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THE POTENTIAL FOR DATING THE OLD SCATNESS SITE, SHETLAND, BY OPTICALLY STIMULATED LUMINESCENCE*

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A series of deposits from the agricultural infield of the multiperiod settlement mound, Old Scatness, were investigated for their potential to yield optically stimulated luminescence dates. Luminescence properties of quartz grains were found to vary through the sequence, but dates were successfully obtained from five deposits, including anthropogenic soils, windblown sands and sands within midden deposits. Single-aliquot equivalent dose measurement was found to be the most appropriate method for dating the deposits. The OSL dates obtained accorded well with the dates provided by archaeological evidence and included the post-medieval, Iron Age, Bronze Age and Neolithic periods of Shetland, while a substantial midden was dated to the Bronze/Iron Age transition.

KEYWORDS: ANTHROPOGENIC SOILS, DATING, OPTICALLY STIMULATED LUMINESCENCE, SCATNESS, SEDIMENTARY QUARTZ, SHETLAND

INTRODUCTION

Optically stimulated luminescence (OSL) is one of three dating techniques that use minerals as radiation dosimeters, the other two being thermoluminescence (TL) and electron spin resonance (ESR). When minerals such as quartz and feldspar are exposed to ionizing radiation from naturally occurring radionuclides in the environment, freed electrons can become trapped in defects within the crystal lattice. Energy is emitted, in the form of light, by a proportion of these charges when they are liberated and recombine. In OSL, this is realized by stimulating the mineral with light of a certain wavelength (energy), which is different to that of the luminescence itself, and measuring the intensity of emission. If the amount of incident radiation required to produce the natural signal is known (the equivalent dose, D_E), and a history of radiation incident upon the sample can be constructed (annual dose), then the date at which the signal was last reset can be found. The OSL signal in sediments is reset by exposure to a few minutes of daylight (bleaching). The technique is most generally applied to quartz and feldspar grains within archaeological materials, such as heated and unheated sediments, pottery, and heated stone (Krbetschek *et al*. 1997; Roberts 1997; Wagner 1998, Ch. 7.2).

The OSL dating of sediments was reviewed by Prescott and Robertson (1997). Most of the published work on British archaeological sites has been conducted at the Oxford University Research Laboratory for Archaeology (Smith *et al*. 1990; Rees-Jones and Tite 1997). The latter have concentrated on alluvial deposits associated with archaeological contexts. In general, these have been successfully dated, and aeolian deposits have also proved acquiescent. Colluvium has

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yielded more variable results, however, and pedogenic deposits have seen little published work (Prescott and Robertson 1997). Reliable dating of such deposits, which have been subject to incomplete bleaching and/or post-depositional mixing, has not been achieved (see, e.g., Huntley *et al.* 1985; Musson and Wintle 1994). However, Lang and Wagner (1996) successfully dated colluvium in loessic layers and pit fills, arising from agricultural activity, using infrared stimulated luminescence (IRSL). In a more recently published study (Gilbertson *et al.* 1999), OSL dates were derived from a variety of source materials, from sites in the Outer Hebrides, including soil horizons and midden deposits.

The general aim of luminescence research at Old Scatness (Burbidge 1998) was to establish the suitability, or otherwise, of deposits at the site for OSL dating using green light stimulation of the quartz grains extracted from of a variety of samples. A further, and technically novel, aspect was to evaluate the dating potential of anthropogenic soils themselves, which were present in the site's agricultural infield area. These could then be used to provide a site chronology based on dates derived from archaeological contexts formed directly by human activity, eliminating the need to rely on association of artefacts or adjacent natural deposits.

THE ARCHAEOLOGICAL SITE AT OLD SCATNESS, SHETLAND

The excavation at Old Scatness lies on the westerly edge of Sumburgh Airport, at the southern end of mainland Shetland (04389, 11106. O.S. Sheet HU 31/41). The site comprises a raised settlement mound containing evidence of regular, if not continuous, human activity from the Bronze Age to the present day. Occupation phases have been identified by excavation of structures and associated midden deposits from the Iron Age, Pictish, Norse and post-medieval periods. Beyond the site there is evidence for a buried soil sequence, representing an arable infield area around the site (Dockrill 1998). The OSL dating programme focused on deposits within this agricultural infield area beyond the confines of the site, rather than within the occupation complex itself. There were three main reasons for this:

(i) Many other methods were available for dating the artefact-rich occupational areas, whereas OSL dating of sediments themselves provides the only way of directly dating a complete agricultural sequence.

(ii) There was considerable variation between on-site depositional sequences, due to the intensity of occupation.

(iii) There were a variety of sediment deposition mechanisms within the occupation complex, many of which were not conducive to the complete bleaching of the constituent minerals.

By using a single stratigraphic section of the buried soil sequence, the maximum amount of otherwise unobtainable information could be gathered from a relatively simple, coherent sequence of contexts.

Previous excavation and coring has defined the area of the agricultural infield at Old Scatness, and test pits had revealed distinctive sequences of buried soils and middens, interleaved with lenses of windblown sand derived from the adjacent beach (Dockrill 1998). Analyses of the micromorphology and artefactual content of the soils yielded evidence of arable cultivation and anthropogenic enhancement by addition of domestic waste and burnt peat and turf (Simpson *et al.* 1998). It was also shown that, while quartz was present throughout and dominated the mono-mineral fractions of the lower sequences, a transition to $CaCO_3$ (shell sand) commenced after the Iron Age (Simpson *et al.* 1998).

Although there were significant polymineral fractions within the deposits, they were considered unsuitable for this initial, and necessarily rapid, study. This was for two main reasons: the mean particle size of the deposits was large (~300 μm; Simpson *et al.* 1998), whereas polymineral dating is applied to the fine-grain fraction (see, e.g., Lang and Wagner 1996), and the presence of radioactive elements in some minerals present would have necessitated time-consuming measurement of the α dose-rate. In addition, any feldspars present may have exhibited anomalous fading, rendering the equivalent dose measurements inaccurate (Duller 1997). Instead, the investigation focused on green light stimulation applied to the coarse grain quartz fraction (90–150 μm). The sub-aerial deposition mechanism of the sand-blow events means that each one contains minerals from the same local (as opposed to ultimate) source, and is most conducive to the bleaching of the OSL signal. Therefore dates from the sands should be more reliable and could be used to cross-check those from the soils.

SAMPLING AND ANNUAL DOSE MEASUREMENT

Samples were taken from a test pit (area H) specifically excavated for the purpose. Area H was situated adjacent to, but outside, the main occupation area and measured 1 m by 1.4 m, with a maximum depth of 1.8 m (Fig. 1). All contexts below the two uppermost soil layers were sampled, and indicative dates were obtained from the artefacts within each context and from its stratigraphic relationship with the main excavated area. Samples were collected by the horizontal insertion of sharpened steel tubes of 1.5 in diameter into a cleaned vertical sediment face. Internal plugs were used to prevent collapse within the tube, and hence contamination of the sample by bleached grains from the section face. Two tubes were inserted into most contexts, one to be used for luminescence measurements and the other for dose-rate measurements. Once the section had been planned and levelled to the site datum, separate water content samples were taken from all contexts and kept in sealed containers.

Direct measurements were made of the gamma activity of contexts 2102, 2105, 2107d and 2110 (Table 1) using a Harwell 0928 four-channel gamma-ray spectrometer with a standard

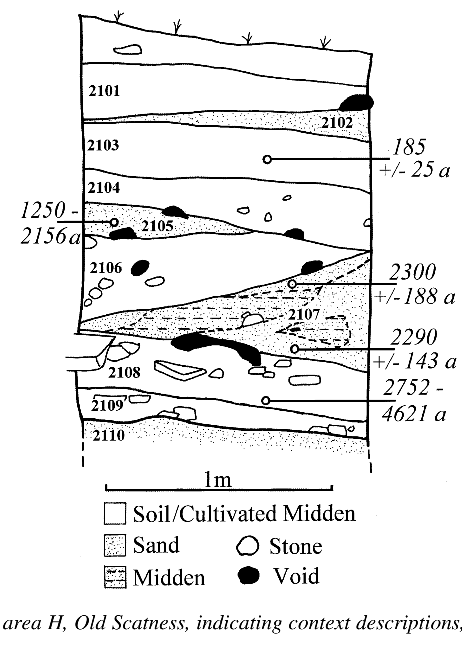

Figure 1 *A section drawing of area H, Old Scatness, indicating context descriptions, sample locations and OSL ages.*

probe containing a 1.75 in by 2 in NaI scintillation crystal. The performance of the spectrometer was checked in the laboratory in a brick reference block and the field measurements processed in the conventional manner (Aitken 1985, 320). The measurement for context 2105 appeared anomalously high and was not used. Dose-rates to the remaining contexts were deduced from interpolation of the direct measurements with the following assumptions. Context 2104 unconformably seals contexts 2105 and 2106, indicating truncation of the sequence at some time after the deposition of the sand lens 2105. It is assumed that the deposits above this unconformity have activity similar to that measured for context 2102, while lower deposits have activities similar to contexts 2107d and 2110. The gamma activity in the region of the unconformity due to both the upper and lower layers was calculated using the tabulated data of Lovborg (Aitken 1985, App. H). The beta dose-rate was determined in the laboratory by beta-TLD (Bailiff 1982). The cosmic dose-rate was calculated for depths between 10 and 170 cm. It was found to change by less than the error term allowed for beta dose-rate, and the mean of values from 10 and 170 cm depth was used. Secular equilibrium was assumed from the Th/U values obtained from the gamma spectrometry data which were not anomalous, but no explicit checks for disequilibrium were made.

In situ water content was measured from each dedicated sample, and used to calculate *in situ* water content for the samples processed for OSL measurement. The volume (as extracted from the tubes) and dry mass of the OSL samples were measured and used to calculate bulk density and porosity, and hence estimate saturated water content. These data were combined to produce W and F as presented in Table 1 (Aitken 1985, 75). The accumulated environmental evidence was considered to indicate that the climate in Shetland had not altered significantly over the timescale involved (see, e.g., Bennett *et al*. 1992). Values of *in situ* water content were therefore used as measured, water retention roughly reflecting clay and organic content, but a 20% error term was included to account for variations. Total dose-rates were calculated following Aitken (1985).

Table 1 *Radioactivity measurements and dose-rates. The standard deviation of the beta activity measurements, extrapolated gamma dose rates and W was estimated as 5%. The standard deviation of F was estimated as 20%. The cosmic dose rate used was 0.22 ± 0.7 Gy ka^{-1} (see text)*

Context	*Sediment type*	*Water content*		*Beta dose-rate (Gy ka^{-1})*		*Gamma dose-rate (Gy ka^{-1})*		*Annual dose (Gy ka^{-1})*
		W	*F*	*Measured*	*Corrected*	*Measured*	*Interpolated*	
2102	Calcareous sand	0.49	0.14			0.59		
2103	Sandy silt loam	0.38	0.34	1.66	1.29 ± 0.11		0.59	2.09 ± 0.13
2104	Sandy silt loam	0.55	0.40					
2105	Quartz sand	0.57	0.17	1.91	1.53 ± 0.12		0.72	2.47 ± 0.14
2106	Sandy silt loam	0.54	0.34					
2107a	Quartz sand	0.40	0.17	1.97	1.63 ± 0.11		0.75	2.60 ± 0.14
2107c	Quartz sand	0.45	0.11	2.02	1.71 ± 0.11		0.76	2.69 ± 0.14
2107d	Sandy clay					0.76		
2107e	Sand + sandy clay mix	0.36	0.39	1.90	1.46 ± 0.13		0.76	2.44 ± 0.15
2108	Sandy clayey silt	0.50	0.56	1.90	1.29 ± 0.14		0.76	2.27 ± 0.16
2109	Sandy clayey silt	0.49	0.54	2.07	1.69 ± 0.12		0.76	2.67 ± 0.15
2110	Quartz sand	0.43	0.19			0.76		

LUMINESCENCE MEASUREMENT

After carefully removing superficial material from the ends of the selected tubes, under subdued red light conditions, OSL subsamples were excavated and dried. The 90–150 μm fraction was isolated by disaggregation and sieving, and then etched in 40% HF for 45 min to remove both the non-quartz component and the outer surface of the quartz grains (Fleming 1970) and 15% HCl to remove fluorides. Samples were re-sieved and aliquots of ~1 mg were prepared by deposition on to 10 mm diameter stainless steel discs. Subsamples were checked under an optical microscope after each sieving. Prior to deposition, typical composition (by number) was roughly 90% clear and frosted quartz, 10% very dark heavy minerals and 0–5% light brown to grey–green unidentified crystals.

OSL measurements were conducted using an automated Risø DA12 reader. Stimulation was provided by a filtered tungsten–halogen lamp (GG420 + interference filter set), giving stimulation in the wavelength range 420–550 nm. The stimulating light was directed on to the sample via a liquid light guide, which gave an incident power of ~16 mW cm^{-2}. Luminescence was detected by an EMI 9235QA photomultiplier via a 5 mm thick stack of two Hoya U340 filters (50% transmittance between 320 and 390 nm). The luminescence measurement was made with the aliquot held at 125 °C (Wintle and Murray 1997), and the duration of the OSL stimulation was chosen so that the signal decayed to less than 5% of the initial value during stimulation. A calibrated ^{90}Sr/^{90}Y β source, giving a dose-rate of 0.605 ± 0.018 Gy min^{-1}, was used for irradiation.

It was decided to use a single-aliquot regenerative protocol (Table 2) with a preheat to 180 °C at 2 °C s^{-1}, to reduce sensitization and the effects of inter-aliquot scatter (Wintle 1997, 7.1) observed in initial tests. A low pre-heat was considered appropriate for the Old Scatness samples, since they had been buried for (only) a few thousand years in low ambient temperatures (Murray *et al.* 1997). The pre-heat is used to remove charge from traps which would be unstable over the burial period of the sample, and may be used to re-sensitize the sample (to approximately its natural level) following irradiation in the laboratory, such that the natural and regenerated measurements are comparable (Wintle and Murray 1999). The regenerative beta doses were chosen such that OSL response to B1 was equivalent to the natural signal, while the response to B2 and B3 bracketed the natural signal. The repeated measurement of the response to

Table 2 *The measurement sequence*

Preheat to 180 °C at 2 °C s^{-1}
Measure OSL at 125 °C for 100 s
Regenerative beta dose B1
Preheat to 180 °C at 2 °C s^{-1}
Measure OSL at 125 °C for 100 s
Regenerative beta dose B2
Preheat to 180 °C at 2 °C s^{-1}
Measure OSL at 125 °C for 100 s
Regenerative beta dose B3
Preheat to 180 °C at 2 °C s^{-1}
Measure OSL at 125 °C for 100 s
Regenerative beta dose B1
Preheat to 180 °C at 2 °C s^{-1}
Measure OSL at 125 °C for 100 s

dose B1 allowed a sensitivity correction to be made, assuming sensitization to be linear throughout the measurement cycle. While this assumption is not generally valid, it is thought sufficient for young samples when using low pre-heats (Godfrey-Smith and Haskell 1993). Use of the measurement protocol outlined in Table 2 resulted in sensitization of between −6.0% and +2.1% per measurement cycle for the Old Scatness samples. Equivalent doses were determined by interpolation of the dose-response curve using unweighted linear regression. Typically, three aliquots were used for each sample and the equivalent dose determined from the weighted mean of these measurements.

DISCUSSION

Equivalent doses were measured for three anthropogenic soils (2103, 2108c and 2109-2) and three sand deposits (2105, 2107a and 2107e), two of which were contained within midden deposits, and are presented in Table 3 together with the age values. The OSL results provide a convincing sequence of increasing age with depth, although the high aliquot-to-aliquot variation in equivalent dose for samples 2105 and 2108c prevents precise determination of the age. Root working across the unconformity at the base of 2104 was observed during excavation. This could have resulted in scatter in the D_E of 2105 and to a lesser extent 2107a, a context that also contained lenses of midden material. Another possibility is contamination by feldspars, for which aliquots were not individually tested. The equivalent dose determined for sample 2109-2 is anomalously low when compared to the rest of the sequence. This could be due to inadequate pre-heating or sensitivity correction.

The formation of the area H sequence may be interpreted as follows (Fig. 1). At the base of the sequence lies a thick sand layer (2110), which is not anthropogenically modified and may date to the early Holocene (Dockrill personal communication). It could thus relate to the coversands reported by Gilbertson *et al.* (1999) in the Hebrides, whose work also indicates the possibility of later reworking. A soil was established above this (2109), containing artefacts thought to be Neolithic. Although ard marks were not evident in the base sand, this soil was stratigraphically equivalent to a cultivated soil, assigned to the early Bronze Age (Dockrill 1998; Simpson *et al.* 1998). OSL measurement on the base of the second soil (2108), although yielding two varying ages, indicates that the era in which it was initiated was likely to lie between 4621 ± 312 and 2752 ± 202 years ago. Although unsupportable statistically, the formation of context 2108 as a

Table 3 *Equivalent doses and age values: Ali., aliquot; WM, weighted mean*

Context	*Single-aliquot equivalent doses (Gy)*				*Age values (a)*			
	Ali. 2/20	*Ali. 3/21*	*Ali. 4/22*	*WM*	*Ali. 2/20*	*Ali. 3/21*	*Ali. 4/22*	*WM*
2103	0.46 ± 0.59	0.39 ± 0.42	0.38 ± 0.02	0.39 ± 0.05	221 ± 280	187 ± 201	184 ± 16	185 ± 25
2105	5.33 ± 0.83	3.09 ± 0.08	4.48 ± 0.77	*	2156 ± 194	1250 ± 79	1812 ± 330	*
2107a	6.55 ± 0.79	6.05 ± 1.05	5.60 ± 0.40	5.97 ± 0.49	2521 ± 332	2328 ± 423	2156 ± 194	2300 ± 188
2107e	5.91 ± 1.19	5.68 ± 0.82	5.23 ± 0.84	5.59 ± 0.35	2423 ± 511	2328 ± 366	2143 ± 370	2290 ± 143
2108c		10.48 ± 0.19	6.24 ± 0.21	*		4621 ± 312	2752 ± 202	*
2109	2.70 ± 0.21	1.92 ± 0.22	3.75 ± 0.31	2.88 ± 0.92				

* Weighted means were not calculated for 2105 and 2108c due to the very large scatter in the data–association cannot be demonstrated.

soil under cultivation would indicate the older value as a better indication for its commencement. It is therefore probable that the primary soil is Neolithic, and that 2108 was cultivated from the early Bronze Age.

The OSL ages (Fig. 1) showed that the midden sequence 2107 was deposited effectively as a single event, and that this was most likely to have occurred around 300 BC. It may therefore be associated with a refocusing of the settlement in the late Bronze Age or early Iron Age (the transition is rather diffuse in Shetland). The age obtained from 2105 indicates that this was deposited within 1 ka of 2107, and may therefore be broadly assigned to the Iron Age. However, evidence for the mixing of younger material into 2105 suggests that formation of the cultivated midden sequence, 2106, should be associated with the Broch period rather than the Pictish.

The soils above the unconformity at the base of 2104 appear to be part of a coherent sequence, leading to the post-medieval age found from 2103, but their date of initial formation after the late Iron Age remains unclear. Elsewhere on site, 17th-century ceramics had been found in layers immediately underlying calcareous sands, stratigraphically equivalent to 2102, which are thought to date to the 18th century (Dockrill and Bond 1999).

The age range measured from 2108 strengthens the argument for a pre-Broch settlement on the site, contemporary with the nearby prehistoric house site of Sumburgh (Lamb 1985). Accumulation of refuse on the site infield around the Bronze Age/Iron Age transition (2107), and the subsequent creation of 2106 during the Iron Age, indicate a short period of intensive non-agricultural activity on this area of the site, which may have been related to expansion or refocusing of the settlement. The age measured from 2103 implies either intensive cultivation, thoroughly bleaching the OSL, followed by a late C18 sand blow (2102), or that sand inundation came later in this area than elsewhere on the site.

CONCLUSIONS

The OSL ages obtained from the area H sequence of Old Scatness have demonstrated that the technique can be successfully used on this site. The luminescence signal of the soils and sands was primarily derived from sub-aerial quartz, indicating that OSL using quartz can be successfully applied to similar depositional environments elsewhere. The associated aims of the project, to investigate the potential for dating anthropogenic soils by OSL, and to define the phases of agricultural activity were also fulfilled, but would benefit from further work. The results indicated that a site chronology can best be defined by dating both the anthropogenic contexts, and the natural ones constraining them. Despite the behavioural unsuitability of the uppermost and lowermost contexts for dating, and the various problems with the measurement protocols employed, a coherent chronology was determined, spanning the majority of the infield sequence at Old Scatness.

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