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Radiocarbon dating from 40 to 60 ka BP at Border Cave, South Africa

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Abstract

We present 21 radiocarbon dates on 19 charcoal samples from the sedimentary sequence preserved in Border Cave, South Africa. The background radiocarbon activity for charcoal from the cave was determined to be 0.050 ± 0.018 percent modern carbon, from the analysis of a radiocarbon-dead sample from unit 5WA. Radiocarbon ages for individual samples ranged from 25.2 to >58.2 ka BP.

The error-weighted mean ages for successively older strata are $38.5+0.85/-0.95$ ka BP for unit 1WA, $50.2+1.1/-1.0$ ka BP for units 2BS.LR.A and 2BS.LR.B, $56.5+2.7/-2.0$ ka BP for unit 2BS.LR.C and $59.2+3.4/-2.4$ ka BP for unit 2WA. This radiocarbon chronology is consistent with independent chronologies derived from electron spin resonance and amino acid racemization dating. The results therefore provide further evidence that radiocarbon dating of charcoal by the ABOX-SC technique can yield reliable radiocarbon ages beyond 40 ka BP. They also imply that Border Cave 5, a modern human mandible, predates >58.2 ka BP and that the Middle Stone Age (Mode 3)—Later Stone Age (Mode 5) transition of Border Cave was largely effected between ~56.5 and ~41.6 ka ago.

1. Introduction

Bird et al. (1999) described a new pretreatment and analytical protocol for the radiocarbon dating of charcoal, and particularly for 'old' charcoal greater than 40 ka BP in age. The pretreatment involves subjecting hand-picked charcoal fragments to acid and base washes, followed by a period in an acid-dichromate oxidizing solution (the ABOX pretreatment). Innovations in target preparation included using stepped combustion (SC) to further remove contamination during combustion to carbon dioxide, pre-oxidation of iron powder used to catalyse the conversion of carbon dioxide to graphite, and the use of a 'double vacuum' extraction line to ensure the complete exclusion of atmospheric contamination.

Analyses of 'radiocarbon-dead' graphite by Bird et al. (1999) suggested a total blank for the methodology of 0.04 ± 0.02 percent modern carbon (pMC), but the analysis of 'radiocarbon-dead' charcoal suggested that in practice the blank might approach 0.1 ± 0.02 pMC for natural samples exposed for long periods to contamination from the local environment. The current study seeks to test further the ability of the methodologies described by Bird et al. (1999) to generate reliable radiocarbon dates on charcoal of greater than 40 ka BP in age, using a suite of charcoal samples in the "difficult" 40–60 ka range from Border Cave in South Africa.

Border Cave (Fig. 1), near the crest of the Lebombo Mountains in northern KwaZulu-Natal province, contains a ~4 m thick sequence of alternating white ash (WA) and brown sand (BS) strata, with cultural debris throughout, that accumulated between ~200 000 years ago and the present (Beaumont et al., 1978; Grün and

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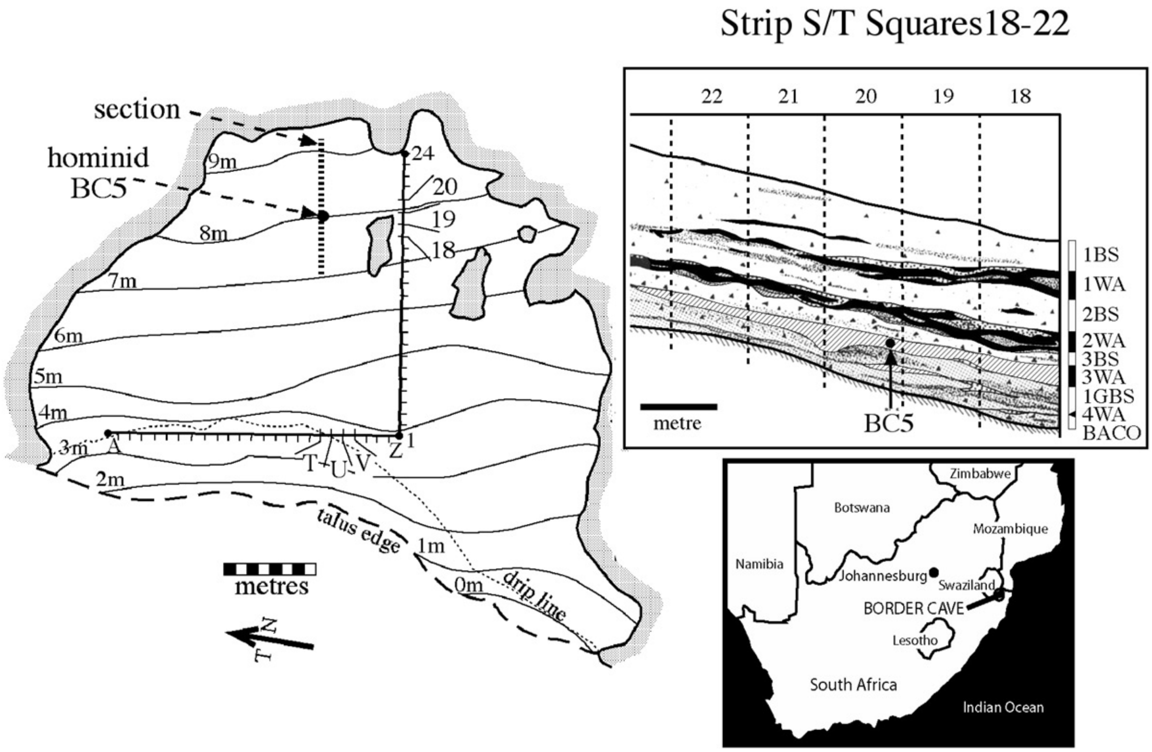

Fig. 1. Location and plan of Border Cave showing contours of the floor of the cave (in metres), the excavation grid and the location of the grid squares from which samples were obtained for the current study, near hominid locality BC5. Also shown is a stratigraphic section, approximately east–west, along the south wall of strips S&T, close to where samples were collected for this study (modified from Beaumont, 1980). BS = brown sand; WA = white ash.

Beaumont, 2001). This site constitutes an ideal test for the ABOX-SC methodology because of its exceptionally clear stratigraphy and the presence in even the lowermost levels of large well-preserved charcoal fragments that thereby permit the direct determination of an appropriate background radiocarbon activity level. Furthermore, results can be assessed by means of independent ages provided by the electron spin resonance (ESR) of teeth (Grün et al., 1990; Grün and Beaumont, 2001; Grün and Ward, 2002), the amino acid racemization (AAR) of ostrich eggshell (Miller and Beaumont, 1989; Miller et al., 1999) and previous radiocarbon results (Beaumont, 1980; Beaumont et al., 1992).

One archaeological objective was to obtain a minimum age for strata directly above Border Cave 5, a modern human mandible from the edge of a shallow interment in underlying unit 3WA of square T20 (Fig. 1). Another was to establish the timespan covered by a dramatic increase between units 2BS.LR.C and 2BS.UP in the incidence of small ($\sim$10 mm long) ‘‘waste’’ rock slivers derived from scaled pieces by bipolar reduction (Beaumont, 1980). The finding of a few such items with mastic-flanked working edges is best taken to mean that this shift reflects an escalating use of informal microliths as inserts for composite tools and weapons (Mulvaney, 1969; Flood, 1983).

2. Samples and methodology

A total of nineteen samples from strata thought to range from $\sim$40 to 60 ka BP were selected from many boxes of Border Cave charcoal in storage at the McGregor Museum. These samples came from six 1WA–2WA units and, in the case of those excavated in 1987, from subdivisions, namely 5 cm spits on Levels (L) within them. An additional sample from unit 5WA, dated by ESR to 147 ± 6 ka (Grün and Beaumont, 2001), was selected to establish an appropriate background radiocarbon activity for the other samples. The methods used for the analysis did not deviate significantly from those described by Bird et al. (1999), except that carbon dioxide produced during the 340°C combustion step was discarded as Bird et al. (1999) has shown this fraction to generally be contaminated by younger carbon.

It was also found that, for the Border Cave samples, carbon dioxide produced from the combustion steps at 650°C and 910°C was very difficult to convert to graphite. This was possibly due to trace contamination by sulphur species carried through the cryogenic purification steps, that may have poisoned the catalytic action of the iron powder during the reduction of carbon dioxide to graphite. Fortunately it was found that this problem could be circumvented in the case of the 910°C fraction by slowly cooling the furnace after combustion over a period of several hours. This either enabled time for the reaction of sulphur contaminants with silver wire in the combustion tube, or allowed sufficient time for reaction at a critical (unknown) temperature to occur.

As a result of these difficulties it was only possible to produce one 650°C target from all the Border Cave samples that were analysed. This sample from unit 2WA in square T20, gave readings of 0.157±0.036 pMC for the 650°C fraction and 0.092±0.022 pMC for the 910°C fraction. This comparison suggests that significant additional contamination is removed by the SC procedure at 650°C, as was also found to be the case for some samples by (Bird et al., 1999).

3. Results and discussion

The results for all samples are presented in Table 1. The 'radiocarbon-dead' sample from unit 5WA.L2 (~147 ka in age) indicates that the background appropriate for the Border Cave charcoal samples is 0.050±0.018 pMC. This blank value has been subtracted from the activities measured for other samples in Table 1, prior to calculation their radiocarbon age. Note that this blank is significantly lower than the value of 0.10±0.02 pMC obtained by the ABOX-SC technique on other 'radiocarbon-dead' charcoal samples by Bird et al. (1999). Indeed, it is not distinguishable from the value of 0.04±0.02 pMC measured for ABOX-pretreated samples prepared from graphite by Bird et al. (1999). This result is consistent with the negligible amounts of moisture movement and biotic activity in the Border Cave sediments, compared with the 'worst case' charcoal samples analysed by Bird et al. (1999), which came from sediments exposed to constantly infiltrating soil solutions.

All but one of the measured radiocarbon activities for the samples differ from zero at two standard deviations and there is good consistency between samples from different levels in the same unit, even at these low values. The radiocarbon activity of the samples generally decreases (Fig. 2), and age therefore increases, in the six units from 1 to 2WA (Fig. 3). An exception to this clear trend are two results from the same square of 2BS.LR in an area of the cave where units A and B are not separable, hence 2BS.LR.A&B. These both returned highly anomalous results that are even younger than a previously obtained age of 39.5 ka BP for a bulk charcoal sample from the directly overlying 1WA unit (P. Beaumont, unpubl. data). Given that fieldnotes record no stratigraphic anomalies below unit 1WA, a

Table 1
Radiocarbon activities (without background subtraction) for Border Cave samples and radiocarbon ages calculated after subtraction of a background of 0.05±0.02 pMC

ANUA-	Sample name	Treatment	Raw radiocarbon (pMC)	Age (yr BP)	Error (±1σ)	Minimum age
Samples						
17304	V20-1WA-LRA-L19	ABOX-900	0.875±0.090	38 540	+850/−950	
17307	V20-2BS-UP-L20	ABOX-900	0.611±0.067	41 640	+940/−1070	
15805	V20-2BS-UP-L21	ABOX-920	0.376±0.046	46 000	+1150/−1340	
17302	V20-2BS-UP-L22	ABOX-900	0.474±0.055	43 880	+1040/−1190	
17306	V20-2BS-UP-L23	ABOX-900	0.303±0.042	48 030	+1360/−1630	
17304	V18-2BS-LRA-L22	ABOX-900	0.276±0.051	48 940	+1740/−2230	
15814	V18-2BS-LRA-L23	ABOX-920	0.282±0.038	48 730	+1360/−1640	
17319	T20-2BS-LR-A&B-LSTRS	ABOX-900	0.998±0.114	37 420	+920/−1050	
19213	T20-2BS-LR-A&B	ABOX-910	4.380±0.220	25 200	+400/−410	
16305	V18-2BS-LRB-L24	ABOX-900	0.178±0.027	53 510	+1870/−2450	
17308	V18-2BS-LRB-L25	ABOX-900	0.342±0.047	46 880	+1290/−1540	
17504	V18-2BS-LRB-L25	ABOX-910	0.252±0.039	49 840	+1580/−1960	
15813	V18-2BS-LRB-L26	ABOX-920	0.239±0.062	50 380	+2380/−3400	
16304	T20-2BS-LRC-STR	ABOX-900	0.151±0.025	55 410	+2210/−3060	
17505	V19-2BS-LRC-L28	ABOX-910	0.124±0.026	57 910	+2950/−4700	
17303	V19-2WA-L29	ABOX-900	0.153±0.027	55 250	+2270/−3200	
18626	V16-Q2-2WA-L3	ABOX-910	0.121±0.019	58 240	+2640/−3950	
19010	V19-2WA-L31	ABOX-910	0.128±0.026	57 490	+2820/−4380	
19011	T20-2WA	ABOX-910	0.092±0.022	62 460*	+4300/−9900	(>58160)
19013	T20-2WA	ABOX-650	0.157±0.036	54 950	+2616/−3900	
Background						
13007	T14-5WA-2	ABOX-920	0.05±0.018			

Results for samples that overlap zero at two standard deviations, after subtraction of background, are marked with an asterisk, and the minimum age is given in brackets.

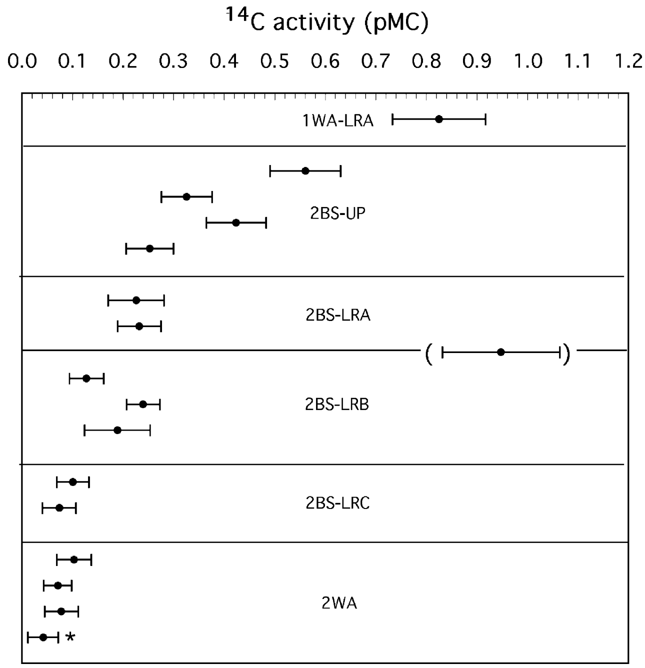

Fig. 2. Measured radiocarbon activities for the Border Cave charcoal samples, after subtraction of a background of 0.05±0.02 pMC. Results are grouped by sedimentary unit. The error shown is one standard deviation and samples that overlap zero at two standard deviations are marked with an asterisk. Brackets indicate sample from T20.2BS.LR.A&B-LSTRS, and the result for sample T20.2BS.LR.A&B (4.38±0.22 pMC) is not shown, as both these samples from unit 2BS.LR.A&B are excluded from consideration (see text). As the samples come from several excavation squares across a complicated and sloping stratigraphy, no depths are given for individual samples, but the samples are arranged in relative stratigraphic order from lowest to highest in the stratigraphy.

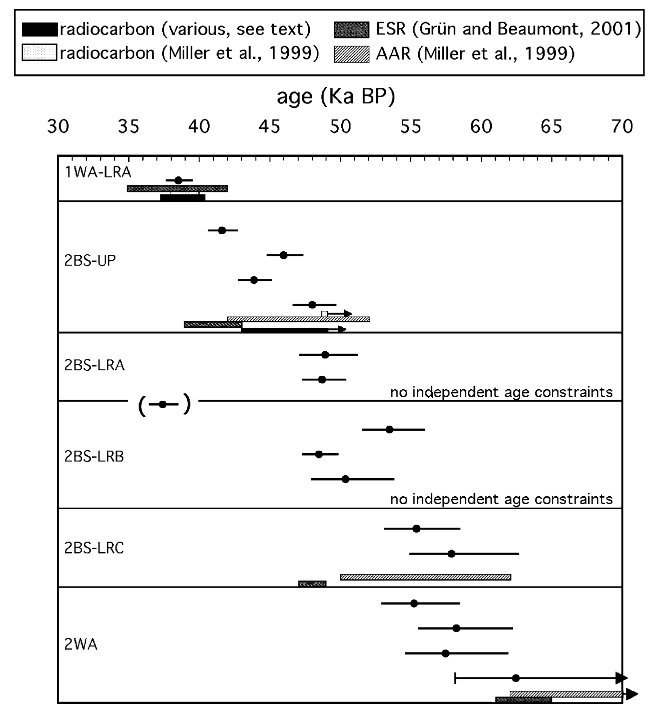

Fig. 3. Radiocarbon ages (±1σ) for the Border Cave charcoal samples, presented as in Fig. 1. In addition the ranges of other independent age determinations obtained by radiocarbon, ESR and AAR for each unit (where they exist) are shown as horizontal bars beneath the radiocarbon dates for each unit derived from this study. Brackets indicate sample from T20.2BS.LR.A&B-LSTRS, and the result for sample T20.2BS.LR.A&B (25.2±0.4 ka BP) is not shown, as both samples from unit 2BS.LR.A&B are excluded from consideration (see text).

most likely interpretation is that the 2BS.LR.A&B sample was mislabelled, either at the time of retrieval or subsequently. The two anomalous results that probably relate to 1BS.LR.A&B, are excluded from further considerations in the discussion that follows.

Four previously dated samples from unit 1WA, all intensively pretreated and measured with a high-precision counter, produced readings that range from 37.7±0.6 to 39.8±0.6 ka BP (Beaumont et al., 1992). The single age of 38.5 ka BP obtained for unit 1WA in this study is therefore in excellent accord with those previous radiocarbon results. This agreement provides an additional check on the applicability of the background value that has been subtracted from our measured radiocarbon activities.

Samples from unit 2BS.UP generated ages from 41.6 to 48.0 ka BP that tend to become older with depth. In contrast, there is no obvious trend with depth through units 2BS.LR.A and 2BS.LR.B, with results from these units having an error-weighted mean age of 50.2+1.1/−1.0 ka BP. The two samples dated from unit 2BS.LR.C both returned ages older than the overlying 2BS units, with an error-weighted mean age of 56.5+2.7/−2.0 ka BP. A charcoal nodule sample, studding the 2WA surface and provisionally labelled 2WA.L29, could relate to either a final portion of that unit or the initial 2BS.LR.C occupation. This sample yielded a date of ~55.2 ka BP, which is seemingly more consistent with the two readings for 2BS.LR.C.

Ages for the three samples from within the 2WA unit have an error-weighed mean value of 59.2+3.4/−2.4 ka BP. The samples from squares V16 and V19 (Table 1) are from the upper and middle levels of unit 2WA, respectively, but in the absence of direct superpositioning, their relative ages are uncertain. Our third sample, was collected from the entire thickness of unit 2WA in square T20, so the location of the single fragment of charcoal used for analysis within the 2WA stratigraphy is also uncertain.

Detailed comparison of the radiocarbon dates presented here with published ESR and AAR chronologies is impeded by the predominant absence of tooth, eggshell and charcoal samples with exactly the same stratigraphic provenances. Comparison is further hampered by the lack of a precise calibration between radiocarbon years and calendar years in the pre-40 ka BP time period. In general, however, there does appear to be a good concordance, within the quoted uncertainties, between the radiocarbon timescale and other published chronologies. There is, in addition, good agreement between the radiocarbon age of 2BS.LR.C and an unpublished burnt flint TL date of 57 ± 7 ka obtained by J. Huxtable at Oxford (pers. comm. to P.B.). Hence, although the uncertainties are large, the data suggest that the offset between calendar and radiocarbon years beyond 40 ka BP is less than a few thousand years. This is consistent with the Lake Suigetsu radiocarbon calibration data of Kitagawa and Van der Plicht (1998) and contrasts with major excursions in offset for radiocarbon ages of less than 40 ka BP as recently reported by Beck et al. (2001).

4. Conclusions

Exceptional stratigraphic clarity and conditions of preservation, together with the ability of the ABOX-SC technique to remove contamination, has resulted in a robust radiocarbon chronology for the 40–60 ka interval at Border Cave. Results show that unit 2BS.UP was deposited over ~7000 years between 41.6 and 48.0 ka BP and that the 2BS.LR.A–2BS.LR.B units accumulated over a maximum period of 4000 years centred on 50.2 ka BP. Readings indicate that the error-weighted mean age of unit 2BS.LR.C is $56.5 + 2.7/-2.0$ ka BP and that samples certainly ascribable to the 2WA unit are older at $59.2 + 3.4/-2.4$ ka BP.

It is suggested from the results presented in this study that large changes in atmospheric radiocarbon levels of the kind that occurred at around 40 ka BP may not have occurred over the preceding ~15 ka. In addition, it should be noted that the modern human mandible, Border Cave 5, from the base of the 3BS–3WA sediment succession in square T20, is clearly substantially older than the >58.2 ka BP date for directly overlying unit 2WA. Finally, these ABOX-SC results show that informal microlithic insert production largely replaced prepared core (Mode 3) technology in northern KwaZulu-Natal between ~56.5 and ~41.6 BP.

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