



# On the pre-dose sensitization of the various components of the LM-OSL signal of annealed quartz; comparison with the case of 110 °C TL peak

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## ABSTRACT

Pre-dose sensitization of various components of LM-OSL signal of a Nigerian annealed quartz sample has been investigated along side with that of 110 °C TL peak in this work. Successive cycles of irradiations and TL/OSL readings using different heating rates were employed to attain pre-dose sensitization. The results showed that the pre-dose sensitization factor of 110 °C TL peak depends strongly on the heating rate of thermal activation. The pre-dose sensitization of 110 °C TL and all components of RT LM-OSL yield HR dependence on the sensitization after TA. This dependence was ascribed to the different heating time associated with each HR. Sensitization of LM-OSL measured at 125 °C generally does not show dependence on HR of TA. This was with the exception of components C<sub>1</sub> and C<sub>3</sub>. Increasing sensitization pattern with increasing HR suggests a correlation between the TL glow-peak at 110 °C, the component C<sub>4</sub> of RT LM-OSL and the component C<sub>3</sub> of the LM-OSL signal at 125 °C. Extension of the present investigation to diverse quartz kinds from different origins was suggested in order to study the prevalence of the pre-dose sensitization on component C<sub>4</sub> of RT LM-OSL. Finally fast heating is suggested for lower sensitization of fast component while applying OSL dating protocols.

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## 1. Introduction

The pre-dose sensitization effect in the 110 °C thermoluminescence (TL) glow-peak of quartz is a result of a combined action of pre-exposure dose and thermal activation (TA) (Zimmerman, 1971). Investigation devoted to separating the individual contributions of each component of pre-dose effect (namely pre-exposure dose and TA) in quartz is scanty in the literature (Koul et al., 2010). While attempting on correlating pre-dose sensitization of TL and optically stimulated luminescence (OSL) in general, Koul and Chougankar (2007) have recently performed a characterization of the luminescence emission of the fast component of the continuous wave optically stimulated luminescence (CW-OSL) signal with pre-dose treatment and concluded that it, like 110 °C TL peak, is sustained by the pre-dose mechanism in the pre-dose effective domain. This finding provides with another argument, besides their same emission spectral region, toward the fact that both TL and OSL share

the same luminescence (L) centers. Kiyak et al. (2008) have performed a component resolved study on the thermally activated characteristics (TAC) of the linearly modulated optically stimulated luminescence (LM-OSL) signals of seven quartz samples from different origin relative to the TAC of their respective TL glow-peaks at 110 °C. They have concluded that the TACs of all individual LM-OSL components of each kind of quartz follow qualitatively the TAC behavior of the respective TL glow-peak at 110 °C. Even though it is now widely established that the two processes share the same luminescence centers, Jain et al. (2003) argued that the pre-dose relationship between 110 °C TL peak and the fast component of OSL may not be valid for other components of the OSL signal.

Furthermore, Kiyak et al. (2008) have also concluded that their fourth component of the room temperature (RT) LM-OSL curve, is closely related with the TL glow-peak at 110 °C. Similar results were also previously reported by Bøtter-Jensen et al. (1999) for their second RT LM-OSL component, as being associated with the 110 °C TL trap. Polymeris et al. (2009) identified a similar second component (C<sub>2</sub>) of the RT LM-OSL signal of their unheated quartz to have similar dose response curves in their studies. Consequent on these findings, Polymeris et al. (2009) suggested the possibility of extrapolating the popular pre-dose method of dating of 110 °C TL

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peak to the RT LM-OSL. However, that proposed possibility cannot stand unless the two features possess identical sensitization patterns. Thus, a parallel sensitization study of 110 °C TL peak and all components of LM-OSL is essential. Such an undertaking will be useful in providing valuable information while addressing the issue of sensitivity changes of quartz that is known to be problematic in luminescence dating (Murray and Roberts, 1998).

Therefore, the purpose of the present work is threefold: (a) to perform a component resolved study toward the contribution of the TA in case of LM-OSL pre-dose sensitization signals measured at RT and 125 °C, (b) to study dependence of pre-dose sensitization of these two luminescence phenomena on the heating rate (HR hereafter) of TA process, and (c) to examine any possible correlation for the sensitizations between 110 °C TL peak and each individual LM-OSL component, investigating thus the claim of Jain et al. (2003).

## 2. Materials and methods

One quartz sample collected from Oro, in Kwara State from Nigeria was analyzed in this work. This was given laboratory name S2. Only annealed quartz was studied for the present study. Following annealing of the natural quartz at 900 °C for 1 h and subsequent fast cooling in the air, grains of dimensions 90–150 µm were obtained, after smashing in an agate mortar and sieving. All luminescence measurements were carried out using a RISØ TL/OSL reader (model TL/OSL-DA-15) equipped with a 0.075 Gy/s  $^{90}\text{Sr}/^{90}\text{Y}$  β ray source (Bøtter-Jensen et al., 2000). The reader was fitted with a 9635QA photomultiplier tube. The detection optics consisted of a 7.5 mm Hoya U-340 ( $\lambda_p \sim 340$  nm, FWHM 80 nm) filter. All measurements were performed in a nitrogen atmosphere. In case of LM-OSL measurements, the ramping rate was 0.04 mW/cm<sup>2</sup>/s, increasing the stimulation light power from zero up to the maximum power (40 mW/cm<sup>2</sup>) over a period of 1000 s.

Successive cycles of irradiations and TL/OSL readings using different HR were employed to attain pre-dose sensitization. In the present study, TA was performed by a TL measurement up to 500 °C. In order to distinguish the contribution of TA from pre-exposure dose, different HRs were employed for the TA TL reading. The experimental protocols were divided into three parts. The sequence of the first part, Part A, for TL is tabulated in Table 1. Part B in Table 2 for RT LM-OSL is the counterpart of its TL Part A. Finally, Part C is identical to Part B, except that the LM-OSL was always preceded by pre-heat to 180 °C and OSL measurements were performed at 125 °C instead of RT. TD was 1 Gy.

The LM-OSL curves were analyzed through a Computer Glow-curve De-convolution Analysis (CGDA) using a general order kinetics expression proposed of Bulur (1996) and later modified by Kitis and Pagonis (2008) onto an expression containing only the peak maximum intensity  $I_m$  and the corresponding time  $t_m$ . These two variables can be extracted directly from the experimental OSL curves. The modified expression used in our computerized procedure is:

$$I(t) = I_m \frac{t}{t_m} \left( \frac{b-1}{2b} \left( \frac{t}{t_m} \right)^2 + \frac{b+1}{2b} \right)^{\frac{b}{1-b}}$$

where  $b$  is the order of kinetics. This latter expression was used providing thus the best test of the first order model assumption correctness for the quartz LM-OSL, since the values of  $b$  were corrected to be 1.0001.

The background signal was simulated by an equation of the form

$$BKG_{LM} = \alpha + ct$$

where  $\alpha$  is the average in the first few seconds of a zero dose LM-OSL measurement, and  $c$  is a constant.

**Table 1**  
Experimental protocol for successive cycle measurements for TL part.

Step	Description	Significance
1	Give test dose (TD) to a subsample and TL measurement up to 180 °C at heating rate of HR <sub>i</sub>	TD serves as regeneration dose and TL is meant for mass normalization/sensitivity monitoring (Table 1 in Murray and Roberts, 1998)
2	Give TD	For pre-exposure dose
3	TL measurement up to 500 °C at heating rate of HR <sub>i</sub>	Measures the thermally unsensitized TL (termed as Sn0) and as thermal activation to 500 °C for the next TL of step 4
4	Repeat steps 2 & 3 four times more	Step 4 measures four (4) sensitized successive cycles TL namely; Sn1, Sn2, Sn3, and Sn4 respectively. Each TL also serves as thermal activation to 500 °C for the next successive cycle TL
5	Give TD	Serves as a pre-exposure dose for step 6
6	TL measurement up to 500 °C at heating rate of 2 °C/s	Monitors influence of varied HRs of TA on each aliquot. Note this step 6 is always read at 2 °C/s heating rate
7	Repeat steps 1–6 for a new aliquot and new HR <sub>i</sub> (Take $i = 0.25, 0.5, 1, 2, 5, \text{ \& } 10$ °C/s)	Meant to observe the effect of TL reading thermal activation resulting from different heating rates

The non-zero intensity value at  $t = 0$  in RT LM-OSL was considered in the CGDA analysis in order to accomplish best fit. This value is not observed in LM-OSL received at 125 °C. Hence, the unwanted initial signal in this work is attributed to the signal arising from the decay of extremely shallow traps that decay in RT. Since these traps decay in RT and do not require optical stimulation, the signal is eliminated by pre-heat to 180 °C and elevated LM-OSL measurement at 125 °C in this work. Therefore, adopting the suggestion of Kitis et al. (2010) that this non-zero intensity value could be attributed to the presence of phosphorescence, a first order phosphorescence component of the form,  $I(t) = I_0 \exp[-\lambda \cdot t]$ , was included in the CGDA analysis. The decay rate,  $\lambda$ , was included as a fitting parameter.

All curve fittings were performed using the software package Microsoft Excel, with the Solver utility (Afouxenidis et al., in press),

**Table 2**  
Experimental protocol for successive cycle measurements for OSL part.

Step	Description	Significance
1	Give test dose (TD) to a subsample and TL measurement up to 180 °C at heating rate of HR <sub>i</sub>	TD serves as regeneration dose and TL meant for mass normalization/sensitivity monitoring
2	Give TD	For pre-exposure dose
3	OSL at room temperature (RT) for 1000 s	Measures the unsensitized RT LM-OSL (termed as Sn0)
4	TL measurement up to 500 °C at heating rate of HR <sub>i</sub>	Measures residual TL and acts as thermal activation to 500 °C for the next cycles
5	Repeat steps 2–4 four times more	Step 5 measures four (4) sensitized successive cycles RT LM-OSL namely; Sn1, Sn2, Sn3, and Sn4 respectively. Each TL also serves as thermal activation to 500 °C for the next successive cycle TL
6	Repeat steps 1–6 for a new aliquot and new HR <sub>i</sub> (Take $i = 0.25, 0.5, 1, 2, 5, \text{ \& } 10$ °C/s)	Meant to observe the effect of TL reading thermal activation resulting from different heating rates

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