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Single grain infrared photoluminescence (IRPL) measurements of feldspars for dating

G.A.T. Duller a,∗, M. Gunn b, H.M. Roberts a

a Department of Geography and Earth Sciences, Aberystwyth University, UK
b Department of Physics, Aberystwyth University, UK

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ABSTRACT

Existing infrared photoluminescence (IRPL) systems have used pulsed infrared stimulation (~830 nm) and measured IRPL emission (at 880 or 955 nm) using time resolved data collection with photomultipliers. Breakthrough of the infrared stimulation light overwhelms the IRPL, but the delayed emission during the laser-off period has been used instead.

This paper describes a system for measurement of the IRPL signal from single sand-sized grains of feldspar. The attachment uses an electron-multiplying charge-coupled device (EMCCD) imaging system, and has two innovations that make it possible to use such a detector to obtain IRPL data. First, the optical detection system has been designed to minimise stray light and maximise the efficiency with which filters reject the stimulation light. This acts to reduce, but not eliminate, the breakthrough. Second, by placing the sample to be measured in a clearly defined sample grid, the spatial resolution provided by the EMCCD has been used to differentiate between regions of the image where IRPL is emitted and adjacent regions where only breakthrough is expected. This allows quantification of the breakthrough and effective subtraction to isolate the IRPL signal from the grains of interest.

The attachment has been used to measure IRPL from single sand-sized grains of feldspar from an aeolian dune from New Zealand. A 1W UV LED (365 nm) is also added to the system and this is effective at resetting the IRPL signal, permitting a single aliquot regenerative dose (SAR) protocol to be used to measure equivalent dose ($D_e$).

Measurement of a known laboratory dose (104 Gy) demonstrates the reproducibility of the attachment, with no overdispersion observed in the resulting single grain $D_e$ values. The recovered dose is within 10% of the given dose. The natural IRPL signal yields $D_e$ values from single grains with low overdispersion (22%) and giving a weighted mean value (103 ± 5.8 Gy) that is consistent with that obtained using post-IR IRSL measurements (105 ± 3.8 Gy). The attachment described here provides IRPL measurements on single grains suitable for exploring the potential of this novel and exciting signal for dating geological sediments.

1. Introduction

The discovery of a luminescence emission at 955 nm in feldspars when stimulated in the near infrared (Prasad et al., 2017) opens up exciting new opportunities for archaeological and geological dating applications. This infrared photoluminescence signal (IRPL) signal, and a second emission at 880 nm (Kumar et al., 2018), are reported to have a number of properties that make them attractive for dating. First, the signal does not deplete during measurement, making it possible to extend the period of stimulation. Secondly, the signal is thought not to suffer from anomalous fading, a major challenge for other methods based on analysis of stimulated luminescence from feldspars. However, an additional observation in initial measurements (Prasad et al., 2017) is that the signal does not bleach rapidly with exposure to daylight, and this could potentially be an impediment to the use of this signal for dating sediments. In previous research using other luminescence signals, where the exposure of sediments to daylight may vary from one mineral grain to another, single grain measurements have proven valuable for dating (Duller, 2008). This paper describes the development of instrumentation for measuring the 955 nm IRPL emission from single sand-sized (~200 μm diameter) feldspar grains. The performance of this instrumentation...
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is degraded significantly when light passes through obliquely (Fig. 1(a) and (b)), and breakthrough of the 830 nm stimulation light into the detector is a major problem. Two of the three systems that Kook et al. (2018) describe pulse the 830 nm IR laser (typically 5 μs on pulse, 95 μs off period) and use time resolved measurements with IR sensitive photomultiplier tubes so that they can ignore the signal during the on-period of the 830 nm stimulation and just use the IRPL emission in the off-period of the 830 nm laser (typically the period 3–92 μs after each IR pulse). In order to make measurements of single grains of feldspar, they based one system around a focussed laser system (Duller et al., 2003). Kook et al. (2018) also describe a system based around use of an electron multiplying charge coupled device (EMCCD) camera. The EMCCD detector described by Kook et al. (2015) has been shown to be a sensitive detector capable of resolving luminescence emissions from single grains of quartz (Thomsen et al., 2015) at ~340 nm. However, unlike typical photomultipliers used for luminescence dating (e.g. ET EMD-9107 or EMI 9635) the EMCCD also has high sensitivity at wavelengths in the yellow (e.g. 580 nm, see Duller et al., 2015) and into the infrared, including up to 955 nm (Kook et al., 2018). Kook et al. (2018) showed an image of IRPL collected using the EMCCD, but no analytical data are shown, possibly because of the high breakthrough of the IR stimulation into the detector. Due to the reset and readout clock speeds, EMCCD images cannot be collected at a rate suitable for time-resolved measurements.

The aim of this work is to design an attachment for the Riso TL/OSL reader to measure IRPL from single mineral grains using an EMCCD.

Two approaches are described that make this possible. Firstly, an optical design is described that optimises the performance of the detection filters in rejecting the stimulation light. Secondly, the spatially resolved nature of the data is exploited to provide an assessment of the breakthrough during every data collection, allowing this breakthrough signal to be effectively subtracted, yielding the IRPL signal. The performance of the attachment is demonstrated in a series of measurements of laboratory and natural doses.

2. Instrument description

Following the work of Kook et al. (2018) the Evolve EMCCD camera (Photometrics) was mounted on a Riso TL/OSL reader, but this was done using a bespoke head and detection optics designed and manufactured at Aberystwyth University to reduce the breakthrough of the IR stimulation (Fig. 2). The head has ports at 45° for optical stimulation of the sample using a 200 mW 850 nm IR laser diode mounted in a Thorlabs TEC temperature-controlled mount. The diode is driven from a Thorlabs bench top laser diode current controller operated in constant power mode with feedback provided by the laser diodes inbuilt photodiode. The laser diode output is cleaned up with an Edmund Optics BP850 × 10 nm OD4 filter to remove the low level but broad tails in its emission. The laser was scattered by a ground glass diffuser to provide uniform illumination with an irradiance of 20 mW cm⁻² at the sample. On the detection side, the 850 nm stimulation was rejected using three Edmund Optics LP925 OD4 filters, and a BP950 × 50 OD4 filter was used to isolate the 955 nm emission (Fig. 1). This is the same filter combination described by Kook et al. (2018). The filters are interference filters designed to operate with light arriving perpendicular to the filter surface, and their transmission properties shift progressively to shorter wavelength as the angle of incidence of light on the filters increases (Fig. 1(a) and (b)). An examination of the Riso DASH head (see Fig. 2 in Lapp et al., 2015) indicates that light can reach the filters at large angles of incidence with a small number of scattering events from the inside of the head and lens tube. The reflectance of the black anodise on the head was measured to be in excess of 20% at 850 nm, and so the likelihood of this happening is large. A critical part of the design of the IRPL head discussed in the present paper has been to control stray light to prevent the stimulation light reaching the filters at large angles of incidence. This has been achieved in two ways. Firstly, long focal length imaging optics along with a baffle located near the sample are used to restrict the angles of rays entering the optical system as shown in Fig. 2. The imaging optics consist of 100 mm and 80 mm Anti-Reflection coated IR achromatic doublets to achieve a magnification of 0.8 with the filters located between the lenses. Secondy, stray light has been minimised by coating all surfaces in the optical assembly with a matt black paint with a reflectance of less than 4% at 850 nm. The combination of these two approaches has meant that it has been possible to reduce the breakthrough to a level where IRPL can be measured using the EMCCD, and this is described in the remainder of this paper.

The head designed for IRPL measurements has eight ports where stimulation or detection units may be placed (Fig. 2) and one of these was fitted with a 300 mW 880 nm LED delivering ~40 mW cm⁻² at the sample. Although an Electron Tubes EMD-9107 PMT was also mounted on the head, the collection efficiency was very poor due to the viewing geometry and limited aperture of the collecting optics, meaning that it was not possible to make IRSL measurements. Where IRSL measurements were needed, a DASH head (Lapp et al., 2015) equipped with 970 nm IR LEDs (180 mW cm⁻²) was used, fitted with a BG3 and BG-39 filter.

Resetting the IRPL signal has been reported as difficult (Prasad et al., 2017) so we mounted a 1W 365 nm Thorlabs LED and collimating lens on the reader (Fig. 2) to provide a computer controlled bleaching unit with an irradiance of 0.6 W cm⁻². The impact of this UV LED on the IRPL
and IRSL signal is discussed later in this paper.

The additional stimulation and bleaching units described above were connected to the External Light Source signals that are built into the DASH head (Lapp et al., 2015), and this allowed full automatic control of the new attachment with the standard software used for measuring sequences on Risø instruments.

### 2.1. Spatial discrimination of IRPL signal and breakthrough

Potassium-rich feldspar separated from an aeolian dune in North Island New Zealand (GDNZ16; Duller, 1996) was used for characterisation of this instrument. The sample had been sieved at 180–212 μm and undergone density separation at 2.62 and 2.58 g cm$^{-3}$ to isolate a potassium-rich fraction. Grains of this material were mounted on single grain discs consisting of an array of 10 by 10 holes, each 300 μm deep and 300 μm in diameter. Optical stimulation at 850 nm was for 1.25 s, and an image was collected every 0.25 s or every 0.1 s (to aid comparison, values in the text are all expressed as counts per 0.1 s). The IRPL image of the single grain disc shown in Fig. 3 was after a dose of ~100 Gy. Prior to this measurement the grains had been preheated at 260 °C for 60 s.

The single grain holder has a defined grid of 100 holes containing grains, and the three locating holes around the margins of the disc allow a coordinate system to be defined that marks where each grain hole is within the image (Duller et al., 1999; Kook et al., 2015). The defined geometry of the sample on the disc makes it possible to discriminate spatially between those regions where one would only expect to observe breakthrough, and those where one would expect to see IRPL (on top of any breakthrough). This spatial discrimination provides an alternative to the time resolved discrimination between signal and breakthrough described by Kook et al. (2018) for photomultipliers.

To measure breakthrough of the 850 nm stimulation into the detector and subtract it from the IRPL signal, two sets of regions of interest were defined for the EMCCD images collected. The first set of regions of interest are centred on each of the holes where the 100 grains are mounted (shown in red in Fig. 4(a)). The second set of regions of interest are centred between the grain holes in an 11 by 11 grid (shown in green in Fig. 4(a)). Thus each grain hole has 4 regions of interest around it where there should be no IRPL, and the average of these four values can be used to define the background due to breakthrough for that hole.

### 3. Initial characterisation of the instrument

Two sets of measurements were made, one using the Electron Multiplying mode of the EMCCD and one in non-EM mode. In non-EM mode the maximum intensity of the observed signal is ~4000 counts per pixel. Kook et al. (2015) recommended defining a region of interest 450 μm in diameter over each single grain hole in order to optimise the signal from each grain while minimising cross-talk from grains in adjacent holes. Summing the signal from this 0.16 mm² area, the intensity is up to 240,000 counts per 0.25 s, but this includes breakthrough from the 850 nm stimulation. Averaging the signal from regions of...
interest (each 0.16 mm² in area) located away from the single grain holes gives a mean signal of 125657 ± 5441 counts or 279 ± 12 counts per pixel per 0.25s. In non-EM mode, CCD readout noise (noise introduced during the process of measuring the charge accumulated in each pixel) is significant, and is 11.1 counts per pixel. The remainder (~268 counts per pixel per 0.25s) of this signal arises from breakthrough of the 850 nm stimulation wavelength through the detection filters. In electron multiplying mode the dynamic range of the EMCCD is diminished, but the read noise is negligible. Analysis of the same sample in EM-mode gave a read noise of 0.003 counts per pixel and a signal of 228 ± 11 counts per pixel per 0.25s (equivalent to 91 counts per pixel per 0.1s).

To assess the reproducibility of the IRPL measurements, and to see whether the signal is depleted with repeated measurement, the sequence shown in Table 1 was used. In this analysis the signals from all 100 regions of interest centred on the grains have been summed, and the signal from the 121 ROIs used for the background scaled by a factor of 1.21 before subtracting it from the 100 ROIs where grains are located. The signal from breakthrough is ~100 counts per pixel per 0.1s, similar to the value of 91 counts per pixel per 0.1s obtained previously. After subtracting this background, the net signal averaged across all 100 grain holes is ~30 counts per pixel, equating to a total signal of ~1.1 million counts per 0.1s (Fig. 4(b)). A small decline in IRPL intensity is observed (0.18% per 2.5 s measurement), consistent with previous reports (Prasad et al., 2017). There is variability of 0.97% in the IRPL signal about a linear fit to the data (Fig. 4(b)), and it is likely that this results from variations in the power output from the 850 nm laser diode.

The intensity of IRPL measurements will depend linearly upon the stimulation power, so having a stable power output from the 850 nm laser diode is important. The breakthrough of the 850 nm stimulation at 470 nm while holding the sample at 300 °C, but this was not possible using the instrument described here. Instead, a 1W 365 nm LED has been mounted on the reader. The sequence shown in Table 2(a) was used to measure the impact of exposure to this UV LED upon the IRPL signal. Since measurement causes negligible depletion of the IRPL signal, a single beta dose was given and preheated, prior to an alternating sequence of IRPL measurement and UV exposure, eventually giving a cumulative length of UV exposure of 20,000 s (Fig. 5). The same sample was then measured using the sequence in Table 2(b) to measure the impact of the UV exposure upon the IRSL and post-IR IRSL signals.

Table 1

| Step | Description |
|------|-------------|
| 1    | 8 β irradiation 665 Gy |
| 2    | TL to 260 °C at 5 °C/s and hold for 60s |
| 3    | IR (880 nm) at 50 °C for 200s |
| 4    | IRPL (850 nm) at 50 °C for 2.5s (5s measurement time, including time before and after IR stimulation with no optical stimulation) |
| 5    | Repeat step 4 a total of 20 times |

4. Impact of 365 nm illumination upon IRPL, IRSL and post-IR IRSL signals

To undertake a single aliquot regenerative dose (SAR) protocol, the luminescence signal needs to be reset after each measurement. The IRPL signal does not decrease rapidly with measurement time, so some other method of resetting the signal is needed. Prasad et al. (2017) used stimulation at 470 nm while holding the sample at 300 °C, but this was not possible using the instrument described here. Instead, a 1W 365 nm LED has been mounted on the reader. The sequence shown in Table 2(a) was used to measure the impact of exposure to this UV LED upon the IRPL signal. Since measurement causes negligible depletion of the IRPL signal, a single beta dose was given and preheated, prior to an alternating sequence of IRPL measurement and UV exposure, eventually giving a cumulative length of UV exposure of 20,000 s (Fig. 5). The same sample was then measured using the sequence in Table 2(b) to measure the impact of the UV exposure upon the IRSL and post-IR IRSL signals and provide a point of comparison with previously published bleaching data for these signals (e.g. Colarossi et al., 2015; Boylaert et al., 2012). For these measurements a DASH head equipped with 870Δ45 nm LEDs was used along with the EMCCD and BG3 and BG39 filters to observe the
sediments. The IRPL signal appears to bleach at a very similar rate as the IRSL signal and has proved to be a very valuable chronometer for dating. It does appear to be well reset in many natural depositional environments and has previously been interpreted as thermal quenching of the IRPL emission. As has been observed many times previously, the IRSL~400 nm IRSL emission remains constant from 7 K to ~200 K, and then increases in intensity by a factor of over 30 times in this temperature range. This increase is due to greater efficiency with which charge is excited from the ground state and subsequently transported via band-tail states to recombination centres (Jain and Ankjaergaard, 2011).

5. Temperature dependence of IRPL and IRSL signals

The temperature dependence of the IRPL signal was investigated in order to decide the optimum temperature at which to make IRPL measurements using the attachment described here. Kumar et al. (2018) demonstrated that over the temperature range from 7 K to 295 K, the 955 nm IRPL emission remains constant from 7 K to ~200 K, and then drops from 200 K to 295 K, but does not reach zero. An aliquot of potassium rich feldspar from GDNZ16 was used to extend the temperature range for such measurements. The aliquot was given a beta dose of 166 Gy, preheated at 320 °C for 60 s and then had the IRSL signal measured using the 880 nm LED while holding the sample at 50 °C. Following this, the IRPL signal was measured at temperatures from 50 to 300 °C (323–573 K). After each measurement, the aliquot was bleached using the UV LED for 1200 s to remove the IRPL signal, and the aliquot was then irradiated again and the cycle repeated. The IRPL signal measured in the range 50–300 °C drops monotonically (Fig. 6), continuing the trend seen by Kumar et al. (2018) at lower temperatures. This drop has previously been interpreted as thermal quenching of the IRPL emission process (Kumar et al., 2018), and is in stark contrast to the increase in the IRSL signal that has been reported many times, as first seen by Duller and Wintle (1991).

A similar experiment as described above was undertaken on the same sample, to provide direct comparison of the behaviour of the IRSL signal at different measurement temperatures. The procedure followed is identical to that used for the IRPL measurement except that following the preheat to 320 °C the IRSL signal is measured at the different temperatures for 200 s in order to deplete the IRSL signal. The IRSL signal increases in intensity by a factor of over 30 times in this temperature range. This increase is due to greater efficiency with which charge is excited from the ground state and subsequently transported via band-tail states to recombination centres (Jain and Ankjaergaard, 2011).

6. Single aliquot regenerative dose measurements

6.1. Measurement of a laboratory radiation dose

A single aliquot regenerative dose (SAR) protocol using the IRPL signal is shown in Table 3. Grains were exposed to the UV LED for 600 s after measurement of the Lx and Tx in order to reduce the IRPL signal. To assess the effectiveness of the procedure the set of grains that had previously been measured to assess the impact of UV exposure upon the IRPL and IRSL signals were used. Grains were given a beta dose of 104 Gy, and then the sequence in Table 3 was followed to build up a dose response curve. The aim of this experiment was to see whether this SAR sequence was able to recycle a dose, and reset the IRPL signal. Fig. 7(a) shows the IRPL signal from a single grain in response to regeneration doses ranging from zero to 166 Gy. The background subtracted from this signal varies depending upon the signal measured in the four adjacent background ROIs, but is typically ~40,000 counts per 0.1s channel per ROI. The IRPL signal does not decline during the 2 s of stimulation at
6.2. Measurement of a natural radiation dose

A set of grains from GDNZ16 that had not previously been measured were placed in a single grain holder, and the sequence shown in Table 3 used to measure the natural IRPL signal and $D_e$ from the grains. Of the 100 grains measured, 22 gave data that passed the rejection criteria outlined above. The IRPL dose response curve was measured to a maximum regenerative dose of 1325 Gy (e.g. Fig. 8(a)) and dose response curves have an average $D_e$ value of 261 Gy, but this value varied from 122 to 432 for individual grains. For the 22 grains for which a $D_e$ value was obtained, the average background was 59291 ± 652 counts per 0.25 s, and the average natural signal (including the background) from the 22 grains was 94201 ± 59291 counts per 0.25 s. Thus the average signal-to-noise ratio is only 0.59. In spite of this low signal-to-noise ratio, dose response curves such as that shown in Fig. 8(a) could be produced, and the radial plot shows that consistent $D_e$ values were obtained (Fig. 8(b)). The overdispersion of the 22 IRPL $D_e$ values is 22%, near the lower end of the range observed in the few examples of single grain IRSL data sets from potassium rich feldspars (e.g. Reimann et al., 2012; Neudorf et al., 2012; Smedley et al., 2016; Riedesel et al., 2018). The weighted mean IRPL $D_e$ value obtained is 103 ± 5.8 Gy, 18% larger than the value of 87.0 ± 3.7 Gy obtained by Duller (1996). However, this earlier value may not be reliable since it used a single aliquot additive dose method that is no longer used, and it measured the IRSL emission at 50 °C but did not make any assessment of anomalous fading. To provide a more appropriate comparison, the $D_e$ value for this sample was measured using a SAR protocol with a post-IR IRSL signal.

The IRPL head was replaced with the DASH head to allow IRSL measurements to be made. A second set of single grains of feldspar from GDNZ16 were mounted on single grain discs and a post-IR IRSL$_{225}$ SAR sequence applied. The SAR sequence is almost identical to that described in Table 3 except that the IRSL measurements were for 100 s while the sample was held at a temperature of 50 °C, and the IRSL measurement was replaced by a post-IR IRSL measurement for 200 s while holding the sample at 225 °C. For the post-IR IRSL$_{225}$ signal, a total of 76 grains of the 100 grains measured passed the rejection criteria and gave a weighted mean $D_e$ of 105 ± 3.8 Gy (Fig. 8), indistinguishable from the IRPL value. The overdispersion of the post-IR IRSL$_{225}$ $D_e$ data is 25%, similar to the IRPL value.

7. Conclusions

The use of an optical system designed to maximise the effectiveness

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**Table 3**

| Step | Signal |
|------|--------|
| 1    | Natural or Laboratory Irradiation |
| 2    | Preheat to 260 °C at 5 °C.s$^{-1}$ and then hold for 60s |
| 3    | IR (880 nm) for 200s, sample at 50 °C |
| 4    | IRPL (850 nm) for 2.5s (0.25s/image) at room temp $L_s$ |
| 5    | UV (365 nm) for 600 s at room temp |
| 6    | Test Dose (52 Gy) |
| 7    | Preheat to 260 °C at 5 °C.s$^{-1}$ and then hold for 60s |
| 8    | IR (880 nm) for 200s, sample at 50 °C |
| 9    | IRPL (850 nm) for 2.5s (0.25s/image) at room temp $T_s$ |
| 10   | UV (365 nm) for 600 s at room temp |
| 11   | Return to step 1 |

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of the LP925 and BP950 rejection filters, and using the well-defined spatial definition of the sample within the image has made possible an IRPL system for single grains based on an EM-CCD detector. Although the signal-to-noise ratio is still low in comparison with the performance typically achieved for IRSL measurements, this IRPL system is able to generate reproducible data, with a typical variability of less than 1% between replicate measurements of the same sample (Fig. 4(b)). The 955 nm IRPL emission is strongly affected by thermal quenching, with the signal dropping monotonically from 50 °C to 300 °C (Fig. 6), and thus subsequent measurements of IRPL have been made at room temperature to maximise the signal. A single aliquot regenerative dose (SAR) method has been applied, using a UV LED to reset the IRPL signal between regeneration cycles. A dose recovery experiment demonstrates the reproducibility of the IRPL measurements, that a dose can be recovered within 10% of the given dose, and the suitability of the UV LED for resetting the signal. The natural D_e for single grains of an aeolian dune sand (GDN16) from New Zealand measured using IRPL (103 ± 5.8 Gy) is consistent with the value derived using a post-IR IRSL signal (105 ± 3.8 Gy). The age control for GDN16 is poorly constrained (Duller, 1996) so it is not possible to assess the accuracy of this D_e determination, but future work using IRPL will focus on analysis of samples with robust independent age control in order to assess the accuracy of ages that can be calculated using this new signal.

Whilst the breakthrough from the 850 nm stimulation is high, one of the advantages of working with an imaging detector and a sample that is clearly defined within the field of view is that the background can be measured by analysis of areas away from the sample and subtracted. Reducing the breakthrough from the 850 nm stimulation will be important in future developments, but subtraction of the background in this way provides a system capable of measuring the IRPL signal from single grains suitable for dating.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Fig. 8. (a) IRPL dose response curve for a single grain of GDN16. (b) Radial plot of D_e values for grains of GDN16. D_e data are shown from IRPL measurements (filled circles) and from post-IR IRSL measurements (open triangles).

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