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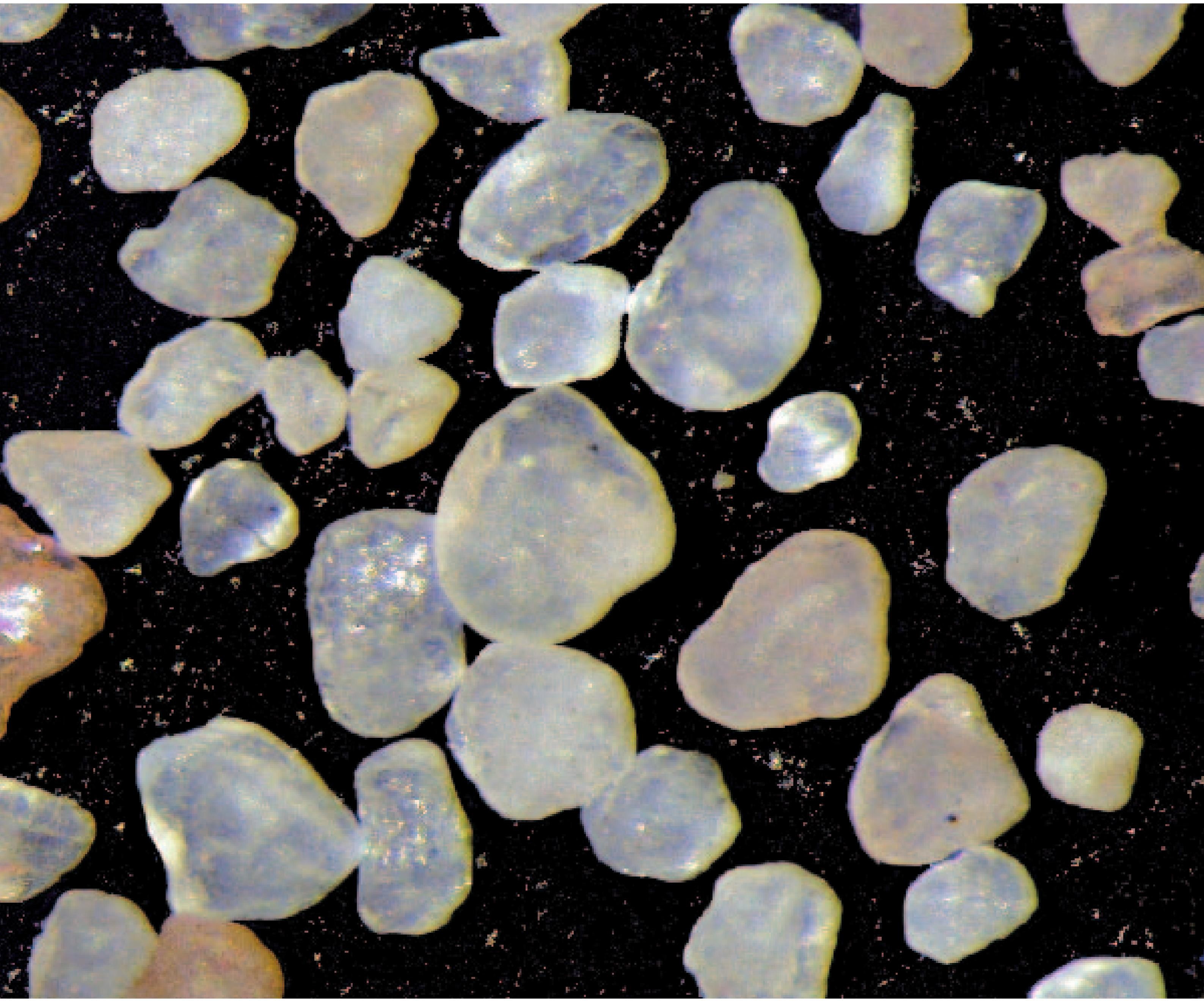
SWALE/URE WASHLANDS SINGLE-GRAIN OPTICALLY STIMULATED LUMINESCENCE (OSL) MEASUREMENTS OF FLUVIAL AND FLUVIOGLACIAL SEDIMENTS

SCIENTIFIC DATING REPORT

Geoff A T Duller and Helen M Roberts



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Summary

Eight samples of fluvioglacial origin and one from a Holocene fluvial deposit were collected from the area around Ripon, with the aim of providing chronological constraints on the deglaciation of the area at the end of the Devensian. The depositional environments are such that there was a concern that not all the mineral grains in the sediment may have been exposed to daylight at deposition. Therefore, luminescence measurements were made on single sand-sized grains of quartz. The signals from these were generally very dim, with only very few grains (0.9%–2.2%) giving detectable optically stimulated luminescence (OSL). In spite of rigorous sample preparation to extract pure quartz, investigations demonstrated that many of these OSL signals originated from non-quartz minerals, probably feldspar inclusions. After rejecting grains with these signals, only 0.1–0.6% of grains remained for the eight fluvioglacial samples, and this was insufficient to allow an age to be determined. For the one fluvial sample a slightly higher proportion (1.0%) of grains was accepted. Statistical analysis was problematic because of the low recovery of suitable grains, but the results broadly agreed with independent age control.

Keywords

Optically Stimulated Luminescence
Geochronology

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1. Introduction

This report describes the measurements and findings of an optically stimulated luminescence (OSL) dating study undertaken as part of a project funded by English Heritage on the Swale-Ure washlands. The overall project studied the fluvial and landscape evolution of the area through the last glacial-interglacial cycle. The specific aspect of the research described in this report is the analysis of a number of samples of fluvial and fluvioglacial sediments collected for OSL measurements in order to assess their depositional age.

OSL provides a method that can directly date the deposition of sediments, recording their last exposure to daylight. In the last 10 years, luminescence dating has become a well-established method for providing an absolute chronology for the deposition of Quaternary sediments (Duller 2004). It is increasingly being used as the method of choice for dating aeolian sediments, and is also being used for analysis of sediments from an ever-increasing diversity of depositional environments. The basis of the method is that naturally occurring minerals (including quartz and feldspars) act as natural dosemeters, recording the amount of ionizing radiation which they absorb. By stimulating such mineral grains in the laboratory, either by heating them or exposing them to light, a luminescence signal is generated. This luminescence is the emission of light from the grains, and the intensity of this light is dependent upon the radiation dose to which grains have been exposed. This latter quantity is known as the equivalent dose and abbreviated to D_e. When this quantity is divided by the rate at which a sample is exposed to radiation during burial (the dose rate) then an age can be calculated.

$$Age\,(a) = \frac{Equivalent\ Dose\,(Gy)}{Dose\ Rate\,(Gy/a)} \qquad \text{Equation 1}$$

(1 Gray (Gy) = 1 Joule/kg)

In the last decade, four major developments have enabled the method to become more reliable. Firstly, in the 1990s a convenient method of optically stimulating a luminescence signal from quartz was developed, using blue light-emitting diodes (Bøtter-Jensen *et al* 1999). Prior to this, optically stimulated luminescence measurements on quartz were difficult to make, requiring large and complex Ar-ion lasers. In consequence, much luminescence research was focussed on the use of another light-sensitive signal, the infrared-stimulated luminescence (IRSL) signal from feldspars. While many useful ages were generated using this signal, a constant concern was that in some studies this signal has been shown to be unstable over short periods of time, leading to an underestimate of the age. This 'anomalous fading' is not entirely understood, and it is not clear whether it is a universal phenomenon in all feldspars (eg Huntley and Lamothe 2001; Spooner 1994) or whether it is restricted to certain types of feldspar (Duller 1997). Quartz is not prone to anomalous fading, and the development of a convenient method of stimulating an OSL signal made it feasible to develop methods based on quartz.

The second significant development was the single aliquot regenerative (SAR) dose procedure (Murray and Wintle 2000; Wintle and Murray 2006). This is a method for the measurement of the radiation dose to which the sample has been exposed during its period of burial (the equivalent dose, D_e). The method has two major advantages. Firstly, it explicitly tests for changes in the sensitivity of the luminescence signal during the procedure and this makes it more reliable than earlier methods used for quartz (Duller and Augustinus 2006). Secondly, because it is a single aliquot procedure (Duller 1991), this means that all the measurements necessary to derive a value of D_e are made on a single sub-sample of the material being dated. This makes it possible to test how reproducible replicate measurements are from the same material.

The third development has been in the analysis of replicate measurements of D_e from a sample, in order to test the reliability of the age. One of the assumptions of luminescence dating is that at the time when the grains were deposited, the luminescence signal from mineral grains was close to zero.

The luminescence signal is sensitive to light, and exposure to more than a few tens of seconds of daylight is sufficient to reduce the luminescence signal to near zero. For aeolian materials this is a reasonable assumption, and studies of modern aeolian sediments support this, with ages as young as a few decades being obtained (Bailey *et al* 2001; Bristow *et al* 2007). For other depositional environments the assumption is more problematic. In fluvially deposited sediments, for instance, some grains may be transported at the base of the water column, thus restricting their exposure to daylight, while others may rise to the top of the water column, or they may be temporarily deposited on bars where exposure to daylight can occur. For the aeolian sediment, all grains would have zero D_e at the time of deposition, and as the sediment remains buried, the D_e of all grains will increase together. For the fluvial sediment, at deposition some grains will have a D_e of zero, while others may retain a signal from their previous period of burial, and thus give D_e values significantly above zero (Olley *et al* 1999). The ability to make replicate measurements of the D_e from a sample makes it possible to explicitly test whether all the grains within a sediment have the same value of D_e or not (Rodnight *et al* 2005; 2006).

The fourth development was a system capable of making measurements on single mineral grains (Duller *et al* 1999; Bøtter-Jensen *et al* 2000). Where sediments are likely to consist of grains which had their luminescence signal reset to different extents at the time of deposition, this may be detected by making standard measurements looking at many hundreds or thousands of mineral grains simultaneously. However, a more detailed analysis can be obtained using measurements on single mineral grains. Using standard equipment, such measurements are prohibitively time-consuming. Duller *et al* (1999) described an automated system which used a focussed laser beam to measure sand-sized (~200µm diameter) grains individually (Fig 1). Using the SAR protocol, this enables a value of D_e to be determined for each mineral grain. This approach makes it feasible not only to identify when samples contain a mixture of grains with different values of D_e, but also to use statistical methods to determine the D_e appropriate for the depositional event of interest (Duller and Murray 2000). This has opened up the possibility of dating complex materials such as glaciofluvial sediments (Glasser *et al* 2006; Duller 2006) that could not be dated confidently using methods based on the analysis of many hundreds or thousands of grains simultaneously.

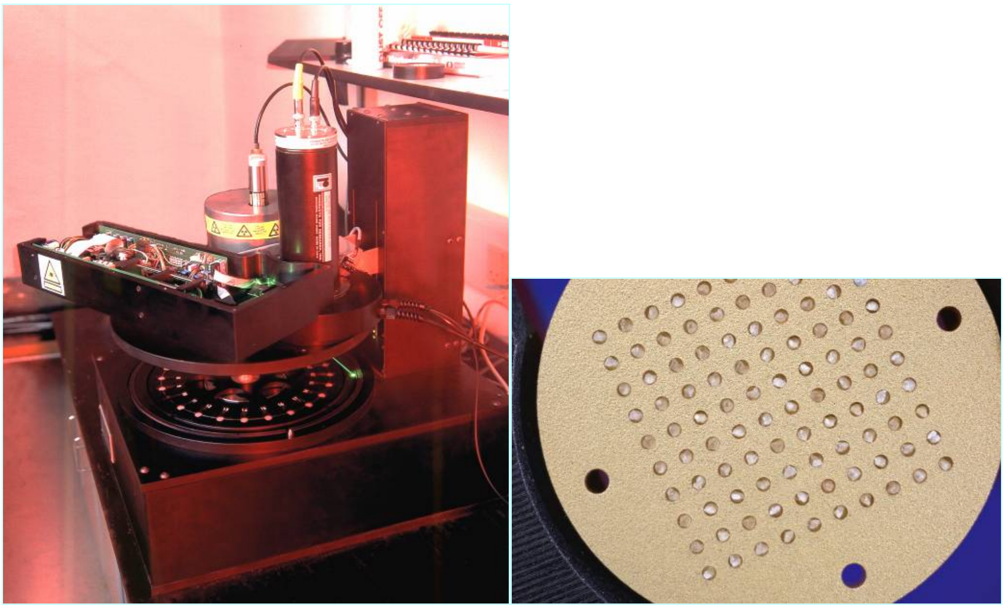


Figure 1: Left: automated system for measurement of optically stimulated luminescence from single mineral grains. Right: a 9.7mm-diameter aluminium disc in which 100 quartz grains are loaded for analysis (images taken from Duller 2004, *J Quaternary Sci*)

As described above, one of the major recent developments has been the Single Aliquot Regenerative dose (SAR) measurement protocol (Murray and Wintle 2000), and this has been applied to single sand-sized grains of quartz in this study. The SAR protocol uses the response to a test dose to correct for any change in luminescence sensitivity occurring in the sample, with all of the measurements necessary for the determination of D_e being made on a single aliquot. Figure 2 illustrates how D_e is obtained from the SAR measurements made. Following measurement of the natural luminescence intensity (denoted by the square symbol on the y-axis of Fig 2), the response (L_x) to a series of artificial radiation doses is measured, and normalised to the response (T_x) to a fixed test dose. A normalised dose-response or 'growth' curve can then be constructed by plotting the ratio L_x/T_x as a function of radiation dose. This enables the natural luminescence intensity to be calibrated to these responses to a given laboratory radiation dose, thereby determining the laboratory equivalent dose, D_e.

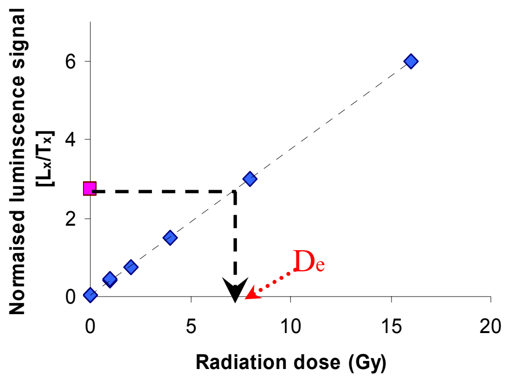


Figure 2: Dose-response or 'growth' curve (diamond symbols) generated from measurements made using the Single Aliquot Regenerative dose (SAR) measurement protocol. The natural luminescence intensity (square symbol) of the aliquot is calibrated against the response to these known artificial irradiation doses to determine the laboratory equivalent dose, D_e

2. Sample collection and dosimetry

Nine samples for analysis by optically stimulated luminescence (OSL) were collected from four sites (Table 1, Fig 3). All nine samples were collected either by inserting an opaque plastic tube into the section, or more commonly by directly placing sediment into a black plastic bag while being shielded from daylight under an opaque tarpaulin. Many of the samples were collected from gravel-rich sediments which could potentially have a heterogeneous dose rate. For seven of the nine samples, measurements of the radiation dose rate were made *in situ* using a portable gamma spectrometer (Table 2).

Eight of the samples were collected from fluvioglacial sediments, consisting of poorly sorted gravels and sands. Two of these samples, from Nosterfield Quarry (Aber78/NO3 and NO4), were associated with a large ice-wedge cast formed within fluvioglacial gravels and sands. The sample from Ripon Racecourse (Aber78/RR1) was from a well-sorted sand associated with Holocene fluvial deposition. More detailed sedimentological descriptions are given in Chapter 2 of Bridgland *et al* (forthcoming). The location of each sample is shown in Figures 4–10.

In addition, the dose rate to samples was assessed using laboratory-based measurements of thick-source alpha counting (TSAC) and GM-beta counting. These were undertaken on the samples after they were dried and finely milled to homogenise them. The results are also shown in Table 2. There is broad agreement between the two sets of analyses. Previous studies in homogeneous sediments have demonstrated comparability between them. For these sediments, where there is likely to be variation in the concentration of radionuclides due to the inhomogeneous nature of the sediments, the best estimate of the dose rate to coarse quartz grains within the sample would come from combining the gamma dose rate measured using the portable gamma spectrometer, and the GM-beta counting. For the two samples where it was not possible to obtain field-based measurements using the gamma spectrometer, the gamma dose rate was calculated using a combination of the beta and alpha counting measurements (Table 2). For the purposes of this table, an average water content of 10 ±2% was assumed during burial, and the contribution of cosmic rays to the dose rate was calculated using the thickness of the overburden. The dose rates were calculated using the conversion factors of Adamiec and Aitken (1998).

Table 1: Location of OSL samples taken for this study of the Swale-Ure.

Sample #	Location	Sample	Grid Reference
1	Marfield Quarry	Aber78/MA1	SE 2145 8312
2	Marfield Quarry	Aber78/MA2	SE 2145 8312
3	Ripon Racecourse	Aber78/RR1	SE 3320 6950
4	Ripon North	Aber78/RN1	SE 3041 7680
5	Ripon North	Aber78/RN2	SE 3068 7665
6	Nosterfield Quarry	Aber78/NO1	SE 2767 8073
7	Nosterfield Quarry	Aber78/NO2	SE 2767 8073
8	Nosterfield Quarry	Aber78/NO3	SE 2767 8073
9	Nosterfield Quarry	Aber78/NO4	SE 2767 8073

Table 2: Dosimetry measurements derived from *in situ* gamma spectrometry and laboratory-based GM beta counting and thick source alpha counting (TSAC), and total dose rate assuming a 10 ±2% water content. The contribution from cosmic dose has been calculated based on the thickness of overburden and is included in the Total Dose rate. See text for details.

	In situ gamma spectrometry			GM beta counting	TSAC		
Sample	K (%)	U (ppm)	Th (ppm)	beta dose (Gy/ka)	U (ppm)	Th (ppm)	Total Dose (Gy/ka)
78/MA1	0.79 ±0.03	2.39 ±0.08	6.02 ±0.21	0.84 ±0.04	2.82 ±0.23	6.40 ±0.76	1.49 ±0.09
78/MA2	0.29 ±0.02	1.69 ±0.08	2.32 ±0.12	0.55 ±0.01	2.11 ±0.09	1.91 ±0.27	0.93 ±0.09
78/NO1	0.38 ±0.02	1.73 ±0.08	2.51 ±0.12	0.78 ±0.01	2.02 ±0.17	3.73 ±0.56	1.08 ±0.06
78/NO2	0.31 ±0.02	1.82 ±0.11	2.47 ±0.14	0.58 ±0.01	2.12 ±0.14	2.77 ±0.46	0.93 ±0.06
78/NO3	0.39 ±0.02	1.94 ±0.10	3.05 ±0.17	1.03 ±0.01	2.34 ±0.20	5.86 ±0.67	1.33 ±0.06
78/NO4	-	-	-	1.06 ±0.02	2.30 ±0.25	7.86 ±0.84	1.66 ±0.08
78/RN1	0.20 ±0.01	1.40 ±0.08	1.98 ±0.11	0.43 ±0.01	1.64 ±0.08	1.54 ±0.24	0.75 ±0.08
78/RN2	-	-	-	0.71 ±0.03	2.22 ±0.10	2.87 ±0.33	1.17 ±0.09
78/RR1	0.38 ±0.02	0.94 ±0.05	2.58 ±0.13	0.44 ±0.02	1.34 ±0.10	2.30 ±0.31	0.78 ±0.08

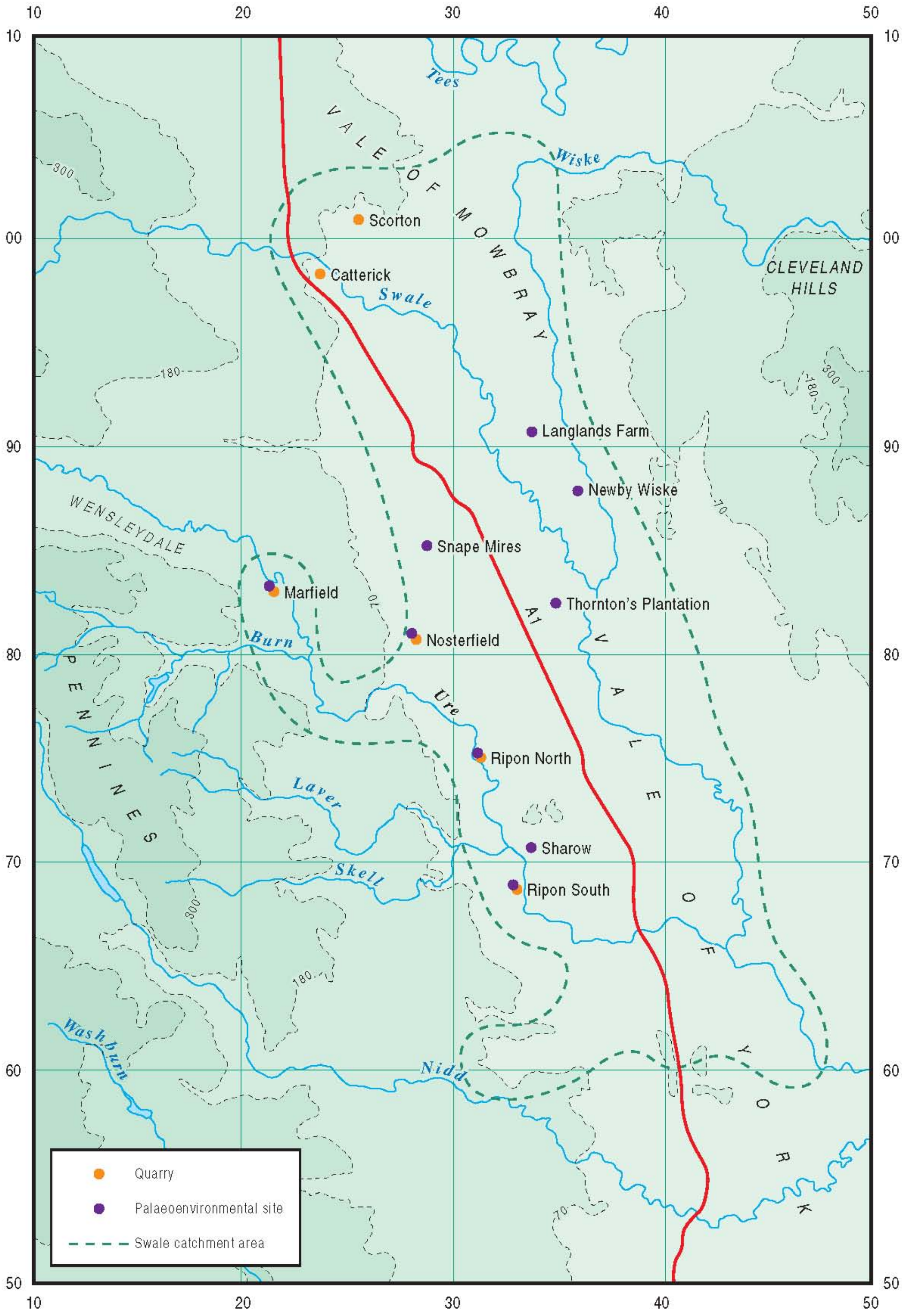


Figure 3: Map showing the study area and sites at Marfield, Nosterfield, Ripon North and Ripon South (Ripon Racecourse) where samples for OSL analysis were collected (Bridgland *et al* forthcoming)

Figure 4: Sample 78/MA1 – Marfield Quarry (G Duller)

Figure 5: Sample 78/MA2 was collected in Marfield Quarry at the back of the section being dug in the photograph (G Duller)

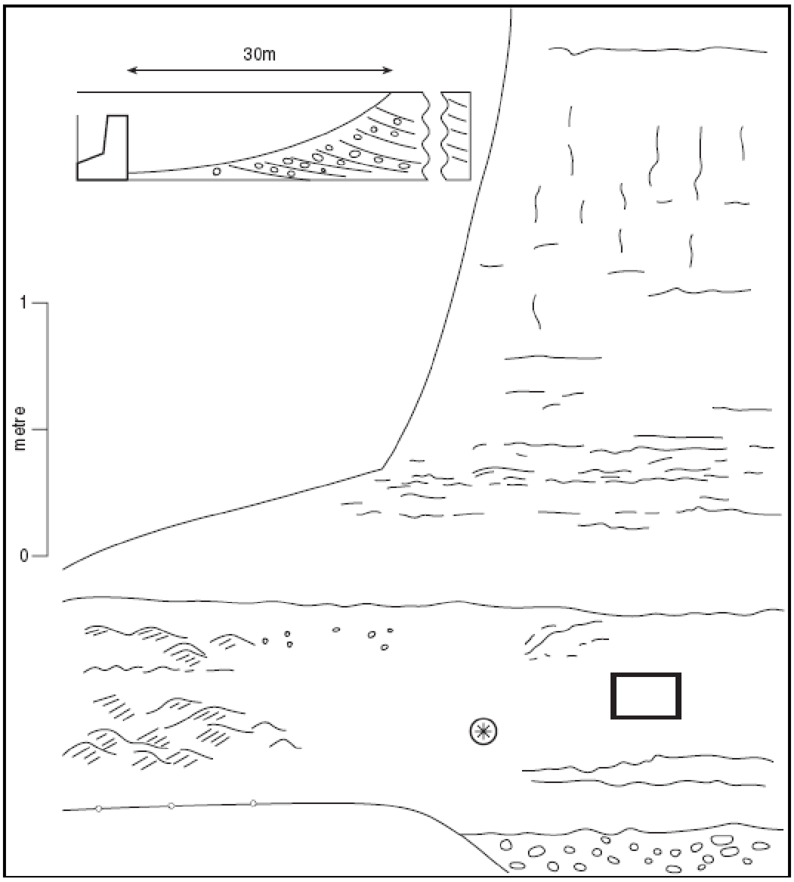


Figure 6: Top: photograph of Ripon Racecourse sample 78/RR1 (G Duller). Bottom: sample position shown by the asterisk inside a circle. Lower diagram is from Bridgland *et al* (forthcoming)

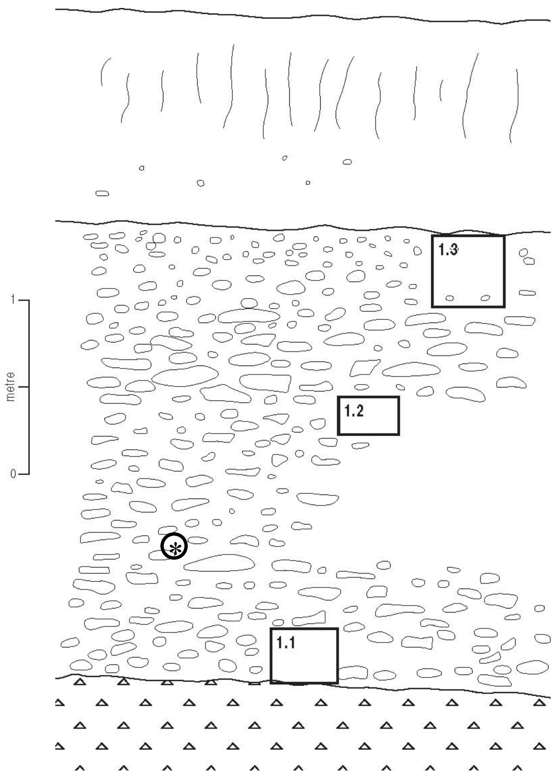
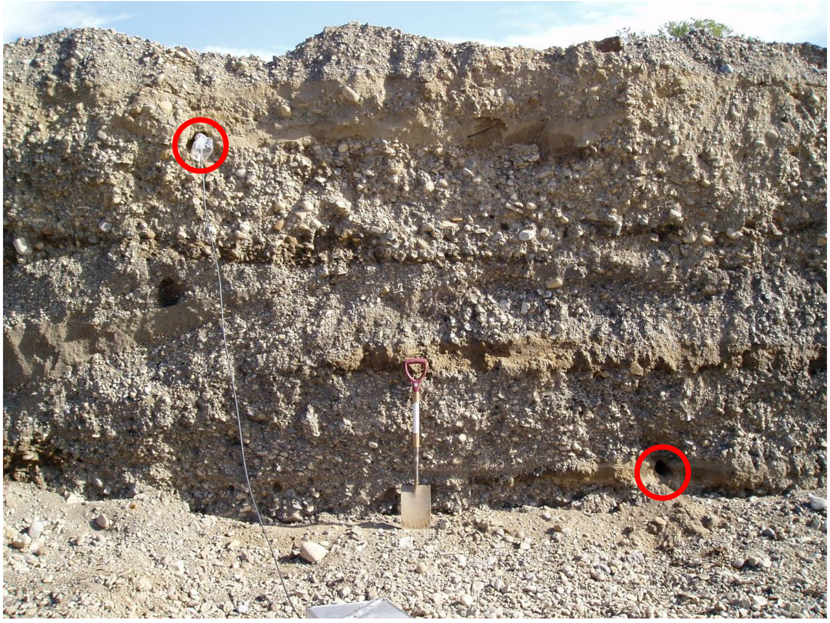


Figure 7: Ripon North trench. Top: sample 78/RN1 was collected under the black tarpaulin in the photograph (G Duller). Bottom: the coarse gravels that were sampled are 1m above the basal till, and ~2m below the overlying medieval alluvium. Sample position shown by the asterisk inside a circle. Lower diagram is from Bridgland *et al* (forthcoming)

Figure 8: Top: photograph of Ripon North river bank section (G Duller). Bottom: sample 78/RN2 was collected from the sandy channel infill on the terrace surface. This terrace is higher than that from which 78/RN1 was collected. Sample position shown by the asterisk inside a circle. Lower diagram is from Bridgland *et al* (forthcoming)



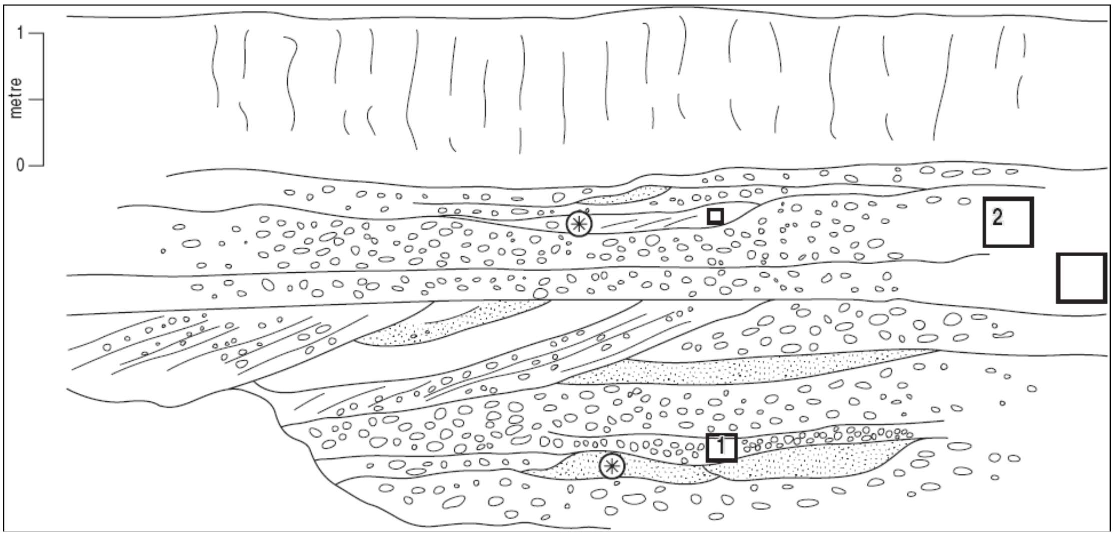


Figure 9: Top: photograph of Nosterfield Quarry (G Duller). Sample 78/NO1 was collected from the bottom right of image, and 78/NO2, being counted for gamma spectrometry, was from the upper left of image. Bottom: sample positions shown by asterisks inside a circle. Lower diagram is from Bridgland *et al* (forthcoming)

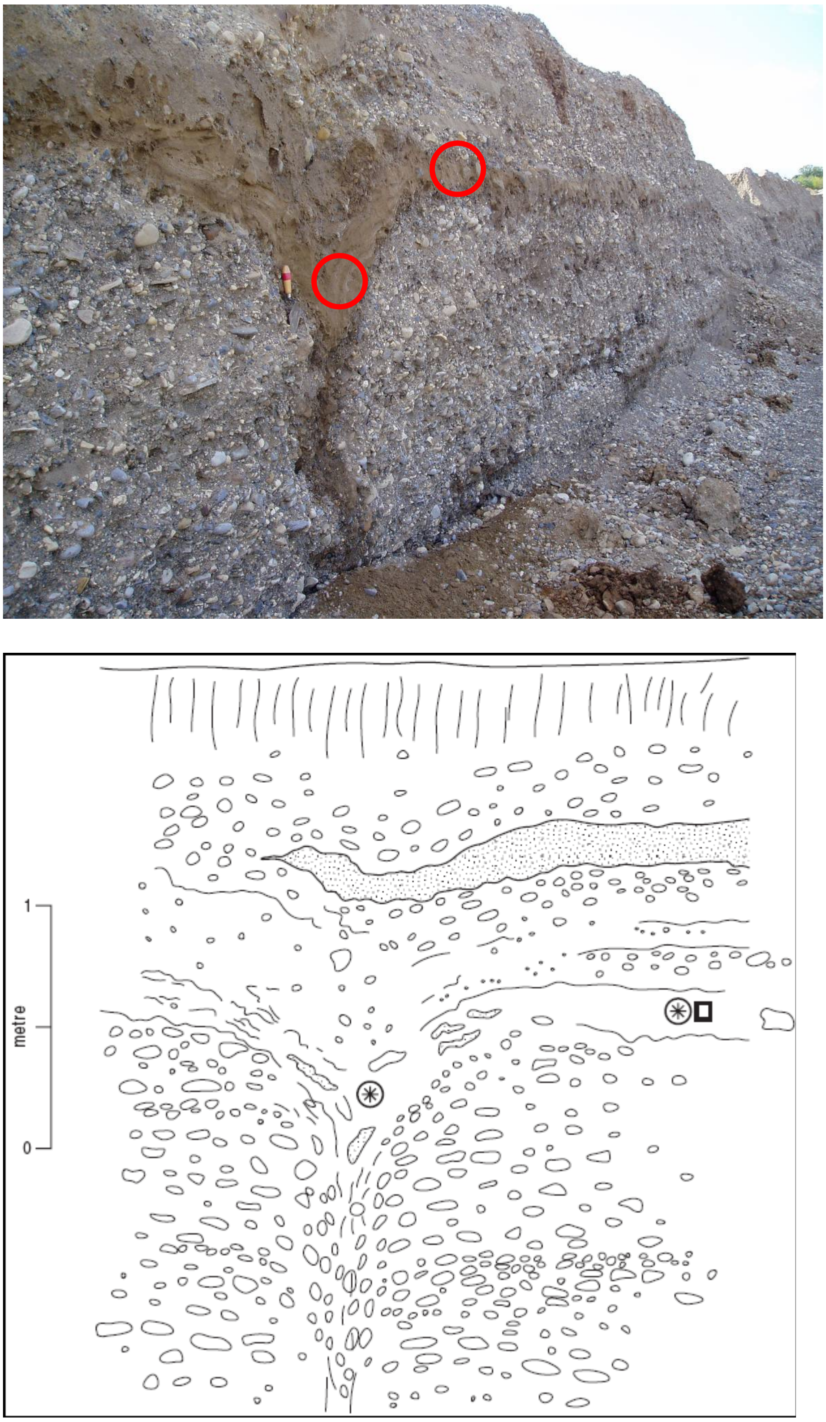


Figure 10: Top: photograph of ice-wedge cast sampled in Nosterfield Quarry (G Duller). Sample 78/NO3 taken from centre of ice-wedge cast ~0.5m below palaeo land surface. 78/NO4 taken from fine sediment associated with the palaeo land surface. Bottom: sample positions shown by asterisks inside a circle. Lower diagram is from Bridgland *et al* (forthcoming)

3. Laboratory Methods

3.1 Laboratory separation of quartz grains

In the laboratory, the OSL samples were prepared under subdued red lighting to avoid inadvertent removal of any of the luminescence signal. Sand-sized quartz grains were separated from the sediments using a standard procedure, following Wintle (1997). It consisted of a combination of 10% HCl to remove carbonates, 20 vols H_2O_2 to remove organics, dry sieving to obtain grains 180–211µm in diameter, and finally density separation using sodium polytungstate solutions at 2.62 and 2.70 g cm^{-3}. Material with a density between these values was then placed in 40% hydrofluoric acid for 40 minutes and then resieved at 180µm. This procedure both removes remaining feldspar grains, and removes the outer ~10µm skin of the quartz grains, which is affected by alpha radiation.

3.2 Luminescence measurements

Prior to measurement of the nine samples studied here, the reproducibility of the SAR procedure on the single-grain system was checked. This was undertaken using 300 grains of quartz which had previously been annealed at 450°C and given a gamma dose of 5Gy at Risø National Laboratory (A S Murray pers comm). The dose in these grains was measured using the SAR procedure (preheat 220°C for 10 seconds, cut heat at 160°C for 0 seconds, and a test dose of 0.7Gy). For a typical grain, Figure 11(a) shows the decay in the luminescence signal during one OSL measurement with the laser in the single-grain system. The laser is a 10mW $NdYVO_4$ solid state laser emitting at 532nm, but because the beam is focussed onto one grain at a time, the power density is very high (~50W cm^{-2}). This leads to a rapid decay in the OSL signal, as expected from quartz grains (eg Duller *et al* 2000). Combining together measurements of the regeneration doses and test doses from this grain, its response to radiation can be characterised (Fig 11(b)). The D_e for this grain is 109.8 ±4.7 seconds, which equates to 5.24 ±0.22Gy.

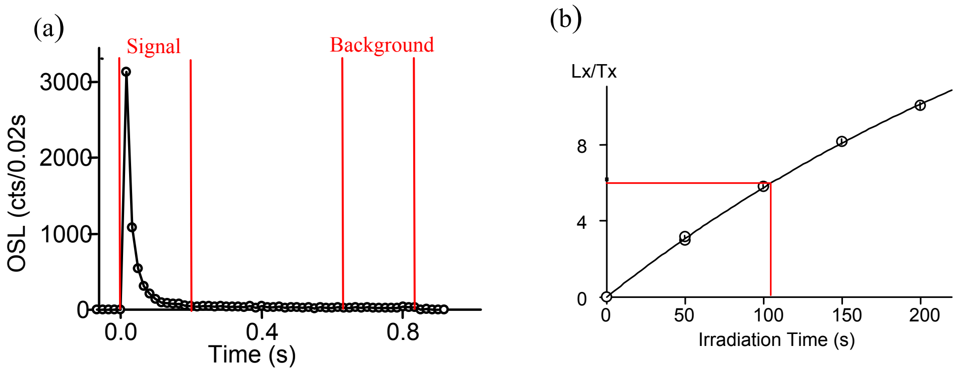


Figure 11: (a) OSL decay curve for a single grain of quartz used for demonstrating the reproducibility of the single-grain system. The parts of the curve integrated to calculate the signal and the background are shown. (b) The SAR growth curve for the same grain of quartz as a function of the irradiation time in the luminescence reader. The calibration for the beta irradiator in this reader is 2.86Gy/minute

The quartz used for these measurements has been specially sensitised prior to its use. Thus all 300 grains which were analysed yielded signals that were sufficiently bright, and reproducible, that they could be used to generate D_e values. Figure 12(a) combines together these 300 D_e values, one for each grain, on a radial plot (Galbraith 1990). On the diagram, each point represents the D_e obtained for a single grain. The more precisely that the D_e value is known, the further the point is plotted to the right of the diagram (the x-axis is the precision). The y-axis indicates the number of standard deviations between the D_e for a grain and a chosen value. For Figure 12(a), the chosen value is 5.0Gy. The result of such a plot is that grains with the same D_e will plot on a straight line originating from the origin. The value of the D_e can be read from the radial axis on the extreme right hand side of the plot. As expected for an artificially irradiated sample, the D_e values all cluster tightly to form a single band, consistent with the known dose of 5Gy. For materials which were incompletely bleached at the time of deposition, the distribution of D_e values may be far more complex (eg Roberts *et al* 1998; Duller 2006).

(a) (b)

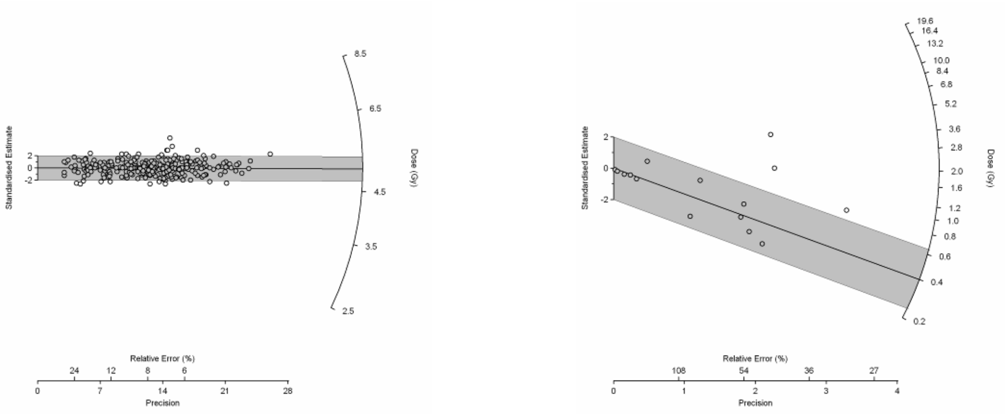


Figure 12: (a) Radial plot showing the doses calculated for 300 individual grains of quartz that had been annealed and given a gamma dose of 5Gy. (b) Radial plot of the grains from sample RR1 from which the signal originated from quartz

4. Results

For each of the nine samples studied here, between 900 and 2000 grains were measured (Table 3) using the single-grain system described above (Fig 1). Previous studies have found that many grains of quartz do not yield any detectable OSL signal (Duller *et al* 2000), with only between 5–10% of grains contributing luminescence. For the samples here, the response to the test dose (T_D 11.9Gy) following the measurement of the natural signal in the SAR sequence was used to assess whether a grain had sufficient signal to be used. A grain was said to have a 'detectable' OSL signal if the intensity in the initial 0.2s of optical stimulation is above the background signal (measured in the last 0.2s of optical stimulation, Figure 11(a)) by three times the variability of the background signal. Between 9 and 44 grains for each sample passed this criterion, equivalent to 0.9–2.2% of grains (Table 3), much lower than has typically been found in other studies.

Table 3: The number of grains measured for each sample, those from which a detectable OSL signal can be observed, and the number of those grains which passed the IR OSL depletion ratio test and other criteria (primarily the recycling ratio). The numbers in brackets are the data expressed as a percentage of the total number of grains measured.

Sample	Grains measured	Grains with 'detectable' OSL signal	Grains passing IR OSL depletion ratio test and other criteria
78/MA1	1000	16 (1.6%)	1 (0.1%)
78/MA2	1000	12 (1.2%)	0 (0.0%)
78/NO1	1000	9 (0.9%)	1 (0.1%)
78/NO2	1200	26 (2.2%)	1 (0.1%)
78/NO3	2000	24 (1.2%)	3 (0.2%)
78/NO4	900	19 (2.1%)	2 (0.2%)
78/RN1	1000	16 (1.6%)	4 (0.4%)
78/RN2	1000	17 (1.7%)	6 (0.6%)
78/RR1	2000	44 (2.2%)	20 (1.0%)

A second criterion which is applied to single-grain measurements is to test whether the grains are sensitive to optical stimulation using infrared (IR) wavelengths (830±30 nm). The aim of this test is to exclude any grains whose luminescence signal does not originate from quartz. The test, known as the IR OSL depletion ratio (Duller 2003), exploits the observation that quartz is not sensitive to IR radiation, while feldspar is. The test yields a ratio, showing the proportion of the OSL signal that is not removed by exposure to IR stimulation. For grains where the OSL signal is dominated by emissions from feldspar, this ratio is significantly less than unity, while for quartz the ratio should be consistent with one (Fig 13). For samples 78/MA1–MA2, 78/NO1–NO4, and 78/RN1–RN2 this test reveals that for almost all of the grains from which a detectable luminescence signal was observed, the signal appears to originate from a non-quartz mineral (Table 3). Given the thorough preparation procedure that was followed, these are likely to be small inclusions of feldspars and other minerals in quartz grains (eg Fragoulis and Readhead 1991). The occasional occurrence of inclusions like this is not uncommon in quartz, and has been observed in previous single-grain studies (eg Jacobs *et al* 2003). What is unusual in the samples from this study is that these inclusions seem to be the dominant source of OSL, and the quartz grains themselves yield negligible OSL. Other standard criteria were applied to each grain (Jacobs *et al* 2003) as well as the IR OSL depletion ratio test, but it was this latter test that caused the majority of grains to be rejected.

Sample 78/RR1 has a marginally higher proportion of grains that can be detected than any of the others, and approximately half of those that are detectable pass the IR OSL depletion ratio test, suggesting that the signals originate from quartz grains. Statistical analysis of this small data set would be problematic, but the data are broadly consistent with a medieval age for this river terrace (Fig 12 (b)), as deduced from the discovery of a leather shoe within the sediment nearby.

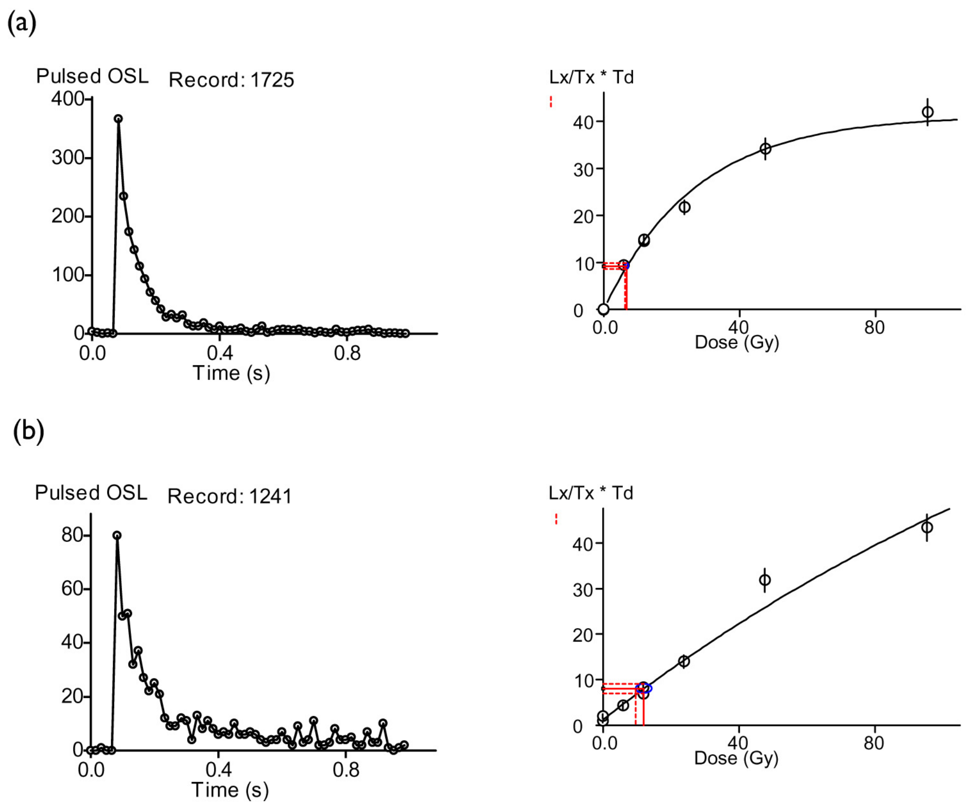


Figure 13: Test dose response (T_D = 11.9Gy) and SAR growth curve for two grains from sample Aber78/RN2. (a) Grain 25 of disc 15 has an IR-OSL depletion ratio of 1.00 ±0.11 and is therefore assumed to be a quartz grain, while (b) grain 41 of disc 5 has an IR-OSL depletion ratio of 0.08 ±0.04 and the signal is therefore thought to be dominated by feldspar. Note the different growth curves shapes in figures (a) and (b). That thought to be dominated by quartz is close to its saturation level by ~80Gy, while the grain in (b) shows almost linear growth over this range of doses. This difference is consistent with the expected characteristics of quartz and feldspar respectively

5. Conclusions

It is common experience that the OSL from quartz originating in different areas may vary significantly in brightness, though the causes of this are not understood. The optically stimulated luminescence emission from the quartz measured in these samples is unusually low. This may be related to the hydrothermal source of much of the quartz in the catchment, but at this stage this is only conjecture. Some OSL emissions are observed, but these are almost entirely derived from contaminants within the quartz. Single-grain measurements have been useful in demonstrating the nature of this contamination, and avoiding the pitfall of incorrectly using such signals to derive age estimates which would almost certainly be erroneous. The one sample from which a small proportion of quartz grains do yield OSL signals (78/RR1) does not have a sufficient number of grains, nor sufficiently bright grains, to enable statistical analysis, but the results are consistent with the known age of the sediment based upon archaeological remains found there.

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