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ESR and U-series analyses of teeth from the palaeoanthropological site of Hexian, Anhui Province, China

ESR and U-series analyses of teeth from the palaeoanthropological site of Hexian which contained *Homo erectus* remains, illustrate the limited effectiveness of stand-alone ESR and U-series age estimates on faunal materials. The problem lies in the unknown U-uptake history causing very large uncertainties in the age results of both techniques. This study demonstrates the particular strength that lies in the integration of ESR and U-series dating analyses allowing the estimation of the U-uptake history. We obtained a combined ESR/U-series age estimate of 412 ± 25 ka (average of six analyses on two teeth). This pinpoints the deposition of the faunal remains to the time of the transition between oxygen isotope stages 12 and 11. This is in agreement with the faunal composition which show a mixture of cold adapted northern mammals and more subtropical-tropical southern elements. The age also implies that the advanced Hexian *Homo erectus* occurred at a similar time as the less advanced *Homo erectus* specimens at Locality 1 at Zhoukoudian (LI-LIII).

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Received 29 July 1996

Revision received 24

October 1997 and accepted

13 December 1997

Journal of Human Evolution (1998) **34**, 555–564
Article No. hu970211

Introduction

Excavations in 1980 and 1981 in Longtandong (Dragon Pool Cave), a small limestone cave in Hexian County, Anhui Province (118°20'E; 31°35'N) produced portions of several human crania, a mandible with two teeth and nine isolated teeth (Huang *et al.*, 1982; Wu & Poirier, 1995). The most complete individual, PA 830, consists of the greater part of the calvarium of a young male individual, with much of the base broken away (Wu & Dong, 1982, 1985: their Figure 5.7). Assessment of the considerable body of faunal remains found in association with the human remains suggested Middle Pleistocene age (Huang *et al.*, 1982). In a deposit with a total depth of less than six metres, Huang and his co-workers found five distinct layers (from bottom to top): Layer 1: a yellow-grey sandy clay, approximately 1.5 m thick; Layer 2: a yellow brown sandy clay of variable thickness (0.7 to 1.4 m) containing the human remains and nearly all other faunal material; Layer 3: a yellow-green silty clay of 0.1 to 0.3 m thickness; Layer 4: a brownish red clay, approximately 2.3 m thick and Layer 5: a brown-red to brown clay of 0.2 to 0.4 m thickness. The faunal remains were concentrated in a limited space of 2 m by 6 m (width and length) and a thickness of about 1 m. The taphonomy of the faunal remains suggested a rapid deposition of Layer 2 (Huang *et al.*, 1982). No direct archaeological evidence was discovered although it is possible that some faunal remains show cut marks, which would suggest that the site was inhabited by the people whose remains were found there.

The Hexian calvarium is conventionally ascribed to *Homo erectus* and shows progressive features, with apparent similarities to a specimen (HIII) found in the upper part (Layer 3) of Locality 1 (The Lower Cave) at Zhoukoudian. Fossils from both sites share a low cranial vault, low frontal squama, details of form of the supraorbital and occipital

tori, relatively thick vault bone and well-developed angulation of the occipital and nuchal planes (Wu & Dong, 1985). There are certain differences in the remains of the two sites—the Hexian specimen shows a slightly flatter frontal squama, greater breadth of the frontal bone as a whole, a higher arch to the temporal bone's upper squamous margin and a greater dimension between inion and endinion.

The overall similarities and differences between the Hexian and the very important sequence from Zhoukoudian raise several crucial chronological and evolutionary issues. Given the time over which Zhoukoudian was occupied (Wu, 1983; Wu *et al.*, 1985; Grün *et al.*, 1997) and the uncertainties of dating its earliest and latest limits, any increased precision for sites with similar morphological remains is useful for the understanding of the Zhoukoudian sequence, over and above the significance to such sites like Hexian itself. Second, some of the detailed anatomical differences between Hexian and Zhoukoudian, like those between Lantian and Zhoukoudian, suggest regional and broader patterns of diversity and evolution. One interpretation of the affinities and significance of Hexian and Lantian is that they are a more southerly *H. erectus* form, in both geographic and environmental senses. This could be merely a minor regional variant of a broad East Asian morphology or, given the flattened frontals and other details, represent the expression of widespread gene flow linking the northern morphology of Zhoukoudian with the southern morphology of the Javan hominids. An accurate chronological assessment of the Hexian remains and the site is therefore important to environmental and evolutionary issues within China and beyond.

Layers 3 and 4 at Zhoukoudian have been correlated to oxygen isotope stages 8 and 9, representing an age range of 245 to 330 ka (Huang, 1993a,b, 1995; Grün *et al.*, 1997).

The fauna associated with the Hexian hominid represents a mixture of warm and cold adapted mammals and an age of about 200 ka was assigned to the site (Huang *et al.*, 1982). A U-series dating study of bone material (Chen & Yuan, 1988) yielded combined $^{231}\text{Pa}/^{230}\text{Th}$ ages in the range of 130 to 220 ka. Earlier ESR studies (Huang *et al.*, 1994, 1995*a,b*) yielded results of about 160–220 ka (early uranium uptake) and 250–350 ka (linear uranium uptake) for a rhinoceros tooth and a deer tooth from Layer 2.

Samples

Two rhinoceros tooth samples (1117 and 1118) were excavated by PHH at the site and further three bovid teeth were provided from the collection of the IVPP (1079 to 1081). All samples originated from Layer 2 and were analysed by ESR. Samples 1117 and 1118 were also analysed by U-series. The basics of both dating methods have been repeatedly described in detail (ESR: Grün, 1989*a,b*, 1993; Ikeya, 1993; U-series: Ivanovich & Harmon, 1992).

Experimental

Several subsamples were cut from each tooth. Dentine and enamel were separated with a dentist's diamond drill and a surface layer (S1/S2 in Table 1) 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 was determined using the procedures outlined by Grün & Brumby (1994). U and Th concentrations were determined by ICP-MS (Grün & Taylor, 1996).

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 20.97 s), 120 G sweep width and 2 mW microwave power.

One *in situ* gamma measurement was carried out at the site yielding a value of 674 $\mu\text{Gy/a}$. 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 sediment analyses. Considering an assumed water content of $10 \pm 5\%$ and an average depth of 11 ± 2 m for the cosmic dose rate, an external gamma dose rate of $1491 \pm 360 \mu\text{Gy/a}$ was obtained (excluding the U-result of sample 1117 which is several standard deviation removed from the average of the other U-analyses).

Dose rate conversion factors were calculated according to Nambi & Aitken (1986) and the cosmic dose rate according to Prescott & Hutton (1988). 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 to 0.15: see Grün & Katzenberger-Apel, 1994), it was obtained repeatedly for Chinese tooth samples (Chen *et al.*, 1994; Liang *et al.*, 1995). A water content of $10 \pm 5\%$ was also used for the calculation of the external beta dose rates from the sediment as well as the dentine.

The analytical procedures for U-series analyses of dental material have been described by McDermott *et al.* (1996). Enamel and dentine samples were dissolved completely in 2–5 ml of sub-boiled teflon-distilled 7 M HNO_3 to which a few drops of concentrated HClO_4 had been added. At the dissolution stage a known weight of a mixed ^{229}Th – ^{236}U spike was added, and

following dissolution the spike was allowed to equilibrate with the sample for several days in a sealed teflon reaction vessel at 150°C. The acid was then evaporated to near-dryness under heat-lamps in a clean-lab, and the sample was re-dissolved in 7 M HNO₃. Ion-exchange chromatography was carried out in two stages (4 and 0.5 ml columns) using anionic ion exchange resin which had been pre-washed in 6 M HCl/H₂O and pre-conditioned in 7 M HNO₃. The major elements which comprised the matrix of the samples were eluted in 7 M HNO₃. Thorium was then eluted in 6 M HCl and U in 1 M HBr. The purified U and Th samples were loaded on separate out-gassed graphite-coated double-assembly Re filaments and the ²³⁴U/²³⁶U, ²³⁵U/²³⁶U, ²³⁰Th/²²⁹Th and ²³²Th/²²⁹Th ratios were measured in ion-counting mode using a high abundance sensitivity (≈10 pb), low dark noise (<0.03 cps) thermal ionization mass-spectrometer at the Open University (MAT 262 RPQ-II). Total procedural blanks were negligible at <40 pg for ²³²Th and <10 pg for ²³⁸U.

Samples 1117EN/DE and 1118EN/DE (EN=enamel; DE=dentine) were analysed by FM and these results have been used for the combined U-series/ESR results. Sample 1118NR was subsequently re-collected from the tooth and analysed by GY at ANU. The chemical pre-treatment of this sample differed only marginally from the above procedure (for details of the chemical pre-treatment see Grün *et al.* (1998) and mass spectrometric measurement see Stirling *et al.* (1995). The U-series results show good agreement between the two laboratories involved. The small differences in the results are most likely due to inhomogeneities in the samples.

The combined U-series/ESR age estimates were calculated with the mathematical algorithms developed by J. Chadam for open system modelling which were first presented by Grün *et al.* (1988). This model

uses a parameter, *p*, which describes the mode of uranium uptake. A *p*-value of -1 corresponds to closed system (EU) and a value of 0 to linear U-uptake (LU) (see below). Negative *p*-values indicate intermediate U-uptake histories whilst positive values indicate delayed U-uptake. In this particular case we had to force the program to assume a closed system for the analytical results of the dentine of sample 1118, because the U-uptake model does not allow for U-leaching (the high U-series results may have been caused by a slight U-loss).

Results

Tables 1 and 2 show the results of the ESR and U-series analyses, respectively. The ESR age estimates were calculated for early (EU) and linear (LU) U-uptake (see Ikeya, 1982). As shown in Figure 1, the ESR results show a relative large scattering. The EU average to 344 ± 48 ka and the LU to 465 ± 94 ka. Because ESR analyses as such do not allow the assessment of the U-uptake history, it is assumed that the correct age of the sample usually lies between the EU and LU age estimates (see Grün & Stringer, 1991). For Hexian, the best age estimate lies therefore in the range of 289 to 542 ka.

The U-series results show a similar large scattering. The closed system ages lie between about 150 and 650 ka. If the same open system rule applied to the U-dating, namely that the correct age lies probably between EU and LU models, the U-series results would cover the age range of about 150 to 1300 ka, because LU U-series ages are approximately double the EU ages (for more details see Bischoff *et al.*, 1995).

However, when combining the U-series and ESR results for open system modelling (Grün *et al.*, 1988; McDermott *et al.*, 1993; Grün & McDermott, 1994), the average age of samples 1117 and 1118 are 420 ± 21 and 405 ± 27 ka, respectively (see Table 3 and

Table 1 Results of chemical analysis and ESR age estimates for samples from Hexian

Sample No.	D _E (Cy)	U(EN) (ppm)	U(DE) (ppm)	TT (µm)	SI/S2 (µm)	U (ppm)	Th (ppm)	K (%)	Sediment					Early U-uptake					Linear U-uptake				
									Cosmic+γ-D (µGy/a)	β-D (µGy/a)	EN-D (µGy/a)	DE-D (µGy/a)	Total D (µGy/a)	Age (ka)	EN-D (µGy/a)	DE-D (µGy/a)	Total D (µGy/a)	Age (ka)	EN-D (µGy/a)	DE-D (µGy/a)	Total D (µGy/a)	Age (ka)	
1079A	871 ± 54	0.50	22.87	1450	50	7.51	9.9	1.56	1491 ± 360	361 ± 33	319 ± 44	439 ± 48	2610 ± 367	334 ± 51	144 ± 21	207 ± 23	2204 ± 363	395 ± 70					
1079B	772 ± 25	0.62	22.48	1450	50	7.51	9.9	1.56	1491 ± 360	361 ± 32	382 ± 51	421 ± 46	2655 ± 369	291 ± 41	173 ± 25	199 ± 22	2224 ± 365	347 ± 58					
1080A	767 ± 26	0.70	19.13	1450	50	9.41	7.7	1.11	1491 ± 360	350 ± 32	431 ± 44	359 ± 36	2631 ± 366	292 ± 42	195 ± 22	169 ± 17	2205 ± 362	348 ± 58					
1080B	841 ± 22	1.24	19.70	1500	50	9.41	7.7	1.11	1491 ± 360	340 ± 30	874 ± 106	387 ± 41	3082 ± 379	272 ± 34	405 ± 51	184 ± 20	2420 ± 365	347 ± 53					
1081A	1289 ± 31	1.71	25.56	1500	50	5.46	10.9	1.73	1491 ± 360	327 ± 29	1281 ± 150	524 ± 56	3623 ± 395	356 ± 40	606 ± 73	252 ± 27	2676 ± 370	482 ± 68					
1081B	1548 ± 35	2.87	18.87	1400	50	5.46	10.9	1.73	1491 ± 360	346 ± 31	2140 ± 237	405 ± 53	4382 ± 434	353 ± 36	1024 ± 117	197 ± 21	3058 ± 380	506 ± 64					
1081C	1270 ± 74	1.65	17.23	1400	50	5.46	10.9	1.73	1491 ± 360	186 ± 17	1250 ± 143	374 ± 39	3301 ± 390	385 ± 51	591 ± 72	180 ± 19	2448 ± 368	518 ± 84					
1117A	1653 ± 54	1.40	95.17	1600	25	40.5	1.57	0.20	1491 ± 360	875 ± 81	878 ± 74	1708 ± 150	4952 ± 405	334 ± 29	416 ± 36	825 ± 73	3607 ± 388	485 ± 50					
1117B	1542 ± 63	0.95	93.00	1400	25	43.3	2.1	0.29	1491 ± 360	1038 ± 97	582 ± 52	1812 ± 163	4923 ± 411	313 ± 29	273 ± 25	872 ± 80	3674 ± 383	420 ± 47					
1117C	1685 ± 62	1.62	88.17	1400	25	42.0	1.8	0.25	1491 ± 360	1003 ± 94	1001 ± 84	1726 ± 156	5221 ± 413	322 ± 28	473 ± 42	834 ± 77	3801 ± 383	443 ± 48					
1118A	1839 ± 77	1.23	73.92	1800	25	13.0	4.0	0.50	1491 ± 360	305 ± 28	1081 ± 91	1479 ± 134	4356 ± 396	422 ± 42	519 ± 44	718 ± 66	3033 ± 370	606 ± 78					
1118B	2130 ± 115	1.65	80.18	1900	25	13.0	4.0	0.50	1491 ± 360	292 ± 33	1453 ± 120	1538 ± 143	4774 ± 406	420 ± 43	700 ± 58	749 ± 70	3232 ± 372	620 ± 80					
1118C	1719 ± 82	1.20	86.0	1850	25	13.0	4.0	0.50	1491 ± 360	299 ± 34	1043 ± 88	1669 ± 150	4502 ± 401	382 ± 39	501 ± 43	810 ± 74	3101 ± 374	554 ± 71					

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 ²³⁴U/²³⁸U from U-series analyses in enamel and dentine; water in dentine and sediment 10 ± 5 wt.% (for beta dose rate); ICP-MS uncertainties for U and Th: 5%, flame photometry detection limit K sediment: 0.05%.

Table 2 Results of U-series analysis (activity ratios)

Sample No.	U (ppm)	$^{234}\text{U}/^{238}\text{U}$	$^{230}\text{Th}/^{232}\text{Th}$	$^{230}\text{Th}/^{234}\text{U}$	Age (ka)
1117 EN	1.2790 ± 0.0018	1.0386 ± 0.0037	>1000	0.7616 ± 0.0042	153.5 ± 1.8
1117 DE	86.065 ± 0.239	1.0055 ± 0.0033	>1000	0.8109 ± 0.0027	180.6 ± 1.6
1118 EN	1.3095 ± 0.0027	1.3228 ± 0.0060	>1000	0.9924 ± 0.0048	280.8 ± 5.4
1118 ENR	1.1121 ± 0.0005	1.2823 ± 0.0014	877 ± 4	0.9977 ± 0.0045	295 ± 11
1118 DE	67.897 ± 0.277	1.3057 ± 0.0058	>1000	1.0896 ± 0.0050	554^{+100}_{-60}

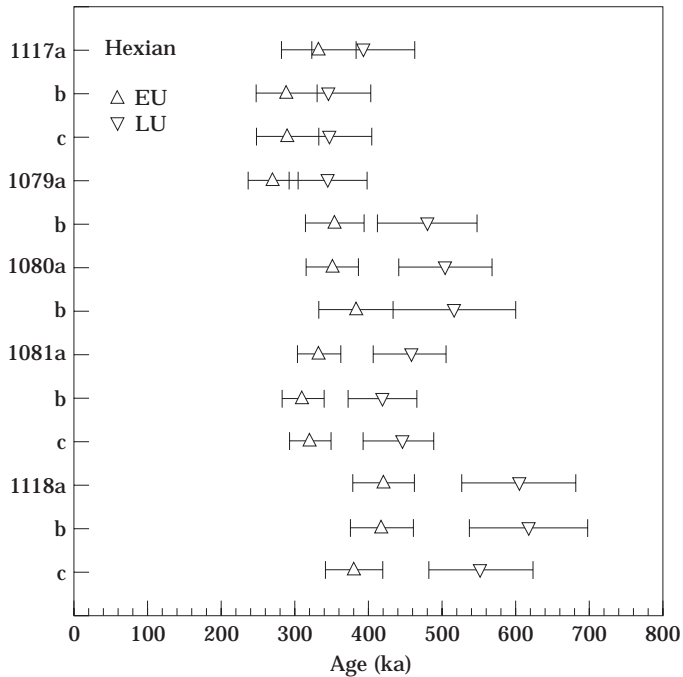


Figure 1. ESR age estimates on teeth from Hexian.

Figure 2), resulting in an average age of 412 ± 25 ka.

Discussion

The U-series results of sample 1117 lie well within the range established by an earlier U-series analysis of teeth from Hexian (Chen & Yuan, 1988) which were in the range of about 75 to 180 ka ($^{230}\text{Th}/^{234}\text{U}$), 60 to >120 ka ($^{231}\text{Pa}/^{235}\text{U}$) and 130 to 220 ka (combined $^{231}\text{Pa}/^{230}\text{Th}$ ages) whilst the results of sample 1118 are clearly older. This can be attributed to delayed U-uptake

into the samples studied by Chen & Yuan (1988) which probably had a similar U-uptake history as sample 1117 (see below).

The ESR dating results of this study of 344 ± 48 ka (EU) and 465 ± 94 ka (LU) are considerably older than those obtained by Huang *et al.* (1994, 1995a,b) which were in the range of 160 to 220 ka (EU) and 250 to 350 ka (LU). At the present stage, the discrepancy between the two sample sets is difficult to explain.

As repeatedly stated in numerous ESR dating studies (e.g., Grün & Stringer, 1991), the correct age of a sample most probably

Table 3 U-uptake parameters and combined U-series/ESR age estimates

Sample No.	p(EN)	p(DE)	Age (ka)
1117A	0 ± 0.20	-0.30 ± 0.15	443 ⁺⁷⁸ ₋₅₁
1117B	-0.16 ± 0.17	-0.41 ± 0.13	393 ⁺⁶⁶ ₋₄₇
1117C	-0.06 ± 0.18	-0.34 ± 0.14	424 ⁺⁷¹ ₋₄₉
1118A	-0.88 ± 0.02	-1 ± 0	420 ⁺⁴⁵ ₋₃₅
1118B	-0.88 ± 0.02	-1 ± 0	420 ⁺⁴⁵ ₋₃₆
1118C	-0.93 ± 0.01	-1 ± 0	374 ⁺³⁴ ₋₂₇

lies somewhere between the EU and LU ESR age estimates and there is no way of postulating a particular U-uptake model for a particular site without any further analysis. In the case of Hexian, the best age estimate of the Hexian fauna, and the hominid, would be somewhere between the EU and LU estimates of about 400 to 560 ka.

The combination of the U-series with the ESR results allows open system modelling for uranium (Figure 3). It can clearly be seen that the mode of U-uptake for the constituents of samples 1117 and 1118 are distinctively different. Sample 1117 shows a nearly linear U-uptake whereas sample 1118 shows nearly closed system (keeping in mind the restriction of closed system for the dentine—the combined U-series/ESR age result would not be much different if the isotopic ratios of the enamel were used for the dentine). The U-series ages of the dentine are significantly older than of the enamel. This is in agreement with detailed U-series analyses on two teeth (Grün *et al.*, 1998) which showed delayed U-uptake of enamel compared to adjacent dentine. The combined results show much less scatter than any of the separate analyses. This study also demonstrates, as for other sites such as Skhul in Israel (Grün & McDermott, 1994), that there is no single U-uptake mechanism neither within a site nor for the constituents of a single tooth (Grün *et al.*, 1998). On the other hand, the p-values found for sample 1117 and 1118 are all between -1 (=EU)

and 0 (=LU), confirming the general rule that the correct age of a sample usually lies somewhere between those limits (see above).

The U-series result of the dentine sample 1118DE is older than the corresponding ESR result. This points to the possibility of U-leaching. If U-leaching had occurred in recent times, it would be in the range of <6%. However, such a small value is well within the uncertainties in the analytical values of the samples. Swisher *et al.* (1996) recently argued for considerable U-loss from teeth (up to 50%) in order to explain discrepancies between U-series and ESR data sets in a study concerning the *H. erectus* sites in Java, Ngandong and Sambungmacan. The present data set indicates that small U-loss is possible, as has been observed in other studies (e.g., Badone & Farquhar, 1982), but not on the scale proposed by Swisher *et al.* (1996). This was also pointed out by Grün & Thorne (1997).

The combined U-series/ESR age estimates of samples 1117 (420 ± 21) and 1118 (405 ± 27 ka) indicate a deposition of the site around the transition of oxygen isotope stages 12 to 11 at 415 ka (Prell *et al.*, 1986; Shackleton *et al.*, 1990). The faunal remains indicate a mixture of cold adapted northern mammals and more subtropical-tropical southern elements (Huang *et al.*, 1982). This has been either interpreted as representing one or more interglacial (warm-humid) and glacial (cold arid) cycles in Eastern China (i.e., presenting a relatively long age range of at least 100 ka) or as a transitional climatic phase (which could be significantly shorter). Our results favour the latter interpretation.

This dating study indicates that the Hexian *H. erectus* specimen occurred more likely in the same time range as the less advanced *H. erectus* specimen LI-LIII rather than the morphologically similar specimen HIII at Locality 1 at Zhoukoudian. One should mention, however, that ESR analyses

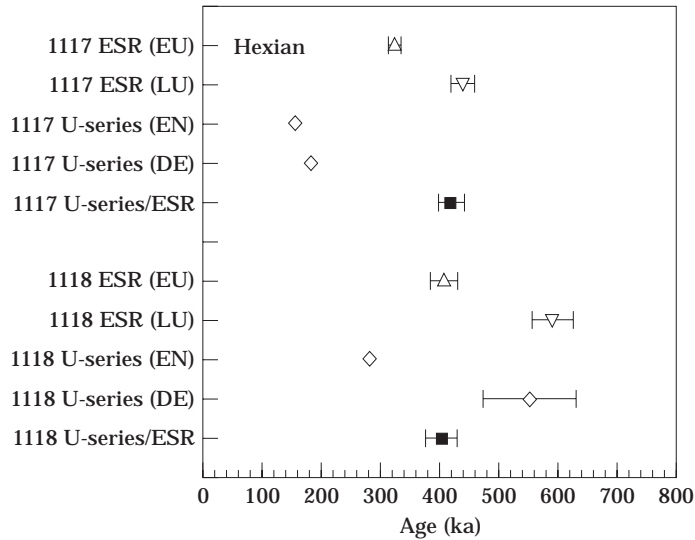


Figure 2. Age estimates for samples 1117 and 1118 (EN=enamel; DE=dentine). Both the ESR and U-series age estimates on the two teeth imply that the two teeth have distinctively different ages. The combined U-series/ESR age estimates are virtually indistinguishable and give the best age estimate for the site.

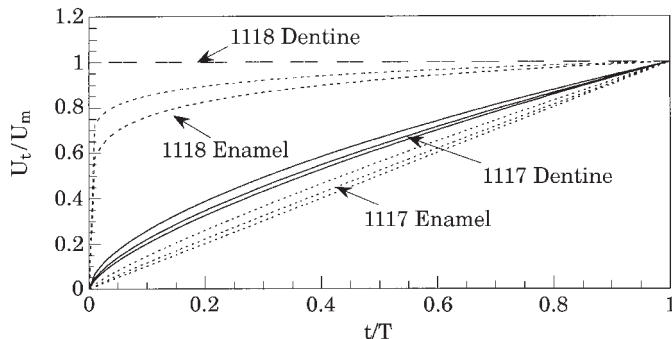


Figure 3. Modelled U-uptake histories. Sample 1118 shows U-uptake which is near to a closed system model whereas the constituents of sample 1117 are closer to a linear uptake model.

of Layer 3 at Zhoukoudian indicated a somewhat older age than previously proposed (EU: 324 ± 30 ka; LU: 350 ± 28 ; Grün *et al.*, 1997), i.e., the age of HIII could be older and therefore be closer to the Hexian specimen.

The proposed co-occurrence of morphologically different hominids at Hexian and Zhoukoudian is not necessarily an indication that the dating results are incorrect. Given the uncertainties of morphological

variation, gene flow and environmental zone across East and Southeast Asia, coincidence of morphologies and dates are unlikely to be approachable with the small number of human remains that is presently known.

Summary

Our study of teeth from Hexian demonstrates the restraints that apply to ESR and

U-series dating of faunal elements from archaeological sites. When applied on their own, it is not possible to reconstruct the U-uptake history of the teeth which seriously affects the reliability of both dating methods. The unknown U-uptake causes larger uncertainties in the dating results particularly of teeth with higher U-concentrations (>2 ppm U in dentine). For Hexian, the U-series analysis cover a range of 150 to 1300 ka and ESR about 290 to 560 ka. The integration of the results of the two methods allow the assessment of the uranium uptake history and the calculation of combined age results. Six repeated analyses on two teeth yielded an average age of 412 ± 25 ka. This pinpoints the deposition of the faunal remains of Layer 2 to the time of the transition of oxygen isotope stages 12 to 11. Our results imply that the advanced *H. erectus* of Hexian occurred at about the same time as the less advanced *H. erectus* specimens LI-LIII at Locality 1 at Zhoukoudian.

Acknowledgements

We wish to thank T. Prior, CSIRO, Canberra, for the use of the gamma source and L. Taylor, QDRC, for irradiating the sample and carrying out the elemental analyses. We thank M. McCulloch and G. Mortimer, RSES, Canberra, for their support in the U-series analyses and the use of the ICP-MS. This study was supported by the Exchange program between the Australian Academy of Sciences and the Academia Sinica with grants to RG and HPH. The Boise Fund provided travel funds to CBS. We thank the referees for constructive comments and Mrs F. M. Grün for corrections.

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