

Rainer Grün

*Quaternary Dating Research Centre,
Australian National University,
Canberra ACT 0200, Australia*

Pei-Hua Huang

*Department of Earth and Space
Sciences, University of Science and
Technology of China, Hefei, 230026,
P.R. China*

Xinzhi Wu

*Institute of Vertebrate Palaeontology and
Palaeoanthropology, Academia Sinica,
P.O. Box 643, Beijing 100044, China*

Chris B. Stringer

*The Natural History Museum,
Department of Palaeontology, Cromwell
Road, London SW7 5BD, England*

Alan G. Thorne

*Division of Archaeology and Natural
History, Australian National University,
Canberra ACT 0200, Australia*

Malcolm McCulloch

*Research School of Earth Sciences,
Australian National University,
Canberra ACT 0200, Australia*

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ESR analysis of teeth from the palaeoanthropological site of Zhoukoudian, China

An ESR dating study on teeth collected from layers 3, 6/7 and 10 at Locality 1, Zhoukoudian provides results that are in general agreement with an earlier multi-dating study and confirm an age range of 300–550 ka for the *Homo erectus* remains in the Peking Man Cave. Uncertainties due to U-uptake and the external gamma dose rates do not allow very precise age estimates for the respective layers. © 1997 Academic Press Limited

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Introduction

Zhoukoudian Locality 1 is a karstic cave site about 50 km southwest of Beijing. The cave lies on the northern slope of Longgu-shan (Dragon Bone Hill) and is developed in Middle Ordovician limestone. The cave is about 48 m high (between 80 and 128 m a.s.l.), 107 m long (Ren *et al.*, 1981) and was filled with sediments. The stratigraphic sequence has been subdivided into 17 layers (Yang *et al.*, 1985) (see Table 1). The cave evolution and the stratigraphic chronology were studied by Huang (1993a,b, 1995).

The remains of about 40 *H. erectus* individuals and more than 100,000 artefacts have been excavated at this site since excavations started in 1921. The hominids are commonly known as “Peking Man”, *Sinanthropus pekinensis*. Two hominid teeth were found at Locality 1 in 1921, followed by a third one in 1927. The first hominid skull-cap (E) was discovered in layer 11 (locus E) in December 1929 (Pei, 1929). In 1936, three skull-caps (LI, LII and LIII) were excavated from layers 8–9 (locus L). A total of five calvaria and other cranial fragments and

Table 1 Depositional cycles and ages of the cave deposits of Zhoukoudian

Layer	Thick-ness (m)	Lithological description	Depositional character	Previous age estimates (ka)	Loess sequence	Oxygen isotope stages and boundaries (ka)	Magnetic polarity	
1	1.5	Brownish yellow breccia with silt	Colluvium		L2	6	B R U H N E S N O R M A L E P O C H	
2	1.7	Brownish red silty breccia and travertine	Sedimentation of impound water	221 ± 84-E(1) 230 ⁺³⁰ ₋₂₃ -U(3)	S2	190 7		
3	3.6	Coarse and large breccia with skull cap HIII	Colluvium and collapse of cave walls and roof	256 ⁺⁶⁰ ₋₄₀ -U(3) 249 ± 51-U(4) 282 ± 59-E(2)	L3	245 8		
4	6.9	Coloured ash and clayey silt	Sheet flow and evidence for the use of fire	292 ± 26-T(8) 306 ± 56-F(6) 316 ± 80-E(2) 320 ± 64-E(2)	S3	295 9		
5	0.4	Travertine	Chemical precipitation from underground flow			330		
6	7.1	Fine and coarse breccia and huge blocks	Colluvium and collapse of cave walls and roof	355-U(5) 368 ± 85-E(2)	L4	375 10		
7	1.5	Greyish yellow fine silt	Sedimentation from underground flow	385 ± 85-E(2) 396 ± 46-E(2)	S4	415 11		
8-9	4.0	Breccia and blocks, skull-caps LI-LIII	Colluvium and collapse of cave walls and roof	423 ± 80-E(2)	L5	460 12		
10	0.6	Coloured ash and silty clay	Sheet flow and evidence for the use of fire	462 ± 45-F(7)	S5a	520 13		
11	0.8	Greyish brown breccia with skull cap EI	Disintegration of cave wall	585 ± 105-E(2)	S5bc	555 14		
12-1	0.9	Brown coarse sand with fine gravels	Sedimentation from underground flow		S5de	615 15		
12-2	0.6	Brown coarse sand and breccia	Disintegration of cave wall	669 ± 84-E(1)	L6	660 16		
13-1	0.3	Reddish brown silty clay	Sheet flow		S6	690 17		
13-2	3.0	Breccia	Disintegration of cave wall		L7	765 18		
13-3	1.5	Brown silty clay	Sheet flow		S7	785 19		
								Bruhnes Matuyama Boundary

Loess sequence: Liu & Ding (1980), Liu (1985), Wang & Sadao (1985), Kukla (1987); Oxygen isotope stage boundaries: Prell *et al.* (1986), Martinson *et al.* (1987), Shackleton *et al.* (1990).

Dating results: U, U-series; T, thermoluminescence; F, fission track; E, electron spin resonance. (1) Preliminary results (Huang *et al.*, 1991a); (2) Huang *et al.* (1993a); (3) Zhao *et al.* (1985); (4) Yuan & Chen (1980); (5) Xia (1982); (6) Guo (1989); (7) Liu *et al.* (1985); (8) Pei (1985).

teeth were found before 1937 all of which were kept in the Peking Union Medical College. In late 1941, these specimens, along with the rest of the collections were sent from Beijing to be shipped to the U.S. for safety. However, the shipment was lost and the whereabouts of the collection remains unknown.

In 1966, the fragments of a calvarium (HIII) were excavated from layer 3 (locus H). These remains fit replicas of two other fragments that had been excavated in 1934 and 1936 from layer 3 and these pieces together form a virtually complete skull cap. They and some other fragments and teeth found after 1949 are the only remains of Peking Man that are still in China. The history of excavations at Zhoukoudian has been summarized by Wu & Dong (1985).

Chronology

The problems of dating the sedimentological sequence at Zhoukoudian have been addressed in a multidisciplinary study (Wu *et al.*, 1985). Palaeomagnetic measurements showed that the Bruhnes/Matuyama (B/M) boundary occurred between the 13th and 14th layer (Qian *et al.*, 1985). This means that layers 14–17 are older than 785 ka (Shackleton *et al.*, 1990; Spell & McDougall, 1992). An erosional surface has been observed between layers 15 and 16. However, it is difficult to assess the time span represented by this hiatus as the palaeomagnetic results for layers 14–17 are uniform and no reversal has been observed in this section.

The description of the sedimentology of layers 1–13, as well as the dating results, are summarized in Table 1. The dating methods applied include U-series, fission track and thermoluminescence (TL). U-series dating was applied on fossil bone, teeth and deer antler from layers 2–6 (Xia, 1982; Zhao *et al.*, 1985; Chen & Yuan, 1988; Yuan & Chen, 1991). Fission track ages were determined on fired sphene grains from ash layers from units 4 (Guo 1989) and 10 (Liu *et al.*, 1985). TL was applied on fired quartz grains of layer 4 (Pei, 1985). A further electron spin resonance (ESR) dating study was carried out on tooth enamel using samples from layers 3, 4, 6–9, 11 and 12 (Huang *et al.*, 1993). Further ESR age estimates on a travertine in layer 2 and a rhinoceros tooth from layer 12 have to be regarded as preliminary (Huang *et al.*, 1991*a,b*). Layers 13–17 contained no animal fossils so that no ESR dating studies could be carried out. An ESR dating attempt (not listed in Table 1) was carried out by Ikeya & Miki (1981) and Ikeya (1985) on bone samples from layers 6 and 10, resulting in ages between 200 and 550 ka.

According to the chronological framework summarized in Table 1 and the relationship of the layers with the boundaries of the oxygen isotope record, it is possible to estimate the age of the hominid remains: about 555–520 ka for sample EI from layer 11, 460–415 ka for specimen LI to LIII from layers 8–9, and 295–245 ka for HIII from layer 3 (Huang, 1993*a*).

We decided to complement the earlier ESR dating study by the analysis of horse and bovid teeth from the collection at Zhoukoudian (1067–1071) and two further rhinoceros samples collected by HPH (1139, 1140). The samples relate to layers 3 (1067, 1068), 6/7 (1139) and 10 (1069–1071, 1140). The basic principles of ESR dating have been recently reviewed in detail (Grün, 1989*a,b*, 1993; Ikeya, 1993).

Experimental

Several subsamples (denoted A, B, C) were separated from each tooth. Dentine and enamel were separated with a dental drill using diamond bits and a surface layer (S1/S2 in Table 2)

was removed from each side of the enamel in order to eliminate the volume that has been irradiated by external alpha rays. Ten aliquots of the enamel were irradiated using a calibrated gamma source with doses of: 0, 109, 198, 368, 927, 1362, 2100, 3608, 4965 and 7147 Gy. The past irradiation dose, D_E , and the associated errors were determined using the procedures outlined by Grün & Brumby (1994). U and Th analyses were obtained by induced coupled plasma mass spectrometry (ICP-MS) and K by flame photometry.

ESR measurements were carried out on a Bruker ECS 106 spectrometer with a 15 kG magnet and a rectangular 4102 ST cavity. The powder samples were recorded with the measurement parameters routinely applied in this laboratory: accumulation of eight scans with 1.015 Gpp modulation amplitude, 10.24 ms conversion factor, 20.48 ms time constant, 2048 bit spectrum resolution (resulting in a total sweep time of 20.972 s), 120 G sweep width and 2 mW microwave power.

One *in situ* gamma measurement was carried out in layer 10 yielding a value of 716 $\mu\text{Gy/a}$. However, this measurement was made in a small gap in the limestone containing very little sediment. It was, therefore, preferred to calculate the external gamma dose rates from the small sediment samples that were still adhering to the teeth. Considering an assumed water content of $10 \pm 5\%$, an external gamma dose rate of $1312 \pm 113 \mu\text{Gy/a}$ was obtained for layer 10, 829 $\mu\text{Gy/a}$ for layer 6/7 and 711 $\mu\text{Gy/a}$ for layer 3. In this study, we applied an α -efficiency value of 0.25. Although this value is somewhat higher than measured on enamel samples from Europe (0.11–0.15: see Grün & Katzenberger-Apel, 1994), it was obtained repeatedly for Chinese tooth samples (Chen *et al.*, 1994). A water content of $10 \pm 5\%$ was also used for the calculation of the external beta dose rate. The cosmic dose rates were calculated according to Prescott & Hutton (1988) and the dose rate conversion factors from Nambi & Aitken (1986) were used.

We also carried out mass spectrometric U-series analysis on a speleothem sample from layer 2. Unfortunately, due to the low U-concentration (22 ppb) and significant ^{232}Th concentrations it was not possible to calculate a meaningful $^{230}\text{Th}/^{234}\text{U}$ age estimate.

Results and discussion

Table 2 shows the results of the chemical and ESR analyses, as well as the ESR age calculations for early (EU) and linear (LU) U-uptake (see Ikeya, 1982). Figure 1 shows the ESR age estimates along with the age ranges of the oxygen isotope stages that were assigned to the respective layers.

Unlike the previous ESR studies at Zhoukoudian, we have calculated the ESR age results for both modes, early and linear U-uptake. It can clearly be seen that the average U-concentrations in enamel and dentine increase from the younger to the older units, thus causing a larger discrepancy between the EU and LU age estimates for the older layers. The ESR results for layer 3 are somewhat older than the age assignment to oxygen isotope stage 8 (see Table 1). For layer 6/7 the previous age assignment falls almost exactly between the EU and LU ESR age estimates, and for layer 10 the assignment to stage 13 (460–520 ka) agrees with average age results for the linear U-uptake model (504 ± 43 ka). As can be seen in Table 2, the external gamma dose rate is only a minor component of the total dose rate. If the *in situ* gamma dose rate of 716 $\mu\text{Gy/a}$ was used in the age calculations, the ages would shift 20% towards older ages, and the previous age assignment would fall between EU and LU values.

Table 2 Results of chemical analysis and ESR age estimates for samples from Zhoukoudian

Sample No.	D_E (Gy)	U(EN) (ppm)	U(DE) (ppm)	TT (μm)	SI/S2 (μm)	U (ppm)	Th (ppm)	K (%)	Sediment				Early U-uptake				Linear U-uptake			
									γ -D ($\mu\text{Gy/a}$)	β -D ($\mu\text{Gy/a}$)	int.D ($\mu\text{Gy/a}$)	DE-D ($\mu\text{Gy/a}$)	Total D ($\mu\text{Gy/a}$)	Age (ka)	int.D ($\mu\text{Gy/a}$)	DE-D ($\mu\text{Gy/a}$)	Total D ($\mu\text{Gy/a}$)	Age (ka)	int.D ($\mu\text{Gy/a}$)	DE-D ($\mu\text{Gy/a}$)
<i>Layer 3</i>																				
1067A	336 ± 13	0.07	4.86	900	25	1.40	8.11	1.27	7.11	± 114	276 ± 28	49 ± 7	152 ± 19	1188 ± 119	283 ± 30	22 ± 3	70 ± 9	1079 ± 117	312 ± 36	
1067B	354 ± 9	0.13	2.81	1000	25	1.40	8.11	1.27	7.11	± 114	257 ± 26	94 ± 15	84 ± 16	1146 ± 118	309 ± 33	42 ± 6	39 ± 8	1049 ± 117	338 ± 39	
1068A	320 ± 10	0.04	3.68	1550	25	1.02	4.02	0.83	7.11	± 114	117 ± 11	30 ± 4	85 ± 10	943 ± 115	340 ± 43	13 ± 2	39 ± 5	880 ± 114	363 ± 48	
1068B	331 ± 13	0.03	3.68	1850	25	1.02	4.02	0.83	7.11	± 114	100 ± 9	25 ± 4	75 ± 9	911 ± 114	363 ± 48	11 ± 2	35 ± 4	857 ± 114	386 ± 54	
<i>Layer 6/7</i>																				
1139A	526 ± 26	0.92	18.0	2500	25	1.62	6.51	1.40	8.29	± 64	126 ± 11	665 ± 92	275 ± 31	1895 ± 117	278 ± 22	313 ± 45	132 ± 15	1400 ± 80	376 ± 28	
1139B	496 ± 17	0.37	17.0	2300	25	1.62	6.51	1.40	8.29	± 64	136 ± 12	277 ± 36	286 ± 32	1528 ± 81	325 ± 21	128 ± 17	136 ± 15	1229 ± 69	404 ± 27	
1139C	477 ± 13	0.16	16.6	1800	25	1.62	6.51	1.40	8.29	± 64	171 ± 15	119 ± 19	339 ± 40	1458 ± 80	327 ± 20	54 ± 10	160 ± 19	1214 ± 69	393 ± 25	
<i>Layer 10</i>																				
1069A	1307 ± 52	0.87	37.1	1400	25	4.02	12.2	2.15	13.12	± 113	370 ± 34	659 ± 94	936 ± 109	3277 ± 186	399 ± 28	312 ± 44	450 ± 53	2444 ± 137	535 ± 37	
1069B	1462 ± 36	1.00	47.8	1250	25	4.02	12.2	2.15	13.12	± 113	404 ± 38	753 ± 108	1292 ± 151	3761 ± 220	389 ± 25	359 ± 51	625 ± 74	2700 ± 149	541 ± 33	
1070A	1403 ± 35	0.82	37.7	1300	25	3.09	11.4	1.93	13.12	± 113	0 ± 0	606 ± 88	2131 ± 176	4052 ± 227	346 ± 21	294 ± 43	480 ± 56	2651 ± 149	529 ± 33	
1070B	1661 ± 137	0.98	39.3	1350	25	3.09	11.4	1.93	13.12	± 113	0 ± 0	741 ± 107	2291 ± 189	4344 ± 245	382 ± 38	360 ± 52	495 ± 58	2795 ± 156	594 ± 59	
1070C	1420 ± 81	0.92	48.8	1350	25	3.09	11.4	1.93	13.12	± 113	331 ± 31	694 ± 100	1257 ± 146	3594 ± 213	395 ± 32	331 ± 48	608 ± 71	2582 ± 145	550 ± 44	
1070D	1145 ± 43	0.69	49.2	1200	25	3.09	11.4	1.93	13.12	± 113	363 ± 35	499 ± 73	1336 ± 153	3510 ± 207	376 ± 22	236 ± 35	643 ± 75	2554 ± 144	448 ± 30	
1071A	1073 ± 27	0.23	33.2	950	25	3.66	13.0	1.78	13.12	± 113	431 ± 44	169 ± 24	1049 ± 118	2961 ± 171	362 ± 23	79 ± 12	500 ± 57	2322 ± 134	462 ± 30	
1071B	1060 ± 28	0.25	30.3	950	25	3.66	13.0	1.78	13.12	± 113	431 ± 44	184 ± 27	959 ± 114	2886 ± 169	367 ± 24	86 ± 12	456 ± 55	2285 ± 134	464 ± 30	
1071C	1050 ± 29	0.33	22.1	1300	25	3.66	13.0	1.78	13.12	± 113	0 ± 0	247 ± 33	1384 ± 120	2843 ± 168	370 ± 24	117 ± 16	279 ± 43	2049 ± 128	512 ± 35	
1140A	839 ± 20	0.17	26.6	2200	25	2.32	12.5	1.85	13.12	± 113	201 ± 16	132 ± 21	475 ± 54	2120 ± 128	396 ± 26	61 ± 10	225 ± 26	1799 ± 117	466 ± 32	
1140B	855 ± 28	0.17	26.8	2100	25	2.32	12.5	1.85	13.12	± 113	210 ± 18	132 ± 20	499 ± 57	2153 ± 129	397 ± 27	61 ± 10	236 ± 27	1819 ± 118	470 ± 34	
1140C	878 ± 26	0.17	25.0	2000	25	2.32	12.5	1.85	13.12	± 113	220 ± 19	132 ± 21	485 ± 55	2149 ± 129	409 ± 27	61 ± 10	230 ± 26	1823 ± 118	482 ± 34	

EN, enamel; DE, dentine; TT, total enamel thickness; SI/S2, surface layer removed from each side of the enamel samples. Error in D_E after Grün & Brumby (1994); beta dose attenuation after Grün (1986); Alpha efficiency: 0.25 ± 0.02; initial $^{234}\text{U}/^{238}\text{U} = 1.4 \pm 0.2$ in enamel and dentine; water in sediment 10 ± 5 wt.-% (for beta rate); ICP-MS uncertainties (U, Th): 10%. Flame photometry detection limit (K-SED): 0.05%.

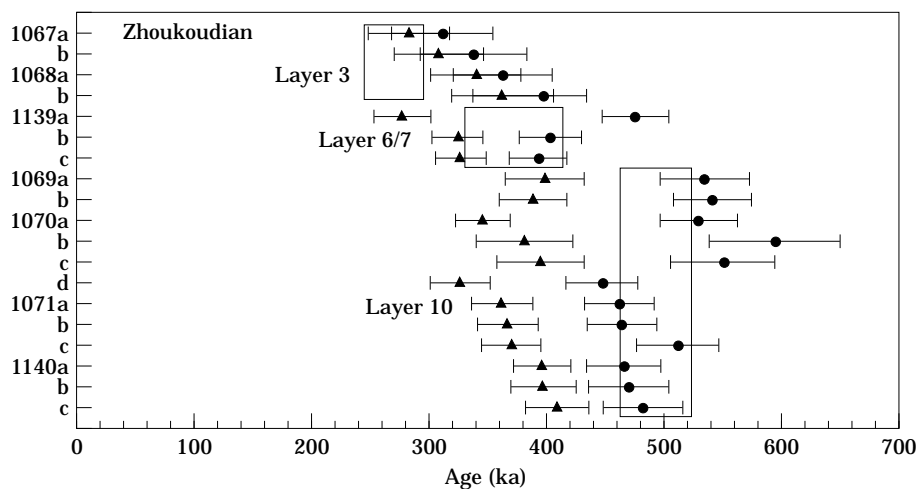


Figure 1. ESR age estimates for tooth samples from Zhoukoudian. The boxes represent the durations of the oxygen isotope stages that have been assigned to the respective layers (see Table 1). No precise depth information was available for the samples. (●) LU; (▲) EU; (□), expected age range for layers.

This study demonstrates the problems of working with museum specimens where it is difficult to establish the external gamma dose rate from very small sediment samples. For example, layer 10 is a relatively thin unit sandwiched between two breccias. Apart from the fact that the precise position of the teeth with respect to the breccia layers was not recorded, the simulation of the external gamma dose rate from fine grain sediment samples recovered from tooth samples is fraught with large uncertainties when the environment consists of inhomogeneous, coarse grain sediments (Schwarcz, 1994). Unfortunately, we were not able to carry out a detailed gamma spectrometric survey at the site which would require drilling rather large holes into the sediments. It also emphasizes a general problem of ESR dating because it is not possible to postulate the specific mode of uranium uptake for a given site without further U-series analysis on the same teeth (see McDermott *et al.*, 1993; Grün & McDermott, 1994). Usually, the correct age lies between the EU and LU age estimates (Grün & Stringer, 1991), but there are also cases where a very late U-uptake must have occurred (see e.g. Grün *et al.*, 1988; Grün, 1996). In general, the ESR results do not contradict the previously published multi-dating study (Wu *et al.*, 1985) and confirm that the hominid remains found in layers 3–11 are in the range of about 300–550 ka. Although a more detailed ESR/U-series dating study in conjunction with a detailed gamma spectrometric survey of the site may give more precise age estimates for the various layers, it is unlikely that an increased accuracy can be obtained that would allow correlation of specific oxygen isotope stages to a particular sedimentological unit beyond stage 8 (corresponding to layer 3).

Tighter age constraints can be expected from mass spectrometric U-series analyses if speleothem samples from layers 2 and 5 as well as matrix samples from other layers are analysed using isochrone techniques. This was recently successfully applied to matrix samples recovered from the Singa hominid (McDermott *et al.*, 1996).

The age estimates confirm the long-time span of the hominid deposits in the Peking Man Cave, and are somewhat older than previous estimates. In terms of human evolution, the older Zhoukoudian *H. erectus* samples are penecontemporaneous with fossils from western Eurasia

and Africa assigned by some to late *H. erectus* or to *Homo heidelbergensis*, while the youngest material may date from a time when the Neanderthals were already beginning to differentiate in Europe (Stringer & Gamble, 1993). These age estimates are broadly consistent with correlations with Eurasian mammalian faunas spanning Cromerian to Holsteinian stages but further contradict Aigner's long-held view (e.g. Aigner, 1987) that the hominid deposits represent only one interglacial phase.

Conclusion

The ESR age estimates presented in this study confirm the results of the previous multidating study (Wu *et al.*, 1985). They indicate that *H. erectus* occupied the Peking Man Cave at Zhoukoudian in the range of about 300,000–550,000 years.

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