



COMPONENT RESOLVED IR BLEACHING STUDY OF THE BLUE LM-OSL SIGNAL OF VARIOUS QUARTZ SAMPLES

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Abstract: The present work provides an initial component resolved analysis concerning the effect of infra-red (IR) exposure at elevated temperatures on the blue LM-OSL signal of quartz (stimulated at 470 nm). The study was performed on a total of seven quartz samples, among which five originated from Turkey, one from Greece and one synthetic quartz sample. For these quartz samples, the presence of 6 or even 7 independent LM-OSL components was previously reported, after the application of a computerized decomposition analysis. IR bleaching of each one of these components is studied and compared to the respective signal reduction due to the same thermal treatment solely. It is clearly demonstrated that IR stimulation at temperatures above 50°C does not deplete only the fast component in most sedimentary quartz samples studied. Net depletion of fast and medium components resulting from IR exposure is sample-dependent and occurs faster as the stimulation temperature increases. Weak IR bleaching of slow components is also reported in some cases, being more effective for stimulation temperatures up to 100°C. No depletion of either the medium or the slow components was detected for stimulation temperatures above 150°C. Finally, IR does not stimulate any of the LM-OSL components in the case of the synthetic quartz sample.

Keywords: Infra-red (IR) stimulation, quartz, bleaching, decomposition, component – resolved analysis, LM-OSL.

1. INTRODUCTION

The prime motivation that led to the initial trial of optical dating (Huntley *et al.*, 1985) was to achieve a better method of dating sediments than was possible with thermoluminescence (Aitken, 1985; 1998). Optically stimulated luminescence (OSL) from quartz is thought to arise from different traps, each having a different photo-ionization cross section. In dating protocols, OSL is usually measured during optical stimulation at steady stimulation power at a specific elevated temperature, resulting in a continuous wave OSL (CW-OSL) signal that decays with time but not according to a single exponential. The age is determined by using the initial part of the CW-OSL signal minus a background based on the signal level at

the end of the stimulation period. Thus, this net initial signal includes contributions from all fast, slow and medium OSL components. In some quartz samples the fast component dominates the initial CW-OSL signal. However, in others the contribution from other less light-sensitive signals can be significant (Jain *et al.*, 2005). The presence of medium and slow components in that initial OSL signal can give rise to erroneous dose estimates while using the Single Aliquot Regenerative-dose (SAR) protocol (Murray and Wintle, 2000).

Therefore, it is desirable to use a well-separated fast OSL component in equivalent dose routines. It has been shown that the fast component can be separated from other component signals by analytical or instrumental procedures, such as curve fitting and/or linearly modulated OSL (LM-OSL) (Bulur, 1996; Jain *et al.*, 2005). However, these procedures were proved to be extremely time-consuming and model dependent. Alternatively, Jain

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et al. (2005) recently demonstrated that in the temperature region between 120 and 190°C it is possible to preferentially deplete the fast component only using Infrared (IR) stimulation at 880 nm (FWHM 45 nm). They reported that IR stimulation for 1500 s (115 mW·cm⁻²) resulted in 10% depletion of the fast component at 160°C and 22% at 190°C, while no depletion of either the medium or the slow components was detected. Based on that result, Jain *et al.* (2005) suggested the application of a new approach to dose estimation for quartz using a single aliquot regenerative dose protocol involving a differential measurement of the amplitude of the fast OSL component. The differential signal is calculated from two brief OSL measurements bracketing bleaching by IR photons at 160°C. However, they have not performed deconvolution analysis; they only monitored the depletion of the LM-OSL signal at the short stimulation time region, where normally the fast component dominates.

The appearance of the first published account of possible long wavelength stimulated luminescence from quartz comes from Godfrey-Smith *et al.*, in 1988, using combined 753 and 799 nm stimulation wavelengths at room temperature. At the same year, Hütt *et al.* (1988) presented evidence of a long wavelength resonance in some types of feldspars at near infra-red wavelengths. Later on, Short and Huntley (1992) attributed the IRSL measured at ambient temperatures using diode stimulation centred on 950 nm not to quartz but to feldspar micro-inclusions. In 1994, Spooner showed that IR stimulation (~860 nm) induced extremely low depletion rates, producing measurable luminescence only at temperatures greater than 70°C, while in a similar study Bailey (1998) reported that IR stimulation (875 ± 80 nm) yielded significant amounts of measurable luminescence from quartz only when the sample temperature was greater than 200°C.

The present work provides an initial component resolved analysis concerning the effect of IR exposure (880 nm) at elevated temperatures on each one of the components of the blue LM-OSL signal of quartz (stimulated at 470 nm). The study was performed on a total of 7 quartz samples, among which one is a synthetic quartz sample, five originated from Turkey and one from Greece.

2. SAMPLES AND EXPERIMENTAL PROCEDURE

The samples used were six sedimentary quartz samples, namely Altinkum, Atakoy, Pendik, Sile, Patara and Chalkidiki (laboratory references ALT, ATK, PDK, SLE, PTR, and CHL respectively), along with an artificial quartz sample provided by MERCK Company (laboratory reference MERC). Samples ALT, ATK, PDK and SLE were recovered from the coastal area around Istanbul and described in some detail previously by Kiyak and Canel (2006). The PTR sample is a proposed tsunami-laid deposit from the ancient city of Patara, located on Antalya's Mediterranean coast in Turkey. The CHL sample, collected from the coast of Chalkidiki region in Northern Greece, was earlier studied by Polymeris *et al.*, 2006a. Quartz purity of samples as well as the absence of

any feldspar inclusions was verified by preliminary XRD measurements.

All measurements were performed using an automated Risø TL/OSL reader (model TL/OSL-DA-15, Bøtter-Jensen *et al.*, 2000), equipped with an internal ⁹⁰Sr/⁹⁰Y beta source (~0.1 Gy s⁻¹). Blue light emitting diodes (LEDs) (470 nm, FWHM 40 nm, ~40 mW cm⁻²) and IR LEDs (880 nm, FWHM 80 nm, ~135 mW cm⁻²) were used for stimulation. Luminescence signal detection was made using an EMI 9635QA photomultiplier tube, fitted with Hoya U-340 filters of 7.5 mm total thickness.

Two experimental protocols were used in the study (as presented in **Table 1**) in order to compare the behaviour of the samples under different experimental conditions. Our protocol is a slight variation of the protocol used by Jain *et al.* (2003), in which they used LM-IR light intensity from 0 to 230 mW cm⁻².

As presented in **Table 1** one aliquot of each sample was pre-sensitised to 650°C and immediately afterwards irradiated with 10 Gy beta dose; it was preheated to 200°C and then stimulated by LM-IR light from 0 to 130 mW cm⁻² at various temperatures T °C (25, 50, 100, 25, 150, 200 and 25°C) for 5 ks. A measurement cycle at 25°C was repeated after every two cycles to monitor sensitivity changes. A blue stimulated LM-OSL at 125°C was recorded after LM-IR measurements. In the second protocol, similar measurement sequence was used (**Table 1**), for a second aliquot that was held at the same temperatures T °C (25, 50, 100, 25, 150, 200 and 25°C) for 5 ks without IR stimulation during the 4th step; this was to ascertain the charge depletion resulting from holding the sample at elevated temperature for 5 ks during the 4th step of protocol 1. One aliquot of each sample was used for each protocol.

3. METHOD OF ANALYSIS

All LM-OSL curves obtained were analyzed by a computerized decomposition procedure assuming first-order kinetics for all possible components (Jain *et al.*, 2003; Kiyak *et al.*, 2007). The first-order kinetics assumption is based on a direct extrapolation from the experience gained from the thermoluminescence (TL) studies of the glow-curve of quartz, where no higher order kinetics glow-peaks exist. The first-order kinetics equation describing a LM-OSL peak proposed by Bulur (1996) was used. However, the original Bulur's Eq. (1), was transformed, so that instead of the parameters n_0 and σ , to contain the parameters I_m and t_m directly obtained from the experimental curves. The transformed form given by Polymeris *et al.* (2006b) is:

Table 1. Protocols used in the study.

Protocol 1 (IR Stimulation)	Protocol 2 (Thermal treatment)
1. Pre-sensitised to 650°C	1. Pre-sensitised to 650°C
2. Irradiation with 10 Gy	2. Irradiation with 10 Gy
3. PH to 200°C for 10 s	3. PH to 200°C for 10 s
4. LM-IR stimulation at T °C for 5 ks	4. PH at T °C for 5 ks
5. Blue stimulated LM-OSL at 125°C for 10 ks	5. Blue stimulated LM-OSL at 125°C for 10 ks

$$I(t) = 1.6488 \frac{I_m}{t_m} t \cdot \exp\left(-\frac{t^2}{2t_m^2}\right) \quad (3.1)$$

where:

I_m and t_m are the values of OSL intensity and time at the maximum of the LM-OSL peak, respectively, and t the stimulation time. Eq. (3.1) was used for the decomposition of all experimental data. The curve fitting was performed using the MINUIT program (James and Roos, 1977), whereas the goodness of fit was tested by the Figure Of Merit (FOM) of Balian and Eddy (1977), given by:

$$FOM = \sum_i \frac{|Y_{Exper} - Y_{Fit}|}{A} \quad (3.2)$$

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. The background was simulated by an equation of the form:

$$bg = z_d \left(C + \frac{t}{P} \right) \quad (3.3)$$

where:

z_d is the zero dose OSL signal after blue stimulation and P the total stimulation time. The parameter z_d is evaluated experimentally, by measuring the background OSL signal at non-irradiated samples. C is a constant, very close to unity. The increase of the background with time is clearly demonstrated by the pioneer LM-OSL paper by Bulur *et al.*, (2002). Furthermore, the formula 3.3 is the most common formula for the background, since it was successfully applied to all previous studies dealing with decomposition of LM-OSL curves resulting from quartz.

4. RESULTS AND DISCUSSION

Linearly modulated blue and IR stimulated luminescence curves

For most of these quartz samples, the presence of 6 or even 7 independent Blue LM-OSL components was previously reported (Kitis *et al.*, 2007; Kiyak *et al.*, 2007; 2008). LM-OSL curve shapes and decomposition analysis examples for samples ALT, ATK, PDK and SLE were previously reported by Kiyak *et al.* (2007) using six individual LM-OSL components, while for sample CHL by Kiyak *et al.*, (2008) using seven individual LM-OSL components. Therefore blue LM-OSL curve shapes analyzed and resolved into their individual components are presented in **Figs. 1** and **2** for the quartz samples PTR and MERC respectively. Six LM-OSL components were used for the decomposition of the former, while seven for the latter, which provides signal with relatively low intensity, due to the fact that it is a quartz sample of synthetic origin.

The values of the t_m used for the decomposition were chosen in the region reported previously by these preceding authors, after an extra correction according to the formula:

$$\frac{t_{m1}}{t_{m2}} = \sqrt{\frac{T_1}{T_2}} \quad (4.1)$$

where:

T_1 and T_2 are the different stimulation times. This correction is inevitable, since the stimulation time in the case of the present study is 10 ks, while in the cases of previously reported papers the stimulation time was much less. Therefore, each peak maximum position shifts to lower stimulation times as the stimulation duration increases. The FOM values were of the order of 2% (in cases of LM-OSL glow curves of low intensity) and better.

Fig. 3 presents IR LM-OSL curves for the PTR and ALT quartz samples. For each one of these, 4 curves are presented, namely IR LM-OSL signal after stimulation at 50, 100, 150 and 200°C. The LM-IR glow curves for the ALT sample are presented as typical for all other quartz samples of Turkish origin. The signal is of extremely low intensity, especially in the case of low stimulation temperatures up to 100°C. For stimulation temperatures be-

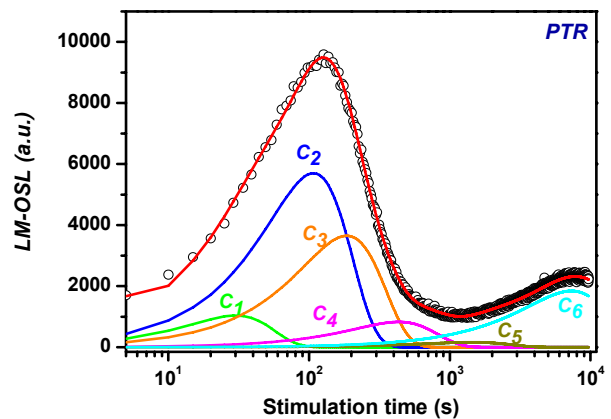


Fig. 1. LM-OSL curve of Patara quartz sample received at 125°C (lab code PTR), deconvoluted into 6 individual components. Dose 10 Gy, PH to 200°C for 10 s. The time axis is given in log scale in order to observe, clearly, all components at short stimulation times.

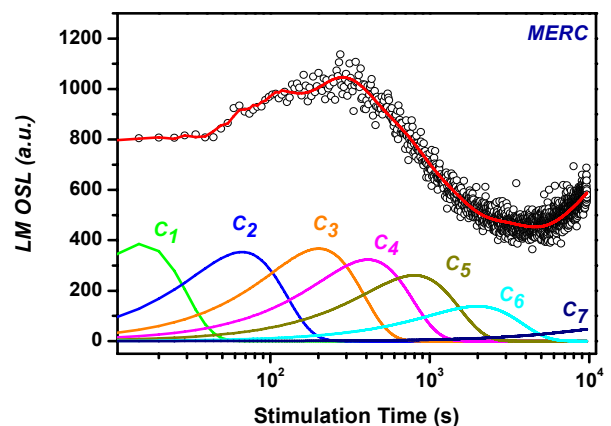


Fig. 2. Component resolution of the LM-OSL curve of the Merck synthetic quartz sample received at 125°C (lab code MERC). Six individual components were used for the deconvolution. Dose 10 Gy, PH to 200°C for 10 s. The time axis is given in log scale in order to observe, clearly, all components at short stimulation times.

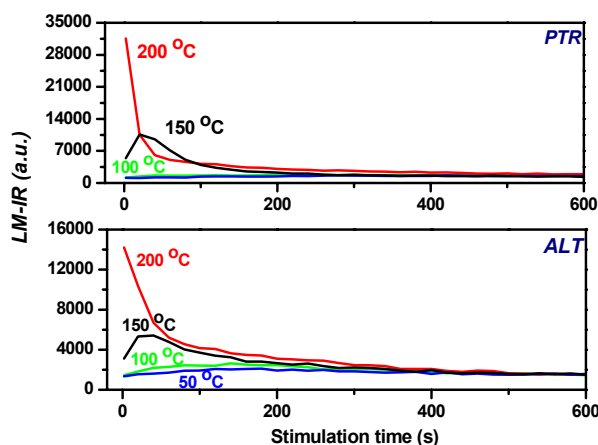


Fig. 3. IR-LM glow curves for two different quartz samples; PTR (upper figure) and ALT (lower figure). IR LM curves for four different stimulation temperatures are presented for each sample. Dose 10 Gy, PH to 200°C for 10 s.

tween 50 and 150°C, the signal forms a wide peak for short stimulation times; the maximum position is shifted towards lower stimulation times as the stimulation temperature is increased. Despite the fact that stimulating at 200°C provides a signal yielding much better statistics, its shape is dramatically changed, i.e. there is not a peak any more and the signal is monotonically depleted with time as in the case of Continuous Wave OSL (CW-OSL).

Signal-to-noise ratio is still very low, especially for long stimulation times. Therefore, the IR LM-OSL signals were not decomposed and analyzed into their individual components.

One notable exception is observed for the PTR quartz sample. For stimulation temperatures up to 100°C, the LM IR glow curves are totally flat. However, for higher stimulation temperatures, the LM IR signal is of great intensity, also forming a peak for short stimulation times at 150°C, while at 200°C not only is very intense, but it is also monotonically depleted also, as in the case of Continuous Wave OSL (CW-OSL).

Component Resolved IR bleaching

IR bleaching was carried out to investigate the depletion rates of each one of the blue LM-OSL components of quartz as a function of the stimulation temperature. The high temperature annealing during the stimulation causes significant depletion of the blue LM-OSL signal. In order to monitor the depletion due to purely IR stimulation, the second experimental protocol was applied, namely the aliquots were held at the same elevated temperatures without IR stimulation in order to study the loss of signal due to thermal decay alone.

For most of the quartz samples studied, IR stimulation at elevated temperatures bleaches the entire blue LM-OSL curve. This feature is illustrated in **Fig. 4**, where for each one of the samples studied, blue LM-OSL curves are

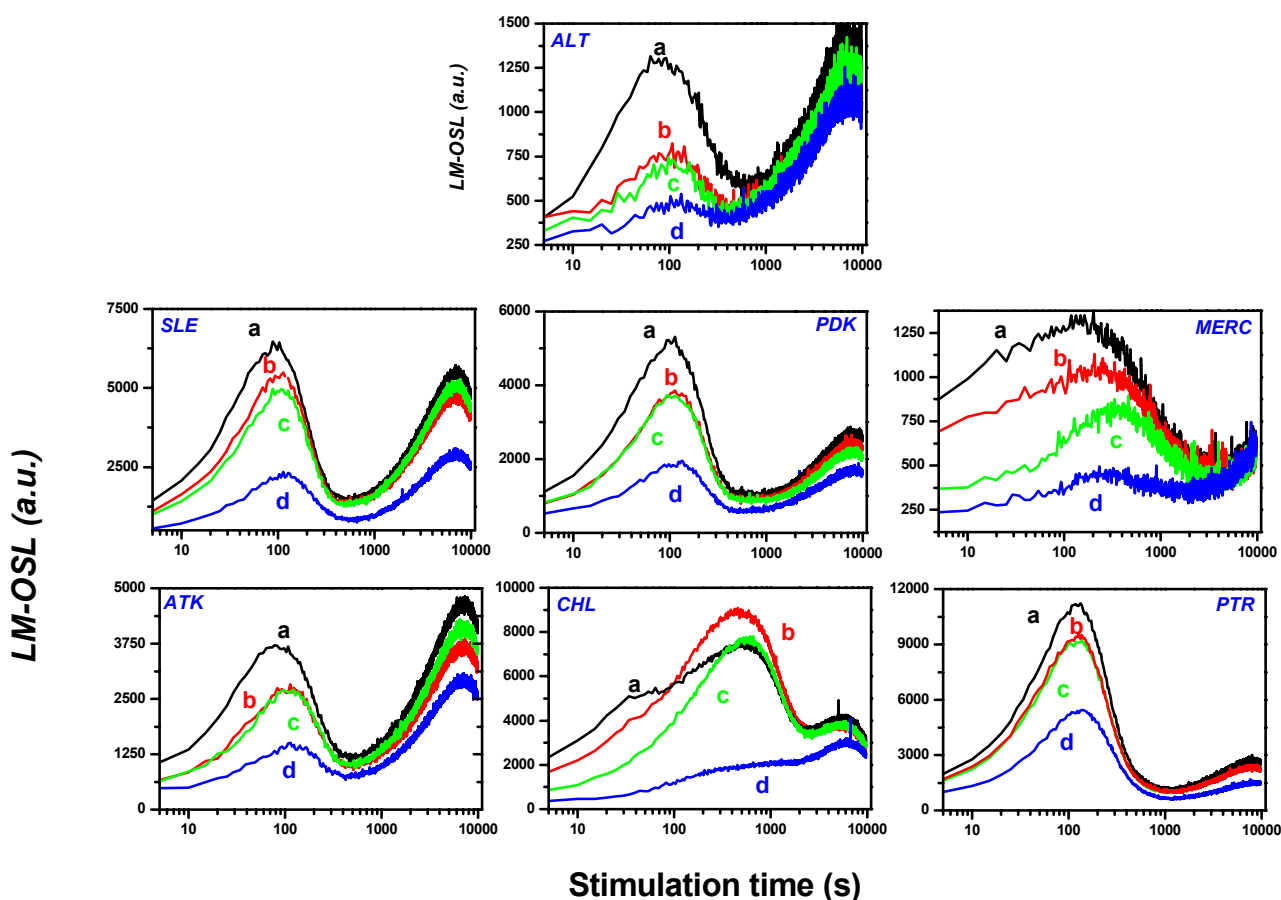


Fig. 4. Blue LM-OSL curves for all quartz samples studied, under four different conditions of IR stimulation; without IR exposure (curve a), after IR stimulation at 25°C (curve b), at 100°C (curve c), and at 200°C (curve d). The time axis is given in log scale.

presented under four different conditions of IR stimulation; without IR exposure (curve a), after IR stimulation at 25°C (curve b), at 100°C (curve c), and at 200°C (curve d). It is interesting to point out that curves in **Fig. 4** are not corrected for sensitivity changes, since the measurement cycles at 25°C, repeated after every two cycles, indicated sensitivity changes of the order of 5% per two cycles or even less. Depletion resulting only from IR stimulation is recorded for the fast components, even at the lowest stimulation temperature, implying that quartz's insensitivity to IR stimulation at room temperature firstly suggested by Short and Huntley (1992) may be sample dependent. This result is also contradictory to the conclusions suggested by Jain *et al.* (2003, 2005). These authors reported that only the quartz fast component appears to be optically stimulated by IR at temperatures between 120 and 190°C and that no depletion of either the medium or fast component was detected at the same temperature region. These results were consistent for the quartz samples studied of Indian, Danish and Australian origin. However, they have not performed deconvolution analysis; they only monitored the depletion of the LM-OSL signal at the short stimulation time region, where normally the fast component dominates.

All OSL components C_i , appear under the same names for all the samples, due to the strong correlation between individual components from different quartz samples. This correlation is strongly supported by the values of trap parameters determined by the decomposition procedure. Those parameters are the t_{\max} maximum position t_{\max} as well as the photo-ionization cross section σ . From **Figs. 1** and **2** it becomes prominent that in all quartz samples studied each component C_i appears at almost the same t_{\max} position. This is the first and most interesting correlation between several components from different samples. Furthermore, all components at the same t_{\max} position yield similar photo-ionization cross section values. Both t_{\max} (for 3600 s of stimulation) and σ values were previously reported for samples ATK, ALT, PDK and SLE by Kiyak *et al.* (2007). The aforementioned correlations between several components from different quartz samples are strongly supported by those numbers. The σ values for the other quartz (CHL, PTR and MERC) samples are adequately similar to further support this correlation. Furthermore, the photo-ionization cross section values are reproducible during the bleaching process to within $\pm 15\%$ for the first three components to less than 25% for the others, providing thus the best test of the first order model assumption correctness.

The individual behaviours of components C_1 , C_2 and C_3 are similar. Furthermore, the values of the t_m for those three components are very close. Therefore, these three components are treated together as a sum for all quartz samples under study, except the MERC quartz sample, where the components C_1 , C_2 , C_3 and C_4 are treated together for the same reasons. Depletion of components C_1 , C_2 and C_3 is presented in the lower part of **Fig. 5**, while depletion of components C_1 , C_2 , C_3 and C_4 are presented in the lower part of **Fig. 6** for the MERC quartz sample. The high temperature annealing (curves (a) of **Fig. 5**) causes moderate depletion of the blue LM-OSL signal,

except the case of the PTR quartz sample, where the signal is sensitized. IR stimulation at elevated temperatures (curves b of **Fig. 5**) further depletes the blue LM-OSL signal, not only in the temperature region between 150 and 200°C as it was previously reported and is true for the samples SLE and CHL, but even for lower stimulation temperatures. This is exactly the case for samples ALT, ATK, PDK and PTR, where the IR stimulation is effective even at the low stimulation temperature of 50°C. For the MERC quartz sample, the strong depletion of the LM-OSL is attributed only to the thermal treatment, since IR stimulation does not cause further depletion to components 1 to 4.

Medium components C_4 and C_5 are also treated together for the six sedimentary quartz samples for the same reasons. The effects of both annealing as well as IR stimulation at high temperatures are presented as curves (a) and (b) respectively in the middle panels of **Fig. 5**. While annealing in the temperature region 50-200°C causes significant depletion to these components in all quartz samples, the effect of the IR stimulation is sample-dependent, causing strong depletion in the case of the ATK sample, moderate one in the case of the ALT sample and small depletion for samples PTR and SLE. Finally, for samples MERC, CHL and PDK the IR stimulation does not deplete further the blue LM-OSL signal of the medium components. Furthermore, bleaching of the medium components is stronger in the low stimulation temperature region, namely for temperatures up to 100°C.

Explanation of the similar behaviour of the separate OSL components when the first order kinetics model is assumed becomes a difficult task. This similar behaviour of some separate OSL components could be simply attributed to the fact that first order model is not a good approximation and the signal of some components originates probably from one trap. However, this would have not been the case for sequential LM-OSL components. Nevertheless, this assumption regarding the weakness of the first order kinetics model should be presented openly, despite the fact that very good reproducibility of the photo-ionization cross section values during the IR bleaching process is a clear experimental result that strongly supports the first order model assumption correctness. Assuming the latter, similar behaviour of LM-OSL components could be possibly attributed to the use of the same recombination centre.

However, the most interesting result is the weak IR bleaching of slow components, whose depletion is presented in the upper panels of **Fig. 5**. It is clearly demonstrated that annealing at high temperatures does not cause significant loss of the signal in all quartz samples, unless the temperature is 200°C. Furthermore, it is also prominent that in some quartz samples, such as ATK, ALT, PDK and PTR, weak IR bleaching of slow components takes place; while for the three other samples (SLE, PTR and CHL) IR does not stimulate any of the slow LM-OSL components. This slow LM-OSL component IR bleaching is more effective when the stimulation occurs at the temperature region between 100 and 150°C. Finally, as in the case of the medium components, no depletion of the slow components was detected for stimulation temperatures above 150°C.

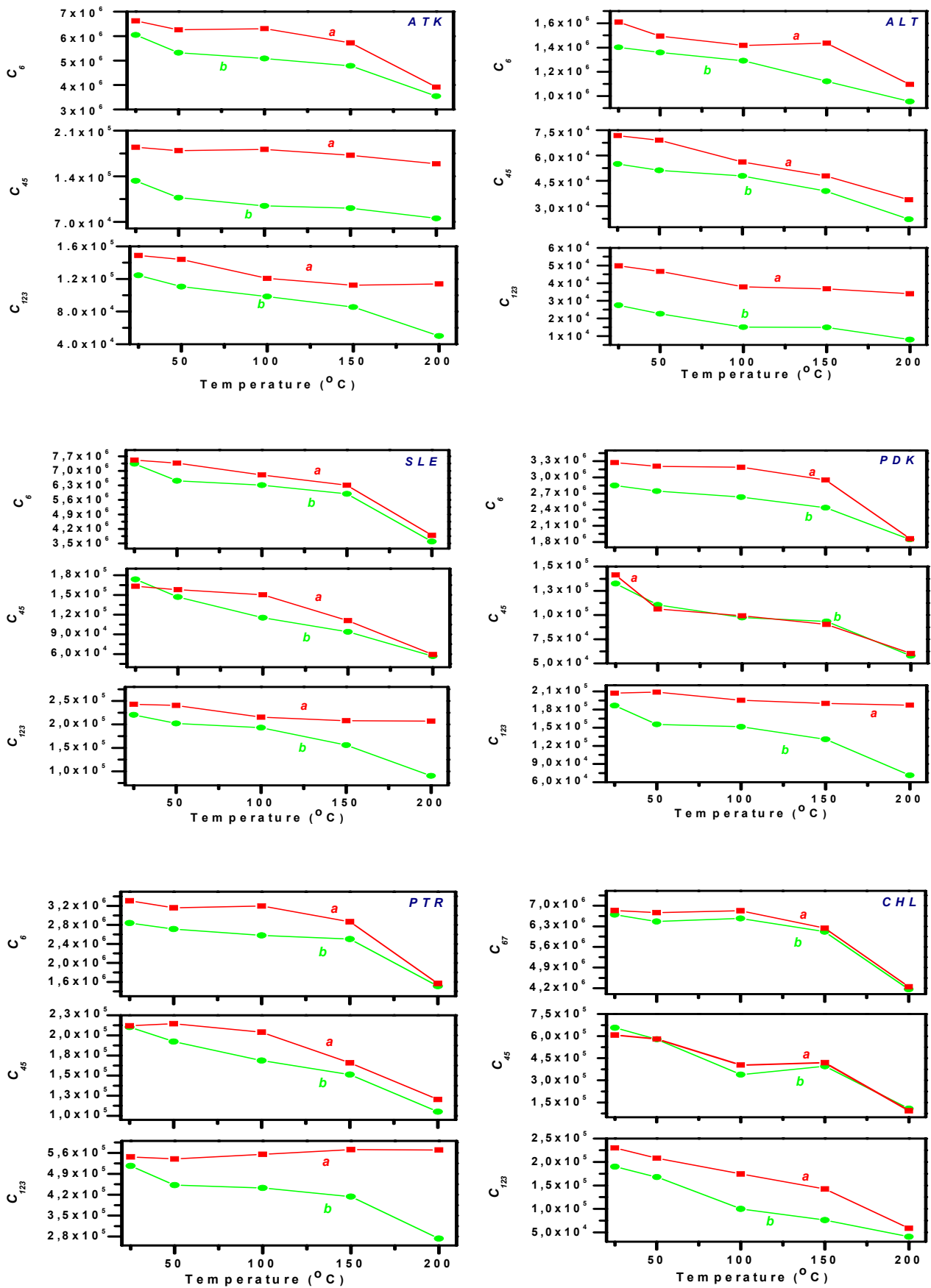


Fig. 5. Component resolved depletion curves of the blue LM-OSL signal for all sedimentary quartz samples studied, after elevated temperature annealing (curves a) and IR stimulation at elevated temperatures (curves b) as a function of the temperature applied; sum of the fast components C₁, C₂ and C₃ (lower figures), sum of medium components C₄ and C₅ (middle figures) and slow component C₆ (and C₇ in case of CHL sample, upper figures)..

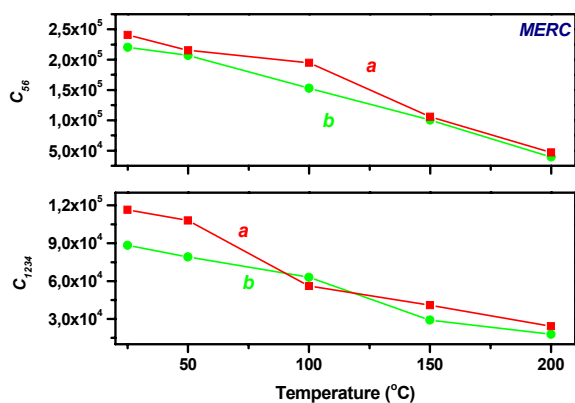


Fig. 6. Component resolved depletion curves of the blue LM-OSL signal for the MERC synthetic quartz sample, after elevated temperature annealing (curves a) and IR stimulation at elevated temperatures (curves b) as a function of the temperature applied; sum of the fast components C_1 , C_2 , C_3 and C_4 (lower figure), sum of medium components C_5 and C_6 (upper figure). IR does not stimulate any of the LM-OSL components.

6. CONCLUSIONS

IR bleaching of the blue LM-OSL components of quartz is studied and compared to the respective signal reduction due to the same thermal treatment solely for seven different quartz samples. IR LM-OSL curves are also presented. It is clearly demonstrated that IR stimulation at temperatures above 50°C does not deplete only the fast component in most sedimentary quartz samples studied. Net depletion of fast and medium components resulting from IR exposure is sample-dependent and occurs faster as the stimulation temperature gets higher. Weak IR bleaching of slow components is also reported in some cases, being more effective for stimulation temperatures in the region between 100 and 150°C. No depletion of either the medium or the slow components was detected for stimulation temperatures above 150°C. Finally, IR does not stimulate any of the LM-OSL components in the case of the synthetic quartz sample.

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