Centre for Archaeology Report 25/2005

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ISSN 1473-9224

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Optically Stimulated Luminescence (OSL) Dating of Sediments from a Middle Palaeolithic Site at Lynford Quarry, Norfolk

Jean-Luc Schwenninger¹ and E J Rhodes²

Summary

This report presents details of the application of optically stimulated luminescence (OSL) dating to sediment samples collected during the excavation of a Middle Palaeolithic open-air site in Lynford Quarry near Mundford, Norfolk. The dates were obtained from sand-sized quartz grains and palaeodose determinations were made using a multi-grain single-aliquot regenerative-dose (SAR) measurement protocol. The environmental dose rate was calculated using the results obtained from instrumental neutron activation analysis (INAA) and *in situ* gamma-ray spectroscopy measurements.

Keywords

Luminescence Dating Geochronology

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Introduction

A relic Middle Devensian palaeochannel was discovered in February 2002 during an archaeological watching brief at Lynford Quarry (TL 825 948), near Mundford, in Norfolk. Thick deposits consisting of organic sandy sediments were buried under several meters of bedded sands and gravel. These organic sediments contained large *in situ* faunal remains as well as Mousterian stone tools and debitage.

Such well-preserved Middle Palaeolithic open-air sites are exceedingly rare and the site was immediately identified as being of national and international importance. Archaeological excavations were carried out by the Norfolk Archaeological Unit under the direction of W A Boismier from the 8th of April to the 11th of September 2002 with funding provided by English Heritage through the Aggregates Levy Sustainability Fund. A key research objective was to establish a chronological framework for the sedimentary sequence and to provide a date for the associated archaeological materials.

In total, 17 samples were collected for luminescence dating. An initial series of seven samples (X1098-X1104) was collected on the 8th of July 2002 by J-L Schwenninger in order to assess the potential for OSL dating. Radioactivity measurements were made *in situ* with an EG&G Ortec MicroNomad Nal gamma-ray spectrometer. Further details regarding individual samples are presented in Table 1, and the precise location of some of the samples is shown in Figures 1 and 2.

Preliminary dates based on the processing of four samples (X1098, X1100, X1103 & X1104) collected during the first site visit were communicated within three weeks. The positive outcome of the pilot study prompted a return visit on the 19th of August 2002. On this occasion, a further eight samples (X1160-X1167) were collected from various sedimentary units in order to complete the sampling of the stratigraphic sequence. An interim OSL report based on the results from the four samples was submitted to the Norfolk Archaeological Trust in December 2002 and these findings were subsequently reported in Boismier *et al* (2003). In October 2003, an additional two samples (X1837 & X1838) were submitted for dating. These were obtained from sandy sediments exposed in machine-excavated test pits close to the archaeological site.

Field code	Lab. Code	Excavation Code	Context	Height (m OD)	Comments on sedimentary units	
LYN03-01	X1098	30-126	20327	6.102	Yellow sand lens in lower gravel	
LYN03-02	X1099	30-125	20003	8.362	Dark brown organic sandy silt	
LYN03-03	X1100	30-124	20003	8.532	Dark brown organic sandy silt	
LYN03-04	X1101	30-123	20002	8.655	Greenish-grey sand	
LYN03-05	X1102	30-122	20005	8.752	Greenish-grey sand	
LYN03-06	X1103	30-127	20015	8.723	Light grey sand	
LYN03-07	X1104	30-128	20002/20003	9.107	Orange sand	
LYN03-08	X1160	30-265	20357	7.750	Greyish-brown silty sand overlying gravel	
LYN03-09	X1161	30-266	20390/20403	7.700	Dark brown organic stony sand	
LYN03-10	X1162	30-267	20371	8.000	Greenish-brown organic stony sand	
LYN03-11	X1163	30-264	20254	7.614	White silty sand below organic sand	
LYN03-12	X1164	30-263	20205	9.908	Yellow sand between sandy gravel	
LYN03-13	X1165	30-262	20317	11.04	Brownish-grey stony sand	
LYN03-14	X1166	30-268	20285	11.481	Brownish-grey sand overlying upper gravel	
LYN03-15	X1167	30-269	20305	10.656	Pale yellow sand between gravel	
LYN03-16	X1837	30-385	Test pit 15	~12.56	Yellow sand	
LYN03-17	X1838	30-387	Test pit 17	~17.30	Yellow sand	

 Table 1
 Sample details.

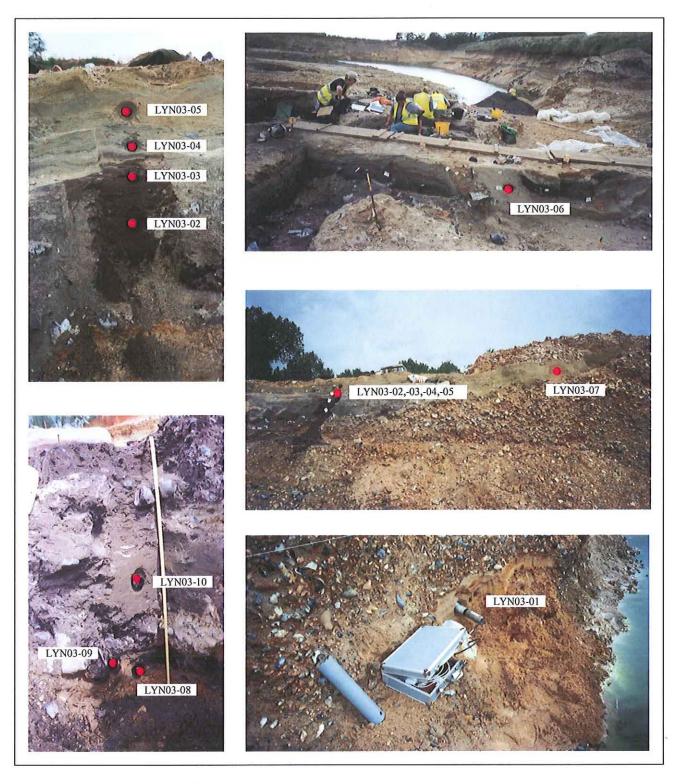


Figure 1 Location of OSL samples.

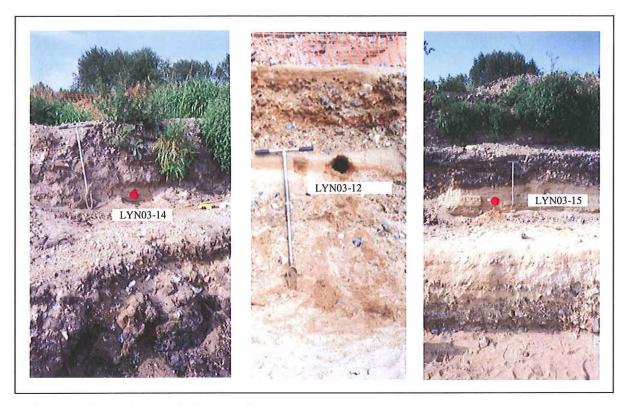


Figure 2 Location of OSL samples.

Methods

The physical basis of luminescence dating

When ionising radiation (predominantly alpha, beta, or gamma radiation) interacts with an insulating crystal lattice (such as quartz or feldspar), a net redistribution of electronic charge takes place. Electrons are stripped from the outer shells of atoms and though most return immediately, a proportion escape and become trapped at meta-stable sites within the lattice. This charge redistribution continues for the duration of the radiation exposure and the amount of trapped charge is therefore related to both the duration and the intensity of radiation exposure. Even though trapped at meta-stable sites, electrons become 'free' if the crystal is subjected to heat or exposed to light. Once liberated, a free electron may become trapped once again or may return to a vacant position caused by the absence of a previously displaced electron (a 'hole'). This latter occurrence is termed 'recombination' and the location of the hole is described as the 'recombination centre'. As recombination occurs, a proportion of the energy of the electron is dissipated. Depending upon the nature of the centre where recombination occurs, this energy is expelled as heat and/or light. Therefore, when the crystal grain is either heated or illuminated following natural or artificial laboratory irradiation (the 'dose') the total amount of light emitted (luminescence) is directly related to the number of liberated electrons and available recombination sites. This is the fundamental principle upon which luminescence dating is based.

In cases where the duration of dosing is not known (as is the case for dating), estimates can be made from laboratory measurements. The response (the sensitivity) of the sample to radiation dose (ie the amount of light observed for a given amount of laboratory radiation, usually β -radiation) must be established. From this relationship the equivalent radiation exposure required to produce the same amount of light as that observed following the natural environmental dose can be determined, and is termed the palaeodose or 'equivalent dose' (D_e). The palaeodose (measured in Gy) is therefore an estimate of the total dose absorbed during the irradiation period. When the dose rate (the amount of radiation per unit time, measured in μ Gy/a) is measured (or calculated from measured concentrations of radionuclides), the duration of the dosing period can be calculated using the equation:

Duration of dosing period = Palaeodose ÷ dose rate.

The technique of optical dating was first applied to quartz by Huntley *et al* (1985), and methodological details were further developed by Smith *et al* (1986) and Rhodes (1988). The technique was demonstrated to work well for aeolian samples by Smith *et al* (1990), and has further proved to provide useful age estimates for a range of sedimentary contexts ranging from aeolian (eg Stokes *et al* 1997) to glacial (Owen *et al* 1997) and fluvial contexts (Wallinga *et al* 2001, 2004). Further developmental research has introduced palaeodose measurement protocols that use a 'single aliquot regenerative-dose' (SAR) protocol (Murray and Wintle 2000). These protocols generally have the potential to provide improved accuracy (eg through correction of sensitivity change, interpolation rather than extrapolation of the palaeodose values) as well as increased precision. In some cases they may also provide an indication of incomplete zeroing of the luminescence signal at the time of deposition. Recent research within the laboratory (Rhodes *et al* 2003) has demonstrated the high precision and accuracy that may be achieved with this technique.

Sample preparation

The laboratory procedures were designed to yield pure quartz, of a particular grain size range, from the natural sediment samples. In order to obtain this material, samples were taken through a standard preparation procedure, as outlined below. All laboratory treatments were performed under low intensity laboratory safe-lighting, from purpose-built filtered sodium lamps (emitting at 588 nm).

After removal of the exposed ends of the sampling containers, the unexposed central portion of the sample was wet-sieved and the 180-255 μ m grain size was used for dating (see Appendix 1 for details of specific samples). The chosen fraction was treated with diluted hydrochloric acid (10%) to remove carbonates and then treated in concentrated hydrofluoric acid (48%) for 100 minutes. This treatment serves two purposes: (i) to dissolve feldspar grains, and (ii) to remove (etch) the outer surface of quartz grains (the only part of each quartz grain exposed during burial to natural alpha radiation). Any heavy minerals present were subsequently removed by gravity separation using a sodium polytungstate solution at 2.68 g cm⁻³. Finally, each sample was re-sieved to remove heavily etched grains. The order of the heavy-liquid

separation and second sieving are on occasion reversed for practical reasons, and for samples with extremely low yields, either or both of these treatments may be omitted after careful consideration. The prepared quartz samples were mounted on 1cm diameter aluminium discs for luminescence measurement using viscous silicone oil.

Various tests for sample purity are made. Sub-samples of the prepared material are examined using optical microscopy and the sample is exposed (within the Risø measurement system) to infrared (IR) light. Quartz generally does not produce measurable IR luminescence at room temperature whereas feldspar, which can suffer from anomalous fading of the infrared stimulated luminescence (IRSL) and OSL signals, or may be less rapidly bleached in some environments, produces an intense luminescence when stimulated with IR. The presence of a strong IRSL signal is therefore used as an indication for the presence of feldspar contaminants and is a criterion for rejection. In the rare cases where samples are rejected due to presence of high levels of IRSL, the prepared sediment sample is treated for ~ 2 weeks in concentrated fluorosilicic acid (H_2SiF_6 ; silica-saturated HF) which effectively dissolves non-quartz material. If following this treatment, IRSL persists then the sample is subjected to a further two week H_2SiF_6 acid treatment before proceeding to the dating phase (luminescence measurement) and the results are interpreted with caution and the possible contamination of the sample discussed.

The measurement sequence adopted for dating all the samples included a post-IR blue OSL procedure (Banerjee *et al* 2001) designed to deplete any feldspar contribution to the OSL signal, by preceding each OSL measurement with an IRSL measurement. The IR exposure reduces the size of feldspar contributions, besides providing an alternative means to determine a palaeodose. For samples with strong IRSL signals, significant feldspar contribution to the OSL may remain, and this is considered in the interpretation of the dates.

In order to determine the attenuating effect of pore water on the environmental dose rate of the sediments, additional samples were collected in the field and hermetically sealed. The moisture content of the sample was determined in the laboratory by weighing the sample before and after oven drying at 50°.

The single aliquot regenerative-dose (SAR) protocol

The SAR method is a regeneration procedure where the light level of the natural signal is converted into Gy via an interpolation between regenerated (ie known dose) points. The natural and regenerated signals are measured using the same aliquot. Sensitivity change commonly observed in quartz TL/OSL has previously precluded meaningful results being obtained this way. A key development reported by Murray and Wintle (2000) is that sample (aliquot) sensitivity is monitored following each OSL measurement (L_i) using the OSL response to a common test dose (S_i). Plots of L_i / S_i provide the necessary (sensitivity change corrected) data for interpolation. The procedure is further outlined in Figure 3.

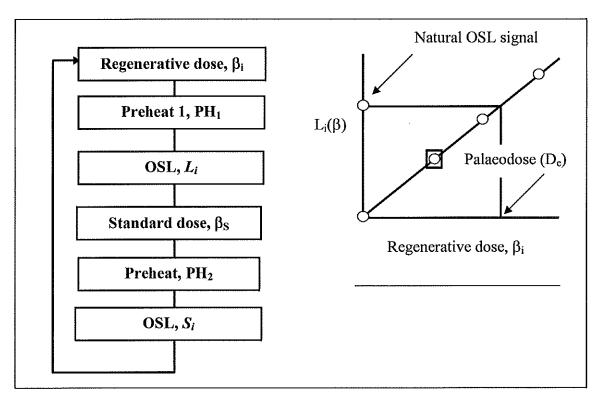


Figure 3 The SAR method. The procedure illustrated here is described in further detail in the text.

Steps 1-6 are repeated n times in order to produce the data points required for interpolation (the first dose β_1 being zero, to give a measure of the natural signal). Typically n=7 (ie the natural plus 6 regeneration points, including one zero dose point and one repeat point). PH₁ and PH₂ are usually different although Murray and Wintle (2000) report no dependence of the palaeodose on either (over the range of 200-280°C). The OSL signal is integrated over the initial part of the decay (to ~10% of initial intensity) and the background is taken as the light level measured at the end of each OSL measurement.

Murray and Wintle (2000) have introduced two further steps in to the measurement procedure. The first is the re-measurement of the first regenerated data point (indicated by the box in the explanatory Figure 3 above). The ratio of the two points (the "recycling ratio") provides an assessment of the efficacy of the sensitivity correction and the accuracy of the technique (large differences being suggestive of an ineffective technique). The recycling ratio (ideally unity) is typically in the range 0.95-1.05. The second additional step is a measurement of the regenerated OSL due to zero dose. This value gives a measure of the degree of thermal transfer to the trap(s) responsible for OSL during pre-heating. The ratio of this value to the natural OSL value (both corrected for sensitivity change) gives the "thermal transfer ratio" and ideally this should be in the range of 0.005-0.020.

Measurement procedures and conditions

Luminescence measurements were made using automated Risø luminescence measurement equipment. There are currently three different systems within the Luminescence Dating Laboratory that can be used for routine dating, the major difference between them being the optical stimulation sources. In two systems, optical excitation is provided by filtered blue diodes (emitting ~410-510nm), and in the third a filtered Halogen lamp (emitting ~420-560nm) is used. In all three systems, infrared stimulation is also provided using either an array of IR diodes or a single IR laser diode (depending on the measurement system). Luminescence is detected in the UV region on all systems, using EMI 9635Q bialkali photomultiplier tubes, filtered with Hoya U340 glass filters. Sample irradiation is provided in all cases by sealed ⁹⁰Sr sources at a rate of 1.5-3 Gy/minute depending on the system used.

The mean palaeodose for each sample was obtained from 12 aliquots (see Appendix 3 for further details regarding the statistics used in palaeodose and error calculations). All OSL measurements were made at 125°C (to ensure no re-trapping of charge to the 110°C TL trap during measurement) for between 50 and 100s, depending on the measurement system used. The signal detected in the initial 1st to 2nd seconds (with the stable background count rate from the last 12 to 24 seconds subtracted) was corrected for sensitivity using the OSL signal regenerated by a subsequent beta dose (β_s) . To ensure removal of unstable OSL components, removal of dose quenching effects, and to stimulate re-trapping and ensure meaningful comparison between naturally and laboratory irradiated signals, pre-heating was performed prior to each OSL measurement. Following each regenerative dose (β_i) and the natural dose, a preheat (PH₁) at 260°C for 10s was used for those samples with a palaeodose higher than 10 Gy. For the two Holocene samples (X1165 and X1166) a reduced pre-heat temperature of 220°C was applied. Following each test dose (β_s), a pre-heat (PH₂) of 220°C for 10s was applied to the older samples and in the case of the younger samples, this was reduced to 200°C for 10s (see Section 2.3 for further details of the SAR method). All the OSL measurements incorporated a post-IR blue OSL stage in which each OSL measurement is preceded by an IRSL measurement at 50°C, to reduce the effects of any residual feldspar grains (Banerjee et al 2001) but the SAR procedure is otherwise unchanged.

For every sample, a routine internal laboratory procedure referred to as DELIA (D_e Luminescence Initial Assessment) was applied prior to the main SAR measurement in order to determine their approximate palaeodose value. This consisted in the use of a simplified version of the SAR measurement protocol applied to a limited number of three test discs in order to determine the internal variability, the OSL and TL signal form and sensitivity, as well as the magnitude of any IRSL signals. This considerably assists in the optimal selection of regenerative and test dose values, number of aliquots to measure, and the preheat combination selected. Quartz samples showing high levels of IRSL at this stage are given an extended (usually 14 days) treatment in fluorosilicic acid (H_2SiF_6). Only one sample (X1104) required additional H_2SiF_6 treatment.

3 Results

The OSL dating results including age estimates, palaeodose, and environmental dose rate measurements are summarized in Table 3. Further details regarding individual samples and radioactivity data may be found in Appendix 1. Factors affecting the dose rate determinations and the statistics used in error calculations are described in more detail in Appendices 2 and 3. The age estimates presented here include the preliminary results reported for four samples in the Interim OSL report submitted in December 2002. Adjustments for the correct burial depth and the true water content, which were not available at the time the initial samples were measured, resulted in minor changes to the preliminary dates reported in the pilot study.

Field code	Lab. code	Palaeodose (Gy)	Dose rate (mGy/a)	In-situ <i>γ</i> -ray spectrometry	OSL age (ka) ± 1 sigma
LYN03-01	X1098	47.90 ± 2.80	0.61 ± 0.04	Yes but poor geometry	78.6 ± 6.7
LYN03-02	X1099	56.55 ± 2.51	0.87 ± 0.06	Yes	64.8 ± 5.5
LYN03-03	X1100	60.86 ± 3.83	1.04 ± 0.07	Yes	58.3 ± 5.6
LYN03-04	X1101	66.84 ± 2.93	1.20 ± 0.06	No	55.9 ± 3.9
LYN03-05	X1102	67.64 ± 2.65	1.27 ± 0.05	Yes	53.4 ± 3.3
LYN03-06	X1103	41.30 ± 1.83	0.86 ± 0.04	Yes	48.0 ± 3.2
LYN03-07	X1104	72.50 ± 3.10	1.19 ± 0.06	Yes	60.7 ± 4.3
LYN03-08	X1160	60.00 ± 3.38	0.92 ± 0.08	Yes	65.0 ± 6.9
LYN03-09	X1161	47.88 ± 2.20	0.69 ± 0.05	No	69.9 ± 6.1
LYN03-10	X1162	45.86 ± 1.61	0.77 ± 0.05	Yes	59.5 ± 4.9
LYN03-11	X1163	45.82 ± 2.25	0.80 ± 0.04	Yes but poor geometry	57.4 ± 4.2
LYN03-12	X1164	15.23 ± 0.98	0.44 ± 0.02	Yes	34.7 ± 2.9
LYN03-13	X1165	0.72 ± 0.03	0.70 ± 0.03	Yes	1.03 ± 0.06
LYN03-14	X1166	0.71 ± 0.05	0.83 ± 0.04	Yes	0.85 ± 0.08
LYN03-15	X1167	23.12 ± 0.78	0.71 ± 0.04	Yes	32.4 ± 2.2
LYN03-16	X1837	115.93 ± 9.20	0.65 ± 0.09	No	175.6 ± 27.7
LYN03-17	X1838	131.35 ± 14.20	0.78 ± 0.09	No	169.2 ± 26.9

Table 3 Summary of OSL dating results. The results are based on luminescence measurements of sand-sized quartz (180-255µm). All samples were measured using a modified single aliquot regenerative-dose (SAR) post-IR blue OSL protocol (Murray and Wintle 2000, Baneriee et al 2001). Gamma dose rates are based on in situ 2-ray spectroscopy measurements. Beta dose-rate values were calculated using the concentrations of uranium, thorium, and potassium as determined by neutron activation analysis (NAA). The presence of large stones and the lack of sufficiently deep sections occasionally prevented making direct measurements. In these instances, the gamma dose rate was calculated either from the concentrations of radioactive elements as determined by NAA (X1168 & X1169) or from interpolated gamma dose-rate values of neighbouring samples (X1101 & X1161). Corrections were made in the age calculation for the water content of the sediment samples using the correction factors outlined in Aitken (1985) and taken from Zimmermann (1971). The contribution of cosmic radiation was calculated as a function of latitude, altitude, burial depth, and average over-burden density according to the formulae of Prescott and Hutton (1994). Further details regarding individual samples may be found in Appendix 1.

Very low IRSL values were occasionally observed for some aliquots, suggesting good quartz separation had been achieved during sample preparation. OSL age estimates are based on sand-sized quartz grains extracted from each sample and the measurement of six or 12 aliquots. Dose rates were calculated by combining the results of neutron activation analysis (NAA) and on-site gamma-ray spectroscopy measurements. The latter provided *in situ* gamma dose-rate measurements whereas the beta dose rate values were calculated from the concentrations of K, Th, and U as determined by NAA.

All samples displayed well-defined luminescence signals and other OSL characteristics were also found to be well suited for optical age determination. Saturation has not provided a limitation to the dating of these samples. A moderate and acceptable degree of inter-aliquot variability was observed with standard deviations of 8-15%, typical of Pleistocene fluvial samples. Most samples showed excellent recycling ratios, having mean sample recycling ratios less than 1% from unity. The size of the mean thermal transfer signal was generally below 2% and only rarely found to be up to 5% for individual aliquots.

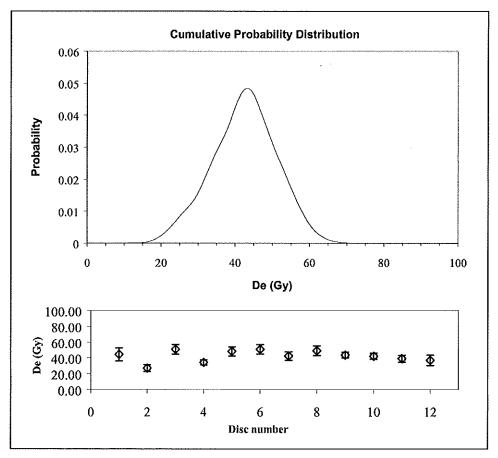


Figure 4 Example of the probability distribution (top) and scatter diagram (bottom) of palaeodose (D_E) estimates obtained from 12 multi-grain single aliquots prepared from sample X1103.

Overall, the observed luminescence characteristics (low variability, good sensitivity, good recycling, and low thermal transfer values) suggest that the calculated age

estimates are reliable. Occasionally, one or two aliquots gave higher palaeodose values, and these were rejected from the age analysis, interpreted as aliquots containing grains suffering incomplete bleaching. In all cases, this made little difference to the calculated ages. The luminescence dating results appear to be broadly consistent with the archaeological expectations, suggesting that the measured OSL signals were sufficiently stable, and the large majority of mineral grains had been well bleached and generally undergone complete zeroing at the time of deposition.

For samples LYN03-16 and LYN03-18, which were not collected by laboratory staff, no on-site radioactivity measurements are available and the environmental dose rate is based entirely on the concentrations of radioactive elements as determined by NAA. For this reason, these age estimates should be interpreted with some degree of caution. Both samples provided age estimates which are substantially older than those directly associated with deposits from the main palaeochannel at the archaeological site. Although no gamma dose-rate measurements are available, it is unlikely that any difference between the true dose rate and the one derived from NAA could account for this substantial gap in age. Indeed, the samples were collected from thick relatively homogenous deposits of sand and away from major sedimentary boundaries, a situation for which one would expect relatively good agreement between *in situ* and laboratory-based dose-rate measurements.

The age estimates obtained from samples in various sections generally appear to be in stratigraphic order. Sample LYN03-01 (see Figure 1) provided the oldest date for the base of the stratigraphic sequence and was collected from a thick sand lens within the fluvial gravels and underlying the main palaeochannel. The dates for the organic sands within the main palaeochannel (see Figure 2) suggest that these accumulated between c 65ka and 57ka. Two series of four [LYN03-02 to LYN03-05] and three [LYN03-08 to LYN03-10] samples were collected at two sampling localities with an additional single sample [LYN03-11] collected elsewhere. No in situ gamma dose-rate measurements could be obtained for samples LYN03-04 and LYN03-09, due to closely spaced sampling. In both these instances, a linear interpolation of gamma dose rates was used between overlying and underlying measurement locations with augmented errors. Although, this approach seems justified, one can not exclude some degree of age over- or under-estimation. This may explain the apparent age inversion noticed between sample LYN03-09 dated to 69.9 ka and the underlying sample LYN03-08 dated to 65.0ka. Both samples however have overlapping errors.

OSL dates from the beds of sand immediately overlying the main palaeochannel provided dates ranging from 53.4ka to 60.7ka [LYN03-04, LYN03-05, LYN03-07]. A later channel feature cutting through these sands and truncating the northern edge of the main palaeochannel was dated to 48.0ka. This sample [LYN03-06] was collected from a thick sand unit within the fill of the U-shaped channel which consisted of a sequence of gravel, sand, and organic beds (see Figure 1). This younger channel was succeeded by a series of braided river channels as evidenced by a variety of sandy units and gravel deposits exposed in several sections. Four radiocarbon dates (GrN-28395, GrN-28396, GrN-28397, GrN-28398) obtained from contexts 30377 and 30378 in the East facing section exposed on the Western edge of the pit provided dates ranging from c 30ka to 36k. Unfortunately, no related OSL samples were

collected from these sedimentary units although, samples LYN03-12 and LYN03-15 (see Figure 2) obtained from contexts 20205 and 20305 elsewhere on site, provided similar age estimates of respectively 34.7 and 32.3ka.

Overlying the sequence of braided river channels was a succession of meandering Holocene channels and sediments which occasionally contained organic matter as well as archaeological finds. Two samples [LYN03-13 and LYN03-14] were secured from these upper sediments in order to complete the geochronological framework for the site. Both these samples also enable to check the reliability of the OSL dating by comparison with radiocarbon dates. In the case of sample LYN03-13, collected from context 20317 in the South facing section along the northern edge of the excavation, it is possible to directly compare the OSL age estimate with the radiocarbon date obtained from plant debris in the lowest monolith of sample 30085. The OSL age estimate of 1030 ± 60 years (AD 910-1030) is in good agreement with two ¹⁴C dates of 1050 ± 110 BP (GrN-28399) and 1310 ± 80 BP (GrN-28400) obtained on two samples from context 30085. Both radiocarbon samples give a calibrated date range of AD 660-980 based on their combined weighted mean. OSL sample LYN03-14 is also in good agreement with the radiocarbon dates, giving as expected, a slightly younger date of 850 ± 80 years BP (AD 1070-1233).

4 Conclusion

The observed luminescence characteristics (good sensitivity, low variability, good recycling, and relatively low thermal transfer values) suggest that the calculated age estimates are reliable. Although, sampling for optically stimulated luminescence (OSL) dating was not always optimal due to the lack of deep sections available in the central area of the site and the removal of overlying sediments through past quarrying activity and archaeological excavation, the OSL dating programme must be considered as having been highly successful. Optical dates based on the measurement of the signal from quartz have provided a complete and relatively secure chronological framework for the sedimentary sequence of the Middle Palaeolithic open-air site. OSL dating clearly has very high potential for dating fluvial sediments of this kind and over this time scale in this region. This case study also illustrates the exciting possibilities for building robust chronologies for aggregate deposits formed as a result of fluvial activity and for developing regional palaeo-environmental reconstructions.

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