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Optical dating of potassium feldspar using far-red ($\lambda > 665$ nm) IRSL emissions: a comparative study using fluvial sediments from the Loire River, France

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Abstract

UV-blue emissions from feldspars have often proved to be unacceptable dosimeters for age estimation given the intrinsic problem of anomalous fading. The potential of exploiting the far-red (>665 nm) IRSL emissions from potassium feldspars as a means of avoiding the malign effects of anomalous fading has recently been proposed (Studies on red thermoluminescence and infrared stimulated luminescence. Unpublished D.Phil Thesis, University of Oxford, 2001). While that research demonstrated that it was possible to detect the far-red signal in the coarse grain feldspar samples studied, it did not present any empirical data for actual geological samples with or without independent age control. The purpose of the present paper is to expand on the work of Fattahi (2001) by undertaking a direct comparison of SAR D_e's and corresponding optical ages for a suite of Holocene through Late Pleistocene fluvial terrace deposits from the upper reaches of the Loire River, France. Here we describe the behaviour of the far-red emissions for these samples and present some initial dating results. The far-red IRSL emission ages obtained are compared to UV-blue IRSL emission ages for the fluvial samples and to corresponding optical ages previously generated on quartz fractions from the same samples. Initial results are promising but show some inconsistencies. Basic experiments demonstrate that this is not attributable to insufficient sensitivity correction by SAR in the far-red emissions, neither was anomalous fading detected in either the far-red or UV-blue emissions over short storage times. It is suggested that refinement of pre-treatment and measurement conditions should enable more successful dating of feldspars with far-red emissions and that further research on the potentially stable far-red signal is necessary.

1. Introduction

This paper builds on the recent research of Fattahi (2001) and Fattahi and Stokes (2003a, b, c) which has explored the suitability of red (>600 nm) IRSL emissions from feldspars for dating applications. In particular it focuses on the 'far-red' (>665 nm) IRSL emission band, and investigates its potential as an alternative means to the more routinely used IRSL approaches that are often affected by anomalous fading. In order to test the performance of far-red IRSL emissions in dating applications, we here undertake a comparison of far-red emission age estimates on a series of fluvial deposits with independent age control (Colls, 1999).

Initial results are outlined from a series of investigations of the far-red emissions of potassium feldspar samples taken from a sequence of fluvial terraces found along the upper reaches of the Loire River, France (Colls, 1999; Colls et al., 2001). Preliminary far-red IRSL ages are reported for a limited number of samples and compared with both UV-blue IRSL age estimates and quartz OSL ages for the same samples. The suitability of using the SAR protocol for far-red emissions, the results of short-term anomalous fading tests, and the characteristics of the far-red emission signal for these samples are also discussed.

2. The potential for exploiting far-red emissions

The use of conventional blue-UV IRSL emissions from feldspars for dating purposes has been widely applied in a range of sedimentary contexts, but has often

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provided unsatisfactory agreement with independent age control owing to a series of behavioural problems, notably anomalous fading (see e.g. Duller, 1997). Anomalous fading of feldspar IRSL has been intensively researched in a number of studies (e.g. Spooner, 1992, 1994; Lamothe and Auclair, 1997, 2000; Huntley and Lamothe, 2001), but no successful means of avoiding this malign effect have yet emerged. It has been demonstrated that the red (>600 nm) thermoluminescence (RTL) signal of volcanic feldspar does not suffer from anomalous fading in comparison to blue-UV emissions of the same samples (Zink and Visocekas, 1997; Visocekas, 2000). Assuming a similarity in physical properties of RTL and far-red emission IRSL, Fattahi (2001) has proposed that, as a logical extension of these findings, the far-red IRSL emissions of feldspar could also provide a non-fading alternative to UV-blue IRSL emissions. If successfully exploited this would potentially offer a more accurate means of dating potassium feldspars using optical techniques.

Until recently, exploitation of far-red IRSL emissions for dating purposes has been limited by technical difficulties associated with insufficient rejection of the potentially high background of the measuring system at the detection wavelengths. This problem has recently been successfully circumvented by modification of the photodetection system to include cooled photomultiplier tubes and various far-red signal pass filter combinations (Fattahi and Stokes, 2003a).

Fattahi (2001) demonstrated the possibility of effectively detecting red IRSL emissions by these means, and showed that these wavelengths appeared to exhibit little or no fading in comparison to the blue-UV emission bands on a limited number of coarse grain feldspar samples. However, a more systematic examination of far-red emissions from geological samples has not yet been undertaken. This is necessary for a more thorough investigation of the properties of red IRSL emissions and to assess its suitability as an alternative dating technique. Initial empirical data from part of a larger study is provided here for terrace sediments taken from the upper reaches of the Loire River in France.

3. The Loire terraces

The Loire River (see Fig. 1 of Colls et al., 2001), one of the largest rivers in Europe, exhibits a suite of seven well-developed Holocene through late Pleistocene terraces along its reaches (Straffin et al., 2000). The terraces of the upper reaches of this river, around the confluence of the Loire and Arroux rivers near Digoin, contain chronological control on the deposited sedimentary record (Fig. 1). A programme of optical dating on the terrace sediment quartz fractions and radiocarbon dating of the younger terraces has recently been undertaken (Colls, 1999; Colls et al., 2001), revealing that climatic controls associated with glacial-interglacial cycles have been key in the formation of these features. The resulting optical age estimates generated were both stratigraphically sensible and reproducible, and showed good agreement with radiocarbon dates where available. Direct comparison of ages generated from far-red IRSL emissions of feldspars with UV-blue IRSL ages for these terrace deposits, along with these independent optical and AMS dates from the same samples (see Tables 1 and 3, Colls et al., 2001) therefore provides an ideal and objective basis for systematically assessing the suitability of far-red IRSL emissions for dating purposes.

4. Experimental procedures

Refined sand-sized (90–212 μm) feldspar was obtained from the same samples previously used for quartz optical dating of the terraces by Colls (1999). Samples were prepared using standard feldspar procedures adopted in the Oxford lab (see Colls, 1999 for details).

All readings were made using a modified Risø model TA-15 automated TL/OSL system with a cooled (−20°C) bialkaline D716A PMT (S11 "green tube"), providing a greater quantum efficiency in the far-red portion of the spectrum than a conventional blue-sensitive EMT 9635Q bialkali PMT (Fattahi and Stokes, 2002a). Selection of suitable filter combinations is critical to maximise far-red IRSL emission detection, when using such PMT's with higher quantum efficiencies in the infrared band. Lai et al. (2002) have investigated a range of filter combinations for the detection of far-red IRSL. Optimal far-red IRSL signal-to-noise ratios were found for these particular samples when using a combination of a long-pass RG665 filter and an Omega 740SP heat rejection filter positioned in front of the S11 PMT, providing a detection window from 665–740 nm (Lai et al., 2002).

Aliquots were stimulated using an infrared laser diode unit ($\lambda 833 \pm 5$ nm, 560 mW cm^{-2}). Additionally, an 830 IL12 interference filter and a RG780 filter were positioned in front of the infrared laser diode in order to reduce any incident photons reaching the PMT directly either from the short-wavelength 'tail' of the infrared stimulation source, or as a result of random scattered light and other components (Lai et al., 2002).

5. Initial dating results

Sample D_e determination is based on the SAR protocol (Murray and Wintle, 2000). Six aliquots of each sample were prepared and analysed using four regeneration dose cycles whose values were varied for each sample to 'bracket' best estimates of D_e. A preheat

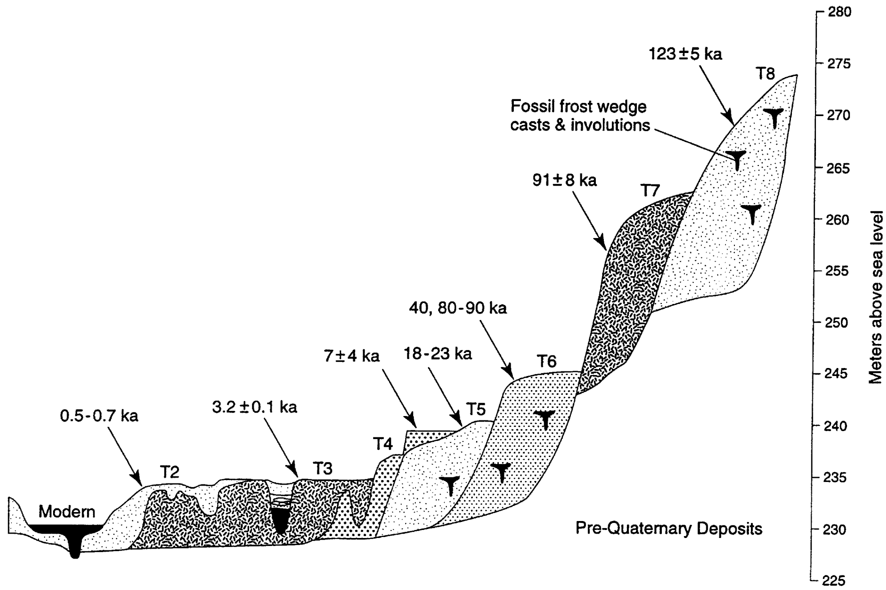

Fig 1. Existing optical (quartz OSL) chronology of the terrace sequence (*source*: Colls, 1999)

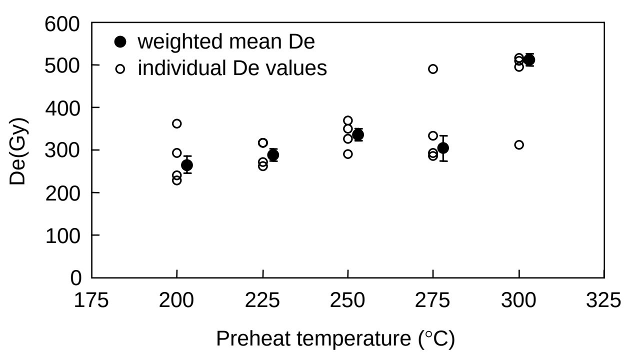

Fig 2. Far-red IRSL emission preheat plateau test for sample 1023/2. Duration fixed at 60 s; 4 D_e values measured on separate aliquots for each preheat temperature; plateau region observed at 200–275°C

of 250°C for 60 s was selected on the basis of far-red IRSL preheat plateau tests undertaken on sample 1023/2 (Fig. 2) (a similar plateau region was also observed in the UV-blue signal for the same sample).

IRSL decay was measured over 100 s at 75°C (see later for discussion), using the first 4 s integral for D_e determination and subtracting the final 20 s integral for background estimation. The same measurement and pre-treatment conditions were maintained for both UV and far-red IRSL emission D_e measurements (performed on separate aliquots) to enable the most direct and simple comparison of resultant D_e and age estimates.

Feldspar dosimetric determinations (Table 1) are based on quartz dose rate calculations for the same samples (found using portable 4-channel NaI gamma spectrometry), with the additional feldspar internal dose rate contributions calculated by assuming a 12% mean potassium content for the feldspar samples and using the appropriate absorption factors for the grain sizes used.

Modification of the photodetection system provided satisfactory detection of the far-red signal above background counts for the first two samples tested. The far-red emission age for sample 1024/1 agrees with the UV-blue emission age within 2 standard errors, although both are an underestimation of the corresponding quartz optical age (Table 1). For sample 1023/2, the far-red IRSL age is an underestimation of both the UV-blue age and the quartz age. The discrepancy between the far-red and UV-blue IRSL ages for this sample may in part relate to inter-aliquot signal scatter (e.g. shown in Fig. 2). UV-blue IRSL ages were in good agreement with quartz OSL ages within errors for most of the 5 terrace units studied, but provided age underestimations for sample 1024/1. Both the far-red and UV-blue IRSL emission ages obtained are stratigraphically sensible.

The far-red IRSL decay curves characteristically displayed smooth, highly reproducible forms (e.g. Fig. 3a). Interestingly, most decay curves displayed an initial peak in the initial channels similar to that often observed when measuring the 'yellow peak' IRSL emissions (Fig. 3a) (Krbetschek, pers. comm.). Growth curves were also generally well-behaved and displayed good recycling ratios (i.e. well-within the ±10% levels deemed to be acceptable) (Fig. 3b), as demonstrated elsewhere by Fattahi and Stokes (2003a).

Table 1
Comparison of initial age estimates from far-red IRSL emissions using SAR with those obtained from UV-blue IRSL emissions, quartz OSL and radiocarbon dating. Errors quoted are at 1 sigma level. Dose rate data based on that obtained by Colls et al. (2001)

Terrace unit	Sample	Feldspar					Quartz			^{14}C dates
		Far-red IRSL			UV-blue IRSL		OSL			Age (ka)
		Dose rate (Gy/ka)	D_e (Gy)	Age (ka)	D_e (Gy)	Age (ka)	Dose rate (Gy/ka)	D_e (Gy)	Age (ka)	
T2	1021/1	8.61±0.21			5.01±0.87	0.58±0.01	8.28±0.20	5.64±0.27	0.64±0.04	0.12±0.05 to 1.42±0.06
T4	1020/2	6.31±0.21			54.5±1.5	9.4±0.4[a]	5.98±0.20	43.8±22.4	7.3±3.75[a]	5.59±0.06
T5	1024/1	5.59±0.19	63.9±4.2	11.4±0.9	81.2±0.2	12.9±0.4	5.26±0.18	108.3±8.5	20.6±1.8	
T6	1023/2	5.32±0.19	280.8±17.6	52.8±3.8	352.6±8.6	66.3±2.9	4.99±0.18	429.9±24.8	61±2.5	
	16/1	3.99±0.1			529.6±48.9	133±12	3.66±0.09	450.4±12.9	123±5	

[a] Age estimates based on minimum rather than weighted mean D_e values owing to presence of partial bleaching in sample.

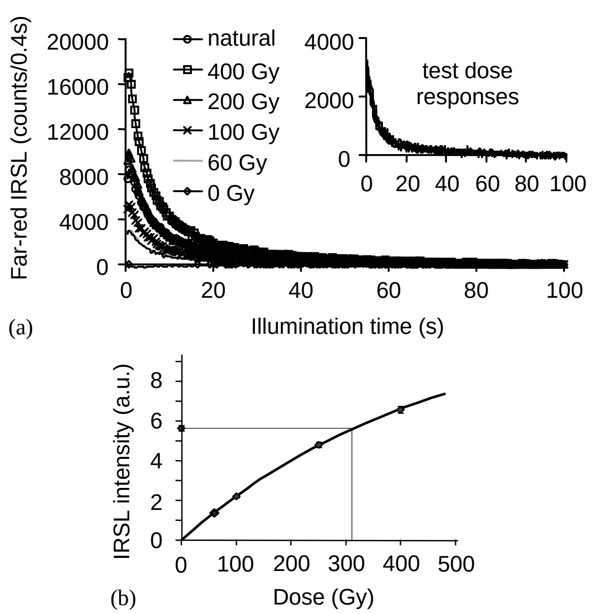

Fig 3. Characteristic far-red IRSL emission (a) decay curve (minus background). Inset: consistent test dose responses for natural and regenerative doses shows absence of major sensitivity change (b) growth curve (fitting: exponential plus linear) for sample 1023/2

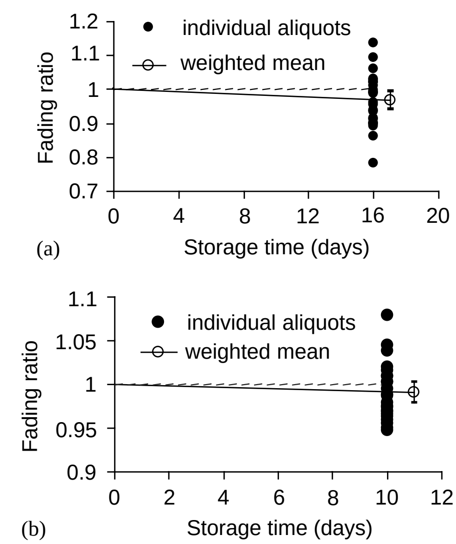

Fig 4. Results of anomalous fading storage tests for (a) far-red IRSL emissions (b) UV-blue IRSL emissions. Fading ratio: pre-storage/post-storage sensitivity corrected IRSL signal; 24 aliquots of sample 1023/2 used in each case; preheat of 250°C for 60 s used prior to IRSL signal measurements.

6. Evaluation of anomalous fading

Short-term storage tests were used to detect whether or not the far-red IRSL signal of the samples were afflicted with anomalous fading. Fading was checked by comparison of sensitivity corrected IRSL signals after irradiation of 100 Gy and storage, with that measured immediately following initial irradiation (i.e. before storage). Fading tests were carried out for both blue and red IRSL emissions to provide a direct comparison of anomalous fading attributes between the two signals. 24 aliquots of sample 1023/2 (chosen for its bright far-red IRSL signal characteristics, abundance of material available, and its extensive use in previous far-red IRSL investigations) were stored at c.70°C for 16 days for the far-red IRSL emission test, and 10 days for the UV-blue IRSL emission test.

No statistically significant fading was identified over the short storage times for either the far-red (Fig. 4a) or UV-blue (Fig. 4b) IRSL emissions for sample 1023/2. The existence of anomalous fading cannot be completely ruled out from these initial experiments, given the short duration of the lab storage tests compared to natural signal storage times, but its absence in the large number of aliquots monitored is encouraging support that this feldspar sample exhibits a stable, non-fading optical signal. Apparent age underestimations of the far-red IRSL ages compared to quartz optical ages for the two samples measured is not therefore believed to be attributable to anomalous fading. Longer storage tests are planned using a range of different samples whose

UV-blue IRSL signal is known to fade, for a more thorough assessment of far-red fading characteristics.

7. Far-red IRSL dating considerations

7.1. SAR suitability

Inadequate correction of sensitivity change by the SAR protocol could also potentially account for far-red (and UV-blue) IRSL age underestimation compared to quartz ages for the samples. In order to demonstrate the applicability of the SAR procedure for D_e determination using far-red emissions two tests were undertaken. Firstly, recovery of a known dose was attempted on 4 aliquots of sample 1023/2 using a modified SAR protocol involving 3 instead of 4 regenerative dose points. A 100 Gy laboratory dose was successfully recovered for the aliquots tested (e.g. Fig. 5a), yielding a weighted mean D_e of 105 ± 7 Gy and a mean recycling ratio of 0.97.

Secondly, to test whether SAR is adequately correcting for any sensitivity change in far-red IRSL D_e determination we undertook repeat measurements of a single dose over numerous cycles (Fig. 5b). The results show that test dose correction of sensitivity change is satisfactory over 5 repeat dose cycles, with variation of less than 5% observed for the aliquots tested. Monitoring of test dose responses to natural and regenerative doses during D_e determination using SAR also showed the absence of any major sensitivity changes for the samples at different measurement stages (see inset Fig. 3a), and the "internal checks" of SAR (i.e. recycling and recuperation ratios) were satisfactory for the samples tested. Together this evidence suggests that inadequate sensitivity correction by SAR for far-red IRSL D_e determination is not an issue in these cases.

7.2. Background signal

Repeat measurements of IRSL signals on aliquots following main natural and regenerative dose IRSL signals were used to monitor background. These revealed that a non-constant far-red IRSL background signal was a common feature in the initial stimulation channels for these samples. Tests using un-dosed discs revealed that this non-constant initial background has a strong dependency on both IR stimulation power (Fig. 6a) and measurement temperature (Fig. 6b), with a greater initial decay observed at lower temperatures and higher stimulation powers. These observations prompted the adoption of a measurement temperature of 75°C, and the use of 60% IR stimulation power during the D_e determination described earlier.

These results suggest that it is not advisable to use normal 'late-light subtraction' in far-red IRSL emission measurement. Modification of the SAR procedure to directly measure the background signal for subtraction using a repeat IRSL measurement following the main natural or regenerative IRSL signal measurement is therefore recommended for D_e determination.

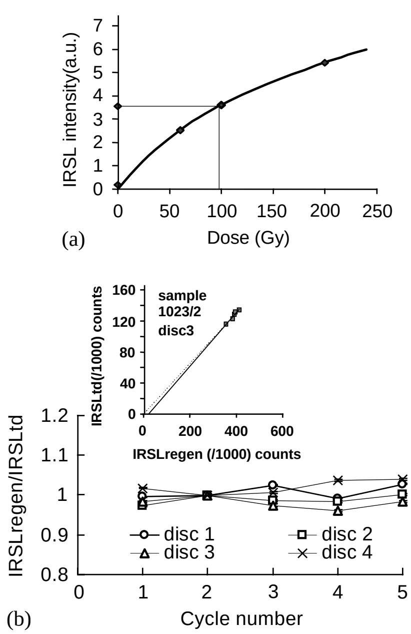

Fig 5. (a) Example (100 Gy) dose recovery growth curve for an aliquot of sample 1023/2 using 3 regenerative points of 60, 100 and 200 Gy, (b) repeat measurement of a single 100 Gy dose, indicating adequate sensitivity correction by use of a 50 Gy test dose. Preheat used: 250°C for 60 s.

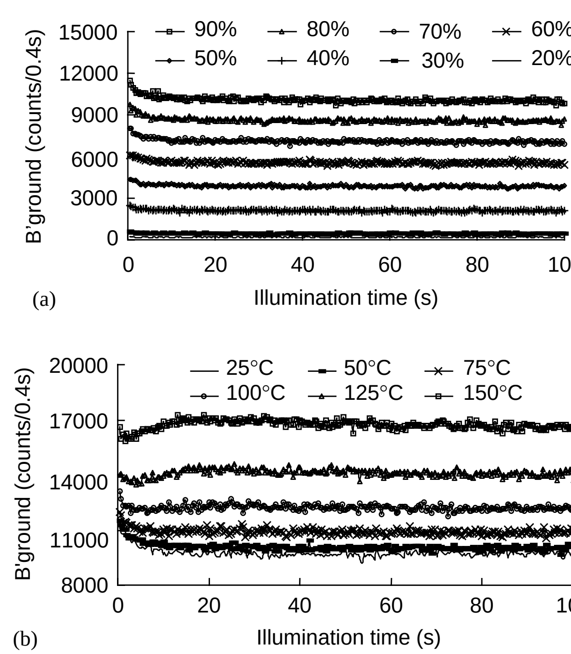

Fig 6. Far-red IRSL non-constant initial background signal dependence on (a) IR stimulation power and (b) IRSL measurement temperature.

8. Conclusions

1. Far-red IRSL emissions are present and detectable in both natural and laboratory dose signals of feldspar samples from the Loire River terraces. This supports similar findings by Fattahi and Stokes (2003b) for other fluvial samples and Lai et al., (2003) for fine-grained aeolian samples. Together this provides strong evidence for the ubiquitous nature of far-red IRSL signals in natural feldspar samples.
2. Initial far-red IRSL age estimates are not in total agreement with quartz OSL or UV-blue IRSL ages, but results are encouraging and samples show well-behaved and reproducible growth curves.
3. Apparent age underestimations are not attributable to inadequate sensitivity correction by SAR or to anomalous fading of the far-red signal. However, longer storage tests are needed to more thoroughly assess far-red fading characteristics.
4. Refinement of pre-treatment and measurement conditions should enable more successful dating of coarse grain feldspars using far-red IRSL emissions. Initial investigations into laboratory lighting conditions indicate a potential for far-red IRSL natural signal depletion, and this may account for the far-red age underestimations observed. Modification of background signal subtraction methods should also provide improved D_e determination. Work is ongoing to date more of the terrace units using far-red IRSL emissions

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