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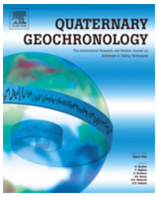

Research paper

ESR chronology of alluvial deposits in the Arlanzón valley (Atapuerca, Spain): Contemporaneity with Atapuerca Gran Dolina site

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ABSTRACT

The Sierra de Atapuerca (Northern Spain) is characterized by a well-developed karst system where several major archaeological sites have been discovered, attesting an almost continuous hominin occupation of the area during the whole Pleistocene period. Previous geomorphological studies showed a connection between genesis of the karst system and the evolution of the nearby Arlanzón river Valley. However, numerical dating results were missing to refine the chronostratigraphical framework of the Arlanzón valley's fluvial incision. To address this, we applied the Electron Spin Resonance (ESR) dating method to sedimentary optically bleached quartz grains from several fluvial terraces. Nine samples were collected from five of the 14 identified terraces. The ESR age results are stratigraphically coherent and in general agreement with both previous geomorphological observations and available palaeomagnetic data. Consequently, an ESR chronology of the geological evolution of the Arlanzón valley is proposed, which can be then correlated to the sedimentary sequence of the palaeoanthropological site of Atapuerca Gran Dolina. Our results provide important information about the chronology of hominid occupation in this area during Early and Middle Pleistocene.

1. Introduction

The Sierra de Atapuerca (Burgos, Northern Spain) is characterized by a well-developed karst system where an almost continuous hominid occupation has been documented since 1.2 Ma, through the discovery of a series of major archaeological sites, such as Sima del Elefante, Gran Dolina, Sima de los Huesos, Galeria or Portalón, among others (Arsuaga et al., 1997; Carbonell et al., 1999, 2008; Pérez-Gonzalez et al., 1999; Rosas et al., 2001; Bischoff et al., 2007). Previous investigations combining geomorphological evolution analysis of the Sierra de Atapuerca landscape (Benito-Calvo, 2004) and the study of the karst systems (Ortega, 2009) revealed a connection between the karst formation and the evolution of the nearby Arlanzón River located southwards (Fig. 1). While the chronology of the archaeological sites is now well established (among others: Berger et al., 2008; Bischoff et al., 2007; Carbonell et al., 2008; Falguères et al., 1999; Moreno, 2011), geochronological data are still missing to constrain the evolution of the fluvial incision of the Arlanzón valley.

To obtain numerical dates on the Pleistocene fluvial system of Arlanzón River, we applied electron spin resonance (ESR) dating to optically bleached quartz grains extracted from sediments. This method has already been successfully used in fluvial contexts, such as the Somme River terrace system in the Northern part of France (Laurent et al., 1998; Bahain et al., 2007), the main tributaries of the Loire River in the Centre Region, France (Voinchet et al., 2010), and the alluvial terrace sequences in Zhangjiajie, northwest Hunan Province, China (Yang et al., 2011).

The aim of this paper is to present the first ESR age results obtained on the terrace system of the Arlanzón River and to

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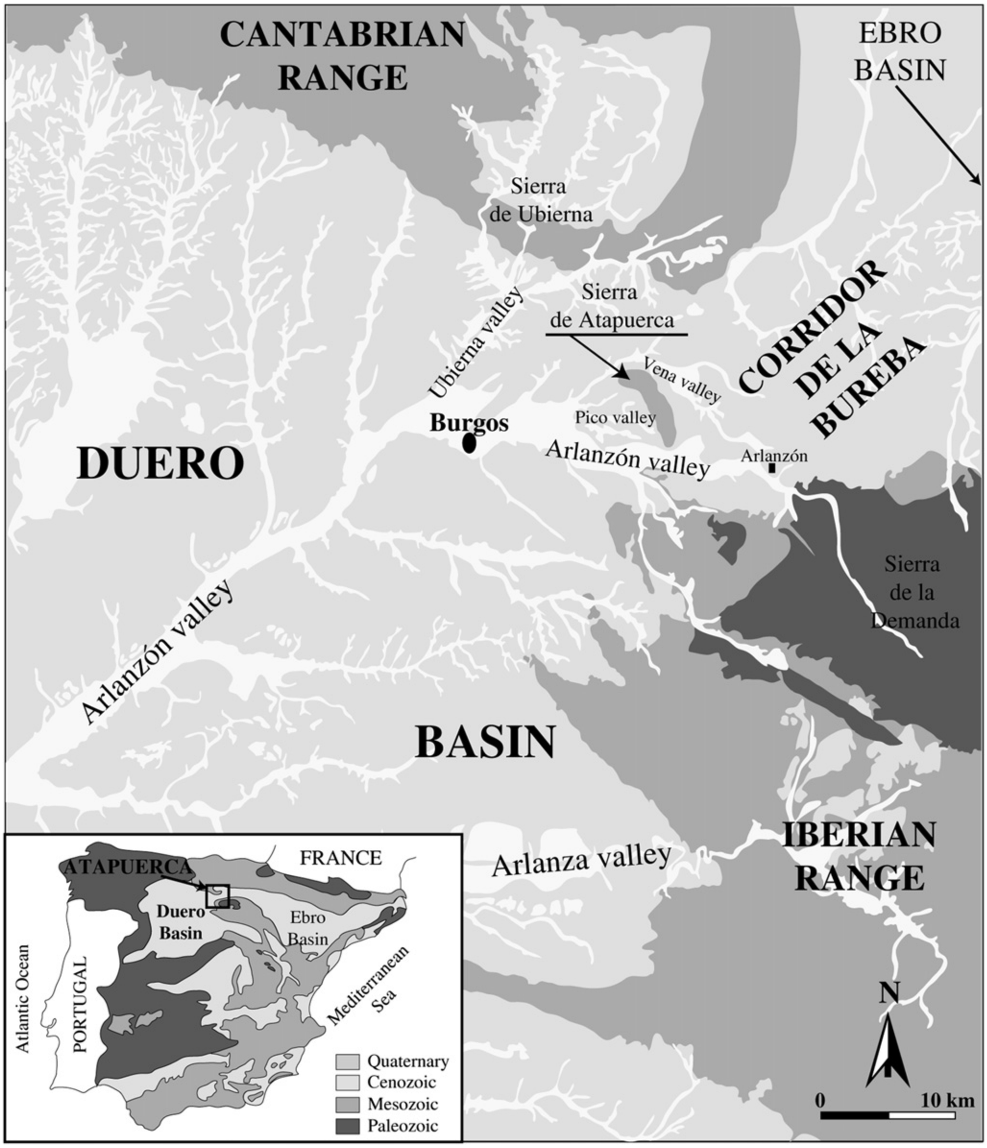


Fig. 1. Geological context of the Sierra de Atapuerca (Modified from Benito-Calvo et al., 2008).

attempt a correlation between the Gran Dolina karstic site and the incision process of the valley.

2. The Arlanzón terrace system

The studied area is located in the North–East part of the Cenozoic Duero River Basin (north-central Iberian Peninsula), which lies between the Iberian and Cantabrian ranges and connects with the Ebro River basin (Fig. 1). The Duero River region constitutes a large endorheic basin filled up during the Palaeogene and Neogene with alluvial and lacustrine detrital sediments (Benito-Calvo et al., 2008). During Upper Miocene and Pliocene, a connection between the Duero basin and the Atlantic Ocean was opened, initializing the incision of the present-day fluvial network of the Arlanzón River and its tributaries (Benito-Calvo et al., 2008; Ortega et al., 2010). This region has then been affected by a tectonic activity during the Late Cenozoic, causing the formation and development of these valleys.

The Arlanzón valley is characterized by a stepped terrace system where 14 terraces have been identified in addition to the present floodplain. These terraces are named from $T1_{AZN}$ to $T14_{AZN}$ (from top to bottom), and are distinguished by relative altitudes (Fig. 2). The fluvial deposits mainly consist of gravels occasionally interstratified by sandy or clayish beds (Benito-Calvo and Pérez-González, 2002; Benito-Calvo, 2004). Previous palaeomagnetic studies carried out on some of the terraces indicated a normal polarity for the $T5_{AZN}$ terrace and a reverse polarity for the $T4_{AZN}$, suggesting the presence of the Brunhes–Matuyama boundary in between (Benito-Calvo et al., 2008).

3. Material and methods

ESR dating of fluvial quartz is based on the detection of various radiation induced paramagnetic centers associated to defects present in the crystalline structure of the quartz (Weil, 1984). Various centers can usually be observed by ESR spectroscopy in quartz, but those showing the best potential to date Quaternary fluvial sediments are the aluminium (Al) (Voinchet et al., 2004) and the titanium (Ti) centers (Tissoux et al., 2007; Gao et al., 2009). Despite of the better bleaching characteristics of the Ti center, its

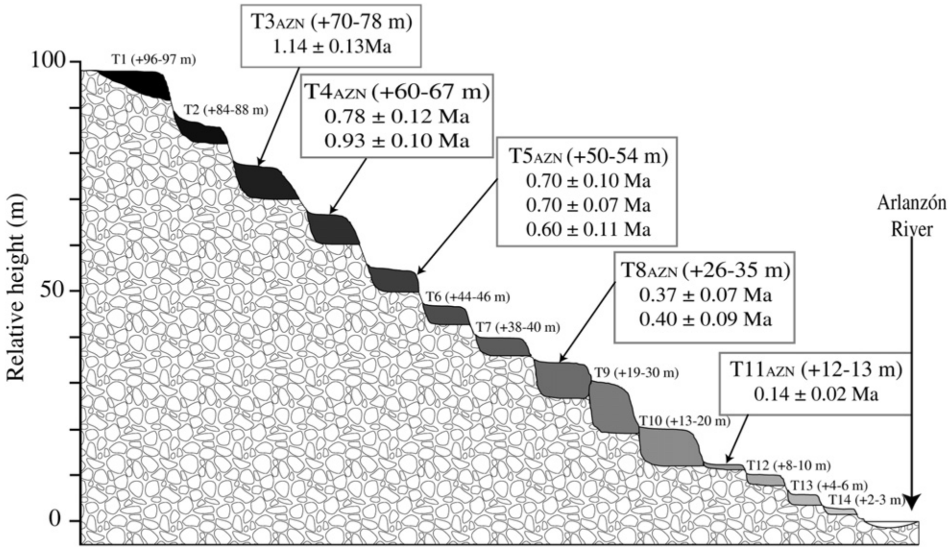


Fig. 2. ESR age results obtained for the Arlanzón valley stepped-terraces system.

ESR intensity was too close to the background in our samples, precluding any reliable and reproducible measurements. Consequently, the Al center was exclusively used in this study.

The ESR signal of the Al center is bleached by sunlight exposure (Yokoyama et al., 1985) and mechanical stress (Liu and Grün, 2011) during the transport phase prior to the sediment burial. However, these phenomena cannot fully reset to zero the ESR intensity and a residual ESR signal has to be assessed and subtracted from the measured ESR intensities before any equivalent dose calculation (see Voinchet et al., 2003).

In this study, five out of the 14 identified terraces of the Arlanzón river system were sampled (Fig. 2): T3$_{\mathrm{AZN}}$ (1 sample), T4$_{\mathrm{AZN}}$ (2 samples), T5$_{\mathrm{AZN}}$ (3 samples), T8$_{\mathrm{AZN}}$ (2 samples) and T11$_{\mathrm{AZN}}$ (1 sample). Relative altitudes of all terraces are given in Table 1.

Sample preparation followed Voinchet et al. (2007) for the extraction of the 100–200 μm fractions of quartz grains. Each sample was divided in twelve aliquots: in addition to the natural reference, 10 were irradiated from 400 to 25 000 Gy with a panoramic ^{60}Co source (Dolo et al., 1996) following an exponential dose step distribution. In addition, one aliquot was optically bleached for ~1500 h using a SOL 2 (Dr Hönle) solar simulator (details in Moreno, 2011).

Aluminium ESR intensities were measured at low temperature (~110 K) with an EMX Bruker X-band spectrometer, using the following experimental conditions: 5 mW microwave power, 1024 points resolution, 100 kHz modulation frequency, 0.1 mT modulation amplitude, 40 ms conversion time, 40 ms time constant and 90 G sweep width.

The angular dependence of the ESR signal due to the sample heterogeneity was taken into account by measuring each aliquot three times after a rotation of 0°, 120° and 240° in the cavity. Furthermore, data reproducibility was checked by running ESR measurements during three distinct days. Consequently, a data set of nine ESR intensities was obtained for each aliquot.

The ESR intensity of Al center was extracted from peak-to-peak measurement between the top of the first peak at $g = 2.018$ and the bottom of the 16th peak at $g = 2.002$ of the Al hyperfine structure (Toyoda and Falguères, 2003). The nine ESR intensities per aliquot were then normalized and considered as independent.

To eliminate the incongruous measurements mainly due to electromagnetic fluctuation in the gun diode, we did not take into account the extreme values of the nine ESR intensities obtained for each aliquot. An average value was then calculated from the five

Table 1
ESR ages and associated data obtained for the samples from Arlanzón valley and replaced within the synthetic general alluvial sequence proposed by Benito-Calvo (2004) (*Bl*, bleaching percentage; *Dcos*, cosmic dose; D_a, total dose rate; D_E, equivalent dose; n.c., not calculated); (γ-doses measured in laboratory are in brackets).

	Terrace	Sample	Water %	*Bl* %	Dα (μGy/a)	Dβ (μGy/a)	Dγ (μGy/a)	*Dcos* (μGy/a)	D_a (μGy/a)	D_E (Gy)	Age (Ma)
Arlanzón Valley	T1$_{\mathrm{AZN}}$ (+92 to 97 m)										
	T2$_{\mathrm{AZN}}$ (+84 to 88 m)										
	T3$_{\mathrm{AZN}}$ (+70 to 78 m)	AZN 05-05	10 ± 5	52	78 ± 3	935 ± 22	671 ± 67	154 ± 8	1864 ± 71	2121 ± 226	1.14 ± 0.13
	T4$_{\mathrm{AZN}}$ (+60 to 67 m)	AZN 05-03	10 ± 5	58	60 ± 2	1404 ± 26	(877 ± 21)	154 ± 8	2521 ± 33	2350 ± 255	0.93 ± 0.10
		AZN 05-04	10 ± 5	55	124 ± 4	1251 ± 25	(775 ± 20)	154 ± 8	2330 ± 32	1827 ± 277	0.78 ± 0.12
	T5$_{\mathrm{AZN}}$ (+50 to 54 m)	AZN 05-01	10 ± 5	54	115 ± 3	1408 ± 23	(921 ± 18)	77 ± 4	2522 ± 29	1523 ± 276	0.60 ± 0.11
		AZN 05-02	10 ± 5	55	87 ± 2	1395 ± 25	(1041 ± 21)	77 ± 4	2601 ± 33	1822 ± 265	0.70 ± 0.10
		AZN 08-05	10 ± 5	54	101 ± 2	1848 ± 27	1251 ± 125	75 ± 4	3275 ± 128	n.c.	n.c.
		AZN 08-06	10 ± 5	59	99 ± 2	1700 ± 30	1289 ± 129	75 ± 4	3128 ± 132	2200 ± 209	0.70 ± 0.07
	T6$_{\mathrm{AZN}}$ (+44 to 46 m)										
	T7$_{\mathrm{AZN}}$ (+38 to 42 m)										
	T8$_{\mathrm{AZN}}$ (+26 to 35 m)	AZN 08-03	10 ± 5	54	88 ± 2	1609 ± 30	973 ± 97	196 ± 10	2767 ± 102	1101 ± 252	0.40 ± 0.09
		AZN 08-04	10 ± 5	49	70 ± 2	1329 ± 30	918 ± 92	217 ± 11	2404 ± 97	878 ± 161	0.37 ± 0.07
	T9$_{\mathrm{AZN}}$ (+19 to 30 m)										
	T10$_{\mathrm{AZN}}$ (+13 to 20 m)										
	T11$_{\mathrm{AZN}}$ (+12 to 13 m)	AZN 08-10	12 ± 5	44	84 ± 2	1450 ± 27	1219 ± 122	175 ± 9	2915 ± 125	410 ± 51	0.14 ± 0.02
	T12$_{\mathrm{AZN}}$ (+8 to 10 m)										
	T13$_{\mathrm{AZN}}$ (+4 to 6 m)										
	T14$_{\mathrm{AZN}}$ (+2 to 3 m)										

central values which are considered as the most representative of each aliquot. Such a value constitutes the experimental data used for the fitting of the dose-responses curves.

The equivalent dose values (D_E) were calculated with the Microcal Origin 8.0 software. The single saturating exponential (SSE) function, traditionally used to describe dose–response curves of quartz grains (e.g. Liu et al., 2010; Voinchet et al., 2010), does not fully fit the experimental data points obtained from the Arlanzón valley samples, leading to a significant overestimation of the equivalent dose value (e.g. Duval et al., 2011). Therefore, we have used a fitting by a single saturating exponential plus linear function (SSE + LIN) because this function describes better the experimental data points (Fig. 3) (see Duval et al., 2009). Data were weighted by the inverse of the squared intensity ($1/I^2$). The errors associated to D_E values are assessed according to the Microcal Origin 8.0 software using the Levenberg–Marquardt algorithm by chi-square minimization.

The total dose rate (D_a) was obtained from the sum of the α-, β-, γ- and cosmic-ray contributions. *In situ* measurements of the natural radioactivity of the sediment were carried out using a NaI probe connected to an Inspector 1000 multichannel analyser (Canberra). *In situ* gamma dose rates were calculated using the threshold approach (see details in Mercier and Falguères, 2007). External α and β contributions were assessed from the radioelement contents (U, Th and K) determined by high resolution γ-spectrometry in laboratory (Yokoyama and Nguyen, 1980). The following parameters were used for the age calculations: dose-rate conversions factors from Adamiec and Aitken (1998), a *k*-value of 0.2 ± 0.1 (Yokoyama et al., 1985), α and β attenuations from Brennan et al. (1991) and Brennan (2003), water attenuation formulae from Grün (1994) and a cosmic dose rate calculated from the equations of Prescott and Hutton (1994). A water content of 10% was assumed. The internal dose rate was considered as negligible because of the low contents of radionuclides usually found in quartz grains (Yokoyama et al., 1985).

The errors associated with annual doses as well as equivalent doses, and with ESR age estimates are given at 1σ.

4. Results and discussion

Based on the combination of results derived from magnetostratigraphic and geomorphologic studies, Benito-Calvo (2004) established a preliminary relative chronostratigraphical framework of the Arlanzón fluvial system. Firstly, the floodplain and the lowest terrace, T14$_{AZN}$, were correlated to the Holocene. Then, the two following terraces, T13$_{AZN}$ and T12$_{AZN}$ were associated to the Upper Pleistocene. The intermediate terraces, from T11$_{AZN}$ to T4$_{AZN}$, were attributed to the Middle Pleistocene. Finally, the three upper terraces, from T3$_{AZN}$ to T1$_{AZN}$, were correlated to the Lower Pleistocene. In addition, since the paleomagnetic study showed a normal polarity for T5$_{AZN}$ and a reversed polarity for the base of T4$_{AZN}$, the Brunhes-Matuyama (B-M) boundary was placed during the formation of T4$_{AZN}$ terrace (Benito-Calvo et al., 2008).

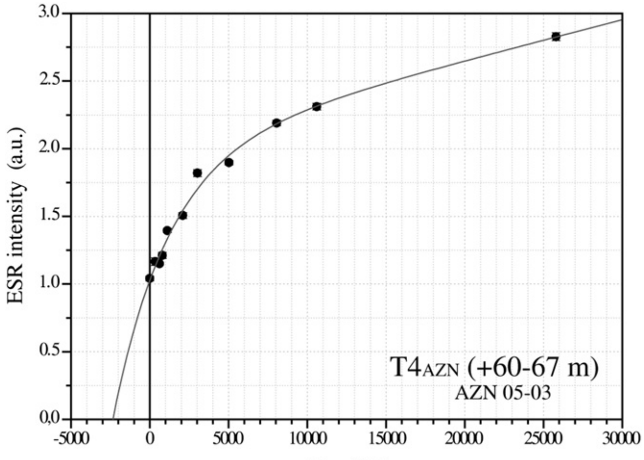


Fig. 3. ESR dose–response curve of the sample AZN 05-03 taken from T4$_{AZN}$ terrace (+60–67 m).

The ESR results and associated data are shown in Table 1. The sample from T11$_{AZN}$ was dated to 0.14 ± 0.02 Ma, in agreement with its previous Upper Pleistocene attribution. The two samples from T8$_{AZN}$ provided reproducible ages of 0.37 ± 0.07 Ma and 0.40 ± 0.09 Ma, as well as the three samples from T5$_{AZN}$, with 0.70 ± 0.10 Ma, 0.70 ± 0.07 Ma and 0.60 ± 0.11 Ma. The ESR age estimates are increasing with the relative altimetry of the corresponding terraces and definitely suggest that fluvial sheets were most likely deposited during the Middle Pleistocene. These results are in agreement with the normal polarity obtained for T5$_{AZN}$ (Benito-Calvo et al., 2008). An ESR age could not be calculated for sample AZN 08-05. The dose response data were too scattered and no dose response curve could be fitted in spite of the use of several distinct functions.

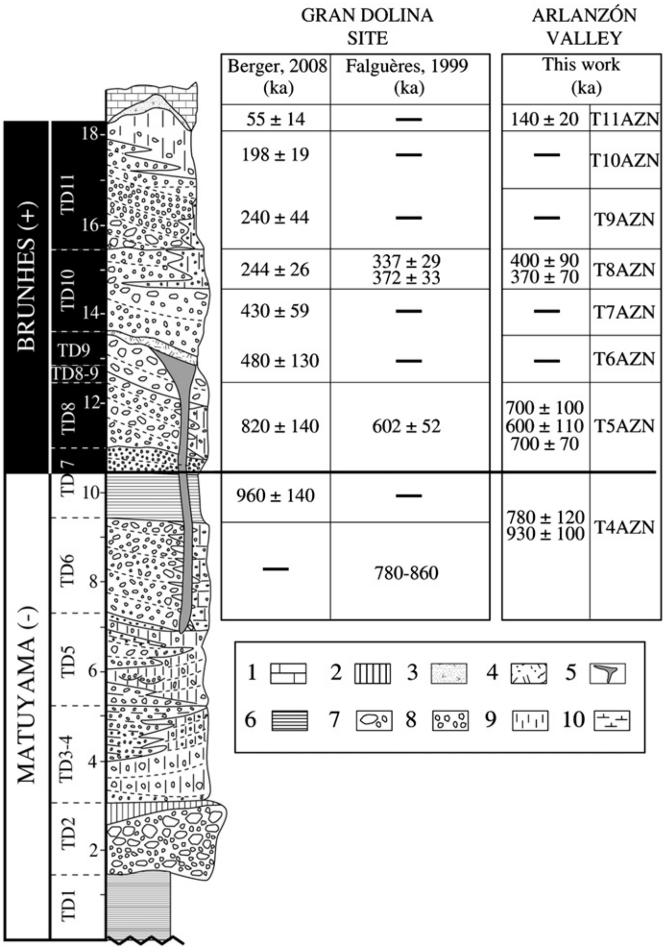
Two ESR results of 0.78 ± 0.12 Ma and 0.93 ± 0.10 Ma were obtained for T4$_{AZN}$ suggesting either a late Lower Pleistocene, or an early Middle Pleistocene age for this terrace, given the large scatter between both ages. These results are statistically not distinguishable, but because the ages are constrained by the B–M boundary (Benito-Calvo et al., 2008), this terrace was most likely deposited during the Lower Pleistocene period. From a geological point of view, it seems also be possible that T4$_{AZN}$ is constituted by two embedded fluvial bodies. Additional sampling should be made in this terrace for clarification.

Lastly, an ESR age of 1.14 ± 0.13 Ma was obtained for T3$_{AZN}$, which dates the deposition of this fluvial unit to the Lower Pleistocene, in good agreement with previous geological and geomorphological data.

5. Chronological relation with Gran Dolina site

Several studies demonstrated a close relationship between the geomorphological evolution of the Arlanzón system and the formation and development of the phreatic endokarst in the Sierra de Atapuerca (Benito-Calvo et al., 2008; Ortega et al., 2010). During the Lower Pleistocene, this endokarst, characterized by a multilevel caves system, was air-opened, as attested by the deposition of allochtonous sediments and the discovery of hominin occupation at the Atapuerca Gran Dolina and at the Sima del Elefante (Carbonell et al., 1995, 2008; Pérez-Gonzalez et al., 2001).

The Gran Dolina site shows an 18 m-thick sedimentary infilling, divided into 11 stratigraphic units, named from TD1 to TD11 (from bottom to top). The chronostratigraphical framework has been established by combination of independent methods, such as combined US-ESR, TL and IRSL, and magnetostratigraphic analyses (Berger et al., 2008; Falguères et al., 1999; Parés and Pérez-González, 1995). A first attempt of correlation between the karst evolution and genesis and the Arlanzón terraces was made by Ortega (2009). ESR ages obtained in the Arlanzón valley in the present study, help to refine this preliminary model. Indeed, the T8$_{AZN}$ terrace can be associated to the top of level TD10 (TD10-1 & TD10-2) and the T5$_{AZN}$ terrace may be contemporaneous with the TD8 level and the top of the level TD7. The TL ages (Berger et al., 2008) and ESR/U–Th ones (Falguères et al., 1999) are coherent with the ESR results obtained on fluvial quartz (Fig. 4). They suggest a Middle Pleistocene age for the T8$_{AZN}$ terrace and an early Middle



GRAN DOLINA SITE		ARLANZÓN VALLEY	
Berger, 2008 (ka)	Falguères, 1999 (ka)	This work (ka)	
55 ± 14	—	140 ± 20	T11AZN
198 ± 19	—	—	T10AZN
240 ± 44	—	—	T9AZN
244 ± 26	337 ± 29 372 ± 33	400 ± 90 370 ± 70	T8AZN
430 ± 59	—	—	T7AZN
480 ± 130	—	—	T6AZN
820 ± 140	602 ± 52	700 ± 100 600 ± 110 700 ± 70	T5AZN
960 ± 140	—	780 ± 120 930 ± 100	T4AZN
—	780-860		

Fig. 4. Lithostratigraphy and magnetic polarity of the Gran Dolina section (updated from Parés and Pérez-González, 1999) with the locations of the luminescence (TL & IRSL) ages (Berger et al., 2008) and ESR/U-series ages (Falguères et al., 1999). Attempt to correlation with Arlanzón valley stepped-terraces system. (1) Mesozoic limestone at the Gran Dolina roof; (2) speleothem; (3) terra rossa; (4) guano of bats; (5) unconformity and loamy-clayey-sandy filling; (6) laminated loamy clays; (7) gravels and boulders; (8) gravels; (9) lutites and clays; (10) marlstone.

Pleistocene age for the $T5_{AZN}$ terrace. Finally, the hominid-bearing level TD6 in which *Homo antecessor* was unearthed and the base of the level TD7 are contemporaneous with $T4_{AZN}$ deposits corresponding to a Lower Pleistocene age.

6. Conclusion

The ESR results obtained in the Arlanzón valley are consistent and reinforce the chronostratigraphic framework established by the combination of geomorphologic and palaeomagnetic data (Benito-Calvo, 2004; Benito-Calvo et al., 2008). The agreement between palaeomagnetic data and the ESR ages obtained for $T4_{AZN}$ and $T5_{AZN}$ confirms the potential of the ESR method on the fluvial deposits of the Sierra de Atapuerca area and shows their contemporaneity with human-bearing deposits of Gran Dolina site.

Cave infilling sediments may be quite complex, because constrained by the cave's geometry and evolution with time, so that their correlation with the external fluvial history is difficult. In the Gran Dolina, the sedimentary sequence is chronologically constrained by a few punctual absolute ages for some of the layers. In absence of a complete chronology of the entire sequence, a correlation with Arlanzón valley would be hazardous. Consequently, given the promising results presented here, future studies have to be focused on a continuous ESR sampling of Gran Dolina deposits (Moreno, 2011), combined with additional sampling of the terraces that could not be dated in this work, in order to carry out a reliable correlation between both systems.

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