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Testing Post-IR IRSL protocols for minimising fading in feldspars, using Alaskan loess with independent chronological control

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ABSTRACT

Concern over anomalous fading has been the biggest single factor responsible for deterring the widespread use of the infra-red stimulated luminescence (IRSL) or thermoluminescence (TL) signal from feldspars for luminescence dating. There has therefore been great interest in the use of the recently proposed Post-IR IRSL signal, because it has been shown to significantly reduce the degree of anomalous fading observed in feldspars and therefore potentially provides a means of circumventing the issue. This study undertakes a systematic investigation into various preheat and Post-IR IRSL measurement conditions proposed in the literature, by using two samples from the Halfway House loess section in Alaska which bracket the Old Crow tephra which has been dated using fission track methods. Preheat plateau tests show a dramatic change in equivalent dose with Post-IR IRSL measurement conditions, and further tests reveal that these changes are driven by preheat temperature rather than Post-IR IR stimulation temperature. Dose recovery tests on laboratory-bleached material mimic the findings of the natural preheat plateau test data, and sensitivity change between the first and second Single Aliquot Regenerative dose (SAR) measurement cycle is found to be responsible. Comparison of the Post-IR IRSL ages with the independent age control shows that, for the samples in this study, the Post-IR IR signal stimulated at 290 °C is inappropriate for dating. However, use of lower preheat (250–300 °C) and Post-IR IR stimulation temperatures from 225 to 270 °C gave rise to ages which were in agreement with the independent age control.

1. Introduction

Optical dating using feldspars potentially offers a significant advantage over quartz due to the greater upper age limit which can be achieved using feldspars. However, the single greatest factor which has prevented the widespread uptake of luminescence dating using feldspars is the phenomenon of anomalous fading. Anomalous fading (Wintle, 1973) is the decay of an unstable luminescence signal which, if uncorrected for, results in underestimation of the true age of the sample using either infra-red stimulated luminescence (IRSL) or thermoluminescence (TL) signals from feldspars (Spooner, 1992, 1994). Some authors argue that anomalous fading is ubiquitous (e.g. Huntley and Lamothe, 2001), affecting all feldspars, whilst other authors find no evidence for anomalous fading in their studies (e.g. Frechen et al., 2001; Preusser et al., 2005). Conventional methods of luminescence dating using feldspars and polymineral fine-grains have tried to measure rates of anomalous fading, and where anomalous fading is detected, correction methods based on models of the process are used to compensate for the loss of signal over time. However, there is no universally agreed model or method for detecting and measuring anomalous fading. The correction method of Huntley and Lamothe (2001) has been widely applied to young samples, where the equivalent dose (D_e) falls within the linear part of the dose–response curve, but in this region feldspars offer little advantage over quartz, which typically spans the same dose range and is not reported to experience anomalous fading. It is the region beyond the linear portion of the feldspar dose–response curve which holds the greatest interest for extending the limits of reliable optical dating, yet in this region the corrections for anomalous fading (e.g. the Dose Rate Correction (DRC) method of Lamothe et al. (2003), and the model of Kars et al. (2008)) become more complex and hence are less commonly applied.

In contrast to attempts to measure and correct for fading in feldspars, recent efforts have focused on isolating an optical signal from feldspars that minimises fading, hence potentially removing the need for such complex signal corrections. Thomsen et al. (2008) first reported that the IRSL signal observed at 225 °C following a prior IR stimulation at 50 °C was much more stable than the conventional IRSL signal (measured at 50 °C) for the same coarse-

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grain potassium-rich feldspar sample; a conventional feldspar preheat temperature of 250 °C for 60 s was used. This 'Post-IR IRSL' signal (hereafter referred to as Post-IR_{50} IR_{225}) gave much lower laboratory anomalous fading rates (g-values ~0.5–1.5%/decade) than conventional IRSL measurements made at 50 °C (IR_{50}, g-values ~1.5–3.5%/decade).

Buylaert et al. (2009) used the same measurement conditions as Thomsen et al. (2008) to examine whether the Post-IR_{50} IR_{225} method could be successfully applied to coarse-grain potassium-rich feldspars extracted from natural sedimentary samples. The fading-corrected Post-IR_{50} IR_{225} and IR_{50} ages were found to be in agreement with each other from a few thousand years to >260 ka (Buylaert et al., 2009), but the lower fading rate observed for the Post-IR_{50} IR_{225} signal offers the advantage that an age derived from this elevated temperature IRSL signal is less dependent on the anomalous fading correction than is the IR_{50} signal. The IR_{50} stimulation is thought to recombine close, unstable, donor-acceptor pairs, and the lower apparent fading rate observed for the Post-IR_{50} IR_{225} signal is due to recombination of the remaining distant, and hence more stable, donor-acceptor pairs (Thomsen et al., 2008). The implication of this, is that increased preheat and IR stimulation temperatures are likely to give rise to increasingly stable Post-IR IRSL signals (Thomsen et al., 2011). Using coarse-grained potassium-rich feldspars, Murray et al. (2009) investigated the practical upper limit to preheat temperature, which in turn governs the practical upper limit of IR stimulation temperature (as the latter should be lower than the former). They concluded that it may be possible to use preheat temperatures as high as 320 °C, implying that IR stimulation temperatures of ~300 °C may therefore be used (i.e. much greater than previously used). Following these observations, Thiel et al. (2011) used a preheat temperature of 320 °C for 60 s, followed by IR stimulation at 50 °C, and then a second IR stimulation at 290 °C to obtain a 'Post-IR_{50} IR_{290}' signal used for dating polymineral fine-grains prepared from Austrian loess. Thiel et al. (2011) noted that low fading rates were observed using the Post-IR_{50} IR_{290} signal (g-values <1.5%/decade), and that the natural Post-IR_{50} IR_{290} signal from a ~780 ka sample was observed to be in saturation when compared to a laboratory-generated dose–response curve, concluding that there was no fading of this signal in nature. Some concern was expressed about the size of the residual doses (15–20 Gy) remaining after laboratory bleaching using either a Hönle Sol2 solar simulator or using 24 h of daylight bleaching. Thiel et al. (2011) concluded that fading-uncorrected Post-IR_{50} IR_{290} ages with no residual subtraction are the "preferred ages" for the loess sequence studied, although they also comment that "firm evidence to support this choice is not available".

Although the use of the Post-IR IR signal was first suggested more than 3 years ago, there has still been very little work conducted to assess the accuracy of this approach using independent numerical dating techniques. This study systematically investigates the newly proposed post-IR IRSL protocols discussed above, comparing the findings against independent age control which is unrelated to any other luminescence method. Post-IR IRSL methods were developed for coarse-grained feldspars and are designed to minimise fading rates; in this study, these methods are tested using polymineral fine-grain samples prepared from an Alaskan loess site, Halfway House. Several previous TL/IRSL studies have been conducted at the Halfway House site (e.g. Wintle and Westgate, 1986; Berger et al., 1994; Oches et al., 1998; Berger, 2003) with mixed success, and fading-corrected IRSL studies (Auclair et al., 2007) have also been conducted. Critically for this study, however, the Halfway House site has independent age control in the form of a key tephra marker bed named the Old Crow tephra, dated using the isothermal plateau fission track (ITPFT) technique. Fading rates and luminescence signal characteristics are examined, and the ages generated are compared with those of previous studies. Finally, the reliability of these different feldspar dating methods is assessed.

2. Study site

The Halfway House loess section (Fig. 1) is exposed as a road-cut ~30 km southwest of Fairbanks, Alaska, and located near the Tanana River. The loess section is ~11 m in thickness, with the Old Crow tephra being found between ~8 and 9 m depth below the modern day surface (Muhs et al., 2003, 2008). The Old Crow tephra is a thick (>20 cm at Halfway House) marker bed which represents the most widespread Quaternary eruption in eastern Beringia (Preece et al., 2011) and hence acts as a key stratigraphic marker. Westgate et al. (1990) generated a weighted mean isothermal plateau fission track (ITPFT) age (Westgate, 1989) of 140 ± 10 ka (based on two determinations using sample UT501, and four using UT613, with ages ranging from 120 to 160 ka) for hydrated glass shards from the Old Crow tephra. Later, based on four ITPFT ages (129 ± 14 ka (UT1434), Péwé et al., 2009; and 146 ± 28 ka (UT613), 156 ± 45 ka (UT501), and 120 ± 23 ka (UT613), Preece et al., 1999), a weighted mean glass-ITPFT age of 131 ± 11 ka was calculated by Péwé et al. (2009). This value was then revised downwards by 5% in the light of corrections made to the age monitor, giving a glass-ITPFT age of 124 ± 10 ka (Preece et al., 2011). Stratigraphic and palaeoevironmental evidence supports a very late Marine Isotope Stage 6 (i.e. a cold-stage) age for the Old Crow tephra (Reyes et al., 2010), consistent with the upper limit of the ITPFT age.

Two loess samples, 82HH-2 and -5, were taken for luminescence dating from below and above the Old Crow tephra respectively at the Halfway House section (Fig. 1), using cylindrical sample tubes of 5 cm diameter. The independent age control provided by the Old Crow tephra serves as a check on the suitability and accuracy of the various newly proposed IRSL techniques designed to minimise fading rates.

3. Sample preparation and equipment

All luminescence measurements were made using polymineral fine-grain material prepared using the standard methods outlined below. Samples were treated with a 10% v.v. dilution of 37% hydrochloric acid, followed by 20 vols. hydrogen peroxide, to remove carbonates and organic material respectively. The 4–11 μm polymineral fraction was separated according to Stokes Law, settling for 20 min over 20 cm depth of 0.01 N sodium oxalate to remove the >11 μm fraction, prior to settling a number of times for 4 h over 20 cm to isolate the 4–11 μm fraction. Polymineral fine-grain aliquots were prepared by dispensing 1 mg of material suspended in acetone onto each 9.8 mm diameter aluminium disc. Luminescence measurements were made using several automated Risø TL/OSL readers, equipped with infra-red (IR) (870 Δ 40 nm) LEDs, and $^{90}Sr/^{90}Y$ beta sources for irradiations delivering between ~2.2 and 5.7 Gy/min. Detection was achieved through a combination of Corning 7-59, and Schott BG39 and GG400 filters. Luminescence measurements were made using a variety of preheat and stimulation conditions as described below, but in each case a Single Aliquot Regenerative dose (SAR) protocol (Murray and Wintle, 2000) was used, and the preheat conditions preceding measurement of the natural or regenerative dose signals were the same as those used prior to measurement of the test dose response (Blair et al., 2005). In all cases, the duration of the preheat was 60 s. The IRSL signal measured at 50 °C ('IR_{50}') or the Post-IR IR signal measured at elevated temperatures ('Post-IR_{50} IR_{elev}') was derived from the initial 1.2 s minus the final 20 s of each 100 s IR stimulation.

The dose rate to the samples (Table 1) was assessed using a combination of thick-source alpha counting using *Daybreak*