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Research paper

Preliminary results of combined ESR/U-series dating of fossil teeth from Longgupo cave, China

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ABSTRACT

Longgupo Cave site, located in Wushan County, Chongqing, China has attracted continuous attention since its discovery of hominid remains in association with late Pliocene-early Pleistocene fauna and numerous lithic artefacts. In 2003–2006, new excavation was carried out on this site, allowing the description of a detailed stratigraphy of the highly complex cave infillings and the sampling of teeth for combined ESR/U-series analyses. Here we report preliminary dating results of seven herbivorous fossil teeth from different archaeological layers of the lowest geological unit (C III). Uranium-series analyses indicate that no obvious uranium leaching has occurred and all the teeth (except one) underwent a very recent uranium uptake history. The obtained US-ESR results show that the age of six teeth are basically consistent, between ~1.4 and 1.8 Ma. At the same time, we observed an inverse correlation of two samples with the stratigraphical sequence. This could be caused by the distinct uranium uptake history of one sample, high uranium content in the enamel for another or bad estimation of external dose rate. Due to the complexity of the stratigraphic sequence, supplementary in situ gamma dose rate measurement should be performed for all the samples during the following excavations in order to confirm this preliminary ESR/U-series chronology.

1. Introduction

Longgupo cave (30°51′47″N, 109°39′56″E), also called "Wushan hominin site", is located in Wushan County, 20 km away from the south of the Yangtze River near the eastern border of Chongqing, China (Fig. 1). It was discovered in 1984 and subsequently excavated in 1985–1988 by the Institute of Vertebrate Paleontology and Paleoanthropology (IVPP) of Beijing and the Chongqing Museum of Natural History (Huang and Fang, 1991). The site attracted more attention since the discovery of a fragment of mandible with two teeth and an upper incisor. Huang and Fang (1991) initially assigned the fossils as a new subspecies of *Homo erectus* (*Homo erectus wushanensis*), then Huang et al. (1995) claimed that the fossils show affinities with *Homo habilis* and *Homo ergaster* remains. Most recent debate of Longgupo fossils focused on whether the remains belong to ape or human (Schwartz and Tattersall, 1996; Wu, 2000; Elter et al., 2001; Elter, 2009; Ciochon, 2009). However, Longgupo cave is nevertheless a crucial archaeological site because of the undoubted evidence of 16 *Gigantopithecus* teeth and numerous stone artifacts (Huang et al., 1995; Boëda et al., 2011a), which could provide the first indication of non-hominin tool-using ape in East Asia (Dennell, 2009).

New excavations of Longgupo site were conducted in 2003 and 2006 by a Chinese–French team (Boëda et al., 2011a), providing detailed information about the site formation process and the stratigraphical sequence (Boëda et al., 2011b; Rasse et al., 2011). In order to complete the chronological framework of the site, fossil

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Fig. 1. Location of Longgupo site, China (black triangle indicates other early hominid sites in China).

teeth were sampled for ESR/U-series study during the new excavation. In this paper, we present preliminary results obtained on seven samples.

2. Geological and paleontological background

Longgupo site is located on the Triassic limestone hill slope that was filled with Plio-Pleistocene deposits from Miaoyu River, and then covered with breccias. The sequence was initially subdivided into twenty horizontal 1 m-thick levels (Huang et al., 1995), but Rasse et al. (2011) demonstrated a more complex stratigraphy (Fig. 2). The cave infilling began by alluvial deposits: black clays on the base C III 10, which altimetrically corresponds to levels 20–13 described by Huang et al. (1995), then coarse fluvial sediments during the deposition of archaeological complexes C III (south wall)/III' (north wall) (levels 12–6) (Fig. 2), and complex II (levels 5–2) until the complete fill of the cave. During the middle and late Pleistocene the site was carpeted by the collapsed coarse breccias along the slope.

The site was then disturbed by gravity deformations: the alluvial sediments, rendered plastic by the high proportion of clayey beds, were deformed following the slope and by karstic collapse along the north and south limestone walls of the site (Rasse et al., 2011).

The faunal assemblage of Longgupo site contains specimens from 68 genera, including microfauna (Huang and Fang, 1991). The occurrence of *Gigantopithecus blacki* makes it possible to compare with the *Gigantopithecus* fauna of South China. Other taxa, such as *Procynocephalus*, *Macaca*, *Sinomastodon*, *Equus yunnanensis*, *Ailuropoda microta*, and *Nestoritherium*, were also found in Liucheng *Gigantopithecus* Cave, an early Pleistocene site of Guangxi Province (Wu and Olsen, 1985), which indicates the two sites have more or less the same age. The presence of the rodent *Mimomys peii* suggest also a time range of late Pliocene to early Pleistocene (Huang and Fang, 1991).

During the excavation in 2003–2006, 854 artifacts were unearthed at Longgupo, including worked cobbles, unipolar flakes, bipolar objects, knapping fragments and hammerstones (Boëda and Hou, 2011). 90% of the raw material was local Triassic limestone, and the other 10% were exogenous materials. The hardness and presence of natural fracture planes in the Triassic limestone are the main reasons of different operational processes and high number of knapping accidents. The archaic age with such uncommon raw materials makes Longgupo industry difficult to compare with other Chinese sites, and the unique technological option indicates it follows a separate line of development with contemporaneous African sites (Boëda and Hou, 2011).

3. Previous geochronological studies

Liu et al. (1988) carried out the first geochronological study of Longgupo sequence using paleomagnetism method, and concluded that the archaeological layers were deposited within a normal polarity period, that they proposed to correlate with Reunion subchron, dated to 2.15–2.13 Ma by Baksi and Hoffman (2000). In fact, several normal magnetozones were identified in the sequence (corresponding to levels 2–3, 7–8, 13 and 20 respectively), but it is rather difficult to replace these zones into the new stratigraphical sequence published by Rasse et al. (2011). Boëda et al. (2011b) emitted some doubts on these paleomagnetic results, according to the highly complex cave fillings that they judged ill-suited for paleomagnetic analysis. The previously observed magnetozones could in fact correspond to the duplication of the same magnetozone along the slope or by faulting. Zhu et al. (2003) also mentioned that the natural remanent magnetization (NRM) intensity values for the Longgupo cave sediments were too weak to be measured accurately.

ESR method was used by Huang et al. (1995) to determine the age of fossil bearing levels. A bovid tooth from level 4 was then dated at 1.02 $\pm$ 0.12 Ma using linear uptake (LU) model (i.e. assuming that uranium was absorbed continuously by the sample). Huang et al. (1995) assigned therefore the magnetically normal levels 2–3 to the Jaramillo subchron, and the magnetically normal

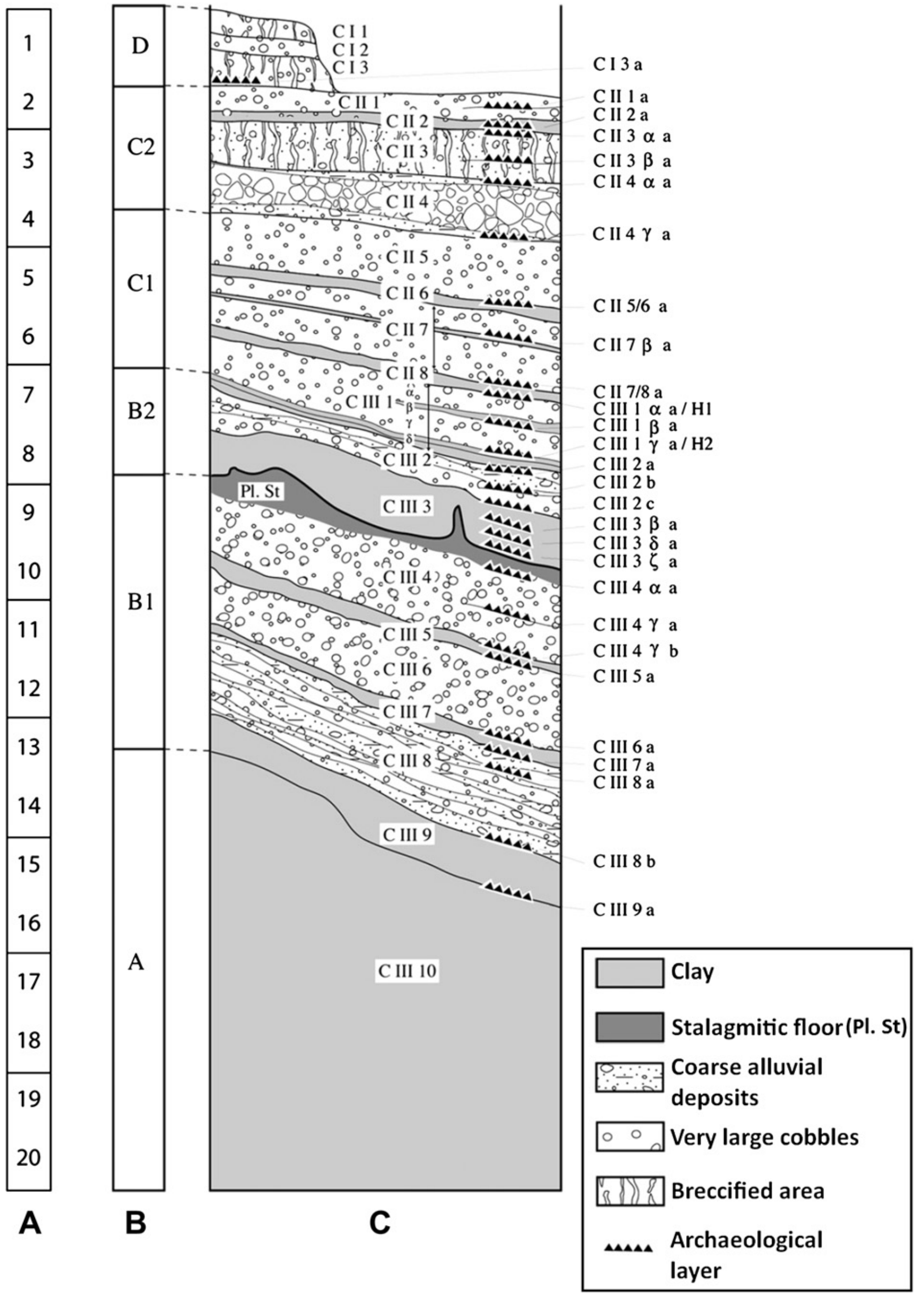

Fig. 2. Stratigraphy of Longgupo site based on the Chinese-French excavation during 2003 and 2006 (A-stratigraphic scale of Huang et al. (1995); B-stratigraphic division in 2003 and 2006; C-stratigraphic profile and archaeological layers; stratigraphic unit C III' was recovered from north wall and correspond to unit C III in south wall).

hominin-bearing levels 7–8 to the Olduvai subchron, and they concluded that Longgupo hominin remains should be as early as 1.90 Ma. These attributions seem to be doubtful nowadays regarding the difficulty of correlating the ancient sediment samples with the new stratigraphical sequence mentioned above.

Chen et al. (2001) also used ESR methods to analyze 3 mammalian teeth from levels 2, 4 and 5. Presuming an early uptake (EU) history (i.e. that uranium uptake took place in a very short time after the sample burial), they calculated the ages of three samples as 1.10 ± 0.23, 1.34 ± 0.24 and 1.28 ± 0.14 Ma, respectively. It should be noticed that Chen et al. (2001) used the same external dose rate to calculate the age of three teeth sampled from different archaeological layers. The sampling realized during 2003–2006 excavation gave us a great opportunity for detailed chronological study of Longgupo site and to compare the new dating results with previous ones despite some discrepancies between the two stratigraphical sequences.

4. Materials and methods

In the present work, combined ESR/U-series method was used in order to model U-uptake history of the fossil tooth. Grün et al. (1988) proposed a function introducing an uptake parameter p (US model): $U(t) = U_0\ (t/T)^{p+1}$, where $U(t)$ is the uranium concentration at the time t, U_0 is the present day uranium concentration, and T is the age of the sample. A p-value of -1 corresponds to EU case, $p = 0$ to LU one and $p > 0$ to indicate a recent uptake of uranium. The US model allows the calculation of a p parameter for each dental tissue, but its application is restricted to p values higher than -1, i.e. uranium loss from the dental tissues cannot be modeled (Grün, 2009a). The model has already shown its potential of dating early hominid sites, at Swartkrans in South Africa (Curnoe et al., 2001) or Atapuerca Gran Dolina in Spain (Falguères et al., 1999), but the limits were clearly demonstrated in Early Palaeolithic sites of Nihewan Basin, China (Han, 2011) and Orce Basin, Spain (Duval et al., 2011a).

The analyzed seven herbivorous teeth were sampled in different layers of Longgupo site from both south (LGP06S02, LGP06S04, LGP06S05) and north areas (LGP06N02, LGP06N05, LGP06N06, LGP06N09). All the teeth present only enamel and dentine dental tissues, hence "sediment-enamel-dentine" geometry was considered for age calculation. The two dental tissues were separated mechanically with a dental drill, outer enamel surface was cleaned to remove the effects of external alpha radiation, then grinded, sieved (100–200 μm) and split into 10 aliquots for gamma irradiation from 125 to 32,000 Gy with a calibrated ^{60}Co GammaCell 220 research irradiator. U-series analyses were performed on each dental tissue by α-spectrometry, following the standard procedure of Bischoff et al. (1988). Radon loss was assessed from combined γ and α-spectrometry measurements (Bahain et al., 1992).

The ESR measurements were carried out on a Bruker EMX ESR spectrometer (X band, 9.82 GHz) at room temperature with the following acquisition parameters: 1 mW microwave power, 1024 points resolution, 100 kHz modulation frequency, 0.1 mT modulation amplitude, 20 ms conversion time and 5 ms time constant. Each sample was measured 4 times in order to check the reproducibility of the data.

The ESR intensity was extracted from peak-to-peak amplitudes (T1-B2) of the ESR signal of enamel (Grün, 2000). In the present study, the equivalent doses (D_E) were determined by fitting the experimental data points with a double saturation exponential (DSE) function using Origin 8.0 software, with weighting by $1/I^2$ (Fig. 3). For early Pleistocene samples, this function often better describes the analytical data points than conventional single saturation exponential (SSE) function (Duval et al., 2009; Han et al., 2011) and it could indicate the presence in tooth enamel of two distinct CO_2^- species with different thermal stabilities and saturation behaviors (Grün et al., 2008). In this study, the error of D_E values determined by DSE function ranges between 3% and 10%.

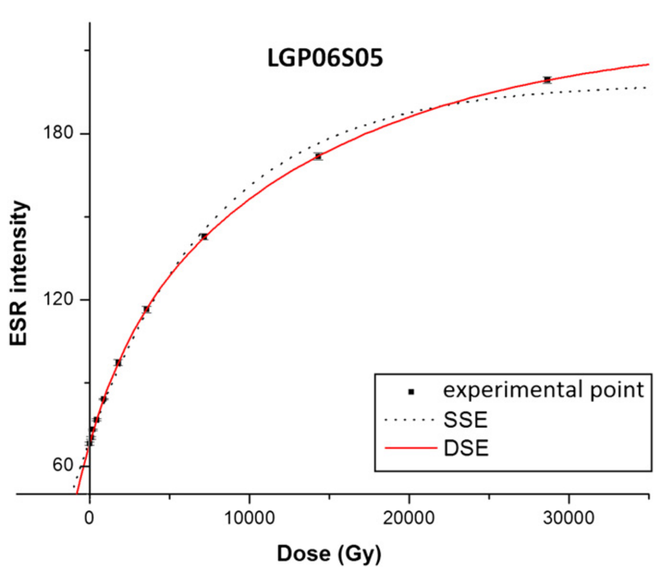

Fig. 3. Dose response curves of Longgupo LGP06S05 sample fitted with two functions (SSE: Single saturation exponential; DSE: Double saturation exponential).

As Longgupo is a typical "lumpy site" where sediments are composed by clays and pebbles mixture, the gamma dose rate measured in situ is advisable. However, in the present study, due to the accessibility during the excavation, in situ gamma dose rates measured by portable NaI gamma spectrometer in 2003–2006 are only available for three samples. Sediments associated with each tooth samples were also collected at the same time for dose rate determination in laboratory by gamma spectrometry using low background high purity Ge detector (Table S1).

The US-ESR ages of the samples were calculated using DATA software (Grün, 2009b) with the following parameters and assumptions: alpha efficiency of 0.13 ± 0.02 (Grün and Katzenberger-Apel, 1994); dose rate conversion factors of Adamiec and Aitken (1998); Monte-Carlo beta attenuation factors (Marsh et al., 2002) based on the thickness of the enamel layer, before and after its preparation; water contents of 3 wt% in the enamel, 7 wt% in the dentine and 10 wt% in sediment were assumed.

5. Results and discussion

The analyzed ^{230}Th/^{234}U activity ratios of Longgupo samples are shown in Table 1. The uranium concentration ranges from 0.32 to 6.73 ppm for enamel and from 21.08 to 105.48 ppm for dentine tissue. The ^{230}Th/^{234}U activity ratios of the samples are generally homogenous, ranging from 0.697 to 0.836, except for LGP06S04 for which ^{230}Th/^{234}U ratios are close to 1 in both two tissues. The apparent U-series ages of Longgupo dental tissues range from 122 ka to 171 ka, except LGP06S04, and indicate a very recent uranium uptake. These characteristics allow dating Longgupo teeth with US model (Han, 2011), while it is not so common for early Pleistocene sites, as recently illustrated by Grün et al. (2010) and Duval et al. (2012).

Table 2 shows US-ESR age results including the different dose rate components calculated with DATA program. For all the Longgupo teeth, the external gamma dose rate contributes more than 50% to the total dose rate. Table S1 shows the gamma dose rate values derived from in situ and laboratory measurements. The laboratory gamma dose rates differ greatly from the in situ values, especially for the sample LGP06S02 and LGP06N09 which were collected from layer C III H1 and the basement of layer C III'6 respectively, perhaps in relation with the numerous limestone fragments embedded in these layers. We have then preferentially used the in situ gamma dose rate measured in these layers for age calculation of the corresponding teeth.

The calculated p-values (Table 2 and Fig. 4) are all positive, ranging from 2.0 to 4.8, except for LGP06S04 dental tissues (p values close to -0.7). It confirms that most of the teeth have experienced a recent uranium uptake history which could have been induced by water stagnation during the humid periods of the middle and late Pleistocene, perhaps in relation with the upper breccia deposition (Boëda et al., 2011b). The sample LGP06S04 underwent a distinct uranium uptake history indicated by lowest p-values in relation with high ^{230}Th/^{234}U activity ratios (~1, see Table 1). For this tooth, the internal dose rate of enamel and beta dose rate of dentine are about two times higher and more than five times higher than in the other samples respectively. This is probably the main reason why the age result of LGP06S04 seems underestimated when compared with other samples. In addition, this recent age might also be partially linked with possible external dose rate over estimation, since the corresponding layer seems rich in fossil bones (field observation), and these remains are characterized by late uranium uptake (see U-series data obtained for

Table 1
U-series data analyzed by alpha and gamma spectrometers and removed thickness of enamel of Longgupo samples.

Sample no.	New stratigraphic layer	Level[a] (Huang et al., 1995)	Tissue	U-content (ppm)	^{234}U/^{238}U	^{230}Th/^{234}U	^{222}Rn/^{230}Th	U-series age (ka)	Enamel thickness (μm)	Enamel removed (dentine side/ sediment side) (μm)
LGP06S02	C III H1	6–7?	Enamel	3.02 ± 0.08	1.211 ± 0.035	0.697 ± 0.025	0.602	123.8 + 9.1/−8.3	1457	40/58
			Dentine	105.48 ± 1.71	1.149 ± 0.016	0.716 ± 0.026	0.336	131.8 + 9.9/−9.0		
LGP06S04	C III 3	8–9?	Enamel	3.08 ± 0.09	1.352 ± 0.040	0.992 ± 0.043	0.543	276.1 + 68.3/−42.4	1153	26/33
			Dentine	79.31 ± 2.00	1.419 ± 0.031	1.007 ± 0.033	0.282	282.2 + 48.9/−34.3		
LGP06N02	C III' 5	10–11?	Enamel	5.37 ± 0.10	1.315 ± 0.024	0.699 ± 0.024	0.745	122.3 + 8.0/−7.4	2010	90/68
			Dentine	40.57 ± 0.54	1.213 ± 0.015	0.769 ± 0.016	0.310	148.9 + 7.1/−6.6		
LGP06N05	C III' 6	11–12?	Enamel	2.19 ± 0.07	1.357 ± 0.037	0.836 ± 0.030	0.633	171.8 + 16.3/−14.1	1105	51/40
			Dentine	28.37 ± 0.61	1.296 ± 0.027	0.819 ± 0.027	0.333	166.4 + 13.9/−12.3		
LGP06N06	C III' 6	11–12?	Enamel	4.60 ± 0.09	1.273 ± 0.023	0.769 ± 0.022	0.663	147.1 + 9.4/−8.6	1391	37/114
			Dentine	21.08 ± 0.36	1.193 ± 0.019	0.745 ± 0.019	0.393	140.4 + 7.8/−7.2		
LGP06N09	C III' 6 base	11–12?	Enamel	0.32 ± 0.01	1.311 ± 0.043	0.716 ± 0.033	0.545	127.8 + 12.1/−10.8	2640	62/72
			Dentine	36.32 ± 0.55	1.181 ± 0.016	0.829 ± 0.017	0.270	176.8 + 10.2/−9.2		
LGP06S05	C III 7	12?	Enamel	6.73 ± 0.10	1.247 ± 0.018	0.777 ± 0.019	0.621	150.8 + 8.5/−7.9	1227	49/60
			Dentine	41.94 ± 0.59	1.112 ± 0.014	0.757 ± 0.019	0.328	148.2 + 8.7/−8.0		

[a] The archaeological levels with question marks indicate the uncertain relationship of the old and new stratigraphic division.

Table 2
D_E values, dose rate components and US-ESR ages with corresponding *p*-values of Longgupo samples.

Sample no.	Layer	Depth[a] (m)	D_E (Gy)	($\beta + \gamma$) sediment + cosmic (μGy/a)		Internal dose ($\alpha + \beta$) enamel (μGy/a)	β dentine (μGy/a)	Total dose rate (μGy/a)	*p*-enamel	*p*-dentine	US-ESR age (ka)
LGP06S02	C III H 1	6	2574.2 ± 125.7	1578 ± 150	In situ	147 ± 44	131 ± 36	1856 ± 152	4.43 ± 1.00	3.92 ± 0.93	1387 ± 160/140
LGP06S04	C III 3	8	3221.4 ± 129.5	1846 ± 172	In situ	1045 ± 1190	700 ± 550	3590 ± 1319	−0.67 ± 0.32	−0.70 ± 0.23	897 ± 107/237
LGP06N02	C III' 5	10	1931.9 ± 117.6	999 ± 120		303 ± 106	41 ± 11	1342 ± 141	4.66 ± 1.22	2.90 ± 0.69	1440 ± 200/182
LGP06N05	C III' 6	11	2216.7 ± 119.4	1054 ± 102		212 ± 81	65 ± 18	1330 ± 121	2.09 ± 0.84	2.34 ± 0.56	1667 ± 176/183
LGP06N06	C III' 6	11	2444.9 ± 226.5	982 ± 95		303 ± 74	28 ± 6	1312 ± 114	3.71 ± 0.46	4.21 ± 1.39	1863 ± 208/211
LGP06N09	C III' 6 base	12	1785.9 ± 45.9	1033 ± 100	In situ	15 ± 4	35 ± 11	1082 ± 87	4.80 ± 0.70	2.12 ± 0.52	1651 ± 166/133
LGP06S05	C III 7	12	2406.6 ± 126.9	1166 ± 84		510 ± 158	71 ± 17	1746 ± 173	2.56 ± 0.68	2.92 ± 0.65	1378 ± 156/150

[a] The burial depth of the samples were estimated based on the stratigraphic section in Boëda et al. (2011b) with uncertainty of ±2 m.

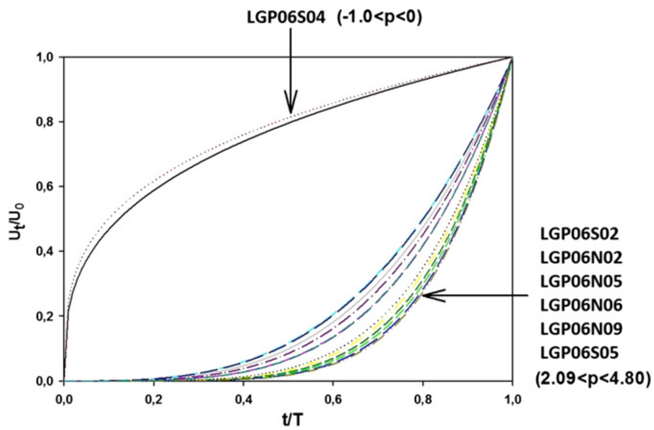

Fig. 4. *p*-values calculated for seven teeth samples in this study (the uppermost one shows a value between −1 and 0, while others range between 2.0 and 4.8).

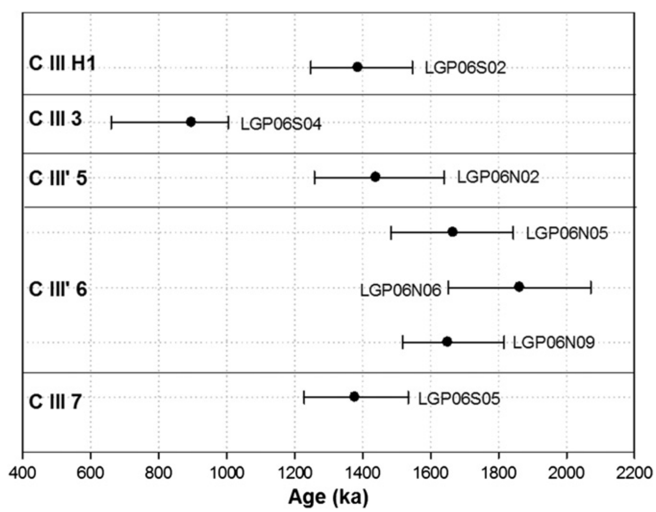

Fig. 5. Preliminary dating results of seven Longgupo tooth samples by combined ESR/U-series methods.

dental tissues in Table 1). The US-ESR age result obtained for LGP06S05 sample (layer C III'7) is slightly younger than the mean age obtained for three samples of the overlaying level C III'6. This age underestimation could be attributed to the high uranium content of the enamel (6.7 ppm) and an inverse correlation between alpha efficiency and U-concentration (Bahain et al., 1992; Duval et al., in press).

In this study, there is no apparent U-uptake evolution with burial depth of the samples, and the close apparent U-series ages indicate most of the samples undertake very recent uranium uptake maybe in relation with drastic changes of the hydrogeochemical conditions during middle and late Pleistocene.

According to our results, the ages obtained for six tooth samples are consistent with the stratigraphical sequence and allow the dating of C III and C III' units between 1.4 and 1.8 Ma (Fig. 5). This result seems younger than the previous age estimation of Liu et al. (1988) and Huang et al. (1995), and it is now possible to parallelize the deposition of the dated archaeological layers with Matuyama Chron posterior to Olduvai subchron end and prior to Jaramillo one.

6. Conclusion

Seven teeth from Longgupo early Pleistocene site, China, were studied by combined ESR/U-series method. The analyzed tooth samples do not present apparent uranium leaching, allowing the application of US model and the young apparent U-series ages indicate a recent uranium uptake history of the dental tissues. The calculated US-ESR ages of the samples are therefore older than the conventional ESR ages bracketed by EU and LU models, previously used as references for the chronology of Longgupo site (Huang et al., 1995; Chen et al., 2001). This study restates also the incorrectness of the viewpoint that ESR age of a teeth sample always lies between EU and LU model ages. As the *p*-values of dental tissues differ from each sample, U-series analysis is obviously essential for ESR dating of tooth in order to reconstruct the uranium uptake history in the dental tissues.

The combined ESR/U-series dating work in this study permits to propose a preliminary age for Longgupo site. Six of the seven analyzed teeth collected from C III–C III' unit present an age comprised between 1.4 and 1.8 Ma. This result confirms the antiquity of Longgupo site which could have an impact on the chronology of hominid presence in southern China.

In order to complete and refine the preliminary US-ESR age results of Longgupo site, future works need to be focused specifically on the following points. Firstly, supplementary in situ gamma dose rate measurement should be performed for all the samples during the next excavation in 2011–2015, in order to improve the existing chronology of the site. Then, additional U-series analyses using mass spectrometry techniques could help us to get a better precision on the U-series data, and thus on the final US-ESR ages. In addition, laser ablation ICP-MS U-series analysis could provide more information about the isotopic spatial distribution of U and Th in the dental tissues (Grün, 2009a; Duval et al., 2011b). Lastly, supplementary samples from the upper layers of the sequence (C I and C II units) need to be collected in order to better constrain the chronology of the whole sedimentary sequence. The forthcoming geochronological results of Longgupo site will help us better understand the human migration and settlement in East Asia and particularly in China.

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Appendix A. Supplementary material

Supplementary material associated with this article can be found in the online version, at doi:10.1016/j.quageo.2012.03.006.

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