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Detection of far-red IRSL from loess

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Abstract: *It has recently been proposed that it may be possible to extend the age range of luminescence dating of loess using the far-red (λ=665-740nm) emission from feldspar, as it is thought not to exhibit anomalous fading. Studies on red luminescence have been hindered due to technical difficulties in suppression of background and other factors. Recently modifications to apparatus (esp. photo-multiplier plus filter combinations) have been reported demonstrating that red IRSL (λ=590-700nm) may be observed from coarse-grained feldspar (Fattahi and Stokes, 2002a). However, this modified system was not able to detect far-red IRSL (λ=665-740nm) from old (>800ka) Chinese loess. In this short note we describe further modifications to the system which have successfully enhanced the far-red IRSL signal, and at the same time reduced background signal levels. As a result, routine measurements of far-red IRSL from loess are possible.*

Introduction

The luminescence dating of feldspar using UV-blue emissions has been hindered by the ubiquitous presence of anomalous fading and associated age underestimation (e.g. Lamothe and Auclair, 1999; Huntley and Lamothe, 2001). Red TL (λ>600nm) of feldspar has been demonstrated not to exhibit anomalous fading (Zink and Visocekas, 1997). The work of Zink and Visocekas (1997) was, however, focussed on a relatively small number of samples and exploited relatively low temperature (<370°C) red TL. A logical extension of their investigations is to study red IRSL from feldspar (Fattahi, 2001).

Recently, Fattahi and Stokes (2002a) have successfully demonstrated that it is possible to detect red IRSL (λ=590-700nm) from coarse grain potassium-rich feldspar by careful selection of novel photo-multiplier (PMT) and filter combinations. In further testing we have found that their system is not able to observe far-red IRSL (λ>665nm) from loess sample as old as 800 ka (expected D_e c. >3,000Gy). Here we describe further developments to the detection system that allow far-red IRSL from loess with doses as low as 50Gy to be measured.

Selection of detection window

There are a variety of luminescence emissions of feldspar from 280 to 800nm (Krbetschek et al., 1997). The conventionally used UV/blue emissions suffer from anomalous fading (e.g. Lamothe and Auclair, 1999). The pioneering work by Zink and Visocekas (1997) has shown that while blue TL of volcanic feldspars (λ<600nm) suffers severely from anomalous fading, red TL (λ>600nm) from the same samples does not fade anomalously. Fattahi and Stokes (2002b) reported that orange-red IRSL (λ=590-700nm) from potassium feldspar derived from sediments exhibited anomalous fading, but at a level much lower than blue IRSL, and that far-red IRSL (λ=665-700nm) showed no fading. It seems that the further toward IR we are able to detect red IRSL, the more stable a signal we observe. We interpret this as a reflection of a progressive reduction of the influence of the broad yellow-orange (~570nm) emission centre which has previously been recognized to exhibit fading (e.g. Fattahi and Stokes, 2002b).

It has been demonstrated that loess has a strong far-red emission (λ=665-740nm) in both natural and laboratory irradiated polymineral samples (Krbetschek et al., 1997; Lai et al., 2002). Moreover, far-red IRSL is highly reproducible and amendable to Single Aliquot Regenerative (SAR) techniques (Fattahi and Stokes, 2002c; Lai et al., 2002; Arnold et al., 2002). As a result, we here further focus on the wavelength band of 665-740nm (far-red IRSL) as the optimum detection window (filter combination is Schott RG 665 + Omega 740 SP, Fig 1). The difficulty in detecting far-red IRSL at this wavelength is the suppression of high background related to the close proximity of the IR stimulation source (λ=830 ± 5nm).

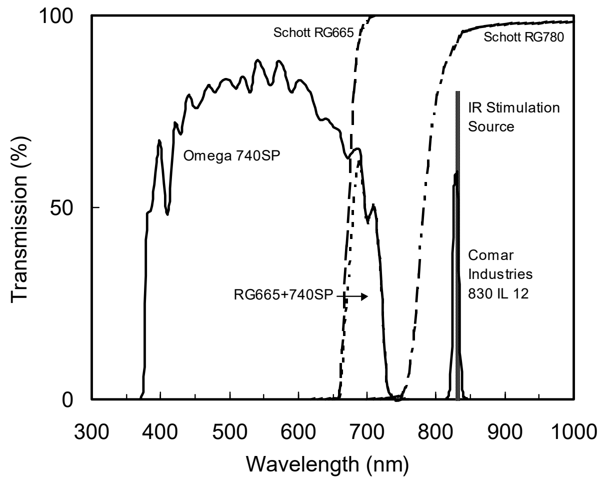

Figure 1.
Detection and stimulation windows.

The sources of background

There are two main contributions to background---- dark counts from the photo-multiplier tube (PMT), and reflected light from IR stimulation (Fattahi, 2001). The dark count of the extended range PMTs used for red emission studies is constant and can be reduced by an order of magnitude down to about 200 c/s by cooling the PMTs down to ~-15°C (Fattahi and Stokes, 2002a). The remaining primary contribution to the background signal is from reflected incident IR stimulation photons and related emissions, due to the close positioning of detection (665-740nm) and stimulation (830 ± 5 nm) windows.

A suitable PMT should be chosen for the purpose of detecting far-red IRSL, and at the same time reducing the background from IR stimulation. There is no ideal PMT with high quantum efficiency (QE) at far-red emission and 0% QE at other wavelengths (see Fig 1 in Fattahi and Stokes, 2002a). While the conventionally used EMI bialkaline 9635 PMT (blue) is highly sensitive to UV/blue emission and has also been used for quartz orange-red TL (c. 600-620nm) detection (e.g. Miallier et al., 1991), it has no QE at wavelengths greater than 650nm, making it unsuitable for far-red emission detection. The EMI bialkline S20 9650 (red) PMT has high QE (5%) at 700nm. However, it has a QE of 1% at 830nm (the stimulation peak), which typically results in high background (~>10^6c/s). The EMI Biakaline D716A S11 (green) PMT has a QE of 0.1% at 700nm and a QE of less than 0.01% at 830nm, making it acceptable for far-red IRSL detection (Fattahi, 2001; Stokes and Fattahi, 2002).

In efforts to detect far-red IRSL from fluvial coarse grain feldspar, Fattahi and Stokes (2002a) have made the following adjustment to the standard Risø TA-15a TL/OSL system (Bøtter-Jensen, 1997), which incorporates an IR laser diode (400mW, 830±5nm) and a ^{90}Sr/^{90}Y beta radioactive source:

1. An extended EMI D716A S11 (green) PMT was used.
2. An S 600 Photocool Thermoelectric Refrigerated Chamber was fitted to cool the PMT (c. ~ - 20°C). This reduces PMT dark counts by an order of magnitude, down to c. 200 c/s.
3. The far-red IRSL was detected using a combination of Schott RG665 + 2*Corion FR400S + Schott BG39 filters, with an estimated detection band of 665-700nm (Fattahi and Stokes, 2002c).

The above modifications make it possible to detect far-red IRSL from fluvial coarse grain feldspar (bright sample), with background at a level of below 500 c/s (Fattahi, 2001). However, using this configuration it has not been able to detect far-red IRSL from loess. Figure 2 shows IR exposure decay curves measured using this configuration. While coarse grain feldspar (sample 15/1, 90-120μm, age c. 23.2 ± 1.8ka) gave a relatively high signal to noise ratio (3.64), no signal was distinguishable above background for a natural loess sample (age c. 800ka, D_e > 3,000Gy, grain size of 4-11 μm). By increasing the grain size to 11-78μm it is possible to detect a small red emission IRSL signal from this sample (initial signal = 400 c/s above background, ~0.13 c/Gy), but at a level which is not suitable for routine dating application.

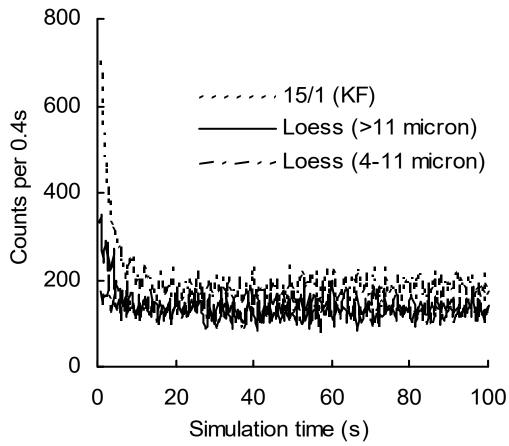

Figure 2.
Far-red IRSL signal level of loess and fluvial coarse grain feldspar using the detection system development by Fattahi and Stokes (2002a). IRSL was measured at 150°C.

No.	S11 PMT (Green) Filter combination	Sample: 15/1 S (Signal)	N (Noise)	S/N	S-N (Net signal)
1	740SP+RG665	301,158	188,157	1.6	113,002
2	FR400S+RG665	346,106	281,803	1.2	64,303
3	2*FR400S+RG665	37,542	6,950	5.4	30,592
4	2*FR400S+740SP+RG665	25,954	4,489	5.8	21,465
5	FR400S+740SP+RG665	61,418	20,178	3.0	41,241
6	BG39+2*FR400S+RG665	3,677	815	4.5	2,862
7	BG39+FR400S+RG665	7,598	1,862	4.1	5,737
8	BG39+740SP+FR400S+RG665	6,668	2,459	2.7	4,208
9	BG39+740SP+RG665	28,499	19,549	1.5	8,950
10	SWP685+2*FR400S+RG665	5,527	1,097	5.0	4,430
11	SWP685+FR400S+RG665	15,244	6,018	2.5	9,227
12	SWP685+740SP+FR400S+RG665	8,952	1,903	4.7	7,048
13	SWP685+740SP+RG665	19,697	4,630	4.3	15,067
14	SWP685+RG665	7,579,798	7,077,072	1.1	502,727
	S20 PMT (Red) **Filter combination**	**Sample: 1023/2** **S** **(Signal)**	**N** **(Noise)**	**S/N**	**S-N** **(Net signal)**
15	2*FR400S+RG665	716,193	655,953	1.09	60,240
16	2*FR400S+RG665+SWP685	36,918	34,904	1.06	2,013
17	2*FR400S+RG665+SWP685+BG39	36,354	35,175	1.03	1,179
18	2*FR400S+RG665+SWP685+HA3	250,189	234,695	1.07	15,494
19	2*FR400S+RG665+SWP685+740SP	162,993	152,562	1.07	10,431
20	2*FR400S+RG665+740SP	184,213	172,559	1.07	11,653
21	2*FR400S+RG665+BG39	343,481	324,731	1.06	18,750

Table 1*.*

Results of filter combination and PMT tests

Bright potassium-rich samples (90-120μm) 15/1 (age c. 22.2 ± 1.8ka) and 1023/2 (age c. 86 ± 6ka) (Colls, 1999) were used. It has been demonstrated that far-red IRSL from these samples exhibit no sensitivity change (Fattahi, 2001). Sample were mounted on stainless steel discs, bleached and administered a dose of 110Gy. IRSL was measured at 30°C for 100s after preheat at 250°C for 10s, and the IR diode power is kept at 90%. IRSL was then re-measured to obtain a background. The far-red IRSL signal was integrated over the first 1 s. The thickness of filters used: 740SP 4mm; FR400S 8mm; BG39 1mm; others 3mm.

Tests of additional signal pass filters

The purpose of using IR-cut filters in front of a photo-multiplier tube is to block the reflected light from IR laser diode. The IR-cut filters available are Schott BG 39, Omega 740 SP, Corion FR 400S, and Delta SWP BL 685 (Fig 3). Fattahi and Stokes (2002a) presented data for a variety of IR-cut filter combinations. However, most of their testing focused on a detection band of 590-700nm. This orange-red region feldspar emission has been shown to exhibit anomalous fading (Fattahi and Stokes, 2002b) due to possible influence of a 570nm emission centre. We have tested additionally filter combinations with far-red (λ=665-740nm) transmission, together with both S11 (Green) and S20 (red) PMTs (Table 1).

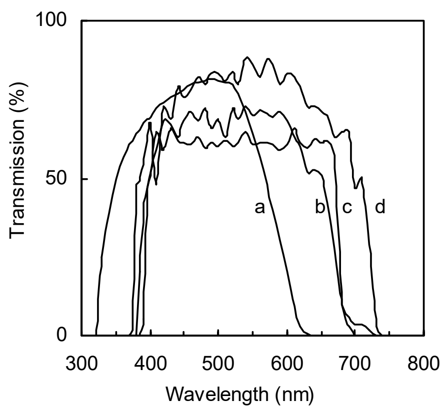

Figure 3.
Transmission characteristics of IR-cut filters (redrawn from Fattahi and Stokes 2002a). (a) Schott BG 39; (b) Corion FR 400S; (c) Delta SWP BL 685; (d) Omega 740 SP.

The S20 (red) PMT was found to result in high backgrounds using all filter combinations and yields poor signal to noise ratios. For the S11 (green) PMT, the filter combination of 2*FR400S + 740SP + RG665 gives the highest signal to noise ratio (5.8), and produces high net signal (21 kc/s) with a background level of 4.5 kc/s. The filter combination of 2*FR400S + RG665 gives a signal to noise ratio of 5.4, and produces higher net signal (30 kc/s) with a background level of 6.9 kc/s. The combination of BG39 + 2*FR400S + RG665 has the lowest background level (815 c/s) with a signal to noise ratio of 4.5, but produces very low net signal (2.9 kc/s). The BG39 is not ideal for far-red IRSL detection, as the transmission at 700nm is less than 1%. As a result, the combination of RG665 + BG39 focuses the band of below 700nm (665-700nm) which has possibly more influence from the 570nm emission peak. For bright, coarse-grained potassium-rich feldspar samples, the combinations of 2*FR400S+RG665 and 2*FR400S + 740SP + RG665 are suitable for far-red IRSL detection (λ=665-700nm), combined with a cooled S11 (green) PMT.

The combination of 740SP and RG665 filters results in a very high net signal (113 kc/s), but also gives a high background (188 kc/s). This high signal pass combination would be suitable for loess sample, if the background could be reduced. There are two possible means by which the background derived from the IR stimulation source might be reduced: (1) Shifting the wavelength of the stimulation source to a longer value, while remains within the feldspar resonance; (2) Filtering the existing IR source (λ=830 ± 5m) in order to remove or reduce any associated short wavelength emissions. In the absence of alternative IR sources at our disposal, we have investigated filtering of the existing source.

Long-pass and interference filtering of the IR stimulation sources

To maximize far-red IRSL emission the detection window should closely match that of the far-red emission centre (c. λ=720-740nm, Krbetschek et al., 1997). Our attempts to exploit this wave band have consistently resulted in high backgrounds (c. 250 kc/s with IR laser diode power at 90%) which we attribute to reflected and scattered Raman and other emissions from the IR source (Fig 4a,b).

We have tested a number of filter combinations to restrict short wavelength (i.e. λ<c. 780nm) transmission from the IR source. Firstly, various thickness (3-12mm) of Schott RG780 (Fig 1) long pass filter was attached to the front face of the IR laser diode. The IR laser stimulation source was then switched on at a constant power and the reflected IR from a blank stainless steel disc was detected. We found that 6mm of RG 780 was capable of reducing the background down to c. 124 kc/s (Fig 4c). Addition of further RG 780 filters was problematic given the limited amount of space available within the Risø diode array housing. We additionally tested an interference filter to try to both reduce background and minimise the total thickness of filters. For this purpose we used a Comar Industries 830IL12 filter (transmission 830 ± 12nm, Fig 1). The filter was tested alone, and in combination with 6mm RG780. We found that the optimum background (c. 4.2 kc/s) was achieved via a combination of both filters (Fig 4d).

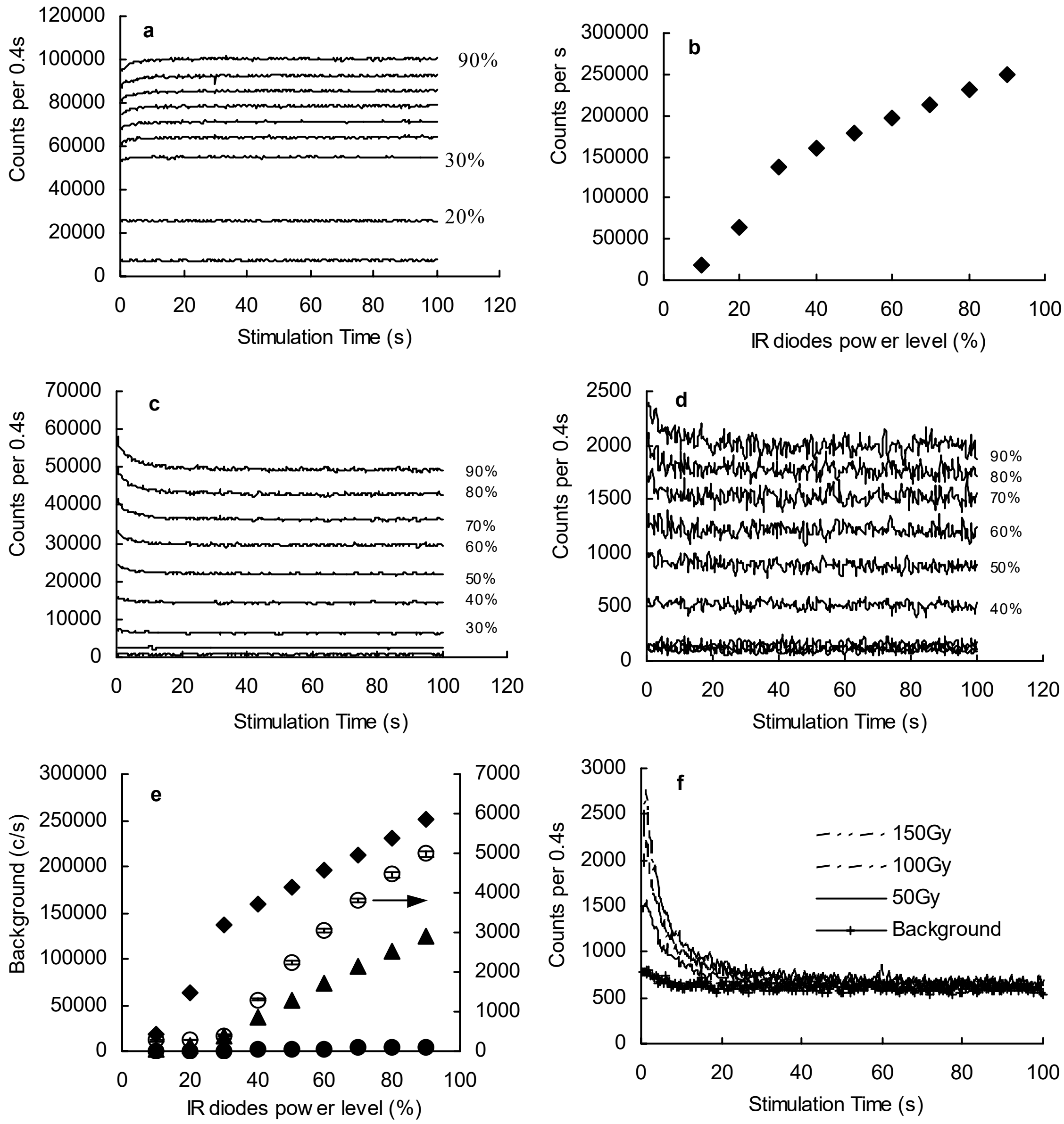

Figure 4.

Background reduction for far-red IRSL detection via additional filtering of IR light source. IRSL was measured at 30°C for 100s on a blank stainless steel disc except in (f). Detection filter combination is RG665 + 740SP. PMT is S11 (green). (a) Background vs stimulation time for a range of IR laser diode power levels; (b) Background counts vs IR diode power for (a); (c) Background vs stimulation time for a filtered IR diode source (filter is 6mm RG780); (d) Background vs stimulation time for a filtered IR diode source (filter combination is 6mm RG780 + 830IL12); (e) Comparison of background levels of filter combinations in front of IR diode. Full circle: 6mm RG780 and 830IL12; full triangle: 6mm RG780; full diamond: none; Empty circle (right Y-axis) is the enlargement of full circle for clarity. All error bars are smaller than the symbols. (f) IR exposure decay curves of far-red IRSL from laboratory irradiated loess, using the configuration in (d). IRSL was measured at 30°C for 100s after preheat at 250°C for 1min. Sample is L9/M, grain size 11-78μm, polymineral.

Discussions

We have extended the investigations of Fattahi (2001) by exploiting the combined result of filtering both the incident (IR) and reflected samples-derived luminescence. By filtering the IR laser diode with both glass and interference filters we successfully reduced the background to an acceptable level, and at the same time by exploiting Omega 740SP and Schott RG665 filters in front of the S11 (green) PMT, maximized the signal from the sample. Figure 4f shows measurements of dose response of far-red IRSL from loess using the present PMT + filter configurations. A 50Gy dose generated a net initial signal level of 1.8 kc/s with a signal to noise ratio of 2.0 and a background level of 1.9 kc/s. The 150Gy has a net initial signal level of 4.3 kc/s with a signal to noise ratio of 4.0.

Conclusions

For routine analysis of far-red IRSL from loess we recommend the use of an EMI biakaline D716A (green) PMT (S11). The EMI S20 9650 (red) PMT is less suitable due to the difficulties in suppression of background. The filter combination of Omega 740SP + Schott RG665 is chosen for the detection of far-red IRSL (λ=665-740nm) from loess. This combination produces high net signal from sample, but result in high background, and low signal to noise ratio. The background can be reduced to an acceptable level (<2,000 c/s) by attaching additional filters (a 830IL12 plus 6mm of RG780) in front of IR laser diode. The system developments described here enables routine detection of far-red IRSL from loess samples.

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Reviewer

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