



## Research paper

## New radiometric dates on the lowest stratigraphical section (TD1 to TD6) of Gran Dolina site (Atapuerca, Spain)



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## ABSTRACT

Ages based on independent methods, such as combined ESR-U series, luminescence, and magnetostratigraphic analyses have been obtained for the upper stratigraphical section of Gran Dolina site (TD6 to TD11 levels). However, the chronostratigraphical framework of this European Paleolithic key site remains incomplete because of its great antiquity and the lack of reliable methods. This paper provides new radiometric dates by electron spin resonance applied to optically bleached quartz grains for the whole stratigraphic sequence. The results agree with the previous chronostratigraphical framework for the upper part of the stratigraphical sequence. The ages for the *Homo antecessor* remains from TD6 layer range between 800 and 900 ka. The lowest layers attributed to endogenous sediments (TD1) could be contemporaneous with the Sima del Elefante TE9 human bearing layer dated to 1.2 Ma. The results suggest a human occupation of possibly more than 1 Ma at the Gran Dolina site. This study confirms moreover the potential of ESR dating method applied on quartz in karstic environment.

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## 1. Introduction

The Sierra de Atapuerca (Northern Spain) is characterized by a well-developed karst system where several archaeological sites were found after the construction of a railway trench, "Trinchera Atapuerca", at the end of the XIX century (Fig. 1). Among all the famous sites discovered at Trinchera Atapuerca, the infilling of the Gran Dolina represents one of the most complete stratigraphic Pleistocene sequences, with a thickness of about 18 m which is divided into 11 lithostratigraphic units named TD1 to TD11, from bottom to top (see Supplementary data) (Gil et al., 1987; Pérez-González et al., 2001). A magnetostratigraphic study divided this

sequence into a Middle (TD8 to TD11) and a Lower (TD1 to TD7) Pleistocene part due to a reverse-to-normal polarity change between TD7 and TD8, interpreted as the Brunhes-Matuyama boundary (Parés and Pérez-González, 1995). This paleomagnetic interpretation is consistent with the biostratigraphic record (Cuenca-Bescós and García, 2007) and the ages obtained on the TD6 to TD11 levels by ESR/U-series on fossil teeth (Falguères et al., 1999) and thermoluminescence (TL) (Berger et al., 2008) (see Supplementary data). The lower levels (TD1 to TD6) offer the opportunity of improving the knowledge about the hominin population before the Brunhes-Matuyama boundary but dating these remains and their enclosing sediments is difficult (Duval et al., 2012).

Since the first ESR dating application in 1975 by Ikeya on a speleothem from Akiyoshi Cave in Japan, numerous ESR dating applications have been developed on a wide range of materials such as marine and continental carbonates, tooth enamel and quartz grains from eolian, fluvial and littoral sediment (Grün, 1989; Bahain

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Fig. 1. Location and plan of the Sierra de Atapuerca cave systems showing the main archaeological sites (Modified from G. E. Edelweiss).

et al., 1995; Falguères et al., 1999; Voinchet et al., 2004; Grün, 2006). Thanks to the ubiquity of quartz, its use as a geochronometer to date sediments is particularly interesting in Quaternary Geology and archaeology because it often represents the only one suitable material for geochronological studies.

ESR dating applied to optically bleached quartz grains extracted from sediments (ESR-OB) is based on the detection of radiation induced paramagnetic centers. Similarly to optically stimulated luminescence (OSL) dating, the ESR signal measured in quartz may be reset by optical bleaching, allowing thus to date not the formation of the mineral but the last sunlight exposure moment (see Supplementary Data). It has been successfully used to date alluvial terraces and associated prehistoric sites over the last decades (Yokoyama et al., 1985; Voinchet et al., 2004; Tissoux et al., 2008; Liu et al., 2013; Moreno et al., 2012), but so far the specific application to karstic environment has never been reported. This paper provides the first attempt on dating karstic sediments by ESR dating of sedimentary quartz. Results were cross-checked with those of other techniques for the upper part in order to evaluate the reliability of ESR-OB ages for the lower part of Gran Dolina sequence.

## 2. Material and method

Each unit of the Gran Dolina stratigraphic sequence was sampled, except TD2. The compaction and hardening of the latter, probably related to post sedimentary fluid migration, avoids any sampling and precludes any *in situ* dose rate measurements. 39 samples were collected, with a special attention to the main archaeological units like TD6 and TD10 (Fig. 2). Where possible, the ESR-OB samples were taken as close as the samples previously collected for luminescence (Berger et al., 2008) or paleomagnetism (Parés and Pérez-González, 1999) studies.

In quartz, two main ESR paramagnetic centers are classically

used for dating: aluminium (Al) and the titanium (Ti) centers (Falguères et al., 1991; Voinchet et al., 2003; Tissoux et al., 2007, 2008). ESR intensities of Ti-center signals from Gran Dolina samples were very close to the ESR background, precluding any reliable and reproducible measurements. Thus, Al-center was exclusively used in this study. As the optical bleaching of this paramagnetic center is not complete in nature, the residual unbleachable signal intensity of each sample was determined and subtracted from the measured ESR intensities before any equivalent dose calculation (see Voinchet et al., 2003). ESR measurements were performed with a Bruker EMX spectrometer (X-band) at Muséum National d'Histoire Naturelle, Paris, at 108 K, using a nitrogen gas flow system. Details of sampling protocol and measurements are available in Supplementary data.

## 3. Results and discussion

All the ESR-OB results are presented in Table 1. Taking into account the high heterogeneity of the layers, the results obtained for each level will be presented separately.

### 3.1. The lowermost Gran Dolina unit (TD1 to TD7)

22 samples were analyzed from the lowermost part of Gran Dolina site. In TD1 unit, TD08-01 and TD08-02 were sampled in an indurated sandy layer partially laminated whereas TD08-01bis was taken in sandy lutite close to a speleothem. TD08-01bis yields a low total dose rate ( $D_a$ ) compared with the two other samples likely due to the proximity of a calcitic formation less rich in radioelements than sediments. This sample yielded an age of  $920 \pm 190$  ka. The ESR results obtained for TD1 range between  $790 \pm 60$  ka and  $1.25 \pm 0.13$  Ma suggesting a late Lower Pleistocene age. These results were compared with the normal magnetization observed in TD1 layer which has been preliminarily correlated to Jaramillo subchron (0.99–1.07 Ma) or Cobb Mountain event (1.22–1.24 Ma) (Parés and Pérez-González, 1999; Pérez-González et al., 2001). The large error range obtained on ESR-OB samples does not allow any correlation with a specific normal event and just suggests an age older than 800 ka.

The levels TD3-4 and TD5 form a 10 samples set. TD08-03 and TD08-04 were taken in a silty to fine sandy with limestone pebbles facies and TD08-05 and TD08-06 in a sandy facies with gravels and blocks located 75 cm above. TD08-07 was sampled 60 cm above TD08-06. All samples show equivalent dose values ( $D_E$ ) ranging between 1200 and 2300 Gy. A relatively large variation in the dose rate evaluation is also observed yielding a higher  $D_a$  ( $\sim 2578 \mu\text{Gy/a}$ ) for the first facies than in the second facies which exhibits a mean  $D_a$  of  $1939 \mu\text{Gy/a}$ . This resulted in younger ages for samples TD08-03 and TD08-04 compared to those from TD08-05, TD08-06 and TD08-07. Five other samples were collected in TD5, which is represented by large blocks among a fine sandy matrix capped by a small clayey level. Three of them (TD08-09, TD08-11 and TD08-12) did not yield any ages because of unknown signals were superimposed with the Al-center signal, thus precluding any  $D_E$  determinations. These signals could be related to manganese (Grün, personal comm.) or to a large iron signal. The two others samples show total dose rates and equivalent doses similar to those of the TD3-4 level. The opening of the cave occurred simultaneously and together with the TD3-4 and TD5 levels deposition. These deposits correspond to the first exogenous sediments of the Gran Dolina stratigraphic sequence. Biostratigraphic studies proposed an age of approximately 1 Ma for these levels (Made, 1999) and a correlation with the marine isotopic stage (MIS) 22 (Cuenca-Bescós and García, 2007). Except for TD08-03 and 04, which are definitely too young probably in association to the difficulty of reconstructing the

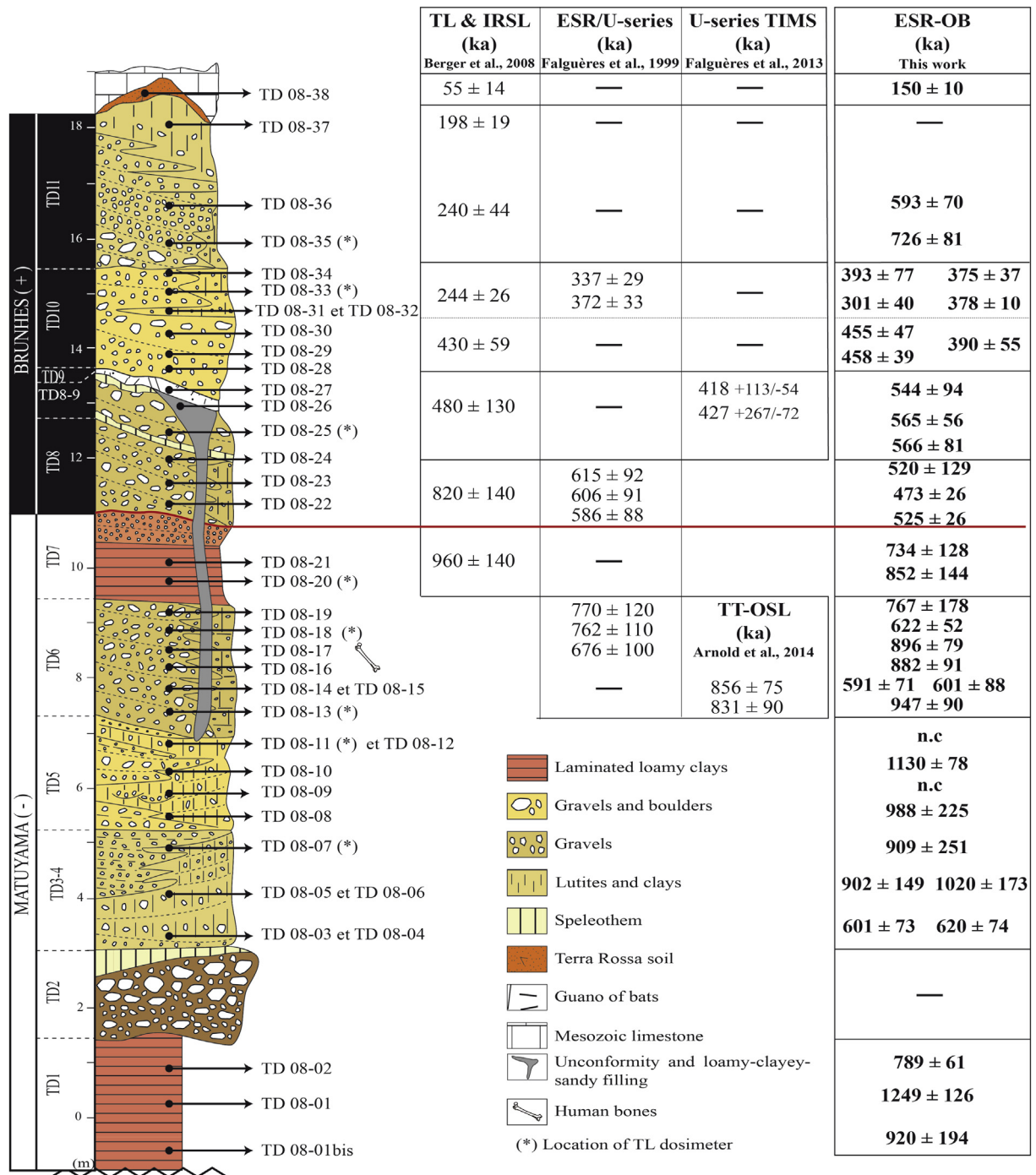


Fig. 2. Stratigraphic profile of the Gran Dolina infilling with the location of the 39 samples studied in this work and the previous radiometric data obtained by ESR/U-series data (Falguères et al., 1999), U-series TIMS (Falguères et al., 2013), TL/IRSL (Berger et al., 2008) and TT-OSL (Arnold et al., 2014). Comparison with the ESR-OB results obtained in this work.

external dose, the other ESR-OB results suggest an age ranging between  $900 \pm 150$  ka and  $1.13 \pm 0.08$  Ma for TD3-4 and TD5 levels, which are coherent with these biostratigraphic data.

Seven samples were taken and analyzed from TD6 layer, which has archaeologically been divided in three sub-horizontally layers from the bottom to the top (Bermúdez de Castro et al., 2008): TD6-3 (TD08-13, 14, 15), TD6-2 also called “Aurora stratum” (Parés and Pérez-González, 1995) in which the *Homo antecessor* humans remains (Bermúdez de Castro et al., 1997) were found (TD08-16, 17, 18) and TD6-1 (TD08-19). The  $D_a$  varies between 2765 and 3552  $\mu\text{Gy/a}$ , while the  $D_E$  values fall into two groups:  $\sim 1700$  Gy

and  $\sim 2700$  Gy providing ages ranging between  $600 \pm 90$  ka and  $950 \pm 80$  ka. The dose rate reconstruction is very difficult due to the heterogeneity and the presence of large amount of lithic artifacts and bones in this level. The ages are clearly dependent on the reliable estimation of the dose rate causing underestimation in several samples. Two samples (TD08-20 and 21), taken in TD7, show homogeneous  $D_a$  and  $D_E$  ranging between 1620 and 1920 Gy yielding ages of  $850 \pm 140$  ka and  $730 \pm 130$  ka respectively.

In TD6, in spite of a quick lateral variation of the  $D_a$  inducing strong changes in the age calculation, four samples are dated to  $770 \pm 180$  ka

**Table 1**  
ESR ages and associated data obtained for the samples from Gran Dolina site (Bl, bleaching percentage;  $D_{\text{cos}}$ , cosmic dose;  $D_{\text{a}}$ , total dose rate;  $D_{\text{E}}$ , equivalent dose; n.c., not calculated).  $\gamma$ -dose measured in laboratory are in regular,  $\gamma$ -dose measured with TL dosimeter are in bold and  $\gamma$ -dose measured *in situ* with a portable spectrometer are in italic.

Level	Sample	Bl %	$D_{\alpha}$ ( $\mu\text{Gy/a}$ )	$D_{\beta}$ ( $\mu\text{Gy/a}$ )	$D_{\gamma}$ ( $\mu\text{Gy/a}$ )	$D_{\text{cos}}$ ( $\mu\text{Gy/a}$ )	$D_{\text{a}}$ ( $\mu\text{Gy/a}$ )	$D_{\text{E}}$ (Gy)	Age (Ma)
TR	TD 08-38	49	$105 \pm 4$	$1691 \pm 46$	$1290 \pm 39$	$168 \pm 8$	$3254 \pm 61$	$482 \pm 44$	<b><math>0.15 \pm 0.01</math></b>
TD11	TD08-37	—	$52 \pm 2$	$987 \pm 21$	$680 \pm 17$	$131 \pm 7$	$1851 \pm 27$	n.c.	<b>n.c.</b>
	TD08-36	59	$71 \pm 2$	$1403 \pm 29$	$945 \pm 24$	$110 \pm 6$	$2530 \pm 37$	$1500 \pm 175$	<b><math>0.59 \pm 0.07</math></b>
TD10	<b>TD08-35</b> <sup>a</sup>	59	$37 \pm 2$	$714 \pm 22$	<b><math>612 \pm 61</math></b>	$94 \pm 5$	$1457 \pm 68$	$1058 \pm 107$	<b><math>0.73 \pm 0.08</math></b>
	TD08-34	64	$120 \pm 3$	$2036 \pm 36$	<b><math>637 \pm 63</math></b>	$90 \pm 5$	$2883 \pm 50$	$1134 \pm 219$	<b><math>0.39 \pm 0.08</math></b>
	<b>TD08-33</b> <sup>a</sup>	63	$120 \pm 3$	$2036 \pm 36$	<b><math>637 \pm 63</math></b>	$81 \pm 4$	$2874 \pm 79$	$864 \pm 113$	<b><math>0.30 \pm 0.04</math></b>
	TD08-32	55	$120 \pm 3$	$2036 \pm 36$	<b><math>637 \pm 63</math></b>	$85 \pm 4$	$2878 \pm 50$	$1080 \pm 103$	<b><math>0.38 \pm 0.04</math></b>
	TD08-31	54	$124 \pm 3$	$1968 \pm 35$	$1459 \pm 29$	$98 \pm 5$	$3649 \pm 46$	$1380 \pm 32$	<b><math>0.38 \pm 0.01</math></b>
	TD08-30	57	$122 \pm 3$	$1629 \pm 40$	$1263 \pm 33$	$78 \pm 4$	$3093 \pm 52$	$1408 \pm 143$	<b><math>0.46 \pm 0.05</math></b>
	TD08-29	58	$116 \pm 7$	$1998 \pm 85$	$1459 \pm 70$	$72 \pm 4$	$3645 \pm 110$	$1669 \pm 133$	<b><math>0.46 \pm 0.04</math></b>
	TD08-28	51	$109 \pm 2$	$1641 \pm 30$	$1237 \pm 25$	$70 \pm 4$	$3058 \pm 39$	$1194 \pm 166$	<b><math>0.39 \pm 0.05</math></b>
TD9	TD08-27	61	$97 \pm 3$	$1617 \pm 32$	$1187 \pm 27$	$73 \pm 4$	$2974 \pm 42$	$1618 \pm 280$	<b><math>0.54 \pm 0.09</math></b>
	TD08-26	58	$107 \pm 4$	$1766 \pm 45$	$1298 \pm 37$	$65 \pm 3$	$3236 \pm 59$	$1827 \pm 178$	<b><math>0.56 \pm 0.06</math></b>
TD8-9	<b>TD08-25</b> <sup>a</sup>	60	$152 \pm 4$	$1865 \pm 42$	<b><math>895 \pm 90</math></b>	$71 \pm 4$	$2983 \pm 106$	$1687 \pm 235$	<b><math>0.57 \pm 0.08</math></b>
TD8	TD08-24	56	$71 \pm 3$	$1243 \pm 35$	$882 \pm 29$	$83 \pm 4$	$2279 \pm 45$	$1184 \pm 294$	<b><math>0.52 \pm 0.13</math></b>
	TD08-23	55	$71 \pm 3$	$1243 \pm 35$	$882 \pm 29$	$83 \pm 4$	$2279 \pm 45$	$1078 \pm 55$	<b><math>0.48 \pm 0.03</math></b>
	TD08-22	56	$71 \pm 3$	$1243 \pm 35$	$882 \pm 29$	$83 \pm 4$	$2279 \pm 45$	$1197 \pm 54$	<b><math>0.53 \pm 0.03</math></b>
	TD08-21	57	$66 \pm 3$	$1173 \pm 33$	<b><math>920 \pm 92</math></b>	$59 \pm 3$	$2219 \pm 101$	$1629 \pm 275$	<b><math>0.73 \pm 0.13</math></b>
TD6	<b>TD08-20</b> <sup>a</sup>	58	$63 \pm 2$	$1216 \pm 26$	<b><math>920 \pm 92</math></b>	$58 \pm 3$	$2256 \pm 98$	$1922 \pm 177$	<b><math>0.85 \pm 0.14</math></b>
	TD08-19	53	$128 \pm 3$	$1930 \pm 41$	$1437 \pm 34$	$56 \pm 3$	$3552 \pm 53$	$2723 \pm 633$	<b><math>0.77 \pm 0.18</math></b>
	<b>TD08-18</b> <sup>a</sup>	53	$129 \pm 2$	$1744 \pm 24$	<b><math>953 \pm 95</math></b>	$55 \pm 3$	$2882 \pm 100$	$1793 \pm 135$	<b><math>0.62 \pm 0.05</math></b>
	TD08-17	56	$111 \pm 2$	$1676 \pm 29$	$1253 \pm 24$	$54 \pm 3$	$3093 \pm 37$	$2771 \pm 242$	<b><math>0.90 \pm 0.08</math></b>
	TD08-16	54	$112 \pm 4$	$1826 \pm 46$	$1337 \pm 38$	$53 \pm 3$	$3327 \pm 59$	$2936 \pm 298$	<b><math>0.88 \pm 0.09</math></b>
	TD08-15	56	$123 \pm 3$	$1903 \pm 43$	<b><math>886 \pm 89</math></b>	$51 \pm 3$	$2962 \pm 55$	$1752 \pm 208$	<b><math>0.59 \pm 0.07</math></b>
TD5	TD08-14	58	$119 \pm 3$	$1898 \pm 33$	<b><math>886 \pm 89</math></b>	$51 \pm 3$	$2953 \pm 42$	$1775 \pm 258$	<b><math>0.60 \pm 0.09</math></b>
	<b>TD08-13</b> <sup>a</sup>	61	$104 \pm 3$	$1725 \pm 43$	<b><math>886 \pm 89</math></b>	$50 \pm 3$	$2765 \pm 105$	$2619 \pm 228$	<b><math>0.95 \pm 0.08</math></b>
	TD08-12	—	$81 \pm 3$	$1641 \pm 41$	<b><math>585 \pm 59</math></b>	$44 \pm 2$	$1729 \pm 79$	n.c.	<b>n.c.</b>
	<b>TD08-11</b> <sup>a</sup>	—	$62 \pm 2$	$1116 \pm 25$	<b><math>585 \pm 59</math></b>	$45 \pm 2$	$1808 \pm 67$	n.c.	<b>n.c.</b>
	TD08-10	55	$70 \pm 3$	$1130 \pm 39$	$819 \pm 32$	$42 \pm 2$	$2062 \pm 51$	$2330 \pm 150$	<b><math>1.13 \pm 0.08</math></b>
	TD08-09	—	$52 \pm 2$	$929 \pm 26$	$658 \pm 21$	$41 \pm 2$	$1680 \pm 33$	n.c.	<b>n.c.</b>
	TD08-08	56	$52 \pm 2$	$983 \pm 25$	$676 \pm 20$	$40 \pm 2$	$1751 \pm 32$	$1730 \pm 393$	<b><math>0.99 \pm 0.22</math></b>
TD3-4	TD08-07	52	$30 \pm 1$	$506 \pm 14$	$757 \pm 20$	$39 \pm 2$	$1332 \pm 27$	$1211 \pm 334$	<b><math>0.91 \pm 0.25</math></b>
	TD08-06	54	$70 \pm 2$	$1270 \pm 23$	$887 \pm 19$	$37 \pm 2$	$2264 \pm 30$	$2042 \pm 336$	<b><math>0.90 \pm 0.15</math></b>
	TD08-05	53	$50 \pm 2$	$899 \pm 24$	$628 \pm 19$	$37 \pm 2$	$1614 \pm 31$	$1646 \pm 278$	<b><math>1.02 \pm 0.17</math></b>
	TD08-04	46	$74 \pm 2$	$1356 \pm 23$	$947 \pm 19$	$35 \pm 2$	$2412 \pm 30$	$1449 \pm 174$	<b><math>0.60 \pm 0.07</math></b>
	TD08-03	57	$83 \pm 3$	$1557 \pm 44$	$1069 \pm 36$	$34 \pm 2$	$2744 \pm 57$	$1701 \pm 171$	<b><math>0.62 \pm 0.06</math></b>
TD1	TD08-02	55	$85 \pm 3$	$1861 \pm 44$	<i><math>1231 \pm 123</math></i>	$28 \pm 1$	$3205 \pm 135$	$2530 \pm 166$	<b><math>0.79 \pm 0.06</math></b>
	<i>TD08-01</i>	56	$85 \pm 3$	$1861 \pm 44$	<i><math>1231 \pm 123</math></i>	$28 \pm 1$	$3205 \pm 135$	$4004 \pm 366$	<b><math>1.25 \pm 0.13</math></b>
	<i>TD08-01 bis</i>	54	$41 \pm 2$	$827 \pm 31$	$878 \pm 88$	$27 \pm 1$	$1774 \pm 97$	$1631 \pm 332$	<b><math>0.92 \pm 0.19</math></b>

<sup>a</sup> Position of TL dosimeters.

to  $950 \pm 80$  ka in accordance with the magnetostratigraphical data, the ESR/U-series dates (Falguères et al., 1999) and the TT-OSL data (Arnold et al., 2014). TD7 provides ESR-OB ages around  $730 \pm 130$  ka and  $850 \pm 140$  ka with large error range. They are in agreement with those obtained by TL ( $960 \pm 120$  ka, mean age) and fit with the B/M boundary at the top of TD7. Another possibility would be to attribute the changing polarity to the Jaramillo event instead of B/M reversal. This eventuality would correspond to the older part of Jaramillo, ca 1.07 Ma, which disagrees with all TD6 radiometric data. It becomes also difficult to place the B/M boundary in the upper layer and the ages are in contradiction with the ESR dates obtained on teeth and quartz which are reproducible in the upper part of TD8. Moreover, no stratigraphical gap is observed in this part of the sequence. In conclusion, ESR-OB results on the lowermost part of Gran Dolina site are in agreement with the palaeomagnetic data, i.e. older than 800 ka below the TD7 level and they are in favor of a B/M boundary at the top of TD7 as initially published (Parés and Perez-Gonzalez, 1995, 1999).

### 3.2. The uppermost Gran Dolina unit (TD8 to TD11)

17 samples were analyzed from the uppermost part of Gran Dolina (TD8 to TD10/11). In TD8 level, formed by lumpy sediment with a many gravels, blocks and debris flows without matrix, three samples were taken (TD08-22, 23, 24). The heterogeneity of sediment could have had an impact of the laboratory dose rate calculation. Quartz samples yielded  $D_{\text{E}}$  values ranging between 1080 and

1200 Gy and using the same dose rate value ( $2279 \pm 45$   $\mu\text{Gy/a}$ ), the resulting ages are between  $473 \pm 26$  ka and  $525 \pm 26$  ka. They seem slightly smaller in comparison with those obtained by ESR/U-series on teeth (Falguères et al., 1999). TL ages obtained in the base of TD8 and TD7 ranging between  $820 \pm 140$  ka and  $960 \pm 140$  ka, support an older chronology locating the Kamikatsura event at the top of TD7 as suggested by Parés et al. (2013) and positioning B/M boundary upper in the sequence.

The TD8-9 and TD9 layers are constituted by breccias, speleothems, silt and clay. Samples from these levels display ages in good agreement between each other ( $566 \pm 81$  ka,  $565 \pm 56$  ka and  $544 \pm 94$  ka) and a good reliability with the ESR/U-series ages on teeth coming from TD8 and the TL-IRSL sample which yields an age of  $480 \pm 130$  ka. In addition, the last occurrence of *M. savini* recorded in TD8, assigns an age younger than ca. 600 ka to upper deposits.

Seven quartz samples have been analyzed from TD10, which was archeologically divided into 4 sub-layers (TD10-4, at bottom to TD10-1). Regarding the  $D_{\text{a}}$  and the  $D_{\text{E}}$ , a remarkable agreement can be observed for the seven samples resulting in ages ranging between  $\sim 380$  and  $460$  ka for TD10, TD10-33 being slightly smaller due to a lower  $D_{\text{E}}$  value. These data are confirmed by two TIMS measurements performed on calcite samples located at the base of TD10 and providing ages of  $417 + 113/-54$  ka and  $427 + 267/-72$  ka which could indicate a maximum age for TD10 level (Falguères et al., 2013).

According to (Faluères et al., 1999) the TD10 age ranges between  $308 \pm 46$  ka and  $418 \pm 63$  ka is in agreement with the TL age ( $430 \pm 59$  ka) for the middle part of this level (Berger et al., 2008). The TIMS U-series analyses performed at the basis of TD10 yielded a minimum age of 420 ka and our quartz samples confirm an age about 400–450 ka for TD10 in agreement with the previous data. The TL age of  $244 \pm 26$  ka obtained for the upper part of TD10 is in contradiction with the rest of the geochronological data. According to our knowledge, no stratigraphical gap was observed inside TD10 justifying such a lapse of time. This would support the first interpretation of Berger et al. (2008) that the TL results from upper part of TD10 level are too young because of “unremoved anomalous fading components”. In summary, the ESR-OB results reinforce the hypothesis of the contemporaneity of TD10 deposition with the base of Galería infilling (GIIa) and the Sima de los Huesos sequence, both containing handaxes and representing the first occurrence of Acheulian tradition at Atapuerca (Faluères et al., 2013; Arsuaga et al., 2014).

In TD11 layer, one of the three samples analyzed (TD08-37) showed the unknown superimposed signal on Al-center as observed in TD5 samples precluding any age calculation. The rest of the samples, showing a large variation on  $D_a$  values, exhibit ages which are not in agreement with neither the stratigraphical order nor previous chronological data.

The *terra rossa* soil capping the Gran Dolina sequence yielded an age of  $150 \pm 10$  ka in agreement with the ESR result obtained for the T11<sub>AZN</sub> terrace of Arlanzón river of  $140 \pm 20$  ka (Moreno et al., 2012). Both results are older and disagree with TL age of  $55 \pm 14$  ka (Berger et al., 2008).

#### 4. Conclusions

Our results show that the ESR dating on optically bleached quartz (ESR-OB) is applicable to karstic infilling sediments showing a relatively good consistency with the previous chronostratigraphical framework and follow a general trend of increasing ages with depth (Fig. 2).

This study provides, for the first time, radiometric dates for the whole stratigraphic sequence (TD1 to TD11). The ESR-OB ages obtained for the upper part (TD6 to TD11) are coherent with the previous chronological framework and reinforce the Brunhes-Matuyama boundary at the top of TD7 level. This confirms an age ranging between 800 and 900 ka for TD6 level, from which *H. antecessor* were discovered. A mean age of 350–450 ka was obtained for the last human occupation of Gran Dolina, located in TD10 layer and representing the first Acheulian evidence at Atapuerca, coeval with la Sima de los Huesos and the base of Galería complex. Newly obtained ages for the lower layer suggest an age older than 800–900 ka for the first levels of Gran Dolina stratigraphic sequence but any correlation with a normal event (*Jaramillo* or *Cobb Mountain*) can be made because of the large error range obtained on ESR-OB samples.

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#### Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.quageo.2015.05.007>.

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