

PRECISION AND ACCURACY IN THE OPTICALLY STIMULATED LUMINESCENCE DATING OF SEDIMENTARY QUARTZ: A STATUS REVIEW

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Abstract: Optically stimulated luminescence (OSL) dating of light-exposed sediments is used increasingly as a mean of establishing a sediment deposition chronology in a wide variety of late Quaternary studies. There has been considerable technological development in the last few years – in instrumentation, in the preferred mineral, and in various measurement protocols. New approaches to the latter, especially with the introduction of the single-aliquot regenerative-dose (SAR) protocol, have given rise to an increasing number of ages in the literature based on the OSL signals from quartz. This paper examines the reliability of these results by reviewing both published and unpublished SAR quartz ages for which some independent age control exists. It first discusses studies of modern (zero age) sediments, and the implications of these results for the importance of incomplete bleaching, especially in water-lain sediments, i.e. sediments for which the initial light exposure is expected to have been insufficient to reduce the apparent dose at deposition to a negligible fraction of the final burial dose. It then compares OSL and independent ages derived from various types of sediments, including aeolian, fluvial/lacustrine, marine and glacio-fluvial/lacustrine. It is concluded that, in general, the ages are accurate, in that there is no evidence for systematic errors over an age range from the last century to at least 350 ka. Nevertheless, the published uncertainties of a small fraction of OSL ages are probably underestimated. We conclude that OSL dating of quartz is a reliable chronological tool; this conclusion is reflected in its growing popularity in Quaternary studies.

1. INTRODUCTION

Optically stimulated luminescence (OSL) can be used to estimate the time elapsed since buried sediment grains were last exposed to daylight. This method of sediment dating makes use of the fact that daylight releases charge from light-sensitive traps in the defects in crystals such as quartz and feldspar. The release of trapped charge by light resets the OSL signal; this process is commonly referred to as bleaching. When grains of quartz are buried and hidden from light, they begin to accumulate a trapped-charge population due to the effects of ionising radiation, such as that arising from radionuclides naturally present in the deposit. This trapped-charge population increases with burial time in a measurable and predictable way. As a result, the time elapsed since sediment grains were buried can be determined by measuring both the OSL signal and sensitivity from a sample of sediment, and by estimating the flux of ionising radiation to which it has been exposed since burial.

Until the late 1990s, the great majority of OSL dates were based on infrared (IR) stimulation of feldspars (Wintle, 1997; Aitken, 1998), but over the last few years both the preferred mineral and the laboratory techniques used to measure OSL have undergone major changes. It was always thought that quartz had considerable potential in optical dating, but it was little used because aliquot-to-aliquot reproducibility was poor, and because it was insensitive to IR. It requires visible light stimulation, which was usually derived from an expensive laser installation (e.g. Rhodes, 1988). Since then, a readily accessible measurement technology has become widely available, with the development of cheap green and blue light sources (these were at first filtered from the broad-band emission of an incandescent filament (Bøtter-Jensen and Duller, 1992); more recently they have been based on bright blue light emitting diodes (Bøtter-Jensen *et al.*, 2000). At the same time, measurement protocols have undergone major development. Until 1997, estimation of the equivalent dose (i.e. the dose absorbed during burial,

D_e) for quartz required the use of many aliquots (typically 50-100, each of about 10 mg). With the development for quartz of first the single-aliquot additive-dose protocol (Murray *et al.*, 1997) and more recently the single-aliquot regenerative-dose (SAR) protocol (Murray and Roberts, 1998; Murray and Wintle, 2000), all the measurements for quartz required to estimate the D_e can now be made on one aliquot. This has two particular advantages: (1) the uncertainties on the average D_e are now based on external estimates of precision – i.e. the standard uncertainty on the mean is estimated from several independent, interpolated measurements of D_e , rather than from the uncertainties associated with, for example, extrapolating a single modelled growth curve fitted to the data derived from many aliquots; and (2), for the first time, the distribution of doses within a sample can be examined explicitly (e.g. by making D_e measurements on single grains). These developments have resulted in a considerable increase in the number of published quartz dates, almost all of which are based on the SAR protocol. Age estimates based on quartz have been now produced by many laboratories over an age range of a few decades to more than 500 ka, and this method has already found considerable application in the dating of Quaternary sediments.

In this paper, we consider the evidence for the reliability of these SAR quartz OSL ages. The SAR protocol is first described and some internal checks on performance outlined. The process of bleaching or resetting the OSL signal is then discussed, along with the complications of interpreting the OSL signal when charge transfer is significant; the importance of these effects is considered by examining the reported apparent ages of modern sediments. We then review both published and unpublished quartz ages for a variety of sediment types and age ranges. In some cases these are known to be poorly bleached, but in all cases some age control exists; this allows the reliability of the OSL ages to be assessed.

2. THE SINGLE ALIQUOT REGENERATIVE DOSE (SAR) PROTOCOL

There are many published descriptions of the single aliquot regenerative dose (SAR) protocol and its applications (e.g. Murray and Olley 1999; Murray and Wintle, 2000; Bailey *et al.*, 2001; Stokes *et al.*, 2001) and it is not appropriate here to do more than outline its principle features, and to highlight the main assumptions. **Table 1** sets out a typical SAR measurement cycle. The sample is given a dose (D_0) during burial (i.e. before sampling). In the laboratory, the sample is first preheated to some temperature in the range of 160 to 300°C (usually for 10 s) and the natural OSL signal (L_0) measured. The sample is then given a test dose (D_1), heated to 160°C and the test dose OSL signal (T_0) is measured; this completes the first (natural) measurement cycle. To begin the second cycle, a regenerative dose (D_1) is first administered; the sample is then heated to the same preheat temperature as in the first cycle and the OSL signal measured (L_1). The sample is then given the same test dose as before (D_1), heated to 160°C and the test dose OSL signal (T_1)

measured; this regenerative cycle is then repeated as many times as desired, with the regenerative dose (D_i) the only variable. These regenerative doses are usually chosen to give values of the sensitivity corrected OSL ratio ($R_i = L_i/T_i$) which bracket the natural response ($N = L_0/T_0$). The test dose (D_i) is usually a small fraction (~10%) of the natural dose, although Murray and Wintle (2000) have shown that this is not a necessary requirement. The natural dose is then estimated by interpolating the ratio N onto a plot of R_i against D_i (**Fig. 1**).

For the protocol to be useful, any sensitivity changes that may occur from one measurement cycle to the next must be accurately measured by the OSL response to the test dose (D_i), i.e. $L_i \propto T_i$ for constant D_i . If this requirement is met, the corrected OSL ratio $R_i (= L_i/T_i)$ should be independent of treatment history, i.e. independent of prior dose or thermal treatment. This is most easily tested by repeating a particular value of D (e.g. $D_5 = D_1$) after

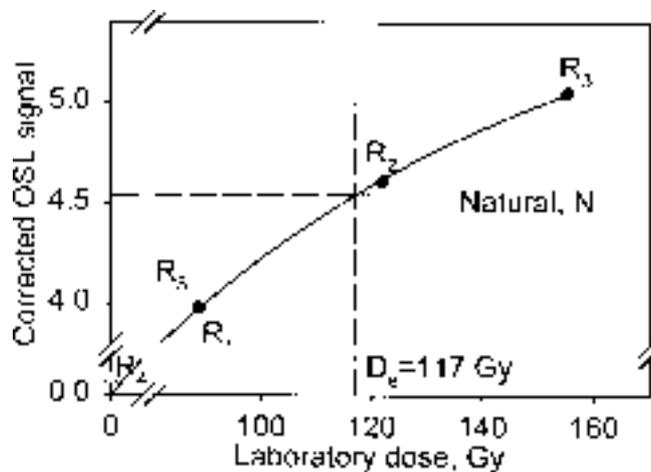


Fig. 1. Typical single-aliquot regenerative-dose growth curve, showing the natural OSL ratio $N (= L_0/T_0)$, see text and Table 1) interpolated onto the regenerated growth curve, for a single 8 mg aliquot of a glaciofluvial sample from northern Russia (laboratory code 992528). The recycled value (R_3 at the lowest regenerated dose is shown as an open circle ($R_3/R_1 = 1.02$ for this aliquot), and the recuperated signal for which $D_4 = 0$ Gy, as an open triangle ($R_4 = 1\%$ of the natural signal N).

Table 1. Generalised single-aliquot regenerative-dose protocol (after Murray and Wintle, 2000).

Step	Treatment	Observe ^d
1	Give dose ^a , D_i	-
2	Preheat ^b (160-300°C for 10 s)	-
3	Stimulate ^c for ~100 s at 125°C	L_i
4	Give test dose, D_t	-
5	Heat ^b to 160°C	-
6	Stimulate ^c for ~100 s at 125°C	T_i
7	Return to 1	

^aFor the natural sample, $i=0$ and D_0 is the natural dose.

^bAliquot cooled to <60°C after heating. In step 5, the TL signal from the test dose can be observed, but it is not made use of in routine applications.

^cThe stimulation time is dependent on the stimulation light intensity and wavelength (eg. 40 s for blue light diodes, 100 to 125 s for green-light sources).

^d L_i and T_i are derived from the stimulation curve, typically the first 1 to 10 seconds of initial OSL signal, minus a background estimated from the last part of the stimulation curve.

various larger values have been used, and comparing the two OSL ratios (in this case R_5 and R_1); for the same dose the two values of R should be the same ($R_5/R_1 = 1$, the so-called recycling ratio). Preheating the sample can also cause recuperation of the OSL signal (i.e. heating after OSL measurement can give rise to a new OSL signal, even in the absence of an ionising radiation dose). To test for this, a SAR cycle is measured with $D_i = 0$ Gy (usually $i = 4$). The ratio R_4 should then be zero, but in practice is usually some small % of N . Rejection criteria can be proposed based on these two test measurements (e.g. $0.9 < R_5/R_1 < 1.1$, and $R_4/N < 5\%$), but there is little evidence of how sensitive the value of D_e is to these criteria.

The performance of the SAR protocol with respect to laboratory doses can be checked by the two tests described above, but it is important to realise that there is no readily available test that the assumptions hold in the first measurement cycle, in particular that laboratory induced sensitivity variations in L_0 are faithfully reflected in variations in T_0 . Murray and Wintle (2000; their Figure 3) did test this assumption using the response of the 110°C TL peak before and after OSL measurement of the natural signal, but this approach requires that any sensitivity changes in the measurement of the OSL signal are reflected in changes in the 110°C TL peak; this cannot be assumed, and must be tested from one sample to another.

For feldspar, Wallinga *et al.* (2001) have suggested the routine use of a dose recovery test, in which a sample is first optically zeroed without any heating above room temperature, and then given a known dose using a laboratory source. This dose is measured as if it were an unknown natural dose (i.e. beginning with a preheat). The important point here is that the first heating of the sample occurs after the administering of the dose to be measured; at least for feldspar, there is clear evidence that the first heating of a sample changes its luminescence sensitivity, probably by changing the rate of charge trapping. Of course, any useful measurement protocol should be able to measure this (known) laboratory dose accurately. While this does not necessarily imply that a natural dose can also be measured accurately, it does serve to increase confidence in the measurement procedure. This approach should probably be made more use of with quartz.

3. RESETTING THE OSL SIGNAL – BLEACHING

If a clean quartz grain is exposed to daylight, the so-called “fast component” of the OSL signal will be reduced to a negligible level within a few seconds. However, in nature this is rarely the case. Quartz sand grains are rarely “clean”; they usually have iron and manganese oxide surface coatings, and they may have adhering clay grains. The transport medium is rarely transparent; even during aeolian transport (usually considered to be the most ideal bleaching circumstance) other grains can attenuate the light. Nevertheless, even in aqueous transport, there may be circumstances where bleaching is complete; sand repeatedly washed up and down a lake or marine beach will receive considerable exposure to light, and sand depos-

ited by a high energy event (e.g. a storm or tsunami) is likely to have been derived from shallow-water deposits that had already been thoroughly light exposed. In many freshwater environments, however, turbidity may be the controlling factor; in some low gradient rivers at low latitudes most of the incident light is absorbed in the first 1 m of water or less (Oliver, 1990).

Predicting the rate of bleaching is complicated further by the nature of a transport process. Turbulent mixing and saltation combine to move even coarse sand-sized grains into and out of the regions of the transport medium which receive the highest light fluxes. If the flux gradient is steep (as in a turbid river) this will have the effect of exposing the grain to light for a short period, before returning it to effectively complete darkness. It is even possible that the majority of bleaching in turbid river systems occurs only during low flow periods when the water is clear and shallow, and sand transport has ceased. Then only those sand grains resting on the surface of the bed will get sufficient light exposure to fully bleach the OSL signal. As a result of these various processes, the bleaching history of every grain will be different, and only when all grains have had more than sufficient light exposure will a sediment sample be completely bleached. This is usually assumed to be true for aeolian transported material, but it is at least questionable for sediments transported in other ways.

Incomplete bleaching results in grains being deposited with a heterogeneous distribution of residual trapped charge. If large sample aliquots (i.e. aliquots made up of many grains) are used for the measurement of the OSL signal, these residual trapped charges give rise to an apparent dose that results both from charge trapped during

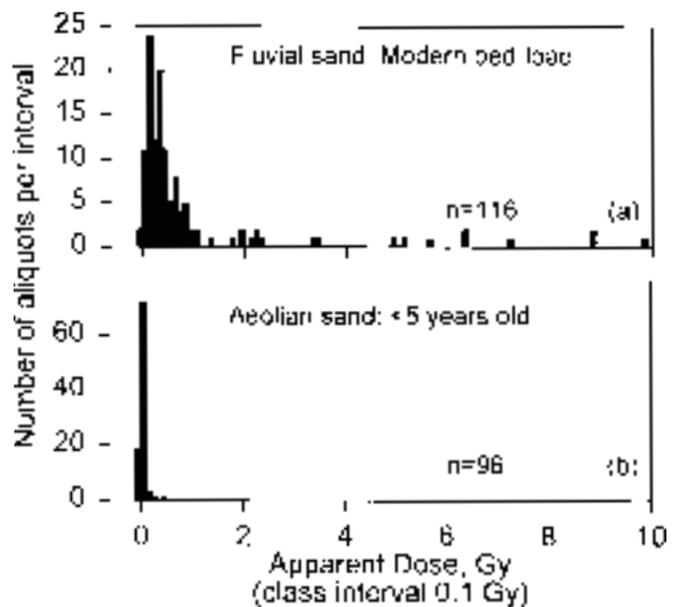


Fig. 2. (a) Apparent doses from 116 small aliquots of 125–180 μm diameter grains of quartz (laboratory code ME95002/2) extracted from a recent sand deposit on the Murrumbidgee River, New South Wales, Australia. Each aliquot contained between 60 and 100 grains. (b) Apparent doses in 96 aliquots from a modern aeolian dune sand (125–180 μm diameter), known to be less than 5 years old, from the east Queensland coast, Australia (DS1/1; both figures taken from Olley *et al.*, 1998).

burial and the residual trapped charge already present at the time of burial. The net effect is an overestimate of the age. However, if small aliquots are used, made up of only a few grains, or even single grains, a distribution of apparent doses will be measured, skewed to larger values. In such cases, the lower values are more likely to give the true age since burial. **Figure 2** illustrates this comparison using two modern sediments, one aeolian and one river sand (Olley *et al.*, 1998), both measured using aliquots of about 100 grains. Effectively all the aliquots from the aeolian sand are well bleached, but only about 5% of those taken from the river sand are consistent with zero dose.

It is possible that during some depositional events none of the sediment grains would receive sufficient exposure to fully bleach the OSL signal. Especially in very young deposits (<100 years) with evidence of heterogeneous bleaching at deposition, it may be that OSL ages should be viewed as maximum ages of burial.

4. PREHEATING AND CHARGE TRANSFER

Some heating of the aliquots is always applied before measurement of any OSL signal. In the SAR protocol described by Murray and Roberts (1998) and improved by Murray and Wintle (2000), the OSL signal is measured while the aliquot is held at 125° C. This is intended to minimise charge cycling through the 110° C TL trap, and so increase the rate of decay of the OSL signal, and this procedure has been adopted widely. Additional heating prior to OSL measurement is also common. There is a tendency for younger samples to be measured after heating to lower temperatures (typically 200 to 220° C for 10 s; Murray *et al.*, 1995; Olley *et al.*, 1998; Hilgers *et al.*, 2001; Murray and Clemmensen, 2001; Banerjee *et al.*, 2001; Wallinga *et al.*, 2001; Stokes *et al.*, 2001) than are used for older samples. The latter are more usually heated to 260 or 280° C for 10 s prior to measurement (Strickertsson and Murray, 1999; Strickertsson *et al.*, 2000; Roberts *et al.*, 1998; 1999; 2001; Wallinga *et al.*, 2001), although there are several examples of ‘preheat plateaus’ which show that the apparent dose is insensitive to the temperature of heating prior to measurement, at least in the range 160 to 300° C (Murray and Olley, 1999; Murray and Wintle, 2000; Roberts *et al.*, 1999).

There are good reasons why younger samples should be measured with lower preheat temperatures. Thermal transfer, or the transfer of charge by heating from light-insensitive but thermally-stable traps into the light-sensitive traps, is probably not important in older samples; this is shown by the lack of sensitivity of D_e to preheat temperature mentioned above. This appears not always to be true for younger samples. Various authors have mentioned or presented preheat plateaus for young samples which increase with temperature (Murray and Clemmensen, 2001; Bailey *et al.*, 2001; Rhodes, 2000; Wallinga *et al.*, 2001; Hilgers *et al.*, unpublished), and Banerjee (2000) has also discussed this issue. It is likely that this is common in samples which have received enough light exposure to empty the OSL trap, but insufficient to empty the

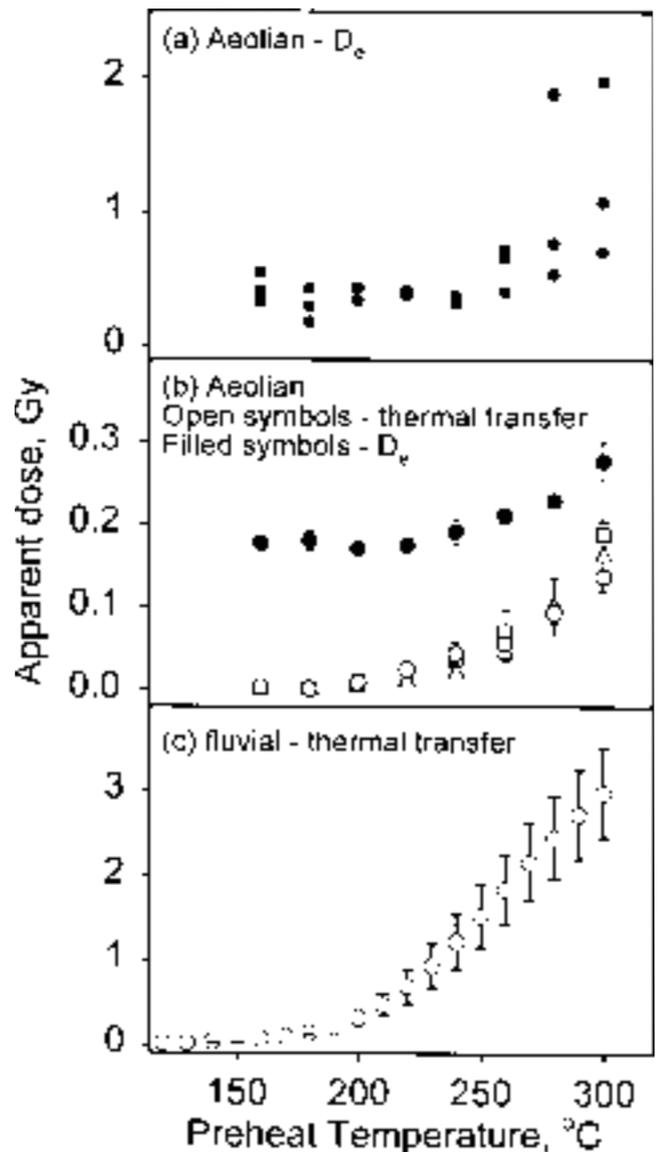


Fig. 3. (a) D_e as a function of preheat temperature for an aeolian sand sample (age 310 ± 90 years) from the coast of Wales (redrawn from Figure 3 of Bailey *et al.*, 2001).

(b) Filled circles: preheat plateau for quartz sample F1 of the dune section ‘Postdüne’, located north of Berlin in Brandenburg, Germany (age: 140 ± 10 years). Aliquots were measured in groups of 3 using 8 different preheat temperatures from 160° up to 300° C (held for 10 s each; the cycle was repeated, 48 aliquots were measured in all). Six regeneration cycles were measured using the following doses for beta-irradiation 0, 0.38, 0.63, 0.88, 0, 0.38 Gy (test dose: 0.25 Gy), blue light emitting diodes for optical stimulation (100 s at 125° C), and a cut heat of 160° C. Each data point shows the average and standard uncertainty of 6 aliquots, except for 240° C ($n=30$) and 260° C ($n=24$).

Open symbols: Data were obtained for 24 aliquots processed as described above, but all aliquots were first stimulated for 150 s with blue diodes at 125° C to remove the natural OSL signal. The test doses applied were 25 % of the D_e (0.05 Gy, open square), 160 % (0.31 Gy, open circle) and 1600 % (3.1 Gy, open triangle). All taken from Hilgers *et al.* (unpublished).

(c) Apparent values of D_e resulting from repeated heating of a single aliquot of 300 year old fluvial bedload from a channel of the Rhine-Meuse system in the Netherlands (see Wallinga *et al.*, 2001). The results were checked for the absence of sensitivity change.

less light-sensitive and thermally-shallow traps, such as the TL traps at 160, 240 and 280° C. These traps can be expected to retain a significant charge population at the time of deposition, even although the OSL trap was well bleached (in fact, these TL traps may even contain an equilibrium trapped-charge population, where rate of trapping equals rate of thermal release at ambient temperature). If this sample is then heated to a temperature sufficient to release some or all of this charge in shallow traps, a fraction of the released charge can be retrapped by the OSL trap. This gives then rise to a finite OSL signal from a sample that was, in fact, well bleached at deposition. This phenomenon will become increasingly less important as the OSL trap stores charge from the ambient radiation flux after burial, because the retrapping process seems to be inefficient. This effect is illustrated in **Fig. 3b,c** (open symbols). Here the aliquots were all first bleached using blue light (at 125° C in **Fig. 3b**; at room temperature in **Fig. 3c**), but without prior heating. This emptied the OSL trap but left any shallow traps (except the TL trap at 110° C) relatively untouched. The aliquots were then subject to the usual SAR protocol, using the various preheat temperatures shown. At low temperatures, the apparent D_e is very small, but at higher temperatures there is significant thermal transfer during the first preheat into the empty OSL trap, and the apparent D_e (filled symbols) begins to rise. In both the young aeolian sample (**Fig. 3b**, open symbols; Hilgers *et al.*, unpublished data) and in the young fluvial sample (**Fig. 3c**; Wallinga *et al.*, unpublished data) the phenomenon is detectable at temperatures as low as 200° C, and rapidly becomes significant at temperatures above this. The effect of this transfer on the variation of D_e with preheat temperature is clearly seen in the preheat plateau for the two aeolian samples (**Fig. 3a** – Bailey *et al.*, 2001 – and **Fig. 3b**, filled circles). It is clearly desirable to use low temperature preheating for young samples, especially those that are likely to be less well bleached. It is less clear that there is a compelling reason to use higher temperature preheating for older samples; as discussed above, all published preheat plateaus for older samples appear relatively flat.

5. MODERN SEDIMENTS

One way to test the hypothesis that samples are well bleached at the time of deposition is to examine modern (i.e. zero age) analogues. This is increasingly standard practice in dating studies, and although such analogues are not always available at or close to the sampling site of interest (especially for deposition environments associated with continental glaciation events) such studies can be very informative. The following sections summarise what is known about various classes of modern sediments using the SAR protocol.

Aeolian – Olley *et al.* (1998) calculated the average dose in an Australian aeolian sediment believed to be <5 years old (using the data of **Fig. 2a**). They derived an average D_e of 0.020 ± 0.006 Gy, using a preheat of 200° C for 10 s; this corresponds to an age of about 20 years, using

typical dose rates (note that throughout the symbol ‘ \pm ’ is followed by the standard uncertainty on the mean, and that the number of significant digits is given as presented by the original author). Bailey *et al.* (2001) took a sample from the top of a parabolic dune, currently subject to wind erosion, on the coast of north Wales, and obtained an age of 20 ± 10 years, averaged over the preheat range 160 to 260° C (heated for 10 s). Murray and Clemmensen (2001) sampled wind-blown sand deposited on concrete on the west coast of Denmark, and known to have been deposited in the last 35 years; they obtained an age of 36 ± 5 years, using a preheat of 200° C for 10 s. One author (Murray) has other unpublished examples of ages ~10 years from modern aeolian sediments from this geographical area.

Fluvial – Murray *et al.* (1995) used a simplified regeneration protocol to examine the residual doses in 4 modern fluvial sediments, covering catchment scales from a few hectares up to >100,000 km². In all these catchments, flood flows were known to be very turbid. In their approach only a single regeneration cycle was used, with no test dose. Their results are considered here, because the OSL response to dose (the growth curve) at low doses is known to be linear, and because the sensitivity change at such low doses will be negligible if the preheat temperature is kept low (they used 200° C for 10 s). They observed average doses of between 0.3 Gy and 3 Gy, corresponding to approximate ages of a few hundred up to a thousand years. The lowest apparent age was derived from the only overbank deposit; the other three were samples of in-channel deposits. They measured the OSL signals using small aliquots of only about 150 fine-sand sized grains; this sample size was chosen to deliberately increase the scatter in the data, and so increase the probability that they would observe aliquots containing only well-bleached grains. In 3 out of the 4 distributions they observed some aliquots with doses consistent with zero, and in the fourth sample, the smallest doses were consistent with an age of about 60 years. This supports the hypothesis that even in turbid systems there are likely to be some grains that are well-bleached. Olley *et al.* (1998) sampled a modern in-channel sand deposit from the Murrumbidgee River in eastern Australia, and used a 200° C preheat for 10 s. They measured an average apparent age of 340 ± 70 years (calculated using their average D_e and dose rate values). Further measurements, using small aliquots of 80 to 100 grains each (see **Fig. 2a**), demonstrated that not all grains in this deposit were fully bleached; if they used only the lowest 5% of aliquots, they derived an age that was consistent with zero (3 ± 4 years). These authors also examined the grain size dependence of the dose distributions in sediments from the same river, and found the rather surprising result that the fraction of well-bleached grains appeared to increase as the grain size increased (in the range <63 μ m to 250 μ m).

Colls *et al.* (2001) sampled various deposits on the River Loire in central France. They used a relatively high temperature preheat (280° C for 10 s), which almost certainly resulted in some thermal transfer, and probably contributed to the finite age of 300 ± 60 years they

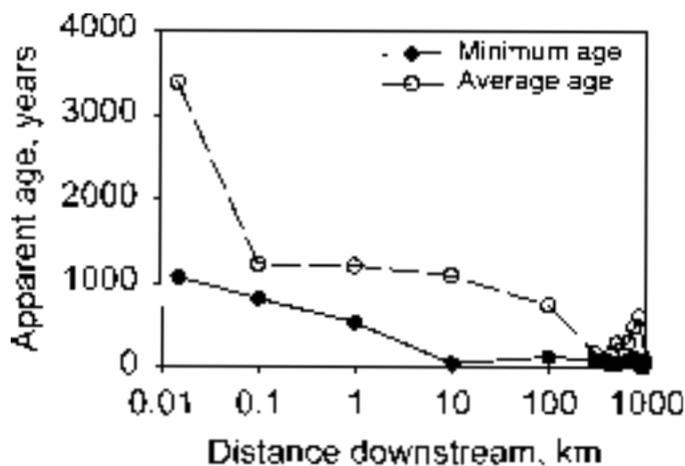


Fig. 4. Apparent D_e for bedload samples collected within the channel of the Loire River in France (after Stokes *et al.*, 2001).

obtained for a modern point-bar deposit. This study used average estimates of D_e – no attempt was made to correct for incomplete bleaching, e.g. by measuring distributions of dose in small aliquots. Stokes *et al.* (2001) conducted a more systematic study of modern sediments in two river basins, the Colorado (USA) and the Loire (France). In the Colorado system they collected 4 samples from the Columbus Point Bar in Texas, and in the Loire they collected 13 samples along the length of the channel, from source to mouth. The Colorado study used the full SAR protocol, whereas that in the Loire used the simplified protocol of Murray *et al.* (1995). In the Colorado study they observed average ages between 95 and 648 years. Their data also suggested that the apparently older samples had not been completely bleached, but their at-

tempt to correct for this only reduced the range to between 70 and 290 years. However, all their measurements involved relatively large aliquots (>1000 grains) and so some averaging was probably unavoidable. In the Loire study, the average ages decreased with distance from 3.3 ka near the source, to about 80 years at the mouth (Fig. 4; open circles). If some allowance was made for incomplete bleaching (filled circles), this pattern changed to about 1 ka at the source, decreasing to only a few decades within the first 10 km, and staying low thereafter.

Marine – In a study of a possible tsunami deposit on the Scilly Isles (UK), Banerjee *et al.* (2001) sampled modern sub-aqueous coastal sand, from a few centimetres below the sediment surface and more than 2 m below water level at low astronomical tide. Using a preheat of 200° C for 10 s, they calculated an average OSL age for the 180-210 μ m fraction of 2.0 ± 1.7 years.

6. HOLOCENE AND LATE PLEISTOCENE SEDIMENTS

The effects of incomplete bleaching are most likely to be obvious in younger samples (e.g. those from the Holocene) and so there have been many studies of aeolian material from this period, but water-lain deposits tend to have been avoided. Nevertheless there is an increasing awareness that incomplete bleaching in fluvial and marine systems may not be as large a problem as previously feared, and this has led to an increasing interest in such sediments.

Aeolian – Strickertsson and Murray (1999) sampled a sand of historically known (300-400 years) age from the west coast of Jutland, Denmark, and obtained an OSL age of 290 ± 20 years, using preheats between 240 and 280° C for 10 s. Bailey *et al.* (2001) dated a small dune field on

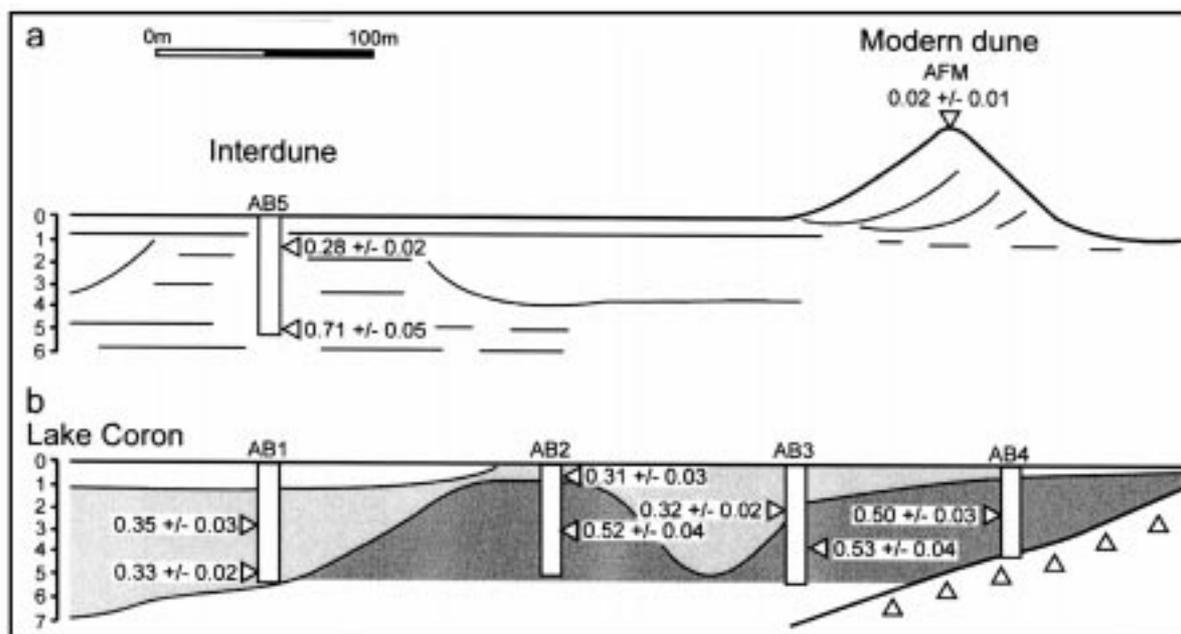


Fig. 5. Schematic cross-sections of (a) the interdune area and modern parabolic dune and (b) aeolian sand stratigraphy at Lake Coron, on the coast of Wales, UK, derived from ground penetrating radar images. The OSL ages (ka) and sample locations are also shown (modified by Bailey [pers. comm.] from Bailey *et al.*, 2001).

the west coast of north Wales (Fig. 5; note that these ages have been revised slightly by Bailey, pers. comm., from those originally published). They obtained an age for the oldest phase of sand movement of 710 ± 50 years, averaged over the preheat range 160 to 260° C (heated for 10 s). This is consistent with the historical evidence that this dune field began to accumulate in AD 1331. They also used ground-penetrating radar to identify two deposition units, which were then sampled from 4 separate boreholes. This provided 4 samples from the younger unit, of average age 328 years (sample standard deviation $\sigma=15$), and 3 for the older, of average age 517 years ($\sigma=12$). They assumed that the samples were saturated throughout the burial period, and do not provide an uncertainty for their water content values or for other corrections introduced to compensate for systematic errors. However, these are presumably not trivial, because their overall combined standard uncertainty on an individual age varies between 20 and 40 years, significantly in excess of their observed reproducibility.

Murray and Clemmensen (2001) describe a sequence of ^{14}C dated palaeosols interleaved with aeolian sand units from a site on the west coast of Jutland in Denmark (Fig. 6). Their values of D_e were independent of preheat temperature (in the range 160 to 280° C, held for 10 s), and the agreement with ^{14}C over the age range 4500 years to about 100 years was very satisfactory. Their youngest results (between 70 and 180 years) were known with considerably better calculated uncertainties (between 9 and 40 years) than the immediately underlying calibrated ^{14}C ages (± 140 years); this illustrates the importance of uncertainties arising from the shape of the ^{14}C calibration curve in the last few centuries. (Note that all ^{14}C dates discussed in this paper are calibrated by the original authors unless otherwise stated.)

Radtke *et al.* (2001) undertook a comparison of quartz and feldspar, single- and multiple-aliquot dating of dune sands immediately above and below the Laacher-See tephra layer at a site near the Rhine, close to Mainz, Germany. Using the SAR protocol, and a preheat of 260° C

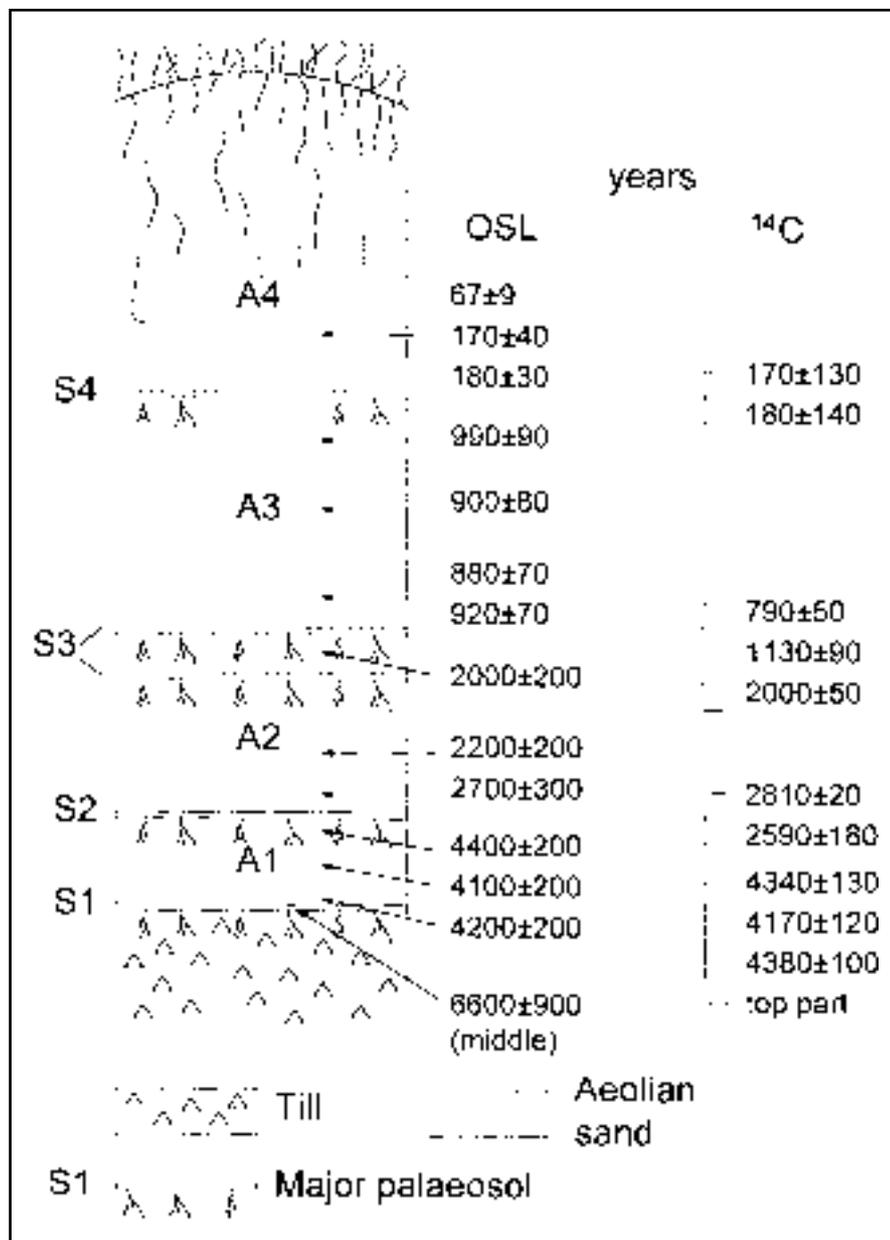


Fig. 6. Diagrammatic section of the Lodbjerg site in western Jutland, Denmark (based on Murray and Clemmensen, 2001), showing relationship between OSL and calibrated ^{14}C dates. S3 is a complex and less well-defined palaeosol. The midpoints of the calibrated ^{14}C ages are shown, with uncertainties of half the range; the ages include an additional 40 years to convert to calendar years.

for 10 s, they obtained quartz ages of 14.6 ± 1.5 ka below the tephra, 11.9 ± 1.0 ka in the tephra layer, and 13.4 ± 1.2 ka above the tephra. All these ages are consistent with the well-known age for the Laacher-See tephra of 13.155 ka, based on AMS ^{14}C measurements of decadal samples of poplar buried by the tephra (Friedrich *et al.*, 1999). Hilgers *et al.* (2001) undertook a related study near Eberswalde, north of Berlin, Germany, at a dune sand site that contains both the Laacher-See tephra and a well-described palaeosol, dated by Schlaak (1993) to the Allerød interstadial (12.9–14 ka). Their study also included several ^{14}C ages from younger layers, and with one exception the relevant OSL ages were in good agreement. However, the relative stratigraphy of the earlier ^{14}C samples compared to the OSL samples in these younger layers was not unambiguous, and so these comparisons are not considered here. They used a preheat of 260°C for 10 s; for the sample immediately overlying the Laacher-See tephra, they obtained an age of 11.2 ± 1.3 ka, and for the samples encompassing the palaeosol, they obtained 11.9 ± 1.1 ka (above) and 15.1 ± 1.4 ka (below). All three of these dates are in excellent agreement with their respective age controls.

Rich and Stokes (2001) dated a variety of aeolian samples from the southern High Plains (between New Mexico and Texas, USA). The sites were of archaeological significance, and many had independent ^{14}C age estimates already available. The reliability of association between these independent ages and the OSL samples is unclear, especially since some of the ^{14}C dates were undertaken in the 1950s. Nevertheless for 11 samples for which a comparison with SAR data can be drawn, the mean ratio of SAR to independent age is 1.12 ± 0.17 . These samples cover an age range from a few hundred years to about 11 ka.

Mangerud *et al.* (2000) report two comparisons of aeolian OSL dates with ^{14}C from two sites in north western Russia. One is at the Pymva Shor archaeological site, where a reindeer bone (uncalibrated ^{14}C age given as 10.27 ± 0.08 ka; after calibration 12.0 ± 0.3 ka) was found

in an aeolian sand unit which provided two OSL dates of 12.0 ± 0.9 and 13.6 ± 1.2 ka. The other is at their Kuya Bridge site, where they give an uncalibrated ^{14}C age of 12.3 ± 0.06 ka (on organic material extracted from bulk sediment sample); after calibration this gives 14.3 ± 0.5 ka. Despite the problems often associated with such samples, this agrees very well with the OSL age from the same aeolian sand unit, of 14.6 ± 1.2 ka.

Freshwater sediments – Olley *et al.* (1998) measured the apparent age of a flood deposit known to have been deposited in 1926. The average apparent age determined from large aliquots (~ 2000 grains) was between 400 and 700 years (preheat 200°C for 10 s), but by using small aliquots (60 to 100 grains), and comparing with the distribution of a modern analogue (see Fig. 2a) they derived an age of 67 ± 5 years, in good agreement with the known age. Olley *et al.* (1999) later examined the dose distribution in single grains from this sample and from a sample with a calibrated ^{14}C age of 1530 years (before 1954) and a 95% confidence age range of 1400–1690 years. Measured doses for single grains from each sample are shown in the form of a radial plots in Fig. 7a, b. The shaded region on each plot represents the expected burial dose (0.27 ± 0.02 Gy and 5.4 ± 0.6 Gy respectively). The dose measured for a grain can be read by extrapolating a line from the y-axis origin through the data point until the line intersects the radial axis (log scale) on the right-hand side. This gives the dose estimate in Gy, and its standard uncertainty can be read by extending a line vertically from the data point to intersect the x-axis. The x-axis has two scales: one plots the relative standard uncertainty of the dose (called relative error by Olley *et al.*, 1999; see Fig. 7) and the other (precision) plots the reciprocal standard uncertainty of the log dose estimate. Therefore, values with the highest precisions and the smallest relative uncertainties plot closest to the radial axis on the right of the diagram, and the least precise estimates plot furthest to the left (note that the calculated uncertainties are usually dominated by counting statistics in this type of study). The y-axis provides a further aid to data display, by plotting standardised

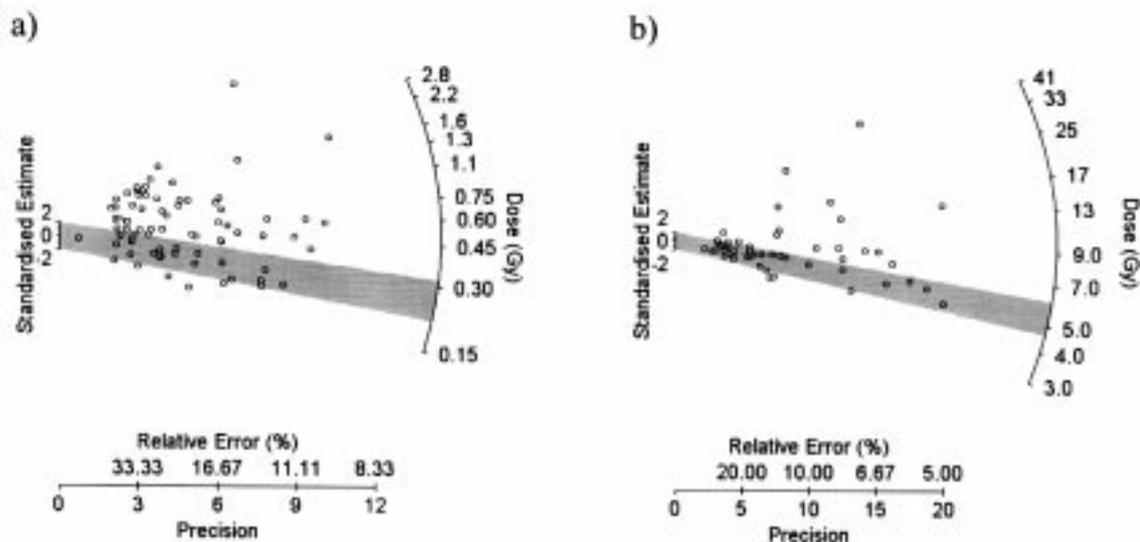


Fig. 7. Radial plots of measured doses for single grains from (a) sample ME95041B and (b) sample WK96008. In each plot, the shaded region represents the expected burial dose (after Olley *et al.*, 1999).

estimates of log dose. These are calculated by subtracting a reference value (such as the pooled log dose for all aliquots, or another log dose of interest) from each of the log doses and then dividing each of the differences by the associated standard uncertainty. Galbraith *et al.* (1999) provide further details, and a worked illustration, of how radial plots may be used to display OSL data. Olley *et al.* (1999) concluded on the basis of these data that the populations of single grains with the lowest doses gave ages consistent with the expected burial ages.

Olley and Hancock (unpublished data) have compared ^{210}Pb and OSL ages on two freshwater sediment cores from south-eastern Australia. In each case the samples were poorly bleached and the OSL ages were based on the lowest doses observed in single grains of quartz, measured with a preheat temperature of 220°C . In the first example, from the Bega estuary in New South Wales, the OSL age of 70 ± 8 years at 25 cm compared well with the ^{210}Pb age of 63 ± 8 years at 24.5 cm. In the second example, from Blue Lake, one of the highest lakes in Australia, the ^{210}Pb age of 128 ± 10 years at 52 cm is consistent with the OSL age of 170 ± 20 years at 60–62 cm if a constant rate of sediment supply is assumed. Olley and Taylor (unpublished data) have also produced a similar comparison between the first appearance of the fallout nuclide ^{137}Cs and an OSL age on lake sediments from the Arapiles Lake Complex, Wimmera, Australia. The first appearance of ^{137}Cs in a sediment core from this region occurs at about AD 1958 (i.e. 44 years ago). The OSL age of the sediments from this horizon gave an age of 29 ± 15 years. Again the sample was poorly bleached and the OSL age was based on the lowest measure doses in single aliquots of quartz each consisting of ~ 10 grains (preheat of 220°C for 10 s).

Wallinga *et al.* (2001) have dated a sequence of abandoned river channels from the Rhine-Meuse system in the Netherlands. These channels had already been extensively studied, and the ages of the younger channels are considered to be well known. **Fig. 8** summarises the comparison of OSL ages (preheat 200°C for 10 s using average values of D_e derived from small aliquots) with the independent chronology; the agreement is excellent except for that from the youngest sample, known from historical sources to have been deposited about 300 years ago. The corresponding OSL age is 920 ± 100 years, and the difference presumably arises from incomplete bleaching, since the

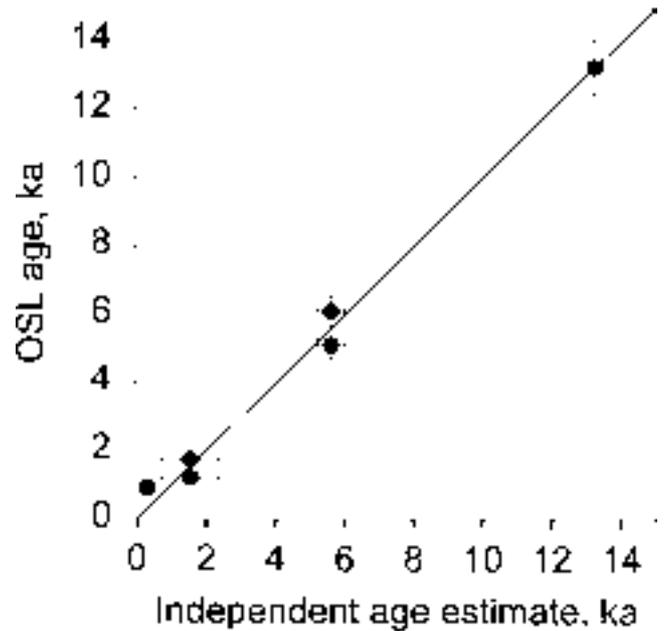


Fig. 8. OSL ages plotted against independent age estimates for fluvial channel deposits in the Rhine-Meuse system in the Netherlands (after Wallinga *et al.*, 2001).

thermal transfer characteristics of these samples had been explicitly studied (those of the youngest sample are shown in **Fig. 3c**).

Houmark-Nielsen (unpublished data) has two comparisons of OSL and ^{14}C ages from Late-Glacial lacustrine deposits in Denmark (**Table 2**, lines 4 and 5). The OSL ages both used 260°C preheats for 10 s, and the agreement with the independent age is very satisfactory. Larsen *et al.* (1999) report one comparison at their Chelmokhta site from northern Russia, where five ^{14}C ages (on wood) of between 10.7 and 11.5 ka can be compared with an OSL age on lacustrine sand of 13.7 ± 1.1 ka.

Strickertsson and Murray (1999) took samples from freshwater Bølling-Allerød (14.1–14.5 ka) and Younger Dryas (11.1–12.9) sediments, from Nørre Lyngby on the west coast of Jutland in Denmark (the independent age controls are discussed and referenced in Strickertsson and Murray, 1999). They used preheats of between 240 and 280°C for 10 s, and obtained SAR ages of 13.6 ± 1.1 ka (Bølling-Allerød) and 9.8 ± 0.7 and 8.2 ± 0.6 (Younger Dryas). The latter pair in particular appears to slightly

Table 2. Summary of comparisons between OSL and calibrated ^{14}C ages from various fluvial and lacustrine sites in Denmark (Houmark-Nielsen, unpublished).

Locality	Sample	OSL Age [ka]	^{14}C Age [ka]	Organic material	OSL/ ^{14}C ratio
Lønstrup	970203	30 ± 2	33 ± 2	plant	0.91 ± 0.08
	970204	29 ± 2	32 ± 3	moss	0.91 ± 0.11
Lodbjerg	980203	30 ± 3	33 ± 1	plant	0.91 ± 0.10
	980201	13.6 ± 1.0	13.9 ± 0.5	plant	0.98 ± 0.08
Bovbjerg	990205	13.8 ± 0.9	14.0 ± 0.5	plant	0.99 ± 0.06
Møn, Kobbøl Gård	990219	26 ± 2	32 ± 2	gyttja	0.81 ± 0.08
	990220	29 ± 2	30 ± 2	seed	0.97 ± 0.09
	000230	28 ± 2	27 ± 1.0	teeth	1.04 ± 0.08
Average:					0.94 ± 0.03

underestimate the independently known age. We do not consider their Podzol Bh-horizon sample, because the authors state that this soil was formed on Younger Dryas sediments, but must have received considerable light exposure at later times, up until the soil was capped by the 300 year old aeolian event discussed earlier.

Marine – Stokes *et al.* (2001) have reported a set of 5 OSL ages obtained using the silt-sized quartz fraction from a deep sea core from the Indian Ocean, for which a well controlled age model already existed. There was no dependence of D_e on preheat temperature, in the range 200 to 280° C, and preheat temperatures of 200 and 240° C were employed in routine analysis. Fig. 9 includes their 2 OSL ages younger than 20 ka; the agreement with

the independent age control is excellent. Strickertsson and Murray (1999) also took marine samples from their Nørre Lyngby section. These were from the Older and Younger Yoldia Clay, with independent ages based on calibrated ^{14}C dating of *in-situ* shell of about 24 ka, and 17.2 ± 0.4 ka. Their SAR OSL ages were independent of preheat temperatures in the range 240–280° C (for 10 s), and the one OSL age (25.3 ± 1.8 ka) from the Older Yoldia Clay was in good agreement with the expected value of about 24 ka. However, there was a possible systematic underestimation of the expected ages for the Younger Yoldia Clay, by about 8% averaged over 4 results. Strickertsson *et al.* (2001) report further ages on the Younger Yoldia clay, from Stensnæs, about 2 km from the earlier site. These two ages (21.1 ± 0.9 and 26.2 ± 1.4 ka) were significant overestimates compared to the expected age range, and the authors suggest that the deposits may have been misidentified in the field (the Older Yoldia Clay [24 ka] is very similar to the Younger). The sand layer above the supposed Younger Yoldia Clay was believed to be Upper Saxicava in the field, with an expected age of about 12.5 ka. In fact an OSL age of 17.5 ± 1.3 ka was obtained, also casting doubt on the field identification.

7. OLDER SEDIMENTS

Aeolian – Watanuki *et al.* (submitted) have examined the silt-sized (4–11 μm) quartz fraction in loess, deposited on two river terrace sites (Niigata and Tochigi) in central Japan, using 240 and 260° C preheats. This material was blown across from the Chinese mainland, but it is unusual in that there is age control based on tephra layers interleaved with the loess; these various tephra layers have been dated, using ^{14}C and fission tracks, and provide an independent chronology extending over the age range 30 to 660 ka. Their data are summarised in Fig. 10. Despite a very large correction to dose rates arising from a modern water content of around 100%, the agreement with the independent evidence is good. The unusually large age range for such fine-grained sediments can be, at least in part, attributed to the low dose rate, of about $1 \text{ Gy} \cdot \text{ka}^{-1}$.

In a study of an early human occupation site in south western Australia, Turney *et al.* (2001) report a comparison of a number of charcoal ^{14}C ages with 5 OSL quartz ages based on small aliquots (~eighty 100 μm grains per aliquot; preheats in the range 160 to 300° C). The sediment is aeolian, but was probably washed into the cave site during periods of exceptionally heavy rainfall. Only one pair of ages is in the age range usually regarded as reliable for ^{14}C (layer 9 at 239 to 249 cm; 24.93 ± 0.34 ^{14}C years BP [28 ± 2 ka after calibration] and OSL 25.5 ± 1.4 ka). Although the older ages obtained using conventional pretreatments showed clear evidence of the ‘radiocarbon barrier’ (Roberts *et al.*, 1994), those obtained using a new acid-base-wet-oxidation pretreatment with stepped combustion (ABOX-SC) provided ages beyond 40 ka. Two of these, at 41.5 ± 1.3 and 46.7 ± 1.9 ^{14}C ka BP, can be directly compared with OSL ages of 44.4 ± 2.1 and 47.1 ± 2.6 ka. Turney *et al.* (2001) suggest that these ^{14}C ages can be

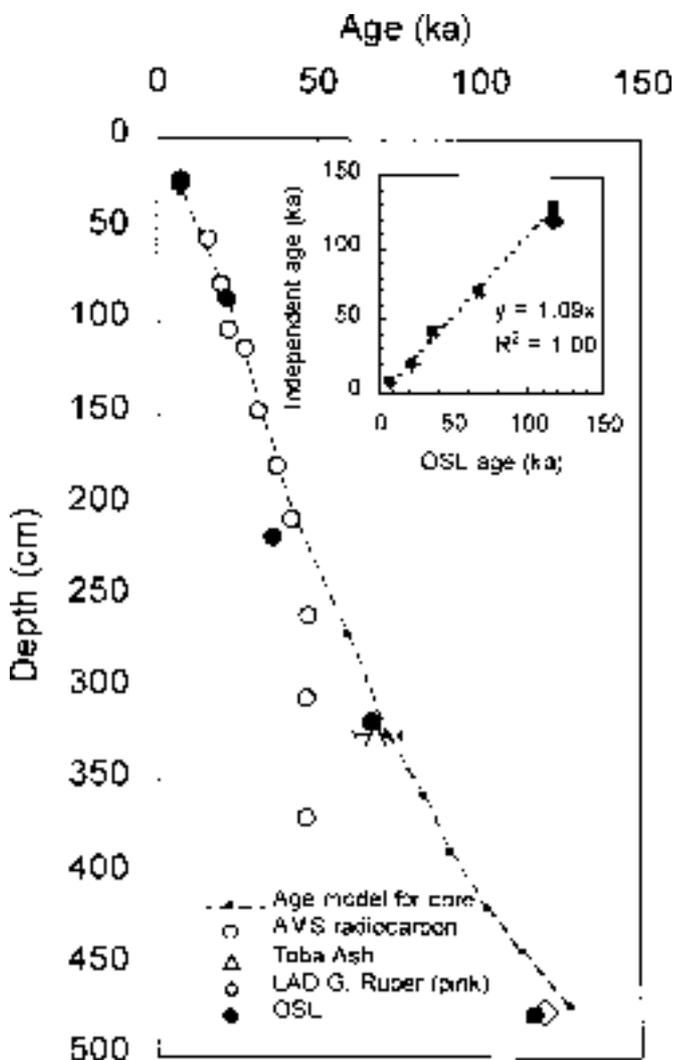


Fig. 9. Chronological models for core 70KL, taken from Stokes *et al.* (submitted). OSL dates are based on silt-sized quartz extracted from a deep sea core in the Indian Ocean. AMS radiocarbon ages include a 400 year sea water correction and are calibrated. Other age control includes the Toba Ash (variably dated to between 68 ± 7 ka by fission track dating and 73–75 ka by Ar-Ar), a key biostratigraphic marker horizon, LAD G. *ruber* (pink) (which provides a maximum age of 120 ka) and comparison of the oxygen isotope record with the SPECMAP-Stack (small markers indicate the tie-points; correlation coefficient=0.94). Details of these independent age controls are given by Stokes *et al.* (submitted).

calibrated by the addition of 1 to 2 ka, based on Kitagawa and van der Plicht (1998) and Voelker *et al.* (1998).

Freshwater – Tanaka *et al.* (2001) have worked with river terrace samples from the Kanto Plain in Japan, using preheating for 10 s at 240° C. At one site age control was provided by the Nakadaichi tephra (55–60 ka, Yamagata site), and their SAR age of 55.6 ± 1.3 ka, calculated using the present day water content, was in good agreement with this. Roberts *et al.* (2001) reported an OSL age of 55 ± 9 ka for sediments from Mammoth cave, Australia, which were bracketed by flowstones with $^{230}\text{Th}/^{234}\text{U}$ ages of 44.4 ± 1.3 ka (above) and 55.2 ± 2.2 ka (below). They also reported $^{230}\text{Th}/^{234}\text{U}$ ages from two other sites which were stratigraphically concordant with OSL dates.

Marine – Larsen *et al.* (1999) present one comparison from their Trepuzovo site, where an (uncalibrated) ^{14}C age on wood (*Picea* twigs) gave 42.6 ± 1.5 ka BP, compared with an OSL age of 54 ± 4 ka for the surrounding marine sand unit.

Stokes *et al.* (2001) have reported a set of 3 older OSL ages in their study of a marine core from the Indian Ocean discussed above. Fig. 9 shows that these older ages are in good agreement with the independent age model, although there is a slight systematic tendency to underestimate the independent age.

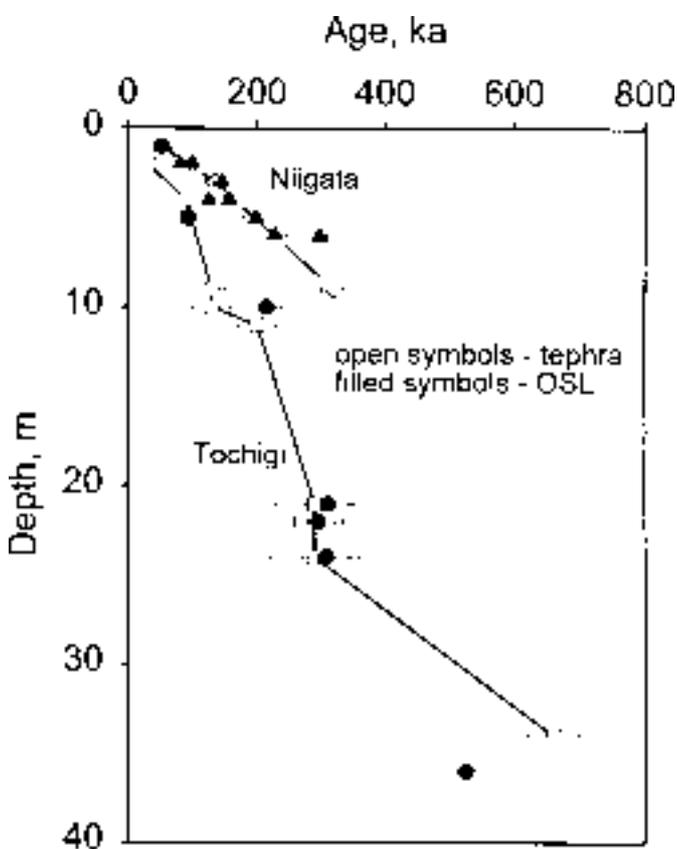


Fig. 10. Comparison of OSL ages from silt-sized quartz grains with an independent tephra chronology (Watanuki *et al.*, submitted). The tephra ages are based on ^{14}C and fission track dating. Unusually high water contents (around 100%) contributed to the low dose rates of about $1 \text{ Gy}\cdot\text{ka}^{-1}$.

Sigaard *et al.* (unpublished) have obtained two dates on a mixed pebble and sand unit near Ejby on Zealand, Denmark; they identified this as an Eemian beach deposit (assigned an age of between 115 and 129 ka by comparison with oxygen isotope stage 5e). The average OSL age obtained for this unit was 135 ± 8 ka. Mangerud *et al.* (2000) discuss two sites in northern Russia with deposits identified as Eemian. They give three OSL dates, of 111 ± 12 , 97 ± 7 and 64 ± 6 ka at site Sula 22, from a shallow marine unit 8 to 9 m thick. A similar 3 m thick Eemian deposit was identified at the adjacent site Sula 21, and this gave two OSL dates of 104 ± 11 and 91 ± 15 ka. These two sites are only 4 km apart, and Mangerud *et al.* (2000) consider the correlation between the two clear. The unweighted average OSL age is 93 ± 9 ka (101 ± 5 ka excluding the 64 ka outlier).

Murray *et al.* (unpublished data) have sampled an extensive sequence of Eemian marine sediments near Gammelmark, in Jutland, Denmark. The sequence is about 12 m high, and grades from clean sand in the upper layers to clayey silt at the bottom. Late Saalian freshwater sediments underlie the sequence. There was no apparent trend in the SAR OSL ages with depth, and all 24 ages are summarised in Fig. 11. The unweighted mean age (excluding the two outliers, and including uncertainties arising from systematic sources) is 119 ± 7 ka, with an overall relative sample standard deviation of 8% (these data are also discussed in Murray *et al.*, 2002). This variability should be compared with the average relative standard deviation based on the individual estimates of uncertainty on each age, of 5% (calculated by averaging the individual variances), and suggests that the internally derived uncertainties are slightly underestimated. This may be connected with the two results (out of 24) which are clearly anomalous, underestimating the known age by about 40%. The authors have evidence that this underestimate arises from a dose-rate anomaly, possibly because of radionuclide redeposition occurring as the sand cliff is

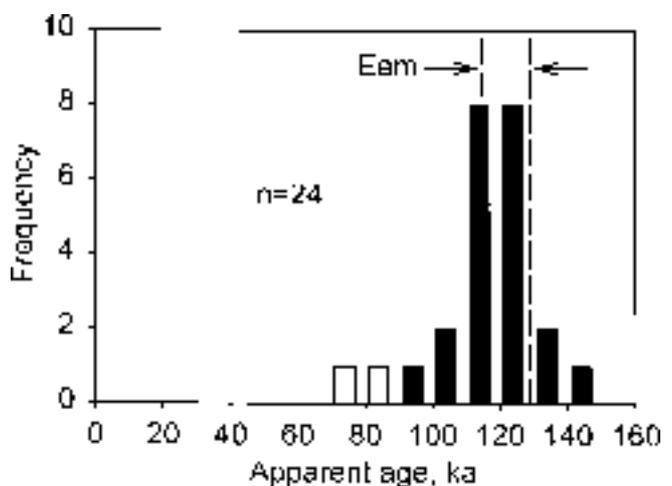


Fig. 11. Distribution of dates (obtained using a preheat of 260° C for 10 s) from 24 samples of Eemian coastal marine sand taken from a coastal cliff section near Gammelmark, south east Jutland. There was no systematic change in age with depth in the section, and the unweighted mean is 119 ± 7 ka ($n=22$, i.e. excluding the lowest two ages).

eroded back by storm action. It is clearly possible that similar but smaller and undetected variations in dose rate with time could contribute to the observed scatter in the accepted ages. It is also interesting that the gross underestimation observed here is similar to that found for one sample by Mangerud *et al.* (2000) reported above; this sample was also recovered from a retreating sediment face.

Glaciofluvial/glaciolacustrine – As part of his study of the deglaciation history of Denmark, Houmark-Nielsen has obtained several SAR quartz OSL ages with good ^{14}C age control. The samples come from a variety of locations, and all the older samples are from lacustrine muds and sands. These results are summarised in **Table 2**, together with the two younger samples discussed earlier; the agreement between OSL and the independent chronology is satisfactory, although there may be a small systematic discrepancy.

8. DISCUSSION

Fig. 12 and **Table 3** summarise all the non-modern age comparisons discussed above. In order not to bias the data towards multiple OSL ages with a single independent age control (e.g. the 22 results from Gammelmark; Murray *et al.*, unpublished) results from a single horizon at a given site have been averaged (the number of ages is given in the column labelled (n) in **Table 3**). Those comparisons where the relationship with the age control is poorly defined have also been omitted (e.g. Rich and Stokes, 2001). The older uncalibrated ^{14}C ages of Turney *et al.* (2001) have been calibrated approximately by adding 1.5 ± 1.0 ka to the uncalibrated data (based on Kitagawa and van der Plicht, 1998, and Voelker *et al.*, 1998). The data are shown with logarithmic axes; the inset to the lower graph shows the same data using linear axes.

Because of the logarithmic axes, the modern results have been omitted from the figure. However, it is interesting to note that although there are several average D_e estimates (i.e. measured using an large number of grains in each aliquot) from modern freshwater deposits (e.g. Stokes *et al.*, 2001), many of which show the expected evidence for incomplete bleaching, there is only one such estimate from sediment of known finite age (Wallinga *et al.*, 2001). This reflects the increasing acceptance that the study of dose distributions using small numbers of grains, or even single grains, is the standard method if incomplete bleaching is suspected.

It is also interesting that in none of the dose distribution studies of modern sediments (or of young non-modern sediments) using small aliquots or single grains, is there any suggestion that the dose distribution approach overestimates the known age. Thus we conclude that, at least in the sediment results reviewed here, there were always some grains that were completely bleached when compared to a time scale of years to decades.

Turning now to **Fig. 12**, it is clear that there is no evidence for any systematic difference between the OSL ages and the independent age estimates over the entire age range – although it must be acknowledged that there are

only a few comparisons in the range between about 50 ka and 125 ka (because of the lack of reliable independent ages), and the data from beyond about 125 ka are all from one study (Watanuki *et al.*, unpublished). The average ratio of the OSL to independent ages is 0.984 ± 0.016 ($n = 52$) unweighted, and 0.978 ± 0.009 weighted (both omitting the freshwater result of 3.1 ± 0.4 at 300 years). Nevertheless, individual points are not all statistically consistent with the line of unit slope. This can be seen more readily in the upper part of the figure, where the OSL/independent age ratios are shown; 5 out of the 53 results lie more than 3 standard deviations from unity. Even ignoring the result of 3.1 ± 0.4 at 300 years (because the source of error in this case is known to be incomplete bleaching), it can be concluded that uncertainties in the ratio of OSL to independent age are underestimated in some cases. Unfortunately it is difficult to deduce where the error lies – in the measurement of the independent estimate of age, in the association of this age with that of the sedimentary horizon, or in the luminescence age itself.

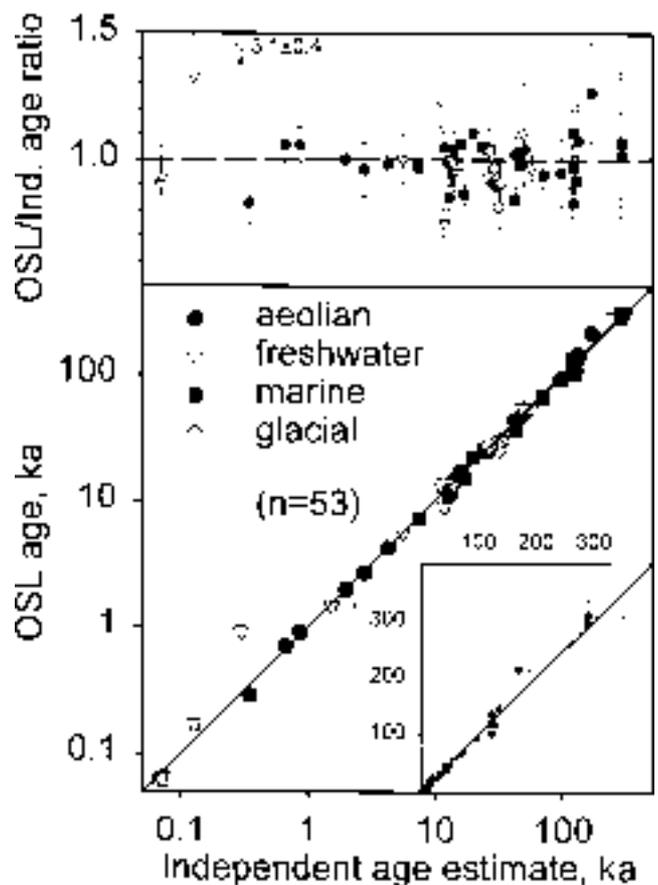


Fig. 12. Summary of age comparisons from all sites discussed in this paper. Note the logarithmic horizontal axis. The OSL to independent age ratios are shown in the top part of the figure, and the inset shows the age data using linear axes. Samples of known modern (i.e. zero) age are not shown. One other comparison has been omitted – from Larsen *et al.* (1999) – because the ^{14}C age of 42.6 ± 1.5 ka involved conventional pretreatment and is uncalibrated. Two other pairs, from Wallinga *et al.* (2001; see Figure 8), are omitted from the upper part of the diagram only, because the independent age (1.54 ± 0.84 ka) is poorly known.

Nevertheless, some comment can be made on the luminescence ages. Combined uncertainties range from 50% (Olley and Taylor, unpublished) to 2.3% (Stokes *et al.*, submitted). Six out of the 53 results are assigned combined uncertainties of 3% or less by their authors (not including the effects of averaging). It is of concern that only limited information is available on uncertainty analysis in some of the data sets reviewed here; in particular,

a discussion of the analysis of random and systematic sources of uncertainty is often missing. This is despite two well-known early publications (Aitken and Allred, 1972; Aitken 1976; both summarised in Aitken, 1985) which outline these contributions, and discuss methods for combining them in the final age. In any comparison of luminescence ages with independent dating results, it is very important that the uncertainties on the luminescence ages

Table 3. Summary of all age comparisons used in Figure 12.

Class	Aliquot and grain size	Preheat, 10 s [°C]	OSL Age [ka]	(n)	Independent Age [ka]	Reference
Aeolian	l, c	240-280	0.29±0.02	1	0.35±0.03	Strickertsson and Murray, 1999
	l, c	160-260	0.71±0.05	1	0.669	Bailey <i>et al.</i> , 2001 & pers. comm.
	l, c	260	13.00±0.7	3	13.155	Radtke <i>et al.</i> , 2001
	l, c	260	11.2±1.3	1	13.155	Hilgers <i>et al.</i> , 2001
	l, c	260	13.1±0.9	2	13.45±0.3	Hilgers <i>et al.</i> , 2001
	l, c	260	12.6±0.7	1	12.0±0.3*	Mangerud <i>et al.</i> , 1999
	l, c	260	14.6±1.2	1	14.3±0.5*	Mangerud <i>et al.</i> , 1999
	l, c	160-280	4.23±0.1	6	4.31±0.07	Murray and Clemmensen, 2001
	l, c	160-280	2.7±0.3	1	2.81±0.02	Murray and Clemmensen, 2001
	l, c	160-280	2.0±0.2	1	2.00±0.05	Murray and Clemmensen, 2001
	l, c	160-280	0.92±0.04	4	0.87±0.04	Murray and Clemmensen, 2001
	l, f	240	93±10	1	98±9	Watanuki <i>et al.</i> , 2002
	s, c	160-300	25.5±1.4	1	28±2*	Turney <i>et al.</i> , 2001
	s, c	160-300	44.1±2.1	1	43±2*	Turney <i>et al.</i> , 2001
	s, c	160-300	47.1±2.6	1	48±2*	Turney <i>et al.</i> , 2001
	l, f	240	215±22	1	170±20	Watanuki <i>et al.</i> , 2002
	l, f	240	311±33	1	290±60	Watanuki <i>et al.</i> , 2002
	l, f	240	296±39	1	290±30	Watanuki <i>et al.</i> , 2002
	l, f	240	308±36	1	290±70	Watanuki <i>et al.</i> , 2002
	l, f	260	53±3	1	51±1	Watanuki <i>et al.</i> , 2002
l, f	260	145±12	1	135±15	Watanuki <i>et al.</i> , 2002	
Fluvial	s, c	200	67±5 a	1	70 a	Olley <i>et al.</i> , 1998
	l, c	240-280	13.6±1.1	1	14.3±0.1	Strickertsson and Murray, 1999
	l, c	240-280	9.0±0.6	2	12±0.3	Strickertsson and Murray, 1999
	s, c	200	0.92±0.10	1	0.3	Wallinga <i>et al.</i> , 2001
	s, c	200	1.49±0.10	1	1.54±0.84	Wallinga <i>et al.</i> , 2001
	s, c	200	5.62±0.35	1	5.62±0.41	Wallinga <i>et al.</i> , 2001
	s, c	200	13.26±0.8	1	13.24±0.07	Wallinga <i>et al.</i> , 2001
	sg	220	70±8 a	1	63±8 a	Olley and Hancock (unpub.)
	sg	220	170±20 a	1	128±10 a	Olley and Hancock (unpub.)
	s, c	220	29±15 a	1	42 a	Olley and Taylor (unpub.)
	l, c	260	13.6±1.0	1	13.9±0.5	Houmark-Nielsen (Table 2)
	l, c	260	13.8±0.9	1	14±0.5	Houmark-Nielsen (Table 2)
	l, c	260	13.7±1.1	1	11.1±0.3	Larsen <i>et al.</i> , 1999
	l, c	240	55.6±1.3	1	58±2	Tanaka <i>et al.</i> , 2001
	s, c	160-300	55±9	1	49.8±4	Roberts <i>et al.</i> , 2001
Marine	l, c	240-280	25.3±1.8	1	24±2	Strickertsson and Murray, 1999
	l, c	240-280	14.9±0.6	3	17.2±0.4	Strickertsson and Murray, 1999
	l, c	240-280	17.3±1.5	1	16.2±0.7	Strickertsson and Murray, 1999
	l, c	260	119±7	22	122±7	Murray <i>et al.</i> (unpub.)
	l, c	260	135±8	2	122±7	Sigaard <i>et al.</i> (unpub.)
	l, c	260	101±4	4	122±7	Mangerud <i>et al.</i> , 1999
	l, f	200, 240	7.31±0.18	1	7.50±0.09	Stokes <i>et al.</i> , 2002
	l, f	200, 240	22.1±0.4	1	20.0±0.12	Stokes <i>et al.</i> , 2002
	l, f	200, 240	36.3±0.8	1	43±2	Stokes <i>et al.</i> , 2002
	l, f	200, 240	67±2	1	71±4	Stokes <i>et al.</i> , 2002
	l, f	200, 240	117±3	1	128±6	Stokes <i>et al.</i> , 2002
	Glacial	l, c	260	30±2	1	33±2
l, c		260	29±2	1	32±3	Houmark-Nielsen (Table 2)
l, c		260	30±3	1	33±1	Houmark-Nielsen (Table 2)
l, c		260	26±2	1	32±2	Houmark-Nielsen (Table 2)
l, c		260	29±2	1	30±2	Houmark-Nielsen (Table 2)
l, c	260	28±2	1	27±1	Houmark-Nielsen (Table 2)	
Note:	<ol style="list-style-type: none"> 1. Aliquot size: l - large, s - small, sg - single grain. Grain size, c - coarse, f - silt. 2. (n) is the number of OSL ages included in the average given. 3. * indicates radiocarbon age calibrated by the present authors. 					

include contributions from all known components, including uncertainties arising from systematic effects (of course the same applies to the independent ages, but such problems are outside the scope of this paper). Some sources of error that are difficult to avoid (given the methods used in the papers reviewed here) include conversion from concentration data to dose rate (estimated at ~3%), absolute calibration of concentration measurements (~3%), beta source calibration (~2%), and beta attenuation factor (~2%). These estimated values are of course approximate, but it should be clear that it is difficult to obtain a luminescence age with an overall or combined standard uncertainty of much less than 5%. This is especially true when it is remembered that other sources of systematic errors, such as those associated with water content and cosmic ray contribution, have not been considered in this discussion because they are site dependent, and that uncertainties arising from random errors also contribute to the combined standard uncertainty. It is also interesting to note that if a relative uncertainty of 5% is added in quadrature to all the OSL ages, only one OSL/independent age ratio (0.750 ± 0.065 at 12 ka) lies more than 3 standard uncertainties from unity (again not including the result of 3.1 ± 0.4 at 300 years, because the source of error is known).

In the past, any uncertainties arising from systematic errors have tended to be swamped by those arising from random errors, especially errors associated with measurement of D_e . Now that precisions on the mean D_e of $< 5\%$ are possible using SAR, it is very important that full attention is given to the uncertainties arising from systematic effects – increasingly these will limit the combined uncertainty in a luminescence date. Only then will it be possible to determine whether the apparently significant deviations of the type shown in Fig. 12 are simply a result of the underestimation of the effects of known sources of error, or whether they reflect a hitherto unidentified source.

9. CONCLUSIONS

There is now a considerable number of comparisons available between quartz SAR ages and independent age control, from a variety of aeolian and water-lain sediments. These very encouraging results confirm that the method has a wide application to many classes of transported material. Incomplete bleaching is very unlikely to give rise to significant errors in aeolian and coastal marine sands, but it can be of significance in Holocene studies of fluvial deposits. However, there is no evidence for significant systematic effects in ages older than about 20 ka, even from fluvial or glacio-fluvial systems, and this should be borne in mind when examining dose distributions in older sediments.

Nevertheless, when due allowance has been made for incomplete bleaching, there remain two or three individual results that are clearly inconsistent with the expected results, and these point the way to more detailed future investigations, especially of dose rates. It is clear that these occasional failures reinforce what is, in any case,

good field practice – that several samples, stratigraphically connected in both horizontal and vertical planes, should be dated from every site. This minimises the risk of undetected gross errors.

It is also strongly recommended that more detailed attention should be paid to formal uncertainty analysis. It has long been recommended good practice in luminescence dating to report both the combined standard uncertainty, assessed in a justified way, and (separately) the component arising from systematic effects; unfortunately this is very rare. With the increased precision available from the SAR protocol, such detailed error analysis is essential if the reporting of unreasonably small uncertainties is to be avoided. We stress that determination and reporting of the uncertainties associated with an age estimate is as important as the determination of the age.

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REFERENCES

- Aitken M.J., 1976: Thermoluminescent age evaluation and assessment of error limits: revised system. *Archaeometry* 18: 233-238.
- Aitken M.J., 1985: Thermoluminescence Dating. *Academic Press*, Oxford. 359 pp.
- Aitken M.J., 1998: An Introduction to Optical Dating. *Oxford University Press*, Oxford: 276 pp.
- Aitken M.J. and Alldred J.C., 1972: The assessment of error limits in thermoluminescent dating. *Archaeometry* 14: 257-267.
- Bailey S.D., Wintle A.G., Duller G.A.T. and Bristow C.S., 2001: Sand deposition during the last millenium at Aberffraw, Anglesey, North Wales as determined by OSL dating of quartz. *Quaternary Science Reviews (Quaternary Geochronology)* 20: 701-704.
- Banerjee D., 2000: Thermal transfer and recuperation in quartz OSL and their consequences regarding optical dating procedures. In: Murthy K. V. R. *et al.*, eds, *Luminescence and its Applications I*. Luminescence Society of India C 1/2000: 86-93.
- Banerjee D., Murray A.S. and Foster I.D.L., 2001: Scilly Isles, UK: optical dating of a possible tsunami deposit from the 1755 Lisbon earthquake. *Quaternary Science Reviews (Quaternary Geochronology)* 20: 715-718.
- Bøtter-Jensen L., Bulur E., Duller G.A.T. and Murray A.S., 2000: Advances in luminescence instrument systems. *Radiation Measurements* 32: 523-528.
- Bøtter-Jensen L. and Duller G.A.T., 1992: A new system for measuring OSL from quartz samples. *Nuclear Tracks and Radiation Measurements* 20: 549-533.
- Colls A.E., Stokes S., Blum M.D. And Straffin E., 2001: Age limits on the Late Quaternary evolution of the upper Loire River. *Quaternary Science Reviews* 20: 743-750.
- Friedrich M., Kromer B., Spurk M., Hofmann J. and Kaiser K.L., 1999: Paleo-environment and radiocarbon calibration as derived from Lateglacial/Early Holocene tree-ring chronologies. *Quaternary International* 61: 27-39.

- Galbraith R., Roberts R.G., Laslett G., Yoshida H. and Olley J., 1999:** Optical dating of single and multiple grains of quartz from Jinmium rock shelter, Northern Australia. Part I: Experimental Design and Statistical Models. *Archaeometry* 41: 339-364.
- Hilgers A., Murray A.S., Schlaak N. and Radtke U., 2001:** Comparison of quartz OSL protocols using Lateglacial and Holocene dune sands from Brandenburg, Germany. *Quaternary Science Reviews (Quaternary Geochronology)* 20: 731-736.
- Kitigawa H. and van der Plicht J., 1998:** Atmospheric radiocarbon calibration to 45,000 yr B.P.: Late Glacial fluctuations and cosmogenic isotope production. *Science* 279: 1187-1190.
- Larsen E., Lyså A., Demidov I., Funder S., Houmark-Nielsen M., Kjær K.H. and Murray A.S., 1999:** Age and extent of the Scandinavian ice sheet in northwest Russia. *Boreas* 28: 115-132.
- Mangerud J., Svendsen J.I. and Astakhov V.I., 2000:** Age and extent of the Barents and Kara ice sheets in Northern Russia. *Boreas* 28: 46-80.
- Murray A.S. and Clemmensen L.B., 2001:** Luminescence dating of Holocene aeolian sand movement, Thy, Denmark. *Quaternary Science Reviews (Quaternary Geochronology)* 20: 751-754.
- Murray A.S. and Olley J.M., 1999:** Determining sedimentation rates using luminescence dating. In: Bruns P. and Hass H.C., eds., *Determination of Sediment Accumulation Rates*. GeoResearch Forum, Trans Tech Publications, Switzerland: 121-144.
- Murray A.S., Olley J.M. and Caitcheon G.C., 1995:** Measurement of the equivalent doses in quartz from contemporary water-lain sediments using optically stimulated luminescence. *Quaternary Science Reviews (Quaternary Geochronology)* 14: 365-371.
- Murray A.S. and Roberts R.G., 1998:** Measurement of the equivalent dose in quartz using a regenerative-dose single aliquot protocol. *Radiation Measurements* 29: 503-515.
- Murray A.S., Roberts R.G. and Wintle A.G., 1997:** Equivalent dose estimation using a single aliquot of quartz. *Radiation Measurements* 27: 171-184.
- Murray A.S. and Wintle A.G., 2000:** Luminescence dating of quartz using an improved single-aliquot regenerative-dose protocol. *Radiation Measurements* 32: 57-73.
- Murray A.S., Wintle A.G. and Wallinga J., 2002:** Dose estimation using quartz OSL in the nonlinear region of the growth curve. *Radiation Protection Dosimetry*, in press.
- Oliver R.L., 1990:** Optical properties of waters in the Murray-Darling Basin, southeastern Australia. *Australian Journal of Marine and Freshwater Research* 41: 581-601.
- Olley J.M., Caitcheon G.C. and Murray A.S., 1998:** The distribution of apparent dose as determined by optically stimulated luminescence in small aliquots of fluvial quartz: implications for dating young sediments. *Quaternary Science Reviews (Quaternary Geochronology)* 17: 1033-1040.
- Olley J.M., Caitcheon G., and Roberts R.G., 1999:** The origin of dose distributions in fluvial sediments, and the prospect of dating single grains of quartz from fluvial deposits using OSL. *Radiation Measurements* 30: 207-217.
- Radtke U., Janotta A., Hilgers A. and Murray A.S., 2001:** The potential for OSL dating Lateglacial and Holocene dune sands with independent age control of the Laacher See tephra (12880 a) at the Section 'Mainz-Gonsenheim'. *Quaternary Science Reviews (Quaternary Geochronology)* 20: 719-724.
- Rhodes E.J., 1988:** Methodological considerations on the optical dating of quartz. *Quaternary Science Reviews (Quaternary Geochronology)* 16: 275-280.
- Rhodes E.J., 2000:** Observations of thermal transfer OSL signals in glacial quartz. *Radiation Measurements* 32: 595-602.
- Rich J. and Stokes S., 2001:** Optical dating of geoarchaeologically significant sites from the southern High Plains and South Texas, USA. *Quaternary Science Reviews (Quaternary Geochronology)* 20: 949-960.
- Roberts R., Bird M., Olley J., Galbraith R., Lawson E., Laslett G., Yoshida H., Jones R., Fullagar R., Jacobsen G. and Hua Q., 1998:** Optical and radiocarbon dating at Jinmium rock shelter, northern Australia. *Nature* 393: 358-362.
- Roberts R.G., Flannery T.F., Ayliffe L.K., Yoshida H., Olley J.M., Prideaux G.J., Laslett G.M., Baynes A., Smith M.A., Jones R., and Smith B., 2001:** New ages for the last Australian megafauna: continent-wide extinction about 46,000 years ago. *Science* 292: 1888-1892.
- Roberts R.G., Galbraith R., Olley J., Yoshida H., and Laslett G., 1999:** Optical dating of single and multiple grains of quartz from Jinmium Rock Shelter, Northern Australia. Part II: results and implications. *Archaeometry* 41: 365-395.
- Roberts R.G., Jones R. and Smith M.A., 1994:** Beyond the radiocarbon barrier in Australian prehistory. *Antiquity* 68: 611-616.
- Schlaak N., 1993:** Studie zur Landschaftsgenese im Raum Nordbarnim und Eberwalde Urstromtal. *Berliner Geographische Arbeiten* 76: 146 p.
- Stokes S., Bray H.E. and Blum M.D., 2001:** Optical resetting in large drainage basins: tests of zeroing assumptions using single-aliquot procedures. *Quaternary Science Reviews (Quaternary Geochronology)* 20: 880-885.
- Stokes S., Ingram S., Aitken M.J., Sirocko F. and Anderson, R., 2002:** Alternative chronologies for Late Quaternary (last interglacial – Holocene) deep sea sediments via optical dating of silt-sized quartz. *Quaternary Science Reviews (Quaternary Geochronology)*, submitted.
- Strickertsson K. and Murray A.S., 1999:** Optically stimulated luminescence dates for Late Pleistocene and Holocene sediments from Nørre Lyngby, Northern Jutland, Denmark. *Quaternary Science Reviews (Quaternary Geochronology)* 18: 169-178.
- Strickertsson K., Murray A.S. and Lykke-Anderson H., 2000:** Optically stimulated luminescence dates for Late Pleistocene sediments from Stensnæs, northern Jutland, Denmark. *Quaternary Science Reviews (Quaternary Geochronology)* 20: 755-759.
- Tanaka K., Hataya R., Spooner N.A. and Questiaux D.G., 2001:** Optical dating of river terrace sediments from Kanto plains, Japan. *Quaternary Science Reviews (Quaternary Geochronology)* 20: 826-828.
- Taylor B.N. and Kuyatt C.E., 1994:** Guidelines for evaluating and expressing the uncertainty of NIST measurement results. *National Institute of Standards and Technology, NIST Technical Note 1297*, US Government Printing Service: 24 pp.
- Turney C.S.M., Bird M.J., Fifield L.K., Roberts R.G., Smith M., Dortch C.E., Grün R., Lawson E., Ayliffe L.K., Miller G.H., Dortch J. and Creswell R.G., 2001:** Early human occupation at Devil's Lair, southwestern Australia 50,000 years ago. *Quaternary Research* 55: 3-13.
- Voelker A.H.L., Sarntheim M., Grootes P.M., Erlenkeuser H., Laj C., Mazaud A., Nadeau M.-J. and Schleicher M., 1998:** Correlation of marine ^{14}C ages from the Nordic Seas with the GISP2 isotope record: implications for the ^{14}C calibration beyond 25 ka BP. *Radiocarbon* 40: 517-534.
- Wallinga J., Murray A.S., Duller G.A.T. and Törnqvist T.E., 2001:** Testing optically stimulated luminescence dating of sand-sized quartz and feldspar from fluvial deposits. *Earth and Planetary Science Letters* 193: 617-630.
- Watanuki T., Murray A.S. and Tsukamoto S., 2002:** Quartz and polymineral luminescence dating of Japanese loess over the last 0.5 Ma: comparison with an independent chronology. *Earth and Planetary Science Letters*, submitted.
- Wintle A.G., 1997:** Luminescence dating: laboratory procedures and protocols. *Radiation Measurements* 27: 769-817.

