Optical dating of Holocene sediments from a variety of geomorphic settings using single grains of quartz

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Received 19 May 2003; received in revised form 1 September 2003; accepted 9 September 2003

Available online 19 November 2003

Abstract

This paper presents an improved method for the optical dating of Holocene sediments from a variety of geomorphic settings. We have measured the equivalent dose ($D_e$) in individual grains of quartz, using green laser light for optical stimulation, and have simulated the $D_e$ distributions for multiple-grain ‘synthetic’ aliquots using the single-grain data. For 12 samples of known (independent) age, we show that application of a ‘minimum age model’ to the single-grain and ‘small’ (10-grain) aliquot $D_e$ data provides the most accurate estimate of the burial dose for nine of the samples examined (3 aeolian, 5 fluvial, and 1 marine). The weighted mean $D_e$ (as obtained using the ‘central age model’) gives rise to burial age overestimates of up to a factor of 10 for these nine samples, whether single grains, small aliquots, or ‘large’ (100-grain) aliquots are used. For the other three samples (two aeolian and one fluvial), application of either the minimum age model or the central age model to the single-grain, small aliquot, and large aliquot $D_e$ data yields burial ages in accord with the independent age control. We infer that these three samples were well bleached at the time of deposition. These results show that heterogeneous bleaching of the optical dating signal is commonplace in nature, and that aeolian transport offers no guarantee that the sample will be well bleached at the time of deposition. We also show that grains sensitive to infrared (IR) stimulation can give rise to low $D_e$ values, which will result in significant underestimation of the burial dose and, hence, of the age of deposition. We demonstrate that use of a modified single-aliquot regenerative-dose protocol incorporating IR stimulation prior to green light stimulation deals effectively with contamination by IR-sensitive grains. We conclude that application of the modified protocol to single grains or small aliquots of quartz, using the lowest $D_e$ population to estimate the burial dose, is the best means of obtaining reliable ages for Holocene sediments from a wide range of depositional environments.

Keywords: Optical dating; Radiocarbon dating; Holocene deposits; Burial ages; Australia

1. Introduction

Methods to determine the ages of Holocene materials with accuracy and precision are of keen interest to geomorphologists, archaeologists, and other Quater-
nary scientists. Although radiocarbon \((^{14}C)\) dating has proven extremely useful in many contexts, there are several instances where \(^{14}C\) dating remains problematic. Foremost among these are marine deposits formed within the last 200 years (as corrections for ‘reservoir’ effects and the calibration of \(^{14}C\) ages result in large age uncertainties; Stuiver et al., 1998) and Holocene fluvial deposits (as shown by the non-modern \(^{14}C\) ages of many wood fragments recovered from recent fluvial deposits; Blong and Gillespie, 1978).

Recent developments in optical dating of quartz have shown the potential to date coastal dunes formed within the last 100 years to a precision of better than \(\pm 20\%\) (e.g., Murray and Clemmensen, 2001; Murray-Wallace et al., 2002; Ballarini et al., 2003). Such deposits are generally composed of wind-blown sediment grains that have been exposed to sufficient sunlight to zero the optically stimulated luminescence (OSL) signal during the most recent transport event. In such instances, the measured equivalent dose \(D_e\) values obtained from single aliquots composed of multiple or individual grains of quartz (e.g., Olley et al., 1998, 1999; Lepper et al., 2000; Fuchs and Lang, 2001; Lepper and McKeever, 2002; Fuchs and Wagner, 2003; Zhang et al., 2003). Here we apply the most mathematically rigorous of these methods, the so-called ‘minimum age model’ (Galbraith and Laslett, 1993; van der Touw et al., 1997; Galbraith et al., 1999), to the \(D_e\) values obtained from aliquots of varying size for 12 samples of independently known age. We show that the application of this model to single-grain and small aliquot \(D_e\) data produces estimates of \(D_b\) that yield optical ages in good agreement with the known ages.

2. Optical dating

When quartz grains are buried, they begin to accumulate a trapped-charge population that increases in a measurable and predictable way in response to the ionising radiation dose to which the grains are exposed. Exposure to sunlight releases the light-sensitive trapped charge, thereby resetting the OSL signal; this process is commonly referred to as ‘bleaching’. The time elapsed since sediment grains were last exposed to sunlight can be determined by measuring the OSL signal from a sample of sediment, determining the \(D_e\) that this represents, and estimating the rate of exposure of the grains to ionising radiation since they were buried (Huntley et al., 1985; Aitken, 1998). The latter parameter of interest is termed the dose rate \(D_r\) and the burial age of well-bleached grains may be obtained from the following equation:

\[
\text{Burial age (years)} = D_e \, (\text{Gy}) / D_r \, (\text{Gy/year})
\]

\((\text{Gy = gray, where } 1 \text{ Gy} = 1 \text{ J/kg})\)

When clean quartz grains are exposed directly to sunlight, the OSL signal is reduced to a negligible level within a few seconds (Wintle, 1997; Aitken, 1998). However, incomplete or non-uniform bleaching is commonplace in many depositional environments (Murray and Olley, 2002), due to surface coating on grains and/or poor exposure to sunlight during sediment transport. This results in grains being deposited with a heterogeneous distribution of residual trapped charge and a correspondingly wide range of measured \(D_e\) values. For such sediments, Olley et al. (1999) suggested that the population of grains with the lowest measured \(D_e\) values provides the most accurate estimate of \(D_b\): the burial dose to which those grains that were well bleached at deposition have been exposed since the most recent transport event.

This recommendation was not supported, however, by the single-grain \(D_e\) distribution presented by Spooner et al. (2001) for an Australian dune sand for which the time of deposition was considered to be well constrained by \(^{14}C\) dating. In this case, the lowest population of \(D_e\) values underestimated the burial age by several thousand years. In this paper, the apparent disagreement between the results of Olley et al. (1999) and those of Spooner et al. (2001) is examined further and resolved using an improved methodology. We
also extend these new methods to an additional 11 Holocene sediment samples from a variety of geomorphic environments (locations shown in Fig. 1) and for which we have independent age control.

3. Sample details

Sample NR99008 was collected from the Naas River valley in the Australian Capital Territory (35.7°S, 149.0°E). The Naas River is a headwater tributary of the Murrumbidgee River, one of Australia’s largest rivers. The sample was collected from a sand and gravel unit at a burial depth of 6.9 m, close to the base of a ~8 m-high river terrace. A sample of wood collected from a log found 10 cm below this sediment sample (Kilham, 1999) yielded a 14C age of 3170 ± 60 BP (Wk-7552), which corresponds to a calibrated age of 3380 years and a 1σ age range of 3340–3470 years (Stuiver et al., 1998). A full description of the stratigraphy of the Naas River deposit is given in Eriksson et al. (submitted for publication).

Sample S2-1 was collected from a gravel pit dug into the floodplain of the Murrumbidgee River near Wagga Wagga, New South Wales (35.0°S, 147.0°E). The pit exposes a 20 m-deep sequence of sediments.
The sample was taken at a depth of 4.3 m from a sand unit, which has been interpreted as a buried source-bordering dune (for details of the site stratigraphy, see Chen et al., 2002). A charcoal sample consisting of numerous fragments approximately 1–2 mm in size was also collected from this horizon for \(^{14}C\) dating. The charcoal fragments were clustered together in a tight pocket set in the sand (Spooner et al., 2001) and are thought to have originated from burning of the root of an eucalypt tree which grew on the dune (Chen et al., 2002). This sample yielded a conventional \(^{14}C\) age of 3540 ± 140 BP (ANU-9820), which gives a 1σ calibrated age range of 3630–4070 years. Problems encountered with single-grain optical dating of sample S2-1 have been reported previously (Spooner et al., 2001) and are examined further here.

Wangrah Creek (35.9°S, 149.3°E) is a 50 km\(^2\) headwater drainage basin of the Murrumbidgee River in southeastern New South Wales. The catchment has a continuous gully network eroded through Quaternary alluvium, which is derived from soils developed on Ordovician slates and sandstones (Prosser et al., 1994). Sample WK96008 was collected from a depth of 2.55 m in a vertical bank exposure along the main channel, approximately 8 km from the head of the valley. Small charcoal fragments collected from the same sediment horizon as the OSL sample yielded a valley. Small charcoal fragments collected from the channel, approximately 8 km from the head of the valley, were supplied from a major incision event upstream (Wilkins, 2002). Sample NU2_1 (36.2°S, 149.2°E) was collected from the clayey-sand deposit that fills an historic channel of the Numeralla River, located in the headwaters of the Murrumbidgee River. The channel was active at the time of European settlement (ca. A.D. 1840) but was infilled shortly afterwards by sediment supplied from a major incision event upstream (Wilkins, 2002). Sample NU2_4 was collected from a stratigraphically equivalent clayey-sand unit in the same area. Parish records and survey reports indicate that both samples have burial ages of between 120 and 160 years.

Samples GE96004 (burial depth 11.5 cm) and GE96005 (burial depth 12.5 cm) were extracted from a sediment core collected from the portion of the Barmah Forest (36.0°S, 145.0°E) situated on the lower Murray River floodplain, in southeastern Australia. European and American pine trees were introduced to Australia about 150 years ago. *Pinus* pollen grains, belonging mostly to *P. radiata*, are common in the regional pollen record after about A.D. 1880 (Ogden, 1996). We have, therefore, assigned a date of A.D. 1880 ± 20 to the first appearance of pine pollen in the core, at a depth of 20 cm (C. Kenyon, personal communication). The mineral magnetic susceptibility and chemistry of the floodplain sediments change at a core depth of 12 cm (C. Kenyon, personal communication). Sample GE96004 is, therefore, expected to have a burial age of ~55 years, and sample GE96005 a burial age of between 55 and 125 years.

Jacka Lake and Lake St. Mary (36.8°S, 141.8°E) are saline groundwater discharge lakes situated in the Wimmera District of semi-arid northwestern Victoria. When the lakes contain water, they act as traps for wind-blown ‘Lowan Sands’. The Lowan Sands form an aeolian cover of Quaternary age on the NNW–SSW trending sub-parallel ridges that lie to the west of the lakes, and that mark still-stands of a regressive Pliocene sea (Lawrence, 1975). They are dominated by moderately well sorted medium- to fine-grained quartz sands, are characterised by a lack of bonding clay, carbonate and soil development, and have been derived mostly from source areas located several tens to hundreds of kilometres to the north and west of the present dunefields (Pell et al., 2001). The sediments of Jacka Lake and Lake St. Mary consist predominantly of massive muds. Sample JK 46 cm was collected from a depth of 46 cm in Jacka Lake and is bracketed by two \(^{14}C\) ages on bulk sediment containing ostracod shells: 460 ± 140 BP (ANU-9805) at 23 cm depth and 1350 ± 130 BP (ANU-9806) at 59 cm depth. These yield 1σ calibrated age ranges of 315–625 years and 1090–1390 years, respectively. Samples NWJ 16 cm and NWJ 68 cm were collected from depths of 16 and 68 cm, respectively, from a core in the northwestern part of Jacka Lake. These samples are bracketed by \(^{14}C\) ages on bulk sediment extracted from the same core at depths of 8 cm (1130 ± 70 BP; ANU-9808), 27 cm (3480 ± 90 BP; OZA033), and 70 cm (5410 ± 90 BP; ANU-9809). The corresponding 1σ calibrated age ranges are 950–1170, 3630–3870 and 6000–6300 years, respectively. Sample SM15 was
collected from Lake St. Mary at a depth of 15 cm. Radiocarbon measurements on bulk sediment from the same level indicated that it contained 104.3% modern carbon, which corresponds to an age of $\sim$ 40 years. The anthropogenic fallout radionuclide $^{137}$Cs is also present in this sediment, and indicates an age of less than 40 years (Olley et al., 1991).

Sample HR5_60 (18.6°S, 146.3°E) was collected at a depth of 60 cm from a relict beach ridge on the Herbert River floodplain in far northern Queensland. This beach ridge lies near the landward margin of a Holocene beach ridge plain, inland of which is the Herbert River alluvial plain. The ridge has been quarried for sand and gravel, leaving a vertical face approximately parallel to the long axis of the ridge. Numerous charcoal fragments, associated with fire hearths, are present at depths of between 55 and 65 cm. Each hearth sits in a well-defined layer that extends laterally along the length of the beach ridge. These layers represent previous, now buried, surfaces that provide tight stratigraphic constraints on the charcoal and sediment samples. The preservation of the hearths attests to the lack of significant post-depositional disturbance. Charcoal samples from depths of 55, 60 and 65 cm gave $^{14}$C ages of 2630 ± 250 BP (DA883), 3680 ± 270 BP (DA884), and 5740 ± 330 BP (DA885), respectively, which correspond to 1σ calibrated age ranges of 2350–2990, 3640–4420 and 6190–6950 years.

4. Sample preparation and analytical methods

A portion of each sample was taken for water content determination and for measurement of the lithogenic radionuclide concentrations. For the OSL analyses, sand-sized grains of quartz (180–212 or 212–250 μm in diameter) were extracted from each sample using standard purification procedures (e.g., Aitken, 1998). The quartz grains were then etched in 40% hydrofluoric acid for 50 min to remove the outer 10 μm rinds and to completely remove any feldspar grains; however, any feldspar inclusions internal to the quartz grains will remain untouched. Finally, acid-soluble fluorides were removed in 15% hydrochloric acid.

Burial doses were determined from measurement of the OSL signals emitted by single grains of quartz and from ‘synthetic’ 10-grain and 100-grain aliquots that were constructed from these data. The etched quartz grains were loaded on to custom-made aluminium discs drilled with a 10 × 10 array of chambers, each of 300 μm depth and 300 μm diameter (Bøtter-Jensen et al., 2000). The OSL measurements were made on a Risø TL/OSL DA-15 reader using a green (532 nm) laser for optical stimulation, and the ultraviolet emissions were detected by an Electron Tubes 9235QA photomultiplier tube fitted with 7.5 mm of Hoya U-340 filter. Laboratory irradiations were conducted using a calibrated $^{90}$Sr/$^{90}$Y beta source mounted on the reader.

Equivalent doses were determined using either the single-aliquot regenerative-dose (SAR) protocol (e.g., Murray and Wintle, 2000; Yoshida et al., 2000), here referred to as ‘standard SAR’, or a modification of this protocol referred to as ‘modified SAR’ (Table 1). In both cases, a full dose-response curve was constructed for each grain. For the standard SAR protocol, the OSL signals were measured for 1 s at 125°C (laser at 90% power), using a preheat of either 200 or 240°C (held for 10 s) for the ‘natural’ and regenerative doses, and a cut-heat to 160°C for the test doses (6.5 Gy). The OSL signal was determined from the initial 0.1 s of data, using the final 0.2 s to estimate the background count rate. In the modified SAR protocol, each disc was exposed to infrared (IR) radiation for 40 s at 125°C prior to measurement of the OSL signal to

<table>
<thead>
<tr>
<th>Step</th>
<th>Treatment</th>
<th>Step</th>
<th>Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Give dose, $D_i$</td>
<td>1</td>
<td>Give dose, $D_i$</td>
</tr>
<tr>
<td>2</td>
<td>Preheat (200 or 240°C for 10 s)</td>
<td>2</td>
<td>Preheat (200 or 240°C for 10 s)</td>
</tr>
<tr>
<td>3</td>
<td>Stimulate with green laser for 1 s at 125°C</td>
<td>3</td>
<td>Stimulate with infrared diodes for 40 s at 125°C</td>
</tr>
<tr>
<td>4</td>
<td>Give test dose, $D_t$</td>
<td>4</td>
<td>Stimulate with green laser for 1 s at 125°C</td>
</tr>
<tr>
<td>5</td>
<td>Cut-heat to 160°C</td>
<td>5</td>
<td>Give test dose, $D_t$</td>
</tr>
<tr>
<td>6</td>
<td>Stimulate with green laser for 1 s at 125°C</td>
<td>6</td>
<td>Preheat (160°C for 10 s)</td>
</tr>
<tr>
<td>7</td>
<td>Return to Step 1</td>
<td>7</td>
<td>Stimulate with infrared diodes for 40 s at 125°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>8</td>
<td>Stimulate with green laser for 1 s at 125°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>9</td>
<td>Return to Step 1</td>
</tr>
</tbody>
</table>

*a For the natural sample, i = 0 and $D_0$ is the natural dose.
bleach any IR-sensitive signal, and a preheat of 160 °C for 10 s was used in preference to a cut-heat for the test doses to ensure that all of the grains were heated to 160 °C. This modified protocol is similar to that applied originally to mixed-mineral fine grains (4–11 μm in diameter) from colluvium (Banerjee et al., 2001) and loess (Roberts and Wintle, 2001) to measure the OSL signal from quartz in the presence of feldspar.

Grains were rejected if they did not produce a measurable OSL signal in response to the 6.5 Gy test dose, had OSL decay curves that did not reach background after 1 s of laser stimulation, or produced natural OSL signals that did not intercept the regenerated dose–response curves (‘Class 3’ grains of Yoshida et al., 2000). The reported $D_e$ uncertainties for each grain are based on counting statistics, the reproducibility with which the laser beam can be positioned (Truscott et al., 2000) and curve-fitting uncertainties. For each sample, the population of grains with the lowest $D_e$ (which we show to be conformable with the burial dose, $D_b$) has been estimated using the minimum age model, which has been described and tested in simulations by Galbraith and Laslett (1993), van der Touw et al. (1997) and Galbraith et al. (1999).

The dose rates were determined from the radionuclide concentrations in the sediment subsamples, which were analysed by high-resolution gamma spectrometry (Murray et al., 1987). Independent checks on calibration were performed using various standards from the U.S. National Bureau of Standards, and from IAEA intercomparisons. The total uncertainties associated with the calculated burial ages incorporate those calculated for $D_b$ using the minimum age model, those assigned to the long-term water content of each sample, and the systematic uncertainties associated with conversions from activity-concentration data to dose rates, the absolute calibration of activity-concentration measurements, the calibration of laboratory beta sources, and the determination of beta-dose attenuation factors.

5. Results and discussion

5.1. Dose rates

The lithogenic radionuclide concentrations for the samples are summarised in Table 2. These measurements were made on dried and powdered samples. Dry dose rates were calculated using the conversion factors of Stokes et al. (2003). The water contents measured for the samples that were collected adjacent to the OSL samples are reported in Table 3. These values have been used to adjust the dry dose rates, following Aitken (1985). We have assumed that the measured water contents, which were assigned uncertainties of ± 5%, are representative of those pertaining to the full period of sample burial. The cosmic-ray dose rates were calculated from Prescott and Hutton (1994). Beta-attenuation factors were taken from

<table>
<thead>
<tr>
<th>Sample name</th>
<th>$^{238}$U</th>
<th>S.E.</th>
<th>$^{226}$Ra</th>
<th>S.E.</th>
<th>$^{210}$Pb</th>
<th>S.E.</th>
<th>$^{232}$Th</th>
<th>S.E.</th>
<th>$^{40}$K</th>
<th>S.E.</th>
</tr>
</thead>
<tbody>
<tr>
<td>NR99008</td>
<td>31</td>
<td>5</td>
<td>17.9</td>
<td>0.4</td>
<td>17</td>
<td>3</td>
<td>29.1</td>
<td>0.7</td>
<td>782</td>
<td>15</td>
</tr>
<tr>
<td>WK96008</td>
<td>50</td>
<td>3</td>
<td>49.5</td>
<td>0.8</td>
<td>39</td>
<td>4</td>
<td>70.6</td>
<td>0.8</td>
<td>608</td>
<td>11</td>
</tr>
<tr>
<td>NU2_1</td>
<td>31.1</td>
<td>1.4</td>
<td>31.4</td>
<td>0.3</td>
<td>26.1</td>
<td>1.4</td>
<td>43.5</td>
<td>0.5</td>
<td>562</td>
<td>6</td>
</tr>
<tr>
<td>NU2_4</td>
<td>27</td>
<td>2</td>
<td>22.6</td>
<td>0.3</td>
<td>20.8</td>
<td>1.8</td>
<td>32.3</td>
<td>0.3</td>
<td>730</td>
<td>8</td>
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<tr>
<td>GE96001c</td>
<td>48</td>
<td>6</td>
<td>45.8</td>
<td>1.5</td>
<td>52</td>
<td>5</td>
<td>73.3</td>
<td>1.2</td>
<td>590</td>
<td>19</td>
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<tr>
<td>SM15</td>
<td>27</td>
<td>14</td>
<td>23.0</td>
<td>1.0</td>
<td>–</td>
<td>–</td>
<td>57.8</td>
<td>1.1</td>
<td>621</td>
<td>16</td>
</tr>
<tr>
<td>NWJ 16 cm</td>
<td>22</td>
<td>6</td>
<td>25.8</td>
<td>1.6</td>
<td>22</td>
<td>7</td>
<td>7.2</td>
<td>0.2</td>
<td>147</td>
<td>13</td>
</tr>
<tr>
<td>NWJ 68 cm</td>
<td>18</td>
<td>5</td>
<td>7.5</td>
<td>1.0</td>
<td>6</td>
<td>3</td>
<td>10.6</td>
<td>0.7</td>
<td>248</td>
<td>10</td>
</tr>
<tr>
<td>JK 46 cm</td>
<td>12</td>
<td>7</td>
<td>11.9</td>
<td>1.3</td>
<td>12</td>
<td>2</td>
<td>26.6</td>
<td>1.2</td>
<td>494</td>
<td>26</td>
</tr>
<tr>
<td>HRS 60</td>
<td>16.3</td>
<td>0.7</td>
<td>13.7</td>
<td>0.2</td>
<td>10.6</td>
<td>1.1</td>
<td>32.8</td>
<td>0.3</td>
<td>560</td>
<td>7</td>
</tr>
</tbody>
</table>

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*a* The data used to calculate the dose rate for sample S2-1 are reported in Spooner et al. (2001) and are not repeated here.

*b* For the dose rate calculations, $^{226}$Ra activity concentrations are assumed to be supported by $^{230}$Th activity concentrations.

*c* Collected from the same depth interval as OSL samples GE96004 (11.5 cm) and GE96005 (12.5 cm) and used to estimate the beta and gamma dose rates for both samples.
Mejdahl (1979), and the effective internal alpha dose rate (applied to all samples) has been estimated using an alpha-efficiency $a$ value of $0.04 \pm 0.02$ (as measured previously for quartz grains from southeastern Australia; e.g., Bowler et al., 2003). The calculated total dose rates ($D_t$), together with their total (random and systematic) uncertainties, are listed in Table 3.

5.2. OSL data

The single-grain $D_e$ estimates for sample NR99008 determined using the standard SAR protocol are displayed in a radial plot in Fig. 2A. The measured $D_e$ (in Gy) for a grain can be read by tracing a line from the $y$-axis origin through the point until the line intersects the radial axis (log scale) on the right-hand side. The corresponding standard error for this estimate can be read by extending a line vertically to intersect the $x$-axis. The $x$-axis has two scales: one plots the relative standard error of the $D_e$ estimate (in %) and the other (‘Precision’) plots the reciprocal standard error. Therefore, values with the highest precisions and the smallest relative errors plot closest to the radial axis on the right of the diagram, and the least precise estimates plot furthest to the left. Galbraith et al. (1999) provide further details, and a worked illustration, of how radial plots may be used to display OSL data. The shaded region in the plot indicates those $D_e$ values that yield optical ages consistent, at the $1\sigma$ confidence interval, with the calibrated $^{14}$C age of a small log collected from 10 cm below the OSL sample. At least two populations of grains are evident in Fig. 2A: a large population of grains with a $D_e$ value consistent with the $^{14}$C age obtained from the underlying log of wood and a small population of grains with a $D_e$ value centered at $3.2 \pm 0.2$ Gy (estimated using the minimum age model). The latter population of grains yields an optical age of $990 \pm 100$ years, which is less than half that of the $^{14}$C age of the log. These grains are unlikely to have intruded the host deposit after deposition by mixing processes, as single-grain optical dating of three sediment samples collected from stratigraphically higher in the same exposure as sample NR99008 indicates that this alluvial sequence accumulated rapidly (Eriksson et al., submitted for publication: sample NR99006, depth 6.60 m, age $3150 \pm 300$ years; sample NR99009, depth 1.15 m, age $1730 \pm 300$ years; and sample NR99007, depth 0.80 m, age $1830 \pm 160$ years). However, the difference between the optical age obtained from the low-$D_e$ population and the $^{14}$C age might be

![Image](https://example.com/image.png)

**Table 3** Mode of transport, sampling depths, water contents (% of dry weight), dose rates ($D_t$), estimates of dose over-dispersion ($\sigma^2$), calculated mean ages, burial (minimum age model) dose estimates ($D_b$), and calculated burial ages.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Mode of transport</th>
<th>Depth (cm)</th>
<th>Water (%)</th>
<th>$D_t$ (mg Gy/year)</th>
<th>$\sigma^2$ (%)</th>
<th>$D_e$ (Gy)</th>
<th>Mean age (years)</th>
<th>$D_b$ (Gy)</th>
<th>Burial age (years)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NR99008</td>
<td>Fluvial</td>
<td>690</td>
<td>15.0</td>
<td>$3.23 \pm 0.23$</td>
<td>23</td>
<td>$11.9 \pm 0.5$</td>
<td>$3700 \pm 330$</td>
<td>$9.3 \pm 1.0$</td>
<td>$2900 \pm 370$</td>
</tr>
<tr>
<td>S2-1</td>
<td>Aeolian</td>
<td>430</td>
<td>9.3</td>
<td>$3.51 \pm 0.08$</td>
<td>24</td>
<td>$16.10 \pm 0.25$</td>
<td>$4590 \pm 350$</td>
<td>$14.0 \pm 0.7$</td>
<td>$3990 \pm 240$</td>
</tr>
<tr>
<td>WK96008</td>
<td>Fluvial</td>
<td>255</td>
<td>4.0</td>
<td>$3.50 \pm 0.30$</td>
<td>51</td>
<td>$8.0 \pm 0.3$</td>
<td>$2300 \pm 200$</td>
<td>$5.4 \pm 1.0$</td>
<td>$1550 \pm 300$</td>
</tr>
<tr>
<td>NWJ 68cm</td>
<td>Fluvial</td>
<td>80</td>
<td>5.6</td>
<td>$3.15 \pm 0.24$</td>
<td>1</td>
<td>$0.33 \pm 0.03$</td>
<td>$105 \pm 15$</td>
<td>$0.34 \pm 0.03$</td>
<td>$110 \pm 15$</td>
</tr>
<tr>
<td>NWJ 16cm</td>
<td>Fluvial</td>
<td>70</td>
<td>6.6</td>
<td>$3.34 \pm 0.23$</td>
<td>85</td>
<td>$0.85 \pm 0.20$</td>
<td>$255 \pm 65$</td>
<td>$0.46 \pm 0.05$</td>
<td>$140 \pm 20$</td>
</tr>
<tr>
<td>GE96004</td>
<td>Fluvial</td>
<td>11.5</td>
<td>20.2</td>
<td>$3.63 \pm 0.30$</td>
<td>55</td>
<td>$1.75 \pm 0.16$</td>
<td>$480 \pm 60$</td>
<td>$0.14 \pm 0.05$</td>
<td>$40 \pm 15$</td>
</tr>
<tr>
<td>GE96005</td>
<td>Fluvial</td>
<td>12.5</td>
<td>20.2</td>
<td>$3.63 \pm 0.30$</td>
<td>47</td>
<td>$1.49 \pm 0.20$</td>
<td>$410 \pm 60$</td>
<td>$0.25 \pm 0.05$</td>
<td>$70 \pm 15$</td>
</tr>
<tr>
<td>SM15</td>
<td>Aeolian</td>
<td>15</td>
<td>42.0</td>
<td>$2.59 \pm 0.17$</td>
<td>42</td>
<td>$0.34 \pm 0.06$</td>
<td>$130 \pm 25$</td>
<td>$0.10 \pm 0.10$</td>
<td>$40 \pm 40$</td>
</tr>
<tr>
<td>NWJ 16cm</td>
<td>Aeolian</td>
<td>16</td>
<td>40.0</td>
<td>$0.82 \pm 0.07$</td>
<td>26</td>
<td>$1.68 \pm 0.05$</td>
<td>$2050 \pm 190$</td>
<td>$1.30 \pm 0.13$</td>
<td>$1590 \pm 215$</td>
</tr>
<tr>
<td>NWJ 68cm</td>
<td>Aeolian</td>
<td>68</td>
<td>51.1</td>
<td>$0.88 \pm 0.05$</td>
<td>25</td>
<td>$6.20 \pm 0.20$</td>
<td>$7050 \pm 520$</td>
<td>$4.4 \pm 0.3$</td>
<td>$5000 \pm 470$</td>
</tr>
<tr>
<td>JK 46cm</td>
<td>Aeolian</td>
<td>46</td>
<td>49.2</td>
<td>$1.63 \pm 0.11$</td>
<td>21</td>
<td>$1.04 \pm 0.06$</td>
<td>$640 \pm 60$</td>
<td>$1.0 \pm 0.2$</td>
<td>$615 \pm 130$</td>
</tr>
<tr>
<td>HR5_60</td>
<td>Marine</td>
<td>60</td>
<td>12.0</td>
<td>$2.50 \pm 0.17$</td>
<td>27</td>
<td>$17.8 \pm 0.5$</td>
<td>$7100 \pm 600$</td>
<td>$10.8 \pm 0.5$</td>
<td>$4300 \pm 380$</td>
</tr>
</tbody>
</table>

$^a$ The relative standard deviation of the single-grain $D_e$ distribution after taking into account the measurement uncertainty for each grain. These estimates of over-dispersion were obtained using the central age model of Galbraith et al. (1999). If measurement uncertainties were the only source of variation in $D_e$, then $\sigma$ would be zero.

$^b$ Dose rate as calculated and reported by Spooner et al. (2001).
Fig. 2. Radial plots of single-grain $D_e$ estimates (Gy) for sample NR99008 determined using (A) the standard SAR protocol and (B) the modified SAR protocol incorporating IR stimulation. The shaded region indicates data that are consistent with the 1σ calibrated $^{14}$C age range. The dashed line in plot (A) indicates the $D_b$ of 3.2 Gy obtained using the minimum age model.
explicable in terms of the residence time of wood in the natural environment. For example, \( ^{14}C \) ages on wood fragments have been reported to differ by 1500 or more years from the age of deposition of the sediments in which they were found (Blong and Gillespie, 1978). But we decided to investigate the possibility that the optical age had been underestimated as a result of laboratory procedures.

Other samples from this site had shown a response to IR stimulation following a beta dose given in the laboratory, which we took to be indicative of residual feldspar grains because it is generally thought that the OSL source traps in quartz are not stimulated by IR radiation at room temperature (Aitken, 1998), or only weakly so (e.g., Godfrey-Smith and Cada, 1996). This IR response persisted after repeated treatments with fluorosilicic acid, which will dissolve all exposed feldspar grains and leave the quartz grains intact. The persistence of an IR signal suggested that we may be dealing with feldspar grains, or some other IR-sensitive minerals or impurity complexes, which were internal to the quartz grains and, hence, protected from the acid. Such inclusions formed the basis of the dating study of a stranded beach-dune sequence in southeastern Australia by Huntley et al. (1993).

We investigated the IR response of grains from the two \( D_e \) populations shown in Fig. 2A. The grains were first bleached in sunlight and then given a 6.5 Gy dose (as a surrogate natural dose), which was followed by OSL stimulation using the green light from the laser. The same grains were then given another 6.5 Gy dose, and exposed to IR radiation for 40 s at 125 °C before green light stimulation. Examples of the resultant OSL decay curves for a grain from the higher \( D_e \) population and for a grain from the low-\( D_e \) population are shown in Fig. 3A and B, respectively. For the grain from the higher \( D_e \) population, the decay curves induced by green light stimulation with and without prior exposure to IR radiation are indistinguishable; the other grains from this population behaved in like manner. By contrast, the post-IR decay curve obtained during green light stimulation of the grain from the low-\( D_e \) population differed markedly from those measured prior to the IR exposure (see Fig. 3B). The post-IR OSL decay curve was almost completely eroded, and the other low-\( D_e \) grains behaved similarly. This indicates that the low-\( D_e \) population consists of IR-sensitive grains.

As a result of this finding, we modified the standard SAR protocol by introducing IR stimulation at 125 °C before each OSL stimulation using green light, and then analysed a fresh set of grains from sample NR99008. The resulting single-grain \( D_e \) estimates are shown in Fig. 2B. The low-\( D_e \) population evident in Fig. 2A is absent in the data set obtained using the modified SAR protocol, and we conclude that IR-sensitive grains were solely responsible for the low \( D_e \) values. Similar results were obtained for other samples from this site (Olley, unpublished data). The burial dose estimated by applying the minimum age model to the data in Fig. 2B is 9.3 ± 1.0 Gy, which yields an optical age of 2900 ± 370 years. This age is stratigraphically consistent with the calibrated \( ^{14}C \) age for the log from the underlying deposit (1σ age range: 3340–3470 years), bearing in mind that the OSL sample is located 10 cm above the log, and with the single-grain optical ages for the three overlying sediment samples (Eriksson et al., submitted for publication).

We then considered whether IR-sensitive grains were responsible for the problems reported by Spooner et al. (2001) for single-grain dating of sample S2-1. Fig. 4A is a plot of the measured \( D_e \) values presented by Spooner et al. (2001) for aliquots composed of ~2000 grains (‘7 mm’ aliquots), ~50 grains (‘1 mm’ aliquots), ~10 grains (‘0.38 mm’ aliquots) and individual grains of sample S2-1. These data were collected on a similar Risø reader to that used here, but using blue-plus-green light (420–560 nm) from a filtered halogen lamp for optical stimulation, with the ultraviolet OSL emissions being passed through two 2 mm-thick Hoya U-340 filters. Quartz grains of 180–212 μm diameter were mounted on stainless-steel discs using silicone oil, and the \( D_e \) values were obtained using the SAR protocol described by Roberts et al. (1998). This involved the use of preheats of 240 °C for 10 s for the natural and regenerative doses, cut-heats of 160 °C after each test dose, and illumination of the samples for 100 s at 125 °C. The OSL emissions were integrated over the first 20 s of illumination, using the final 20 s integral as background. The reported \( D_e \) uncertainties are based on counting statistics, and incorporate the calibration uncertainty associated with the laboratory beta source.
The shaded region in Fig. 4A indicates those \( D_e \) values that yield optical ages consistent with the 1σ calibrated \( ^{14}C \) age range (3630–4070 years). Many of the data points plot below the minimum expected \( D_e \) of 12.7 Gy. The equivalent doses obtained from the 10-grain aliquots and from the single grains range from <6 to >30 Gy. Spooner et al. (2001) noted that the multiple-grain aliquots and single grains that had \( D_e \) values of less than ~10 Gy exhibited a significantly slower initial rate of OSL decay in the test and regenerative dose cycles than did aliquots and grains with \( D_e \) values of greater than 10 Gy.

The single-grain \( D_e \) estimates for sample S2-1 obtained using a green laser and the standard and modified SAR protocols are presented in Fig. 4B. The shaded region again brackets the OSL data that are consistent with the \( ^{14}C \) age, using the sample ‘bulk’ dose rate for all grains. The data collected using the standard SAR protocol (displayed as open circles) show a similar spread in \( D_e \) to those reported by Spooner et al. (2001), ranging from 4.6 ± 1.4 to 30 ± 7 Gy. A number of high-precision \( D_e \) estimates fall below the \( D_e \) range of 12.7–14.3 Gy required to match the calibrated \( ^{14}C \) age. To take account of the analytical uncertainties associated with each \( D_e \) value, we have used the minimum age model to determine the \( D_e \) value associated with the population of grains that were bleached most completely at the time of deposition and that should most closely approximate the true burial dose. The resultant estimate of \( D_b \) is 11.6 ± 0.7 Gy, which is too small because of the inclusion of low-\( D_e \) grains that have an IR-sensitive signal.

By contrast, the data collected using the modified SAR protocol (Fig. 4B, closed circles), with IR
stimulation prior to illumination by green laser light, lack the high-precision low-$D_e$ values evident in the previous data set. The minimum age model gives a $D_b$ estimate of 14.0 ± 0.7 Gy, which falls squarely within the expected range. These results suggest that IR-sensitive grains were responsible for the difference between the $^{14}$C age for sample S2-1 and the optical age determined by Spooner et al. (2001) from single grains and from small aliquots of quartz. They also illustrate that the modified SAR protocol, incorporating IR stimulation prior to green light stimulation, deals effectively with contamination by IR-sensitive grains.

We considered two possible explanations for the low-$D_e$ values measured from IR-sensitive grains:

1. the IR-sensitive grains are, or contain, feldspar, so the natural OSL signal may have decayed during the period of sample burial due to ‘anomalous fading’, a phenomenon that has been shown to afflict many feldspars (e.g., Huntley and Lamothe, 2001). Clarke and Rendell (1997) have also
Fig. 5. Radial plot of single-grain $D_e$ estimates (Gy) for sample WK96008 determined using the modified SAR protocol. The shaded region indicates data that are consistent with the $D_b$ estimated using the minimum age model.

Fig. 6. Radial plots of single-grain $D_e$ estimates (Gy) for fluvially deposited samples (A) NU2_1 and (B) NU2_4. The dashed lines indicate the $D_b$ values estimated using the minimum age model.
shown that the IR-stimulated ultraviolet emissions from sodium feldspars, in particular, are thermally unstable at laboratory timescales. In this study, however, grains were preheated prior to optical stimulation and this should reduce the magnitude of any age shortfall due to fading of the ultraviolet emissions from feldspars (Clarke and Rendell, 1997). For the young samples analysed here, it would also require much higher rates of fading than those reported by Huntley and Lamothe (1997) to account for the age underestimates associated with the low-$D_e$ values obtained from single grains.

(2) The samples examined in this study (and by Spooner et al., 2001), were prepared under dim ‘red’ (>590 nm) illumination using Lee 106 filters. Red and longer wavelengths are suitable for uncontaminated quartz, but IR-sensitive contaminants in the separated quartz grains could have been partially or completely bleached by this light source, resulting in the depletion of the natural IR-stimulated luminescence (IRSL) signal (e.g., Lamothe, 1995). Potential contaminants include feldspars, other IR-sensitive minerals and, as suggested by Godfrey-Smith and Cada (1996), aluminium impurity complexes in the crystal lattice of natural quartz. Affected grains would yield $D_e$ values that were lower than the true burial dose when measured using the standard SAR protocol. We consider this explanation to be the more likely of the pair.

Our results and modified SAR procedure are consistent with previous recommendations to employ IR stimulation to identify and/or deplete the OSL signal from feldspars (and other IR-sensitive source traps) that would otherwise be stimulated by green and blue light. For example, Jain and Singhvi (2001) proposed that IR stimulation for 300 s at 220 °C was most effective at removing the feldspar-related OSL signal (the latter heat treatment was also shown to remove the thermally unstable IR-stimulated ultraviolet emission from feldspars; Clarke and Rendell, 1997), and Duller (2003) suggested that an IR stimulation step be added to the end of the standard SAR protocol to identify and reject contaminated samples.

Fig. 7. Single-grain $D_e$ estimates (Gy) plotted against OSL counts in response to a 6.5 Gy test dose for floodplain sediment samples (A) GE96004 (open circles: 200 °C preheat; closed circles: 240 °C preheat) and (B) GE96005. The solid horizontal lines denote the $D_b$ values estimated using the minimum age model.
quartz grains. By incorporating IR stimulation in our modified SAR protocol, we are extending the approach originally proposed for mixed-mineral fine grains (Banerjee et al., 2001; Roberts and Wintle, 2001) to sand-sized quartz grains from a variety of geomorphic settings. We note, however, that previous studies have suggested that the OSL and IRSL signals from feldspars do not originate from identical source traps (see review by Duller, 1997), so the modified SAR protocol is likely to reduce, rather than eliminate, any adverse effects from IR-sensitive grains.

In the following sections, we illustrate that use of the modified SAR protocol improves the accuracy of the optical ages obtained for a range of Holocene fluvial, aeolian and marine sediments, for which the age of deposition is tightly constrained by other methods. Table 3 lists, for each sample, the mode of sediment transport, the depth from which it was collected, the field water content (expressed as % of dry weight), the total dose rate ($D_r$), the relative standard deviation of the $D_e$ distribution after allowing for statistical estimation error ($\sigma$), the weighted mean dose estimate obtained using the central age model.

![Graphs](image-url)
(\(D_c\)), the calculated mean age, the burial dose estimate obtained using the minimum age model (\(D_b\)), and the calculated burial age.

5.3. Fluvial sediments

Sample WK96008 was collected from a floodplain in the upper part of the Wangrah Creek catchment and sediment grains would have been transported less than 5 km prior to deposition. Measured equivalent doses for single grains from this sample are shown in a radial plot in Fig. 5. The spread in measured \(D_e\) values (3.7 ± 1.0 to 47 ± 6 Gy) indicates that the sample was neither fully nor uniformly bleached prior to burial. The over-dispersion parameter, \(\sigma\), represents the relative standard deviation of the \(D_e\) distribution after allowing for measurement uncertainties (Galbraith et al., 1999). If the latter were the only source of variation in \(D_e\), then \(\sigma\) would be zero. By contrast, sample WK96008 has a \(\sigma\) value of 51% (Table 3), which indicates a substantial spread in \(D_e\) above and beyond that associated with statistical estimation error. The weighted mean (central age model estimate) of all measured \(D_e\) values is 8.0 ± 0.3 Gy, which corresponds to a calculated age of 2300 ± 200 years. This is significantly older than the calibrated \(^{14}\)C age of 1530 years (1\(\sigma\) age range: 1420–1590 years). By contrast, the minimum age model yields an estimate of \(D_b\) of 5.4 ± 1.0 Gy. This produces an optical age of 1550 ± 300 years, which is consistent with the \(^{14}\)C age.

Single-grain \(D_e\) data for samples NU2_1 and NU2_4 are presented in Fig. 6A and B, respectively. Both samples were collected about 100 km from the headwaters of the Numeralla River catchment, and are known from documentary records to have been deposited between 160 and 120 years ago. Using the minimum age model, their burial doses are estimated to be 0.34 ± 0.03 Gy (NU2_1) and 0.46 ± 0.05 Gy (NU2_4). These yield ages of 110 ± 15 and 140 ± 20 years, respectively, which are consistent with the expected depositional ages. The weighted mean \(D_e\) (estimated using the central age model) for sample NU2_1 (0.33 ± 0.03 Gy) is consistent with the burial dose estimated using the minimum age model and gives an age of 105 ± 15 years. The agreement between the expected burial age and the optical ages determined using the minimum age model and the central age model indicates that this fluvial sample was well bleached at the time of deposition. In contrast, the weighted mean \(D_e\) value for sample NU2_4 (0.85 ± 0.20 Gy) yields an age of 255 ± 65 years, ~ 50% greater than expected. For this sample, only the grains with the lowest \(D_e\) values produce estimates of \(D_b\) in agreement with the true (known) burial age. The differential bleaching histories of these two samples are reflected also in their \(\sigma\) values: 1% for sample NU2_1 as compared to 85% for sample NU2_4 (Table 3). These estimates of over-dispersion indicate that measurement uncertainties account for nearly all of the spread in \(D_e\) for sample NU2_1, whereas additional sources of variation are required to explain the substantial spread in \(D_e\) for sample NU2_4.

Samples GE96004 and GE96005 were extracted from a sediment core collected more than 1000 km...
from the headwaters of the Murray River catchment. Preheats of 200 and 240 °C for 10 s were applied to grains from sample GE96004. The resulting $D_e$ distributions were indistinguishable, so the data were combined for subsequent analysis. Despite the potentially long transport distance of these two samples, which might be expected to facilitate bleaching of the grains to acceptably low residual $D_e$ values (e.g., Stokes et al., 2001), the spread in measured equivalent doses ($\sigma$ value of 42%) indicates that not all of the grains in either sample were fully bleached prior to burial; the corresponding $\sigma$ values are 55% (GE96004) and 47% (GE96005), indicating that the observed $D_e$ dispersions cannot be explained solely in terms of single-grain measurement uncertainties. The burial doses estimated using the minimum age model are $0.14 \pm 0.05$ Gy (GE96004) and $0.25 \pm 0.05$ Gy (GE96005), which correspond to burial ages of $40 \pm 15$ and $70 \pm 15$ years, respectively. These optical ages are consistent with their expected depositional ages of ~55 and 55–125 years. In contrast, the optical ages determined using the weighted mean $D_e$ values (1.75 ± 0.16 and 1.49 ± 0.20 Gy, respectively) overestimate the burial ages by up to a factor of 10.

5.4. Aeolian sediments

As the quartz grains analysed from Lake St. Mary and Jacka Lake were transported on to the lake beds by wind, rather than by water, we refer to them as aeolian, rather than lacustrine, sediments.

The single-grain $D_e$ data for the sediment sample from Lake St. Mary (SM15) are presented in Fig. 8A. These data were collected using either a 200 °C preheat (open circles) or a 240 °C preheat (closed circles), but the data sets are indistinguishable so they have been combined for subsequent analysis. The spread in measured equivalent doses ($\sigma$ value of 42%) indicates that some of the grains were incompletely bleached prior to burial. The $D_b$ for sample SM15 is estimated to be $0.10 \pm 0.10$ Gy (using the minimum age model), which gives a burial age of 40 ± 40 years. This is consistent with the $^{14}$C and $^{137}$Cs age estimates of <40 years. The optical age determined using the weighted mean $D_e$ overestimates the burial age by >200% (see Table 3).

The spread in single-grain $D_e$ data for samples NWJ 16 cm (Fig. 8B) and NWJ 68 cm (Fig. 8C) suggests that both samples were heterogeneously bleached at the time of deposition. The minimum age model estimates of the burial doses ($1.30 \pm 0.13$ and $4.4 \pm 0.3$ Gy, respectively) give ages of 1590 ± 215 years (NWJ 16 cm) and 5000 ± 470 years (NWJ 68 cm). Sample NWJ 16 cm is bracketed by 1σ calibrated $^{14}$C age ranges of 950–1170 and 3630–3870 years, and sample NWJ 68 cm is bracketed by $^{14}$C age ranges of 3630–3870 and 6000–6300 years. In both cases, the optical age obtained from the $D_b$ value estimated using the minimum age model is consistent with the $^{14}$C age constraints. The weighted mean optical age for sample NWJ 16 cm (2050 ± 190 years) also falls within the $^{14}$C age range, but, given the spread in measured $D_e$ values (Fig. 8B), the minimum age model estimate is likely to be closer to the true depositional age.

Fig. 10. Single-grain $D_e$ estimates (Gy) plotted against OSL counts in response to a 6.5 Gy test dose for beach ridge sample HR5..60. The solid horizontal line denotes the $D_b$ estimated using the minimum age model and the shaded region indicates data that are consistent with the 1σ calibrated $^{14}$C age range.
age. For sample NWJ 68 cm, the weighted mean optical age of 7050 ± 520 years is significantly older than the calibrated age range for the underlying 14C sample. At 26% (NWJ 16 cm) and 25% (NWJ 68 cm), the \( \sigma \) values for these two samples are much smaller than those obtained for most of the fluvial samples discussed above, indicating less over-dispersed \( D_e \) distributions. This result accords with expectations that aeolian sediments are apt to be bleached more completely than fluvial sediments at the time of deposition.

Sample JK 46 cm from Jacka Lake is bracketed by calibrated 14C age ranges of 315–625 and 1090–1390 years. The single-grain \( D_e \) values for this sample are presented in Fig. 9. In general, the data are much less dispersed than those collected for NWJ 16 cm and NWJ 68 cm, but two of the 44 points (\( D_e \) values of 4.4 ± 1.7 and 0.21 ± 0.07 Gy) fall well outside the shaded region. The inclusion of these two values yields an estimate of over-dispersion of 21%, whereas their omission reduces \( \sigma \) to zero. The latter indicates that measurement uncertainties can account for all of the spread in \( D_e \) and that the single-grain equivalent doses are consistent with a common \( D_e \). For sample JK 46 cm, therefore, the spread in \( D_e \) values at the time of deposition was small compared to the dose exposure since burial. The \( D_b \) estimated from all 44 \( D_e \) values using the minimum age model is 1.0 ± 0.2 Gy, which gives an optical age (615 ± 130 years) consistent with the 14C ages above and below the OSL sample. As the data are mostly consistent with a single \( D_e \) distribution, the weighted mean provides a valid, and more precise, measure of the burial dose, in this case 1.04 ± 0.06 Gy (using all data). This estimate of \( D_b \) corresponds to a burial age of 640 ± 60 years, which is indistinguishable from the minimum age model estimate of the burial age and consistent also with the bracketing 14C ages.

5.5. Marine sediments

Sample HR5_60 was collected from a relict beach ridge on the Herbert River floodplain in far northern Queensland. Along this stretch of coastline, beach ridges are formed as the result of storm surges (e.g., Nott and Hayne, 2001). Charcoal fragments collected from an in situ buried hearth in the same sediment horizon as sample HR5_60 yielded a 1σ calibrated 14C age range of 3640–4420 years. The calculated total dose rate for this sample is 2.51 ± 0.17 mGy/year, so the expected burial dose is between 9.1 and 11.1 Gy. The single-grain \( D_e \) values for this sample are displayed in Fig. 10, and the shaded region denotes \( D_e \) values consistent with the expected \( D_b \) of 9.1–11.1 Gy. Most of the measured \( D_e \) values are greater than the expected \( D_b \), and the \( D_e \) distribution is significantly over-dispersed (\( \sigma \) value of 27%), indicating that a majority of the grains were insufficiently bleached at the time of deposition. However, a small population of grains does yield \( D_e \) values consistent with the expected \( D_b \), and the minimum age model predicts a burial dose of 10.8 ± 0.5 Gy. This corresponds to an age of 4300 ± 380 years, which matches the independent 14C age control.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>( L_o ) (%)</th>
<th>Age(10) (years)</th>
<th>( D_{10} ) (Gy)</th>
<th>Age(100) (years)</th>
<th>( D_{100} ) (Gy)</th>
<th>Age(100) (years)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NR99008</td>
<td>8</td>
<td>&lt;3470</td>
<td>9.6 ± 0.6</td>
<td>2970 ± 320</td>
<td>10.7 ± 1.0</td>
<td>3300 ± 420</td>
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<tr>
<td>S2-1</td>
<td>28</td>
<td>3630–4070</td>
<td>14.9 ± 0.5</td>
<td>4250 ± 370</td>
<td>15.8 ± 0.2</td>
<td>4500 ± 370</td>
</tr>
<tr>
<td>WK96008</td>
<td>77</td>
<td>1420–1590</td>
<td>5.7 ± 0.3</td>
<td>1640 ± 160</td>
<td>7.9 ± 0.2</td>
<td>2300 ± 200</td>
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<tr>
<td>NU2_1</td>
<td>19</td>
<td>120–160</td>
<td>0.32 ± 0.05</td>
<td>100 ± 20</td>
<td>0.32 ± 0.04</td>
<td>100 ± 20</td>
</tr>
<tr>
<td>NU2_4</td>
<td>25</td>
<td>120–160</td>
<td>0.42 ± 0.13</td>
<td>125 ± 40</td>
<td>0.46 ± 0.10</td>
<td>140 ± 30</td>
</tr>
<tr>
<td>GE96004</td>
<td>38</td>
<td>~ 55</td>
<td>0.32 ± 0.08</td>
<td>90 ± 25</td>
<td>1.68 ± 0.06</td>
<td>460 ± 45</td>
</tr>
<tr>
<td>GE96005</td>
<td>38</td>
<td>55–125</td>
<td>0.38 ± 0.10</td>
<td>100 ± 30</td>
<td>0.90 ± 0.05</td>
<td>250 ± 25</td>
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<tr>
<td>SM15</td>
<td>33</td>
<td>&lt;40</td>
<td>0.07 ± 0.02</td>
<td>30 ± 10</td>
<td>0.11 ± 0.03</td>
<td>40 ± 10</td>
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<tr>
<td>NWJ 16 cm</td>
<td>61</td>
<td>950–3870</td>
<td>1.52 ± 0.06</td>
<td>1850 ± 180</td>
<td>1.60 ± 0.03</td>
<td>1950 ± 180</td>
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<tr>
<td>NWJ 68 cm</td>
<td>32</td>
<td>3630–6300</td>
<td>6.8 ± 0.3</td>
<td>7720 ± 610</td>
<td>8.60 ± 0.10</td>
<td>9760 ± 650</td>
</tr>
<tr>
<td>JK 46 cm</td>
<td>16</td>
<td>315–1390</td>
<td>1.00 ± 0.05</td>
<td>615 ± 50</td>
<td>1.00 ± 0.06</td>
<td>615 ± 60</td>
</tr>
<tr>
<td>HR5_60</td>
<td>92</td>
<td>3640–4420</td>
<td>14.2 ± 0.5</td>
<td>5660 ± 470</td>
<td>16.9 ± 0.25</td>
<td>6730 ± 510</td>
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</tbody>
</table>
Hence, despite the very wide spread in measured $D_e$ values for this sample, the true burial dose was successfully determined using a combination of the single-grain $D_e$ measurements and the minimum age model.

By contrast, several other methods advocated recently for determining the $D_b$ of partially bleached deposits produce substantial age underestimates: 1880 \pm 240 years using the technique of Zhang et al. (2003), 2150 \pm 290 years using the method of Fuchs and Lang (2001), and 2500 \pm 300 years using the procedure of Olley et al. (1998). The latter approach, however, cannot strictly be applied in this instance because, unlike Olley et al. (1998) who used the $D_e$ distribution of a modern sample to define an appropriate upper $D_e$ limit for $D_b$ determination, no such data are available for this site; we have, instead, simply calculated the $D_b$ from the lowest 5% of $D_e$ values. Another method proposed recently to estimate the $D_b$ of partially bleached deposits (Lepper and McKeever, 2002) gives an age that is too old, with an uncertainty that is too large to provide useful age resolution within the Holocene (5200 \pm 3200 years).

In contrast to these other approaches for estimating $D_b$, the minimum age model takes into account the statistical properties of the process that generated the $D_e$ values. The model estimate of $D_b$ and the associated uncertainty are founded on well-established statistical principles, and the model has been thoroughly tested in simulations (e.g., van der Touw et al., 1997). In this paper, we have demonstrated that, in addition to being statistically rigorous, the minimum age model provides accurate estimates of $D_b$, with useful precision, as shown by the concordance between the burial ages and the known depositional ages of 12 Holocene samples.

5.6. Synthetic aliquots

For each of these 12 samples, we have simulated the $D_e$ distribution obtained for forty-eight 10-grain and 100-grain aliquots using the single-grain data. Grains were selected at random from the measured population, which included grains that did not emit any detectable OSL. The proportion of luminescent grains in the samples ranged from 8% to 92% (Table 4). The $D_e$ value for each synthetic aliquot was calculated by summing the (natural) test dose OSL intensities (a measure of intrinsic grain brightness) for all grains on an aliquot and then weighting each of the single-grain $D_e$ values by their fractional contribution to this sum. The $D_b$ for each sample was then estimated from the 48 independent $D_e$ values generated for both the 10-grain and 100-grain aliquot data sets, using the minimum age model. The results are reported in Table 4, together with the calculated burial ages. For all but three of the samples (GE96004 (fluvial), NWJ 68 cm (aeolian), and HR5...60 (marine)), the minimum age model estimates for the 10-grain aliquots are consistent with the known burial ages. Samples GE96004, NWJ 68 cm and HR5...60 are the most poorly bleached of the samples examined in this study, with fewer than 10% of the luminescent grains having $D_e$ values consistent with the expected burial doses. For these three samples, the 10-grain aliquots give rise to burial ages that are too old by approximately 125%, 54% and 32%, respectively; the true burial ages can only be determined accurately from the single-grain data. This simulation illustrates that while ‘small’ aliquots may often yield reliable estimates of the burial age, even for water-lain sediments, there are a sufficient number of instances, including aeolian samples (such as NWJ 68 cm), that caution should be exercised when dating Holocene sediments using multiple-grain aliquots.

This cautionary advice is amplified by the results of the 100-grain aliquot simulation. Estimation of the $D_b$ using these data and the minimum age model yields age overestimates for six of the 12 samples, with the burial age for sample GE96004 being too old by almost an order-of-magnitude. These findings reiterate those of Olley et al. (1999), who found that, for heterogeneously bleached sediments, the probability of an optical age exceeding the true age of deposition increases with the number of grains on an aliquot.

6. Summary and recommendations

The results from samples NR99008 and S2-1 show that the presence of IR-sensitive grains can give rise to spurious low-$D_e$ populations, which will result in the significant underestimation of the burial dose and, hence, the depositional age. The new single-grain data
presented here for sample S2-1 show that the presence of IR-sensitive grains was responsible for the apparent disagreement between the results of Olley et al. (1999) and Spooner et al. (2001). For sample NR99008, repeated treatment with fluorosilicic acid, which dissolves exposed feldspars but leaves quartz grains untouched, did not remove the mineral inclusions or impurity complexes responsible for the IRSL signal. Unfortunately, therefore, use of this acid treatment does not guarantee the removal of IR-sensitive grains. However, application of the modified SAR protocol reported here, which incorporates IR stimulation prior to green light stimulation, greatly reduces the malign effects arising from contamination of quartz grains by IR-sensitive inclusions or impurity complexes. Accordingly, we recommend that this modified SAR protocol be used routinely for optical dating of quartz.

For all of the samples examined in this study, an accurate estimate of the burial dose was obtained by applying the minimum age model (Galbraith et al., 1999) to $D_e$ measurements made on single grains of quartz using the modified SAR protocol (see Fig. 11). Only three of twelve samples were sufficiently well bleached that other methods of optical dating would

Fig. 11. Comparisons of single-grain optical ages determined using the minimum age model (open circles) and central age model (closed circles) with independent age estimates (thick horizontal lines) for 12 Holocene samples. Error bars on the optical ages are at one standard error. The depositional environment of each sample is described in detail in Section 3.
have provided a reliable estimate of the true burial age. For the other nine samples examined here, which includes sediments that underwent aeolian transport prior to deposition, use of multiple-grain aliquots or the weighted mean of single-grain $D_e$ measurements gave rise to an overestimate of the burial age. These results show that heterogeneous bleaching of the OSL signal is commonplace in nature, and that aeolian transport is not a sufficient guarantee that the sediment grains will be well bleached at the time of deposition; a similar caveat concerning the bleaching of aeolian samples was expressed by Lian and Huntley (1999).

We conclude that application of the modified SAR protocol to single grains of quartz, using the minimum age model to estimate the burial dose from the lowest $D_e$ population, is the best available means of obtaining reliable burial ages for Holocene sediments from a variety of depositional environments. Following Galbraith et al. (1999), however, we caution that the minimum age model may not be appropriate for samples in which the spread in $D_e$ values is due primarily to factors other than partial bleaching (as may be the case for many Pleistocene sediments), nor for samples that have undergone post-depositional disturbance, resulting in the intrusion of grains from younger strata. In the latter instance, the minimum age model may not yield an accurate estimate of the true burial age of the host deposit. Finally, it would be instructive to combine the ‘dose distribution’ approach advocated here with methods of ‘signal analysis’, which exploit differences in the relative bleaching rates of the most light-sensitive component of quartz OSL (as used in this study) and the less rapidly bleached OSL components (e.g., Bailey et al., 2003; Yoshida et al., 2003). Both strategies offer the prospect to identify fully bleached grains, incompletely bleached grains, and grains that have experienced post-depositional transport in soils and sediments.

Acknowledgements

We thank Jacqui Olley for sample preparation, Chris Leslie for gamma spectrometry analyses, Ian Prosser for the Wangrah Creek radiocarbon age, Christine Kenyon for the Barmah Forest floodplain samples, Lynda Radtke for the radiocarbon ages for the Jacka Lake samples, Gary Caitcheon for comments on an early version of this manuscript, and Heinz Buettikofer for drafting the radial plots. Richard Roberts thanks the Australian Research Council for the support of a Senior Research Fellowship, and Tim Pietsch thanks Gerald Nanson for useful advice and discussions. We also thank Mark Macklin, Andreas Lang and Takashi Oguchi for reviews and comments that improved the manuscript.

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