



# Investigating the contribution of recuperated TL to post-IR IRSL signals in a perthitic feldspar

X.L. Wang<sup>a,\*</sup>, A.G. Wintle<sup>b,c</sup>

<sup>a</sup> SKLLQG, Institute of Earth Environment, Chinese Academy of Sciences, Xi'an 710075, China

<sup>b</sup> Institute of Geography and Earth Sciences, Aberystwyth University, Aberystwyth SY23 3DB, UK

<sup>c</sup> McDonald Institute for Archaeological Research, University of Cambridge, Downing Street, Cambridge CB2 3ER, UK

## HIGHLIGHTS

- The measured post-IR IRSL signal in feldspar has a non-IR-stimulated component.
- This contribution is the result of isothermal decay of a recuperated TL signal.
- The recuperated TL signal is due to photo-transfer during IRSL production.
- In dating protocols, this isothermal contribution should be removed by heating.

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## ABSTRACT

Using a museum specimen of perthitic feldspar, the characteristics of post-IR IRSL production at 200 °C after different prior IR bleaching at 100 °C were investigated. It was found that the post-IR IRSL signal had an isothermal TL contribution that was unexpected following a previous preheat at 320 °C; this is the result of isothermal decay of recuperated TL peaks resulting from photo-transfer that occurred when the previous IRSL signal was measured at a lower temperature. The isothermal TL contribution to the post-IR IRSL signal depends on prior IR bleaching conditions. Since the recuperated TL signal comes from photo-transfer during IRSL production, this signal should also suffer from anomalous fading. Thus, it is suggested that this isothermal TL contribution to the measured post-IR IRSL is removed by the inclusion of an additional step, a cut-heat to 300 °C, in the post-IR IRSL dating protocol.

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## 1. Introduction

Recently it has become popular to use post-IR IRSL signals for dating potassium-rich feldspar grains from sandy sediments and the silt-sized mixed mineral component of loess (Li and Li, 2011; Buylaert et al., 2012). Measurements on very old sedimentary samples have shown that the post-IR IRSL signals are in saturation (Li and Li, 2011; Buylaert et al., 2011; Thiel et al., 2011; Thomsen et al., 2011). This implies that the measured post-IR IRSL signals suffer negligible anomalous fading and therefore can be used for dating.

Two laboratory protocols have been developed for dating sediments using a post-IR IRSL signal. In the first, an IR exposure is given at a relatively low temperature, e.g. 200 s at 50 °C, following

an initial preheat (e.g. 320 °C for 60 s) and the post-IR IRSL is then measured at an intermediate temperature (e.g. 290 °C) (Buylaert et al., 2012). In the second, it has been suggested that several 100 s IR exposures are applied, increasing the temperature of stimulation in 50 °C steps from 50 °C to 250 °C following an initial preheat at 300 °C for 10 s (Li and Li, 2011). Both these methods have been tested using samples with independent age control (Buylaert et al., 2012; Fu et al., 2012) and good agreement has been reached.

Both protocols involve a range of different thermal treatments, both with and without IR stimulation. Following the initial preheat (e.g. for 60 s at 300 °C) of an aliquot of feldspar, IR exposure at a lower temperature will result in photo-transfer of electrons into thermally emptied traps, as first reported by Duller (1995). This phenomenon has been reported by others (e.g. Murray et al., 2009; Thomsen et al., 2011; Tsukamoto et al., 2012) in their studies of the post-IR IRSL signal from a range of feldspars. The presence of such a recuperated TL peak will result in an isothermal TL signal when the aliquot is then held at a temperature that can empty those

\* Corresponding author.

E-mail address: [wxl@loess.llqg.ac.cn](mailto:wxl@loess.llqg.ac.cn) (X.L. Wang).

traps. In their study of time-resolved IRSL signals from feldspars, Morthekai et al. (2012) also considered the contribution of isothermal TL to the signal measured during the on time of each stimulation pulse.

In the current study, we wished to determine the contribution of isothermal TL to the post-IRSL signal as measured under conditions similar to those in the two protocols. We designed a series of experiments to investigate both the luminescence signals resulting from these thermal treatments and the effect of varying the IR stimulation times and power applied prior to the post-IR IRSL signal being measured. The aim was to obtain a greater understanding of the processes that occur during these post-IR IRSL measurement protocols.

## 2. Sample and experimental details

The experimental sample (IEE39) is a museum specimen of perthitic feldspar, with more than 93% alkali feldspar and less than 7% plagioclase feldspar as determined by electron microprobe analysis. A  $K_2O$  content of 12.3% was obtained by X-ray fluorescence spectroscopy and thin-section analysis under the microscope showed that the alkali feldspar component was orthoclase. The sample was crushed and 4–11  $\mu m$  grains deposited on stainless steel discs. Fine grain aliquots prepared in this way were used to reduce disc to disc variability in order that one disc could be used for each measurement.

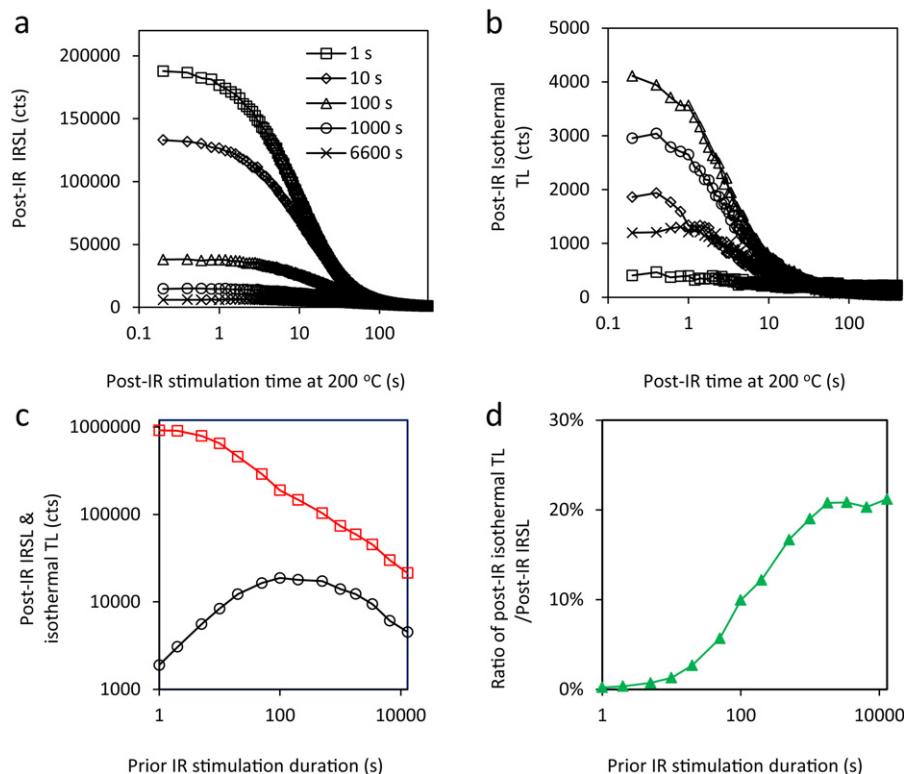
All luminescence measurements were performed using a Daybreak 2200 automated OSL reader equipped with combined blue ( $470 \pm 15$  nm) and infrared (IR) ( $880 \pm 80$  nm) LED units, and a  $^{90}Sr/^{90}Y$  beta source (delivering 0.086 Gy/s for 4–11  $\mu m$  grains

attached to stainless steel discs) for irradiation. The nominal maximum IR stimulation power is  $\sim 45$  mW/cm<sup>2</sup>, but experiments were made with 80% power, unless otherwise stated. Luminescence emissions were detected by an EMI 9235QA photomultiplier tube, in front of which were 3 mm thick Corning 7-59 and BG-39 glass filters.

The first step in each of the measurement sequences used in this paper is a TL measurement made by heating the sample to 500 °C at 5 °C/s to remove any geological IRSL signal. Following this heating, no IRSL was observed for IR stimulation at 100 °C for 500 s, and no post-IR IRSL was observed when subsequently stimulated at 200 °C. A regeneration dose of 200 Gy was then given to each aliquot for all experimental sequences to produce IRSL or post-IR IRSL signals. An initial preheat temperature of 320 °C was selected, as suggested by Murray et al. (2009) to remove charge from thermally unstable traps and used by others in post-IR IRSL dating protocols (e.g. Thiel et al., 2011; Buylaert et al., 2012). The first IR stimulation temperature was 100 °C, the lowest stable IR stimulation temperature achievable in the Daybreak reader. The post-IR IRSL was measured at 200 °C; this was the lowest stimulation temperature at which Li and Li (2011) found negligible anomalous fading. A heating rate of 5 °C/s was used to reach these temperatures.

## 3. Experimental sequences and results

To study the production of post-IR IRSL and the other feldspar luminescence signals remaining after different prior IR stimulation conditions, five experimental sequences were designed, with a different batch of fresh aliquots being used for each set of experimental conditions.



**Fig. 1.** a: Post-IR IRSL measured at 200 °C as a function of stimulation time (log scale); each decay curve relates to a different prior IR exposure (selected from the values given in the text) at 100 °C as described in the text and Sequence 1. b: Post-IR isothermal TL measured at 200 °C as a function of time (on a log scale) in step 2.5 of Sequence 2 (symbols refer to the same IR stimulation times as in a); note the data set for a prior IR stimulation time of 6600 s is second from bottom. c: Normalized post-IR IRSL and post-IR isothermal TL (integrated from 0 to 1 s taken from decay curves in a and b, respectively) plotted as a function of the duration of the prior IR exposure. d: Ratio (expressed as a percentage) of data sets given in c plotted as a function of the duration of the prior IR exposure.

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