Luminescence Dating at Diepkloof Rock Shelter – New Dates from Single-grain Quartz

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Abstract: Diepkloof Rock Shelter, in South Africa's Western Cape, has been the subject of two different luminescence dating projects in recent years (Jacobs et al. 2008 and Tribolo et al. 2013) with somewhat divergent results. This paper presents dating analysis of three additional samples from Diepkloof, as well as one sample each from Kathu Pan 6 and Hollow Rock Shelter, also in South Africa. These samples were collected in 1995, prior to the other two projects. Analysis of single-grain quartz shows that the samples are near saturation. Conventional rejection criteria, when applied to dose recovery data, result in an underestimation of dose for high administered doses. Applying the fast ratio (Durcan and Duller 2011, Duller 2012) as a rejection criterion removes this underestimation and preferentially rejects grains from the samples with low equivalent dose values. This makes the ages older than they would have been without this criterion. Age results are older than those presented by Jacobs et al. (2008), and slightly older or equivalent to those provided by Tribolo et al. (2013).

Key Words: Middle Stone Age, Diepkloof Rock Shelter, OSL Dating, Quartz Saturation

1. Introduction

Diepkloof Rock Shelter, near the Atlantic shore of South Africa's Western Cape, contains a remarkable sequence of Middle Stone Age (MSA) deposits that documents the transitions among several recognized technological complexes, including Howiesons Poort (HP) and Still Bay (SB). Both HP and SB have figured prominently in discussions of origins of human modernity (e.g., Jacobs et al. 2008). Early work in the shelter involved small test excavations, in 1973 and 1986. Then in 1999 a South African-French team began extensive excavations at Diepkloof, lasting until the present (Parkington et al. 2013). Results of this work have recently been published in a special issue of this journal.

Much attention has been paid to the chronology at Diepkloof, particularly that provided by luminescence dating, because most of the MSA deposits are beyond the range of radiocarbon. Notably, a discrepancy in dates has been reported between results from an Australian lab (Jacobs et al. 2008) and a French lab (Tribolo et al., 2009, 2013), with the French dates generally older than corresponding Australian dates, at least for the older part of the sequence. Some agreement between the labs is reported for the younger part of the sequence, although the French team only has limited dates for this section. The different chronologies between the two labs also support different interpretations of HP and SB (Porráz et al. 2013a). Some of the disagreement between the French and Australian studies have to do with differences in technological typology (Porráz et al. 2013b), but this paper deals only with the dating.

This paper presents luminescence analyses of three samples from Diepkloof and one sample each from two other MSA sites in South Africa, all of which were collected in 1995, and none of which have been previously published (Figure S1).

2. Sites and Sampling

Initial luminescence samples at Diepkloof were collected in 1988 by John Vogel and Stephan Woodburn of the Quaternary Dating Research Unit in Pretoria. They collected two samples from the 1973 test pit. In 1995 the author (University of Washington, Seattle) collected three additional samples. These also were drawn from the 1973 test pit, long before the expanded excavation. The dating results from the Pretoria and Seattle labs were compiled into a report, but it was never published, partly because knowledge of the Diepkloof technological sequence was imperfectly known at the time. The author has recently reanalyzed the three Seattle samples using single-grain OSL dating (optically stimulated luminescence) of quartz and multi-grain IRSL dating (infrared stimulated luminescence) of feldspar, and these are reported here to provide additional data in regards to the chronology of the site.

The excavations at Diepkloof have been divided into three interconnecting sectors: trench, main and back (Figure S1B and Figure 2 in Parkington et al. 2013). Most of the Australian and French samples were collected from the trench or main section, although the French collected some heated lithic samples from the back section, where the 1973 test pit is located. How the Seattle samples correlate with the Australian and French samples is a little difficult because different stratigraphic terminology was used in the 1973 test unit as opposed to other parts of the excavation. The top of the 1973 unit (Figure S1C) consists of a thin layer of sediments related to the Late Stone Age (LSA). Below

that is a massive layer with abundant microstratigraphy consisting of stacked lenses of ash and charcoal, originally called the orange-black layer. This corresponds with lithostratigraphic unit 4 of Miller et al. (2013), wherein the sedimentary sequence in other parts of the excavation is described. Two of the Seattle samples were collected from this stratum, one, UW247, about 24 cm below the LSA material, and one, UW325, near the bottom about 80 cm below UW247. Below the orange-black layer is a brown sandy layer, with less microstratigraphy, which corresponds with lithostratigraphic unit 3 of Miller et al. (2013). UW260 was collected from this stratum. The orange-black layer contains Late Howiesons Poort and Intermediate Howiesons Poort lithics, using the terminology of Porraz et al. (2013b). UW247 appears to be Late Howiesons Poort or Post-HP. UW325 appears to be Late or Intermediate Howiesons Poort and UW260 appears to be Intermediate Howieson Poort.

For comparative purposes the reanalysis of samples from two other sites is reported. One, UW287, is from Hollow Rock Shelter, a single-component Still Bay site in the mountains east of Diepkloof. Also called Sevilla 48, the site consists of shallow, undifferentiated slope wash deposits that have collected in a cavity underneath a large boulder. The deposits appear relatively undisturbed, including what is interpreted to be an intact hearth. The deposits yielded 69 Still Bay points from two excavations in 1993 and 2008 (Högberg and Larsson 2011). No other diagnostic artifacts were found in the shelter, although one backed piece, typical of HP, was found outside the shelter. Some preliminary OSL ages from samples collected in 2008 have been reported by Högberg and Larsson (2011). The UW sample was collected about midway in a 26-cm thick section extending from bedrock to the surface of the deposits (Figure S1E).

The other sample UW264, is from a Howiesons Poort stratum at Kathu Pan 6 in the Northern Cape. Kathu Pan (Figure S1D) is a former ethereal lake, or vlei. A stratigraphic section, revealed by a sinkhole in the limestone bedrock, includes a 1 m-thick sandy layer, containing HP artifacts, sandwiched between two calcareous layers. The sample was collected from the center of this layer. Sinkholes in the limestone substratum have yielded exposed stratigraphic sections at various places around the lake, extending in age from the Early to Middle to Late Stone Age. Originally investigated by Beaumont (Beaumont and Morris 1990), the sites have recently been the focus of chronological studies, mostly directed at the Early Stone Age (Wilkins et al 2012, Porat et al. 2010).

3. Methods

The samples were collected by driving opaque tubes into exposed profiles and capping the ends. The exposed ends were removed in the laboratory under red-orange light and used for dose rate measurements. The unexposed portion was sieved to separate the sand fraction, which was then treated with HCl and H₂O₂ to remove carbonates and reduce organics. For quartz the 180-212 μm fraction was treated with 48% HF for 40 minutes, rinsed with HCL and water, and then placed in a solution of lithium metatungstate of density 2.67 g/cm³. After this procedure, grains lighter than 2.67 are considered mostly quartz. Any feldspar contaminants can be identified during measurement by application of infrared stimulation. A portion of the 180-212 μm fraction was set aside to isolate potassium feldspars. This followed the same procedure except no HF etch was performed, and the density solution was set at 2.58 specific gravity. Grains lighter than 2.58 were used for measurement.

Luminescence was stimulated on single-grains of quartz using a 532nm laser delivering 45 W/cm², attached to a Risø TL-DA-15 reader. (A small number of grains were measured on a Risø TL/DA-20). Detection was through 7.5 mm U340 (ultraviolet) filters. Exposure was for 0.8 s on each grain at 125°C, with the first 0.05s used for analysis and the last 0.25s for background. Radiation dose was provided by a ⁹⁰Sr/⁹⁰Y beta dose, which delivers a dose on average of about 0.08 Gy/s. The source, however, produces a dose gradient, which differs in the UW equipment by a factor of 2 across a single-grain disk. This was accounted for in measuring the dose given to each grain. Equivalent dose (D_e) was determined using the single-aliquot regeneration method (Wintle and Murray 2006), employing nine regeneration doses including a zero dose, two doses of the same magnitude at beginning and end, and two doses which were preceded by an infrared stimulation. Regeneration doses as high as 250 Gy were employed. No optical wash at the end of each SAR sequence was employed, contrary to both Jacobs et al. (2008) and Tribolo et al. (2013), because there was no evidence for charge buildup through the measurement cycle for most grains. The test dose was about 3 Gy. The preheat employed was variable as discussed later. Luminescence growth with dose was described using a saturating exponential function. In some cases a saturating exponential plus linear function was employed if a regular saturating exponential could not fit the data, but this was not used in cases where the reason for poor fit was because the natural signal was higher than any of the regeneration signals.

Grains were rejected for analysis if they failed any of the following criteria: (1) the ratio between the corrected signals for the two identical regeneration doses (recycle test) fell outside 0.8-1.2 range, a range which encompasses 2 standard errors for most grains; (2) corrected signals from the zero dose (recuperation) of more than 10% of the natural signal and visually showing a decaying signal; (3) failure of the corrected natural signal to intersect the growth curve because it was higher than the corrected signal from the largest regeneration dose or the derived D_e is more than twice the value of the characteristic dose, D_0 , of the exponential function, which implies the sample is close to saturation (Wintle and Murray 2006); (4) presence of feldspar as indicated by visually reduced signals from the two points on the growth curve where the OSL measurement was preceded by infrared stimulation; (5) D_e values that were not significantly different from zero; and 6) poor signals, as indicated by net natural signals less than three times the background standard deviation, or test dose signals with errors of more than 40%. These criteria are similar to those used by Jacobs et al. (2008) and presumably by Tribolo et al. (2013), although they did not specify anything beyond “standard criteria”. One important difference is in criteria 3, where neither study mentions whether they rejected any grains because the D_e was greater than 2 D_0 . This might not always be a valid criterion, but there was no relationship in this study between high D_0 values and D_e , suggesting that using this criteria did not bias against older grains. A dose recovery test, where D_e is determined on a zeroed sample given a known dose, was performed on each sample using different preheats and different administered doses, as discussed later. Before the administered dose, the samples were zeroed by exposure to the laser for 1 s at 125°C.

Luminescence in feldspars was stimulated by a 150mW/cm² 830nm IR laser, attached to a Risø TL/OSL DA-20 reader, set to 30% power and passed through an RG 780 filter. Emission was collected through a blue filter pack, allowing transmission in the 350-450nm range. IRSL measurements were made at 50°C using a preheat of 250°C for 1 minute. Exposure time was the same as for quartz. Test

151 doses were about 6 Gy. The beta source on this instrument delivers an average dose of 1.2 Gy/s, with a
152 similar gradient ranging over two orders of magnitude. D_e determination followed the same procedure
153 as for quartz and used the same criteria for selection except of course criteria 4. Fading rates were
154 measured on single grains following Auclair et al. (2003), and fading correction followed Huntley and
155 Lamothe (2001). Multi-grain aliquots for feldspars, containing about 1000 grains on each aliquot, were
156 also used, because for the Diepkloof samples, the feldspars were too insensitive to obtain many grains
157 that passed the criteria. The multi-grain aliquots were measured for equivalent dose on either the
158 TL/OSL DA-15 or TL/OSL DA-20 reader. Stimulation was with either 830 nm laser diodes (DA-15) or 875
159 nm LEDs (DA-20). Emission was through the blue filter pack. Stimulation was for 100s successively at
160 50°C and 290°C, with the first 2 s used for analysis and the last 6 s for background. The post-IR IR signal
161 at 290°C was employed to limit fading (Bueyaert et al. 2009) and preferred over lower stimulation
162 temperatures because of the age of the samples. Fading tests were performed on one set of samples
163 (about half the aliquots) but not on a second set to conserve machine time. No feldspars were
164 measured for UW264

165 While preparation of the 180-212 μm grains and all luminescence measurements were done in
166 2012-2013, dose rate measurements were not redone and results obtained in the late 1990s were used.
167 Dose rate on the samples was measured at that time with high resolution gamma spectrometry. Alpha
168 and beta contributions were adjusted by attenuation factors given by Mejdahl (1979) and by etch depth
169 (calculated experimentally to be about 9 μm). The feldspar contains an internal dose rate, which was
170 not measured. Rather, the content was estimated from other samples with similar sensitivity (Feathers
171 2012), $3 \pm 2\%$ for the insensitive Diepkloof samples and $8 \pm 3\%$ for the somewhat sensitive UW287. K
172 content correlates well with sensitivity.

173 Radioactivity was also measured on various rocks, ash or charcoal lenses, salt accumulations,
174 and other indications of possible radioactive heterogeneity which were within 30 cm of the samples.
175 These were measured with alpha counting, flame photometry and delayed neutron analysis (for ^{238}U
176 concentrations). The contributions of these to the total gamma dose rate were estimated in proportion
177 to their size and distance from the sample. They accounted for 20% of the total gamma dose rate for
178 UW247, 60% for UW325, and 15% for UW260. On-site dosimeters of $\text{CaSO}_4(\text{Dy})$ were left at each
179 sample location but never retrieved. Cosmic dose rate was determined after Prescott and Hutton (1988)
180 and also included a correction, at Diepkloof and Hollow Rock Shelter, for the configuration of the
181 shelter, taking into account the thickness of overburden and the zenith angles of unobstructed
182 penetration using equations of Crookes and Rastin (1972). The values for cosmic dose rate obtained for
183 Diepkloof were similar to those obtained by Jacobs et al. (2008) and Tribolo et al. (2013). Because the
184 Diepkloof samples contained abundant organic matter and because it absorbs dose at a different rate
185 than the mineral constituents similarly to water, moisture content was determined by loss on ignition,
186 heating to 1000°C. The samples did not react with HCl, so negligible carbonate was present. The
187 inorganic fractions are largely sand and silt, with little clay.

188

189

3. Dose Rate

Table 1 gives the gamma spectrometer results. Some disequilibrium in the ^{238}U decay chain is apparent for all samples except UW325. For age calculation, the level of disequilibrium was considered constant through time. Also given is the measured moisture content. Table 2 gives the radioactivity of other constituents that were close enough to the samples to contribute to the gamma dose rate. Finally, Table 3 gives total dose rates for both quartz and K- feldspar. The alpha dose rate to quartz was determined from attenuation considerations and the error should encompass any significant internal dose. The total dose rates are similar to those measured by Tribolo et al. (2013) (1.1-1.4 Gy/ka), but somewhat lower than those measured by Jacobs et al. (2008) (1.5-2.2 Gy/ka). The main difference seems to be in the beta dose rate (this paper ranging from 0.51-0.99, for Tribolo et al, ranging from 0.72-0.86, and for Jacobs et al., ranging from 0.94-1.48 Gy/ka). Tribolo et al. (2013) argued that some of the high beta dose measured by Jacobs et al. is due to a recalculation based on radioactivity heterogeneity, a procedure questioned by some (Guérin et al. 2013), but Tribolo et al. concede that higher dose rates cannot only be accounted for by this. The differences may simply be due to difference in sampling location. My analysis also used higher moisture contents, based on measured amounts and including organic contents, than either Tribolo et al or Jacobs et al, both of whom used 5 ± 2 percent.

4. Equivalent Dose and Age – Quartz

4.1. Acceptance rates

Acceptance rates are first investigated. Table 4 gives the number of grains measured, the number of rejections for various reasons, the number accepted (passing all criteria), the rate of acceptance, and the ratio of accepted grains to those rejected either because the natural signal does not intersect the growth curve, the natural signal being higher than the highest regeneration signal, or because the signal is close to saturation. The latter rejected grains are called “too high” for convenience. The acceptance rates range from 6 to 16%, which is not uncommon for quartz. What is more unusual is the large number of “too high” rejections, more in every case than the accepted grains. The ratio of accepted to “too high” decreases with depth at Diepkloof. Tribolo et al. (2013) do not give acceptance rates, but Jacobs et al. (2008) report acceptance rates ranging from 29 to 42%. The ratio of accepted to “too high” ranged from 2.5 to 12. These values are much higher than those reported for the Diepkloof samples here. They might have accepted some near saturated grains that I would have rejected, but this cannot explain such a wide discrepancy. Table 4 does not include 488 grains from UW247 and UW325 measured on the DA20 machine and also using a higher test dose (12 Gy). The acceptance rate for these grains was 30%, similar to that obtained by Jacobs et al. (2008), who also used a test dose of 9 Gy. The higher test dose may account for some of the increase in acceptance rate, but most must be due to different sensitivity of Risø readers. The accepted to “too high” ratio from the DA20 data was 1.0, still much lower than that reported by Jacobs et al. (2008). Perhaps they used higher regeneration doses, but none of their D_e values, as judged from their radial plots, exceeded the 250 Gy maximum regeneration dose used here.

Bailey et al. (2005) argue that the “too high” phenomenon is the result of competitive effects from an unstable reservoir center in quartz. The effect is the result of high laboratory dose rates and may be significant in samples more than 40 ka. They recommend using pulsed irradiation, where the dose is delivered in small increments each followed by a preheat. This was attempted some time ago on 90-125µm grains from UW264 and UW287. A time-consuming process, it made no difference in D_e for UW264 (D_e of about 55 ± 3 Gy), but for UW287 it increased the central age dose from 80 ± 3 to 98 ± 4 Gy. An alternative explanation to such “dose quenching” is that the sensitivity of the test dose signal increases faster than the regeneration dose signal at high doses because of saturation (Duller 2012). If this were true, the uncorrected luminescence signal should increase with dose even if the corrected luminescence signal starts to decrease (Duller 2012). Surveying a sample of grains from UW247 shows this to be the case on most of them.

4.2. Dose Recovery

The dose recovery data were also instructive in this regard. Table 4 shows the rejection/acceptance data for two sets of dose recovery measurements, one using an administered dose of 1000sβ, or about 80 Gy, and the other using an administered dose of 500sβ, or about 40 Gy. The data are combined for all samples. The 500s data show a higher acceptance rate and a much higher accepted/“too high” ratio. The dose recovery using the 1000s dose consistently underestimated the given value, yielding an average derived dose of about 750s. The dose recovery using the 500s dose, on the hand, yielded a much more acceptable dose of about 510s. This raises the possibility the D_e of the samples might be underestimated (Duller 2012). This will be further discussed later.

4.3. Preheat

The SAR protocol for quartz requires a proper thermal treatment, which can vary from sample to sample. Jacobs et al. (2008) used a 260°C 10s preheat after the regeneration doses and a 220°C 5 s preheat after the test dose for all samples. Tribolo et al. (2012) argued that the proper thermal treatment varied from sample to sample and thus they used variable combinations. Both groups applied an optical wash at elevated temperature after each cycle, a step not found necessary here. Finding the proper thermal treatment for single-grain data is difficult, because what is proper likely varies from grain to grain. The best one can do is to find something close to an average optimum (Jacobs et al. 2008). The error in doing that should show up in the over-dispersion, σ_b , of the dose recovery test. If one uses that to evaluate the sample D_e distributions, then determining the proper thermal treatment may not be so critical.

For the samples reported here, three preheats (all for 10s) after the regeneration doses were examined. The preheat after the test dose was kept constant at 200°C cut. The measurements were done on single-grains. Results are tabulated in Table 5 and plotted in Figure S2. Looking at the dose recovery data, the 240°C preheat seems to lead to an overestimation of the dose. That seems to be the case, as well, for the D_e determinations on two samples, UW247 and UW264, but it is not the case for UW260 and UW287, which show D_e decreasing with increasing preheat, nor for UW325, where there is no significant difference in D_e among preheats. Despite these observations, there is no systematic trend

of either increasing or decreasing dose with preheat for the dose recovery or for all samples. In fact, when combining the results from all three preheats, the recovered dose, from the central age model, agrees very well with the administered dose for all samples and for the Diepkloof samples on their own. These results seem to demonstrate the variation in proper thermal treatments among grains, results that are influenced to no small degree by sample size. Note also that the over-dispersion values do not differ much among preheat treatments. Adopting one particular preheat will not lower the over-dispersion. Consequently, results from all preheats are combined. That the error in doing so is not substantial can be gleaned from the finite mixture model, to be discussed momentarily. The D_e values from the 260°C preheat seem low for every sample, but running the finite mixture model either including or not including the 260°C preheat data makes no great difference in the number of components, their D_e values or their relative proportions for any sample.

4.4. D_e distributions

Figure 1 provides decay and growth curves for four Diepkloof samples. Table 6 provides D_e and σ_b of the distributions from all preheats, using the central age model (Galbraith and Roberts 2012), which computes a central tendency and over-dispersion. The σ_b is quite high, much more so than the 20% from dose recovery and what might be considered a minimum for a single-aged distribution. These σ_b values are much higher than those obtained for Diepkloof by either Jacobs et al. (2008), ranging from 16-32%, or Tribolo et al. (2013), ranging from 29-37%. To look at the structure of the distributions, the finite mixture model (Galbraith and Roberts 2012) was applied. This model divides the distribution into single-aged components, each having an assumed normal distribution and σ_b typical of a single-aged sample. I used the 20% σ_b from dose recovery for the typical value. Best fits were evaluated by reference to maximum log likelihood and Bayes Information Criterion (Galbraith and Roberts 2012).

Table 7 gives the results from applying the finite mixture model. To visualize the distributions, Figure 2 displays radial plots for each sample. The two shaded regions represent the two largest components. The graphs are centered at the central age D_e . To further illustrate the similarity in distributions representing different preheats, the open circles represent values obtained after a 220 or 240° preheat, and the solid triangles represent values obtained after a 260°C preheat. For all samples the smallest components were the ones with the lowest D_e values, containing for the most part negligible numbers of grains. These are represented by a line in the plots.

From the radial graphs, the distributions of all samples appear bimodal. Very few grains, if any fall in the region between the statistical range of the two main components, with the possible exception of UW287. Because they are bimodal, using the central age model for the estimation of the D_e corresponding to the depositional age is not appropriate. A possible reason for the high over-dispersion is inhomogeneity in the beta dose rate at a single-grain scale. This possibility was examined by adjusting the dose rate to reflect K-feldspar hotspots (Mayya et al. 2006) or by halving the beta dose rate to reflect nearness to low dose rate material (such as organics), and dividing the low value component by the adjusted dose rate to see if that would bring it into agreement with the age of the high value component. In neither case did this agreement obtain, ruling out micro-dosimetry as a major cause for the over-dispersion. Partial bleaching is another possibility, but the skewness and

discontinuity of the distributions do not suggest that this is a factor. Neither Jacobs et al. (2008) nor Tribolo et al. (2013) argued for partial bleaching. A more likely explanation is post-depositional movement downwards. The strongest cases can be made for UW247, UW325, and UW287, where more than 75% of the grains fell in the older component. For UW247 and UW325, the grains could have originated in the overlying Late Stone Age deposits. It is less clear where the younger grains for UW287 came from, because it was such a shallow deposit, but it could be from surface turbation. Much weaker cases can be made for UW260 and UW264 because the two components were more evenly distributed. Table 7 gives the ages for the oldest component for UW247, UW325 and UW287, and for the two oldest components for UW260 and UW264.

The two second component ages from UW264 and UW260 seem much too young, and difficult to explain by post-depositional processes. UW264 was between two calcareous layers but contained little carbonate itself. The second component of UW260 gives a Late Stone Age age, but how so many grains could have migrated down through the stratum above it (containing UW247 and UW325, which did not show that much post-depositional movement) is uncertain. The relatively clear delineation of the stratigraphy does not suggest such mass movement either. Might the underestimations have a different cause?

4.5. Fast Ratio

Similar to results reported here, Duller (2012) found that for high administered doses, the recovered dose consistently underestimated the given dose (his Figure 3). To address this problem, Duller (2012) adapts the Fast Ratio first developed by Durcan and Duller (2011) for multi-grain aliquots. He makes adjustments for single-grain signals, where the effective stimulation power delivered to the sample is not precisely known. The Fast Ratio is the ratio of the intensity of the initial signal on an OSL decay curve, which should be dominated by the fast component, and another integral further along the decay curve chosen to represent the contribution of the medium component, with a background subtracted from both signals (Durcan and Duller 2011). By restricting analysis to those grains with high Fast Ratios, and therefore dominated by the fast component, he found that not only did the recovered dose (for high doses) approach the given dose, but that the over-dispersion among grains was near zero (his Figure 6). He calculated the appropriate integration periods based on a b value for each component, which is the product of the photo-ionization cross-section and the stimulation light intensity and which is obtained by fitting exponential functions to the decay curve.

For this paper, I used the component fitting routine available on Risø Luminescence Analyst Version 4.12. The single-grain curves were obtained on the three Diepkloof samples using 40% laser power and a 4s stimulation time. Only two components were fit to the curves; for the great majority of grains adding a third component did not significantly improve the fit. For 50 grains the average b value for the fast component was 10.3 ± 3.4 and for the medium/slow component 2.1 ± 0.7 . These values correspond well to those of Duller (2012): 11.1 ± 4.3 and 1.94 ± 1.0 . The first channel, corresponding to 0.02s, was used for the fast component, and the middle component was determined by scaling the integral used by Duller according to the difference in power used (90 instead of 25%). The integral used for the middle component was 0.18-0.22 seconds, which is identical to the values used by Jacobs et al.

(2013) in using the Fast Ratio, also with 90% power, but calculating the integrals in a much more complicated fashion (Z. Jacobs, personal communication): 0.02 and 0.18-0.22s. Given the difficulties in calculating these integrals because of the uncertain power delivery of the green laser, these values can only be approximate.

If using the Fast Ratio as a rejection criterion, the question arises as to how small a ratio is acceptable. Duller (2012) recommended using only grains with Fast Ratio's >20, but as Jacobs et al. (2013) point out that leaves a very small sample size. Duller (2012) also suggested using a plateau test, calculating D_e distributions using a variety of cut-off values and seeing over what range those values are stabilized. This is the approach taken here.

Looking at the dose recovery data first, Figure 3 shows the central age D_e as a function of Fast Ratio for both the 80 Gy (1000s) and 40 Gy (500s) administered doses. Only Fast Ratio values up to 7 were used, because sample size became quite small for larger ratios. The values are the minimum Fast Ratio a grain must have to be included. For the 80 Gy dose recovery, the recovered dose/administered dose ratio increases up to about a Fast Ratio of 2-2.5, where it begins to plateau at a value much closer to 1 than was the case using all the data. For the 40 Gy dose recovery the recovered/administered ratio forms a plateau near one at all Fast Ratios. Figure 4 shows the over-dispersion as a function of Fast Ratio. The over-dispersion declines for both recovery tests, but more so for the 80Gy test. The over-dispersion does not drop to zero, however, as it did for Duller's data (2012). The data in this paper suggest that grains showing a low Fast Ratio are more likely to underestimate the dose and to increase over-dispersion, at least for samples with high D_e values. Why might this be the case is not obvious. The medium component is known to have a slightly higher saturation value than the fast component and one of slower components has a much higher saturation level (Singarayer and Bailey 2003). If what is being called the medium component here has a higher saturation level than the fast component, then at high doses, the medium component may provide enough signal for the regeneration points to allow the natural signal to intersect the growth curve, but the natural signal will be depleted because of less contribution from the saturated fast component. This would only have much effect from samples dominated by the slower component. The fact that the smaller test dose does not result in saturation while the regeneration doses do also plays a role. This issue is explored in more detail in Feathers and Pagonis (2015).

Looking at the sample data, Figure 5 show that all five samples show an increase in D_e as the Fast Ratio increases, plateauing after about 2. The over-dispersion, Figure 6, also decreases to a plateau in about the same place. This suggests that the central age model using all grains is likely to underestimate the true paleodose. The finite mixture model was rerun on the samples with the low Fast Ratio grains (<2) removed, and using for a single-age distribution an over-dispersion of 15%, instead of 20%, reflecting the decrease in over-dispersion for dose recovery. Table 8 shows that all components have increased D_e values. UW247 and UW287 appear to be near single-grain distributions. UW325 and UW260 have one dominant component. The younger components can be interpreted as representing movement downward, mostly from MSA strata and not from the LSA overburden. UW264 also has one dominant component. Table 9 translates these D_e estimates into age by dividing through by the bulk dose rate.

6. Equivalent dose and age -- feldspar

Dating of K-feldspar, which is known to have a higher saturation value, was performed for the three samples at Diepkloof and for UW287 at Hollow Rock Shelter. Single-grain dating was attempted first on 180-212 μm grains. Table 10 shows the acceptance data. The three Diepkloof samples have very low acceptance rates, too low to make single-grain dating time efficient. The reason for low acceptance rates is likely because whatever the grains were that floated in the 2.58 specific gravity solution, they were not pure K-feldspars, but rather something with relatively low K concentrations. For the Diepkloof samples, analysis continued with multi-grain aliquots. For UW287, the acceptance rate was relatively high, but lower than many feldspar samples. Significantly, the number of "too high" grains was much lower than the number of accepted grains. Only one of the 32 accepted grains could not be corrected for fading, because the fading rate ($g=14$) was too high. The weighted average fading rate for all grains was only $3.2 \pm 0.9 \%$ /decade. Equivalent dose from the central age model was 51 ± 6 Gy, with 59% over-dispersion. Figure S3a gives a typical decay and growth curve and fading data. When corrected for fading, the 31 grains that could be corrected, produced an age, from the central age model, of 39.4 ± 5.4 ka, with the over-dispersion reduced to 46.8%. The finite mixture model produced two components, the older of which with 67.4% of the grains gave an age of 57.5 ± 7.4 ka, younger than the quartz age, although within 2-sigma. Whatever the cause of this discrepancy, perhaps small sample size for the feldspars, the feldspars are not producing older ages than the quartz for this sample.

This is not the case for the multi-grain feldspar aliquots for the Diepkloof samples. Stimulation involved an initial IR stimulation at 50°C followed by a second one at 290°C (Post-IR IR) (see Figure S3b). The measured g -value for the 50°C stimulation was 5.2 ± 0.78 (weighted average), while for the 290°C stimulation it was 1.7 ± 0.26 . The number of aliquots measured is small, because the samples did not yield enough K-feldspar grains. Dose recovery for two aliquots from two samples yielded measured/administered ratios of 1.07 ± 0.04 for 50°C stimulation and 1.15 ± 0.14 for 290°C (weighted averages). Table 11 gives the corrected age, over-dispersion and the corresponding quartz age (from Table 7). Except for UW260, the K-feldspar ages are substantially older, but they have high over-dispersion. UW260 has lower over-dispersion, and the age from the 290°C stimulation, is within 2σ of the quartz age. The high over-dispersion could be a sample-size effect, but also from variable fading rates, since they were assumed for half the aliquots. This explains why the over-dispersion for the 290°C stimulations are less dispersed in general, because the fading rate is much lower. The higher ages for the K-feldspar from UW247 and UW325 could be due to partial bleaching, because feldspars do not bleach as rapidly as quartz, or mixing. The feldspar ages should be considered maximums.

7. Discussion

Assuming UW247 is late Howiesons Poort, the age from Table 10 is older than any of the HP samples of Jacobs et al. (2008) and older than all LHP samples of Tribolo et al. (2013, except for one burned lithic date from the bottom of LHP layers. That date of 71 ± 8 ka is in good agreement with UW247. The date from UW325 is out of stratigraphic order, but within 2-sigma of UW247. It is also the most mixed. The age is in the range of HP dates from Jacobs et al. (2008) and of the LHP-IHP transition of Tribolo et al. (2013). The age could be an underestimation, and the feldspar analysis suggests an

older age. The age for UW260 agrees with the IHP ages of Tribolo et al. (2013), most of which were obtained from burned lithics, but two were sediments near the bottom of IHP sediments. UW260 is older than even the Still Bay dates of Jacobs et al. (2008). In general the dates for HP reported here are older than those of Jacobs et al. (2008), and are slightly older or in agreement with those of Tribolo et al. (2013), especially their dates from burned lithics, which may not have the same saturation problems. Age comparisons are given in Table 12. Differences with Jacobs et al. are hard to reconcile. The greatest difference is in dose rate, which is probably not due to sample location, because ages are also different. Diepkloof is a complicated site and further work to isolate any inaccuracies in the published dose rate values is warranted. The University of Washington dose rate values were not reassessed in this paper from their original derivation some 15 years ago. On the other hand, the ages are not so different among laboratories that Howiesons Poort at Diepkloof cannot fit into the general chronological range of the industry in other parts of Africa. The narrow time frame postulated by Jacobs et al. (2008), however, may not be sustainable, particularly given the wide chronological range provided by Tribolo et al. (2013), which is partially supported by the present work.

The age for UW287 from the Still Bay component at Hollow Rock Shelter is younger than the SB dates at Diepkloof of Tribolo et al. (2013), all of which were obtained on burned lithics. It is in the same ballpark as the SB dates at Diepkloof of Jacobs et al. (2008), and for their SB dates from across South Africa (Jacobs et al. (2013). It is also in the same age range as the preliminary OSL dates from Hollow Rock Shelter reported by Högberg and Larsson (2011). The age for UW264 is in the range of IHP at Diepkloof (Tribolo et al. 2013), but it is slightly older, although not outside error terms, for the range of HP reported by Jacobs et al. (2008) from across South Africa.

8. Conclusions

A reanalysis of sediment samples collected in 1995 from Diepkloof Rock Shelter has shown that the quartz samples are near saturation and have a large number of grains rejected because the natural signal either does not intersect the dose response curve or intersects it high on the saturating curve. Dose recovery experiments show that for high administered doses the recovered dose is underestimated. Application of a Fast Ratio criterion (Duller 2012) preferentially removes grains with low D_e values, correcting for the underestimation of the dose recovery and increasing average D_e values for the samples. The same behavior is noted for two other samples from Hollow Rock Shelter and Kathu Pan 6. The adjusted ages are older than the ages that were computed using all the data, although in some cases not by much. This is because the largest component was used as the basis for age, and applying the fast ratio criteria mainly eliminated grains that were concentrated in younger components.

Comparison with other ages obtained for Diepkloof samples shows that the ages reported here are older than comparable sediment samples from Jacobs et al. (2008) and slightly older or equivalent to sediment and burned lithic samples from Tribolo et al. (2013). Best agreement is with the burned lithic samples. Neither Jacobs et al or Tribolo et al. considered the saturation issue, but the disagreements could also reflect other differences, such as in dose rate.

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541

542 **Figure captions**

543 1. a) Natural decay curves for single grains showing variation in fast ratio: UW260, fast ratio = 20;
 544 UW260, fast ratio = 9.5; UW260, fast ratio = 5.2; UW247, fast ratio = 1.0. None of these grains showed
 545 any sensitivity to stimulation with IR. b) growth curves for two of these grains: squares, UW247, fast
 546 ratio =1.0; triangles, UW260, fast ratio = 20. Circles are the natural signals, the dotted lines being the
 547 interpolation into the growth curves at the equivalent dose. Symbols at the same dose represent the
 548 recycle test.

549 2. Radial graphs (see text) for all five samples. The two highlighted references are the two largest
 550 components of the finite mixture model. The third line represents the third, smallest component. The
 551 graphs are centered on the equivalent dose from the central age model. Open circles represent values
 552 obtained after a 220 or 240° preheat, and the solid triangles represent values obtained after a 260°C
 553 preheat.

554 3. Recovered dose as a function of fast ratio. The fast ratio values are the minimum acceptable, so that
 555 the number of accepted grains increases cumulatively to the right. The dotted lines show the 1:1 ratio

556 between administered dose and recovered dose. Recovered dose from central age model; error bars
557 are one standard error.

558 4. Over-dispersion from dose recovery data as a function of fast ratio. Over-dispersion calculated from
559 the central age model; error bars are one standard error.

560 5. Sample equivalent dose as a function of fast ratio. Equivalent dose from the central age model; error
561 bars are one standard error.

562 6. Sample over-dispersion as a function of fast ratio. Over-dispersion calculated from the central age
563 model; error bars are one standard error.

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565 S1. Location information. A) Topographic map of South Africa showing location of sites discussed in
566 text. B) Plan view of excavation at Diepkloof highlighting 1973 test pit where samples from this study
567 were drawn. C) Schematic of north wall of test pit showing sample locations, from top to bottom,
568 UW247, UW325 and UW260. D) Plan view of Kathu Pan archaeological area, showing location of Kathu
569 Pan 6. A profile drawing showing the exact location of UW264 is not available. E) Photo of location of
570 UW287 in Hollow Rock Shelter. The end of a PVC pipe in the excavated profile just below the surface
571 shows the location of the sample.

572 S2. Equivalent dose as a function of preheat for each sample and dose recovery ratio
573 (administered/derived) for all samples combined. The purple line represents a dose recovery ratio of 1.

574 S3. (a) Decay curve, growth curve and fading curve for a single K-feldspar grain from UW287, Hollow
575 Rock Shelter. (b) Decay curves, growth curves and fading curves for 50°C and 290°C stimulations for a
576 multi-grain aliquot from Diepkloof (UW260).

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Table 1. Gamma spectrometry data (ppm) plus moisture content

Sample	Depth (m)	Th-series	U-series ^{238}U - ^{234}U	U-series ^{230}Th - ^{226}Ra	U-series ^{222}Ra - ^{206}Pb	^{40}K	Moisture (%)
UW247	0.45	2.2±0.1	1.2±0.3	0.6±0.1	0.6±0.1	0.75±0.01	16±5
UW325	1.25	5.2±0.3	1.5±0.2	1.5±0.2	1.5±0.2	1.46±0.05	25±5
UW260	1.51	5.2±0.2	1.8±0.2	1.8±0.2	1.3±0.1	1.27±0.03	30±5
UW287	5.0	8.4±0.3	2.0±0.2	2.0±0.2	1.4±0.2	0.39±0.04	6±3
UW264	2.55	2.2±0.2	1.1±0.6	1.1±0.6	0.6±0.1	0.40±0.02	15±5

Table 2. Delayed neutron analysis, alpha counting, and flame photometry results.

Sample	constituent	DNA (ppm)	Alpha Counting (ppm of parent)		Flame photometry (%)
		^{238}U	Th-series	U-series	K
UW247	ash	1.2±0.2	4.3±0.8	0.9±0.1	2.42±0.06
	carbon	0.6±0.2	2.1±0.6	0.63±0.07	2.79±0.06
	rocks		1.3±0.4	0.70±0.06	0.30±0.01
	salt		1.0±0.3	0.38±0.05	3.65±0.20
UW325	salt		1.6±0.3	0.26±0.04	0.63±0.01
	rocks		3.6±0.7	0.52±0.07	0.25±0.01
	ash	1.8±0.2	4.7±0.7	1.1±0.1	1.07±0.03
	sand lens		4.3±0.7	1.7±0.1	0.76±0.04
	carbon		4.8±0.7	1.3±0.1	1.23±0.05
UW260	rock		4.8±0.8	1.2±0.1	0.37±0.01
	ash	2.6±0.2	5.8±0.9	1.6±0.1	1.87±0.10
UW287	bedrock		9.4±1.3	1.5±0.2	0.29±0.01
UW264	adjacent stratum		3.6±0.6	0.32±0.06	0.37±0.01

Table 3. Total dose rates (Gy/ka)

Sample	site	Depth (m)	alpha	beta	gamma	cosmic	total
quartz							
UW247	Diepkloof	0.45	0.01±0.01	0.51±0.03	0.35±0.02	0.03±0.01	0.89±0.04
UW325	Diepkloof	1.25	0.01±0.01	0.99±0.05	0.48±0.03	0.02±0.01	1.49±0.06
UW260	Diepkloof	1.51	0.01±0.01	0.93±0.05	0.56±0.03	0.02±0.01	1.52±0.06
UW287	Hollow Rock	5	0.01±0.01	0.61±0.04	0.53±0.05	0.13±0.03	1.28±0.07
UW264	Kathu Pan 6	2.55	0.01±0.01	0.37±0.03	0.25±0.02	0.15±0.03	0.77±0.05
K-feldspars							
UW247	Diepkloof	0.45	0.03±0.01	0.7±0.1	0.35±0.02	0.03±0.01	1.1±0.1
UW325	Diepkloof	1.25	0.07±0.03	1.1±0.1	0.48±0.03	0.02±0.01	1.7±0.1
UW260	Diepkloof	1.51	0.07±0.03	1.1±0.1	0.56±0.03	0.02±0.01	1.7±0.1
UW287	Hollow Rock	5	0.12±0.05	1.0±0.2	0.53±0.05	0.13±0.03	1.8±0.2

Conversion factors from Guérin et al. (2013); attenuation factors from Mejdahl (1979)

Table 4. Quartz acceptance data.

Sample	# measured	Rejection criteria						Accepted	Acceptance rate (%)	Ratio accept/ too high
		Low signal	recycle	Too high	feldspars	recuperation	Zero dose			
UW247	1371	794	131	226	4	12	6	198	14.4	0.88
UW325	1671	1174	141	191	4	5	3	153	9.2	0.80
UW260	1959	1455	104	241	11	17	3	128	6.5	0.53
UW287	1961	1444	92	229	8	6	7	175	8.9	0.76
UW264	1261	805	61	207	1	5	2	196	15.5	0.95
sβ*	Dose Recovery Data									
1000	1688	1109	92	222	13	7	1	224	13.4	1.01
500	1556	1043	71	174	5	14	4	245	15.7	1.41

* Source delivers about 0.08 Gy/s, so 1000sβ equals about 80 Gy, and 500sβ equals about 40 Gy.

Table 5. Preheat data.

sample	220°C 10s			240°C 10s			260°C 10s		
	N	D _e (Gy)	σ _b (%)	N	D _e (Gy)	σ _b (%)	N	D _e (Gy)	σ _b (%)
UW247	94	52±3	45±5	49	63±5	45±7	55	54±4	36±6
UW325	41	68±9	65±11	48	67±9	68±11	64	58±5	55±7
UW260	33	96±9	42±9	50	78±7	55±8	45	57±6	55±8
UW287	79	80±6	62±7	49	65±8	73±10	47	52±6	66±9
UW264	29	37±3	24±10	93	46±2	36±5	74	38±3	53±6
Dose recovery (administered/recovered)									
All	67	0.99±0.04	15±4	75	0.83±0.04	20±4	103	1.07±0.03	17±3
Dose recovery --all preheats									
All Diepkloof samples				140	0.97±0.03	20±3			
All samples				245	0.98±0.02	20±2			

Table 6. D_e from central age model.

Sample	N	D_e (Gy)	σ_b (%)
UW247	198	55±2	43±3
UW325	153	63±4	62±6
UW260	128	74±4	57±5
UW287	175	67±4	68±5
UW264	196	41±2	43±4

Table 7. D_e values and ages from finite mixture model

Sample	component	D_e (Gy)	Proportion (%)	Age (ka)
UW247	1	25±4	9±4	
	2	62±2	92±4	63±4
UW325	1	6±1	2±1	
	2	28±4	19±6	
	3	86±5	79±6	58±4
UW260	1	11±3	4±2	
	2	51±4	43±7	33±3
	3	116±7	53±7	76±6
UW287	1	6±1	3±2	
	2	29±3	21±4	
	3	94±4	76±4	73±6
UW264	1	6±1	2±1	
	2	31±4	36±14	40±6
	3	54±4	62±14	

Table 8. D_e values from finite mixture model after low fast ratio grains removed

Sample	component	D_e (Gy)	Proportion (%)
UW247	1	43±7	14.8
	2	71±3	85.2
UW325	1	13±2	4.2
	2	55±4	38.6
	3	94±4	57.2
UW260	1	37±4	26.9
	2	61±4	73.1
UW287	1	23±6	7.7
	2	94±4	92.3
UW264	1	37±4	39.2
	2	61±4	60.8

Table 9. Ages after low fast ratio grains removed.

Sample	Provenience	Basis for age	Age (ka)
UW247	Diepkloof 45 cm	Largest component	73 ± 5
UW325	Diepfloof 125cm	Largest component	63 ± 4
UW260	Diepkloof 151cm	Largest component	80 ± 6
UW287	Hollow Rock Shelter	Largest component	73 ± 6
UW264	Kathu Pan 6	Largest component	79 ± 8

Table 10. Feldspar acceptance data.

Sample	# measured	Rejection criteria					Accepted	Acceptance rate (%)	Ratio accept/ too high
		Low signal	recycle	Too high	recuperation	Zero dose			
UW247	194	186	5				3	1.54	
UIW325	196	188	6	1			1	0.51	1.0
UW260	190	188				1	1	0.53	
UW287	192	125	18	6		11	32	16.67	5.3

Table 11. Feldspar data*.

Sample	Stimulation	N	D _e (Gy)	g-value	Corrected age (ka)	σ _b (%)	Quartz age (ka)
UW247	50°C	4	84±20	5.3±0.6	123±34	49±21	73 ± 5
	290°C	4	127±21	1.8±0.6	129±21	25±14	
UIW325	50°C	7	121±5	5.1±0.5	124±19	33±12	63 ± 4
	290°C	7	157±9	1.8±0.6	100±9	16±8	
UW260	50°C	6	127±20	5.3±0.8	117±5	0	80 ± 6
	290°C	6	147±27	2.8±0.1	104±6	89±6	

D_e and age values are from the central age model; g-values are weighted averages.

Table 12 Comparison of ages (weighted means, from central age model, in ka).

Cultural attribution	Diepkloof			
	Högberg and Larsson (2011)	Jacobs et al. (2008)	Tribolo et al. (2013)	This paper
Post Howiesons Poort		50.9 ± 1.3 (n=2)	68 ± 7 (n=1)	
Late Howiesons Poort		60.5 ± 0.8 (n=5)	52 ± 3 (n=4)	72.7 ± 4.6 (n=1)
Intermediate Howiesons Poort (top part)			64 ± 3 (n=7)	63.1 ± 3.9 (n=1)
Intermediate Howiesons Poort (bottom part)		63.3 ± 2.2 (n=1)	82 ± 2 (n=15)	79.7 ± 5.8 (n=1)
Early Howiesons Poort/Still Bay		72.1 ± 1.7 (n=2)	104 ± 3 (n=12)	
Kathu Pan 6				
Howiesons Poort				79.4 ± 8.1 (n=1)
Hollow Rock Shelter				
Still Bay	72-80			73.2 ± 5.9

Figure 1A

Figure 1B

Figure 2

Figure 2

UW247

UW325

UW260

UW287

UW264

Figure 3

Figure 4

Figure 5

Figure 6