Optical dating of dune sand from Blombos Cave, South Africa: I—multiple grain data

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Abstract

An aeolian sand unit overlies the Middle Stone Age deposits at Blombos Cave on the southern Cape coast. These deposits contained culturally-important artefacts, including bone tools and pieces of engraved ochre, as well as a large number of worked lithics. The aeolian sand and two other remnants of the sand dune formed against the coastal cliff were dated using optical dating. To determine the dose received since deposition, measurements were made on 5 mg aliquots of purified quartz grains using the single-aliquot regenerative-dose (SAR) protocol. The results of several internal check procedures are reported and at least 15 replicate dose determinations are presented for each sample. Combining these dose values with measurements of the radioactive content of each sample resulted in an age of $69.2 \pm 3.9$ ka for the unit within the cave, and a mean age of $70.1 \pm 1.9$ ka for all three dune samples. This provides a minimum age for the Middle Stone Age material at Blombos Cave.

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Introduction

Optical dating provides a direct means of dating sedimentary units, with the age of last exposure to sunlight being obtained from measurements of the optically stimulated luminescence (OSL) and the radioactive content (Aitken, 1998). It is especially important for sediments that are beyond the range of radiocarbon dating (~40 ka). Thus, optical dating is appropriate for providing chronological information for sediments relating to the Middle Stone Age in southern Africa. Studies of the OSL behaviour of quartz from Australian sand dunes, recently summarized by Wintle and Murray (2000), have led to the development of an improved laboratory procedure for measuring the radiation dose to which grains have been exposed in their environment; this dose is called the equivalent dose ($D_e$). The decay of elements in the uranium and thorium decay chains, and the decay of $^{40}$K, with a minor contribution from cosmic rays, produce radiation in the environment.
resulting in the delivery of an annual dose to the quartz grains.

Several different luminescence-based dating procedures have been applied to Middle Stone Age sediments from sites in southern Africa, e.g. thermoluminescence (TL) and OSL analysis of quartz at White Paintings Rockshelter, Botswana (Feathers, 1997) and at Die Kelders, South Africa (Feathers and Bush, 2000), and TL and OSL analysis of quartz and infrared stimulated luminescence (IRSL) analysis of K-feldspars and polymineral fine grains at Klasies River and Duinefontein, South Africa (Feathers, 2002).

In this paper we have chosen to apply the single aliquot regenerative dose (SAR) protocol of Murray and Wintle (2000). Our choice is based on the excellent agreement between OSL ages determined by SAR and independent ages for about 50 samples reported by Murray and Olley (2002). This data set included sand-sized quartz from Holocene (Murray and Clemmensen, 2001) and Late Glacial (Hilgers et al., 2001) aeolian sediments with radiocarbon control, and silt-sized quartz from a marine core from the Indian Ocean, with a variety of independent age controls back to 120 ka (Stokes et al., 2003). Our study reports results for three samples of dune sand from Blombos, South Africa, where cave sediments have been found to contain lithic tools, shaped bone artefacts and decorated ochre (Henshilwood et al., 2001a,b; Henshilwood et al., 2002).

Site and sample description

Blombos Cave is one of a number of sites along the southern Cape coast of South Africa, including Die Kelders and Klasies River (Fig. 1), which have been found to contain evidence of Middle Stone Age occupation. The Middle Stone Age deposits contain a large number of finely-worked bifacial points, a range of bone tools (Henshilwood and Sealey, 1997; Henshilwood et al., 2001b) and several pieces of engraved ochre (Henshilwood et al., 2002).

Blombos Cave (34°25′S, 21°13′E) is located 300 km to the east of Cape Town, just to the west of the village of Still Bay. It is currently ~100 m from the shore and ~35 m above sea level. The cave is cut into calcarenite of the Pliocene Wankoe Formation (part of the Bredasdorp Group). The remains of a large Quaternary dune that was...
formed against the coastal cliff are also to be found at Blombos, and these have been assigned to the Waenhuiskrans Formation. Sand from this dune is also found inside the cave, where it forms a continuous, archaeologically-sterile, layer (5–60 cm thick) that separates the Later Stone Age deposits from the artefact-rich, underlying Middle Stone Age deposits (Fig. 2). Its stratigraphic position makes it a key unit, since it provides a minimum age for the Middle Stone Age deposits. Its aeolian origin makes it a good candidate for optical dating. Parts of this eroded dune are to be found as a stack, preserved at present sea level, and as isolated remnants on the cliff face. For the current study, three samples were taken in February 2001. ZB15 was collected from the
uncemented sand layer within the cave, by pushing a 2” diameter opaque plastic tube into the cleaned baulk section. ZB13 was from a cemented remnant at the same altitude above sea level as the cave. ZB20 was collected from the cemented stack that remains in line with the cave entrance. This paper describes the multiple grain OSL measurements made on the samples, while the companion paper (Jacobs et al., 2003) describes the single grain measurements.

Experimental procedures

Sample preparation

The surface layer of samples ZB13 and ZB20 that might have been exposed to sunlight was discarded. Carbonates were dissolved in 10% HCl and organic matter was oxidised in 30 vols H₂O₂. Mechanical dry sieving separated the 212–250 µm diameter grain size fraction for ZB15 and the 180–212 µm grain size diameter fraction for ZB13 and ZB20. Quartz was obtained from these fractions via density separation using a sodium polytungstate solution of specific gravity 2.62 to reduce the potassium feldspar content and specific gravity 2.70 to remove heavy minerals. The separated quartz fraction was etched in 48% HF for 45 minutes to destroy any remaining feldspars and then washed with concentrated HCl for 45 minutes to remove fluorides. The purified quartz grains were then resieved using the lower sieve size (212 µm for ZB15, and 180 µm for ZB13 and ZB20). Grains for single aliquot analyses were mounted on 9.7 mm diameter aluminium discs using ‘Silkospray’ silicone oil. Each aliquot contained ~500–800 quartz grains and weighed about 5 mg.

Experimental apparatus

All single aliquot measurements were carried out using an automated Risø TL/OSL reader (Botter-Jensen et al., 2000). Optical stimulation was with blue light emitting diodes (LEDs) with peak emission at 470 nm, with 5 clusters of 6 LEDs directed at the sample disc. In front of each cluster, a green long-pass filter (Schott GG420) was fitted to attenuate the short wavelength tail of the LED emission (Botter-Jensen et al., 1999). In addition, a 1.0 W laser diode emitting at 830 nm was included in the reader, producing ~500 mW/cm² at the sample (Botter-Jensen and Murray, 1999). The OSL was measured with an EMI 9635Q photomultiplier tube through three 3 mm Hoya U-340 filters, one of which has a ZrO₂/SiO₂ coating to reduce transmission in the red. Beta irradiation was with a calibrated ⁹⁰Sr/⁹⁰Y source giving ~6 Gy/min.

The single aliquot procedure

Introduction

To obtain useful information on the depositional age of quartz samples, it is necessary for there to be an accurate method of converting the OSL signal to a reliable estimate of the equivalent dose (Dₑ). The intensity of the natural OSL signal is compared with the OSL intensities resulting from laboratory irradiation, usually presented as an OSL dose response curve. Studies on quartz from Australian dune sands have shown that there is a difference in the efficiency of luminescence production following irradiation in nature lasting tens of thousands of years, compared with that following a laboratory irradiation given a few minutes after a laboratory light exposure (Wintle and Murray, 1999). This is seen as a change in the OSL sensitivity.

The OSL signal from a test dose can be used to monitor sensitivity change throughout the construction of an OSL dose response curve. This procedure, originally suggested by Murray and Roberts (1998), was developed as the single aliquot regenerative dose (SAR) dating protocol by Murray and Wintle (2000). The SAR protocol involves making a series of OSL measurements (Lᵣ, Lₓ, Tᵣ, Tₓ as listed in Table 1) on each aliquot. The OSL response (Tₓ) to a test dose (Dₓ) given after each regeneration dose (Dₓ) monitors for changes in sensitivity. A sensitivity-corrected (Lₓ/Tₓ) dose response curve is constructed and Dₑ is determined by interpolation of Lᵣ/Tᵣ onto this curve.
curve. This procedure provides a value of $D_e$ for a single aliquot. Application to a number of aliquots results in an equivalent number of $D_e$ values being obtained, giving an opportunity to assess the reliability of the measurements.

**Measurement conditions**

The SAR protocol was used to provide 48 (ZB15 and ZB20) or 24 (ZB13) independent values of the equivalent dose, $D_e$, for each of the three samples. A heat treatment is applied in order to remove any OSL signal that may be derived from traps shallower than the main OSL trap that empties at around 320°C (Smith et al., 1986; Spooner, 1994a). Different aliquots were measured following 10 s preheats at different temperatures deliberately chosen to cause different rates of sensitivity change. For each sample, the first group of aliquots was preheated at 160°C, the next at 180°C, the next at 200°C and so on, up to a maximum temperature of 300°C.

Optical stimulation was made at an elevated temperature (125°C), using a heating rate of 5°C/s to reach this temperature. This is to circumvent re-trapping of charge at the 110°C TL peak during illumination (Wintle and Murray, 1997; 2000). Optical stimulation was for 40 s using blue LEDs at a constant power (90% of full power) to obtain $L_n$ and $T_n$ and $L_x$ and $T_x$. A cut heat of 160°C before the OSL measurement of the test dose was used to eliminate any signals that would interfere with the OSL measurement. Individual dose response curves were constructed for each aliquot using a range of regeneration doses (up to 120 Gy) and a test dose of 12 Gy. A repeated dose point was used to test the accuracy of the sensitivity correction within the dose response curve.

**Optical decay curves**

A 40 s exposure to the LEDs was used for optical stimulation. Fig. 3 shows a typical natural decay curve for ZB15. By 0.8 s, the signal has been reduced to about one third of that in the first 0.16 s. The natural OSL signal obtained in the first 0.8 s typically ranged from 5 000–40 000 (ZB15), 40 000–80 000 (ZB13) and 10 000–80 000 (ZB20) counts. The variation in equivalent dose ($D_e$) as a function of illumination time over 8 s (the entire OSL signal) is negligible. Shown as an inset to Fig. 3 is the plot of $D_e$ obtained for the integrated OSL signal as a function of the integration time. This demonstrates that the selection of the integration time does not have a significant effect on the value of $D_e$. The luminescence intensity (L) used to obtain $D_e$ is that from the first 0.8 s of the optical decay.

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**Table 1**

Single Aliquot Regenerative-dose (SAR) protocol (Murray and Wintle, 2000)

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Preheat for 10 seconds</td>
</tr>
<tr>
<td>2</td>
<td>OSL at 125°C for 40 seconds, measuring the natural intensity, $L_n$</td>
</tr>
<tr>
<td>3</td>
<td>Test dose, $D_t$</td>
</tr>
<tr>
<td>4</td>
<td>Cut heat to 160°C</td>
</tr>
<tr>
<td>5</td>
<td>OSL at 125°C for 40 seconds, measure test dose intensity, $T_n$</td>
</tr>
<tr>
<td>6</td>
<td>Regeneration dose, $D_x$</td>
</tr>
<tr>
<td>7</td>
<td>Preheat for 10s at the same temperature as in step 1</td>
</tr>
<tr>
<td>8</td>
<td>OSL at 125°C for 40 seconds, measure regenerated intensity, $L_x$</td>
</tr>
<tr>
<td>9</td>
<td>Test dose, $D_t$</td>
</tr>
<tr>
<td>10</td>
<td>Cut heat to 160°C</td>
</tr>
<tr>
<td>11</td>
<td>OSL at 125°C for 40 seconds, measure test dose intensity, $T_x$</td>
</tr>
<tr>
<td>12</td>
<td>Repeat steps 6–12 to build up a dose response curve</td>
</tr>
</tbody>
</table>

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decay curve with a background signal subtracted. The background for the natural, regenerative and the test dose signals is the mean of the last 8 s of the relevant natural or regenerative dose decay curves, as suggested by Murray and Wintle (2000).

Testing the appropriateness of the SAR protocol

The primary assumption of the SAR protocol is that the test dose OSL signal is representative of the luminescence sensitivity of the preceding OSL signal. The linear and proportional relationship of these two signals has previously been demonstrated for sedimentary quartz using specifically designed experiments (Murray and Roberts, 1998; Armitage et al., 2000; Murray and Wintle, 2000).

However, an equivalent relationship can be obtained (provided no thermal depletion occurs) using measurements at different temperatures obtained from SAR runs. In Fig. 4 the recycled dose points of one aliquot at each preheat temperature were used to obtain a plot of the test dose response (T_x) versus the preceding regeneration response (L_x). The two data points are consistent with a line forced to pass through the origin in each case, indicating a linear relationship between T_x and L_x. In addition, the data lie on a single line (not shown), except for the 280°C and 300°C preheat points. The data in Fig. 4 indicate that preheating at 240°C caused the sensitivity to increase by about a factor of 3 compared with the sensitivity following the 160°C preheat. This emphasises the need for application of a sensitivity correction in construction of the dose response curve. The deviation of the data obtained using the two higher temperature preheats from the lower temperature trend line is caused by a reduction in L_x as the result of thermal depletion of the OSL trap. Similar data sets were obtained for the other two samples.

Beside the temperature dependence shown in Fig. 4, sensitivity change can also be observed as a function of measurement cycle (e.g. Armitage et al., 2000; Bailey, 2000). In Fig. 5 the changes in response of the test dose through the SAR cycle are shown (the data shown in Fig. 4 are represented by cycles 4 and 7). Values of T_x are normalized to the value of T_n, the test dose response after measurement of the natural OSL (L_n). Data for lower preheats (160–240°C) show an initial decrease in sensitivity and the higher preheats (260–300°C) an increase. Over the 7 measurement cycles, the values increase and decrease by about 30%, similar to the 40% found by Armitage et al. (2000) for one of their samples when a constant regeneration dose was used.

We perform a validity check on the sensitivity correction procedure by repeating a regeneration dose within the SAR measurement cycle; the ratios
of these corrected intensities (the recycling ratio of Murray and Wintle (2000)) should fall within ±10% of unity. All samples conform to this test and exhibit no preheat dependence (e.g. Fig. 6).

Dose response curves

OSL dose response curves were constructed for all aliquots. The regeneration dose points in all aliquots of the three samples were fitted with an equation that is comprised of an exponential plus a linear term.

\[ I = I_0 + I_{\text{max}} (1 - e^{-D/D_0}) + k.D \]

where \( I \) is the corrected luminescence intensity, \( D \) is the given laboratory dose, \( D_0 \) and \( k \) are constants. All data were well fitted by this function and, as is typical in quartz, growth of the OSL response continued above doses of 80 Gy (e.g. Fig. 7).

Dose recovery experiment

An additional check on the applicability of SAR to this material can be obtained by zeroing the natural luminescence signal using a light source, and then giving aliquots a known laboratory dose. The aliquots are then treated in an identical way to those aliquots used to obtain \( D_e \).

For sample ZB15, 23 aliquots were bleached for 100 s with the blue LEDs and given a dose of 48.7 Gy. The \( D_e \) values obtained from these aliquots are displayed in two formats.

In one method (Fig. 8a) each individual \( D_e \) is represented by a Gaussian curve whose central point is the \( D_e \), whose width is the uncertainty in the \( D_e \), and whose area is a fixed value. The Gaussian curves from the different aliquots are then summed to create a probability density function. The individual \( D_e \) values are also shown superimposed on this graph, with the values put in increasing rank order. For these samples, given a known dose of 48.7 Gy, the results give a weighted mean of 48.7 ± 0.6 Gy and appear normally distributed.

The use of probability density functions for displaying single grain data (originally fission track ages) has been criticised (Galbraith, 1998), and radial plots have been used as an alternative method for displaying the \( D_e \) values (Galbraith et al., 1999). In such a diagram (Fig. 8b), each \( D_e \) value is plotted as an individual point on the graph. The position on the x-axis is a measure of the precision with which the \( D_e \) is known. The more precisely the value is known, the further it plots to the right of the diagram. The value plotted on the y-axis is the difference between the \( D_e \) for that aliquot and some reference value. The difference is then divided by the uncertainty in the \( D_e \);
thus the y-axis is the number of standard deviations that that $D_e$ value lies away from the reference value. The choice of reference value is arbitrary, and altering it simply causes the data points to rotate about the origin. A mathematical consequence of plotting these two parameters is that all aliquots with the same $D_e$ will fall on a line radiating from the origin. Thus it is convenient to draw a third ‘radial’ axis on the right hand side of the graph that gives the $D_e$. Such graphs are particularly useful for plotting data which have widely varying uncertainties. By drawing a band between the ‘+2’ and ‘-2’ points on the y-axis, one can delimit the area within which data will lie if they are consistent with a given value of $D_e$ within 2 standard deviations. For the data from the dose recovery experiment, 96% fall within 2$\sigma$ of the known dose (Fig. 8b).

**IR check for feldspar contamination of OSL signal**

Ensuring that luminescence is observed from quartz only is essential. However, Fragoulis and Readhead (1991) suggested that it is possible for quartz grains to enclose microinclusions and Huntley et al. (1993) have observed an IR-stimulated luminescence signal from etched quartz grains. If a potassium-rich feldspar inclusion is found in a quartz grain, then it is possible that it could give rise to a locally significant beta dose from the $^{40}$K decay. It could also cause underestimation of $D_e$ if the OSL signal from the feldspar exhibited anomalous fading (Wintle, 1973).

Stokes (1992) has interpreted the absence of a detectable IRSL signal under exposure to 880$\Delta$80 nm stimulation as indicating an absence of significant feldspar contamination in the final ‘pure’ quartz extracts in his multiple grain measurements. Spooner (1994b) has shown experimentally that feldspar, but not quartz, produces IRSL at room temperature and that stimulation with infrared radiation at room temperature should be able to act as a method of testing for feldspar contamination. However, no author has defined what IRSL intensity is unacceptable, since this relies heavily upon the equipment used, the age of the sample and the relative brightness of the feldspar and quartz.

A related, but not identical, check has been proposed by Duller (2003). This is based on the depletion of the OSL signal from feldspars following exposure to IR. In the current study, the SAR protocol was modified to include this OSL IR depletion ratio check for feldspar contamination at the end of the measurement sequence. The procedure involves two replicate measurements of a given dose. The first measurement ($L_x/T_x$) is as normal within the SAR procedure (same measurements as for the recycle dose point). The second measurement ($L_y/T_y$) is exactly the same as for the first except that, prior to preheating, the aliquot is
exposed to the IR laser diode at room temperature for 100 s. The ratio of \((L_y/T_y)/(L_x/T_x)\) is used to identify possible feldspar contamination, since the presence of feldspars will cause the second measurement \((L_y/T_y)\) to be reduced. Five percent of aliquots showed an OSL response that was reduced by more than two standard deviations after IR exposure and these aliquots were rejected. The advantage of this OSL IR depletion ratio approach over conventional IRSL measurements is that the impact of the feldspar contamination upon the dating procedure is measured directly.

**Dosimetry**

Dose rates were calculated using the conversion factors of Adamiec and Aitken (1998) with corrections for grain size and moisture content (Aitken, 1985) and the data are given in Table 2. The beta dose rate for ZB15 in Table 2 is about 1% lower than that used previously to calculate the total dose rate (Henshilwood et al., 2002) because we had previously omitted to allow for the effect of the HF etch on the beta dose. The external alpha particle contribution is considered negligible since the outer skin (ca. 9 µm) of the grain was removed by the HF etch.

For ZB15 the internal alpha dose rate of the etched and heavy-liquid separated quartz was obtained directly by alpha counting a finely-milled sub-sample. Using an alpha efficiency value of 0.04 (Rees-Jones, 1995), an internal alpha dose rate of 0.036 Gy/ka (36 µGy/a) was calculated. This value is consistent with the range of 0.01–0.05 Gy/ka quoted by Feathers and Migliorini (2001), and a value of 0.036 Gy/ka has been used for all three samples.

The external beta dose rate was determined from six 1 g subsamples of each sample using a Risø GM-25–5 beta counter (Botter-Jensen and Mejdahl, 1988) with a Shap granite standard (6.25 Gy/ka) and magnesium oxide as a background. The beta dose rate was calculated as the mean of these six values. The standard deviation of the average of these subsamples is approximately 3%. The external gamma dose rate has been estimated using the values for the concentration of uranium and thorium determined by thick source alpha counting (TSAC) and potassium by atomic absorption spectrometry (AAS). For the two samples outside the cave (ZB13 and ZB20) field gamma spectrometry measurements were not feasible. For ZB15, the calculated gamma dose rate was consistent with that measured in the field.

Table 2
Dose rate and ages for the sands from Blombos Cave

<table>
<thead>
<tr>
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<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain Size (µm)</td>
<td>212–250</td>
<td>180–212</td>
<td>180–212</td>
</tr>
<tr>
<td>Water Content (%)</td>
<td>10 ± 5</td>
<td>5 ± 2</td>
<td>5 ± 2</td>
</tr>
<tr>
<td><strong>TSAC</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uranium (ppm)</td>
<td>1.83 ± 0.11</td>
<td>2.07 ± 0.15</td>
<td>1.84 ± 0.13</td>
</tr>
<tr>
<td>Thorium (ppm)</td>
<td>2.04 ± 0.34</td>
<td>2.87 ± 0.46</td>
<td>2.70 ± 0.42</td>
</tr>
<tr>
<td><strong>AAS</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Potassium (%)</td>
<td>0.17 ± 0.01</td>
<td>0.20 ± 0.03</td>
<td>0.19 ± 0.01</td>
</tr>
<tr>
<td>Beta Dose Rate (Gy/ka)</td>
<td>0.34 ± 0.02</td>
<td>0.45 ± 0.02</td>
<td>0.44 ± 0.01</td>
</tr>
<tr>
<td>Gamma Dose Rate (Gy/ka)</td>
<td>0.31 ± 0.02</td>
<td>0.40 ± 0.03</td>
<td>0.37 ± 0.03</td>
</tr>
<tr>
<td>Cosmic Ray Dose Rate</td>
<td>0.05 ± 0.01</td>
<td>0.09 ± 0.01</td>
<td>0.05 ± 0.01</td>
</tr>
<tr>
<td>Total Dose Rate (Gy/ka)</td>
<td>0.73 ± 0.03</td>
<td>0.98 ± 0.03</td>
<td>0.89 ± 0.03</td>
</tr>
<tr>
<td>D_e (Gy)</td>
<td>50.3 ± 1.7</td>
<td>68.3 ± 2.4</td>
<td>63.1 ± 1.4</td>
</tr>
<tr>
<td>Age (ka)</td>
<td>69.2 ± 3.9</td>
<td>69.6 ± 3.5</td>
<td>70.9 ± 2.8</td>
</tr>
</tbody>
</table>

*Dose rates have been rounded to two decimal places, but they were summed prior to rounding. The total dose rates include 0.036 Gy/ka as an effective internal alpha dose rate, as discussed in the text.*
The cosmic ray dose rate for the sample inside the cave (ZB15) was estimated from the depth of the overburden, and also making corrections for geomagnetic latitude and altitude (Prescott and Hutton, 1994). The two samples outside would have been part of the Waenhuiskrans dune system for a considerable length of time, with 10–20 meters of sediment overburden. This would have been eroded slowly through time, and an average value for the overburden of 5 m for the sample 35 m above sea level (ZB13) and 10 m for the sample at sea level (ZB20) has been assumed.

The calculation of the moisture content of the samples over long time periods requires some speculation. Two moisture content measurements for the samples inside the cave were made. Firstly, the present day water content, expressed as $100/p_2$ (weight of water/weight of dry sediment) was measured as 5%. A second measurement gave a value of 29% for the free-draining moisture content. The dune layer in the cave is believed not to have ever been fully saturated since it is well above the present water table. Dose rates were calculated using a moisture content of 10% for the sample from inside the cave, and 5% for the samples from outside the cave.

The total dose rate for ZB15 is 4% higher than that used by Henshilwood et al. (2002) owing to the changes in the beta dose rate and inclusion of the internal alpha dose rate. The effect is offset by the change in the model for $D_e$ calculation discussed in the next section.

Equivalent dose and age calculations

Fig. 9 shows a plot of the equivalent dose ($D_e$) versus the preheat temperature for sample ZB15. By using a broad range of preheating temperatures, the pattern of sensitivity changes is accentuated, as previously shown in Fig. 5. Thus, being able to obtain the same $D_e$ value, even when using very different preheat treatments, implies that the correction within SAR for luminescence sensitivity changes is effective, and hence the confidence in the final $D_e$ value is enhanced. For all three samples the lower preheat temperatures (160–180°C), and the highest (300°C) preheat, showed a large scatter of $D_e$ values. From 200 to 280°C the $D_e$ values from 29 (ZB15), 30 (ZB20) and 15 (ZB13) replicate aliquots showed no systematic trend with temperature.

The data for preheats from 200 to 280°C are displayed for ZB15, ZB13 and ZB20 (Figs. 10, 11 and 12) as probability density functions and also as radial plots. Analysis of the luminescence data included a 1.5% systematic uncertainty on every luminescence measurement ($L_x$ and $T_x$, Armitage et al., 2000), and this uncertainty is propagated through the calculations.

To obtain an age for each sand sample, it is necessary to combine the individual values of $D_e$ by taking the weighted mean, with each value of $D_e$ weighted in proportion to the reciprocals of the variance ($\sigma/D_e$). This approach biases the mean value of $D_e$ towards the smaller values when each $D_e$ value has the same relative standard error ($\sigma/D_e$). For single grain SAR analysis, it has been suggested by Galbraith et al. (1999) that it is more appropriate to take the log of each $D_e$ value and obtain a weighted mean of these values, using the reciprocals of the variances related to the log $D_e$ values (approximated by the relative standard error, $\sigma/D_e$). This approach is preferred by Galbraith et al. (1999) since it reduces the multiplicative
effect of error propagation when obtaining $D_e$ by using L/T ratios. They considered two approaches for well bleached samples, the “common age model” and the “central age model”. The former assumes that all grains have an identical $D_e$ value, whereas the latter assumes that the grains received a range of doses, centred on an average value of $D_e$. We have applied these two procedures to the single aliquot data for the three dune samples, and the results are given in Table 3, together with the weighted mean value discussed above. The “central age model” not only gives the central value for $D_e$, but also provides an estimate of the width of the distribution of true $D_e$ values. This is termed the overdispersion of the data set and these values are also given in Table 3. The fact that they are significantly above zero suggests that a range of $D_e$ values are present, perhaps as a result of microdosimetry variation, though this would be expected to show up more in single grain analysis. The values of $D_e$ obtained using the “common age model” and the “central age model” are very close to each other, and in each case systematically higher than the values obtained using the weighted mean. This is because of the different weighting factors used, as discussed above. The error term associated with the weighted mean is the standard error of the mean, and is obtained by dividing $\sigma$ by $\sqrt{n}$ where $n$ is the number of independent estimates of $D_e$. This error term is similar in magnitude to that obtained for the “central age model”. Given

Fig. 10. Values of $D_e$ for ZB15 obtained by SAR, (a) probability plot together with values in ranked order and (b) radial plot (drawn with a reference value of 50.3 Gy).

Fig. 11. Values of $D_e$ for ZB13 obtained by SAR, (a) probability plot together with values in ranked order and (b) radial plot (drawn with a reference value of 68.3 Gy).
the observation that there is some degree of over-dispersion for all three samples, the values of $D_e$ obtained using the “central age model” were chosen as those most appropriate for calculation of the ages, and these values are those given in Table 2. The ages thus obtained are $69.2 \pm 3.9$, $69.6 \pm 3.5$ and $70.9 \pm 2.8$ ka for samples ZB15, ZB13 and ZB20, respectively. The age of $69.2 \pm 3.9$ ka for ZB15, the sand from within the cave, is almost identical to the value of $69 \pm 5$ ka given in footnote 22 of Henshilwood et al. (2002).

The three age estimates confirm that all three samples are indeed related to the large dune that formed against the cliff. A weighted mean age of $70.1 \pm 1.9$ ka was calculated for the Waenhuiskrans Formation, though the uncertainty on this combined age does not take into account the separation of systematic and random uncertainties. This provides a minimum age for the underlying MSA levels found in the cave (Henshilwood et al., 2002).

Even after rejecting those aliquots measured following a preheat of 160, 180 and 300°C, all three samples exhibited a spread in $D_e$ between replicate aliquots (Figs. 10, 11 and 12) and this is reflected in the overdispersion values given in Table 3. This is in contrast with the results of the dose recovery experiment (Fig. 8), in which the overdispersion is 0%. The lack of a systematic trend with preheat (Fig. 9) implies that the spread in $D_e$ is not caused by something inherent to the SAR procedure. The presence of feldspar contamination within some aliquots would cause scatter, but this explanation is unlikely because those aliquots whose IR check suggested the existence of feldspars were rejected. Alternatively, such spread could result from small scale variations in the radiation dose rate to individual grains during the period of burial. Such an explanation is inherently difficult to test, but difficult to reject. Another possible explanation is that the sediment contains a mixture of grains of different age. This seems unlikely, given the aeolian origin of the samples. In order to be confident that this is not the case, and to investigate the larger than expected spread in the $D_e$ values, single grains have been analysed from each sample, and this work is described in Jacobs et al. (2003).

Summary

Three dates have been obtained for remnants of a coastal sand dune at Blombos on the southern Cape coast of South Africa. Optical dating of purified quartz grains was achieved using a SAR protocol. The reliability of the procedure was demonstrated by undertaking a recycling ratio test, by confirming that the $D_e$ estimates are independent of preheat temperature, by recovering a given laboratory dose, and by confirming the purity of the refined quartz via IRSL tests. Comparing the results of the dose recovery experiment with the values of $D_e$ for the three samples, it can be seen...
that the latter show greater scatter. The cause of this additional scatter is unclear, and measurements of single mineral grains are described in Jacobs et al. (this issue) to clarify this.

One of the sand units was found inside a cave, where it overlay the archaeologically-rich layers of the Middle Stone Age. For this sample an age of $69.2 \pm 3.9$ ka was obtained, an age that was confirmed by the other two ages $69.6 \pm 3.5$ ka and $70.9 \pm 2.8$ ka. These dates confirm the antiquity of the artefacts found in the Middle Stone Age deposits.

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