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## THE EFFECT OF TEST DOSE AND FIRST IR STIMULATION TEMPERATURE ON post-IR IRSL MEASUREMENTS OF ROCK SLICES

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**Abstract:** Optically stimulated luminescence (OSL) is increasingly applied to the dating of rock surfaces. There is at present no practical way of separating pure minerals (quartz and feldspar) from hard rocks for OSL measurement without losing the grain-size dependent dosimetric information and there is little information about the performance of the single-aliquot regeneration-dose (SAR) measurement protocol on the post-infrared infrared stimulated luminescence (pIRIR) signals from rock slices. The latter is investigated here. Our data indicate that there is a systematic increase in dose response curve saturation (or  $D_0$ ) with test dose size when the regeneration doses are first given in increasing order, and then decreasing order. This trend disappears if these orders are reversed. The reproducibility of dose response curves is dependent on the size of the test dose (poorer for small test dose). For rock slices given a saturation dose in the laboratory, it is observed that the sensitivity corrected pIRIR<sub>290</sub> signal lies close to saturation level of the dose response curve, for first IR stimulation at temperatures between 50 and 250°C. However, the pIRIR<sub>290</sub> signal from naturally saturated slices lies close to the laboratory saturation levels only for higher first IR stimulation temperatures e.g. 200°C or 250°C. Our data confirm earlier suggestions based on sand-grain measurements that, for older samples, accurate measurements close to saturation require that a higher first IR temperature is used.

**Keywords:** luminescence dating, rock surface, post-IR IRSL (pIRIR), SAR protocol, test dose correction, first IR stimulation temperature.

### 1. INTRODUCTION

Recently, optically stimulated luminescence (OSL) has been proposed as a geochronological tool applicable to rock surfaces (e.g. Vafiadou *et al.*, 2004; Simms *et al.*,

2011; Liritzis, 2011; Sohbati *et al.*, 2011, 2012, 2015; Chapot *et al.*, 2012; Freiesleben *et al.*, 2015). OSL dating techniques have been predominantly developed for quartz and feldspar as target minerals. Although the quartz luminescence signal is more stable than that of feldspar (Aitken, 1985), it is not always possible to find samples with a useful quartz sensitivity when dating solid rocks (Sohbati *et al.*, 2011; Guralnik *et al.*, 2015). In addition, it is difficult to separate pure mineral phases in igneous rock slices without losing grain-size dependent dosimet-

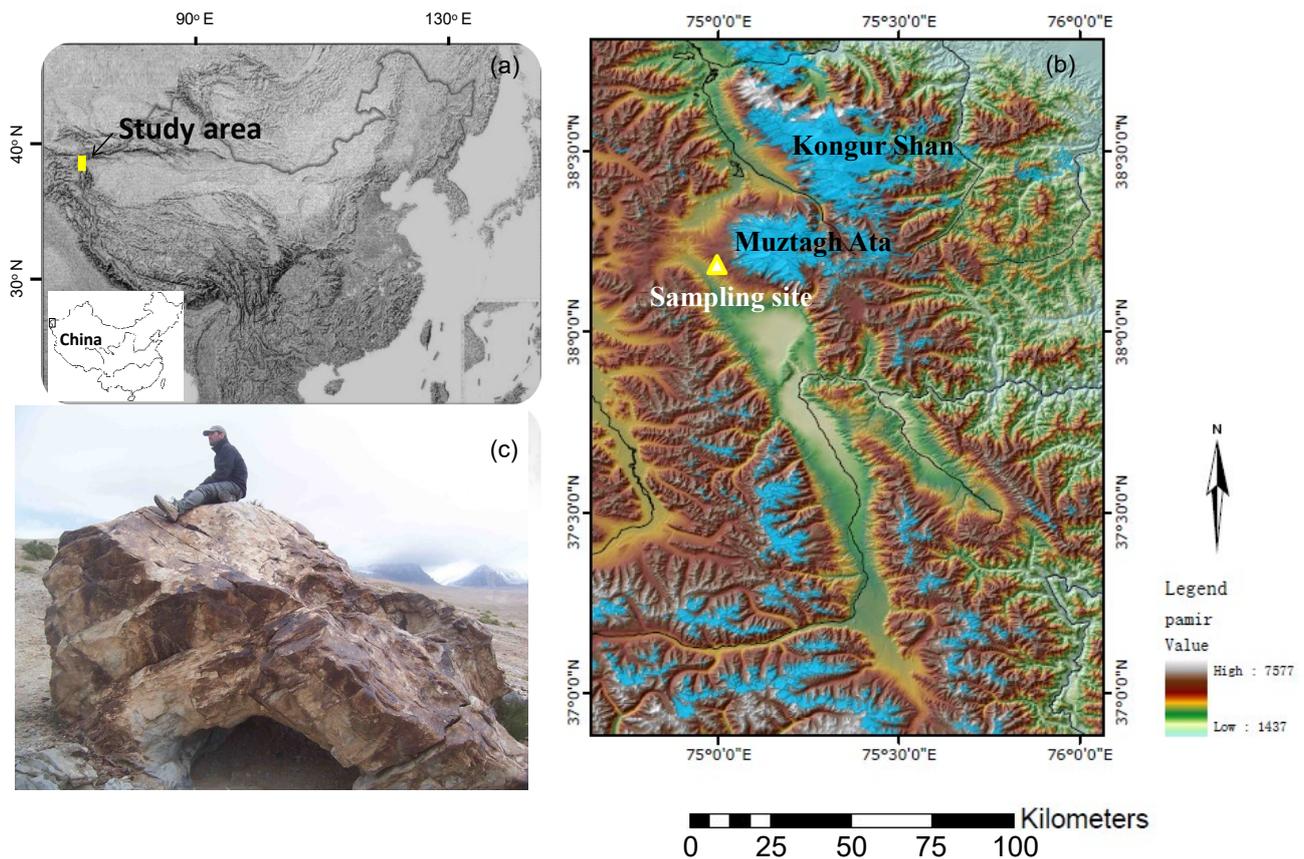
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ric information (Sohbati *et al.*, 2011); however earlier studies have shown that the infrared stimulated luminescence (IRSL) signal in blue emission band from coarse grains is mainly associated with K-rich feldspar (e.g. Baril and Huntley, 2003; Sohbati *et al.*, 2013). Unfortunately, the conventional IRSL signal suffers from anomalous or athermal fading (Wintle, 1973; Aitken, 1985; Spooner, 1994) and as a result has proved to be of limited value in dating. However, the feldspar signal derived from infrared stimulation at elevated temperature, measured after stimulation at about 50°C or higher temperature (the so-called post-IR IRSL or pIRIR; Thomsen *et al.*, 2008; Buylaert *et al.*, 2012; Li and Li, 2012a) seems to circumvent this problem and offers considerable potential for the dating of older material. Over the last few years, the pIRIR signal from feldspar has been increasingly applied to the dating of different types of sedimentary samples (e.g. Buylaert *et al.*, 2009; Li and Li, 2011; Thiel *et al.*, 2011), but there is little information on the performance of the single-aliquot regeneration-dose (SAR) measurement protocol (Murray and Wintle, 2000) on the pIRIR signal from rock slices.

The size of the test dose (e.g. Qin and Zhou, 2012; Buylaert *et al.*, 2012; Yi *et al.*, 2016) and the stimulation temperature (e.g. Thomsen *et al.*, 2008; Li and Li, 2012b) are important parameters in pIRIR dating. In this study, we investigate the effect of test-dose size and first stimulation temperature on the pIRIR<sub>290</sub> signals from rock slices, especially from those close to saturation. This is undertaken using both natural and laboratory saturated samples.

## 2. SAMPLE AND MEASUREMENT INSTRUMENT

Aliquots were taken from a glacial granite gneissic boulder (MUST88) sampled in July 2013 from the Pamir plateau, Western China (Fig. 1) previously dated to ~80 ka by Seong *et al.* (2009) using <sup>10</sup>Be. Several blocks of ~4×4×7 cm were cut from the surface of the boulder using a petrol-driven cut-off saw equipped with a diamond blade. Sample preparation and luminescence signal measurements were carried out at the Institute of Geology (China Earthquake Administration, China) and at the Nordic Laboratory for Luminescence Dating, Aarhus



**Fig. 1.** (a) Study area located at the western part of China, Pamir Plateau. (b) Samples were taken from the SW side of the Muztagh Ata massif. This fig was modified after the Fig. 1A in Owen *et al.*, (2012). (c) Photo of the glacial gneissic boulder (MUST 88) from which the sample was taken (Seong *et al.*, 2009).

University, Denmark and Center for Nuclear Technologies (DTU Nutech), Technical University of Denmark. Under subdued red-orange light, cores ~10 mm in diameter and up to 40 mm long were drilled from these blocks using a water-cooled diamond core drill; these cores were then cut into 1.2 mm thick slices using a water-cooled low-speed saw equipped with a 0.3 mm thick diamond wafer blade, giving a net slice spacing of 1.5 mm. The surface slices were treated with 10% HF for 30–40 min and 10% HCl for 20 min to remove any weathering products. No treatment was undertaken on inner slices (Sohbati *et al.*, 2011).

Rock slices were put in a carousel directly and measured on a Risø TL/OSL reader Model DA-20, using infrared stimulation (870 nm, ~130 mW/cm<sup>2</sup>) and photon detection through a Schott BG 39/Corning 7-59 filter combination (2 and 4 mm, respectively). Beta irradiation used a calibrated <sup>90</sup>Sr/<sup>90</sup>Y source mounted on the reader (Bøtter-Jensen *et al.*, 2010).

### 3. METHODS AND RESULTS

#### Luminescence characteristics of IR<sub>50</sub> and pIRIR<sub>290</sub>

The measurement protocol shown in **Table 1A** (modified after Thiel *et al.*, 2011) was used to measure the IR<sub>50</sub> and pIRIR<sub>290</sub> signals. For calculations employing a net signal, the initial 2 s of the signal minus a background evaluated from the last 10 s of the 200 s stimulation was used. **Fig. 2a** shows the natural IR<sub>50</sub> and pIRIR<sub>290</sub> decay curves for one slice from a depth of ~30 mm into the rock surface from a single core. **Fig. 2b** shows the sensitivity-corrected natural IR<sub>50</sub> and pIRIR<sub>290</sub> signal ( $L_n/T_n$ ) of slices plotted against depth, normalized to the average  $L_n/T_n$  at depths >15 mm. Negligible signal was detected up to a depth of ~8 mm for IR<sub>50</sub> and ~4 mm for pIRIR<sub>290</sub> into the

rock, because of zeroing of the signal near the surface. Based on the fitting of the simple rock-surface-exposure model (Equation 5.5 from Sohbati *et al.* 2011; the solid curves), the IR<sub>50</sub> and pIRIR<sub>290</sub> signals are in field saturation at depths >12 mm and >10 mm, respectively. The values at these depths are scattered, presumably because of variability in the  $D_0$  across the slices. However, the individual pIRIR<sub>290</sub>  $L_n/T_n$  values from these depths lie very close to the saturation level in their respective laboratory dose response curve (**Fig. 2a** inset), while the natural IR<sub>50</sub> signal (solid symbols) is ~50% of laboratory saturation.

#### Inheritance in the test dose response

At high doses, even a small residual remaining after the measurement of  $L_x$  signals could significantly affect the signals from small test doses and so have an effect on the final  $D_e$  estimate (e.g. Qin and Zhou, 2012; Nian *et al.*, 2012; Fu *et al.*, 2015). We first investigated the size of any inherited signal arising due to thermal transfer and/or insufficient zeroing during measurement of the  $L_x$  signals, using the sequence outlined in **Table 1B**. After measurement of the natural signal from various slices and thermo-optically bleaching (IRSL for 200 s at 325°C) any remaining signal, a high regeneration (4400 Gy) dose was given to each slice, which were then measured according to **Table 1B**. **Fig. 3** shows the ratio of the inherited IR<sub>50</sub> and pIRIR signals (steps 1–5, 1–6, **Table 1B**) to the corresponding initial signals (steps 1–3, 1–4) plotted against preheat temperature. For the IR<sub>50</sub> signal this ratio is consistently ~0.2% except at the highest two preheat temperatures (>360°C) where this ratio begins to increase to a maximum of ~0.5% at 400°C. In contrast, the inherited pIRIR signal decreases with temperature from ~2% at 260°C to ~0.6% at 360°C. Assuming that the dose re-

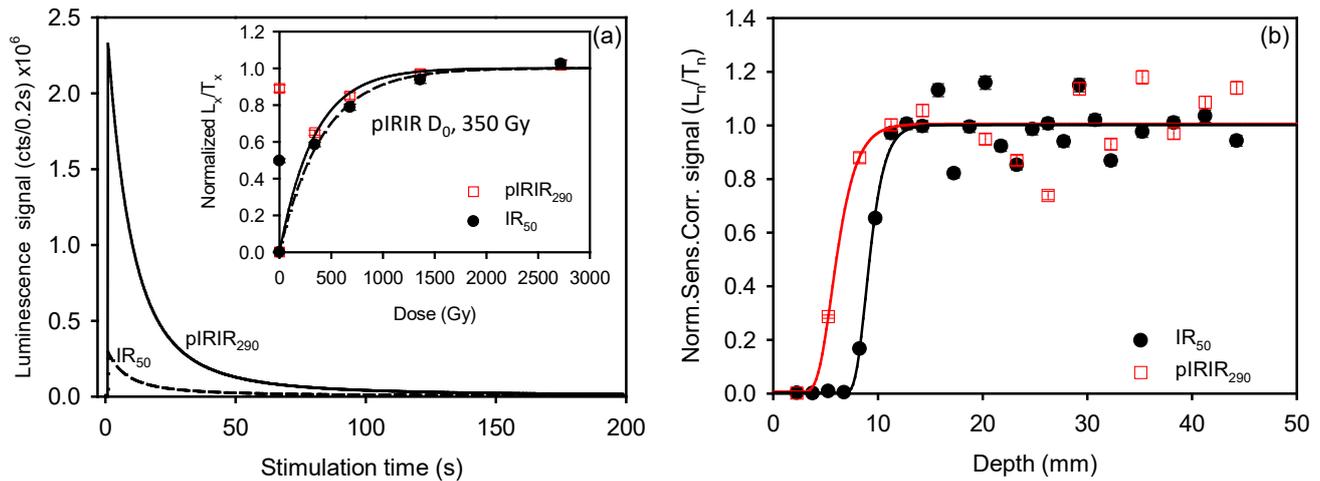
**Table 1.** The post-IR IRSL SAR protocols used in this study

Step	A	B	C <sup>3</sup>
1-1	Natural or Regeneration dose	Dose (4400 Gy)	Natural, Natural+2688 Gy, bleached+2688 Gy or Regeneration dose
1-2	Preheat at 320°C for 100 s	Preheat at T°C for 100 s <sup>1</sup>	Preheat at 320°C for 100 s
1-3	IR at 50°C for 200 s ( $L_{x,50}$ )	IR at 50°C for 200 s ( $L_{sat,50}$ )	IR at T °C for 200 s <sup>2</sup>
1-4	IR at 290°C for 200 s ( $L_{x,290}$ )	IR at (T-30)°C for 200 s ( $L_{sat,T-30}$ )	IR at 290°C for 200 s ( $L_n$ or $L_{sat}$ )
1-5		IR at 50°C for 200 s ( $L_{inherited,50}$ )	
1-6		IR at (T-30)°C for 200 s ( $L_{inherited,T-30}$ )	
2-1	Test dose		Test dose
2-2	Preheat at 320°C for 100 s		Preheat at 320°C for 100 s
2-3	IR at 50°C for 200 s ( $T_{x,50}$ )		IR at T °C for 200 s
2-4	IR at 290°C for 200 s ( $T_{x,290}$ )		IR at 290°C for 200 s ( $T_n$ or $T_{sat}$ )
3-1	IR at 325°C for 200 s (Thermo-optical wash)		IR at 325°C for 200 s (Thermo-optical wash)

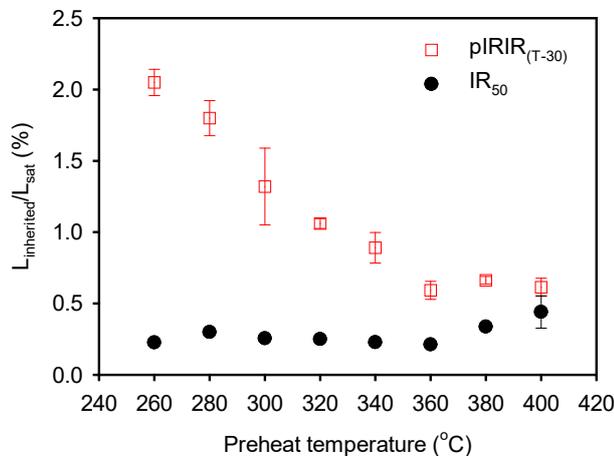
<sup>1</sup>T ranges from 260 to 400°C in steps of 20°C.

<sup>2</sup>T is 50, 200 and 250°C

<sup>3</sup>In experiment C,  $L_n$  is the signal resulting from the natural dose, the natural+2688 Gy or bleached+2688 Gy, as appropriate.  $T_n$  is the corresponding test dose signal



**Fig. 2.** (a) Typical decay curves and (inset) dose response curves of IR<sub>50</sub> (dashed line) and pIRIR<sub>290</sub> (solid line) signals. (b) The variation of the IR<sub>50</sub> and pIRIR<sub>290</sub> “residual signals” with depth into the rock surface.



**Fig. 3.** The ratio of the “residual signal” (steps 1-5, 1-6, Table 1B) to the initial signal (steps 1-3, 1-4) at different preheat and stimulation temperatures. Three slices were measured at each temperature. The error bars represent one standard error.

sponse curve follows an exponential growth, an inherited signal of 2% corresponds to an inherited dose of ~7 Gy (using the D<sub>0</sub> of 350 Gy from Fig. 2 inset). This implies that for samples recording a large dose resulting in signals at or close to saturation, any test dose should be significantly larger than 7 Gy. This is discussed further below (next section).

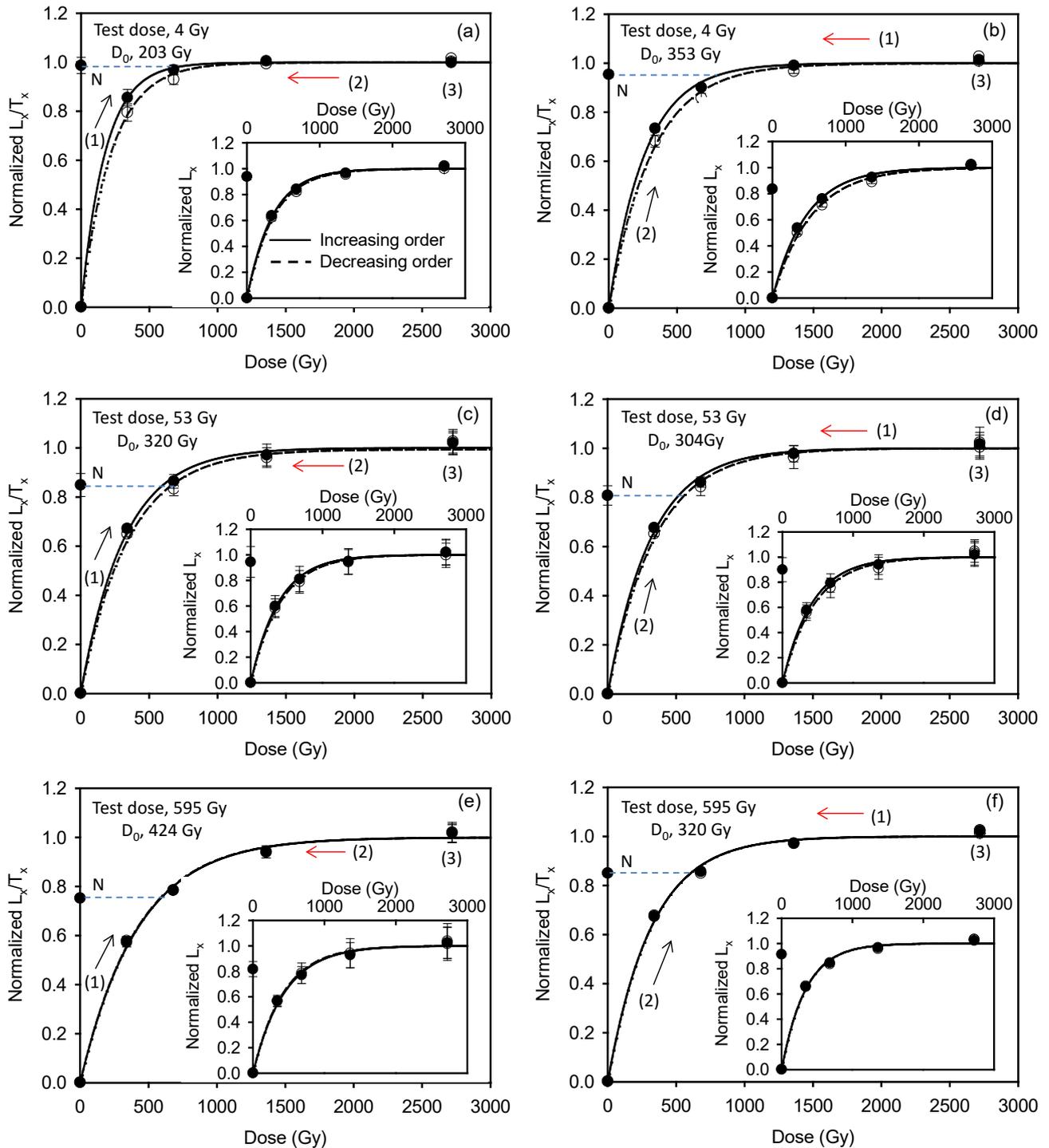
### Measuring rock slices in saturation: effect of test dose size and the order of regeneration dose

Six sets of experiments were undertaken to test the influence of test dose on the pIRIR<sub>290</sub> dose response curves and the accuracy of the measured equivalent doses; these involve changing the order in which the regeneration doses were given (Tso and Li, 1994; Timar-Gabor and

Wintle, 2013), and the size of the test dose. Three saturated slices from the inner part of rock core (depth >15 mm) were used for each set of experiment (i.e. 18 naturally saturated slices in total). The L<sub>x</sub>/T<sub>x</sub> results are shown in Fig. 4, normalized to the saturation value of their corresponding fitted dose response curve (single saturating exponential function). The insets show the L<sub>x</sub> data alone without test dose normalization. In Fig. 4a, 4c, 4e, after the measurement of the natural signal, the regeneration doses were (1) given in increasing order, then (2) repeated, but in decreasing order, and finally in step (3) the highest dose (~2700 Gy) was repeated again. In the second group of experiments (Fig. 4b, 4d, 4f), the regeneration doses were (1) first given in decreasing order and then (2) in increasing order before (3) the highest dose was re-measured. In Fig. 4a and 4b, the test dose was only 4 Gy. These experiments were repeated with a larger test dose of 53 Gy to give Fig. 4c and 4d, and a much larger test dose of 595 Gy to give Fig. 4e and 4f.

Comparison of the shapes of the dose response curves suggests that in the first group of experiments (regeneration doses first given in increasing order) the value of D<sub>0</sub> increases with the size of the test dose (Fig. 4a, 4c, 4e). However, there is no such correlation in the second group where the regeneration doses were initially given in a decreasing order; here D<sub>0</sub> seems to be independent of the test dose size (Fig. 4b, 4d, 4f). For clarity all the dose response curves are summarized in Figs. 5a and 5b. Although the potential natural variation in the D<sub>0</sub> values of individual slices as implied from the luminescence-depth profile (see Fig. 2b) may preclude any conclusive interpretation of these data, this observation is nevertheless noteworthy because each dose response curve is an average of three curves measured from three different naturally saturated slices.

A further comparison of the sensitivity-corrected dose response curves constructed by giving doses in increasing



**Fig. 4.** Laboratory dose response curves for different test doses, where the regeneration doses are given in increasing or decreasing order. For (a) and (b) the test dose is 4 Gy, (c) and (d) ~53 Gy, and (e) and (f) ~595 Gy. The solid and dashed lines represent doses given in increasing and decreasing order, respectively. The numbers (1), (2) and (3) show the order in which the regeneration doses were given. Three slices were measured at each point. The insets show the dose responses of the sensitivity uncorrected signal ( $L_x$ ). All data were normalized to the saturation value of their corresponding fitted dose-response curve. The  $D_0$  values corresponding to the solid  $L_x/T_x$  curves, based on a single saturating exponential fit, are presented in the individual figures.

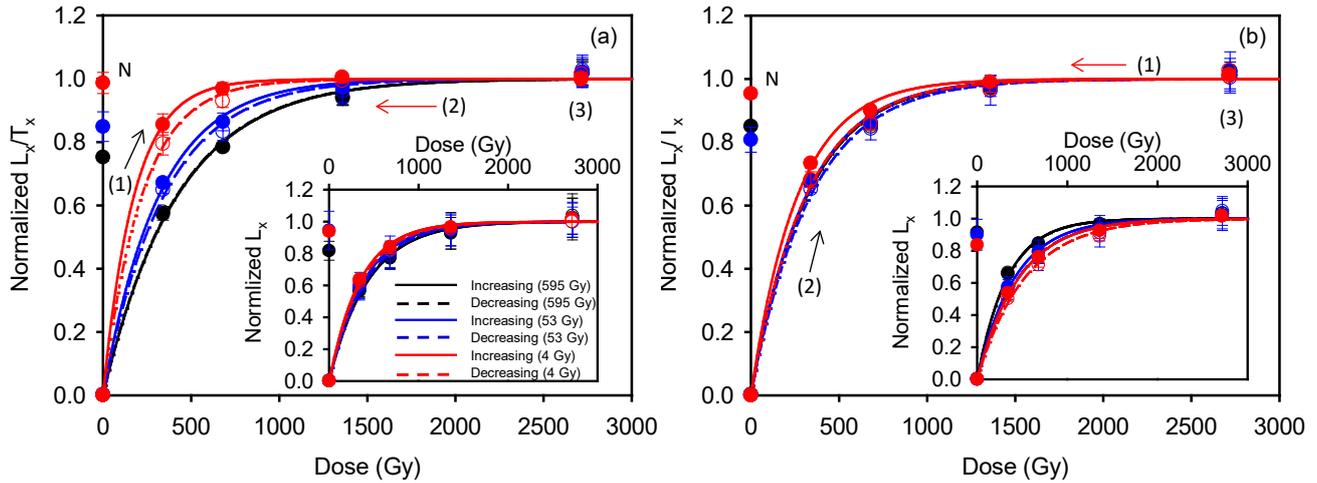


Fig. 5. a) Summary of data in Fig. 4a, 4c and 4e. b) Summary of data in Fig. 4b, 4d and 4f.

and decreasing order (in the same experiment) indicates that their reproducibility improves as the test dose increases (Fig. 4a–f; note the discrepancies between the solid and dashed curves). The reproducibility is poorer when a small test dose (4 Gy) is used to measure dose response curves up to saturation, and independent of the order in which regeneration doses are given (Fig. 4a and 4b). However, it becomes increasingly better when the test dose size is increased (Fig. 4c, 4d, 4e, 4f). In Fig. 4e, 4f, with the largest test dose (595 Gy), the solid and dashed curves overlap.

Although the natural signal appears to be closer to saturation in the dose response curves that have not been corrected for sensitivity change (Fig. 4 insets), this is likely to be an artefact of sensitivity change; Fig. 6 shows that the test-dose response drops by ~10% between the natural and the first regeneration SAR cycle. When the

$L_x/T_x$  data are examined (Fig. 4), the natural signal lies 1% (Fig. 4a) and 5% (Fig. 4b) below laboratory saturation for the 4 Gy test dose, 15 (Fig. 4c) and 19% (Fig. 4d) below for the 53 Gy test dose, and 25 (Fig. 4e) and 15% (Fig. 4f) below for the 595 Gy test dose. The apparent saturation (or close to saturation) of the natural signal for the 4 Gy test dose is possibly a reflection of the inheritance effect discussed above.

#### Effect of first IR stimulation temperature

In this experiment, three groups of slices were measured, where the first IR stimulation temperature was 50°C, 200°C or 250°C (Fig. 7, Table 1C). The purpose of this experiment is to see which prior treatment gives the most reliable dose estimate. Group (1) consisted of naturally saturated slices from the inner part of the rock

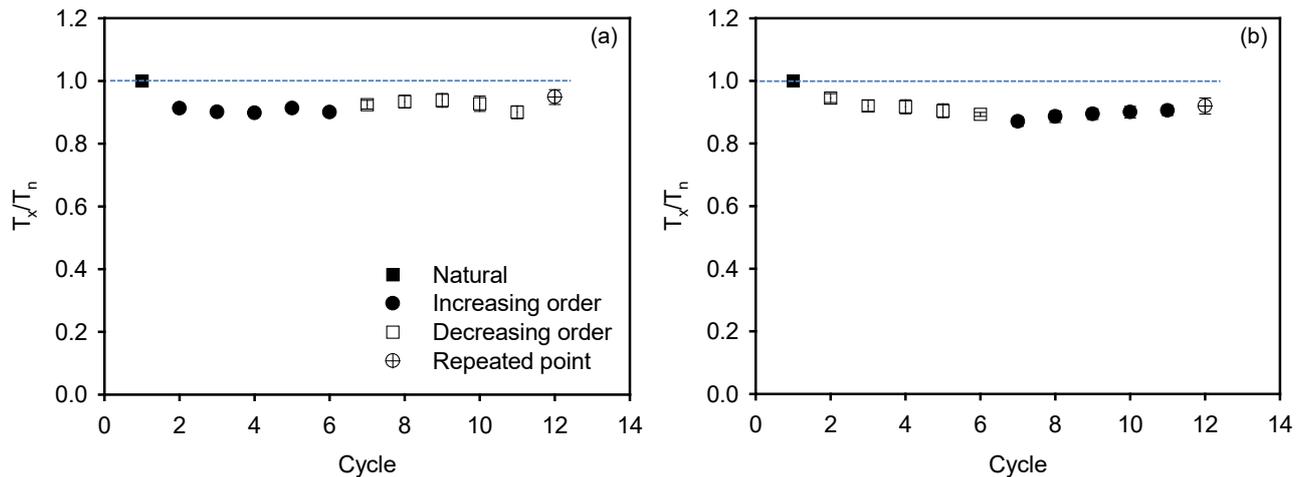


Fig. 6. Sensitivity change monitored by a large test dose (595 Gy). (a) Increasing regeneration doses followed by decreasing doses. (b) Decreasing regeneration doses followed by increasing doses. Three slices were used at each point.

(depth >15 mm). Group (2) contained naturally saturated slices from the inner part of the rock to which a high dose of 2688 Gy had been added. Group (3) contained naturally bleached slices from the surface of the rock to which a high dose of 2688 Gy was added. All these samples were then measured and “ $L_n/T_n$ ” (first cycle measurements resulting from natural, natural+2688 Gy, or bleached+2688 Gy, as appropriate) were derived. Subsequently, a regenerated dose cycle with 2688 Gy was undertaken using all these slices; this cycle gave an estimate of the laboratory saturation signal  $L_{sat}$  and its corresponding test dose response  $T_{sat}$ . The ratios of the “ $L_n$ ” to  $L_{sat}$  and “ $L_n/T_n$ ” to  $L_{sat}/T_{sat}$  are shown as a function of first stimulation temperatures in Fig. 7, for two sizes of test dose, 53 Gy (Fig. 7a, 7c) and 595 Gy (Fig. 7b, 7d). These data are similar to those from a dose recovery test except that we are considering the reproducibility of light levels (luminescence intensities) rather than doses.

Consider first Fig. 7a, 7b where the uncorrected absolute light level ratios ( $L_n/L_{sat}$ ) are presented. In both figures, the natural signals ( $L_n$ ) are within 10% of the regenerated  $L_{sat}$  signals, suggesting that the natural light level is indistinguishable from the regenerated saturated light level, as was also indicated in Fig. 4 insets. However the ratios of  $L_{n+2688}/L_{sat}$  and  $L_{bleached+2688}/L_{sat}$  are all >1 (unfilled diamond and unfilled square symbols in Fig. 7a, 7b); this confirms that the apparent saturation of the natural signal is an artefact of the change in sensitivity during the measurement of the first SAR cycle (see Fig. 6 and discussion above). Fig. 7c, 7d present the sensitivity corrected ratios ( $L_n/T_n$ ). Here most of the  $L_{n+2688}/T_{n+2688}$  and  $L_{bleached+2688}/T_{bleached+2688}$  signals are consistent with the corrected regenerated signals ( $L_{sat}/T_{sat}$ ) at  $2\sigma$ , indicating a good recovery ratio for samples saturated in the laboratory. However, for the naturally saturated slices (group 1), the signal is <0.9 for first stimulation tempera-

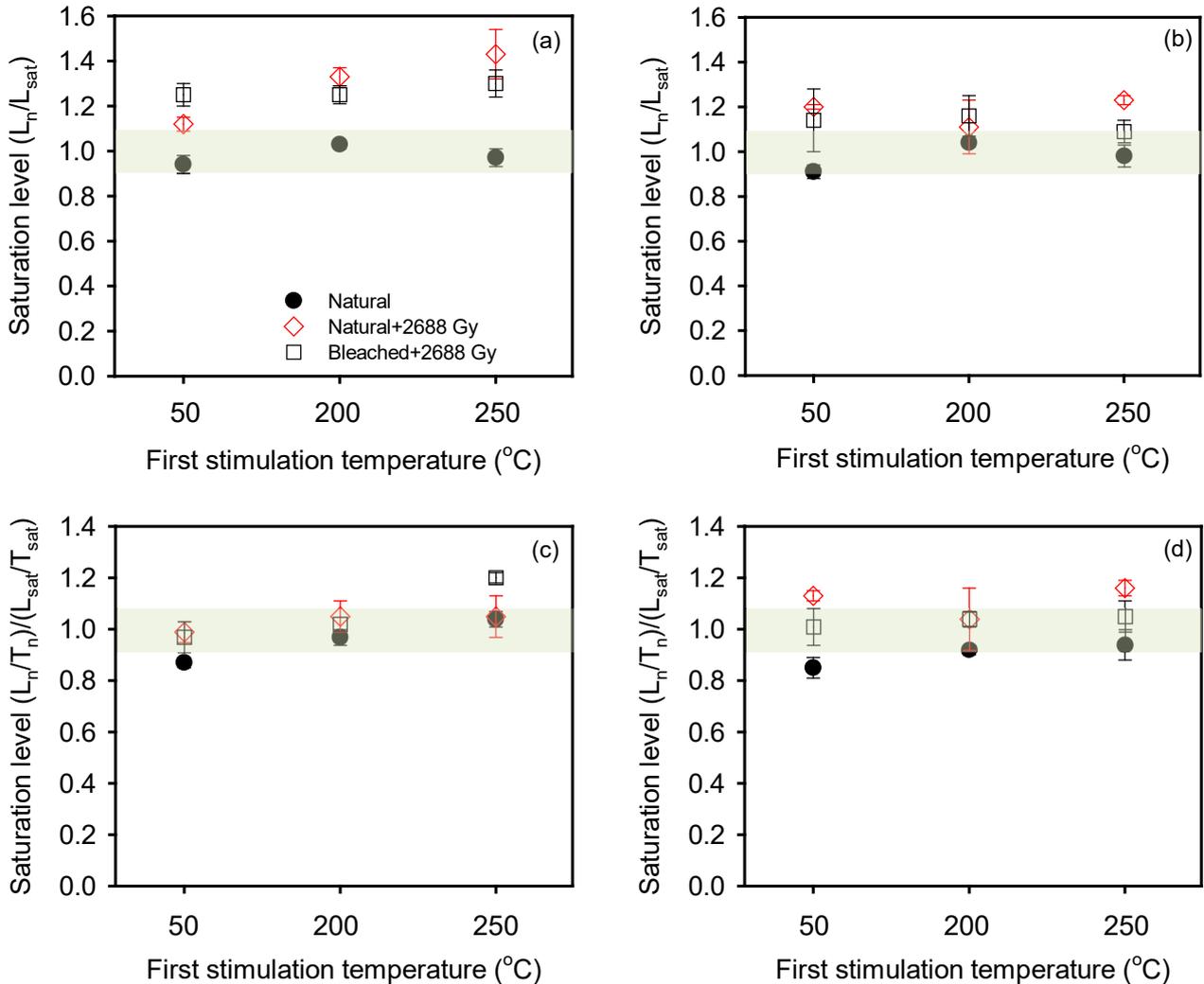


Fig. 7. Effect of first stimulation temperature on the degree of saturation. Test doses in (a) and (c) are ~53 Gy and in (b) and (d) ~595 Gy. The shadowed area covers 10% deviation from unity (i.e. 0.9 to 1.1). Three slices were measured at each temperature. The error bars represent one standard error.

ture at 50°C. This effect is independent of test-dose size and could possibly be because we are sampling a less stable signal with a lower prior IR stimulation temperature. However, there is also a tendency for an increase in the levels of laboratory dosed  $L_{n+2688}/T_{n+2688}$  and  $L_{\text{bleached}+2688}/T_{\text{bleached}+2688}$  with a prior IR stimulation temperature, and in fact some data lie above unity; thus, the increase cannot be solely attributed to signal stability. Our data suggest that an apparent increase in the ratio with IR stimulation temperature could be a combination of both sensitivity change and increased stability. It may also be that the results obtained using a first stimulation temperature of 200°C are closer to unity than those obtained using 250°C, although at this stage we do not wish to place too much confidence in this conclusion.

#### 4. DISCUSSION AND CONCLUSION

We find that the reproducibility of the sensitivity-corrected dose response curve is poorer when a small test dose (4 Gy) is used to measure dose response curves up to saturation; however the small test dose gives the best agreement between the natural signal (expected to be in saturation) and the dose response curve. This result may be attributed to the inheritance effect, which can translate into a dose as high as 7 Gy (section 3 – Inheritance in the test dose response) and thus be a source of significant contamination in the test dose pIRIR<sub>290</sub> signal. We are unable to detect a significant effect of inheritance in the comparison of the large 595 Gy and 53 Gy test doses in the subsequent experiments (reproducibility of laboratory dose-response curves, Fig. 4, or first to second cycle saturation light level ratios, Fig. 7). This is presumably because even the 53 Gy test dose was >7 times larger than any potential inheritance. Fig. 4c–f indicates that laboratory measurements of dose-response curves are reproducible and, when the test doses are much larger than the inheritance dose (7 Gy) in any given experiment, the resulting dose-response curves are independent of the order in which regeneration doses are given. Perhaps surprisingly this is equally true of absolute light levels (Fig. 4 insets) and  $L_x/T_x$  ratios. Although it appears that the absolute light levels ( $L_x$ ) give natural signals closer to saturation, we argue that this is an artefact of a significant change in sensitivity especially in the first SAR cycle (Fig. 6). This argument is supported by the comparison of the non-normalized and test-dose normalized ratios of the “natural” to laboratory saturation levels (Fig. 7). Although the non-normalized ratios are close to unity for the natural signal, these ratios are up to 1.4 (Fig. 7a) for the samples which were saturated in the laboratory ( $L_{n+2688}/L_{\text{sat}}$  and  $L_{\text{bleached}+2688}/L_{\text{sat}}$ ) before the first measurement cycle (test dose), clearly indicating a significant change in sensitivity. This is in contrast to the ratios of the test-dose normalized data which are generally much closer to unity, although there remains some underestimate for a first stimulation temperature of 50°C. It is

interesting to note that Li and Li (2012a) and Yi *et al.* (2016) have both observed an underestimation in  $D_e$  plateau for a first stimulation temperature of 50°C, although several authors (e.g. Buylaert *et al.*, 2012, 2015; Yi *et al.*, 2015) have shown that natural doses up to 650 Gy are independent of first stimulation temperature. It may be that the underestimates derived from the test-dose corrected natural saturation light levels (Fig. 7 for a first stimulation temperature of 50°C) only become significant for large palaeodoses.

Our data indicate that there is a systematic increase in dose response curve saturation (or  $D_o$ ) with test dose size when the regeneration doses are first given in increasing order, and then decreasing order (Fig. 5a). This trend disappears if these orders are reversed (Fig. 5b). We do not at present understand this effect.

In conclusion, for our samples, it appears that when measuring large doses, (i) the saturation of the dose response curve changes as a function of the test dose if the regeneration doses are delivered in an increasing order, but this effect is not so important if the regeneration doses are delivered in the decreasing order, (ii) the size of the test dose should be significantly larger than the apparent dose corresponding to any carryover from the previous measurement, and (iii) it may be better to use a first stimulation temperature of at least 200°C for samples close to saturation, regardless of the size of the test dose.

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