The central lowlands of the Hunter Valley, NSW:

Why so few early sites have been found in this archaeologically-rich landscape

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Appendix 1

Cheshunt Dune Optical Dating

Sample Collection and Preparation

The two sediment samples were collected in steel coring tubes driven into freshly exposed profiles. Each core contained about 600 g sediment; sub-samples were collected for in situ water content measurement and laboratory assay of the radioisotope concentrations; in situ gamma-ray spectrometry was performed in the core holes.

In the ANU laboratory, quartz grains of 90–125 mm diameter were extracted under low-intensity red light in a procedure involving sequential HCl acid digestion, dry sieving, heavy liquid flotation (collecting <2.68 g cm^{-3} fraction), and then etching in 48% HF acid for 40 minutes to remove surface discoloration and the outer 6–8 mm alpha-particle irradiated shell.

Equivalent Dose Measurement

The protocol selected at that time to measure the radiation dose accrued since burial, termed the 'equivalent dose' (ED), was the multiple-aliquot Australian slide method (Prescott et al. 1993). This was chosen on account of its reliability for dating when adequate quartz is available and pre-depositional bleaching and minimal post-depositional mixing can be safely assumed (see for example, Magee et al. 2009). The optical dating technique itself is extensively discussed in Aitken (1998). The Australian slide method combines growth curves measured using multiple-aliquot additive-dose and multiple-aliquot regenerative-dose protocols to construct a composite growth from effectively zero luminescence to near-saturation. ED is then determined by an interpolative process.

Each of the additive-dose and regenerative-dose growth curves was comprised of 64 separate aliquots, each aliquot consisting of approximately 5–6 mg of etched quartz, attached by silicone oil to the central 7 mm diameter of stainless steel discs. OSL was measured using an Elsec Type 9010 automated reader with UV OSL emissions detected by an EMI 9235QA photomultiplier tube optically filtered by one UG 11 and one U-340 filter. Optical stimulation of 500±80 nm was provided using a filtered halogen lamp; irradiations were performed using an Elsec Type 9022 beta irradiator containing a 3.7 GBq ^{90}Sr/^{90}Y beta plaque and delivering a dose-rate of 0.0415±0.0013 Gy/s.

The multiple aliquot additive-dose measurement involved an initial sampling of the 'natural' OSL during a short illumination (typically < 0.5 mJ) for normalisation purposes. Laboratory irradiation was then administered, after which all aliquots receive simultaneous preheating at 220°C for 300 s in a purpose-built oven. The OSL light sum from each aliquot was then measured with the sample held at 20°C. The multiple-aliquot regenerative-dose procedure differs from the additive-dose procedure only in that following the normalisation exposure, 54 discs were reset by 8 hours exposure to an unfiltered 1000 W halogen lamp prior to dosing; the other 10 discs remain unbleached. Following background subtraction and normalisation, the multiple-aliquot additive-dose and multiple-aliquot regenerative-dose growth curves were constructed, each using the 64 aliquots, and then combined using the Australian slide software. For both samples a saturating exponential-plus-linear growth curve was fitted (scaling factor = 1.00) to find ED; the resulting growth curves are shown in Figure A1.

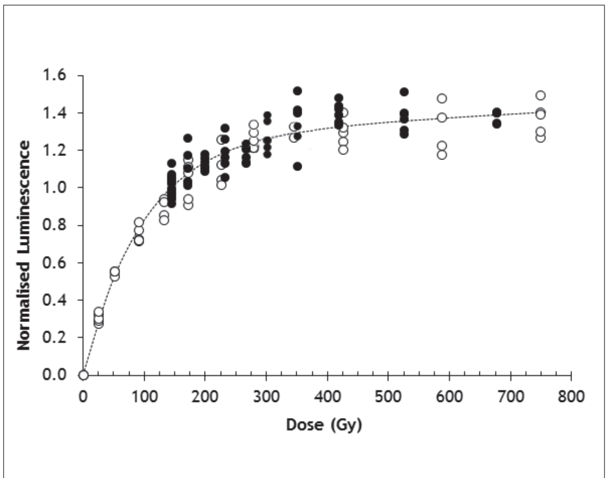

Figure A1 An example of the Australian slide growth curve, using sample CS2.25 (ANU$_{OD}$1364). Black open circles represent regenerative-dose data, black filled circles represent additive-dose data.

Dose-Rate and Age Determination

The environmental dose-rate was calculated by measuring the radioisotope concentration of the sediment, and then applying dose-rate conversion factors and correcting for the soil water content, and adding the calculated values for the cosmic ray dose-rate (using Prescott and Hutton 1994).

Concentrations of the radioisotopes Th and K (including Rb) were measured by neutron activation analysis (NAA), and delayed neutron analysis (DNA) was used to measure U (Becquerel Laboratories, Lucas Heights Science and Technology Centre). Concentrations were also calculated from the data collected using in situ NaI gamma-ray spectrometry. U and Th concentrations in the HF acid-etched quartz used for OSL measurements were assumed as 10% of the bulk sediment activity (following Aitken 1998), and the efficiency with which alpha irradiation induced OSL was assumed as 0.05±0.02 following Questiaux (1990).

Ages were calculated using the 'AGE' program (Grün 1999, 2009), incorporating the dose-rate conversion factors of Adamiec and Aitken (1998). Radioisotope and environmental data are shown in Table A1 along with the ages calculated: CS1.25 dated to about 83±4 ka, and CS2.25 to about 88±4 ka.

Warkworth Sand Sheet Optical Dating

The validity of the original optical dating performed at ANU on three samples collected from Warkworth, as reported in Hughes et al. (2003), was questioned by Scarp Archaeology (2009). Fortunately, portions of these original

Cheshunt	CS1.25			CS2.25		
	$ANU_{OD}1363$			$ANU_{OD}1364$		
Burial depth (m)	1.25			2.25		
Quartz grain diameter (mm)	107.5	±	17.5	107.5	±	17.5
Palaeodose (Gy)	134.4	±	6.1	145.4	±	6.3
In situ water content (%)	13.2	±	0.7	8.7	±	0.4
Saturation water content (%)	19	±	1.9	19	±	1.9
In situ fraction of saturation	0.69	±	0.08	0.45	±	0.05
NAA/DNA						
U (ppm)	0.79	±	0.142	0.76	±	0.137
Th (ppm)	6.55	±	0.131	5.67	±	0.113
K (%)	0.97	±	0.073	1.07	±	0.077
ICPMS						
U (ppm)	1.05	±	0.053	1.07	±	0.054
Th (ppm)	5.38	±	0.269	5.25	±	0.263
Flame photometry						
K (%)	0.9	±	0.027	0.97	±	0.029
XRF						
K (%)	0.874	±	0.035	0.959	±	0.038
NaI in situ gamma ray spectrometry (water corrected)						
U (ppm)	1.202	±	0.057	1.348	±	0.055
Th (ppm)	6.008	±	0.129	4.979	±	0.111
K (%)	0.905	±	0.011	0.898	±	0.022
Weighted means of isotope concentrations						
U (ppm)	1.098	±	0.037	1.174	±	0.037
Th (ppm)	6.181	±	0.087	5.313	±	0.076
K (%)	0.903	±	0.010	0.936	±	0.016
Cosmic ray dose-rate (Gy/ka)	0.182	±	0.027	0.162	±	0.024
Dose-rate components (Gy/ka)						
Internal alpha dose-rate	0.030	±	0.014	0.028	±	0.013
Internal beta dose-rate	0.008	±	0.001	0.008	±	0.001
External alpha dose-rate	0.024	±	0.013	0.023	±	0.013
External beta dose-rate	0.818	±	0.013	0.873	±	0.015
External gamma + cosmic ray dose-rate	0.738	±	0.028	0.722	±	0.025
Total dose-rate (Gy/ka)	1.62	±	0.04	1.65	±	0.04
Age (ka)	83.1	±	4.3	87.9	±	4.4

Table A1 Radioisotope and environmental data, along with the evaluated dose-rate components.

three samples were available for re-analysis in 2013. The samples had been retained in the ANU Luminescence Dating Laboratory archive, and, on closure of this facility in 2010, were transferred to the University of Adelaide 'Prescott Environmental Luminescence Laboratory', along with the remaining sample archive. The transferred material included not only sediment samples suitable for radioisotope, mineralogical and granulometric analysis, but critically also sediment splits from each of the three original OSL sampling core tubes, which had only received minimal pre-treatment at ANU (HCl acid wash and sieving) and then spent the intervening decade in light-safe storage, thus retaining the original OSL. Hence, appropriate quartz grains were available for extraction from these same samples to enable us to apply the single-grain optical dating technique in 2013, as a check on the 2002 results obtained at ANU.

The work performed at the ANU consisted of initially dating the three samples using the multiple-aliquot Australian slide method, as applied at the Cheshunt dune site and described above. Although the OSL behaviour of these samples appeared to indicate that the dates were of high reliability, the availability in 2002 of effective single-aliquot techniques and concerns by the AMBS team that there was potential for considerable bioturbation at the Warkworth site combined to lead to a re-dating of the two Pit 9 samples using 'small aliquots' of several grains each, with ED now measured using the Single Aliquot Regeneration (SAR) protocol (Murray and Wintle 2000). The results obtained were in very good accord with the ages measured using the Australian slide method. Details of the dating using the Australian slide method and the re-dating of Pit 9 using SAR, including the radioisotope, environmental data and dose-rates, are given in Hughes et al. (2003). In the paper above we reported the third dating of these samples in 2013, this time at the University of Adelaide, using single grain optical dating.

Sample Preparation

The three samples had previously undergone the initial steps of quartz grain extraction at ANU, consisting of an acid wash in 10% HCl to digest carbonates, followed by agitation in an ultrasonic bath in a NaOH solution as a dispersant to break up clay aggregates and remove most remaining traces of organic material, then sieving. The >212 µm fraction had been stored in sealed light-proof containers and archived. At the University of Adelaide we resumed preparation of these three samples using our current standard procedures. Grains in the range 212–250 µm were selected by dry-sieving, then density separation was applied first using lithium heteropolytungstate (2.67 gcm^{-3}) to separate out (sink) heavy minerals, secondly of density (2.62 gcm^{-3}) to separate out (float) lighter minerals such as feldspars. The grains of 2.62–2.67 gcm^{-3} were then etched for 40 minutes in 40% HF acid. Etching removes the outer 6–8 µm layer of the grains, thus removing surface discolorations such as oxide coatings and largely eliminating the effect of alpha particle contribution. It also is a final step for removal of occasional non-quartz grains (typically feldspar) that may remain after the density separation procedure. The etched quartz grains were then washed in warm 10% HCl to remove any precipitated fluorides, and finally re-sieved to obtain a well-defined 212–250 µm fraction.

OSL Measurements

OSL measurements were carried out using a Risø TL/OSL-DA-20 reader. Radiation was applied using a ^{90}Sr/^{90}Y β source, with dose calibration factors applied for each individual grain position. This was necessary since the configuration of the radioactive source is such that radiation is not equally distributed over the surface of the disc. OSL was stimulated by a 532 nm laser and detected using an EMI 9235QB photomultiplier optically filtered by one 7 mm thick UV-transmitting Hoya U 340 glass filter.

All measurements were carried out on single-grains of quartz measured using the SAR protocol. This approach has superseded multiple-grain methods as our preferred technique for sediment dating, due to greater reliability of ages measured from water-lain sediments, where a proportion of transported grains may not be sufficiently exposed to light to bleach completely and are therefore not fully 'reset', or sediments where mixing in of grains of differing exposure histories (optical ages) may have occurred.

Sample discs were prepared by placing one 212–250 µm grain into each of 100 300 µm diameter pits laid out in a 10 x 10 array on custom-manufactured 9.7 mm diameter aluminium discs. One disc for each sample was initially used in a pilot study to optimise the distribution of regenerative doses to be applied in the final measurement protocol, in which six discs totalling 600 grains were measured for sample 'Pit 9, 60 cm', and four discs totalling 400 grains each were measured for 'Pit 9, 125 cm' and 'Pit 7, 175 cm'.

In the SAR protocol a series of radiation doses (regeneration doses) is administered, interspersed with constant magnitude 'test doses' to enable correction for OSL sensitivity changes. These data are then used to construct a regenerative dose response curve, onto which the natural OSL is projected and its ED value found by interpolation. Regeneration dose values were selected according to the results of the pilot study. A zero dose to test for recuperation, and a repeat of the first dose to test for recycling, were also included. Grains having a 'recycling ratio' of 80% or better, recuperation less than 15%, and reasonably smooth regeneration and test dose response curves were accepted for ED analysis and subsequent inclusion in age evaluation.

The raw OSL data was analysed using the software program 'Analyst', provided by G. Duller, University of Aberystwyth, UK. Regenerative OSL data were corrected for sensitivity change using the corresponding test dose data and the results used to construct regenerative growth curves for each individual grain. An example of a typical SG regenerative growth curve for a grain that displays acceptable OSL properties is shown in Figure A2.

A proportion of grains in each sample were unsuitable for dating, and were rejected from inclusion in the age analysis through the application of several criteria, including: insufficient detected OSL to be statistically meaningful, poor recycling ratios, poor growth curves and excessive recuperation values. The summary of the grains chosen and those rejected is given in Table A2.

To assist visualisation of the results, the distributions of the measured EDs of the individual grains are presented in three ways (Figure A3).

All of the samples showed ED distributions consistent with an aeolian deposition history (they have rapidly-falling away high-ED tails, indicating the absence of partially bleached components characteristic of fluvial deposits), and a high dispersion consistent with post-depositional bioturbation.

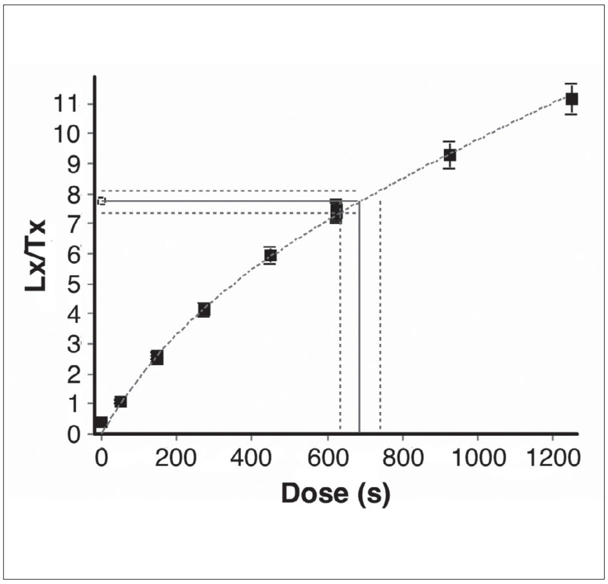

Figure A2 A regenerative growth curve for a single quartz grain, along with the natural OSL and uncertainty region projected onto the Dose axis. The 'Lx/Tx' axis represents the OSL values corrected by the corresponding test doses, and are plotted against the units of Dose in seconds as shown; these are later converted to absorbed radiation dose (Gy) by the calibration factor appropriate to that grain's position on the sample disc.

A representative central ED, termed ED_{Age}, to be used in the age calculation was determined for each sample from an analysis of the individual values. By inspection of the ranked distribution plots, and the probability density functions, outlying ED values which did not conform with the main distribution were removed during the calculation of ED_{Age}. These values indicate the grains may have been moved by bioturbation or incompletely bleached at burial; this involved

	Pit 9, 60 cm	Pit 9, 125 cm	Pit 7, 175 cm
Valid ED	42%	45%	35%
No luminescence	13%	14%	22%
Rejected grains	44%	35%	27%
Saturated	1%	3%	9%
Over-saturated		2%	5%
On linear portion of curve, >3xDo		1%	3%
Supralinear			1%
Total number measured	600	400	400
Number of valid EDs	254	180	141

Table A2 The number of grains measured and the fractions assigned to various categories based on the application of rejection criteria.

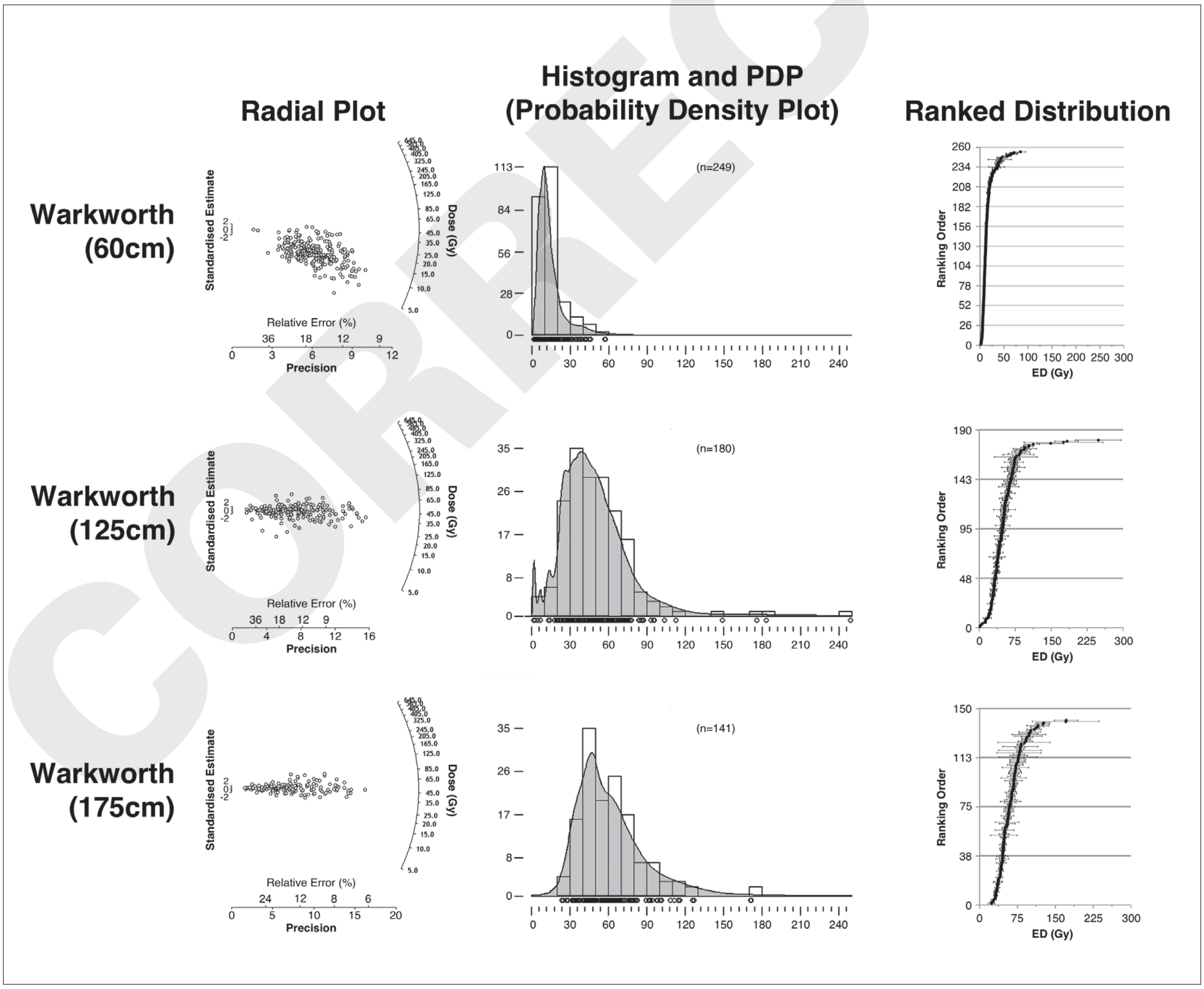

Figure A3 Distributions of the measured EDs of the individual grains: (left) radial plots (Vermeesch 2009) for each of the three samples. The curved y-axis shows ED (on a logarithmic scale) and the x-axis the precision of each measurement. The ED corresponding to an individual point is found by drawing a radius through the point from the origin to where it intersects the y-axis; (centre) histograms and probability density plots (PDP) for each sample; the ED values are weighted by their individual 'precision' proportional to their errors; and (right) ranked measurements with error bars.

exclusion of very few grains. The central value of the remaining EDs was calculated using the central age model of Galbraith and Green (1990) and Galbraith et al. (1999) to give ED_{Age} from which the sample age was calculated. The error terms shown are also given by the central age model, and indicate the uncertainty in the central (peak) value of each distribution rather than the spread in values. The values of ED_{Age}, determined as outlined above, are shown in Table A3. However, as the dispersion observed was significantly greater than is usual (55 % for Pit 9, 125 cm, and 33 % for Pit 7, 175 cm, compared to 10–20% for a well-bleached undisturbed sample), which is interpreted as indicating bioturbation, consistent with field evidence. We consequently also evaluated alternative ages for each sample using a minimum age model (all grains being included in these calculations except the four outliers); results are shown in Table A3.

Field Sample Name	Pit 9, 60 cm	Pit 9, 125 cm	Pit 7, 175 cm
Laboratory Code Number	ANU_{OD}1580	ANU_{OD}1582	ANU_{OD}1581
Total dose-rate (Gy/ka)	1.04±0.03	0.83±0.03	0.94±0.04
Equivalent Dose (Gy) MAM	5.3±0.5	19.0±3.0	37.3±3.0
Age (ka) by single-grains, MAM, 2013	5.1±0.5	22.9±3.7	39.7±3.6
Equivalent Dose (Gy) CAM	12.34±0.5	43.9±1.9	57.2±1.8
Age (ka) by Australian slide method, 2002	13.7±0.5	47.4±2.0	55.1±2.3
Age (ka) by single-grains, SAR, CAM, 2013	11.9±0.6	52.9±3.0	61.0±3.0

Table A3 The dose rates and chosen EDs for each sample, determined from applying the central age model and the minimum age model to the distributions of SG EDs. Also shown are the ages evaluated for each of the three samples.

Age Evaluation

The environmental dose rates were evaluated using the AGE program of Grün (1999, 2009) and, as previously, utilised the radioisotope concentrations, water content values and cosmic ray dose rates reported for each sample in Hughes et al. (2003), with appropriate correction made for beta particle attenuation for the larger grains of quartz used in the single grain dating reported here. The depositional ages of the samples were obtained by dividing their ED_{Age} by the environmental dose rate, and are shown in Table A3 for both the CAM and MAM.

Summary

The uppermost sample, Pit 9, 60 cm shows considerable scatter of the EDs, with minimum age analysis showing about 20% of the grains constitute a population of 5.1±0.5 ka age, and 69% of the grains give the major age peak centred at 11.9±0.6 ka. A small proportion of grains give a maximum age peak of about 30.2±3.4 ka, which is consistent with the observed evidence of bioturbation having acted to incorporate older grains from the underlying material.

The sample Pit 9, 125 cm has dispersion of 55%. Four grains give ages of 1–3 ka; as these represent 1% of the population they are regarded as outliers. Age calculation included all grains except the four outliers, and using the minimum possible gaussian peak gives 22.9±3.7 ka, which we consider to be the youngest realistic age for this sample. Analysis of the major peaks shows two peaks: one at 67.5±5.5 ka (64% of the grains) and a second at 38.6±5.0 ka (34% of the grains). The composite value for the distribution yields the CAM age of 52.9±3.0 shown in Table 6 in the body of the main paper.

The sample Pit 7, 175 cm has dispersion of 33%. This sample has no grains with ages younger than 27 ka, and a tail of older age grains extending to a poorly-defined peak at about 90 ka. Minimum age analysis shows the youngest peak has an age of 39.7±3.6 ka, and the composite value for the distribution yields the CAM age of 61.0±3.0 shown in Table A3. The single-grain distribution for this sample shows the skewed distribution has no young grains but a trail of a few older age grains suggesting bioturbation with grains incorporated from lower deposits followed by a period of barren biological activity and no bioturbation (i.e. buried deeply). The bioturbation of the upper sample subsequently did not appear to have penetrated deeply enough to affect this lower sample.

The central ages measured by the single-grain optical dating technique in re-dating these three samples in 2013 are little changed from the original dates obtained at ANU using the Australian slide method and then 'small aliquot' SAR. These age differences are minor and are attributed as principally due to the capability provided by the single-grain method to strip out the outlying populations of grains mixed into the samples by significant post-depositional bioturbation. The presence of bioturbation meant that the minimum age model was also applied, hence, whatever the extent of bioturbation, the true age of the lower two samples cannot be less than approximately 20,000 ya.