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The effect of preheating on the IRSL signal from feldspar

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ABSTRACT

It is difficult to relate the IRSL signal from feldspar to a particular region of the TL curve; prior IR stimulation reduces the TL signal over a wide range of temperatures. Such data are apparently consistent with the observation from pulse anneal experiments that a laboratory-induced IRSL signal is detectably eroded by a relatively low temperature preheat. These results can be explained by a distribution of trap depths of IR sensitive traps, and/or by changes in recombination probability induced by IR exposure. To investigate the relative importance of these processes, we first examine the relationship between the loss of blue IRSL and TL signals with preheating, and the effect of prior IRSL on the TL signal. Using IRSL measured at 50 °C and a SAR protocol, we then examine the dependence on preheat temperature of equivalent dose (D_e), laboratory fading rate (g), and the resulting luminescence age, from three sedimentary potassium-rich feldspar extracts. We demonstrate that there is no systematic increase in D_e for a preheat temperature range from ~80 °C to ~320 °C (60 s duration). After fading correction, age plateaus vary slightly over the temperature range examined, but there is no evidence for an increase in age with preheat temperature. We therefore conclude that the main dosimetry trap(s) in feldspar are not significantly eroded by laboratory heating for up to 60 s at 320 °C, and we tentatively identify the source of this IRSL as a TL peak lying between 410 and 420 °C; this suggestion is consistent with a kinetic analysis of sensitivity-corrected IRSL data. The corollary to our observations is that shallow (unstable) traps do not give rise to a significant IRSL signal.

1. Introduction

It has been observed that IR stimulation of feldspar at elevated temperature gives lower laboratory fading rates than those observed at the more widely used stimulation temperature of 50 °C (Thomsen et al., 2008). Using a stimulation temperature of 225 °C, Buylaert et al. (2009) have confirmed that these lower fading rates seem to apply in nature. However, these authors did not explore the use of higher temperatures because they used the standard preheat of 60 s at 250 °C, and it was considered necessary to keep the stimulation temperature below the preheat temperature. Given the apparent trend of decreasing tunnelling probability with increased stimulation temperature, it is important to investigate the practical upper limit to preheat temperature. It is equally important to find out what minimum preheat temperature is sufficient to isolate a geologically stable IRSL signal.

Bøtter-Jensen et al. (2003a; pp. 211–214) point out that there is conflicting evidence on the need to preheat feldspar during D_e measurement. Duller and Bøtter-Jensen (1993) examined the ratio of IRSL from a natural sample to which a beta dose had been added, to the IRSL from separate natural samples (i.e. $N+\beta/N$), as a function of preheat temperature, and did not observe any systematic change in this ratio with temperature. On the other hand Duller (1994) used pulse annealing curves to argue that the $N+\beta$ signal contained thermally unstable components which could be removed by a preheat at 220 °C for 10 min, and the need for such a high temperature preheat is now widely accepted.

In this paper we investigate the origins of the IRSL signal by comparing the IRSL response to preheating with the TL response to prior IR stimulation. We use a SAR protocol to investigate the preheat dependence of both equivalent dose (D_e) estimates and rates of anomalous fading, to derive the dependence of age on preheat temperature. Finally we investigate the thermal stability of the IR signal following a high temperature preheat, and compare predictions of TL peak temperature based on these data with the most likely source candidate in the TL glow curve.

2. Measurement facilities and samples

All measurements were undertaken on one of several Risø TL/OSL readers, model 15 or 20 (Bøtter-Jensen et al., 2003b). IR

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stimulation used 875 nm light emitting diodes delivering between 100 and 200 mW cm^{-2} at the sample position; the sample was held at 50 °C during stimulation. Detection was in the blue region of the spectrum, through a combination of Corning 7-59 and Schott BG-39 glass filters. Unless otherwise indicated the IRSL signal is derived from the first 2 s of stimulation less a background from the end of stimulation period. All heating was at 5 °C s^{-1} in a nitrogen atmosphere.

Four different samples were used in this study: H22546 (early MIS5e shallow marine sediment from Russia, expected age ∼130 ka; Murray et al., 2007); 963603 (colluvium from Tanzania; Sørensen et al., 2001); 052225 (fluvial sediment from Portugal; Martins et al., in press) and 981011 (early MIS5e shallow marine sand from Denmark, expected age ∼130 ka; Murray and Funder, 2003). All feldspar extracts were prepared by sieve-separating the desired grain size (180–250 μm), and treating with HCl and H_2O_2 before density separating the grains <2.58 g cm^{-3}. This K-rich feldspar fraction was then etched in 10% HF for 40 min and 10% HCl for 1 h, before mounting as a single layer of grains in 0.1 mm thick stainless steel cups using silicone oil.

3. TL glow curves and IR pulse anneal curves

To illustrate the behaviour of our samples, Fig. 1(a) presents the representative natural and regenerative TL curves for sample 963603, all measured on the same aliquot. In the natural TL, there is a pronounced peak at ∼310 °C and a smaller peak at ∼400 °C; there is no significant signal below 200 °C. In the subsequent regenerated signal (after preheating to 250 °C for 60 s), the same two peaks are clearly visible. However, in the regenerated TL signal that has not been preheated, the high temperature signals are dwarfed by a very strong multiplet lying between ∼100 and 130 °C. Samples H22546, 981011 and 052225 showed similar behaviour, although the second peak in the natural at ∼400 °C was much less pronounced; in H22546, for example, it was only visible as a broad high temperature tail on a peak at ∼320 °C.

Fig. 1(b) presents the corresponding dependence of IRSL on temperature using a standard pulse anneal measurement on an aliquot of 963603. These data were obtained by using a sufficiently short low-power IR stimulation such that the IRSL signal was not significantly depleted by each pulse of IR (see caption to Fig. 1). Then the aliquot was heated to the next preheat temperature, held for 10 s, and the IR measurement repeated. The IRSL signal from the natural (filled circles) does not begin to decrease until above 200 °C, and it has been completely thermally eroded by ∼400 °C. In contrast, the regenerated IRSL signals (open circles) begin to decrease at a much lower temperature. The IRSL decreases slowly at first, and begins to decrease more rapidly by ∼180 °C. The regenerated data remain consistently below the natural data, and show a very similar pattern to the results obtained by Duller (1994).

The changes in these pulse anneal IRSL data seem to reflect the shape of the natural and regenerated TL curves; in the natural the IRSL begins to decrease at ∼200 °C, just where the natural TL signal starts to become significant. Similarly, the regenerative TL curve extends down to lower temperatures and the regenerated pulse anneal IRSL curve begins to decrease in the same temperature range. However any correlation is certainly not proportional; the large loss of low temperature TL peaks (<200 °C) only seems to relate to a very small loss of IRSL (<10%), if any, in the regenerated data, whereas almost all the loss of IRSL in both the natural and regenerated signals seems to occur in the temperature region where only a small amount of the total TL signal is detected.

4. Effect of prior IR stimulation on TL

One way of looking at the relationship between IRSL and TL is to look at the loss of TL signal resulting from prior IR stimulation (e.g. Li and Aitken, 1989; Duller and Wintle, 1991). Fig. 2(a) shows the effect of 0, 10, 100 and 1000 s of IR stimulation on the regenerated TL signal using a single aliquot of 963603. This aliquot was first heated to 500 °C and given a dose of 260 Gy before each measurement; the absence of sensitivity change was confirmed by repeating the 0 s and 64 s measurements. The TL signal is reduced by IR stimulation throughout the temperature range, but most loss seems to occur around 140 and 400 °C. This is more visible in Fig. 2(b), in which the various TL signals following IR stimulation are subtracted from the TL without prior stimulation. These 'lost TL' signals clearly show that two peaks at ∼140 °C and ∼410 °C are reduced by IR stimulation. In both cases there is a strong linear relationship ($R^2 > 0.999$) with negligible intercept, between the net IRSL and the peak height of the lost TL resulting from 7 different IR stimulations in the range of 2–1000 s (only data for 140 °C peak

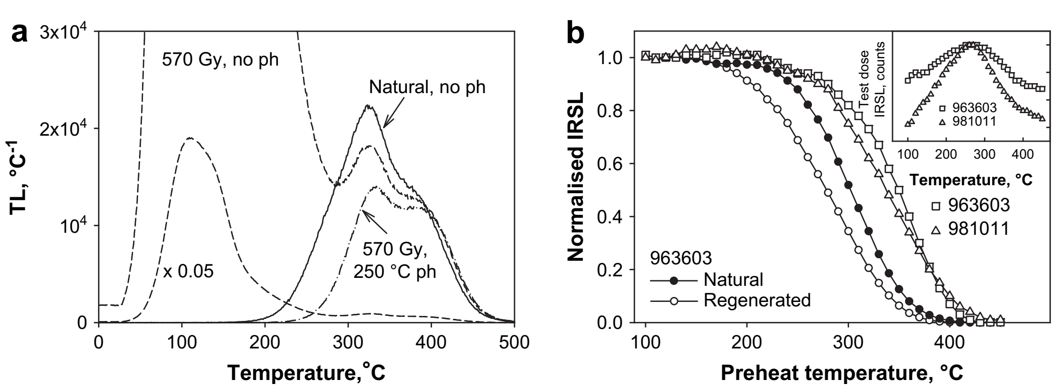

Fig. 1. (a) TL curves from sample 963603. All measurements on the same aliquot: natural without preheating; 570 Gy regenerative dose without preheating, and 570 Gy followed by preheating for 60 s at 250 °C. (b) pulse anneal curves. The aliquot (sample 963603) was stimulated for 0.1 s at ∼1% of full diode power, such that the IRSL signal was not significantly depleted by the stimulation. It was then preheated for 10 s at the lowest temperature shown, and the measurement repeated. This process was repeated with 10 °C increments in preheat temperature until 450 °C (filled circles). A second aliquot was bleached with IR at 45 °C, preheated to 90 °C for 10 s, and bleached again with IR. The aliquot was then given a regenerative dose of ∼600 Gy, and the pulse anneal process repeated (open circles). Finally, a third aliquot of 963603 was used to obtain the sensitivity-corrected IR remaining after preheating for 10 s (open squares). In this experiment, the aliquot was heated to 500 °C, given a 4 Gy dose, preheated to the temperature shown, and measured to give L_i. The measurement process was repeated twice more, to ensure no photo- and/or thermal transfer, and then the dose measurement cycle was repeated, but with a fixed preheat at 90 °C, to give T_i. The data shown (open squares) are the resulting L_i/T_i. The variation of T_i with temperature is shown inset. This last experiment was also repeated for sample 981011 (open triangles).