



## TT-OSL dating of Longyadong Middle Paleolithic site and paleoenvironmental implications for hominin occupation in Luonan Basin (central China)

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### ABSTRACT

Dating middle Pleistocene hominin occupations alongside the reconstruction of paleoenvironments in China between 700 and 100 ka has always been a challenging task. In this paper, we report thermally transferred optically stimulated luminescence (TT-OSL) dating results for a Middle Paleolithic site in the Luonan Basin, central China, which we have named Longyadong Cave. The results suggest that the age of cave infilling and the deposition of sediments outside the cave range between  $389 \pm 18$  and  $274 \pm 14$  ka. These deposits are stratigraphically and geochronologically correlated with the L4 loess and S3 paleosol units of the typical loess–paleosol sequence of the Chinese Loess Plateau, and with Marine Isotope Stages (MIS) 10 to 9, respectively. On the basis of these new ages and the available paleoenvironmental data, it is suggested that the Longyadong hominins might have occupied the site both in glacial and interglacial periods, demonstrating that they coped well with environmental change in this mountainous region in warm/wet and cold/dry climates. The study further implies that the hominins abandoned the Longyadong Cave between  $274 \pm 14$  and  $205 \pm 19$  ka, when it was sealed by alluvial and slope deposits.

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### Introduction

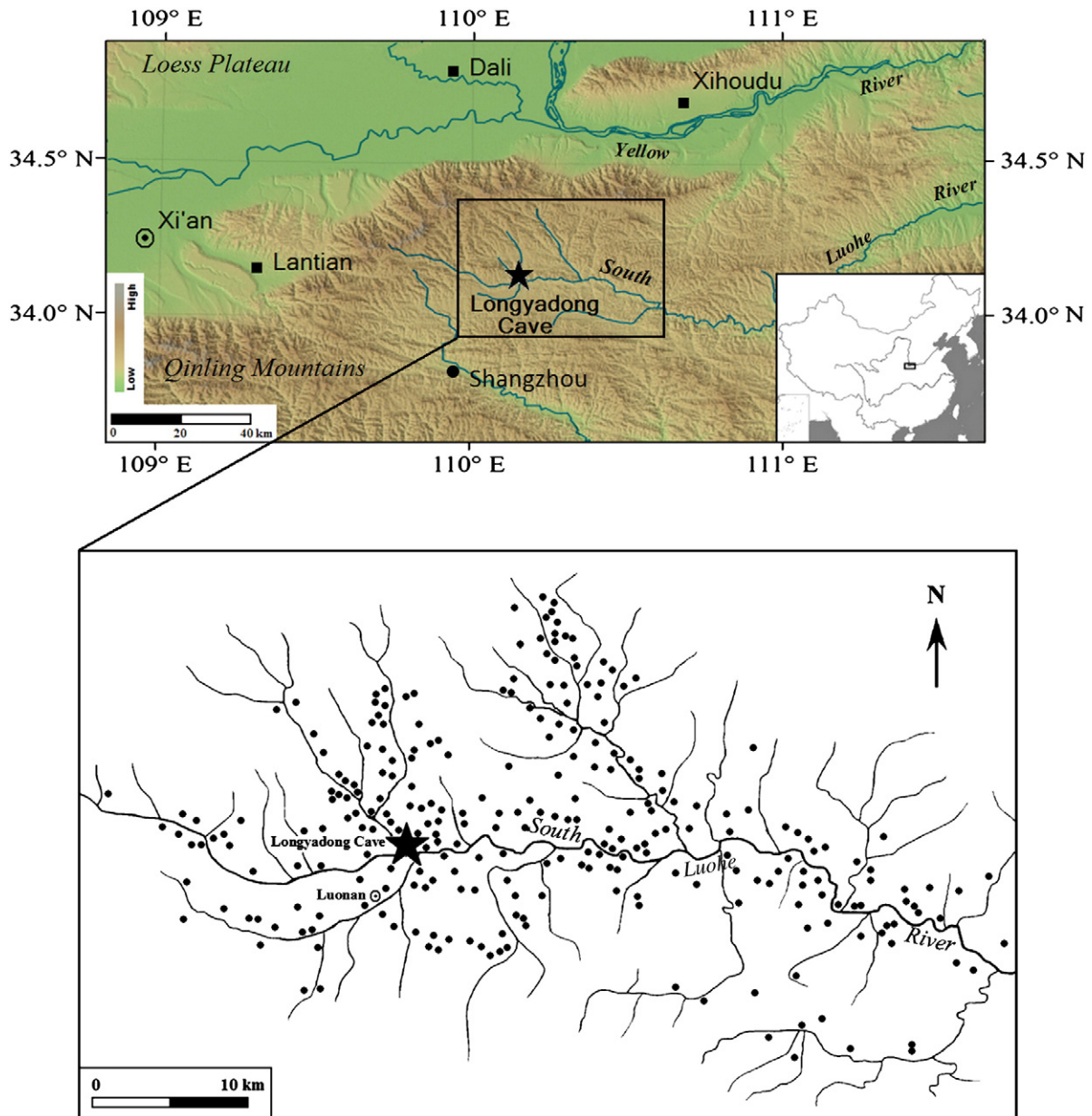
The middle Pleistocene hominin sites in China are critically important to the study of human evolution in East Asia. However, the ages of most sites, in particular those older than 100 ka and younger than the Brunhes–Matuyama boundary (780 ka), are not well established due to limitations of existing dating techniques or the lack of appropriate materials. Dating of the famous Zhoukoudian Locality 1 (Peking Man site) in northern China provides a good example: several dating methods have been applied, but the results are scattered and are still contentious (Wu et al., 1985; Zhao et al., 1985; Yuan et al., 1991; Grün et al., 1997; Shen et al., 2001, 2009). Thus, to apply newly refined dating techniques to these hominin occupations is still of interest.

In this context, the Luonan Basin in Shaanxi Province, central China, seems to be a key area. The Luonan Basin is an intermountain depression with an elevation of 993 m, through which the upper South Luohe River flows in the Eastern Qinling Mountains (Fig. 1). The basin has an annual precipitation of 706 mm and an average annual temperature of 11.0°C; it is covered by a mosaic of grass and reforested and native plant communities. Previous archeological studies have suggested that this region represents the transitional zone between the southern

pebble-core tool industries and the northern flake-core tool industries in China (Zhang, 2002; Shaanxi Provincial Institute of Archaeology and Museum of Luonan County, 2008). Not far from the Luonan Basin are several important early and middle Pleistocene archeological sites, including the Lantian and Dali sites from which the early *Homo* and Archaic *Homo sapiens* remains were recovered (Wang et al., 1979; An and Ho, 1989; Xiao et al., 2002; Zhu et al., 2003; Yin et al., 2011) (Fig. 1). By 2004 a systematic archeological survey program had identified a total of 269 open-air sites, four of which have been partially test-excavated, and one, Longyadong Cave, has been fully excavated (Wang et al., 2004, 2005, 2008; Wang, 2005; Shaanxi Provincial Institute of Archaeology et al., 2007). These sites are located along the upper South Luohe River and its tributaries (Fig. 1) and are situated within the 2nd to 6th river terraces. Loess with thickness from 2 to 25 m is deposited on the terraces, containing distinct loess–paleosol alternations (Lu et al., 2007, 2011; Zhang et al., 2012). Most of these sites, including the base of Longyadong Cave, are on the 2nd terrace. None of the sites have been properly dated until recently, when dates for the Shangbaichuan and Liuwan loess–paleosol sequences on the 2nd terrace (Lu et al., 2007, 2011) provided a preliminary reference time frame for these hominin occupations.

More than 10,000 artefacts have been collected from the sites, being stone tools made of local cobbles found in the South Luohe River. The main percussion techniques used were direct hard hammer percussion and anvil-chipping techniques. Tools include a variety of

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**Figure 1.** Longyadong Cave location on the Upper South Luohe River, Luonan Basin, central China. Also shown in the upper figure are the adjacent archeological sites (solid squares) and the Shangzhou loess section site (solid point). The lower figure shows the open-air Paleolithic sites in the Luonan Basin (modified from Wang, 2005).

Mode I chopper-chopping tools such as choppers, scrapers, points and burins. In addition, many typical Acheulean-like artefacts such as hand-axes, cleavers and bifacially modified trihedrals have been found. From this evidence it has been inferred that there were intensive hominin activities in the basin (Wang, 2005; Wang et al., 2005, 2008; Lu et al., 2007, 2011; Shaanxi Provincial Institute of Archaeology et al., 2007); however, interpretations of hominin behavior and paleoenvironment have not been possible due to a lack of reliable chronologies for hominin occupation.

The technique using thermally transferred optically stimulated luminescence (TT-OSL) signals were developed by Smith and Aitken (Smith et al., 1986; Aitken and Smith, 1988). More recently, Wang et al. (2007) used the TT-OSL signal of quartz to develop a dating method and used Chinese loess deposits to test and validate their procedure. A number of problems with the proposed single aliquot procedure (Wang et al., 2007) have been reported, with several modifications to the single aliquot regenerative (SAR) TT-OSL (SAR-TT-OSL) protocol being proposed (Tsukamoto et al., 2008;

Porat et al., 2009; Stevens et al., 2009; Adamiec et al., 2010; Jacobs et al., 2011; Duller and Wintle, 2012). These studies have significantly improved the reliability of the TT-OSL technique. In this study, we have employed the procedure developed by Stevens et al. (2009), which was also tested on a Chinese loess sample and showed that there is still signal growth up to a dose of at least 12 kGy, so that this method may usefully extend the age range beyond the current limits of conventional OSL for sediments.

#### Stratigraphy and sampling

Longyadong Cave is located on the Huashilang hillslope behind Donghe village, on the second terrace of the South Luohe River approximately 3 km north of the town of Luonan County (Fig. 1). The cave is about 6 m long and 1.5–2.5 m wide. The cave entrance was buried under a mixture of collapsed deposit when it was discovered by local farmers, who sought fossilized bones as valuable “dragon bones”, a type of local herbal ingredient sold in drugstores in the





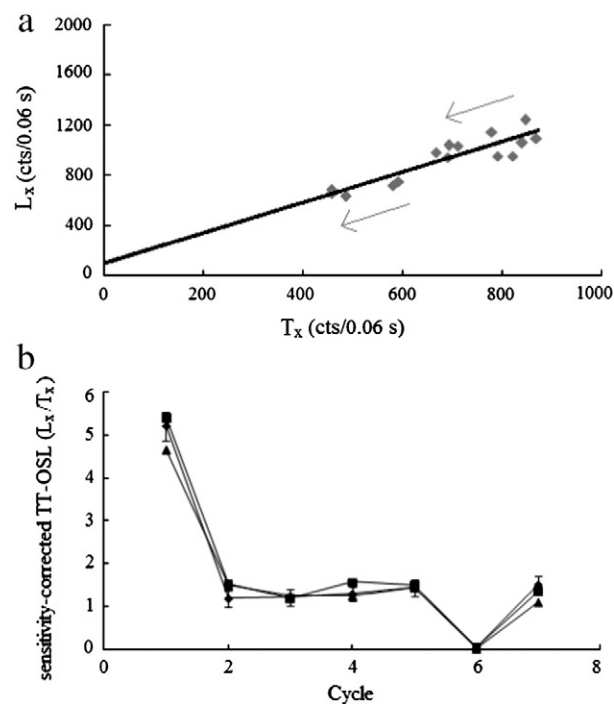
carbonates and organic matter, then etched with 35% fluorosilicic acid ( $\text{H}_2\text{SiF}_6$ ) to remove feldspars, and sieved again. The quartz grains were then mounted as a monolayer with a 5-mm spray mask on the 9.8-mm-diameter aluminum disks using silicone spray oil. The purity of the isolated quartz was checked by infrared (IR) stimulation, and no significant infrared stimulated luminescence (IRSL) signal was detected.

All signals were measured using a Risø TL/OSL-DA-20C/D luminescence reader. Blue LEDs ( $\lambda = 470 \pm 30$  nm) were used for optical stimulation of the quartz grains. The OSL and TT-OSL signals were measured using an EMI 9235QB15 photomultiplier tube and 7.5-mm-thick Hoya U-340 glass filter (Bøtter-Jensen et al., 1999). All measurements were carried out in the Luminescence Dating Laboratory at Nanjing University. For the 40–63  $\mu\text{m}$  grains, an alpha efficiency ( $\alpha$ -value) of  $0.035 \pm 0.02$  (Rees-Jones, 1995; Lai et al., 2003, 2008a,b) was used. Concentrations of  $^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{40}\text{K}$  were obtained by neutron activation analysis (NAA). Sample-specific water contents were determined by laboratory analysis (Table 1). The cosmic ray dose rate was calculated using present-day burial depths (Prescott and Hutton, 1994).

### Equivalent dose ( $D_e$ ) determination

To determine whether the test dose is appropriately monitoring sensitivity change, the relationship between the regeneration ( $L_x$ ) and test dose ( $T_x$ ) signals (Murray and Wintle, 2000) should be tested. If the protocol has been used successfully, the correlation between  $L_x$  and  $T_x$  is linear and the best-fit line passes close to the origin (Stevens et al., 2009). The correlation between  $L_x$  and  $T_x$  by SAR cycle was obtained for sample LYD-1 (Supplementary 2). Figure 3a shows that the relationship between  $L_x$  and  $T_x$  is linear, and passes within  $1\sigma$  of the origin. Figure 3b shows that when the same dose (420 Gy) was repeated five times (cycles 2–5 and 7) following cycle 1, good reproducibility was obtained. This, together with a signal consistent with zero when a zero dose was administered (cycle 6), indicates that no charge was carried over from one measurement cycle to the next and that the high temperature bleaches ( $290^\circ\text{C}$  for 400 s) used to remove this charge did not cause a significant disproportional change in sensitivity between the sensitivity-corrected repeat measurements. Here we prove that this was consistent with the presence of a residual natural TT-OSL signal after laboratory bleaching, due to the difficult-to-bleach nature of the TT-OSL signal (Supplementary 2), and that it is a common phenomenon for Chinese loess (Sun et al., 2012). It is important to note that when bleached for 215 h in the solar simulator, the entire residual signal seems to have been removed, and the  $L_x/T_x$  cycling was approximately consistent across all measurement cycles.

After these tests, measurements of the SAR TT-OSL sequence were carried out (Supplementary 3). A special high-temperature blue light bleach ( $290^\circ\text{C}$  for 400 s) was used to remove any remnant TT-OSL signal following the previous dosing. The TT-OSL signal was calculated from the sum of the first 0.6 s of optical stimulation, from which a background signal was subtracted, estimated from the final 6 s of optical stimulation (total stimulation time 60 s). The sensitivity change correction produced recycling ratios in the 0.90–1.10 range for samples LYD-3, LYD-4 and LYD-5, and in the 0.85–1.12 range for LYD-1 and LYD-12. The average recycling ratio of all aliquots was  $1.01 \pm 0.01$ . All dose response curves were fitted using either a linear,



**Figure 3.** TT-OSL characteristics for sample LY-1. (a)  $L_x$  vs.  $T_x$  after repeating identical doses between each cycle as part of the sequence described by Stevens et al. (2009) on three aliquots of LY-1 ( $L_x = 420$  Gy dose;  $T_x = 280$  Gy dose) following initial bleaching twice at room temperature by blue LEDs in a luminescence reader for 300 s. (b)  $L_x/T_x$  vs. measurement cycle at constant regeneration dose for three aliquots of LY-1 (same data as in part (a)). The first cycle was the remanent natural magnetization after laboratory bleaching as in (a). The sixth cycle was the zero dose point.

saturation-exponential or saturation-exponential-plus-linear function. A representative TT-OSL decay curve is shown in Figure 4a, and the growth curve for one aliquot from sample LY-1 is shown in Figure 4b. Although LY-1 was the oldest sample, no dose saturation was observed up to a dose of 1800 Gy, while  $D_e < 1373$  Gy was comfortably obtained (Fig. 4b). The  $D_e$  frequency distribution of sample LY-1 is presented in Figure 4c; it approximates a normal distribution from which an average can be calculated to obtain a combined  $D_e$  estimate for age determination.

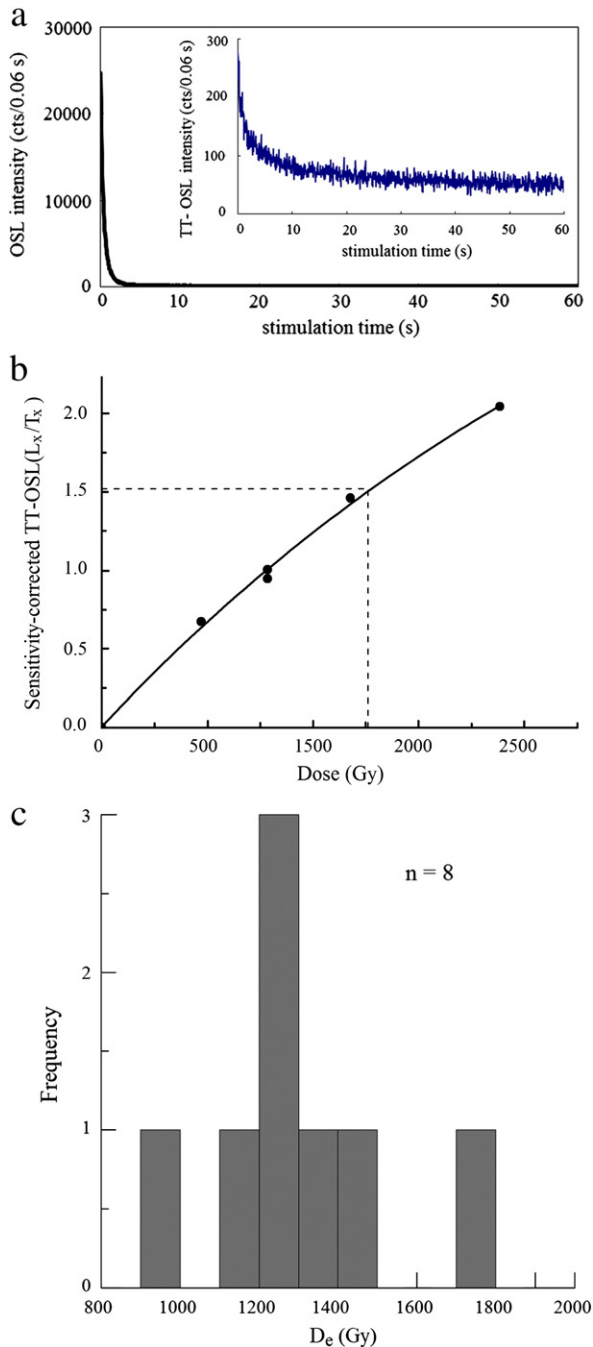
### Results

The  $D_e$  and dose rate information are listed in Table 1, and the TT-OSL and TL age sequences are shown in Figure 4. The mean  $D_e$  values range from  $1378 \pm 54$  to  $846 \pm 42$  Gy (Table 1) and increase with depth, but there is no statistically significant difference between the mean  $D_e$  values of samples LY-1, LY-2 and LY-3. The TT-OSL age estimates are in stratigraphic order, ranging from  $389 \pm 18$  to  $205 \pm 19$  ka (Fig. 5), consistent with previously reported TL ages (Wang and Huang, 2001). The agreement in ages suggests that both the TT-OSL and TL ages are reliable. Therefore, it is suggested that the TT-OSL and TL ages now provide a timeline for early hominin occupation of the Luonan Basin.

**Table 1**

Element concentrations, water content, equivalent dose ( $D_e$ ), total dose rate and TT-OSL ages for the samples collected from the Longyadong Cave sediments.

Sample No.	Depth (m)	$^{238}\text{U}$ (ppm)	$^{232}\text{Th}$ (ppm)	$^{40}\text{K}$ (%)	Water content (%)	Total dose rate (Gy/ka)	$D_e$ (Gy)	Age (ka)
LYD-1	9.0	$3.46 \pm 0.12$	$12.1 \pm 0.35$	$2.48 \pm 0.07$	$19 \pm 3.8$	$3.54 \pm 0.08$	$1373.7 \pm 54.3$	$389 \pm 18$
LYD-2	8.0	$2.57 \pm 0.11$	$10.8 \pm 0.32$	$2.44 \pm 0.07$	$17 \pm 3.4$	$3.45 \pm 0.08$	$1302.8 \pm 54.4$	$377 \pm 17$
LYD-3	7.3	$2.54 \pm 0.10$	$11.2 \pm 0.32$	$2.45 \pm 0.07$	$8 \pm 1.6$	$3.67 \pm 0.08$	$1349.2 \pm 193.5$	$367 \pm 53$
LYD-4	6.0	$2.30 \pm 0.09$	$11.7 \pm 0.34$	$2.34 \pm 0.07$	$4 \pm 0.8$	$3.74 \pm 0.08$	$1116.1 \pm 38.5$	$298 \pm 12$
LYD-5	4.5	$2.40 \pm 0.10$	$15.6 \pm 0.42$	$2.41 \pm 0.07$	$4 \pm 0.8$	$4.13 \pm 0.09$	$846.3 \pm 42.0$	$205 \pm 19$



**Figure 4.** Data for sample LY-1. (a) A representative OSL and TT-OSL decay curve for a single aliquot. The OSL signal was 100 times greater than the TT-OSL signal. (b) Representative growth curve. (c) Frequency histogram of  $D_e$  distribution from the 8 single aliquots measured for this sample.

The luminescence chronology indicates that the initial hominin settlement at Longyadong occurred  $389 \pm 18$  ka at least; Layer 2 is also a cultural layer, which should be older, but no age was obtained for this layer. The cave may have seen continuous hominin occupation from  $389 \pm 18$  until  $274 \pm 14$  ka; however, the gradually filling of the cave over a long period of time may have reduced the available space in the cave, so that after about 270 ka Longyadong Cave was probably too small to be occupied. The cave eventually filled with alluvium and collapsed sediment (Layer 5) between  $210 \pm 11$  and  $205 \pm 19$  ka.

## Discussions

### Paleomagnetic stratigraphy

In order to provide complementary data, additional analyses were performed on the Longyadong sediments. In 2009, we attempted to use magnetostratigraphic analysis to constrain the age of deposition of the bottom sediments in the cave, and to determine whether it could have been as old as the Brunhes–Matuyama boundary ( $\sim 780$  ka). Fifteen oriented cube samples were collected from a freshly cleaned section wall in Trench 1 (Supplementary 1). These samples were then systematically heated in a non-magnetic field, in  $30$ – $50^\circ\text{C}$  steps from room temperature to  $610^\circ\text{C}$ , to remove secondary magnetization. The progressive demagnetization successfully isolated the characteristic remanent magnetization (ChRM) for each of the samples after removing viscous remanent magnetization (VRM) following treatment at  $150^\circ\text{C}$ – $300^\circ\text{C}$ . The demagnetized samples were measured by a 2 G superconductor magnetic meter, and the principal component directions were calculated by a least-squares fitting technique (Kirschvink, 1980). For the analysis (Supplementary 1), inclinations and declinations of all samples were positive, and the Longyadong deposit sequence was well-correlated with the positive magnetic Brunhes Chron (Cande and Kent, 1995). These results demonstrated that the entire Longyadong deposit is younger than 780 ka.

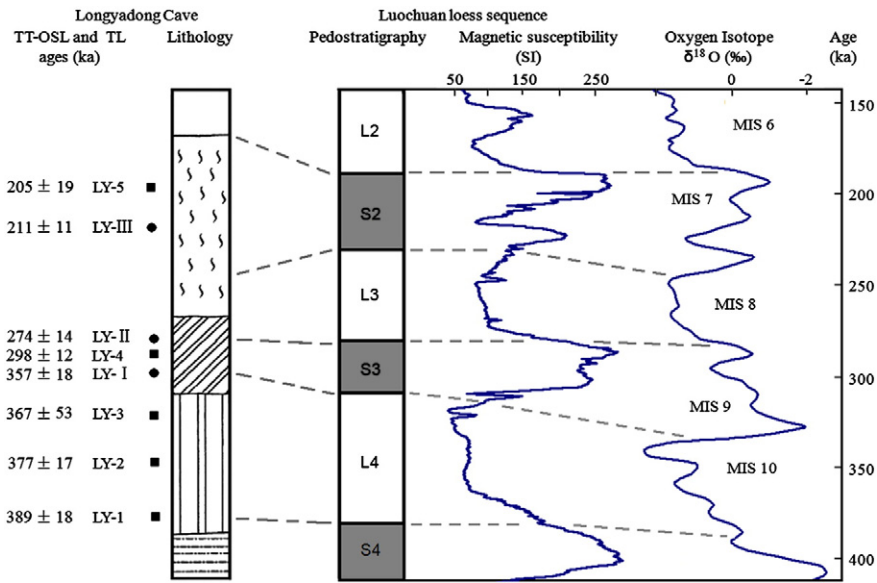
### Correlation with the loess sequence and marine isotopic sequences

Loess has covered a large area of northern and central China since at least the early Pleistocene (Lu et al., 2010). The time scale, stratigraphic correlation, and paleoenvironmental changes of typical Chinese loess deposits are well established (Liu, 1985; Lu et al., 1999; Liu et al., 2000). We compared the Longyadong Cave sedimentary sequence, using the TT-OSL ages as temporal controls, with the dated loess–paleosol sequence in the Luonan Basin (Lu et al., 2007, 2011), and with the typical loess–paleosol sequence of the Loess Plateau (Lu et al., 1999, 2004) and the marine oxygen isotope record (Bassinot et al., 1994; Zachos et al., 2001) (Fig. 5) to further examine the time of the hominin occupation of Longyadong Cave.

Layer 3, aged between  $389 \pm 18$  and  $367 \pm 53$  ka, predominantly consists of loess and should be correlated with the L4 loess unit in the typical loess–paleosol sequence of the central Loess Plateau, for which an orbitally tuned age of between 385 and 334 ka has been reported (Lu et al., 1999), and it has been correlated with Marine Isotope Stage (MIS) 10 in the global ice-volume time series (Bassinot et al., 1994; Zachos et al., 2001) (Fig. 5). By contrast, Layer 4 has ages between  $357 \pm 18$  and  $274 \pm 14$  ka, and consists mainly of soils. It can be correlated to the S3 paleosol that has an orbitally tuned age range between 334 and 279 ka (Lu et al., 1999); it can also be correlated with MIS 9, which was a warm, humid period (Bassinot et al., 1994; Zachos et al., 2001). The middle to upper part of Layer 5 has ages of  $210 \pm 11$  ka (LY-3) and  $205 \pm 19$  ka (LY-5), and can be correlated with the S2 paleosol of the Chinese loess sequence and MIS 7 in the global ice-volume time series; this was a warm, humid interglacial period. No samples were collected from Layer 2, so its age remains uncertain, but since it contained the earliest evidence of hominin occupation it should be older than  $389 \pm 18$  ka, which correlates with the S4 paleosol of the loess–paleosol sequence (Lu et al., 1999).

### The paleoenvironment

Palynological analysis of the loess–paleosol section at the Shangzhou site (location shown in Fig. 1), about 30 km from the Longyadong Cave site, revealed a rich pollen assemblage (Lei, 2000). This loess–paleosol sequence lasted from about 700 to 100 ka. The pollen assemblage is relatively rich in arboreal pollen dominated by



**Figure 5.** Correlation between the Longyadong deposit with the loess stratigraphy in Luochuan and the marine oxygen isotope record (Bassinet et al., 1994). The orbitally tuned ages of the loess sequence at Luochuan are from Lu et al. (1999).

*Quercus*, *Pinus*, *Castanea* and *Betula*, and also much non-arboreal pollen, dominated by *Artemisia*, *Compositae*, and *Chenopodiaceae* (Fig. 6). The paleosol units, relatively rich in *Quercus*, *Castanea* and *Juglans*, indicate a warm, wet climate, reflecting an optimal climate for hominin occupation. In the loess units, pollens are dominated by *Artemisia*, *Compositae* and *Gramineae*, and arboreal pollen from *Pinus*, *Betula* and *Taxodiaceae*, indicating a relatively cold, dry climate (Fig. 6). Less diversity and smaller quantities of arboreal pollen in the loess units than in the paleosol units suggest a harsher environment at the time of loess deposition. Significant environmental changes in this area have also been reconstructed by our field pedogenetic observations and analyses (Zhang et al., 2012).

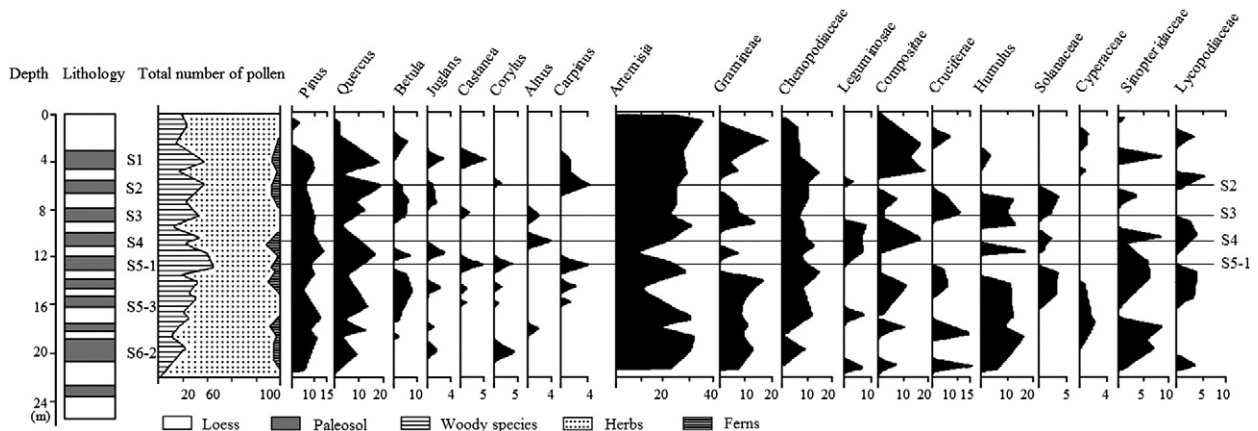
Layer 3 of the Longyadong Cave deposit corresponds to the L4 loess unit in the Shangzhou loess–paleosol sequence (between the S4 and S3 paleosols in Fig. 6). Although a relatively cold, dry climate prevailed, the L4 stage was an important time for hominin activity inside the cave, and demonstrates that early hominins adapted to the cold environment during a glacial period in the Luonan Basin. Pollen assemblages of the S3 paleosol in the Shangzhou loess–paleosol sequence are relatively rich in *Quercus*, *Castanea* and *Juglans* content, indicating a warm, wet climate (Lei, 2000) that may have been most favorable for hominins living in this region.

**Conclusion**

Our study indicates that the  $D_e$  value obtained using the SAR TT-OSL procedure was generally satisfactory for dating the sediments at the Longyadong Cave. The TT-OSL chronology is in good agreement with the interpretations of the sediment sequences and with previously obtained TL ages. Adopting the TT-OSL benchmark age, the loess and paleosol units at Longyadong Cave have ages ranging from  $389 \pm 18$  to  $274 \pm 14$  ka. These correlate with L4 and S3 in the Chinese loess sequence, and clearly indicate paleoenvironmental changes. Thus, the hominins occupied Longyadong Cave for ~115,000 yr, adapting and living through changing environments from the cold, dry to warm, humid climates.

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**Figure 6.** Pollen assemblage of the loess deposit at Shangzhou, about 30 km from the Longyadong site. Modified from Lei (2000).

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <http://dx.doi.org/10.1016/j.yqres.2012.10.003>.

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