

CAN WE USE COSMOGENIC ISOTOPES TO DATE STONE ARTIFACTS?

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ABSTRACT. Two chert artifacts from the region near Luxor, Egypt have yielded concentrations of cosmogenic ¹⁰Be that allow calculation of nominal exposure ages of 326,000 and 304,000 years. Both artifacts are flakes that were collected atop limestone benches of the Eocene Thebes Formation which form cliffs along the west side of the Nile. The site is at elevation 240 m and is about 15 km from the Nile. Tools associated with these artifacts can be attributed to the Late Acheulean or early Middle Paleolithic (the transition has been suggested to have been on the order of 250,000–300,000 years ago). This area, where abundant chert nodules have weathered out, has been a collection, extraction, and fabrication site since the Early Paleolithic (since at least 400,000 years ago). Surface exposure dating records all periods of exposure. That means these ages represent composite ages, comprised of exposures both before and after working. But what fraction of the ¹⁰Be concentration we have measured was acquired before the flakes were produced? Here we propose several approaches to deconvolute the different exposure periods and better approximate the real age of the artifacts. As there is no *a priori* reason that the two ages should agree with the typological ages of the artifacts, nor for the two independent ages to agree, these first results are especially exciting and intriguing.

INTRODUCTION

Lithic artifacts can be dated by determining absolute ages on associated geological materials (using radiocarbon, U-series, electron spin resonance, ⁴⁰Ar/³⁹Ar, luminescence, e.g. Wendorf et al.1993; Aitken 1999) or directly dated with luminescence techniques (e.g. Valladas 1992; Mercier et al. 1995). The former method requires the presence of appropriate materials (organic material, tufa deposits, ash layer, etc.) with a clear stratigraphic relationship to the artifacts and may have methodological limitations. Direct dating of chert tools with luminescence requires that they were heated (to at least 450 °C) in a fire. This may put limitations on the material available and the oldest ages may be constrained by when fire was domesticated (e.g. Mercier et al. 1995). Recently, the dating of the outer surface (or skin) of worked (but unburned) chert artifacts has been described by Schwarcz and Rink (2001).

The method described here (surface exposure dating with cosmogenic isotopes) will allow direct dating of siliceous artifacts themselves. The restricting caveats are that the artifacts must have been continuously exposed since fabrication but must not have been exposed earlier or there must be a way to determine the time of prior exposure. Having fulfilled these caveats, there is no dating limitation, tools and debitage (knapping waste) exposed for several thousand to millions of years can be dated. The isotopes used (e.g. ¹⁰Be, ²⁶Al and ²¹Ne) are produced in situ within a rock due to bombardment by cosmic rays (Lal 1991; Cerling and Craig 1994). Therefore, the concentration measured provides a direct measure of time the rock has been exposed to cosmic rays at or near the surface. We present here the first two ¹⁰Be results for two different chert artifacts. Technically, the analysis of chert does not appear to pose unique problems (see also Boaretto et al. 2000). On the other hand, we are clearly in the early stages of the exposure dating of stone tools. The greatest hurdle will be to constrain the exposure acquired by the chert before they were collected by prehistoric humans.

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THE THEBES MOUNTAINS SITES

Chert artifacts are abundant on the highly weathered plateaux or benches making up cliffs along the Nile at Luxor (Figure 1). The Thebes Mountains, with the Eocene Thebes Formation as the youngest rock unit, were uplifted during Oligocene/Miocene time in tectonism related to the opening of the Red Sea. During the late Oligocene and early Miocene, the Nile (*sensu lato*) cut deeply into the Thebes Mountains (Said 1981, 1993). Downcutting became especially intense during extreme base level lowering related to the Messinian salinity crisis (around the Miocene/Pliocene transition at about 5 Ma) (Said 1981, 1993). Since that time the Thebes Mountains have undergone minor erosion in the form of planation and scarp retreat with deposition of associated alluvial and colluvial material. The artifacts we have analyzed were collected from two different locations (about 3 km apart) atop these nearly flat plateaux at elevation of 240 m (about 150 m higher than the present elevation of the Nile). The sites have not been affected by the repeated entrenchment and alluviation of the Nile (and its antecedents) nor by erosive activity in the tributary canyons or wadis.



Figure 1 The benches of resistant Thebes limestone from which the artifacts were collected

Thebes Formation chert (also termed Eocene Flint) occurs as nodules, stringers, lenses, and discontinuous beds in the limestone bedrock. The nodules accumulate in a lag horizon as the enclosing and underlying less-resistant limestones, marls and shales erode away. This provided a fertile ground for prehistoric man who has intensely collected and extracted chert here since the Early Paleolithic (e.g. Clark 1967, 1970; Vermeersch 1990, 2000). The sampled sites contain a melange of several types of chert artifacts (e.g. hand axes, scrapers, blades) as well as workshop debris, intermixed with unworked chert nodules. In the immediate area of these artifacts no mounds or debris of quarrying were observed. Similarly, we have no indication that the artifacts were ever buried by soil or colluvium. At these sites, the regolith layer is thin (less than 5 cm) and discontinuous.

At the two sampling sites, tools with clear characteristics can be classified as Late Acheulean or early Middle Paleolithic in age. The two artifacts we have analyzed are flakes made during the knapping process; they are not themselves tools. Both artifacts were severely wind polished and bore a well-developed mottled dark brown to black patina of desert varnish. In one of the tools (sample Lux 1), the varnish was present on both the unworked and worked surfaces (Figure 2). On a broken surface, the fresh chert is pale pinkish grey to buff, with a relatively compact texture.



Figure 2 The two analyzed flakes, on the left designated as sample Luxor 2, on the right Luxor 1

CHEMICAL TREATMENT AND AMS MEASUREMENT OF THE CHERT SAMPLES

The chert pieces were crushed and sieved to less than 0.5 mm. The material was cleaned with dilute HCl, then with dilute HF and HNO₃ until an about 50% loss by weight (Kohl and Nishiizumi 1992). 50–100 grams and 0.3 mg ⁹Be carrier were dissolved with HF and HNO₃. Following traditional methods of separation and purification of Be (Ochs and Ivy-Ochs 1996), the ¹⁰Be/⁹Be ratio was measured by accelerator mass spectrometry at the ETH/PSI facility in Zürich (Kubik et al. 1998). ¹⁰Be concentrations are listed in Table 1. Both samples were prepared and analyzed for ²⁶Al, although unsuccessfully. This is due to a low inherent Al content yielding low currents.

Table 1 AMS-measured ¹⁰Be concentrations and calculated “exposure ages.”

Sample nr	Elevation	¹⁰ Be atoms per gram quartz	Nominal exposure age ^a
Luxor 1	240 m	1.56 × 10 ⁶	326,000 ± 22,000
Luxor 2	240 m	1.46 × 10 ⁶	304,000 ± 20,000

^aErrors are at the 1σ level including the statistical (counting) error and the error due to the normalization to the standards and blanks. A sample processing reproducibility error of 5% has been included.

For ¹⁰Be studies, the mineral quartz is used almost exclusively (cf. Ivy-Ochs et al. 1998). This is primarily because one can reliably clean meteoric ¹⁰Be from the surface of quartz grains without leaching any of the in situ ¹⁰Be contained in the quartz structure (e.g. Kohl and Nishiizumi 1992). Even though chert is a microcrystalline form of quartz (lattice size on the order of 5–30 microns), there was still some question as to the influence the intense patina may have on cleaning of meteoric ¹⁰Be. Meteoric ¹⁰Be is produced in the atmosphere at levels several orders of magnitude higher than in a rock surface. It, therefore, poses a serious contamination problem if not completely removed. Because beryllium is very surface reactive, the meteoric ¹⁰Be found in rain water may be present in the Fe and Mn oxide minerals which make up desert varnish (Krinsley 1998; Broecker and Liu 2001). Several ¹⁰Be studies using quartz indicate that the presence of desert varnish does not result

in a meteoric ^{10}Be contamination problem (Brown et al. 1998). This was also shown by agreement between ^{10}Be and ^{26}Al ages on varnished rock surfaces (Liu and Broecker 2000). But perhaps chert behaves differently than quartz. We have noted no remaining varnish upon examination of the chemically-treated chert under a binocular microscope. But to unequivocally prove there is no remaining meteoric ^{10}Be , we are performing sequential dissolution experiments and will remeasure ^{26}Al (with carrier addition) on an aliquot of this chert. We note that Boaretto et al. (2000) also found that flint tools could be effectively cleaned of meteoric ^{10}Be using standard procedures.

DISCUSSION OF AGES

Exposure ages (Table 1) were calculated using the production rates of Kubik et al. (1998) and altitude-latitude scaling parameters based on Lal (1991). No corrections were necessary for thickness of the samples (less than 3 cm) (Masarik and Reedy 1995) nor for shielding from surrounding topography. The nominal ages for the two different chert pieces are $326,000 \pm 22,000$ (Luxor 1) and $304,000 \pm 20,000$ (Luxor 2). These errors are purely analytical and do not include uncertainties in the production rate or scaling which may be up to 20% (e.g. Stone 2000). Interestingly, the slightly older age belongs to the flake with the intensely varnished unworked face.

Surface exposure dating records all periods of exposure, if there has been no intervening burial interval during which radionuclides decay. That means these exposure ages are composite ages made up of:

- the time the chert nodule was enclosed in the limestone bedrock and gradually getting closer to the surface as the limestone around it eroded away, plus
- the time that the eroded out chert cobble was lying on the plateau surface prior to being worked on, plus
- the time after the flake was chipped from a cobble.

The crucial question is what proportion of the exposure was acquired before the cobbles were worked? Had the cobbles been exposed for a few thousand years, for tens of thousands of years or for longer? Unfortunately, ^{26}Al and ^{21}Ne cannot be used to reveal these earlier exposure periods as the composite age represents one continuous exposure.

Even though these ages do indeed fit into the time range expected for Late Acheulean to early Middle Paleolithic artifacts (see below) more information is needed to determine when these flakes were actually made. As a first step, more worked pieces should be analyzed, especially tools which themselves can be better constrained typologically. Do they also yield ages on the order of 300,000 years? One could also check the exposure ages of unworked chert cobbles found on the surface and with a well-developed patina. One would expect to obtain a range of ages as each cobble weathered out at a different time.

The fact that some of the flakes exhibit an intense varnish on worked and unworked faces may prove useful. But varnish does not always grow at a constant rate and may as well flake or peel off, therefore its thickness cannot be quantitatively used as a gauge of time (Liu and Broecker 2000, Broecker and Liu 2001). Nevertheless, perhaps we can glean information about the relative time of exposure prior to and after working by measuring the thickness of the varnish on worked and unworked faces. Another approach that may help to narrow down the time range is to analyze chert nodules located close to collected artifacts but still embedded in the limestone bedrock. From this we should be able to estimate the total exposure time (assuming removal of overlying bedrock at a constant erosion rate). This may help us to deconvolute the three time periods of the composite exposure. It must be

cautioned that there are possible scenarios where the cobbles would have a roughly similar inheritance (controlled by the erosion rate of the limestone). For example, if all the worked pieces were plucked out of exposed limestone sections rather than just collected from loose debris. If the latter were true, the erosion rate of the limestone would only constrain the lower limit of the inherited contribution. In the end, as much information as possible should be garnered and used as input parameters for various weathering out/exposure models.

Is it possible that these are the actual exposure ages for these artifacts? Is such an age reasonable for the typology exhibited by the flakes and associated tools? The two dated pieces belong to the more heavily varnished artifacts known from the Luxor area collections. They are connected with more roughly hewn Late Acheulean inventories prevalent before Middle Paleolithic sites where more refined and also less varnished tools appear. The dates measured are well in accordance with the older range of the estimated time window for the Early to Middle Paleolithic transition. Acheulean artifacts from middle Egypt are known to date from at least 400,000 years ago, with the Final Acheulean at 350,000–400,000 years ago (Vermeersch 2000). The early Middle Paleolithic is centered on 150,000–200,000 (Vermeersch 2000). It is the transition of the Early to Middle Paleolithic which is difficult to pinpoint with absolute dating methods. For example, McBrearty and Brooks (2000) quote the introduction of Middle Stone Age technology at 250,000–300,000 years ago. Conversely, some suggest that the beginning of the Middle Paleolithic was closer to 230,000 (Wendorf et al. 1993), 200,000 (Clark 1988) or even 130,000 years ago (Guichard and Guichard 1968). Our attempt at dating and the intriguing preliminary results presented here are thus at this juncture especially timely.

CONCLUSIONS

Two chert artifacts (flakes) contained ^{10}Be concentrations that gave exposure ages of 326,000 and 304,000 years. Based on the typological age range of the tool assemblage (Late Acheulean to early Middle Paleolithic with the transition possibly around 300,000 to 250,000 years ago) and ages of archeological material present in the Thebes Mountains (Early Paleolithic up into Predynastic; e.g. Vermeersch 2000, 2001), our preliminary results are indeed reasonable. To be conservative one should not consider them to be the actual dates for tool working until the period of exposure prior to fabrication can be better constrained. For assessing this we propose several approaches which must be tested. This includes analyzing additional worked chert pieces from the surface and unworked nodules presently still embedded in the bedrock. These preliminary results are encouraging and highlight the exciting potential of surface exposure dating of siliceous artifacts.

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REFERENCES

- Aitken MJ. 1999. Archaeological dating using physical phenomena. *Reports on Progress in Physics* 62(9): 1333–76.
- Boaretto E, Berkovits D, Hass M, Hui SK, Kaufman A, Paul M, Weiner S. 2000. Dating of prehistoric caves sediments and flints using ^{10}Be and ^{26}Al in quartz from Tabun Cave (Israel): Progress report. *Nuclear Instruments and Methods in Physics Research B* 172: 767–71.
- Broecker WS, Liu T. 2001. Rock varnish: recorder of desert wetness? *GSA Today* 11(8):4–10.
- Brown ET, Bourlès DL, Burchfiel BC, Qidong D, Jun L, Molnar, P, Raisbeck GM, Yiou F. 1998. Estimation of slip rates in the southern Tien Shan using cosmogenic ray exposure dates of abandoned alluvial fans. *Geological Society of America Bulletin* 110(3):377–86.
- Cerling TE, Craig H. 1994. Geomorphology and in-situ cosmogenic isotopes. *Annual Reviews of Earth Planetary Science Letters* 22:273–317.
- Clark JD. 1967. *Atlas of African prehistory*. Chicago: University of Chicago Press.
- Clark JD. 1970. *The prehistory of Africa*. South Hampton: Camelot Press.
- Clark JD. 1988. The Middle Stone Age of East Africa and the beginnings of regional identity. *Journal of World Prehistory* 2:235–305.
- Guichard J, Guichard G. 1968. Contribution to the study of the Early and Middle Paleolithic of Nubia. *The Prehistory of Nubia*. Dallas: Southern Methodist University Press. p.148–93.
- Ivy-Ochs S, Kubik PW, Masarik J, Wieler R, Bruno LA, Schlüchter C. 1998. Preliminary results on the use of pyroxene for ^{10}Be surface exposure dating. *Schweizerische mineralogische und petrographische Mitteilungen* 78:375–82.
- Kohl CP, Nishiizumi K. 1992. Chemical isolation of quartz for measurement of in-situ produced cosmogenic nuclides. *Geochimica et Cosmochimica Acta* 56:3583–7.
- Krinsley D. 1998. Models of rock varnish formation constrained by high resolution transmission electron microscopy. *Sedimentology* 45(4):711–25.
- Kubik PW, Ivy-Ochs S, Masarik J, Frank M, Schlüchter C. 1998. ^{10}Be and ^{26}Al production rates deduced from an instantaneous event within the dendro-calibration curve, the landslide of Köfels, Ötz Valley, Austria. *Earth and Planetary Science Letters* 161:231–41.
- Lal D. 1991. Cosmic ray labeling of erosion surfaces: in situ nuclide production rates and erosion models. *Earth and Planetary Science Letters* 104:424–39.
- Liu T, Broecker WS. 2000. How fast does varnish grow? *Geology* 28(2):183–6.
- Masarik J, Reedy R. 1995. Terrestrial cosmogenic-nuclide production systematics calculated from numerical systematics. *Earth and Planetary Science Letters* 136:381–95.
- McBrearty S, Brooks AS. 2000. The revolution that wasn't: a new interpretation of the origin of modern human behavior. *Journal of Human Evolution* 39(5): 453–563.
- Mercier N, Valladas H, Valladas G. 1995. Flint thermoluminescence dates from the CFR laboratory at GIF: contributions to the study of the chronology of the Middle Paleolithic. *Quaternary Science Reviews* 14: 351–64.
- Ochs M, Ivy-Ochs S. 1996. The chemical behavior of Be, Al, Fe, Ca, and Mg during AMS target preparation from terrestrial silicates modeled with chemical speciation calculations. *Nuclear Instruments and Methods in Physics Research B* 123:235–40.
- Said R. 1981. *The geological evolution of the River Nile*. New York: Springer-Verlag.
- Said R. 1993. *The River Nile*. Oxford: Pergamon Press.
- Schwarcz HP, Rink WJ. 2001. Skinflint dating. *Quaternary Science Reviews* 20:1047–50.
- Stone J. 2000. Air pressure and cosmogenic isotope production. *Journal of Geophysical Research* 105: 23,753–59.
- Valladas H. 1992. Thermoluminescence dating of flint. *Quaternary Science Reviews* 11:1–5.
- Vermeersch PM. 1990. Palaeolithic chert exploitation in the limestone stretch of the Egyptian Nile Valley. *The African Archaeological Review* 8:77–102.
- Vermeersch PM. 2000. *Paleolithic Living Sites in Upper and Middle Egypt*. Leuven: Leuven University Press.
- Vermeersch PM. 2001. 'Out of Africa' From an Egyptian point of view. *Quaternary International* 75(1):103–12.
- Wendorf F, Schild R, Close AE. 1993. Summary and conclusions. *Egypt during the last interglacial*. New York: Plenum Press. p.552–73.