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A 300–600 ka ESR/U-series chronology of Acheulian sites in Western Europe

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Abstract

For a long time, the establishment of the chronology of Acheulian settlements in Western Europe was restrained by the lack of reliable dating methods. During the Middle Pleistocene, the archaeological sites in this area are generally associated with fluvio-lacustrine and karstic contexts. The main geochronological methods (e.g. K/Ar, Ar/Ar, U-series, OSL and TL) could not be applied because of a lack of suitable material for dating, or the expected ages were beyond the upper limit of the dating technique. For several years, the combination of ESR and U-series data has allowed the direct dating of palaeontological remains from the entire Middle Pleistocene period. Combined ESR/U-series dating was applied to several major Acheulian sites in Spain, Italy and France. This paper presents the dating results, their implications for the chronology of the Acheulian in Western Europe as well as the methodological limits of the ESR/U-series dating method caused by the effect of weathering on the uranium uptake into the teeth.

1. Introduction

For a long time, it has been difficult to establish the chronology of the early Acheulian settlements in Western Europe because of the lack of suitable dating methods. Most Acheulian archaeological sites are associated with fluvial or lacustrine environments or karst infillings. The most commonly used geochronological methods, such as ^{40}K – ^{40}Ar , ^{39}Ar – ^{40}Ar , thermoluminescence (TL) or U-series, usually cannot be applied, because the age of the sites is beyond the range of these methods or did not contain materials suitable for dating.

Electron Spin Resonance (ESR) dating was first applied to fossil teeth in the 1970s (Ikeya, 1978) and appeared to have great potential to address this problem. However, ESR dating was greatly hampered by the problem of the unknown U-uptake, which may cause very large errors. Ten years later, a model combining ESR and U-series data was proposed which made it possible to calculate a specific uranium uptake parameter p for each dental tissue and a single age for each tooth (Grün et al., 1988). The development of this combined ESR/U-series (ESR–US) model applied on fossil dental enamel allows the calculation of direct ages for archaeological strata that contain evidence of human occupation. The method is particularly well suited for sites between 300 and at least 800 ka. In many cases, it is the only method which can be applied beyond 500 ka in carbonate and karst environments (Fig. 1). Subsequent applications have resulted in a number of publications reporting the dates of many Pleistocene archaeological sites (Schwarcz et al., 1989; Grün et al., 1996; Falguères et al., 1999).

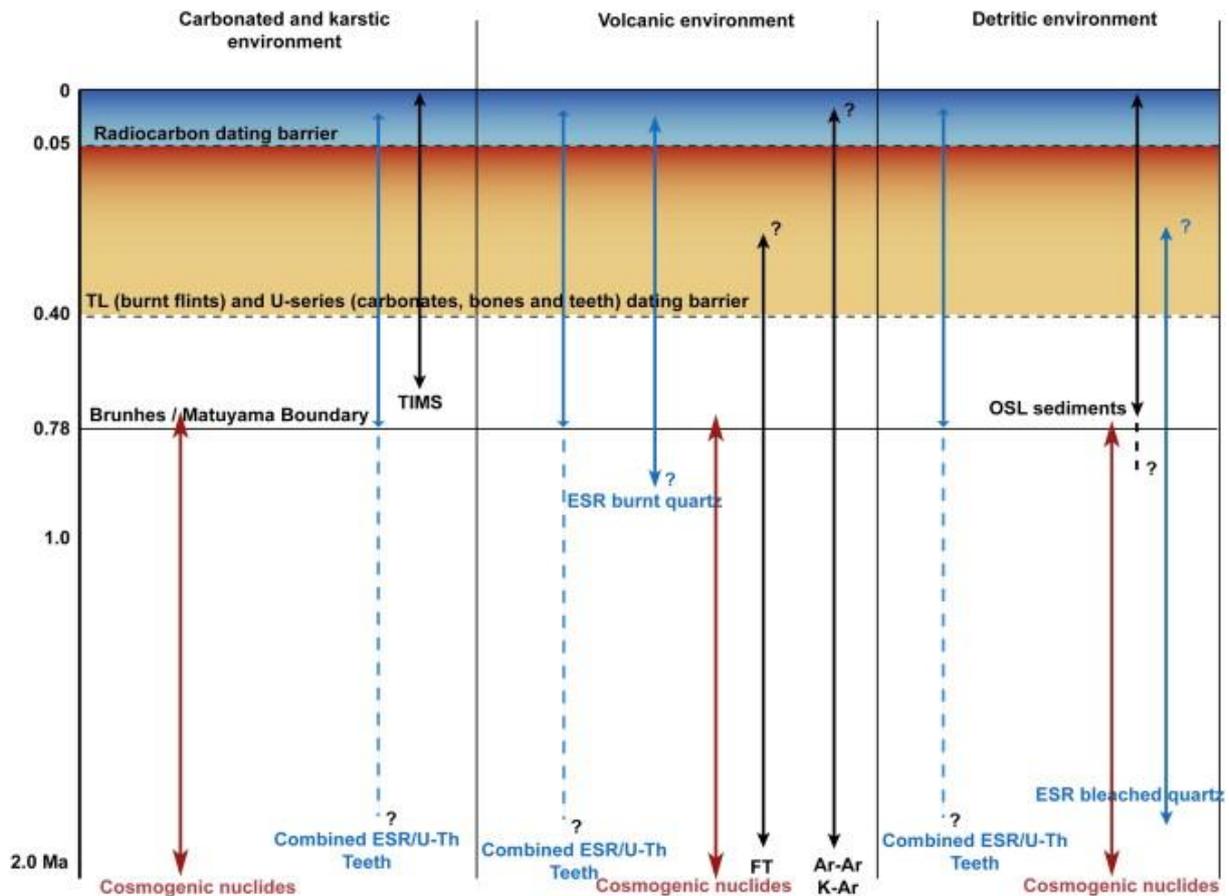


Fig. 1. Applicability range for the main numerical dating methods available for the last two million years. The blue space corresponds to the period covered by radiocarbon dating. The yellow to red space is the domain of TL dating on burnt flints and of U-series dating. The three columns correspond to the main different environments in which archaeological sites are found in Western Europe. In comparison with other methods, the ESR/U-series method applied on teeth has the advantage to be potentially applied on the three domains. For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.

This paper focuses on the period between 300 and 600 ka in Western Europe, and particularly in northern Spain, in Italy, and in France, which is particularly interesting for the arrival of the Acheulian culture, probably in association with a settlement phase from Africa or the Middle East (Goren-Inbar et al., 2000). During the Lower Pleistocene, the oldest European archaeological evidences are restricted to the southern part of Europe (Guadix-Baza Basin, Sierra de Atapuerca, Spain; Ceprano, Italy) and their lithic assemblages do not contain bifaces. On the other hand, the oldest Acheulian sites, dated around 600–700 ka, have been found at a latitude north of 45° in Europe, like Pakefield in Great Britain (Parfitt et al., 2005, Roebroeks, 2005) and La Noira in Central France (Despriée et al., 2009, Despriée et al., 2010). During the same period, the Acheulian culture seems to have been absent or just emerging in the southern part of the continent. Hence, at Isernia la Pineta, a locality in southern Italy dated to 600 ka by $^{39}\text{Ar}/^{40}\text{Ar}$ method (Coltorti et al., 2005), no bifaces were found among the very abundant lithic assemblage (Peretto, 2006) while in the same region, at Venosa, several localities dated to between 500 and 600 ka display Acheulian assemblages (Piperno et al., 1998). In northern France, the probable migration route between the European continent and the British Isles, the oldest Acheulian localities, such as Cagny-la-Garenne in the Somme Valley or La-Celle-sur-Seine, in the Seine Valley, were dated to around 450 ka and earlier, Acheulian evidence is scarce and poor (see Antoine et al., 2010). Thus, the establishment of a reliable chronology is crucial to understand the timing and dynamics of the Acheulian settlements of Europe.

For the period between 300 and 600 ka, it is possible to obtain U-series dates on speleothems which are in stratigraphical relation with the human remains and Acheulian levels. At Arago Cave, France, a set of more than 50 U-series dates on a stalagmitic floor located stratigraphically above the Acheulian palaeoanthropological layers suggests that the human remains unearthed from these levels and associated lithic industry are older than 350 ka (Falguères et al., 2004). Mass spectrometric U-series dating can provide age results with much higher precision. For example, at the Acheulian site of Orgnac 3, France, a stalagmitic formation was dated by Thermal Ionization Mass Spectrometry (TIMS) to a maximum age of 318 ± 9 ka for the upper human levels (Ayliffe, personal communication). This result confirms those previously obtained by ESR and U-series on the same calcitic level (Falguères et al., 1988). However, the use of these methods is restricted to karst sites and to travertine localities such as La-Celle-sur-Seine, where the Acheulian layer is overlain by a thick carbonate tufa dated by TIMS to around 390 ka (Limondin-Lozouet et al., 2006, Limondin-Lozouet et al., 2010).

Hence, a major potential of the combined ESR–US method is to provide an alternative way to establish a reliable chronological framework of the human settlement in non-volcanic contexts. Moreover, as shown by the samples presented in this paper, this method also yields chronological information for the uranium uptake in teeth and geochemical behaviour of uranium in dental tissues, and thus on the taphonomy of the localities.

2. Methods

One of the major difficulties in dating large herbivorous teeth is linked to the complexity of the system which has to be considered. A mammal tooth consists of several tissues (enamel, dentine and cement), which have different chemical compositions (see Hillson, 2005) and sensitivities to diagenetic processes (Piepenbrink, 1989, Kohn et al., 1999, Dauphin and Williams, 2004), inducing differential U-incorporations. Fossil bones and teeth usually behave as open systems, increasing the difficulty to get reliable ages. But because of fossilization and taphonomic conditions according to the nature of the site, cave or open-air locality, the degree of “opening”, in terms of uranium uptake, can be very different from one tissue to another from the same tooth, or from one site to another (Grün, 2009).

The combined ESR–US model allows an estimation of the history of uranium uptake in each dental tissue using a one parameter (p -value) diffusion equation. This approach is different to the conventional methods where the mode of uranium uptake is assumed, such as the early uptake (EU) model which postulates that uranium was incorporated shortly after the sample was buried (Bischoff and Rosenbauer, 1981), and the linear uptake (LU) model where uranium is supposed to have been incorporated at a constant rate since the time of burial (Ikeya, 1982). It was shown by Grün (2009) that there are many instances where neither model is appropriate, and that any assumptions about U-uptake may lead to extreme errors.

In Grün et al. (1988), the uranium content of each dental tissue is then given by the following general equation:

$$U(t) = U_m(t/T)_{p+1} \quad (1)$$

where $U(t) = U$ concentration at time t , U_m = measured U concentration, and T = age. Using the isotopic ratios of U-series and the D_E values, one can calculate the evolution of the internal dose rate. The definition range is limited to p -values greater than or equal to -1 , i.e. to samples exhibiting an ESR–EU age higher than one obtained by the corresponding U-series (Fig. 2).

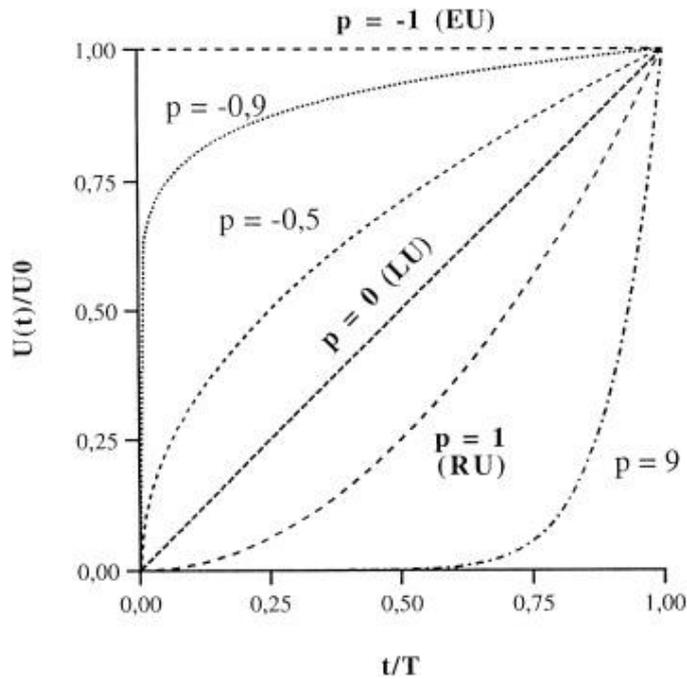


Fig. 2. Curves for uranium uptake as defined by general Eq. (1) for some particular p -values (from Grün et al., 1988).

When the p -values fall in the range between -1 (EU model) and 0 (LU model), the age may be considered as acceptable and representative of the “real” age, even if the sample acquired uranium after burial. This correction procedure describes more accurately the geochemical history of the tooth (Grün et al., 1988, Bahain et al., 1992, Grün and McDermott, 1994, Falguères et al., 1999). However, the large age difference between EU and LU models which can be sometimes observed for the same sample (Grün, 2000) allows for only an approximate chronological estimation of archaeological events. Another case corresponds to a recent uranium uptake by teeth (p -values > 0). The Recent Uptake (RU) model, which assumes a p -value = 1 was proposed by Blackwell et al. (1992). Finally, it happens sometimes that U-series ages are older than the EU–ESR ones. In these cases, p -values cannot be calculated because data are beyond the definition range of the equation. However, in order to try to get an age determination, p -values are set to -1 for such tissues.

The main particularity of the ESR–US model is that a unique p -value for each dental tissue is sought for which the dates of the U-series and ESR agree.

In order to calculate an ESR–US age, several parameters must be assessed (see details in Falguères et al., 1999). $^{230}\text{Th}/^{234}\text{U}$ and $^{234}\text{U}/^{238}\text{U}$ isotopic ratios and U content of each dental tissue are measured by U-series alpha spectrometry. The equivalent dose (D_E) is determined for enamel from ESR measurements using the additive dose method. The dose rate is calculated from radioactivity measurements of the surroundings and of the sample itself, using U-series data, additional gamma-ray measurements (*in situ* and in laboratory) and cosmic dose rate evaluation. The combination of all data allows the calculation of a single age as well as p -values for each dental tissue. The p -values give insights into the U-uptake history.

3. Results and discussion

3.1. Ambrona site, Spain

Once uranium has been incorporated after the burial, it is generally possible to get ages even when the uptake is very recent. A good example is represented by the Acheulian site of Ambrona, Spain (Falguères et al., 2006) where the oldest levels of the archaeological sequence provided samples with apparent Holocene U-series ages for each tissue, in complete disagreement with the typical Acheulian industry, the Middle Pleistocene faunal record and the relatively high D_E values. Fig. 3 shows that the isotopic ratios of these samples are in the domain of reliability (Zone I) delimited between the oblique and the left part of the horizontal lines representing the secular equilibrium and where U-series are characterized by $^{234}\text{U}/^{238}\text{U} > 1$ and $^{230}\text{Th}/^{234}\text{U} < 1$, a precondition to consider U-uptake as a continuous process in fossil teeth. The right part of the diagram (Zones II and III) indicates much more complex processes where $^{234}\text{U}/^{238}\text{U} > 1$ and $^{230}\text{Th}/^{234}\text{U} > 1$ suggesting that U-uptake has stopped a long time ago or that U-leaching had occurred sometime in the past (see details in Osmond and Ivanovich, 1992, Chabaux et al., 2003). Data in Zone IV indicate uranium leaching. For the stratigraphically lowest samples (AS1 and AS2), the extreme left location in the diagram suggests very recent uranium uptake. The ESR-US ages are close to 300 ka with p -values ranging between 16 and 40. These high p -values suggest a very recent uranium uptake for each tooth (and each tissue) and the ESR-US calculated age represents a minimum age for the base of the sequence. On the other hand, for the samples recovered from the upper part of the stratigraphy, p -values ranged between -0.53 and -0.86 which indicate U-uptake between the classical EU and LU models and the ESR-US results yield a minimum age of 340 ka (mean value of AS6 stratum) for the upper part of the Lower complex of Ambrona. For these samples, cement had $^{230}\text{Th}/^{234}\text{U}$ values significantly greater than unity, indicating U-leaching. Combining all data for the site, for the lower layers of Ambrona, the ESR-US model indicated a minimum age of 350 ka. This study has emphasized the limitation of the ESR-US model which can provide only informative ages when p -values are largely greater than 1 (Falguères et al., 2007).

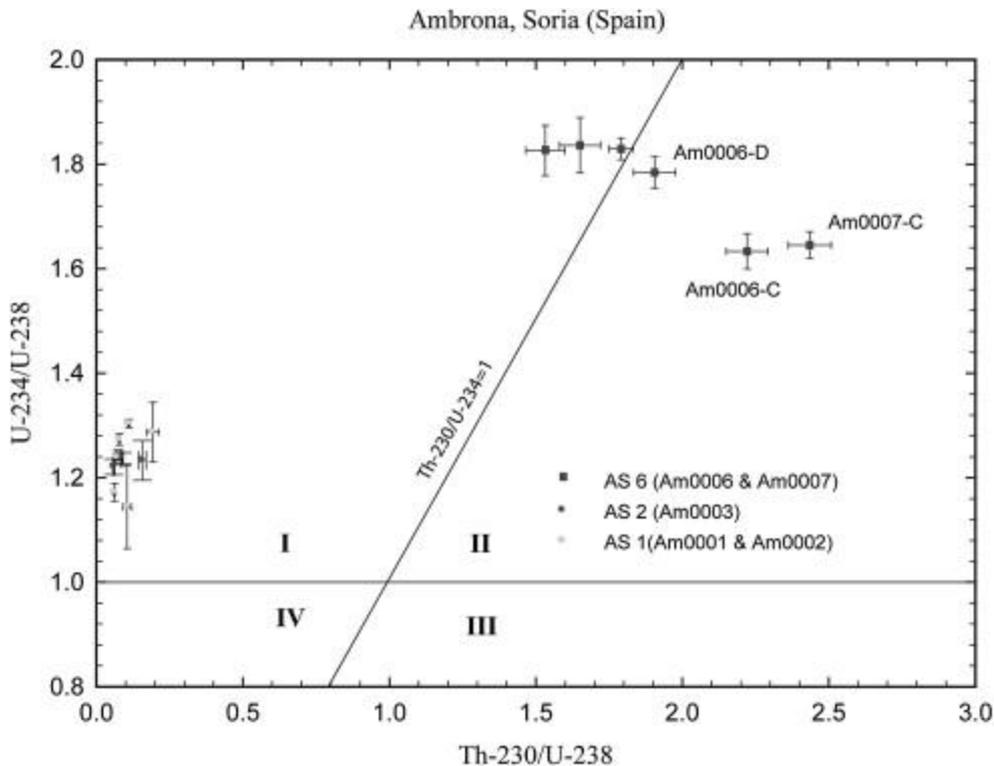


Fig. 3. Theoretical evolution of the ^{238}U - ^{234}U - ^{230}Th disequilibria in the $(^{234}\text{U}/^{238}\text{U})$ versus $(^{230}\text{Th}/^{238}\text{U})$ activity ratio diagram for each dental tissue from Ambrona samples. The diagonal dotted line represents equilibrium between ^{234}U and ^{230}Th . The diagram can be divided into four zones I to IV

(modified from Chabaux et al., 2003): the U-uptake zone (Zone I) with $(^{234}\text{U}/^{238}\text{U}) > 1$ and $(^{230}\text{Th}/^{234}\text{U}) < 1$, the U-leaching zone (Zone IV) where $(^{234}\text{U}/^{238}\text{U}) < 1$ and $(^{230}\text{Th}/^{234}\text{U}) < 1$, and the two other areas (Zones II and III) called complex zones. The Zone I can be interpreted as having the highest reliability for the ESR–US combined method. One can observe that the isotopic data of samples from the lower stratigraphical levels AS1 and AS2 are located in the extreme left part of the uptake area (Zone I) and close to the line where $(^{234}\text{U}/^{238}\text{U}) = 1$ which seems characteristic of a recent uptake. For the youngest samples from AS6 level, the majority of the tissues are close to the equilibrium line, indicating thus an older U-uptake. But it is also important to note that the cement of the two teeth is located in the so-called complex domain, which suggests that the U-mobility in these tissues cannot be interpreted as a single U-uptake or U-leaching, but more complex scenarios have probably occurred.

3.2. Visogliano site, Italy

Visogliano, Italy, is a locality in which human remains were found associated with an Acheulian industry (Tozzi, 1994). Herbivorous teeth from the lowest layers (units 38–44), coeval with the human remains, were dated by the ESR–US model (Falguères et al., 2008). All p -values ranged between -0.26 and -0.78 providing ESR–US ages between 440 and 480 ka for the level 44. Except for the dentine of VI9802 and VI9808, Fig. 4 shows that the isotopic ratios fall in Zone I, in which the ESR–US model could work well. All data suggest that these human bearing levels are contemporaneous with Level G of Arago Cave, in which more than 100 human remains were unearthed with Acheulian artefacts.

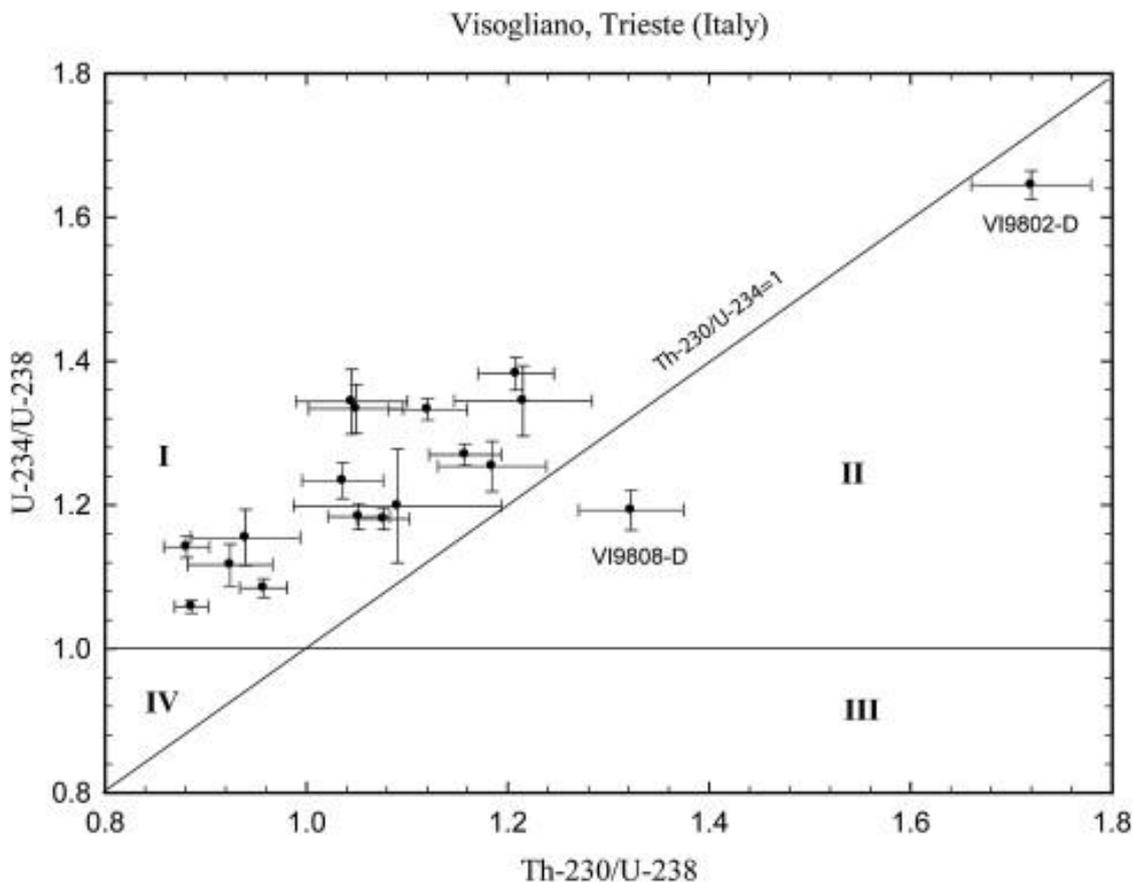


Fig. 4. Theoretical evolution of the ^{238}U – ^{234}U – ^{230}Th disequilibria in the $(^{234}\text{U}/^{238}\text{U})$ versus $(^{230}\text{Th}/^{238}\text{U})$ activity ratio diagram for each dental tissue from Visogliano samples. All the samples are in the domain of reliability (Zone I), close to the equiline, indicating thus a U-uptake process which is not recent (i.e. p parameter which tend towards -1), except two of them which are located in the complex zone.

3.3. Arago Cave, France

Arago Cave's Level G consists of a 30–65 cm thick bone accumulation which has been affected by drastic geochemical alteration (Lumley et al., 1981). In addition, the heterogeneity of the bones' accumulation renders the dose rate assessment for Level G very difficult. This problem emphasizes that dosimetry measurements often represent the most difficult part for obtaining radiometric ages. In Level G, three areas with different diagenetic alterations were observed (non-altered, phosphated and carbonated). Teeth extracted from the carbonated part yielded ages ranging between 450 and 490 ka with p -values between 0 and -1 . Paradoxically, they fit better with palaeontological data and previous radiometric ages than those obtained from the non-altered zone, indicating that the taphonomy of this area is likely more complex than expected particularly with respect to the understanding U-mobilization in the sediments (see Han et al., in press).

3.4. La Micoque site, France

Lastly, when uranium is leached in the teeth, the ages are not directly calculable. In order to get an estimation age, the p -values were set at their minimum ($p = -1$) allowing a calculation. Duval (2008) observed that a correlation exists between the ESR–US ages and the number of blocked p -values ($p = -1$) for Lower Pleistocene teeth: the higher is the number of blocked parameters, the lower is the calculated ESR–US age. This observation was verified for samples from the top of the E Layer of Acheulian French site of la Micoque (Falguères et al., 1997). An ESR–US age calculated for one sample with one blocked p -value (enamel) is older than the ESR–US age of the second sample for which two p -values (enamel and dentine) were blocked. When all p -values in a tooth are blocked, the ESR–US age corresponds to the ESR–EU age. This age can therefore be considered as a minimum one, because the dose rate associated to the dental tissues is the maximum value calculable according to the ESR–US model. In such case, the EU-value is the only that can be obtained.

4. Conclusions

The ESR–US dating is the only method which can be applied to open systems such as bones and teeth, which are often the only available samples in open air sites older than 400 ka. It offers new opportunities for dating sites which cannot be dated by other methods, as in the case of karst areas of Western Europe. Fig. 5 suggests that the Acheulian sites which have a similar latitude (between 40 and 45°N) are coeval within a period ranging between 300 and 500 ka, while the oldest Acheulian sites are located north of 45°N.



Fig. 5. Map of the different dated Acheulian sites between 40 and 45° latitude. Atapuerca Galeria and Sima de los Huesos are added in the figure according to the luminescence chronology and to the U-series dates published by Berger et al. (2008) and by Bischoff et al. (2007), respectively.

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