Red luminescence emission from potassium feldspars stimulated by infrared

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Abstract

The use of the blue–UV Infrared stimulated luminescence (IRSL) from feldspars in luminescence dating has generally been unsuccessful due to age underestimation related to anomalous fading. The red emission from feldspar may however provide a non-fading alternative. A series of experiments are reported here which investigate IR stimulated (λ = 833 ± 5 nm) red luminescence emission from potassium feldspar. The aim is to demonstrate the potential of red IRSL as an alternative to the widely used UV-blue emission IRSL approaches. Five key factors are optimised to increase the dose-related red IRSL signal, and to decrease the background signal levels. These include photomultiplier tube (PMT) characteristics, detection filter combinations, laser diode intensity, measurement temperature, and substrate related effects. Preliminary measurements are described that illustrate the considerable potential of red IRSL for dating applications, including low sensitivity changes from volcanic potassium feldspars and high dose saturation of red IRSL from sedimentary and volcanic potassium feldspars.

Keywords

Luminescence dating; feldspar; red emission; infrared; orange- red IRSL

Introduction

Huntley et al. (1985) demonstrated the possibility of obtaining an optically stimulated luminescence (OSL) signal from feldspar, and the use of OSL in dating applications is now well established. Hütt et al. (1988) published the first optical stimulation spectrum for feldspar showing a large stimulation peak in the near IR (825-1030 nm). This unexpected stimulation peak in the near infrared for feldspars was confirmed in later studies and by a number of authors (see Table 4 and Figure 9 of Duller, 1997; Wintle, 1997; Krbetschek et al., 1997).

The IR stimulated luminescence emission spectra vary considerably round the ternary feldspar compositional diagram (Duller, 1997). Krbetschek et al. (1997) reported emission bands at 280, 330, 410, 560 and 700 nm for the IRSL of potassium-rich feldspars extracted from sediment samples. With respect to signal detection, the commonly used EMI 9635Q PMT is biased towards detection in the blue-violet range with 25% quantum efficiency at 400 nm, around six times higher than that at 570 nm. Luminescence workers have used this capability primarily for the detection of blue-violet potassium feldspar emissions (e.g. Duller, 1994a,b, 1997).

Due to several potential advantages, including the speed of resetting of the luminescence clock by exposure to sunlight (Li and Wintle, 1994), the use of IRSL for dating sediments has attracted considerable attention. However, despite over a decade of research, satisfactory agreement with independent age control has been shown only in a few studies (e.g. young samples in Duller, 1994b; Clarke et al. 1996). In particular, the application of infrared stimulation blue luminescence (IRSL) has been hampered by anomalous fading and the associated problem of age underestimation (e.g. Balescu et al., 2001; Huntley and Lamothe 2001; Lamothe and Auclair, 1997, 1999, 2000; Lian and Shane, 2000; Richardson et al., 1997, 1999; Spooner, 1992, 1994; Wallinga et al., 2000).

Zink and Visocekas (1997) have shown that red (which they termed near-IR) (λ > 590 nm) thermoluminescence (RTL) of at least some species of feldspar do not suffer from anomalous fading, whereas blue emissions (λ < 590 nm) from identical samples do exhibit the effect. They demonstrated that RTL emission in sanidine feldspar possessed a number of characteristics similar to that of the blue range emission. These included similarities in activation energies and sensitivity to sunlight bleaching, with both signals bleaching rapidly in a few hours of exposure. Fattahi (2001) has shown that orange-near red IRSL (λ = 600-660 nm) shows either no or less anomalous fading in comparison with UV IRSL for the feldspar samples examined. A logical extension of these works would be, as has been the case for UV-blue emission of both quartz and feldspar, to establish whether it is possible to observe the red emission under optical stimulation and if so, determine whether red IRSL possesses similar or different characteristics in comparison to UV-blue IRSL. Technological difficulties in developing a system which is capable of detecting in the red, while rejecting other wavelengths, have restricted further research in this area (Duller, 1997). The primary technical limitation in red IRSL detection is the potentially high background of the measurement system. This background is mainly due to two sources: reflection of the incident IR stimulation photons, and thermal incandescence.

Given its potential to circumvent anomalous fading, we consider the development of a suitable detection system and the testing of IR stimulated red emissions to be a priority for luminescence dating and one which we have systematically evaluated (Fattahi, 2001). As with conventional OSL of quartz and IRSL of feldspars, practical advantages in exploiting anti-Stokes radiation encouraged us, in combination with the well-known feldspar stimulation IR resonance, to first consider the use of IR stimulation wavelengths to detect a red emission.

This and a companion paper (Fattahi and Stokes, 2003b) present results of our attempts to assemble suitable optics and maximise an infra-red stimulated red luminescence emission from feldspar. Our ultimate aim is to validate the signals as routine integrating dosimeter for dating purposes.

In this paper we use ‘‘ Red’’ as a name to describe all detection windows used in this study between 600-720 nm; ‘‘IRSL’’ as a name to describe the conventionally employed UV-blue IRSL ($\lambda_{emission}$ < 600 nm); ‘‘orange- red IRSL’’ to describe IRSL in the wavelength emission region c. 600-720 nm; “orange-near-red IRSL” to describe IRSL in the wavelength emission range 600-660 nm; and “far-red IRSL’’ to describe IRSL in the wavelength emission range 665-720 nm.

Experimental conditions

A full description of our experimental apparatus is given in Fattahi and Stokes (2003b) and only a summary is provided here. Experiments were carried out using a Risø TL-DA-15 automated TL/OSL system (fitted with a $^{90}Sr/^{90}Y$ beta source delivering ~7 Gy.min^{-1}). Three photomultiplier tubes were used. These consisted of either an Electron Tubes 9635Q (hereafter called the “blue”) tube, or one of two cooled (T ~ -20°C), extended range PMTs: an Electron Tubes D716A (hereafter called the “green” tube) bialkaline PMT; or an Electron Tubes 9658 (hereafter called the “red” tube) S20 PMT, that are equipped with an S 600 PHOTOCOOL thermo-electric refrigeration chamber which allows active cooling of the photocathode down to 40°C below room temperature.

The system incorporates a powerful focused IR laser diode stimulation source, which provides incident photon energy of up to ~ 400 mW.cm^{-2} at the sample, at a wavelength of 833 ± 5 nm. Luminescence was measured through filter combinations designed to transmit a variety of broad (λ= 590-720 nm) or narrower (λ = 600-660 nm λ = 665-720 nm) red emissions (Fattahi and Stokes, 2003b).

The potassium feldspar samples used in this study were collected from a variety of geographical locations. Sample (OX_{OD} 847/5) is a late Holocene New Zealand dune sand. Sample 99/5/1 is a Mid Pleistocene New Zealand Ignimbrite. Four samples were obtained from terrace deposits in the Upper Loire Valley, France (15/1, 15/2, 14/1 and 21/1), kindly provided by Alison Colls. A fluvioglacial sample from Ontario (Middle Wisconsinan deposits near St. Thomas, Ontario, Canada with Laboratory identification: Y7A). Six further samples were obtained from UAE (WW1A, 2A, 3A, 4A, 1B and WW2B).

Optimisation of the IRSL (>600 nm) emission

We identify five main parameters that affect the signal and background: sample temperature including thermal incandescence, intensity of the IR stimulation source, IR reflection of substrate surface, and PMT and filter combination characteristics. We have considered the effect of these parameters individually, and in combination, for efficient red IRSL detection.

The effect of sample temperature and IR laser diode intensity on background

It is obvious that the probability of eviction of electrons from traps depends on the intensity of the stimulation source. Different models have been suggested for the production of IRSL in feldspar, including a thermally–assisted photon process (Hütt et al., 1988; Trautmann et al., 2000; Poolton et al., 2002a,b). However, the amount of thermal assistance is strongly dependent upon the optical excitation energy chosen (Poolton et al, 2002a). Doubling the stimulation power will double the rate of arrival of photons and therefore the luminescence emitted per unit time from a given trap (Aitken, 1998). Increasing the sample temperature (stimulation temperature) will also increase the eviction of electrons. According to Aitken (1998), near room temperature this eviction rate increases by the order of 1% per degree centigrade. Increasing the stimulation temperature and light intensity therefore has the advantage, for dim samples, of increasing the signal relative to the background due to PMT noise. The potential disadvantages of increasing the IR diode intensity and sample temperature can be divided in two categories. Firstly, the background will increase, with temperature and photon flux, which are themselves due to thermal incandescence and reflection of stimulating photons from a sample. Secondly, thermal quenching, if present, decreases the luminescence centres efficiency as the temperature is increased (e.g. Poolton et al., 1995). As such, our attention has focused on minimising thermal incandescence and reflected stimulation photon flux.

Thermal incandescence has previously hampered the application of the red emission in thermoluminescence dating (e.g. Fattahi and Stokes, 2001; 2003a). The effect of IR reflection originating from a typical 1W infrared (830 ± 5 nm) laser diode unit is an order of magnitude greater than the effect of thermal incandescence (c. 10^{-2} J.m^{-3}) at ~ 500°C at 900 nm, as a background component (Fattahi and Stokes, 2003b).

To explore the effect of IR laser diode intensity on background, we have examined a variety of substrate types while varying the laser intensity (0-100%). For this experiment we used four disks: two aluminum (Al) disks, one of which was painted black (using a marker), and two stainless steel (SS) disks, one of which was again painted black. Figure 1 demonstrates that there is a significant IR intensity-related background signal. It is additionally noted that at higher IR intensities there is an initial small decay component in this background, which is probably related to IR source characteristics. This feature requires further investigation. Figure 2 compares the typical effect of the laser intensity at 30%, 60% and 90% on the 4 disks. Similar patterns are observed for other filter combinations and PMT (but with background intensities varying from some millions to only 100 counts per second). Clearly background represents a significant issue in red IRSL detection system design.

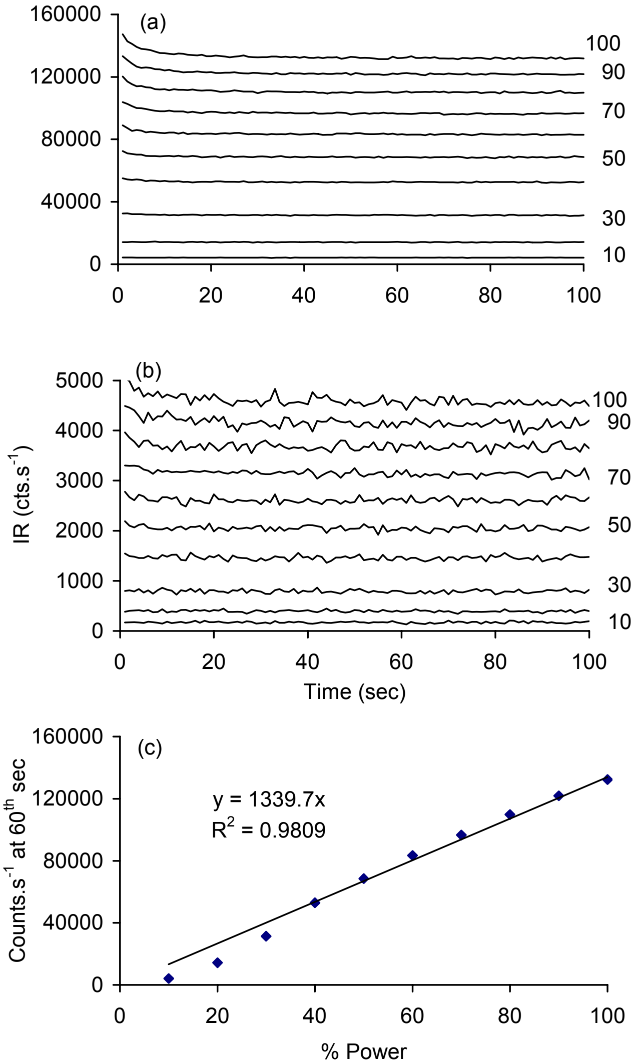

Figure 1: *Reflected IR from a blank stainless steel disk using red PMT and two sets of filter combinations. (a) Omega 740 SP + FR 400S + OG590, (b) Omega 740 SP + 2*FR 400S + OG590. (c) Relationship between the IR counts and laser out put of figure (a). The labels on the data lines give the intensity (% of maximum) of IR laser output. In all groups of filter combinations examined, the same pattern has been observed.*

The widely employed Pilkington HA-3 heat rejection filter used in UV-blue TL and OSL studies cannot prevent the IR transmission in red luminescence studies using a conventional "blue" bialkali PMT (Figure 2a). This confirms previous results reported by Fattahi and Stokes (2000).

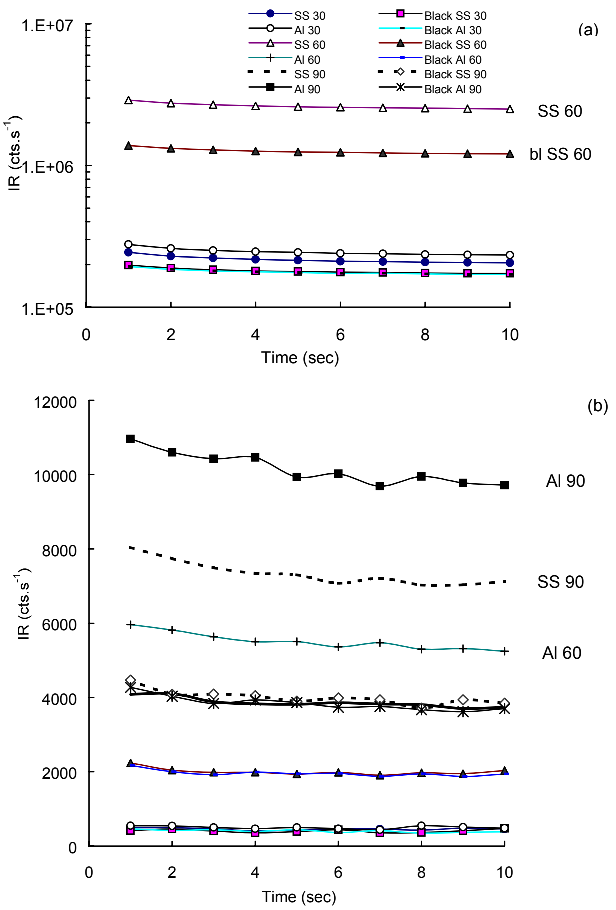

Figure 2: *Reflected IR from, two aluminium (Al) disks, and two stainless steel (SS), one of which is painted black, using two filter combinations. (a) For HA3 + OG 590. (b) For FR 400S + OG 590. The labels on the data lines give the disk used and the intensity (% of maximum) of IR laser output.*

A combination of Omega 740 SP + Corion FR 400S + OG 590 and the "red" PMT decrease the background at 100% laser intensity from > 10^6 to ~1.5 x 10^5 cts.s^{-1} (Figure 2a). Addition of another FR 400S reduces it to ~5 x 10^3 cts.s^{-1}.

Some combinations of IR cut filters are able to decrease the background from in excess of millions of counts per second to a low level comparable to PMT dark noise (a few hundred counts per second). The general pattern noted is that reflected IR is high for both unpainted Al and stainless steel substrate, the former consistently being greater. The levels of IR reflection are clearly related to the IR intensity (Figure 2b). However, when the disk surfaces are painted black, while a background dependence on IR intensity remains, we obtain considerably lower backgrounds (at least half) than for the unpainted discs, and there is no significant difference observed between the two substrate types, as expected. This suggests that further investigation is required to find a substrate that is more suitable for red-IRSL measurements than aluminium or stainless steel. Didier Miallier (personal communication) has investigated that a stainless steel disk that has been exposed to air for a while will always give a significant spurious signal when heated; for circumventing this effect, the disk must be preheated once shortly before a set of measurements.

For some filter combinations, background levels were as low as 100-200 cts.s^{-1} but the signals are correspondingly lower and this limits potential dating applications. A key observation is that background is mainly due to the reflection of incident light, which is directly related to the IR source intensity. In comparison, the PMT dark count is negligible. Therefore, the optimum configuration for red IRSL measurement is use of a substrate with minimum IR reflection, while applying the maximum IR intensity.

The effect of filter and PMT combinations on red IRSL measurements

A range of filter combinations and PMTs was tested using sample 847/5 (Fattahi, 2001). The result demonstrated that each PMT needs a specific filter combination to optimise the signal and signal to noise, and comparing three PMTs with a single filter combination will not demonstrate the optimum results for all tubes.

While there is at present no definitive choice, we consider the green PMT in combination with two FR 400S plus OG 590 or RG 665 as an efficient arrangement for detection of red IRSL decay curves over a broad red emission range (~ 600-720 nm) or far-red (~ 665-720 nm) of spectrum, respectively. An Omega 625DF50 alone, in combination with the green PMT, is suitable in cases where details of the entire decay form and low background levels are required in orange-near-red (~ 600-660 nm) part of the spectrum. If a green PMT is not available, we suggest the red PMT in combination with two FR 400S + BG 39 (1mm) plus OG 590 or RG 665 for detecting of orange-red or far-red IRSL decay curves, respectively. Alternatively, for very bright samples if neither the red or green PMT are available, an Omega 625DF50 or Omega 750 SP + OG590 filters combined with a blue PMT may provide a usable combination for orange-near red portion of the spectrum. The combination of Omega 750 SP + RG665 filters and a blue PMT may provide a usable combination for far-red IRSL detection. While this would result in a very low signal yield, it has the advantage of not requiring active cooling.

Suggestions for stimulation source investigations

The most significant contribution to the background signal is from the IR source, due to the close position of the detection (600–720 nm) and stimulation (~ 830 nm) wavelengths and high intensity of the IR laser. Figure 3 shows the characteristics of the IR laser source used for this study. In our opinion the short tail of the IR laser wavelength has an enormous effect on background.

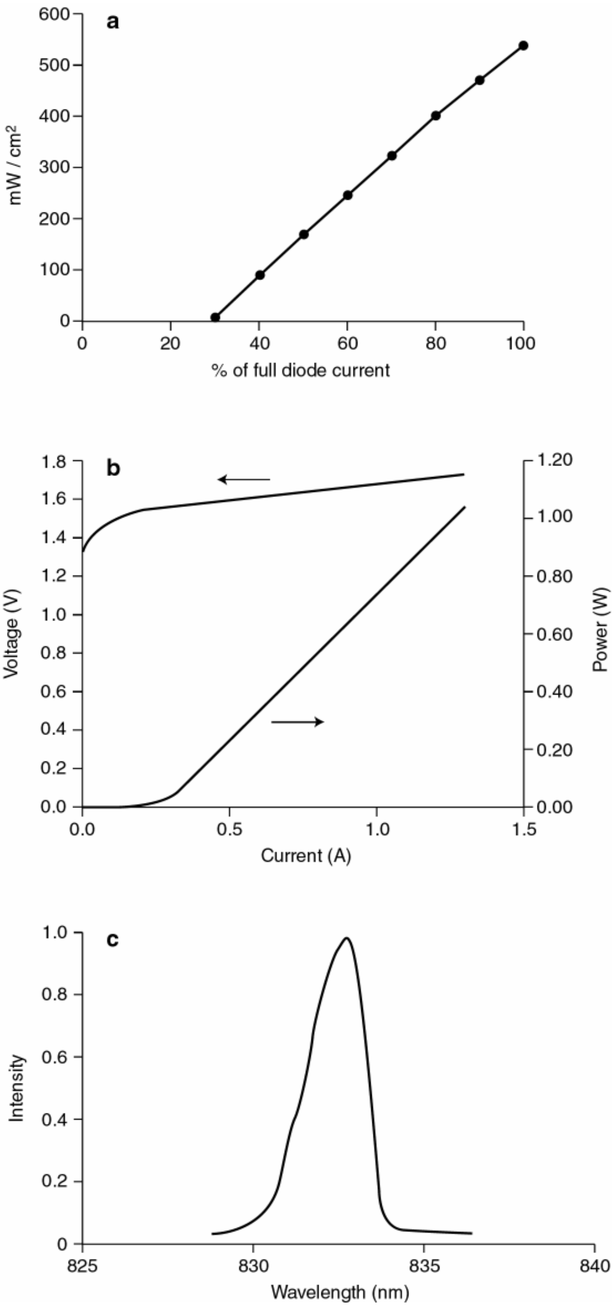

Figure 3: *HPD1110-9mm IR laser characteristics. (a) from Risø catalogue. (b) and (c) from High Power Devices catalogue.*

A variety of IR-cut filters (employed in this study) have been used to reduce the background from IR laser source. But, these filters have also reduced signal detection efficiency from samples. Therefore, the use of IR-cut filters has not satisfied both high signal level and signal-to-noise ratio. There are two alternatives for increasing the signal to noise and reducing the background. Firstly, we suggest using another laser diode that stimulates at higher wavelengths (i.e., > 850 nm). Secondly, we suggest the use of a combination of long-pass (short-cut) and interference filters in front of the HPD1110-9 mm IR laser.

An examination of some basic red IRSL and RTL properties of potassium feldspar

Figure 4 shows the typical stimulation and detection bands used for the following section of this study.

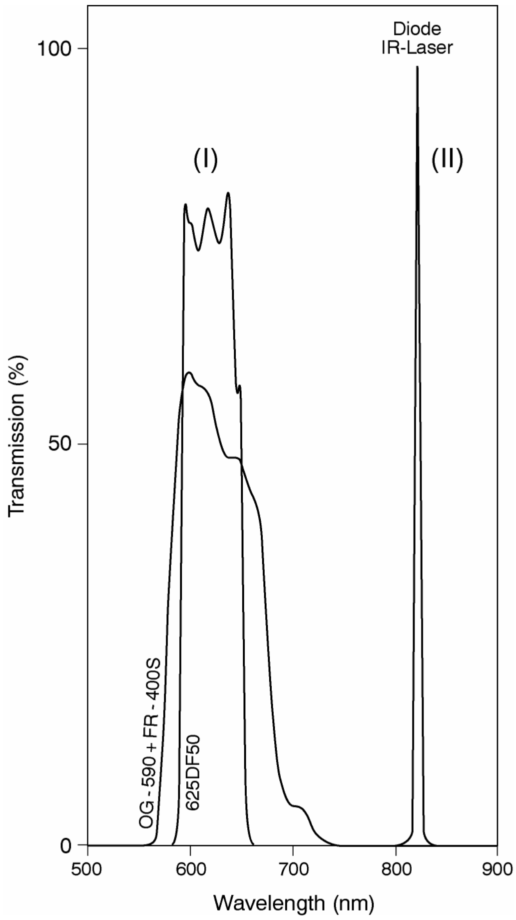

Figure 4: *The stimulation (II) and detection (I) bands used in this study. (I) Transmission through detection filters (Omega 625DF50 and a combination of OG590 + FR 400S). (II) IR stimulation source spectrum (833 ± 5 nm).*

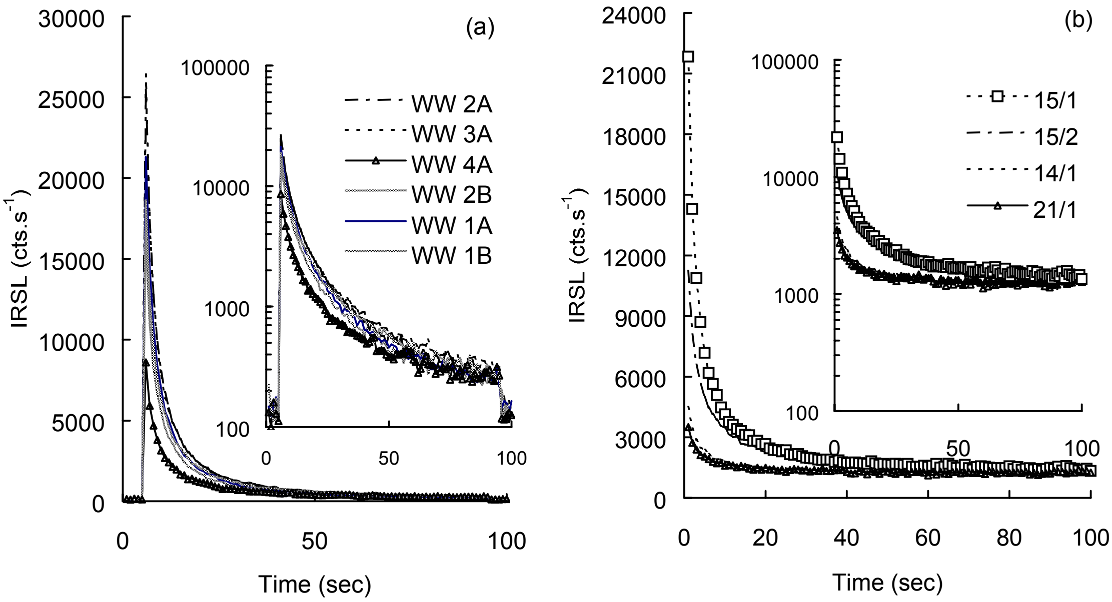

Figure 5: *Natural red IRSL decay curves (pre-heated at 250°C for 60 s) of feldspar samples collected from UAE (WW 1-4A and WW 1-2B) and France (15/1, 15/2, 14/1 and 21/1), respectively. (a) UAE samples detected through EMI 9635Q "Blue" tube and OG 590 + BG 39, inset shows the same curves on a logarithmic y-axis scale. (b) French samples detected through D716A "Green" PMT and RG 665 + FR 400S + BG 39.*

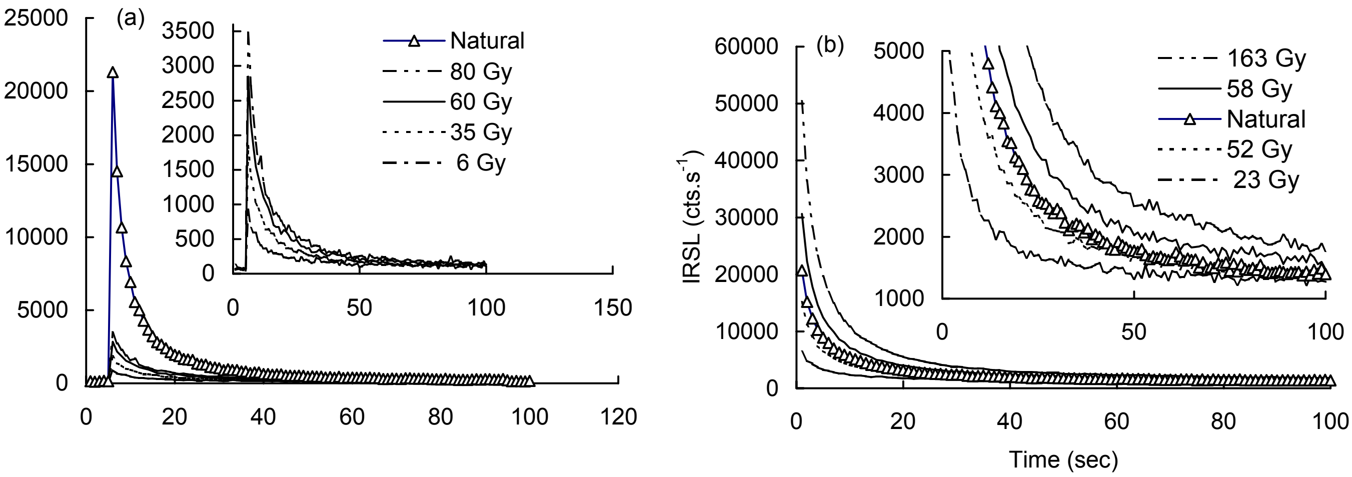

Figure 6: *Natural and laboratory red IRSL decay curves of (a) WW1A sample detected through EMI 9635Q "Blue" tube and OG 590 + BG 39. Inset shows extended y-axis scale curves. (b) 15/1 sample detected through D716A "Green" PMT and RG 665 + FR 400S + BG 39. Inset shows extended y-axis scale curves.*

Observation of the red IRSL decay form

Exposure of natural and laboratory irradiated feldspars to 830 nm IR (80% of maximum intensity) results in the prompt emission of red IRSL, which decreases to near background during the exposure of 100 seconds. Figure 5 shows the red IRSL decay curve observed for a natural feldspar sample measured at 150°C. Natural and laboratory irradiated red IRSL decay curves of samples WW1A and 21/1 are shown in Figure 6. Both curve fitting analysis of these continuous wave red IRSL decay curves and LM- red IRSL analysis indicate that the decay form is best modelled by the sum of three exponential forms with different physical properties (e.g., decay rate and saturation with dose) (Fattahi, 2001).

Red emission IRSL and Red TL sensitivity changes

To explore red emission IRSL and Red TL sensitivity changes, Fattahi (2001) examined the red IRSL decay curves and RTL (0-500ºC) glow curves of two heated potassium feldspar (OX_{OD}847/5 and 99/5/1) by repeat measurements (dose-IRSL-RTL) on samples. He demonstrated that IRSL decay curves and RTL glow curves are highly reproducible.

However, this is valid only for repeated experiments in the laboratory. For dating applications, the sensitivity change between the natural process of irradiation and the laboratory irradiation, unfortunately, can not be asserted, even if no sensitivity change has occurred in the lab.

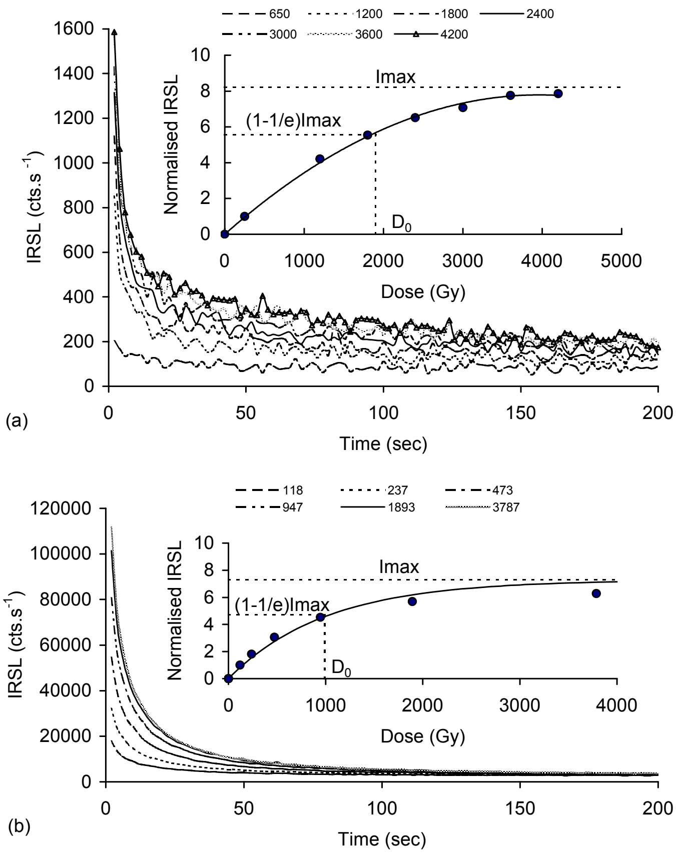

Figure 7: *Regenerated red IRSL decay curves of feldspar extracted from sample 99/5/1 and WW1A potassium feldspars. (a) Top shows the added dose in Gy for 99/5/1 sample. Inset shows the growth curves plotted for initial (first second), D_0 is the characteristic saturation dose. The detection system was D716A "Green" PMT and Omega 625. (b) The same as (a) but for far-red IRSL fromWW1A sample. The detection system was "Green" PMT and RG 665 + FR 400S + BG 39.*

Red IRSL Dose Response

An important potential advantage of red IRSL is its high dose response characteristic. We have constructed regenerated growth curves over extended dose ranges. Figure 7 summarises the red IRSL decay and growth curve data for 99/5/1 and WW1A potassium feldspar samples. The orange-near-red and far-red IRSL exhibits continued growth with doses up to 4 and 2 kGy, respectively. Using the first second of red IRSL minus the background (average of the last 5 seconds) suggests that the growth form is well modelled by a single saturating exponential. The characteristic saturation dose (D_0) for samples 99/5/1 and WW1A are 1800 and 1000Gy, respectively. If these values are taken as representing the maximum range of "easily utilized" dose, and we assume typical environmental dose rates of 1-2 Gy.ka^{-1}, the useable age range of the dosimeter, assuming trap stability over the same period, is in the order of 0.5-2 million years. While for these samples the dose-response data are well modelled by single saturating exponentials model, other results described elsewhere indicate that in some cases an exponential plus linear model provides a better fit.

It should be mentioned that Miallier (personal communication) has found that in some cases, for RTL of old quartz samples, the growth curve has not at all the same shape as the growth curve for samples reset in the lab. He believes that this is probably due to "ageing effects" that will take a long time to become significant (e.g., internal new traps creation under radiation, or defects diffusion) and that are annealed by heating.

The effect of measurement temperature on red IRSL signal

The effect of measurement temperature on orange-near-red and far-red IRSL was examined using sample WW1A, a potassium feldspar. Both IRSL signals showed almost identical pattern. The red IRSL signal first increased up to a sample temperature of 120°C and then decreased to a minimum close to 300°C. Here, we explain the details of the experiments that we performed. Similar experiments were performed using both detection windows.

The red IRSL decay curves of laboratory irradiated WW1A sample, which had been given a 240 Gy dose, were measured during 100 s illumination with IR, while the sample was held at temperatures from 20 to 460°C in 20°C steps (Figure 8). To observe possible phosphorescence components and any thermally stimulated contribution to the red IRSL signal, the aliquot was held at the stimulation

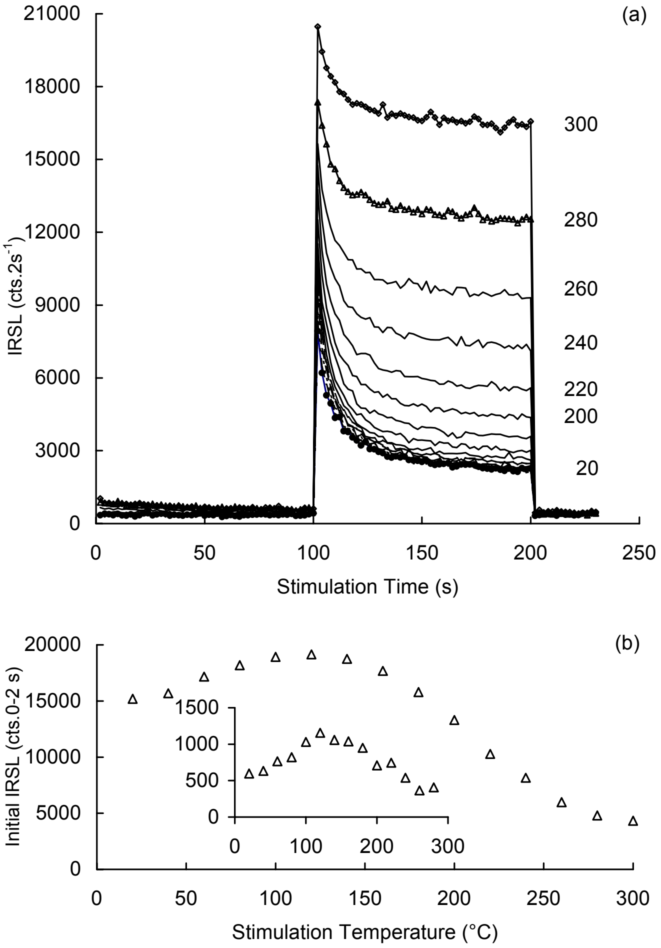

Figure 8 : *The effect of stimulation temperature on red IRSL. (a) The orange-near-red IRSL obtained for potassium feldspar (WW1A), which had been given a 240 Gy dose, before every orange-near-red IRSL measurement at elevated temperature from 20 to 460°C in 20°C steps. (b) Variation in net initial (100-102 s less background estimated from 190-200 s) laboratory irradiated orange-near-red IRSL signals obtained for 100 s at stimulation temperature (after subtracting the data without illumination from the data collected with IR) plotted versus stimulation temperature. A dose of 240 Gy was given prior to every IR stimulation (see text). Orange-near-red IRSL was observed through D716A "Green" PMT and Omega 625. Inset shows the result of similar experiment but using D716A "Green" PMT and RG 665 + FR 400S + BG 39.*

temperature for 100 seconds prior to the red IRSL measurement. After the red IRSL measurements, the luminescence was measured for 30 seconds, at the stimulation temperature but with no illumination, to observe any thermally assisted component. As can be seen in Figure 8a, increasing the temperature significantly increases the background of IRSL (100–200 s). In comparison, the thermal effect (0-100 s and 200-230 s) is very low. This difference could be explained by several possible mechanisms including increases in the IR reflection at higher sample temperatures and thermally sensitive deep traps providing electrons to IR sensitive shallower traps. This component is significantly greater than instrumental and thermal background, and was subtracted from the red IRSL measurements by subtracting the data measured with no illumination from the data measured with IR (100-200 s).

Variations in net initial (100-102 s less background estimated from 190-200 s) laboratory irradiated orange-near-red emission signals are plotted versus stimulation temperature in Figure 8b. The inset shows the result of the same experiments, using far-red IRSL signals. An initial increase by ~ 33% (for orange-near-red) and 150% (for far-red IRSL) of the original signal in the net integral between 20-120°C is followed by a decrease of ~ 66% (for orange-near-red) and ~ 50% (for far-red IRSL) compared with their original IRSL intensity between 120-300°C. Based on Figure 8b, it is advantageous to elevate sample temperature during measurement (c. 120°C). An elevated measurement temperature might additionally remove any phototransfer component in low temperature traps, as has been described in quartz OSL studies (e.g., Murray and Wintle 1998), but could additionally result in complications relating to the thermal erosion and thermal quenching of signal. In the case of orange-near-red IRSL this increase might be due to the effect of yellow peak transfer to higher wavelengths with increasing temperature (Rieser et al., 1997). These factors are discussed elsewhere (Fattahi, 2004).

Conclusions

While IRSL from feldspar has attracted considerable attention for dating applications, satisfactory agreement with independent age control for most samples has been hampered by a series of behavioural problems, in particular, anomalous fading (Duller, 1997). It is reported that RTL (>600 nm) of feldspar does not suffer from anomalous fading (Visocekas, 2000). We have systematically examined the possibility of observing red IRSL from potassium feldspar as an alternative approach. Assuming that there is a similarity in physical properties of RTL and red IRSL (as has been the case for UV-blue emission of both feldspar and quartz), the resulting signal may not exhibit anomalous fading (tested in Fattahi and Stokes, 2003c), and be suitable for dating applications (Fattahi and Stokes, 2003d).

In order to maximise red IRSL measurements, the data suggest that efficient PMT/filter combinations are of the bialkaline D716A PMT + two FR 400S, and either OG 590 or RG 665 for wide red (600-720 nm) or far red (665-720 nm), respectively. The bialkaline D716A PMT and an Omega 625DF 50 filter is the optimal combination for orange-near-red (600-660 nm).

Among the other factors controlling the signal and background are IR intensity and wavelength, substrate IR reflectivity, and measurement temperature. The major findings regarding these three give rise to the following: (1) Increasing IR intensity significantly increases both signal and background. (2) Decreasing IR (830 nm) reflectivity of substrate, can significantly decrease the background. (3) Increasing the stimulation temperature increases the signal and background. Much of the thermal incandescence can be avoided using suitable IR cut filters.

The system can easily observe red IRSL decay curves from natural and laboratory irradiated feldspars, and provides a capability of detecting red IRSL from doses as small as 6 Gy.

Preliminary investigations have shown that the red IRSL characteristics of potassium feldspars are very promising for dating applications, including reproducibility of IRSL curves, well-behaved growth curve form, and high dose saturation.

We believe that red IRSL, and particularly emissions > 665 nm, open up interesting new areas for luminescence research, which should be fully tested as an alternative to the generally poorly behaved UV-blue emission from feldspar.

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