error $\mathcal{E}$ was induced to each data point of the TL intensity. In other words, for a data point x, the scattering effect can be induced as:

$$\xi = x + a(x)z\,, \mathcal{E} = \xi - x \qquad (19)$$

where $a(x)$ is adjustable standard error and z is standard normal distribution of mean 0 and standard deviation 1. In the present study, $a(x)$ was introduced as a fraction of the peak maximum intensity. Fig. 8 illustrates the scatter data effect on the fitting quality and uncertainty estimation of a single glow peak.

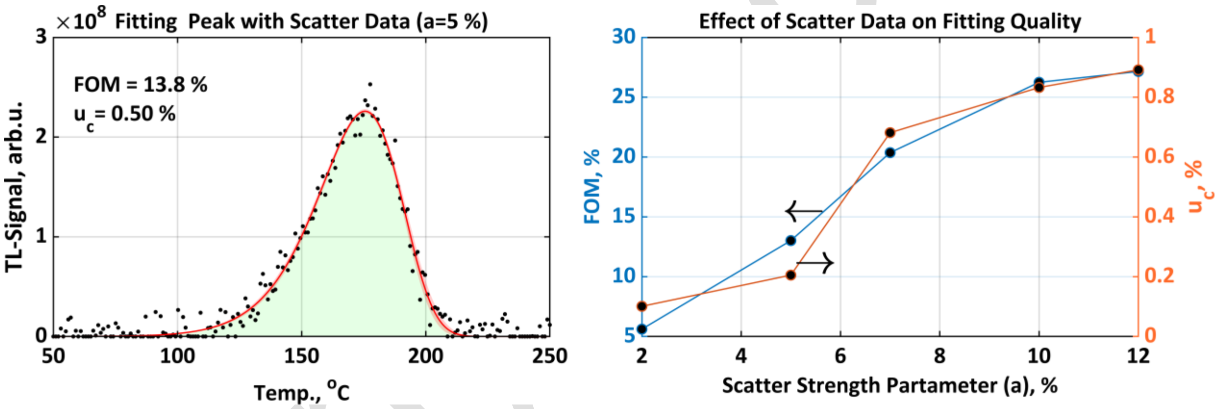

Fig. 8. Effect of scatter data on quality of fitting and model's uncertainty estimations.

At $a = 5\%$, a high $FOM = 13.8\,\%$ value indicating unsatisfactory fitting was obtained. The high FOM values are due to the high dispersion of the scatter data appearing at the low- and high-temperature tails of peak. On the other hand, $u_c = 0.5\%$ was obtained referring to a successful fit. This successful fit is because the entire range of the single glow peak was fitted by the model, and therefore, it was able to determine the trapping parameters with an acceptable precision. Nevertheless, this may not be the case with experimental data.

Scatter data is usually observed in case of low dose levels. The RefGC#10 of GLOCANIN project represents the glow curve of LiF:Mg,Ti detector irradiated by 0.2 mGy. The deconvolution analysis of RefGC#10 is presented in Fig. 9.

High FOM and u_c values were estimated for the RefGC#10. These high values are attributed to the scatter data in the TL signal. It is worth noting that the uncertainty u_c over the temperature range of glow curve increases in the area where the peaks are overlapping to each other. This is because in these regions, the uncertainty of TL signals is affected by both the scatter data and the overlapping effect. On the other hand, it reveals that the overlapping between peaks is dominant compared to the scatter data.

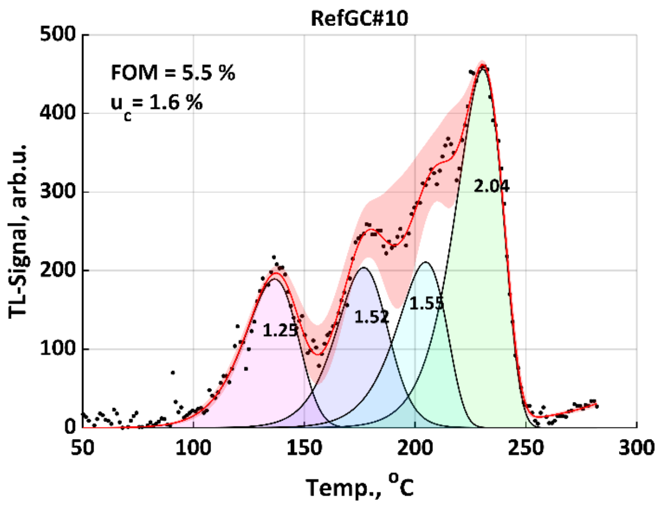

Fig. 9. The deconvolution analysis of GLOCANIN RefGC#10. The activation energy obtained from the deconvolution analysis, in unit of eV, is denoted on the maximum of each peak. The deconvolution analysis was assessed using the FOM and $\boldsymbol{u_c}$ criteria.

4. Conclusions

Critical drawbacks have been observed when using the FOM criterion to assess the deconvolution analysis of TL glow curves. The FOM may not provide reasonable assessment for the deconvolution analysis of TL glow curves when the glow peaks are overlapping with each other. Furthermore, it provides a misleading assessment for the deconvolution analysis of glow curves recorded with large channel size. These drawbacks could be recovered with the proposed assessment criterion which is based on the uncertainty of the deconvolution process.

The developed uncertainty criterion can provide detailed information about the performance of the fitting model at each region in the glow curve. In this way, using the TL signals in regions where the uncertainty is high can be subjected to further investigation or used with cautions. On the other hand, it can also provide an overall assessment for the deconvolution process of TL glow curve.

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