Thermal ionization mass spectrometry U-series dating of a hominid site near Nanjing, China

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ABSTRACT

Mass spectrometric U-series dating of speleothems from Tangshan Cave, combined with ecological and paleoclimatic evidence, indicates that Nanjing Man, a typical *Homo erectus* morphologically correlated with Peking Man at Zhoukoudian, should be at least 580 k.y. old, or more likely lived during the glacial oxygen isotope stage 16 (~620 ka). Such an age estimate, which is ~270 ka older than previous electron spin resonance and alpha-counting U-series dates, has significant implications for the evolution of Asian *H. erectus.* Dentine and enamel samples from the coexisting fossil layer yield significantly younger apparent ages, that of the enamel sample being only less than one-fourth of the minimum age of Nanjing Man. This suggests that U uptake history is far more complex than existing models can handle. As a result, great care must be taken in the interpretation of electron spin resonance and U-series dates of fossil teeth.

Keywords: *H. erectus,* U-series, TIMS, speleothem, tooth.

INTRODUCTION

Precise and accurate dating of hominid fossils, particularly beyond the range of the ^{14}C method, is key to the understanding of many fundamental issues in human evolution. Several dating methods, such as electron-spin resonance (ESR), thermoluminescence (TL), amino acid racemization (AAR), U-series, K-Ar, and palaeomagnetism, have been used for dating hominid sites in different time ranges (see Wintle, 1996, for review). Among them, ESR dating of fossil teeth and TL dating of burnt flints were commonly used (e.g., Grün and Stringer, 1991), but their reliability is compromised by the need to calibrate numerous free parameters and by the uncertainty in U uptake history (for ESR) (e.g., Grün et al., 1998, 1999). The AAR method (e.g., Brooks et al., 1990) involves the assumption of an ''average'' paleotemperature, which is clearly an oversimplification for materials of mid-Pleistocene age. Paleomagnetic dating is reliable, but the age resolution is rather poor. The K-Ar method, particularly single-grain laser ablation ^{39}Ar-^{40}Ar dating, has been remarkably successful for dating volcanic lava or ash flows intercalated with hominid-bearing deposits (e.g., Swisher et al., 1994; Hou et al., 2000), but the majority of hominid sites do not have volcanic materials readily available for dating. The U-series method, particularly since the development of mass spectrometric techniques (Edwards et al., 1987; Ludwig et al., 1992), has revolutionized Quaternary chronology, especially for the time range beyond the ^{14}C limit, up to 600 ka. This method has proven remarkably successful for dating flowstones interlayered with hominid deposits (e.g., Schwarcz, 1992) and calcretes on hominid fossils (McDermott et al., 1996), but its reliability for dating fossil bones and teeth, like that of ESR, depends on knowing the sample's U-uptake history (e.g., McDermott et al., 1993; Grün et al., 1999).

The distribution, ecological environment, evolution, and destination of *Homo erectus* in China and eastern Asia are fundamental issues in the debate between out-of-Africa and multiregional evolution schools. Advocates of the multiregional evolution model for the origin of anatomically modern *Homo sapiens* (e.g., Thorne and Wolpoff, 1992) proposed that Asian *H. erectus*, which was initially derived from Africa 1–2 m.y. ago, was the direct ancestor of modern eastern Asian populations. The physical difference and genetic similarity among different human populations in the world were attained dynamically through gene flow and selective survival within an interconnected web of ancient *Homo* lineages. In contrast, hypotheses in support of the out-of-Africa model (e.g., Stringer, 1994) proposed a possible slower evolution rate for Asian *H. erectus* (than for their African counterparts) (Clark et al., 1994), which was abruptly and completely replaced by *H. sapiens* derived from Africa 100–200 ka ago. This Africa-derived *H. sapiens* is ancestral to all human populations in the world, with little or no genetic contribution from ancient *Homo* elsewhere, such as *H. erectus* in eastern Asia and the Neanderthals in Europe (e.g., Stringer, 1994; Ovchinnikov et al., 2000). Although the out-of-Africa school insists that mitochondrial DNA should be the single most important and reliable supporting ''evidence'' for its model (Actually the DNA clock itself is model-dependent), this model must be tested further in a more reliable chronological context. For instance, the proposal for the so-called slower evolution rate for Asian *H. erectus* is based on an incorrect chronological framework, which has been recently challenged in the light of new dates and discoveries (e.g., Hou et al., 2000; Shen et al., 1996; Shen and Wang, 2000). Recent technical developments in Quaternary chronology also revealed that the established chronological framework for human evolution has been significantly compressed. Important events, such as the earliest dispersion of genus *Homo* out of Africa (Balter and Gibbons, 2000), the arrival of *H. erectus* in Asia (Swisher et al., 1994), the interface between late *H. erectus* and archaic *H. sapiens* (Clark et al., 1994), and the appearance of early modern *H.*

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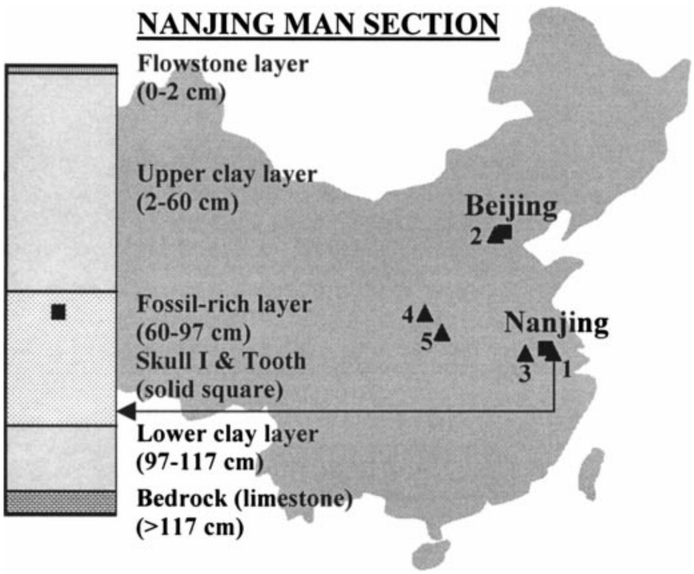

Figure 1. Sketch map showing localities of Nanjing Man (Tangshan) and other relevant *H. erectus* sites in China. Triangles with numbers indicate *H. erectus* sites: 1—Tangshan, 2—Zhoukoudian (Peking Man site), 3—Hexian, 4—Lantian, 5—Yunxian. Cross section at left shows stratigraphic sequence in the small cave where Skull I and a tooth were found.

sapiens (Grün and Stringer, 1991; McDermott et al., 1993, 1996) were pushed to much earlier times. Such a trend is having a significant impact on models concerning human evolution (e.g., Thorne et al., 1999).

Bearing the above important issues in mind, we have undertaken a thermal ionization mass spectrometry (TIMS) U-series chronological study of Nanjing Man, a recently discovered *H. erectus*, which shows dramatic similarities to the hotly debated Peking Man from Zhoukoudian (Xu et al., 1993; Mu et al., 1993). Considering the uncertainties and conflicts in the Peking Man chronology (e.g. Shen et al., 1996; Shen and Wang, 2000; Zhou et al., 2000; Grün et al., 1997), an increased precision and accuracy for the age of the morphologically analogous Nanjing Man will be useful for understanding the Peking Man sequence and the evolution of *H. erectus* in China. In addition, U-series dating of more reliable flowstone will provide an independent assessment of U-uptake history in fossil teeth, a factor that is crucial for the interpretation of their ESR and U-series ages (e.g., Grün et al., 1999). ESR and U-series dates of fossil teeth have played an important role in establishing chronological frameworks for many important hominid sites in the world (e.g., Grün and Stringer, 1991; McDermott et al., 1993; Grün et al., 1997, 1998; Chen and Zhang, 1991), but their reliability needs further evaluation.

NANJING MAN SITE

In 1993, two crania (a male and a female) and one tooth of *H. erectus*, named Nanjing Man, were discovered in a karst cave called Hulu Cave at Tangshan, 26 km east of Nanjing, eastern China (Mu et al., 1993) (Fig. 1). The cave, 80 m long, 35 m wide, 15 m high, and at 85.9 m altitude, was developed in Ordovician limestone. The hominid fossils were excavated from a small cave within the big cave. The stratigraphic sequence in the small cave comprises, from bottom to top, a lower brown clay layer (97–114 cm), a fossil-rich layer (60–97 cm), an upper brown clay layer (2–60 cm) and an overlying flowstone layer (0–2 cm). The female cranium (Skull I) and the tooth were discovered within the fossil-rich layer (60–97 cm). The male cranium (Skull II) was unearthed within the entrance wall to the small cave, where it was associated with a fossil-bearing conglomerate layer. Since it was first discovered by local peasants, its stratigraphical location is still controversial (e.g., Wang et al., 1999). Morphological evidence suggests that the three hominid fossils belong to three individuals of probably different evolution stages. The overlying flowstone layer can be traced throughout the big cave, where it becomes thicker and impure. On top of the flowstone layer grow numerous well-preserved stalagmites and still-dripping stalactites.

The crania and tooth are analogous to those from both Zhoukoudian (Peking Man site) and Hexian; the similarity to those at Zhoukoudian is much greater than that at Hexian, although Hexian is only 50 km southwest of Nanjing (Mu et al., 1993). In particular, Skull II shows the greatest similarity to Peking Man in its frontal bone. However, Nanjing Man's nasal bone is distinctive. Apart from the morphology, faunal compositions coexisting with Nanjing Man are monotonic and remarkably similar to those found at the Peking Man site, which are ubiquitous in northern China (Xu et al., 1993). Typical mammal fossils of south China type, such as *Ailuropoda*, *Megatapirus*, *Tapirus*, *Elaphodus*, and *Capricornis sumatraensis*, are not present. Limited pollen samples recovered from clays adhering to bone fossils are also typical of northern China; no modern subtropical species characteristic of southern China are found (Liu et al., 1998). Such records suggest that the continental temperature at the time when Nanjing Man crania were deposited was significantly lower than that of today. On the basis of such evidence, Xu et al., (1993) and Liu et al. (1998) proposed that Nanjing Man lived during a glacial period.

SAMPLE SELECTION AND ANALYTICAL PROCEDURES

Samples were obtained from the 2-cm-thick flowstone from the small cave, which directly overlies the Nanjing Man fossils. The flowstone is composed of fine-grained pure crystalline calcite; there is no discernible alteration or recrystallization. Three subsamples (GB-B, GM-M, and GB-T) were cut parallel to the growth layer from bottom to top. A stalagmite knocked down by construction workers was also collected. This stalagmite comprises pure radiating calcite crystals perpendicular to concentric transparent growth layers. Two clay-rich layers, possibly representing growth hiatuses, are visible; initially we suspected that these layers correspond to the clay layers bracketing the Nanjing Man fossil layer. Five subsamples (TSS-1 to 5), cut parallel to the growth layer, bracket the clay-rich layers. In addition, three samples of deer teeth (two of dentine and one of enamel) recovered from the fossil-rich layer within the small cave were also collected for this study.

Our analytical procedures are modified after Edwards et al. (1987) and Ludwig et al. (1992). Sample chips weighing 0.2–1.5 g were ultrasonically cleaned, spiked with a ^{229}Th-^{233}U mixed tracer, and completely dissolved in double-distilled HNO_3. After total dissolution, concentrated H_2O_2 was added to decompose organic matter and to ensure complete spike-sample mixing. After standard Fe hydroxide coprecipitation to preconcentrate U and Th, the precipitates were redissolved in 0.2 ml 7 *M* HNO_3 and purified using standard ion-exchange methods (Edwards et al., 1987). U and Th fractions were loaded separately onto zone-refined rhenium single filaments and sandwiched into two graphite layers. A Fisons VG Sector 54-30 thermal ionization mass spectrometer, equipped with a WARP filter and an ion-counting Daly detector, was used to determine U and Th concentrations and isotopic compositions. Both Th and U are measured on the Daly detector in peak-jumping mode. The thermal ionization mass spectrometry (TIMS) procedure allows an average throughput of four to five samples per day. Total procedural blanks are negligible at less than 10 pg for both Th and U. Repeated analysis of the international HU-1 uraninite standard yields an average ^{234}U/^{238}U activity ratio of 1.0015 $\pm$ 0.0009 (2 σ_m, $N = 16$), within error of the value reported by Ludwig et al. (1992) (using decay constants of Edwards et al., 1987). ^{233}U/^{229}Th ratio of

TABLE 1. U-Th ISOTOPIC DATA FOR SAMPLES FROM NANJING MAN SITE, TANGSHAN CAVES, NANJING, EASTERN CHINA

	U (ppm)	^{230}Th/^{232}Th	^{234}U/^{238}U	^{230}Th/^{238}U	^{230}Th Age (ka)	Initial ^{234}U/^{238}U
Flowstone overlying Nanjing Man fossil layer						
GB-T	0.278	11,960	1.2716 ±41	1.3770 ±56	571 +91/-58	2.370
GB-M	0.349	23,750	1.2787 ±25	1.3879 ±50	579 +67/-44	2.440
GB-B	0.213	4,524	1.2809 ±25	1.3894 ±65	566 +82/-51	2.400
Mean					577 +44/-34	
Stalagmite growth layers						
TSS-1	0.166	707	1.3687 ±22	1.5045 ±43	517 +28/-23	2.600
TSS-2	0.216	1,278	1.3183 ±43	1.4389 ±53	544 +64/-42	2.490
TSS-3	0.248	1,491	1.2834 ±21	1.390 ±11	544 +120/-60	2.330
TSS-4.2	0.240	1,164	1.2273 ±18	1.3113 ±52	542 +53/-38	2.060
TSS-5	0.271	22,260	1.2640 ±17	1.3640 ±28	552 +32/-25	2.263
Deer enamel (En) and dentine (De) coexisting with Nanjing Man crania						
En-1	1.723	3,150	1.2479 ±25	0.8995 ±57	130.1 ±1.6	1.358
De-1	59.49	17,130	1.5634 ±23	1.7172 ±93	388 ±14	2.692
De-2	62.41	42,940	1.5160 ±16	1.6456 ±92	375 ±13	2.494

Note: Isotopic ratios refer to activity ratios. ^{230}Th ages are calculated using program ISOPLOT/EX of Ludwig (1999). Decay constants are those in Edwards et al. (1987). All errors are quoted at 2 sigma level. TSS-1 to TSS-5 were cut at 7.1, 20.3, 26.3, 31.3, 32.8, and 40.1 mm from the skin of the stalagmite. The two clay-rich layers are located at 23.6 and 36.4 mm from the skin.

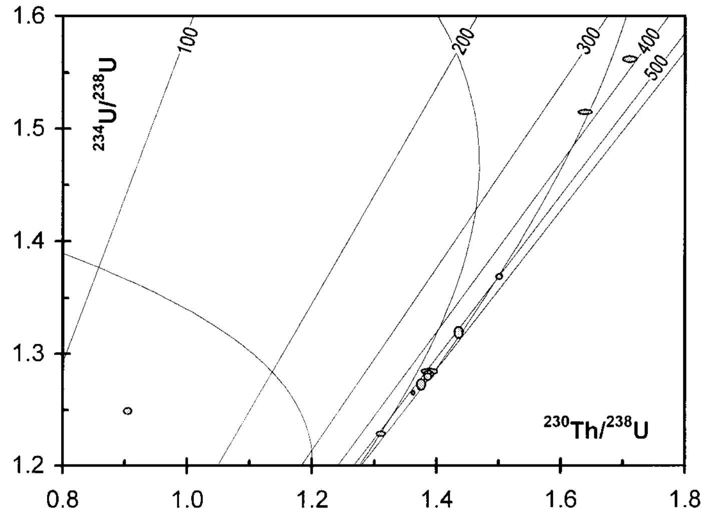

Figure 2. ^{234}U-^{230}Th isotopic evolution of samples from Nanjing Man site. Curved lines are closed-system isotopic evolution for initial ^{234}U/^{238}U activity ratios of 1.5, 2.0, and 2.5. Straight lines with numbers are isochron ages (ka). Ellipses are ratios and errors for individual samples shown in Table 1.

the mixed spike was calibrated against HU-1 standard, whose isotopic systematics are known to be in secular equilibrium, with ^{230}Th/^{234}U activity ratio = 1 (Cheng et al., 1996). This calibration method has the advantage of minimizing errors propagated from decay constant uncertainties for ^{230}Th and ^{234}U, which is particularly important to ensure accuracy for dating samples older than 400 ka, whose isotopic ratios are close to values in secular equilibrium.

RESULTS AND DISCUSSION

The stalagmite and flowstone samples contain much lower U (0.17–0.35 ppm) than the tooth enamel (1.7 ppm) and dentine (59.5–62.4 ppm) (Table 1). All samples have high ^{230}Th/^{232}Th activity ratios (707–42,900), suggesting that initial/detrital Th contribution is negligible in ^{230}Th age calculation.

The flowstone samples yield analytically indistinguishable ages of 566 +82/−51, 579 +67/−44, 571 +91/−58 ka, respectively, which give a mean of 577 +44/−34 ka, using Monte Carlo methods. This mean age is within error of ages of 580 +50/−40 to 600 +60/−50 ka, and 563 +50/−36 ka reported by Cheng et al. (1996) and Wang et al. (1999) for the same flowstone layer, respectively (isotopic ratios being normalized against measurements of HU-1 standard). Such values are considered as the minimum age estimate for the Nanjing Man fossils, at least for Skull I and the tooth in the small cave.

The five subsamples of the stalagmite growth layers yield ages ranging from 517 +28/−23 ka to 552 +32/−25 ka, all consistent with stratigraphic order. These ages confine the time for the formation of the two clay-rich layers to between 544 +64/−42 ka and 552 +32/−25 ka. The maximum 553 +32/−25 ka age is analytically indistinguishable from the mean age of the flowstone. On the basis of these data combined with the field relationship, we consider that the stalagmite is more likely to be younger than the flowstone, and the two clay-rich layers in the stalagmite possibly correspond to flooding events rather than clay horizons bracketing the fossil layer. The continuous nature of the growth layers and the similarity in their initial ^{234}U/^{238}U activity ratios (e.g., Table 1, Fig. 2) also suggest that both the stalagmite and the flowstone may have formed under similar climatic conditions.

Comparison with the SPECMAP deep-sea oxygen isotopic record of Imbrie et al. (1984) suggests that the flowstone and stalagmite formed during oxygen isotope stages 14–15, the mean age of the flowstone being in the warmer stage 15. Although stage 14 represents a colder period, it is significantly milder than other glacial periods, such as stages 6, 12, and 16. Because of this, significant speleothem growth during stage 14 is not unexpected. Thus, considering the fact that the flowstone defines only the minimum age of the Nanjing Man, and the evidence that Nanjing Man fauna formed during a glacial period, we infer that the Nanjing Man crania were deposited during glacial oxygen stage 16 (~620 ka), if not before.

Despite the established similarity between Nanjing Man and Peking Man, their stratigraphic correlation is not clear. We suggest that the Nanjing Man sequence is most likely correlated with layer 6 of Zhoukoudian, a thick (7.12 m) breccia layer with pollen records and fossil assemblage reflecting a cold stage (Zhou et al., 2000). The overlying fifth layer consists of a flowstone layer ~90 cm thick, reflecting a warm period. This flowstone layer yielded TIMS U-series ages ranging from 535 to >600 ka (Shen and Wang, 2000), remarkably similar to the age of the flowstone layer overlying the Nanjing Man fossil layer.

Mu et al. (1993) suggested that although the Nanjing and Hexian hominid sites are less than 50 km apart, the fossils are significantly different. Faunal composition at Hexian shows a mixture of cold-adapted northern mammal fossils and subtropical southern elements, significantly different from the Nanjing Man fauna. Grün et al. (1998) also acknowledged the analogy and difference between Hexian cranium and Skull HIII of layer 3 at Zhoukoudian, the youngest member of the Peking Man family, the Hexian cranium being probably more advanced. The combined ESR and U-series results constrain the age of the Hexian hominid to 412 ± 25 ka, which is identical to the TIMS U-series age of 412 ± 13 ka for a flowstone layer overlying Skull HIII of Zhoukoudian (Shen et al., 1996). Grün et al. (1998) proposed that the observed morphological differences between Hexian and Zhoukoudian reflect regional diversity in *H. erectus* evolution. However, the new age relationship described here suggests that *H. erectus* at Lantian, Yunxian (Chen et al., 1997), Zhoukoudian layers 8–11, Tangshan, Zhoukoudian layer 3 and Hexian (see Fig. 1) may represent different evolution stages of the Asian *H. erectus*, and their regional migration was probably related to cyclic climate changes.

Our study, combined with those recently reported for Zhoukoudian and Hexian, does not appear to support either a possible coexistence of late *H. erectus* and early *H. sapiens* in China (Chen and Zhang, 1991) or an evolution rate of Asian *H. erectus* slower than that of the African counterparts (Clark et al., 1994). These hypotheses were based mainly on unreliable ESR and α-counting U-series dates of fossil teeth—e.g., the 230 ka age for the youngest member of the Peking Man family. The recent dates suggest that *H. erectus* at key sites in China should be older than 400 ka, the vast majority being older than 600 ka, the time of transition between *H. erectus* and *H. sapiens* in Africa (Clark et al., 1994). The antiquity of Chinese *H. erectus* allows more time for them to evolve to *H. sapiens* in Asia.

The three fossil teeth from the Nanjing Man site yield significantly younger apparent ages. The two dentine samples give indistinguishable ages of 388 ± 14 and 375 ± 13 ka, respectively, only two thirds of the minimum age of Nanjing Man. Such ages are comparable to the 201 ± 58 to 395 ± 69 ka (2 σ) ESR and α-counting U/Th dates for teeth from the same fossil layer (Chen et al., 1996), which suggests that U uptake history in the dentine does not follow an early uptake model; rather, it is intermediate between early and linear uptake models, being closer to the linear model. The enamel sample gives an age of 130.1 ± 1.6 ka, which is less than one fourth of the minimum age of Nanjing Man, implying a delayed U-uptake history. The significantly lower initial $^{234}U/^{238}U$ ratio in this enamel also indicates that U must be taken up externally, rather than from coexisting dentine, unlike the conclusion of Grün et al. (1999). Our study provides a means for independent testing of the reliability of open-system modeling and age estimation based on combined ESR and U-series dates of teeth (Grün and McDermott, 1994), as well as the assumption that concordant ESR and U-series dates reflect an early uptake history (McDermott et al., 1993). The open-system modeling approach, which has been used frequently for dating many important hominid sites (e.g., Grün et al., 1998, 1999), may not apply to the enamel in our case.

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