Journal of Archaeological Science 31 (2004) 965–977

Journal of Archaeological SCIENCE

http://www.elsevier.com/locate/jas

Coupled electron spin resonance (ESR)/uranium-series dating of mammalian tooth enamel at Panxian Dadong, Guizhou Province, China

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Received 10 May 2003; received in revised form 10 December 2003; accepted 17 December 2003

Abstract

Panxian Dadong (Guizhou province, southwestern China) is an archaeological cave site within an elaborate multi-genesis karst system that contains three stacked caves. Collaborative Sino-American archaeological excavation and multi-disciplinary studies have been in progress since 1996. An *Ailuropoda–Stegodon* faunal assemblage along with lithics and human teeth has been recovered from the deeply stratified deposits. The represented taxa are generally indicative of a Middle Pleistocene biostratigraphic age. Fifteen mammalian fossil teeth were collected during the 1998–2000 excavations. The enamel was dated by conventional ESR and coupled ESR/U-series dating techniques. The ESR early uptake (EU) and linear uptake (LU) model ages range from 120–300 ka. Uranium–thorium results from four dentine samples depict a variety of uptake histories the samples have undergone throughout the entire 6-m depth of the excavation units. The coupled ages suggest that samples at Panxian Dadong demonstrate linear uptake model behavior and indicate the true burial ages for the tooth samples. The upper and lower unit has mean LU ESR ages of 156 ± 17 ka and 258 ± 47 ka, respectively.

Keywords: Electron spin resonance; U-series dating; China; Panxian Dadong; Middle Pleistocene; Archaeology; *Ailuropoda–Stegodon*; Palaeoclimate

1. Introduction

Panxian Dadong Cave, located in Guizhou province, southwestern China (Fig. 1), is part of a large multi-genesis karst system that contains three connected and integrated stacked caves [32]. It is situated at 24°37′38″N, 104°44′E, at 230 m above the valley floor. The present elevation is due in part to uplift associated with the Qinghai–Xizang (Tibetan) Plateau [4,5,15]. The main chamber is 250 m deep, 23–56 m wide and has a vaulted ceiling ranging in height from 22–30 m. Excavations at Panxian Dadong have yielded a lithic assemblage [16,18] associated with an *Ailuropoda–Stegodon* fauna [21,34] that suggests a relative age of late Middle Pleistocene for the human activity at Panxian Dadong.

There are several Palaeolithic archaeological sites located near Panxian Dadong that have been dated from the late Middle to the early Late Pleistocene by both

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doi:10.1016/j.jas.2003.12.010

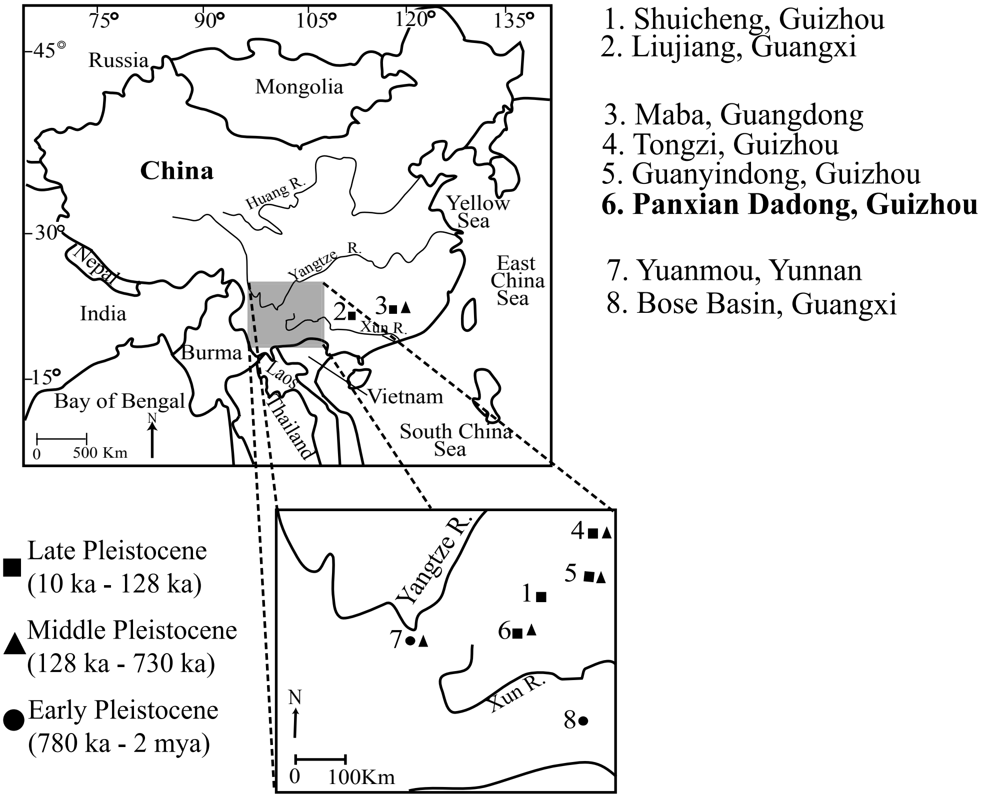

Fig. 1. Map showing the location of Panxian Dadong and neighboring archaeological sites, as well as the relative age of each site.

relative and absolute methods (Fig. 1) [2,9,24,27,29–31,33]. However, Panxian Dadong is unique because it possesses hominid fossil remains (5 teeth), over 6 m of stratified deposits with archaeological material, and well-preserved fauna.

Prior to the application of ESR dating to fossil mammalian teeth, Chinese Palaeolithic sites beyond the range of radiocarbon dating (~40 ka) were dated using the U-series method on teeth, bone and speleothems, or by palaeomagnetic methods. U-series dating of the dental tissues has been integrated with ESR dating techniques to help resolve the modes by which uranium entered the teeth during burial and to decipher the coupled ESR–U-series age [7,8,19]. This is the approach used at Panxian Dadong. Previous ESR results from teeth collected during the 1998–99 excavations concur with the suggested late Middle Pleistocene age based on relative methods. Early uptake (EU) model ages range from 49–276 ka and linear uptake (LU) model ages range from 55–343 ka [20]. During the 2000 field season HLJ and WJR collected teeth and sediment for further ESR dating, U-series dating, and dosimetry refinement. The results are reported here.

2. Site formation processes

The deposit that infilled the cave originates from the breakdown of cave limestone, or soil formed by the erosion of neighboring hills. The latter are rich in iron-pisolites that characterize tropical soils. The cave sediments are brown–yellow clays, red–brown sands, pebbles, breccias, and limestone blocks [16]. Several episodes of calcification (travertine deposition) throughout the stratigraphic sequence reflect laminar seepage of carbonate-saturated water flowing down the cave walls and accreting laterally. The recurrence of travertine layers suggests a succession of climate fluctuations between warm/wet and cool/dry conditions [13]. Narrow, black, weathered travertine crusts in association with the appearance of thin gravel layers characterize the deposit and represent times of very slow deposition (Fig. 2). Most of the stratigraphic units can be easily followed throughout the entire excavated area. Moving from the north end of excavation Area B in Fig. 3, the stratigraphy of the deposit changes from well-cemented subangular pebbles to poorly sorted, matrix-supported gravel. Loose silty clays dominate the strata as one nears the southern edge of Area B.

3. Previous dating of the stratigraphic sequence

Previous absolute age estimates for Panxian Dadong include U-series ages from speleothems and fossil teeth. Three remnant capping speleothems that were attached to the northern cave wall averaged to 130 ka [25]. They

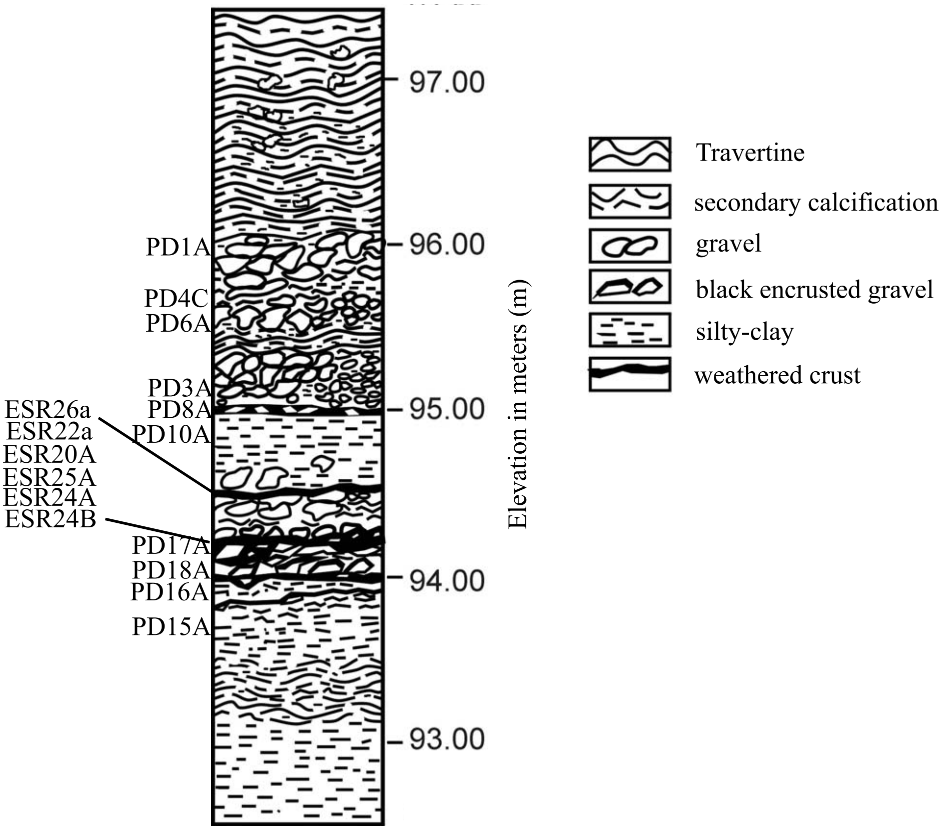

Fig. 2. Stratigraphic column of the northern profile in area B of Figure 2 with samples projected. Note the major hiatus characterized by weathered crusts and encrusted gravels, and the intermittent layers of travertine and secondary calcification (personal communication P. Karkanas). Modified from Rink et al. [20]

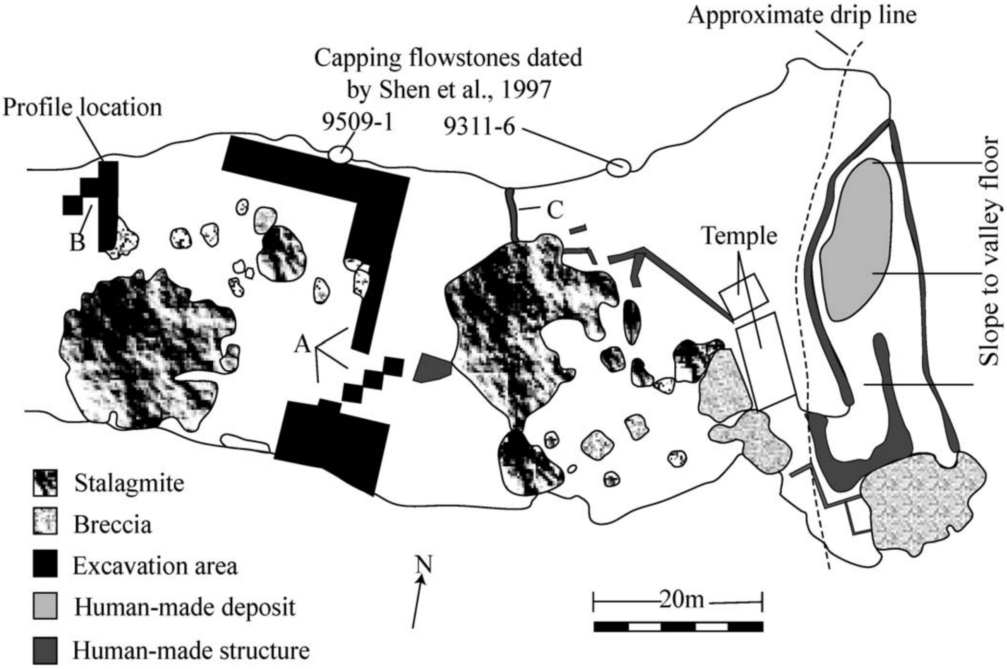

Fig. 3. Plan view of the main chamber. The area marked A denotes the earlier excavations, area B is the excavation where the ESR samples were taken, and C marks the entrance to the cave. The location of the profile in Figure 3 and the capping flowstones dated by Shen et al. [25] are also marked. Modified from Huang et al. [14].

suggested this age was a minimum estimate for the excavation areas. Huang et al. [14] published U-series results ranging from 68–16 ka for several mammalian teeth that were excavated during the 1993 season (Fig. 2, Area A). These younger dates are not consistent with the capstone speleothem dates or the age implied by the faunal assemblage. One possible explanation for these young U-series ages is that the teeth may have experienced non-early uranium uptake making the estimates minimum ages.

ESR ages were reported for 10 teeth excavated during the 1998–99 field season [20]. The samples' stratigraphic locations (z) are recorded as meters below the 100 m established datum. These teeth were divided into two groups: those higher than the 95.00 m level (e.g. 5 m below datum) and those retrieved below 94.20 m. The mean early uptake (EU) model and linear uptake (LU) model ages for the younger group (95.00 m and above) are 137 ± 16 and 156 ± 18 ka, respectively. For the teeth recovered from the lower segment of the excavation (below 94.20 m) the reported EU and LU mean model ages are 214 ± 24 and 262 ± 31 ka, respectively.

4. Samples and methodology

Teeth analyzed in this study include rhinoceros, bovid and cervid molars. Six of these teeth are from excavations above 95 m and four teeth were excavated from 94 m and below. The ten teeth are from squares E46, G46 and F46. Five teeth collected during the 2000 excavation lie between 94.265 and 94.407 m and are all within square I46, and therefore located stratigraphically between the samples from previous seasons. All teeth were collected with the attached sediment and surrounding 5 cm of sediment for beta dose rate approximations. The dated enamels from the 1998–99 excavations were from whole and fragmented teeth, while the samples from 2000 were whole teeth. All sampled teeth show excellent preservation of the dental tissues.

In the field, attached sediment was carefully removed, labeled and set aside for external dose rate analyses at McMaster Nuclear Reactor by neutron activation analyses (NAA). In the laboratory a piece of enamel (approximately 1 cm^2 and planar) with its attached dentine (1.5–2 mm thick) was removed from each tooth with a hand-held diamond wheel saw. The enamel and dentine were separated and the enamel was stripped of ~ 50 μm from both the outside (sediment side) and the inside (dentine side) to eliminate the enamel exposed to alpha radiation. Thickness of the dental tissues was determined with an electronic micrometer indicator with an uncertainty of ± 2 μm. The enamel from each sample was gently crushed with an agate mortar and pestle, and then sieved (<150 μm). The powdered enamel was divided into ten aliquots (10–60 mg each) and then irradiated by an artificial gamma radiation source (^{60}Co) at McMaster Nuclear Reactor hot cell in 10 uniformly increasing step doses from 0 to 175 Gray (Gy). ESR intensities were then measured for each aliquot on a Bruker EMX X-band spectrometer with the microwave power set at 2 mW, a field modulation of 100 kHz, and modulation amplitude of 5.00G. With a SSE (single saturating exponential) function, the ESR intensities were fitted to create a dose response curve using the VFIT software to ascertain the equivalent dose (D_E). The errors attached to the D_E were calculated within VFIT software with a $1/I^2$ weighting scheme of the data (see [1] for protocol). The dentine and enamel were analyzed for U, Th and K concentrations using neutron activation analysis (NAA) and delayed neutron activation analysis (DNAA). See Table 1 for analytical data.

Four dentine samples (PD6D1, PD15D1, ESR20A, ESR24A) that backed the corresponding dated enamels were selected and prepared for ^{230}Th/^{234}U dating and then analyzed using an inductively coupled plasma multicollector mass spectrometer (MC-ICP-MS, IsoProbe). The main advantages of MC-ICP-MS analyses over thermal ionization MS (TIMS) is the greatly reduced sample size, increased precision [10] and sampling resolution, and shorter run times.

The dentine samples were dissolved in 7.5 M HNO_3 with a spike and $FeCl_3$. 3 M NH_4OH was added repeatedly to the samples to form the precipitate and followed by centrifuging. To further reduce the presence of phosphate in the samples they were re-dissolved with 2 M HNO_3 until the sample reached a pH=7.0 and there was no white flocculate visible.

Gamma dose rate measurements were made in-situ within the excavation profile proximal to the excavated teeth using both a Harwell Gamma Scintillometer and thermoluminescence dosimeters (TLDs) of two types (Table 2). The TLDs used in 1998 contain calcium fluoride powder encased in a pure copper tube (1 mm thick) and were left in the profile for a year to record the annual dose (these TLDs were corrected for an internal dose rate of 100 μGy/a). In 1999, Panasonic TLDs, containing $CaSO_4$:Tl sealed in 1.3 mm thick pure copper tube, were inserted 30 cm horizontally into the profiles and left for one year. The TLDs record both the gamma and cosmic radiation; therefore, the gamma dose rates from TLDs have been corrected for the cosmic dose of 6 μGy/a. This small cosmic dose rate is due to the very thick cave roof (50 m), the small entrance to the cave, and the distant location of the samples from the entrance. In 2000 several dosimetry measurements were taken using a Harwell Gamma Scintillometer inserted into 30-cm deep horizontal holes drilled in the profiles for teeth excavated that same season.

The majority of "gamma holes" contained limestone pebbles and clay sediments, creating a lumpy site, as opposed to a smooth site [22]. However, these 30 cm deep horizontal gamma holes were constrained to the

Table 1
Analytical data for teeth from Panxian Dadong

Sample	Elevation in meters[a] (*z*)	Square[b]	Taxon	D_E^c (Gy)	U En[d] (ppm)	U Den[d] (ppm)	U Sed[d] (ppm)	Th Sed[e] (ppm)	K Sed[e] (wt%)	Enamel[f] thickness (μm)	Rem 1 (μm)[g]	Rem 2 (μm)[g]
1998												
PD1A	95.833	E46	Bovid	35.5(0.5)	0.28	13.47	2.00	6.30	0.36	1330 ± 61	58 (29)	48 (24)
PD4C	95.473	F46	Bovid	48.6(2.0)	<0.1	7.96	2.10	5.20	0.65	1525 ± 51	42 (21)	59 (30)
PD6A	95.409	F46	Bovid	73.1(3.1)	<0.1	11.42	3.30	8.00	0.69	1627 ± 85	55 (27)	77 (38)
PD3A	95.188	E46	Rhinoceros	66.7(3.4)	0.16	17.57	1.40	4.40	0.25	1271 ± 53	62 (31)	46 (23)
PD8A	95.111	F46	Rhinoceros	39.0(2.0)	<0.1	6.46	1.50	4.10	0.36	3258 ± 108	92 (46)	61 (32)
PD10A	94.974	G46	Rhinoceros	54.8(1.2)	<0.1	4.30	1.40	5.30	0.31	2012 ± 273	96 (48)	58 (29)
2000												
ESR 26A	94.407	I46	Rhinoceros	64.1(4.2)	0.13	6.15	1.43	6.23	0.60	1885 ± 90	105 (52)	62 (31)
ESR 22A	94.396	I46	Bovid	78.5(6.9)	0.31	9.03	1.62	8.14	0.57	1166 ± 137	90 (45)	108 (54)
ESR 20A	94.371	I46	Cervid	70.7(4.4)	<0.1	7.6	1.63	5.99	0.52	1027 ± 166	111 (55)	71 (36)
ESR 25A	94.314	I46	Bovid	75.5(4.3)	<0.1	9.44	0.85	5.34	0.33	1445 ± 113	91 (46)	76 (38)
ESR 24A	94.265	I46	Cervid	97.3(5.5)	<0.1	11.57	1.12	4.43	0.36	1041 ± 93	73 (37)	94 (47)
ESR 24B	94.265	I46	Cervid	101.1(6.5)	<0.1	6.59	1.12	4.43	0.36	1085 ± 170	98 (47)	86 (43)
1999												
PD17A	94.156	F46	Rhinoceros	82.0(1.4)	<0.1	10.43	1.00	3.80	0.26	1634 ± 121	70 (35)	40 (20)
PD18A	94.035	F46	Rhinoceros	75.8(1.2)	<0.1	14.21	1.00	3.80	0.26	1389 ± 149	79 (40)	64 (32)
PD16A	93.939	F46	Rhinoceros	53.5(0.7)	<0.1	5.14	0.90	3.40	0.19	2457 ± 111	61 (30)	69 (35)
PD15A	93.798	F46	Rhinoceros	78.7(1.6)	<0.1	11.50	1.00	3.80	0.26	1399 ± 168	39 (20)	32 (16)

[a] Elevation is measured below a 100 m established datum.

[b] Squares are 2 × 2 m units.

[c] Equivalent dose (D_E) in Grays (Gy).

[d] Uranium in enamel (En), dentine (Den) and sediment (Sed) determined by delayed neutron counting with a detection limit of 0.1 ppm.

[e] Thorium (Th) and potassium (K) determined by neutron activation analysis. Uncertainty levels were insignificant to calculations of annual doses.

[f] Each sample measured in 40–100 positions using a micrometer indicator with a precision of ± 2 μm; the reported uncertainties are $\pm 1\sigma$.

[g] Amount of enamel stripped from the external side (Rem 1) and the internal side (Rem 2) to remove damage due to alpha radiation. The uncertainty was estimated to be about 50% of the removed portion.

Table 2
In-situ dosimetry locations at Panxian Dadong

Designation	Year	Type of dosimeter	*X* (m)	*Y* (m)	*Z* (m)	Gamma dose rate (μGy/a)[a]	Corresponding tooth
Gam 1	1998	Harwell Spectrometer	555.522	518.122	95.439	252 (50)	PD4, PD6
Gam 2	1998	Harwell Spectrometer	555.494	519.168	95.102	219 (44)	PD8, PD3
D15	1998	CaF_2 TLD	555.747	521.329	95.904	420 (36)	
D16	1998	CaF_2 TLD	555.734	521.206	95.921	355 (27)	
D17	1998	CaF_2 TLD	555.705	521.012	95.871	207 (21)	
Mean of D15,16,17						327 (109)	PD1
Gam 4	1999	Harwell Spectrometer	552.576	519.153	93.765	179 (36)	PD 16
Gam 5	1999	Harwell Spectrometer	555.262	518.883	93.788	180 (36)	PD 15,17,18
Gam 6	1999	Harwell Spectrometer	555.297	517.076	94.906	313 (63)	PD 10
Gam 11	2000	Harwell Spectrometer	554.106	511.665	94.559	166 (33)	ESR 22
Gam 12	2000	Harwell Spectrometer	552.719	513.547	94.400	177 (35)	ESR 20,25,24
Gam 14	2000	Harwell Spectrometer	555.300	513.415	94.472	242 (48)	ESR 26

[a] microGrays per year.

stratigraphic levels from where the teeth were excavated, thereby creating dose rates that can be applied with confidence to the individual samples, assuming lateral continuity. Bulk sediment samples from the "gamma holes" were collected to determine the moisture content of the sediments (% of dry sediment). The results ranged from 10–15% moisture, therefore, a value of 10 ± 10 has been assumed as a reasonable value for the entire burial history of the samples. The 1998–99 ESR ages were calculated using ROSY Ver. 1.41 dating software and the conventional 2000 ESR ages were calculated using ROSY Ver. 2 ESR dating software created by K.-L. Kao and D. Mitchell. This software is identical to ROSY Ver. 1.41, with the exception that it has been modified from a FORTRAN/DOS operating system to a Windows-based interface [16]. Coupled ESR/U-series ages were calculated for four dentine samples analyzed for U-series dates using a computer program developed by R. Grün [6]. Coupled ESR/U-series ages result from a single calculation that uses the combination of relevant isotopic ratios (Th and U) in conjunction with the equivalent dose and dose rate data. The age is then consistent with all of the dose rates and a particular uptake function, known as the *P*-value [7,8].

5. Results

5.1. ESR model ages

In this work, we again report the stratigraphic locations of earlier dated teeth so that a comprehensive picture of all of the dating work at Panxian Dadong can be assembled here. The ESR ages show a trend of increasing age stratigraphically downward through the excavated deposit (Table 5). All but three samples have EU and LU model ages that are statistically indistinguishable within the analytical uncertainties. This overlap is due to the relatively small contribution of doses derived from uranium uptake into the teeth, meaning that the majority of the total dose rate originates from external beta and gamma doses (Table 2). U-series analysis of four dentine samples validates the mode uranium uptake experienced by these samples during burial and provides information to appropriate a valid coupled ESR-^{230}Th/^{232}U age [7,8].

All of the sampled teeth and their locations have been divided into an upper and a lower group. The division boundary is a depositional hiatus occurring between 94–94.3 m. The hiatus, represented by a layer of encrusted gravel (∼30 cm thick), inclines slightly to the south and then quickly thins and becomes horizontal. The upper group (n=6) lies at or above the 95 m horizon and has a range of 118–159 ka EU model ages and 131–181 LU model ages, with the exclusion of PD1. The mean EU and LU ages for this group are 137 ± 15 and 156 ± 17 ka, respectively. PD1, the stratigraphically highest sample, has been excluded from the mean age calculation because it fell outside $\pm 2\sigma$ when it was included in the calculation for the population standard deviation of the mean (Table 5). Therefore, it is considered an outlier and has not been included with this group. The upper group of teeth (94.97–95.47 m) is derived from a section of relatively quick deposition, characterized by poorly sorted, sub-angular gravel with an intermittent layer of travertine (∼20 cm thick). The lower group of samples was excavated below the 94.4 m datum horizon and has EU model ages ranging from 158–296 ka and LU model ages ranging from 185–349 ka. The mean ESR model ages of this group are 211 ± 40 ka (EU) and 258 ± 47 ka (LU). These teeth are derived from an area characterized by intermittent black encrusted gravel, denoting periods of non-deposition to relatively slow depositional rates. Due to the non-depositional condition and the comparatively high uranium content associated with ESR 22A, it is possible that this age is an underestimate, and/or ESR 22A came

Table 3
Dose rate data for ESR dating of teeth at Panxian Dadong

Sample	Z m above datum	γ Dose rate (μGy/a)	β Sed dose rate (μGy/a)	EU α-En dose rate (μGy/a)	EU β-En dose rate (μGy/a)	EU β-Den dose rate (μGy/a)	EU[a] total dose rate (μGy/a)	LU α-En dose rate (μGy/a)	LU β-En dose rate (μGy/a)	LU β-Den dose rate (μGy/a)	LU[a] total dose rate (μGy/a)
PD1A	95.833	327	70	57	18	129	606	26	8	62	499
PD4C	95.473	252	84	0	0	76	412	0	0	35	371
PD6A	95.409	252	98	0	0	105	458	0	0	49	403
PD3A	95.188	219	50	45	13	205	539	21	6	99	402
PD8A	95.111	219	21	0	0	30	271	0	0	14	254
PD10A	94.974	313	33	0	0	33	386	0	0	15	368
ESR 26A	94.407	242	49	41	12	52	403	18	6	24	345
ESR 22A	94.396	166	93	97	26	109	497	45	12	52	374
ESR 20A	94.371	177	87	0	0	115	384	0	0	54	324
ESR 25A	94.314	177	42	0	0	106	332	0	0	50	276
ESR 24A	94.265	177	66	0	0	167	417	0	0	80	329
ESR 24B	94.265	177	60	0	0	99	342	0	0	47	290
PD17A	94.156	180	33	0	0	113	332	0	0	53	273
PD18A	94.035	180	39	0	0	164	389	0	0	78	303
PD16A	93.939	179	19	0	0	33	269	0	0	16	234
PD15A	93.798	180	42	0	0	140	367	0	0	66	294

[a] Total dose rates includes 6 μGy/a of cosmic dose (corresponding to the cave roof thickness of 50 m) plus very small contributions of alpha dose rates for those teeth with U (uranium) in enamel.

Table 4
Analytical U-series data for Panxian Dadong

Dentine sample[a]	^{238}U concentration ppm (±2σ error)	^{230}Th/^{234}U[b]	^{234}U/^{238}U[b]	^{230}Th/^{232}Th[b]	^{230}Th/^{234}U closed system age (ka) (±2σ error)
PD6D1	10.06(0.0024)	0.32	1.18	180	42.14(0.22)
ESR20A	7.37(0.0017)	0.58	1.25	372	91.85(0.40)
ESR24A	11.38(0.0027)	0.73	1.31	2468	132.31(0.29)
PD15D1	11.18(0.0029)	0.61	1.12	4877	99.76(0.40)

[a] Th/U analyses were performed on dentine tissues adjacent to ESR dated enamel by MC-ICP-MS at GEOTOP Laboratory, University of Quebec at Montreal.

[b] Activity ratios.

Table 5
ESR and U-series dating results for teeth from Panxian Dadong

Sample	EU ESR age [ka] (±ka) {±1*s*}	LU ESR age [ka] (±ka) {±1*s*}	Coupled ESR-^{230}Th/^{234}U age (ka)	*P*-value
Upper Group				
PD1A	59 (10)	71 (16)		
PD4C	118 (14)	131 (18)		
PD6A	160 (22)	182 (25)	$208^{+23}/_{-19}$	2.29±0.4
PD3A	124 (14)	166 (21)		
PD8A	144 (23)	154 (26)		
PD10A	142 (23)	149 (23)		
Mean[a]	137 {15}	156 {17}		
Lower Group				
ESR 26A	159 (26)	185 (31)		
ESR 22A	158 (20)	210 (28)		
ESR 20A	184 (30)	219 (34)	$231^{+32}/_{-26}$	0.06±0.2
ESR 25A[#]	228 (39)	274 (44)		
ESR 24A[#]	233 (34)	296 (42)	$294^{+35}/_{-30}$	−0.29±0.12
ESR 24B[#]	296 (54)	349 (58)		
PD17A	247 (29)	301 (42)		
PD18A	195 (21)	250 (32)		
PD16A	199 (26)	228 (17)		
PD15A	214 (24)	268 (36)	$296^{+31}/_{-24}$	0.32±0.17
Mean[b]	211 {40}	258 {47}		

Ages calculated assuming 10±10% moisture in surroundings throughout the burial period.

[a] Mean Age for upper grouping of samples, excluding PD1A. See text for details. Reported {± 1*s*} values are the population standard deviation of the mean.

[b] Mean Age for lower grouping of samples. Same {± 1*s*} as in [a].

from a different environment and therefore has been reworked.

5.2. Coupled ESR-^{230}Th/^{234}U ages

Four dentine samples (PD6D1, PD15D1, ESR20D1, and ESR 24D1) were analyzed MC-ICP mass spectrometry in order to refine the EU and LU uptake model ages. Uranium concentrations within the enamels were extremely low or negligible; therefore U-series dating of the enamel samples was not required. The results are given in Table 4. The ^{230}Th/^{232}Th ratios were high, demonstrating that detrital ^{232}Th is not a concern with the samples and eliminating the need for ^{230}Th age corrections. The errors on the U-series ages are very low, ranging from 0.2–0.5% of the calculated ages. The ^{238}U concentrations measured match the concentrations measured initially by delayed neutron activation analysis (DNAA) ensuring accurate and comparable results.

The *P*-values listed in Table 5 were calculated using the computer program created by Grün [6] and discussed in the methodology section. The values show that different uranium uptake processes have been present at Panxian Dadong throughout the samples' burial history. For example, PD6A is stratigraphically the highest sample and has a *P*-value that is considerably greater than one, 2.29±0.4. This indicates the sample either experienced recent uptake, or episodic uranium gains

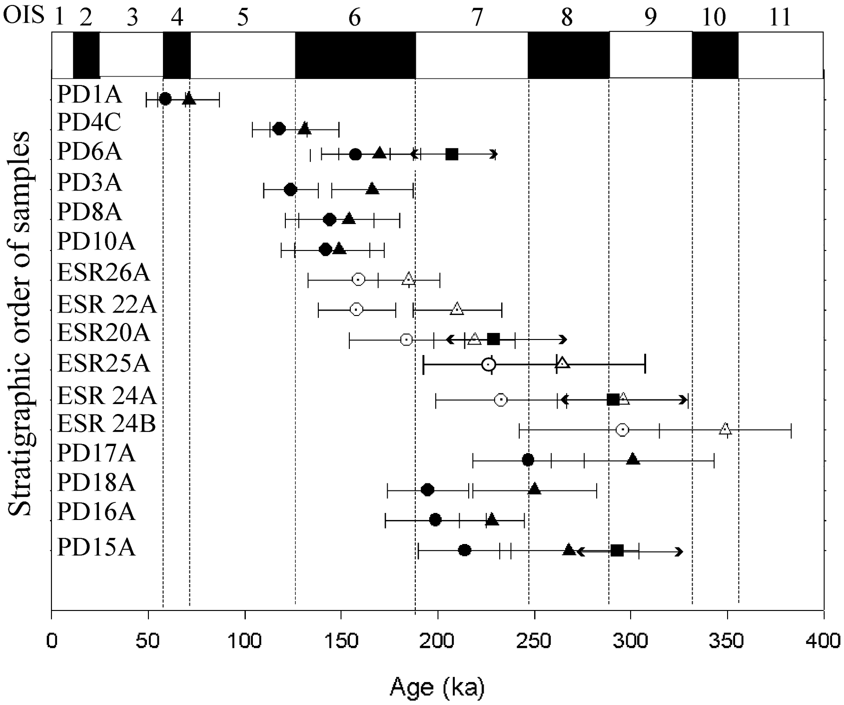

Fig. 4. ESR ages with errors shown in relative stratigraphic location, highest samples at the top. The vertical axis is not scaled. ESR24A and ESR24B come from a single tooth. Closed circles and triangles represent the EU and LU model ages, respectively, from the 1998–99 excavations. Open circles and triangles represent the EU and LU model ages, respectively, from the 2000 season. Closed squares are the coupled ages. Oxygen isotope states (OIS) boundaries are after Martinson et al. [17]. See text for discussion.

and losses. ESR20A experienced very close to linear uptake as suggested by a *P*-value close to zero (0.06). *P*-values between −1 and zero indicate uptake processes between linear and early uptake. Sample ESR24A experienced uptake within this range between linear and early uptake as determined from the *P*-value of −0.29. Finally, PD15A has a *P*-value of 0.32, suggesting a slightly slower than linear uptake. The result of the *P*-values calculated for four dentine samples suggests that a variety of uptake scenarios have occurred throughout the deposit at Panxian Dadong and greatly helps to refine the conventional ESR model ages.

6. Discussion

The high-resolution deep-sea chronostratigraphy of Martinson et al. [17] has been used to correlate Panxian Dadong ESR samples to oxygen isotope stages with the assumption that the continental climate in southwestern China was similar to that recorded in the deep-sea sediments. The range of sample ages span from OIS 10 to OIS 5, as defined by Martinson et al. [17] (Fig. 4). The ESR results reported here extend the absolute age range of the deposits within Panxian Dadong well into the Middle Pleistocene. All teeth are stratigraphically lower than the capping speleothem dated at ~130 ka by Shen et al. [25]. However, this age should be used with caution because the speleothem samples are located approximately 15–20 m to the east of the ESR dating samples (Fig. 3) and the geological association between the speleothems and the sampling area is unclear. The LU mean model age (258 ± 47 ka) has been assumed as a minimum age for the lower group, and this corresponds to the end of OIS 8 (glacial). The EU and LU mean model ages of the upper group indicate deposition during OIS 6 (glacial). However, the U-series analysis suggests that the ages of these teeth are older, and therefore, the mean age, 156 ± 17 (LU), should be taken as a minimum age. Most likely these teeth were buried towards the end of OIS 7.

The ages for the deeply stratified archaeological sequence at Panxian Dadong represent a period that is not well represented by other chronometrically dated sites in southwestern and southern China. The ages for the lower group are significant because a human tooth was excavated within Square I46, near the mammalian dating samples of 2000. The mammalian tooth sample ESR24A/B lies 0.06 m above and less than 0.5 m north of the human tooth and ESR24A's coupled ESR-^{230}Th/^{234}U age is $294^{+35}/_{-30}$ ka. Due to the extremely close spatial relationship between the ESR dating sample and the excavated human tooth we can conclude $294^{+35}/_{-30}$ ka is an excellent correlative age and is the oldest evidence for the genus *Homo* in Guizhou province.

6.1. Comparison with data from other sites

The ESR dates for Panxian Dadong enable us to place the site within a broader regional and temporal framework. Other southwestern and southern Chinese Pleistocene sites with chronometric dates that can be compared with Panxian Dadong are listed in Table 6 and their locations are indicated in Fig. 1. Yuan et al. [33] and Chen and Yuan [3] reported U-series ages on bone and/or dental tissues from some of these sites. Only ages that show $^{230}Th/^{234}U$ and $^{231}Pa/^{235}U$ ages in agreement (within error), and are therefore closed-systems, will be discussed.

The chronologically oldest sites in the region are Yuanmou in Yunnan province and the Bose Basin sites to the east in Guangxi. These sites provide evidence that hominids were occupying open-air localities in southwestern and southern China in the Early Pleistocene. The Yuanmou early *Homo erectus* locality is situated in a large sedimentary basin with nearly 700-m thick deposits of fluvio-lacustrine strata [35]. Several localities have been investigated, although most of the attention has focused on the hominid-yielding site. Huang and Grün [12] report ESR model ages ranging from 670 ka (EU) to 1.6 ma (LU) from fossil tooth enamel. More recently Hyodo et al. [15] reported palaeomagnetic data suggesting that the Yuanmou hominid-bearing level should be assigned to the early Bruhnes chron (approximately 0.7 ma). The Bose Basin has $^{40}Ar/^{39}Ar$ ages from three in-situ tektites (Australasian) from archaeological localities that give an overall weighted mean isochron age of 803 ± 3ka [11]. The Bose archaeological materials are known from several terraces of the Youjiang River. The settings of both the Bose and Yuanmou localities suggest that riverine environments provided important resources as well as pathways for migration for early hominids.

After these late Lower/early Middle Pleistocene localities, there is a substantial gap in the archaeological/dating record for the region—until the later Middle Pleistocene when hominid activity is documented in Guizhou province at Panxian Dadong and the roughly contemporaneous site of Guanyindong. Located only 180 km to the northeast of Dadong, Guanyindong is another cave site yielding lithics associated with an *Ailuropoda–Stegodon* fauna. Chronometric dates were reported by Yuan et al. [33], Shen and Jin [24] and Shen [23]. According to Yuan et al. [33], the U-series age from bones and teeth is approximately 115–157 ka. Most recently, Shen [23] provided U-series dates for speleothems from Guanyindong. He suggests that the upper portions of the sequence are younger than 40 ka, while the deeper deposits range from 50–240 (and may be further constrained by stratigraphic analysis to 100–190 ka). As Shen [23] points out, these results are not entirely consistent with the faunal data. For example, there is one *Gomphotherium* tooth that, according to these U-series estimates, would be the latest known example of the taxon. Panxian Dadong and Guanyindong document two notable changes in hominid use of the landscape during the late Middle Pleistocene and continuing through the Late Pleistocene: the exploitation of upland, karstic environments and the use of caves.

Tongzi (Yanhuidong) is another Guizhou cave with hominid teeth, a faunal assemblage similar to that of Panxian Dadong but including taxa associated with younger ages (such as *Cuon javanicus* and *Crocuta ultima*), and a few stone artifacts. Yuan et al. [33] report ages of $181^{+11}/_{-9}$ to 113 ± 11 ka for tooth and bone samples. These dates suggest that portions of the Tongzi sequence are contemporary with the upper group of dates from Panxian Dadong, falling within OIS 6 and 5. Another set of age estimates ranging between 172–192 ka and 241 ka were determined by Wang [26], and most recently Shen and Jin [24] suggested that the archaeological and hominid materials are from sediments sandwiched between speleothems dating to 206 and 240 ka. While these studies suggest that sediments from Tongzi are older than 200 ka, the age of the hominid materials remains uncertain.

At the Maba cave in Guangdong province, a *H. sapiens* calvarium has been dated to the early Late Pleistocene based on U-series dates of $129^{+11}/_{-10}$ ka [33]. The associated *Ailuropoda–Stegodon* fauna is characterized by Upper Pleistocene taxa [9]. Therefore, Maba can be correlated with the upper deposits of Panxian Dadong and the end of OIS 5.

The most recent sites with chronometric dates are the Liujiang (Guangxi) and Shuicheng (Guizhou) caves. These have been assigned to the Late Pleistocene with U-series ages of $>67^{+6}/_{-5}$ and 52 ± 3 ka, respectively [33]. A complete cranium and other hominid remains designated as *H. sapiens* have been recovered within deposits at Liujiang [28], while hominid teeth were found at Shuicheng. Both of these sites postdate the deposits at Panxian Dadong, falling within OIS 3.

7. Conclusions

The ESR data in this study constrain the age of the sampled portion of the deeply stratified sequence at Panxian Dadong to the time frame between 120 and 300 ka. In most cases, the LU model ages on tooth enamel have proven to be the minimum ages with the use of coupled ESR/U-series ages on adjacent dentine of four samples. These age determinations make Panxian Dadong the oldest dated human occupation in Guizhou province. Panxian Dadong has faunal material, human remains and lithics within a well-characterized and well-dated geological sequence whose record extends from

Table 6
Regional archaeological sites comparable with Panxian Dadong

Site	Province	Environment	Oxygen isotope stage	Archaeological/palaeoanthropological materials	Date	Method
Yuanmou	Yunnan	Open air, fluvio-lacustrine basin	17	Hominids, lithics, fauna	670 ka EU, 1.6 ma LU, 0.7 ma	ESR (conventional), palaeomagnetic
Bose	Guangxi	Open air, fluvial basin	20	Lithics	803 ka	Ar–Ar on tektites
Panxian Dadong	Guizhou	Cave, upland	6–8	Hominids, lithics, fauna	156 LU-258 LU (min. mean ages)	ESR (conventional and coupled U-series)
Guanyindong	Guizhou	Cave, upland	3–7	Lithics, fauna	57 ka, 240 ka	U-series
Tongzi	Guizhou	Cave, upland	5–6	Hominids, lithics, fauna	113 ka, 181 ka	U-series
Maba	Guangdong	Cave, upland	5	Hominid, fauna	129 ka	U-series
Liujiang	Guangxi	Cave, lowland	3	Hominids, fauna	>67 ka	U-series
Shuicheng	Guizhou	Cave, upland	3	Hominids, lithics, fauna	52 ka	U-series

the Middle Pleistocene to the Late Pleistocene (OIS 8 to OIS 2).

Acknowledgements

We thank the Natural Sciences and Engineering Research Council of Canada and the Social Sciences and Humanities Research Council of Canada for financial support to WJR and HLJ. We thank Nikki Robinson for help with sample preparation, and Panagiotis (Takis) Karkanas (Ephorate of Palaeoanthropology-Speleology, Athens) for helpful discussions and contributions toward understanding the geological sequence at Dadong.

Major support for the Panxian Dadong excavations and analyses was provided by the National Science Foundation (#9727688); The Henry Luce, Wenner-Gren and Leakey Foundations; and the University of Cincinnati Taft Fund.

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