Late Acheulean hominins at the Marine Isotope Stage 6/5e transition in north-central India

Michael Haslam, Richard G. Roberts, Ceri Shipton, J. N. Pal, Jacqueline L. Fenwick, Peter Ditchfield, Nicole Boivin, A. K. Dubey, M. C. Gupta and Michael Petraglia

Supplementary Information

Chronology

To estimate the time of deposition of the artefact-bearing sediments at Bamburi 1 and Patpara, we collected samples for optically stimulated luminescence (OSL) dating by hammering metal tubes into the cleaned section faces and then sealing the tubes in black plastic bags after removal. In the OSL dating laboratory at the University of Wollongong, the sediment at both ends of each tube was discarded under dim red illumination (as it would have contained grains exposed to sunlight at the time of sample collection), and quartz grains of 180–212 μm in diameter were then extracted from the internal (light-safe) portions using standard preparation procedures (Aitken, 1998). These included the successive application of hydrochloric acid (to dissolve carbonates), hydrogen peroxide (to oxidise any organic matter), dry sieving (to obtain different grain-size fractions) and density separation using sodium polytungstate solutions (to isolate the quartz from the heavy minerals and feldspars). The separated quartz grains were etched with 48% hydrofluoric acid for 40 min to remove the alpha-dosed rind of each quartz grain and destroy any remaining feldspars.

OSL measurements of individual quartz grains were made using the same Risø equipment and single-aliquot regenerative-dose (SAR) procedures as described elsewhere (Petraglia et al., 2007; Jacobs and Roberts, 2007; Jacobs et al., 2008a). The SAR procedure involves measuring the OSL signals from the natural (burial) dose and from a series of regenerative doses (given in the laboratory by means of a calibrated $^{90}Sr/^{90}Y$ beta source), each of which was preheated at 260°C for

10 s prior to optical stimulation by an intense, green (532 nm) laser beam for 2 s at 125°C. The resulting ultraviolet OSL emissions were detected by an Electron Tubes Ltd 9235QA photomultiplier tube fitted with Hoya U-340 filters. A fixed test dose (~10 Gy, preheated at 240°C for 5 s) was given after each natural and regenerative dose, and the induced OSL signals were used to correct for any sensitivity changes during the SAR sequence. As a check on possible contamination of the etched quartz grains by feldspar inclusions, we also applied the OSL IR depletion-ratio test (Duller, 2003) to each grain at the end of the SAR sequence, using an infrared exposure of 40 s at 50°C.

For all grains, the D_e values were estimated from the first 0.2 s of OSL decay, with the mean count recorded over the last 0.3 s being subtracted as background. The dose-response data were fitted using a saturating exponential plus linear function and the sensitivity-corrected natural OSL signal was projected on to the fitted dose-response curve to obtain the D_e by interpolation. The uncertainty on this estimate (from photon counting statistics, curve fitting uncertainties, and an allowance of 2% per OSL measurement for instrument irreproducibility) was determined by Monte Carlo simulation, using the procedures described by Duller (2007) and implemented in Analyst version 3.24. The final D_e uncertainty includes a further 2% (added in quadrature) to allow for any bias in the beta source calibration.

Aberrant grains were rejected using the quality-assurance criteria described and tested previously (Jacobs et al., 2006a). Grains were rejected if they exhibited one or more of the following properties: 1) weak test-dose OSL signals (i.e., the initial intensity of the test-dose signal was less than three times the background intensity); 2) high levels of recuperation (i.e., the sensitivity-corrected OSL intensity measured in the 0 Gy regenerative-dose cycle was more than 5% of the sensitivity-corrected natural OSL intensity); 3) poor recycling ratios (i.e., the sensitivity-corrected OSL values for duplicate regenerative doses differed by more than 2σ); 4) natural OSL signals

equal to or greater than the saturation limit of the dose-response curve (i.e., the sensitivity-corrected natural OSL intensity exceeded that induced by the largest regenerative dose ('Class 3' grains of Yoshida et al., 2000) or lay in the saturated region of the dose-response curve, so a finite estimate of D_e could not be obtained); and 5) significant loss of OSL signal after exposure to infrared stimulation (i.e., the OSL IR depletion ratio was less than unity by more than 2σ, which indicates contamination of quartz grains by feldspar inclusions).

Under these experimental conditions, and using these quality-assurance criteria, we recovered correct dose estimates for individual grains of quartz from BAM-2/1 and PAT-4/1 that had first been bleached with natural sunlight for 2 days and then given a known dose in the laboratory. The mean ratios of measured to given dose (BAM-2/1: 1.01 ± 0.01, n = 126; PAT-4/1: 0.97 ± 0.02, n = 68) are statistically consistent with unity, which shows that the chosen SAR procedures can accurately recover a known dose under controlled conditions. Overdispersion (OD) values of 5–10% were obtained for these dose recovery data sets (BAM-2/1: 5.4 ± 0.8%; PAT-4/1: 9.9 ± 1.4%). Such values are similar to those reported previously for dose recovery tests on single grains of quartz (Roberts et al., 2000; Thomsen et al., 2005; Jacobs et al., 2006b). Overdispersion refers to the relative spread in the dose distribution above and beyond that associated with the measurement uncertainties of individual grains, and was calculated using the Central Age Model (Galbraith et al., 1999). If all of the scatter were due to measurement error alone, then the OD value would be zero.

Of the 3600 grains measured, only 593 grains (i.e., 16.5% of the total) passed all of the quality-assurance criteria (Table 3). Most grains in each sample (58–85%) were rejected because their test-dose OSL signals were indistinguishable from background, resulting in D_e values being obtained from between 55 grains (BAM-1/1) and 215 grains (PAT-3/2). The accepted grains exhibited a rapid rate of initial OSL decay when stimulated by the laser beam; the two left-hand plots in Figure 6 show decay curves for a moderately bright grain from BAM-1/1 and a very bright grain from

BAM-3/2 (in both cases, the laser was switched on at 0.1 s). This behaviour is consistent with the OSL signal arising from the most light-sensitive ('fast') component of quartz OSL, for which the SAR procedure was developed originally (Galbraith et al., 1999). The corresponding dose-response curves are well described by a saturating exponential plus linear function (Figure 6, inset plots), which extends to doses of several hundred grays (1 Gy = 1 J/kg). In these graphs, the dashed line projects from the sensitivity-corrected OSL intensity on the y-axis to intersect the dose-response curve at the D_e, which can be read by drawing a line vertically to intersect the dose axis. Grains that did not exhibit 'plus linear' growth at high doses, or that saturated at low doses, were rejected using the fourth quality-assurance criterion, because only minimum estimates of D_e could be obtained.

The centre column of Figure 6 shows the D_e values of the 55 and 136 quartz grains of BAM-1/1 and BAM-3/2 that passed all of the quality-assurance criteria, and the right-hand column shows the D_e distributions for the two Patpara samples. In these radial plots, each point represents the D_e value for a single grain, which can be read off the graph by drawing a line from the origin of the standardised estimate axis, through the point of interest, until it intersects the D_e axis. The uncertainty on this D_e is obtained by drawing a vertical line from the data point to intersect the relative error axis. D_e values measured with the greatest precision fall furthest to the right, and any shaded band of ± 2 units projecting from the standardised estimate axis should capture 95% of the points if the measurement uncertainties are sufficient to account for the observed scatter (Galbraith et al., 1999). As is typical for quartz grains (Galbraith et al., 2005; Jacobs and Roberts, 2007), the D_e values are spread more widely than can be explained by their measurement uncertainties alone as no single band of ± 2 units can encompass 95% of the points. The shaded bands in Figure 6 are centred on the D_e components that accommodate the majority of grains, as discussed below.

The extent of scatter in D_e is reflected in the overdispersion values of 32–37% for the five samples (Table 3). OD values of up to 20% are common for single-grain data sets, with some reported

examples exceeding 30%, for grains that are known or thought to have been fully bleached at burial and not affected by post-depositional disturbance from sediment mixing or by differences in beta dose rate among grains buried at the same time (summarised in Arnold and Roberts, 2009: Table 4). As shown by a factorial experiment (Galbraith et al., 2005) and by other controlled trials (Roberts et al., 2000; Thomsen et al., 2005; Jacobs et al., 2006b), there are many possible causes of D_e overdispersion that cannot be completely accounted for by experimental measurements in the laboratory, but which are unrelated to field complications such as incomplete bleaching, post-depositional mixing or variations in the beta dose rate to individual grains. For example, non-identical field and laboratory conditions include differences in the intensity and wavelength of the bleaching spectra, the type of ionising radiation and the rate at which dose is delivered to the grains, and the length of time available for any redistribution of trapped charge due to defect migration (Galbraith et al., 2005). Also, the heating and bleaching procedures employed in the SAR sequence will likely not empty the OSL source traps in all grains uniformly, and thermal transfer effects are apt to vary among grains. The results of the dose recovery tests on BAM-2/1 and PAT-4/1 attest to such inherent 'natural' variability: the measured doses were overdispersed by 5–10%, despite having eliminated any field-related problems by exposing the grains to prolonged sunlight before giving them an identical, known dose in the laboratory.

Another possible reason for the large OD values of the Bamburi 1 and Patpara samples is that the sensitivity-corrected natural OSL signals intersect the dose-response curve in the 'plus linear' region, where the low slope will give rise to larger uncertainties than those obtained for grains with D_e values that lie in the steeper portion of the growth curve (<150 Gy). The majority of quartz samples dated worldwide fall in the latter category, so those investigated here have unusually high D_e values by comparison. We can dismiss the possibility that the overdispersion is due to the presence of small D_e values from quartz grains contaminated by inclusions of feldspar, which is well known to produce D_e underestimates (Aitken, 1998), because all such grains (n = 39, ~1% of

the total number measured) had been identified and rejected already using the quality-assurance criteria. But the presence of carbonate nodules in the Bamburi 1 and Patpara deposits makes it highly likely that some of the measured grains were located next to, or encased by, carbonates. As these grains would have experienced greatly reduced beta dose rates (owing to the negligible radioactivity of carbonates compared to most other sediments), their D_e values would also be smaller than those obtained for grains deposited at the same time but juxtaposed by non-carbonate materials. The D_e distributions may be overdispersed, therefore, in part because of between-grain differences in beta dose rate.

Does the extent and pattern of spread in the D_e values support this hypothesis? Certainly, a feature common to all five samples is the existence of grains with D_e values much smaller than those of the majority of grains (Figure 6). We used the Minimum Age Model (Galbraith et al., 1999) to estimate the D_e value for the population of grains with the smallest measured doses, and divided this by the beta-subtracted environmental dose rate (i.e., the total dose rate minus the beta contribution) to determine the burial age of the presumed carbonate-coated grains. For each sample, the calculated age of this population of grains equalled or exceeded that of the majority of grains, which indicates that a negligible beta dose rate is sufficient to explain the smallest D_e values in these distributions.

In the above calculation, the D_e value for the majority of grains was obtained using the Finite Mixture Model (FMM) and was then divided by the total environmental dose rate (i.e., including the beta contribution) to estimate the age. (Details of the methods used to measure the different components of the environmental dose rate are provided below.) The FMM can be used in conjunction with statistical criteria, such as maximum log likelihood and the Bayes Information Criterion, to identify the smallest number of D_e components needed to fit a measured D_e distribution (Roberts et al., 2000). Details of this model, and how to implement it in practice, are provided by David et al. (2007) and Jacobs et al. (2008b). For the samples investigated here, two-

component fits with an OD of 10–15% were sufficient to accommodate the observed scatter in D_e. To determine the OSL ages, we used the FMM to calculate the weighted mean D_e value of the component containing the majority of grains (in effect, a component-specific Central Age Model estimate of D_e). These components are shown as shaded bands in Figure 6, and include 80–90% of the accepted grains for four of the samples and ~60% of the grains for PAT-3/2 (Table 3).

Can the 10–40% of the grains with substantially smaller D_e values be explained as a result of their proximity to carbonates and a correspondingly reduced beta dose rate? A micromorphological study of the Bamburi 1 and Patpara deposits has not been undertaken, but the mass of carbonate in the bulk samples was determined by loss on ignition. The measured carbonate contents of 4.2–8.5% (Table 3) are similar to the proportions of high-precision D_e values (i.e., relative errors <20%) that are significantly smaller than the mean D_e of the majority of grains in each sample. For example, BAM-1/1 has 6 grains with high-precision D_e values substantially smaller than the mean D_e of the major component (381 ± 18 Gy): this represents 11% of the 55 grains for which D_e values could be obtained, which is similar to the carbonate content of 8.5% for this sample. Likewise for PAT-4/1, 4 of 97 grains (4%) have high-precision D_e values significantly smaller than the mean for the major component, and the carbonate content of this sample is 4.2%. The same general relation holds true for the other samples also. D_e values with relative errors >20% are omitted from this comparison, because they are mostly consistent with the weighted mean, owing to their large uncertainties.

Similar results are obtained when a two-component fit to the entire D_e data set is made using the FMM, which assigns to each grain a probability of belonging to the major or minor D_e component. Inspection of these probabilities reveals that the high-precision D_e values are clearly associated with one of the two components, whereas the less precise D_e values have a reasonable likelihood of belonging to either component. Overall, the FMM gives a higher estimate of the proportion of grains in the minor component, but the uncertainty associated with these estimates is

correspondingly large, reflecting the inclusion of both the high- and low-precision D_e values. For example, the minor components of BAM-1/1 and PAT-4/1 consist of 22 ± 7% and 8 ± 4% of the grains, respectively, which are statistically consistent with the bulk carbonate contents of these samples. In summary, therefore, a small number of grains in each sample are likely to have experienced greatly reduced beta dose rates, and the relative abundance of such grains is consistent with the carbonate contents of these samples.

We used the procedure described in Jacobs et al. (2008c) to estimate the beta dose rates experienced by the majority of grains in our samples, from which the OSL ages were obtained. In each case, the D_e value of the major FMM component was divided by the total dose rate calculated using the adjusted beta dose rate, which is shown in Table 3 alongside the original (uncorrected) value for the bulk sample. This adjustment accounts for the fact that if the carbonate-coated grains (which comprise the minor D_e component) had experienced below-average beta dose rates, then the grains in the major D_e component must have received above-average beta dose rates, as the sum of the pair gives the average (i.e., measured) beta dose rate for the bulk sample.

To estimate the adjusted beta dose rate for the major D_e component, we first calculated the mid-point value of the bulk sample beta dose rate and zero (the latter representing grains surrounded by >2 mm of carbonate). This corresponds to the average beta dose rate for the grains in the minor D_e component. Using BAM-3/2 as a worked example, the bulk sample beta dose rate is 1.634 Gy/ka (listed in Table 3 as ‘Beta’), so half of this is 0.817 Gy/ka. This value is then multiplied by the proportion of grains assigned to this component by the FMM (19.1%), to estimate the fraction of the bulk sample beta dose rate associated with these grains (0.817 x 0.191 = 0.156 Gy/ka). The difference between this value and the bulk sample beta dose rate represents the fraction of the latter associated with grains in the major D_e component (1.634 – 0.156 = 1.478 Gy/ka), which is divided by the proportion of grains in this component (80.9%) to estimate the average beta dose rate for

these grains (1.478 / 0.809 = 1.827 Gy/ka, listed in Table 3 as 'Adjusted beta'). Hence, the bulk sample beta dose rate (1.634 Gy/ka) equals the sum of the fractional beta dose rates for the minor and major components: 19.1% of grains at 0.817 Gy/ka plus 80.9% of grains at 1.827 Gy/ka.

To obtain an estimate of uncertainty for the adjusted beta dose rate, we assumed that the range of beta dose rates experienced by the carbonate-affected grains lies between zero and the bulk sample beta dose rate (1.634 Gy/ka) at 2σ and are normally distributed about the mid-point value (0.817 Gy/ka). At 1σ, therefore, the uncertainty on the adjusted beta dose rate of BAM-3/2 is half of 0.817 Gy/ka, and the standard error on the mean is obtained by dividing this value by the square root of the number of grains in the major D_e component (110 grains, calculated as 80.9% of the total of 136 accepted grains). This gives a value of 0.039 Gy/ka, which is added (in quadrature) to the measurement error on the bulk sample beta dose rate (0.080 Gy/ka) to estimate the total uncertainty on the adjusted beta dose rate (0.089 Gy/ka).

The beta dose rate adjustment has only a small effect on the calculated OSL ages. Statistically indistinguishable ages are obtained using the Central Age Model estimates of D_e and the bulk sample beta dose rates. For example, the upper and lower samples at Bamburi 1 change in age from 131 ± 10 and 131 ± 9 ka (Table 3) to 115 ± 10 and 120 ± 9 ka, respectively. Likewise at Patpara, the two samples change in age from 140 ± 11 and 137 ± 10 ka (Table 3) to 132 ± 9 and 128 ± 11 ka, respectively. This comparison shows that the ages obtained for these samples are not sensitive to the dose rate correction procedure, regardless of the proportion of grains in the major and minor D_e components. However, we consider the ages determined using the D_e values for the major FMM component and the adjusted beta dose rates to be more accurate than those obtained using the Central Age Model estimates of D_e and the bulk sample beta dose rates, because the former take into account the known occurrence of carbonate in the deposits.

The environmental dose rate for each sample was calculated as the sum of the beta and gamma dose rates due to the radioactive decay of ^{238}U, ^{235}U, ^{232}Th (and their daughter products) and ^{40}K, plus the contribution from cosmic rays (Table 3). We also included an effective internal alpha dose rate of 0.03 ± 0.01 Gy/ka, which captures (at 2σ) the range of values (0.01–0.05 Gy/ka) measured previously for sedimentary quartz grains from Australia and Africa (Feathers and Migliorini, 2001; Bowler et al., 2003; Jacobs et al., 2006b). The beta dose rates were determined in the laboratory on a dried and powdered portion of each sample using a Risø GM-25-5 low-level beta multicounter, making allowance for beta-dose attenuation due to grain size and hydrofluoric acid etching (Brennan, 2003). This approach does not give information on whether the beta emissions originated from the U or Th decay chains, or from ^{40}K, but instead gives the total counts from these sources. However, because ~60% of the beta dose rate in the ^{238}U series originates from decays in the lower part of the chain, this method of measurement of the U-series contribution is superior to neutron activation analysis or ICP-MS, for example, both of which measure the parental U concentration; determination of the beta dose rate from the latter depends critically on the assumption of secular equilibrium in the ^{238}U chain, which is commonly not the case (Olley et al., 1996, 1997).

The gamma dose rates were measured at Bamburi 1 and Patpara with a portable gamma-ray spectrometer, to take account of any spatial heterogeneity in the gamma radiation field within 30 cm of each OSL sample (as gamma rays can penetrate this distance through sediment and rock). After removing the metal tube hammered into the section at each sample location, the hole was extended to a depth of 30 cm using a hand-auger, and the probe was inserted into the hole. Counts were collected for 30 min with a 2-inch NaI(Tl) crystal. The detector was calibrated using the concrete blocks at Oxford (Rhodes and Schwenninger, 2007) and the gamma dose rate was determined using the 'threshold' technique (Mercier and Falguères, 2007). This approach gives an estimate of the combined dose rate from gamma-ray emitters in the U and Th chains and from ^{40}K, and does not give information on their relative contributions. As with beta counting, however, this

approach is much less sensitive to U-series disequilibria than are methods that calculate the gamma dose rate from the parental U concentration, because ~98% of the gamma dose rate in the ^{238}U series originates from decays in the lower part of the chain. By measuring the gamma and beta dose rates in this manner, we have implicitly assumed that the present state of (dis)equilibrium in the U and Th decay chains has prevailed throughout the period of sample burial. Numerical modelling has shown, however, that this assumption is unlikely to lead to errors in the total dose rate of more than 2–3%, even for the most common time-dependent disequilibria in the ^{238}U series, when techniques such as these are employed (Olley et al., 1996, 1997).

The cosmic-ray dose rates were estimated from published equations (Prescott and Hutton, 1994), taking into account the latitude, longitude and altitude of Bamburi 1 and Patpara, as well as the water content of the samples (Readhead, 1987) and their burial depths, both averaged over the time since deposition. To accommodate the range of plausible alternative scenarios, we calculated the cosmic-ray dose rate for the present burial depth of each sample (which equates to assuming the sediment overburden reached its current thickness soon after sample deposition and maintained it thereafter) and for half the present depth (which equates to a steady rate of accumulation of overburden from the time of sample deposition to the present day). We took the mean of these two determinations and assigned it a relative uncertainty of 10%, which covers both of these scenarios at 1σ, and added (in quadrature) a further 10% for the systematic uncertainty in the primary cosmic-ray intensity (Prescott and Hutton, 1994). The OSL ages are not sensitive, however, to errors in the estimation of the cosmic-ray dose rate, because it constitutes only 4.5–7.0% of the total dose rate for these samples (Table 3).

The beta, gamma and cosmic-ray dose rates were calculated for long-term sample water contents of 7.5% at Bamburi 1 and 6.5% at Patpara. These values are ~4% moister than the measured (field) water contents at both sites (Table 3), but are smaller than the saturation water contents determined

in the laboratory (~22% and ~21% for the Bamburi 1 and Patpara samples, respectively). We used water contents slightly above the field values to compensate for sample collection during the dry season. But we consider much higher time-averaged water contents (i.e., integrated over the period since sample deposition) to be unlikely, for three reasons: 1) the deposits are free draining and not waterlogged; 2) the wet season lasts only one-quarter of each year; and 3) the present and last interglacial periods were more humid than the last and penultimate glacial periods, which account for at least half of the past 140 ka. We assigned an uncertainty of ± 2% to each of the Bamburi 1 and Patpara water contents to accommodate (at 2σ) the field values and any likely variations averaged over the last 140 ka. The chosen water contents are consistent with the value of 10 ± 5% used in a previous OSL dating study of the Son and neighbouring Belan valleys (Williams et al., 2006). The calculated ages are not particularly sensitive to the chosen values, however, increasing only by ~1% for each 1% increase in water content.

We calculated the beta and gamma dose rates using the updated water-content correction factors of Nathan and Mauz (2008), and we also applied their carbonate-content correction factors. For the latter calculation, we used a carbonate content of 6 ± 2% for all samples. Nathan and Mauz (2008) show by numerical modelling that samples with low carbonate contents (<20%) require a negligible correction for time-dependent changes to the beta and gamma dose rates, which are also insensitive to the timing of onset and completion of carbonate formation. For the five samples dated in this study, the age estimates in Table 3 decrease by only 6.2–7.1 ka if the carbonate content is reduced from 6% to zero (which is clearly too low, given the presence of carbonate nodules in the modern deposits). In conclusion, therefore, the OSL ages obtained for the Bamburi 1 and Patpara samples are robust to any reasonable deviations from the chosen D_e values, cosmic-ray dose rates, water and carbonate contents, and accompanying adjustments to the beta and gamma dose rates.

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