²⁶Al/¹⁰Be Age of Peking Man

Guanjun Shen¹, Xing Gao², Bin Gao¹ & Darryl E. Granger³

The chronological position of Peking Man, or *Homo erectus pekinensis*, has long been pursued, but has remained problematic due to lack of a suitable dating method¹⁻⁷. Here we report cosmogenic ²⁶Al/¹⁰Be burial dating of quartz sediments and artifacts from the lower strata of Zhoukoudian Locality 1 where the remains of early members of the Peking Man family were discovered. This study marks the first radioisotopic dating of any early hominin site in China beyond the range of mass spectrometric U-series dating. The weighted mean of six meaningful measurements, 0.75 ± 0.09 (0.11) Ma (million years), provides the best age estimate for lower cultural Layers 7-10. Together with previously reported U-series³ and paleomagnetic⁴ data, as well as sedimentological considerations^{8, 9} these layers may be further correlated to S6-S7 in Chinese loess stratigraphy or marine isotope stages 17-18, in the range of ~0.68-0.75 Ma. These ages are substantially older than previously supposed and may imply hominin presence in northern China throughout early Middle Pleistocene climate cycles.

With an inventory of six fairly complete hominin crania and bones representing at least 40 individuals, 98 species of non-hominin mammalian fossils and tens of thousands of stone artifacts the cave site of Zhoukoudian Locality 1, \sim 50 km southwest of Beijing, China, has remained the largest single source of *Homo erectus* and is one of the most important Paleolithic sites in the world¹.

The site is a sedimentary infill within a vertical karstic fissure. The ~40-m-thick depositional sequence can be divided into 17 layers¹⁰. The lowermost Layers 11-17 are fluvial and contain clasts from the nearby Zhoukou River; Layers 6-10 are breakdown breccia interlayered with silt and sand washed in from the hillslope; Layer 5 is travertine; the uppermost Layers 1-4 are silt and travertine with minor breakdown that accumulated after collapse of the cave's ceiling¹¹. Stone artifacts and hominin fossils have been recovered from Layers 1-10, with a majority from a lower level in Layers 8-9 and an upper level in Layers 3-4 (ref. 1). Mammalian fossils have been found from Layers 1-13; they are fairly uniformly distributed, although some primitive carnivores disappear above Layer 5 (ref. 12).

¹College of Geographical Sciences, Nanjing Normal University, Nanjing, Jiangsu 210046, China

²Institute of Vertebrate Paleontology and Paleoanthropology, Academia Sinica, Beijing 100044, China

³Department of Earth and Atmospheric Sciences, Purdue University, West Lafayette, IN 47907–1397, USA

As part of a multidisciplinary study initiated in the late $1970s^1$, dating was carried out at several Chinese institutions using a variety of techniques. The following age sequence was proposed: ~700 ka (thousand years) for the lowest fossiliferous Layer 13, based mainly on paleomagnetic stratigraphy⁴; ~500 ka for the lowest hominin fossil-bearing Layer 10, based on fission track dating of sphene grains; and ~230 ka for the uppermost Layers 1-3, based on 230 Th/ 234 U dating of fossil materials². These age assignments were generally supported by later 231 Pa/ 235 U⁵, fission-track⁶ and ESR dating⁷. An age range of ~230-500 ka for the hominin-bearing layers has been widely accepted by paleoanthropologists, though with a few critical comments¹³.

In contrast, much older ages were determined by mass spectrometric U-series dating of intercalated pure and dense calcite samples³, known to be a more reliable chronometer^{14, 15}. An age of 400 ± 8 ka was proposed for an upper horizon of Layers 1-2, ~500 ka for the upper part of Layer 5, and ≥ 600 ka for the middle and lower parts of Layer 5.

The suggestion of a much older Peking Man than previously suspected needs to be validated by an independent dating method. However, numerical dating beyond the upper limit of mass spectrometric U-series dating, ~600 ka, is difficult in China, as the lack of contemporary volcanic activity nearly precludes application of 40 Ar/ 39 Ar dating. Fortunately, allogenic cave sediments such as those at Locality 1 may be suitable for burial dating with cosmogenic 26 Al and 10 Be in quartz¹⁶⁻¹⁹. This method is based on the radioactive decay of 26 Al ($t_{1/2} = 717 \pm 17 \text{ ka}^{18}$) and 10 Be ($t_{1/2} = 1.36 \pm 0.07 \text{ Ma}^{20}$). These two nuclides are produced with a known 26 Al/ 10 Be atomic ratio of 6.8:1 in quartz exposed to secondary cosmic radiation near the ground surface. Their initial concentrations depend on the mineral's exposure time, which in turn is controlled by the erosion rate of the host rock. If quartz grains at the surface are washed into a cave with greater than ~10 meters of overburden, then the production of cosmogenic nuclides drastically slows. Because 26 Al decays faster than 10 Be, the 26 Al/ 10 Be ratio decreases exponentially with time. This offers a means for dating quartz burial up to ~3-5 Ma^{16}.

Burial dating was first applied to quartz gravels in caves for deriving river incision rates¹⁷. The method was later applied to hominin sites at Sterkfontein in South Africa¹⁸ and Sima del Elefante at Atapuerca, Spain¹⁹. The advantages of burial dating are its radiometric basis and its independence from other dating methods. However, it must be realized that cave sediments can have complex stratigraphy, particularly in vadose fills. Moreover, if fossils are mixed with quartz sediments with a prior burial history, the age result will be erroneously old.

Six quartz-bearing sand samples were collected, two of them (ZKD-12 and ZKD-13) from fluvial deposits in Layers 12 and 13 and the other four (ZKD-6, ZKD-7, ZKD-8/9 and ZKD-10) from quartz-rich lenses or sublayers in Layers 6, 7, 8/9 and 10, respectively. In addition, four quartzite

artifacts that directly indicate human presence at the site were analyzed from collections made in the 1930s from Layers 8/9. The ²⁶Al and ¹⁰Be concentrations and corresponding burial ages are presented in Table 1.

Three of the four quartzite artifacts yield results consistent within one standard error, with an error-weighted mean of 0.72 ± 0.13 (0.14) Ma. The fourth artifact (ST-3) gives an aberrant result of 1.66 ± 0.21 (0.24) Ma. This particular sample could have been taken from an older cave fill or terrace prior to manufacture. Among the sediment samples, those from Layers 7, 8/9, and 10 yield consistent results, with an error-weighted mean age of 0.78 ± 0.14 (0.15) Ma. This age is slightly older than, but within error of the weighted mean of the results from the three artifacts, suggesting that some sand might enter the cave with a previous burial signal. Sample ZKD-6, collected from a thin sandy lens adhered to the North Wall^{21} gave an aberrant result, 2.78 ± 0.51 (0.54) Ma. Though containing a few fossils, it is now out of stratigraphic contact with the main cross-section. It is therefore possible that this sandy lens dates to an earlier phase of cave formation. Finally, the two samples from the basal fluvial sediments did not yield meaningful age results. Their inherited cosmogenic nuclide concentrations were quite low, indicating rapid erosion in their source area, and leading to large uncertainty. Taken together, we consider the weighted mean of the six meaningful measurements, 0.75 ± 0.09 (0.11) Ma, to best represent the age for Layers 7-10 of Zhoukoudian Locality 1. This age is consistent with both prior U-series results³ and paleomagnetic stratigraphy⁴.

The age may be refined in the context of the cave environment. Several previous studies have correlated sedimentary packages at Zhoukoudian with the Chinese loess stratigraphy and marine isotope stages (MIS)^{8,9}. Layers that predominantly consist of breakdown breccia (6, 8/9) or loess (4) may be correlated with colder, drier periods, while intervening layers (3, 5, 7, and 10) consisting of waterlain sediments and/or flowstones with warmer, more humid periods (Fig. 1). The mammalian fauna support this interpretation, with a preponderance of steppe fauna in the breccia layers, and more forest fauna in the intervening layers^{8,9}. Following the loess timescale²², the well-dated flowstone in Layer 5 may be correlated with a period of prolonged warmth and humidity in China, associated with paleosol 5 (S5) and MIS 13-15 from ~500-600 ka. Layer 6 breccia best corresponds to loess 6 (L6) and MIS 16. Layer 7, containing waterlain sediments interbedded with flowstone would correspond to S6 and MIS 17. Layers 8/9 are primarily breakdown breccia, and would correspond to L7. The waterlain sediments in Layer 10 and the upper part of Layer 11 would then correspond to S7. Both L7 and S7 have been correlated to MIS 18 (ref. 22). Finally, the Brunhes/Matuyama boundary at 0.78 Ma lies between Layers 13 and 14, placing them near the L8/S8 boundary or in MIS 19. These correlations are supported by stable isotope evidence. Teeth from Equus sanmeniensis in Layers 10-11 (interglacial) have a

 δ^{18} O value that is about 3-4 permil higher than those in Layers 8/9 and 4 (glacial, ref. 23), consistent with the climatic correlations. We suggest that Layers 7-10, including the lower cultural level and the first appearance of *H. erectus* at Locality 1, lie within the range of S6-S7 and MIS 17-18 from ~0.68-0.75 Ma. Pending further confirmation, the assignment of the lower cultural level in Layers 8/9 into a cooler, drier episode may imply hominin presence at the site through glacial-interglacial cycles. Together with previous U-series dating of flowstone in Layers 1-2, the presence of *H. erectus pekinensis* at Zhoukoudian Locality 1 is constrained to a total range of 0.40-0.75 Ma.

A reliable chronology is critical for establishing the mode of Middle Pleistocene human evolution in East Asia, which remains highly debated²⁴⁻²⁶. Previously, the chronology of Chinese sites has been largely based on U-series and ESR dating of fossil materials, methods that are known to be vulnerable to post-burial U migration²⁷. ²³⁰Th/²³⁴U dating of speleothem calcites^{3, 28, 29} has repeatedly shown that the previous timescale for Middle-Late Pleistocene hominin sites in China may have been underestimated as a whole. The results of this paper show that such a tendency persists beyond the range of mass spectrometric U-series dating. It is foreseeable that ²⁶Al/¹⁰Be burial dating will be applied to other hominin sites in China and elsewhere, contributing substantially to establishing a robust chronological framework and thereby to a better understanding of human evolution.

METHODS

For quartzose sand, several kilograms of sediment were collected. Silt and clay were removed by a water rinse, and carbonates were dissolved in HCl. The remaining quartz-rich sand was sieved to >0.2 mm and leached several times in hot 5% HF/HNO₃ overnight with agitation. Following magnetic and gravimetric separation, the resulting quartz consisted of two populations: a darker-colored quartz with a high native Al concentration, and a lighter-colored quartz with a lower Al concentration. The darker-colored grains were removed by handpicking. For quartzite artifacts, the samples were thoroughly cleaned in 1% HF/HNO₃ and then crushed to a grain size <0.5 mm. The quartz was further purified by repeated overnight leaching in 1% HF/HNO₃ in an ultrasonic tank.

Purified quartz was dissolved in 5:1 HF/HNO₃, and spiked with ~0.3 mg ⁹Be prepared from beryl. An aliquot was taken for aluminum determination by ICP-OES using the method of standard additions. After evaporation and fuming of fluorides in H₂SO₄, Al and Be were separated on ion exchange columns in 0.4 M oxalic acid, precipitated as hydroxides, and transformed to oxides in a furnace at 1100°C. BeO was mixed with Nb and Al₂O₃ with Ag for ¹⁰Be/⁹Be and ²⁶Al/²⁷Al measurement by AMS at PRIME Lab, Purdue University.

Burial ages are calculated following ref. 16. For samples of this paper, postburial production by muons is safely ignored. Production rates are estimated for latitude 39° N, elevation 120 m³⁰, adjusted for a revised ¹⁰Be half-life²⁰.

- 1. Wu, R. K. et al. Multi-disciplinary Study of the Peking Man Site at Zhoukoudian (Science Press, Beijing, 1985, in Chinese).
- 2. Zhao, S. S. et al. Study of chronology of Peking Man Site. in (1) 239-240.
- 3. Shen, G. J. et al. High-precision U-series dating of Locality 1 at Zhoukoudian, China. J. Hum. Evol. 41, 679-688 (2001).
- 4. Qian, F., Zhang, J. X. & Yin, W. D. Magnetic stratigraphy from the sediment of West Wall and Test Pit of Locality 1 at Zhoukoudian. in (1) 251-255.
- Yuan, S. X., Chen, T. M., Gao, S. J. & Hu, Y. Q. U-series dating of fossil bones from Zhoukoudian sites. *Acta Anthrop. Sin.* 10(3), 189-193 (1991).
- 6. Guo, S. L. et al. Fission track dating of Layer 4 of the Peking Man site. Acta Anthrop. Sin. 10(1), 73-77 (1991).
- 7. Grün, R. *et al.* ESR analysis of teeth from the palaeoanthropological site of Zhoukoudian, China. J. Hum. Evol. **32**, 83-91 (1997).
- Zhou, C. L., Liu, Z. C. & Wang, Y. J. Climatic cycles investigated by sediment analysis in Peking Man's Cave, Zhoukoudian, China. J. Archaeol. Sci. 27, 101-109 (2000).
- 9. Liu, Z. C. Sequence of sediments at Locality 1 in Zhoukoudian and correlation with loess stratigraphy in northern China and with the chronology of deep-sea cores. *Quat. Res.* 23, 139-153 (1985).
- 10. Yang, Z. G. et al. Study of the Late Cenozoic strata at Zhoukoudian. in (1) 1-85.
- 11. Goldberg, P., Weiner, S., Bar-Yosef, O., Xu, Q. Q. & Liu, J. Y. Site formation processes at Zhoukoudian, China. J. Hum. Evol. 41, 483-530 (2001).
- Dong, W. et al. A comparative analysis on the mammalian faunas associated with *Homo erectus* in China. Acta Anthrop. Sin. 19(supplement), 246-256 (2000).
- Aigner, J. S. The age of Zhoukoudian Locality 1: the newly proposed ¹⁸O correspondences. *Anthropos* (Brno) 23:157-173 (1986).
- 14. Ludwig, K. R. & Renne, P. R. Geochronology on the Paleoanthropological time scale. Evol. Anthrop. 9, 101-110 (2000).
- Richards, D. A. & Dorale, J. A. Uranium-series chronology and environmental applications of speleothems. *Rev. Mineral. Geochem.* 52, 407-460 (2003).
- Granger, D. E. & Muzikar, P. F. Dating sediment burial with *in situ*-produced cosmogenic nuclides: theory, techniques, and limitations. *Earth Planet. Sci. Lett.* 188, 269-281 (2001).
- Granger, D. E. A review of burial dating methods using ²⁶Al and ¹⁰Be, in *In situ-produced cosmogenic nuclides and quantification of geological processes* (eds. Siame, L., Bourlès, D. L. & Brown, E. T.) 1-16 (*Geol. Soc. Am. Spec. Pap.* 415, 2006).
- Partridge, T. C., Granger, D. E., Caffee, M. C. & Clarke, R. Lower Pliocene hominid remains from Sterkfontein. *Science* 300, 607-612 (2003).
- 19. Carbonell, E. et al. The first hominin of Europe. Nature 452, 465-469 (2008).
- 20. Nishiizumi, K. et al. Absolute calibration of ¹⁰Be AMS standards. Nucl. Instr. and Meth. B 258, 403-413 (2007).
- 21. Ren, M. E. et al. Development of Zhoukoudian caves and its relation to the life of fossil man. in (1), 155-184.
- 22. Wang, X. S., Yang, Z. Y., Lovlie, R., Sun, Z. M. & Pei, J. L. A magnetostratigraphic reassessment of correlation between Chinese loess and marine oxygen isotope records over the last 1.1 Ma. *Phys. Earth Planet. Int.* **159**, 109-117 (2006).
- 23. Gaboardi, M., Deng, T. & Wang, Y. Middle Pleistocene climate and habitat change at Zhoukoudian, China, from the carbon and oxygen isotopic record from herbivore tooth enamel. *Quat. Res.* 63, 329-338 (2005).
- 24. Rightmire, G. P. Human evolution in the Middle Pleistocene: The role of *Homo heidelbergensis*. *Evol. Anthrop.* **6**, 218-227 (1998).
- 25. Wu, X. Z. The evolution of humankind in China. Acta Anthrop. Sin. 9(4), 312-321 (1990).
- 26. Clark, J. D. et al. African Homo erectus: old radiometric ages and young Oldowan assemblages in the Middle Awash Valley,

Ethiopia. Science 264, 1907-1910 (1994).

- 27. Pike, A. W. G. & Pettitt, P. B. U-series dating and human evolution. Rev. Mineral. Geochem. 52, 607-630 (2003).
- 28. Zhao, J. X., Hu, K., Collerson, K. D. & Xu, H. K. Thermal ionization mass spectrometry U-series dating of a hominid site near Nanjing, China, *Geology* 29, 27-30 (2001).
- 29. Shen, G. J., Gao, X., Zhao, J. X. & Collerson, K. D. U-series dating of Locality 15 at Zhoukoudian, China: implications for hominid evolution, *Quat. Res.* 62, 208-213 (2004).
- 30. Stone, J. O. Air pressure and cosmogenic isotope production. J. Geophys. Res. 105, 23753-23759 (2000).

Acknowledgements:

This project was jointly supported by National Natural Science Foundation of China and Wenner-Gren Foundation. AMS ²⁶Al and ¹⁰Be measurements were supported by an NSF facility grant to PRIME Lab. We would like to thank the Zhoukoudian Site Museum for assistance in sample collection, Dr. Christopher Norton for discussions, the first author's graduate students, Qingfeng Shao, Hong Wang and Li Zhang for assistance in sample collection and pretreatment.

Author contributions:

G.J.S. and D.E.G. contributed equally to conceiving the project, organizing fieldwork, interpreting data and preparing the paper. X.G. provided access to the site and quartzite artifacts. Chemistry was performed by B.G. and D.E.G.

Author information:

Correspondence and requests for materials should be addressed to G.J.S. (gjshen@njnu.edu.cn) or D.E.G. (dgranger@purdue.edu).

Figure legends

Figure 1 Correlation of Zhoukoudian Locality 1 sedimentary column with loess stratigraphy and global climate records. Depositional environments¹¹ can be correlated with alternating loess (L) and soil (S) layers found across the Chinese loess plateau, which have in turn been correlated to the marine isotope record^{8, 9, 22}. The correlations are pinned in time by the Brunhes/Matuyama paleomagnetic boundary in Layers 13-14 (ref. 4), U-series ages of flowstones in Layers 1-2 and 5 (ref. 3), and cosmogenic burial ages of Layers 7-10 reported here. Magnetostratigraphic data⁴ are indicated by closed circles for normal polarity, open circles for reverse polarity, half-closed circles for transitional or uncertain polarity and B/M for Brunhes-Matuyama boundary.

Sample	[²⁶ A1]	[¹⁰ Be]	²⁶ Al/ ¹⁰ Be	Burial age†
	(10^{6} at/g)	(10^{6} at/g)		(Ma)
Sediment				
ZKD-6	0.073 ± 0.018	0.040 ± 0.004	1.82 ± 0.49	$2.78 \pm 0.51 \ (0.54)$
ZKD-7-2	0.550 ± 0.053	0.132 ± 0.009	4.17 ± 0.49	$1.00 \pm 0.39 \ (0.40)$ *
ZKD-8/9	1.252 ± 0.095	0.273 ± 0.008	4.58 ± 0.38	0.75 ± 0.16 (0.17)*
ZKD-10-2	0.568 ± 0.052	0.120 ± 0.006	4.72 ± 0.50	$0.75 \pm 0.37 \ (0.37)^*$
ZKD-12	0.105 ± 0.030	0.021 ± 0.006	5.10 ± 2.01	$0.62 \pm 1.08 \ (1.08)$
ZKD-13	0.106 ± 0.028	0.018 ± 0.005	5.89 ± 2.35	0.31 ± 1.06 (1.06)
Artifacts (8/9)				
ST-1	0.199 ± 0.027	0.040 ± 0.002	4.95 ± 0.72	$0.67 \pm 0.29 \ (0.29)^*$
ST-2	0.476 ± 0.037	0.100 ± 0.003	4.77 ± 0.39	0.73 ± 0.17 (0.17)*
ST-3	0.371 ± 0.039	0.122 ± 0.003	3.04 ± 0.33	$1.66 \pm 0.21 \ (0.24)$
ST-4	0.568 ± 0.083	0.120 ± 0.005	4.72 ± 0.71	0.75 ± 0.29 (0.30)*

Table 1 Cosmogenic nuclide concentrations and burial ages

*Ages included in the weighted mean for Layers 7-10.

 \dagger Uncertainties (±1 σ) are expressed in two ways: the first includes analytical uncertainty only, and should be used when comparing burial ages; the second, parenthetical uncertainty also includes systematic errors in half-lives, and should be used when comparing against absolute ages or other methods.

Zhoukoudian

Sanmenxia loess profile

