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Three archaeomagnetic applications of archaeological interest to the study of burnt anthropogenic cave sediments

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ABSTRACT

Recent archaeomagnetic studies carried out on Mid-to Late Holocene burnt anthropogenic cave sediments have shown that under certain conditions, these materials are suitable geomagnetic field recorders. Archaeomagnetic analyses carried out on these contexts constitute a rich source of information not only for geophysical purposes –in terms of reconstructing the variation of Earth's magnetic field in the past– but also from the archaeological point of view, for example by archaeomagnetic dating. Here, we report three different archaeomagnetic applications to the study of burnt cave sediments: (i) archaeomagnetic dating; (ii) determining palaeotemperatures and (iii) assessing post-depositional processes. The first case study is a dating attempt carried out on a Late Holocene (Bronze Age) burnt level from El Mirador Cave (Burgos, Spain). Using the directional European secular variation curve, several dating intervals were obtained for the last burning of this combustion feature. Considering the archaeological evidence and the independent radiometric (¹⁴C) dating available the possible ages obtained are discussed. This is the first archaeomagnetic dating obtained in these contexts so far. The second case study is an application of the method to determine the last heating temperatures reached by the carbonaceous facies of these fires. Stepwise thermal demagnetization of oriented samples can be used to quantitatively estimate heating temperatures. An intermediate normal polarity component interpreted as a partial thermo-remance (pTRM) with maximum unblocking temperatures of 400–450 °C was systematically identified, revealing the last heating temperatures experienced by this facies. These temperatures were confirmed with partial thermomagnetic curve experiments. Finally, archaeomagnetic analyses on a partially bioturbated burning event were performed in order to evaluate until what spatial extent the burnt sediments were affected by post-depositional mechanical alteration processes. For each case study, the archaeological implications are discussed highlighting the potential of archaeomagnetic methods to retrieve archaeological information.

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1. Introduction

Since the pioneering work of Brochier (1983a,b), the study of Holocene burnt anthropogenic cave sediments has experienced considerable progress. A great number of archaeological excavations as well as the increasing amount of data provided by disciplines such as soil micromorphology (Boschian, 1997; Macphail et al., 1997; Angelucci et al., 2009), palaeobotany (Rasmussen, 1993; Delhon et al., 2008; Cabanes et al., 2009) or

zoarchaeology (Rowley-Conwy, 1998; Martín et al., 2014) among others, is yielding valuable information about the formation and use of these deposits. Archaeomagnetism has emerged as one of these lines of research. Although it has a long tradition in Earth sciences its application in prehistoric archaeology is still sporadic and its potential to retrieve archaeological information remains underutilized.

Broadly speaking, archaeomagnetism deals with the study of the record of the Earth's magnetic field direction and/or intensity changes in the past in burnt archaeological materials. Most archaeological materials contain small amounts of ferromagnetic minerals (s.l.), such as magnetite or haematite. When heated to high temperatures (>500–600 °C) and subsequently cooled these

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minerals acquire a remanent (permanent) magnetization parallel to the ambient magnetic field. Under several conditions this information may be very stable over long periods of time and used in a wide variety of applications, among which dating is likely the most known. However, given their versatility, magnetic methods can provide valuable information ranging from determining palaeotemperatures (e.g., Brown et al., 2009), ash sourcing (Church et al., 2007) or assessing the degree of preservation in archaeological cave fires (e.g., Carrancho et al., 2012). This paper provides a review of some of these applications specifically applied to anthropogenic cave sequences.

These stratigraphic sequences usually contain multiple burning events generated by the periodic burning of organic material (e.g., vegetal remains and dung) produced by livestock penning (Angelucci et al., 2009). Their preservation state is usually good, are generally well-dated by independent methods (namely radiocarbon) and have a broad geographical distribution throughout the Mediterranean region (Angelucci et al., 2009). Therefore they constitute a great source of archaeomagnetic data and the information obtained has both geophysical and archaeological interest. The main goal of this article is to highlight the potential of magnetic methods to answer archaeological questions through three different applications. The first is a dating attempt of a firing event from El Mirador Cave (Spain) using the recently designed directional European Secular Variation (SV) curve for the Neolithic (Carrancho et al., 2013). The second is a methodological application to determine the last heating temperature undergone by these fires. The third consists on evaluating to what extent a burning event might be affected by post-depositional processes. The archaeological and archaeomagnetic implications of these cases studies will be discussed as well as the limits of each application.

2. Materials and methods

2.1. Sites

The studied materials correspond to samples from Neolithic, Chalcolithic and Bronze Age burning events exposed in the Holocene stratigraphies of El Mirador and Portalón de Cueva Mayor caves (Sierra de Atapuerca, Burgos) and El Mirón Cave (Cantabria, Spain; Fig. 1a). For detailed information on the archaeology, stratigraphy and chronology of these sites the reader is referred to Straus and González Morales (2012), Carretero et al. (2008) and Vergès et al. (2008, in this volume). These fires generally contain a

grey/white ash facies of variable thickness (2–10 cm) over a thin (~2 cm) black carbonaceous subjacent facies.

2.2. Sampling

Archaeomagnetic sampling was carried out with the aid of a non-ferromagnetic cylindrical tube which incorporates a built-in orientation system specifically designed for soft (unlithified) lithologies (Carrancho et al., 2013). Its main advantage is that it allows a precise geographical orientation of the samples besides being minimally invasive. The tube is pressed against vertical profiles where the burnt facies outcrop. After the azimuthal reading, the sediment is carefully inserted in cylindrical plastic boxes (Ø 16.5 mm, 17 mm length; volume of about 3.6 cm³) and stored in cold conditions (3–4 °C) until measurement to avoid chemical alterations. Samples for thermal (TH) demagnetization of the natural remanent magnetization (NRM) were oriented by the same means and introduced into home-made plaster cubes (Carrancho, 2010). These contain a cylindrical hole with the same dimensions and volume as the plastic capsules in order to keep the sample in fixed position. The NRM of the plaster cubes is at least two orders of magnitude less than the sample's magnetization. Details of the number and type of samples collected for each case study are given below.

2.2.1. Case study 1 (archaeomagnetic dating)

A burning event (Ci1) from El Mirador Cave (42° 20' 58" N, 03° 30' 33" W; Sierra de Atapuerca, Burgos, Spain) was intensively sampled for archaeomagnetic dating purposes (Fig. 1a–b). The archaeostratigraphic unit where Ci1 is located (MIR103 – Sector 100) has a ¹⁴C (AMS) dating (sample code: Beta – 339094) obtained from a charcoal fragment with a 2σ dating interval of 1510 to 1410 cal. BC (3190 ± 30 BP). Archaeological evidence is limited to few pottery remains suggesting a possible Bronze Age for the MIR103 unit. The objective here was to obtain an archaeomagnetic date of the last heating of this event using the directional European SV curve (Carrancho et al., 2013). The Ci1 burning event is composed of an ash and a carbonaceous facies. The ashes are white on top and reddish brown on the bottom with a total thickness of about 15 cm. Just beneath, a dark carbonaceous (~2 cm) facies is preserved delimiting the surface where burning occurred (Fig. 1). At the top of the lower level, just at the base of the burning event, a burrow can be observed that may have partially affected the structure. A total of 29 oriented samples (22 ashes and 7

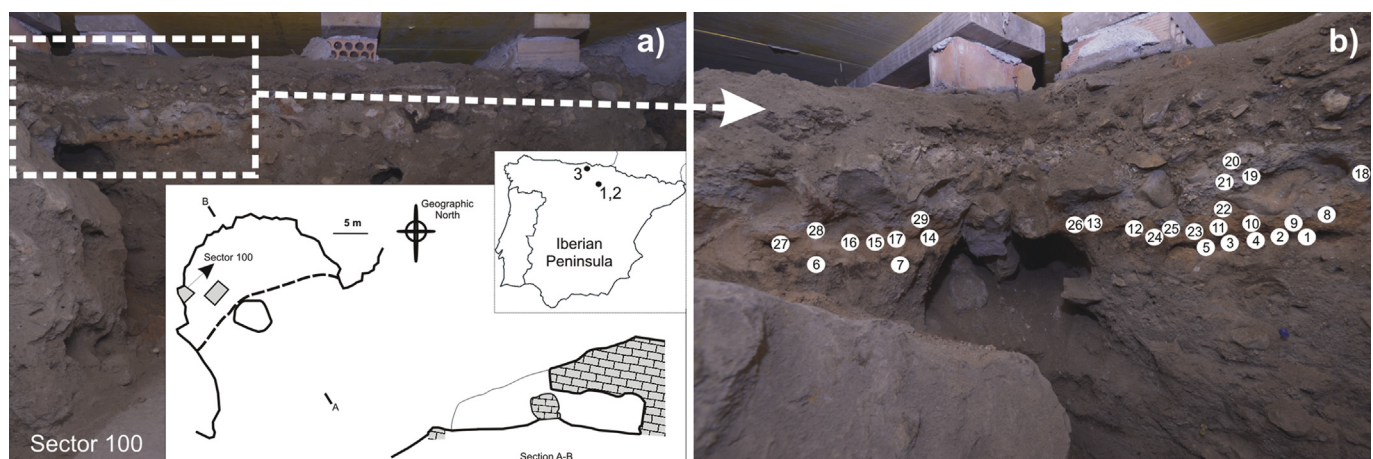


Fig. 1. (a–b) Photographs showing the studied burning event with the location of the samples. The plan and section view of Mirador Cave showing the survey pit where Ci1 is located (sector 100). The location of the three caves studied is shown in the map: 1, 2 (El Mirador and Portalón Caves, Sierra de Atapuerca, Burgos) and 3 (El Mirón Cave, Cantabria).

carbonaceous samples) were collected following the sampling procedure described in Section 2.2.

2.2.2. Case study 2 (estimating palaeotemperatures)

The samples analysed in this case study are representative carbonaceous samples from 6 different Holocene burning events from El Mirador, El Portalón and El Mirón Cave (Spain). They were previously studied along with hundreds of burnt samples in the design of the first directional European PSV curve for the Neolithic (Carrancho et al., 2013). The objective is to show how the identification of partial thermal remanent magnetizations (pTRMs) permits the quantitative estimation of the last heating temperature in the carbonaceous facies. The validity of this approach was verified carrying out thermomagnetic curve analyses on bulk (unoriented) sample from this facies and studying their degree of reversibility (Section 4.2). The sampling procedure was the same as described in Section 2.2.

2.2.3. Case study 3 (assessing post-depositional processes)

In order to test the reliability of the palaeomagnetic method to determine to what extent the mechanical reworking might have affected an anthropic cave fire, an archaeomagnetic study of a Late Holocene burning event from El Portalón Cave (Burgos, Spain; map of Fig. 1a) is reported. This burning event contains a white ash facies (~10 cm) over a ~2 cm dark carbonaceous facies both partially altered by an ancient burrow (Fig. 8). The colour and texture of ashes on the right side of the burning event are somewhat mixed, suggesting that some kind of mechanical reorganization might have occurred. In contrast, the ashes of the central and left part are pure white ashes seemingly *in situ*. This event was intensively sampled collecting 24 oriented samples of both facies (18 ashes and 6 carbonaceous samples). The archaeomagnetic mean direction obtained has been reported by Carrancho et al. (2013). Nevertheless, the objective here is to describe what magnetic features display *in situ* samples compared to those that are reworked. These results will allow testing the criteria established in a similar case study (Carrancho et al., 2012) as well as evaluating the degree of alteration that the structure might have suffered.

2.3. Laboratory methods

All analyses were performed in the laboratory of palaeomagnetism of Burgos University (Spain). The measurement of the natural remanent magnetization (NRM) was carried out with a 2G SQUID magnetometer (noise level 5×10^{-12} Am²). Low-field

magnetic susceptibility at room temperature was measured with a KLY-4 susceptometer (AGICO, noise level 3×10^{-8} S.I.). The NRM directional stability was analysed by stepwise progressive alternating field (AF) and thermal (TH) demagnetization. AF demagnetization was carried out in 18–20 steps up to maximum fields of 100–120 mT with the 2G magnetometer AF demagnetization unit. TH demagnetization was performed using a TD48-SC (ASC) thermal demagnetizer in 15–17 steps up to 660 °C. The Characteristic remanent magnetization (ChRM) direction of every specimen was determined by principle component analysis (PCA; Kirschvink, 1980) including at least four demagnetization steps (usually five or more).

In order to study further the ferromagnetic mineralogy present, different rock-magnetic experiments were carried out with a variable field translation balance (MM_VFTB). These comprised progressive isothermal remanent magnetization (IRM) acquisition curves, hysteresis loops (± 1 T), backfield curves and thermomagnetic curves up to 700 °C in air. These analyses were undertaken on representative bulk sample (~400 mg) both on ash and carbonaceous samples. Curie temperatures of Js-T curves were determined using the two-tangent method of Grommé et al. (1969). Saturation magnetization (M_s), remanence saturation magnetisation (M_{rs}) and coercive field (B_c) were calculated from hysteresis loops after subtracting the paramagnetic contribution. In combination with the coercivity of remanence (B_{cr}) determined from the backfield curves, the domain state distribution was analysed in the Day diagram (Day et al., 1977; Dunlop, 2002).

3. Case 1: archaeomagnetic dating

3.1. Background

Archaeomagnetic dating is based on two fundamental phenomena. First, the ability of ferromagnetic minerals (*s.l.*) to acquire a remanent magnetization when heated and subsequently cooled from high temperatures parallel with and proportional to the geomagnetic field. This mechanism of magnetization is known as thermoremanent magnetization or TRM and is characteristic of structures such as ovens, kilns and hearths. Second, the Earth's magnetic field undergoes subtle variations in direction and intensity on a timescale of 10^2 – 10^3 years on a regional scale. These fluctuations are known as secular variation (SV) and are reproducible for regions no bigger than 500–600 km of radius (Lanos, 2004). Over recent years great efforts have been undertaken to derive regional SV curves for different regions, particularly in Europe. These master curves are composed of directional and/or intensity data of the Earth's magnetic field obtained from previously well-dated burnt archaeological materials (and occasionally also from lava flows). With some exceptions in Eastern Europe (Tema and Kondopolou, 2011; Kovacheva et al., 2014), most European SV curves cover the last 2–3 millennia (Gallet et al., 2002; Schnepf and Lanos, 2005, 2006; Gómez-Paccard et al., 2006; Marton and Ferencz, 2006; Tema et al., 2006; Zananiri et al., 2007).

Standard archaeomagnetic dating works on the basis of comparing the mean direction and/or intensity determined from a site with the SV curve available for the region and period concerned. Many archaeomagnetic dating examples are reported in the literature using directional, intensity data or both combined (e.g., Casas et al., 2007; Ech-Chakrouni et al., 2013). The more archaeomagnetic data added to these regional SV curves the better defined they will be, thus improving the dating technique. More recently, archaeomagnetic dating using geomagnetic field models has become feasible. For instance, the SCHA.DIF.3K European regional model (Pavón-Carrasco et al., 2009) based exclusively on archaeomagnetic directional and intensity data for the last 3

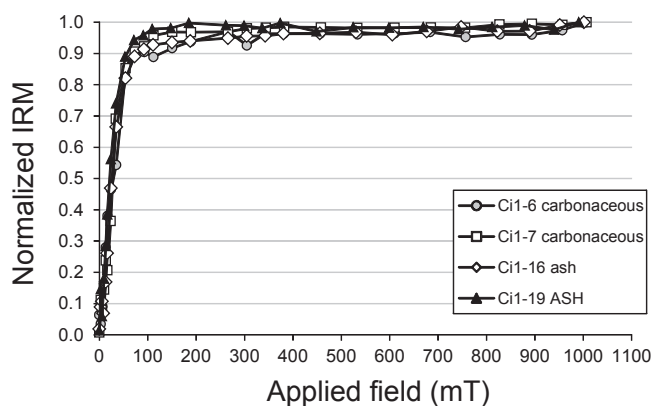


Fig. 2. Four normalized progressive isothermal remanent magnetization (IRM) acquisition curves of representative ash and carbonaceous samples from the Ci1 burning event. Maximum field = 1 T.

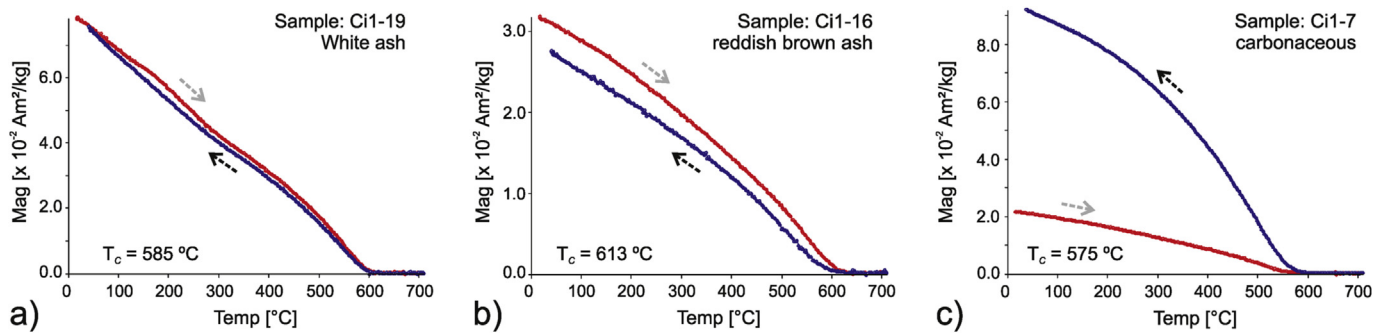


Fig. 3. (a–c) Representative thermomagnetic curves (magnetization vs. temperature) of two ashes and a carbonaceous sample from Ci1 burning event (El Mirador Cave). Heating (cooling) cycles are plotted in red (blue) with their respective arrows. Sample code, facies and magnetization intensity values and the T_c are indicated. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

millennia, directly predicts the geomagnetic field at the site of interest even for regions where no SV curve is available. This avoids any eventual relocation error which has been proved to introduce significant errors (Casas and Incoronato, 2007). There are also global models for longer periods (e.g., Korte and Constable, 2005; Pavón-Carrasco et al., 2010; Korte et al., 2011) but not suited for

archaeomagnetic dating because they include sedimentary data that smooth the geomagnetic field variations through time. Also, new software has been developed to carry out archaeomagnetic dating using various SV models (Pavón-Carrasco et al., 2011).

Archaeomagnetic dating has a typical range of error of a few centuries although there are good examples reaching dating

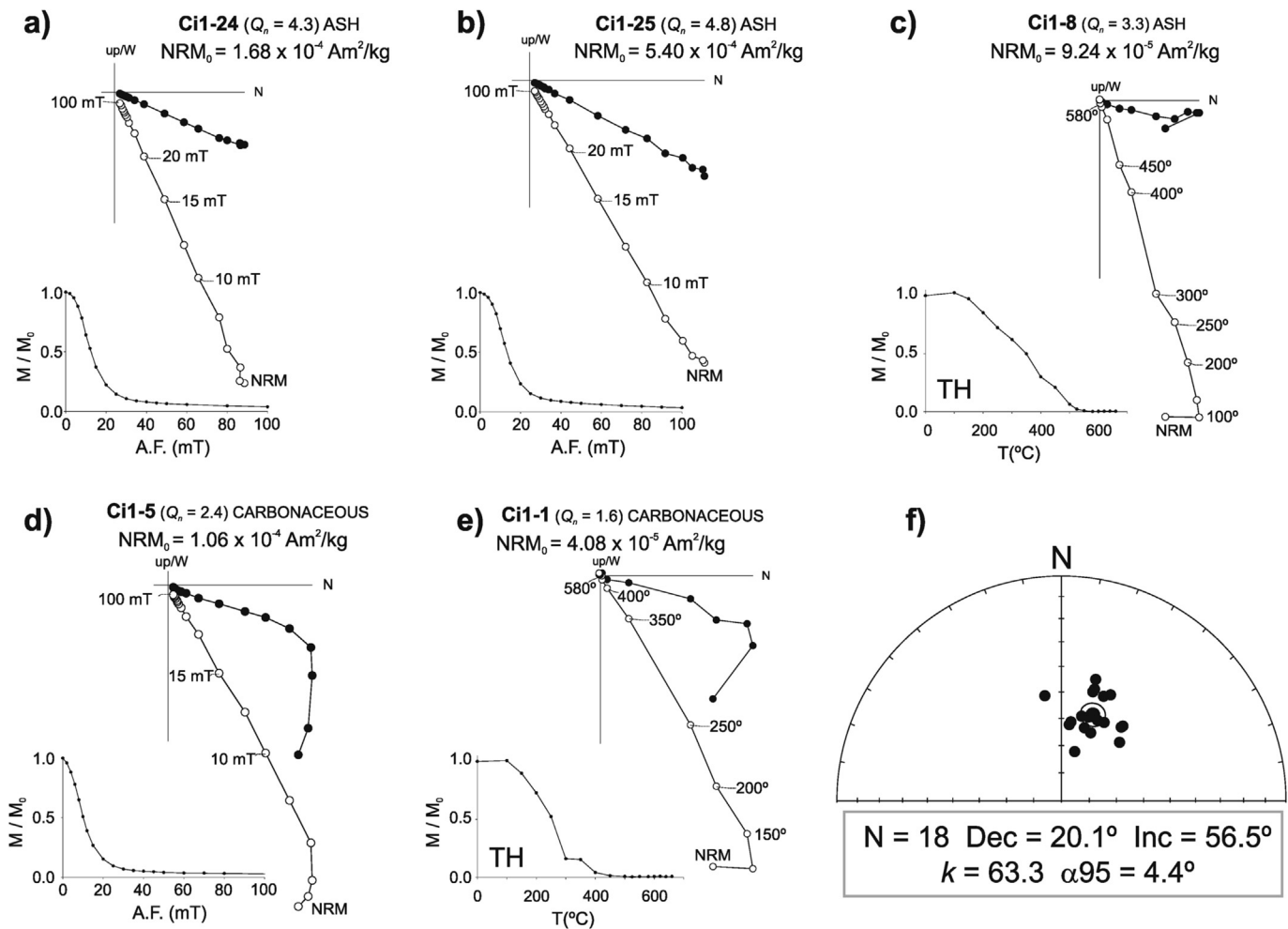


Fig. 4. (a–f). Representative orthogonal NRM demagnetization plots from the Ci1 burning event. Solid (open) circles show projections of vector endpoints onto the horizontal (vertical) plane. The sample code, facies, intensity (NRM_0), Koenigsberger (Q_n) ratio and normalized demagnetization spectra are shown for each sample. AF = alternating field; TH = thermal. (f) Equal area projection of all ChRM directions with the mean direction and α_{95} confidence circle. N = number of samples; Dec = declination; Inc = inclination; k = precision parameter and α_{95} = semi angle of confidence.

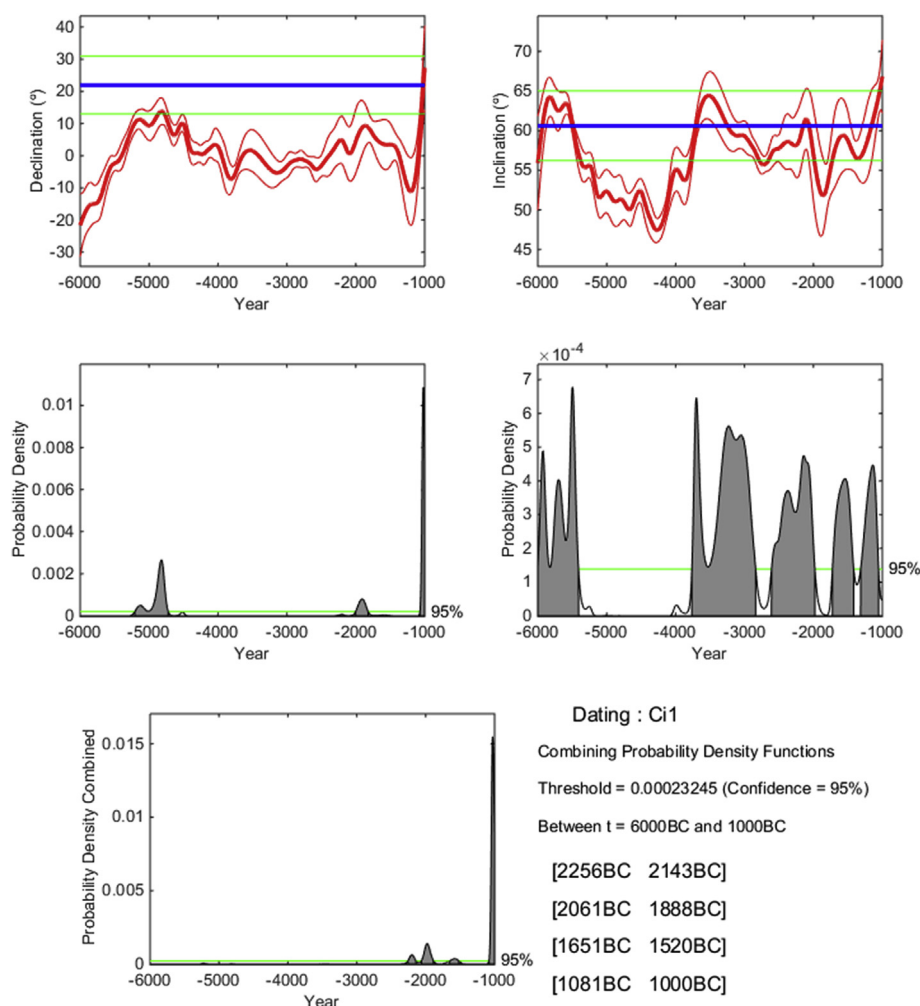


Fig. 5. Probability-of-age density functions (95% of confidence) obtained for the C11 burning event with the Matlab tool from Pavón-Carrasco et al. (2011) for declination and inclination values using the European directional secular variation curve (Carrancho et al., 2013).

resolution of a few tens of years as the one reported from an early 18th century brick kiln by Casas et al. (2007). This depends on several factors such as sampling or analytical errors, inconsistent behaviour of the material or the rate of variation of the Earth's magnetic field. However, dating applicability of the method depends on the length and completeness of the SV curve for the region concerned. The longest and systematic archaeomagnetic records for the last 8 ky exist for Eastern Europe (Tema and Kondopolou, 2011; Kovacheva et al., 2014) but that is not the case for Western Europe as mentioned before. Current efforts aim to temporally and geographically extend SV records using well dated, *in situ* archaeomagnetic materials.

Recent studies carried out on Mid to Late Holocene burnt anthropogenic cave sediments from the Iberian Peninsula (Carrancho et al., 2009, 2012, 2013) and Central Europe (Kapper et al., 2014a, b) have allowed the extension to mid-Holocene times of the archaeomagnetic database and the dating technique. These authors showed how under certain conditions reliable archaeomagnetic directions can be obtained from these materials. As multiple burning events are usually present in these archaeological sequences, various archaeomagnetic data (spanning a time period in the range of hundreds to thousands of years) can be obtained from a single site. Combining 26 new directions obtained from Neolithic, Chalcolithic and Bronze Age burnt levels from three caves in Spain with the existing archaeomagnetic

database for Eastern Europe (Kovacheva et al., 2009; Korte et al., 2011), a directional European SV curve for the Neolithic exclusively based on archaeomagnetic (TRM) data was published (Carrancho et al., 2013). Although new results are being reported (e.g., Hervé et al., 2013a,b), archaeomagnetic data for times prior to around 1000 BC in Western Europe are rather scarce. Burnt anthropogenic cave sediments emerge thus as a new geomagnetic field recorder with a great potential both for geophysical and archaeological purposes.

3.2. Results and discussion

3.2.1. Magnetic properties

Natural remanent magnetization values are between 4.08×10^{-5} and $8.27 \times 10^{-4} \text{ Am}^2\text{kg}^{-1}$ whereas low-field magnetic susceptibility values oscillate between 6.42×10^{-7} and $4.78 \times 10^{-6} \text{ m}^3\text{kg}^{-1}$. The highest values for both parameters correspond to the ashes indicating a major concentration of ferromagnetic minerals in this facies. The Koenigsberger ratio ($[Qn = \text{NRM}/(\chi H)]$ (cf. Stacey, 1967)) where χ is the magnetic susceptibility and H is the local geomagnetic field strength, yielded values between 1.6 and 19.6. These values agree well with others reported for similar materials (Carrancho et al., 2009, 2012; Kapper et al., 2014a,b) and indicates that the NRM is of thermal origin.

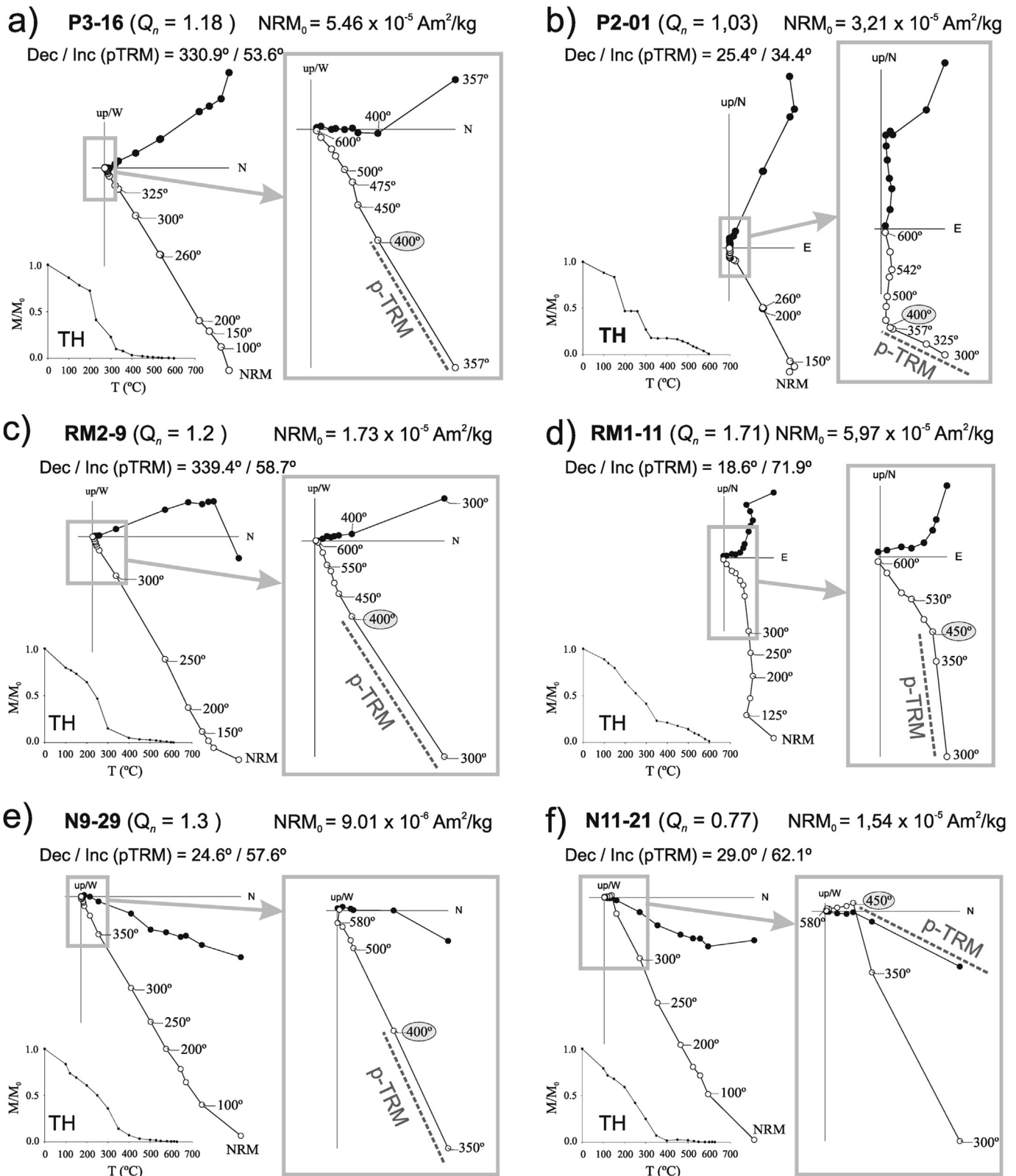


Fig. 6. (a–f) Orthogonal NRM demagnetisation plots of representative carbonaceous samples from different burning episodes of (a–b) El Portalón cave (c–d) El Mirón cave and (e–f) El Mirador Cave. Symbols are as in Fig. 4. The final steps of the diagrams are blown up to denote the presence of a high-temperature component. The maximum unblocking temperatures (max T_{UB}) of the partial thermoremanent magnetization (pTRM) are within grey ellipses indicating the heating temperatures. Dec (declination) and Inc (inclination) of the pTRM component are shown for each diagram.

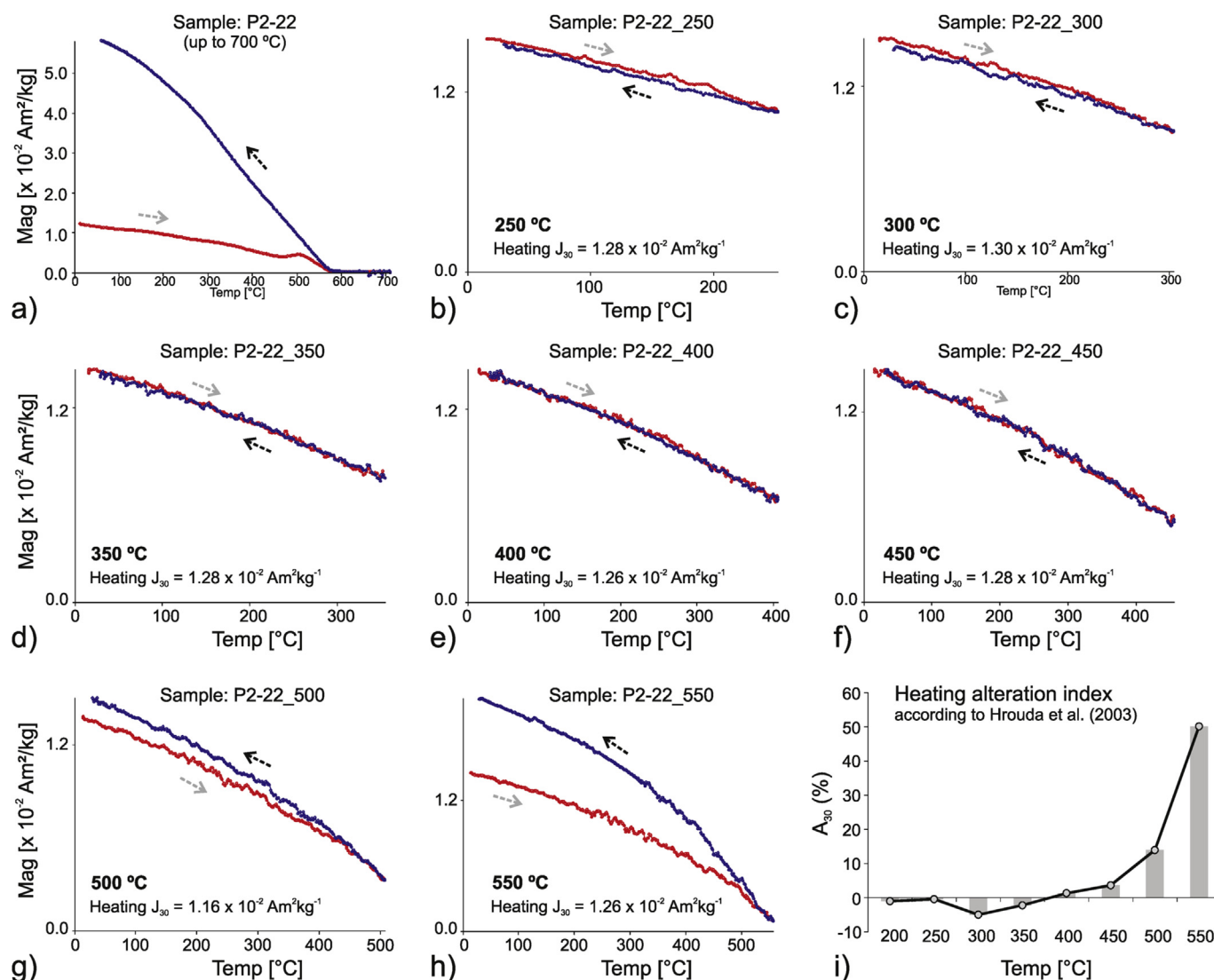


Fig. 7. (a) High-temperature thermomagnetic curve up to 700 $^{\circ}\text{C}$ of the carbonaceous sample P2-22 (sister sample of P2-01; Fig. 5b) from El Portalón Cave. (b–h) Progressive thermomagnetic curves in 50 $^{\circ}\text{C}$ incremental steps from 200 $^{\circ}\text{C}$ to 550 $^{\circ}\text{C}$ carried out on additional sample from P2-22 specimen. The starting magnetization intensities (heating values at 30 $^{\circ}\text{C}$, J_{30}) are indicated for each graph. Symbols are as in Fig. 3. (i) Histogram showing the variation in the alteration index of individual heating/cooling runs of P2-22 sample following Hrouda's et al. (2003) method.

The rock magnetic experiments carried out allowed characterizing the magnetic mineralogy, domain state and thermal stability. The IRM acquisition curves are almost saturated at fields of 150–200 mT indicating that they are dominated by a low-coercivity mineral (Fig. 2). A small fraction of a high-coercivity mineral (up to 5–10% of the SIRM or Saturation of IRM at 1T), most probably haematite, seems also to be present. However, its contribution to the magnetization is not significant. The Curie temperatures (T_C) determined from thermomagnetic curves performed on selected samples are around 580 $^{\circ}\text{C}$ indicating the dominance of magnetite in both facies (Fig. 3). Occasionally, T_C s of up to 615 $^{\circ}\text{C}$ have been observed in some reddish brown ashes pointing out that stable maghaemite might also be present (Fig. 3b). The occasional presence of maghaemite has been already observed in this type of fire (e.g., Carrancho et al., 2009, 2013) and it would imply a thermochemical remanent magnetization (TCRM), making such specimens unsuitable for absolute archaeointensity determinations. The high thermomagnetic reversibility of ashes is noteworthy, particularly the white one (Fig. 3a). Conversely, carbonaceous specimens exhibit much lower thermomagnetic

reversibility producing secondary magnetite on cooling (Fig. 3c). This indicates that they underwent lower heating temperatures as is explained in more detail in case study 2 (Section 4).

3.2.2. NRM directional stability and archaeomagnetic dating

Fig. 4(a–f) illustrates representative NRM orthogonal demagnetization diagrams of both facies and the stereographic projection with all the individual Characteristic remanent magnetization (ChRM) directions determined. All specimens show a secondary viscous component of normal polarity easily removable in the first steps of the magnetic cleaning (<10–15 mT or <200–250 $^{\circ}\text{C}$) particularly evident in carbonaceous specimens (Fig. 4d–e). The NRM stability of the ashes is defined by a stable, high intensity normal polarity component almost demagnetized at 80–100 mT decaying univectorially towards the origin (Fig. 4a–b). AF demagnetized carbonaceous specimens exhibit also a single component (Fig. 4d) or occasionally two-component magnetizations partially overlapping. In the latter case, these specimens were not considered to calculate the ChRM direction.

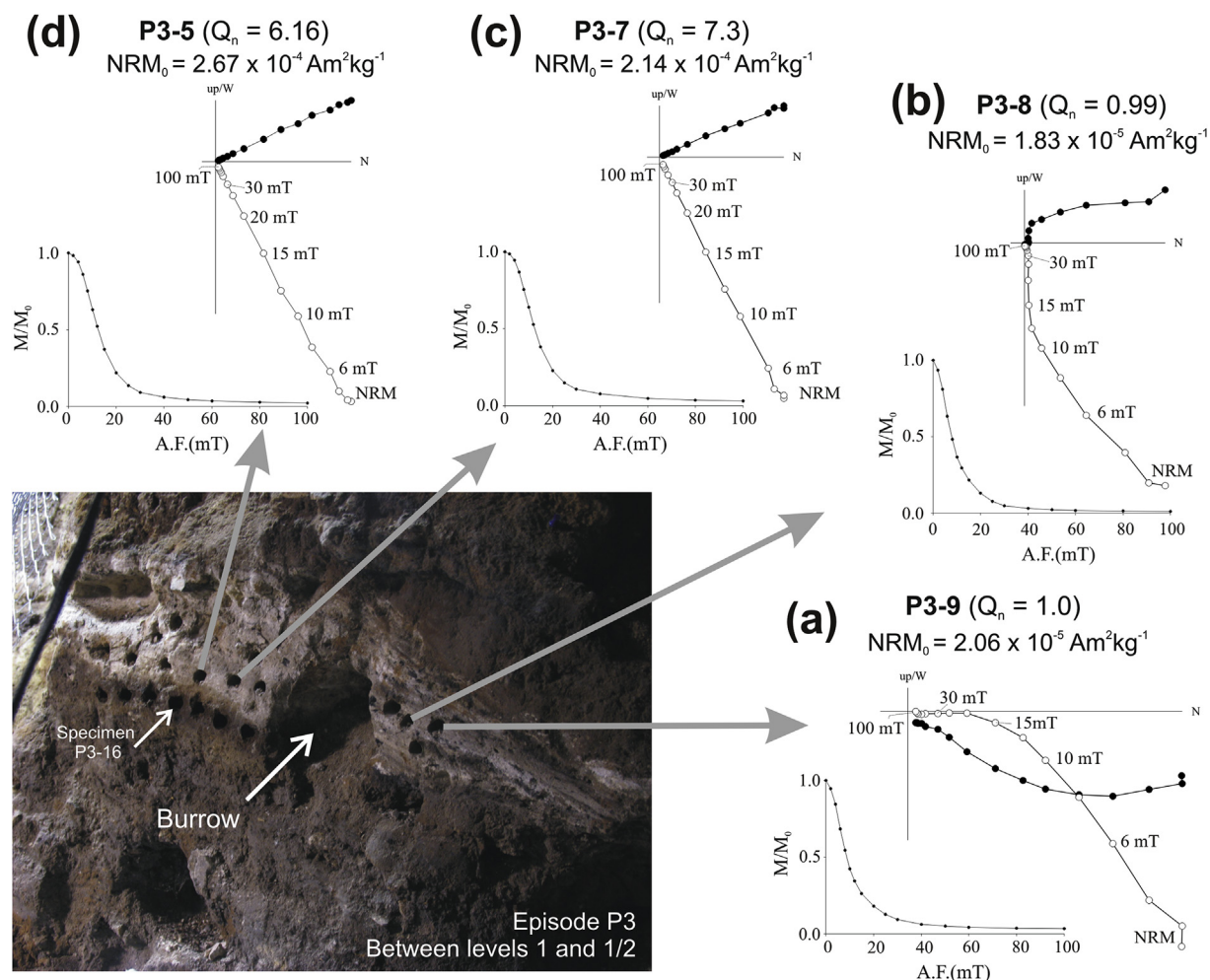


Fig. 8. Representative orthogonal NRM demagnetization plots of a partially bioturbated burning event from El Portalón Cave. (a–b) Diagrams of two ashes showing anomalous behaviour. Note how ashes from the right part of the photo are somewhat mixed. (c–d) Diagrams of two ashes from the central-left part (*in situ*). Symbols are as in Fig. 4 and 6. See Section 5 for explanation.

Three out of 5 specimens sampled for TH demagnetization of the NRM broke during laboratory analyses. The two remaining specimens (Fig. 4c and e) correspond to an ash and a carbonaceous specimen, respectively. The ChRM direction in the ash was determined between 250 °C to 580–600 °C. The ChRM direction in the carbonaceous specimen was defined between 250 °C and 450 °C, reflecting a partial thermo-remnant magnetization (pTRM) likely caused by moderate heating that this facies underwent. This is consistent with the irreversible thermomagnetic behaviour of this facies (e.g., Fig. 3c) as is more detailed in Section 4 (case study 2). AF demagnetization is adequate to determine successfully the ChRM direction because the main remanence carrier is a low-coercivity mineral.

Carrancho et al. (2013) established various quality selection criteria to identify anomalous behaviours and determine the reliability of these structures to obtain archaeomagnetic directions. These are: (i) presence of all the sedimentary facies for each burning event (ashes over underlying carbonaceous facies); (ii) Koenigsberger (Q_n) ratio values > than 1 indicating a stable thermoremanence (TRM) or a partial TRM; (iii) absence of any indication of mechanical alteration in the sediments (e.g., mixed or truncated facies), and (iv) a majority of demagnetisation diagrams with univectorial NRM among the ashes. Following these criteria, 8 specimens were rejected for the calculation of the mean

archaeomagnetic direction (the three broken specimens excluded). These specimens have the lowest NRM intensities, Q_n ratios <1.0 and anomalous directions or multicomponent demagnetization diagrams. As it is discussed further in case study 3 (Section 5), all these features are indicative of some type of post-depositional reworking. It is worth mentioning that most rejected specimens come from parts close to the burrow, which are potentially affected by reworking (Fig. 1). The mean direction obtained (Fig. 4f) has a Declination = 20.1°; Inclination = 56.5°; $k = 63.3$; $\alpha_{95} = 4.4^\circ$, according to Fisher (1953) statistics.

Probability density functions of possible dates for declination and inclination were obtained comparing the results with the directional European PSV curve (Carrancho et al., 2013) at the site coordinates using the archaeomagnetic dating tool of Pavón-Carrasco et al. (2011). The probability functions were combined to obtain the most probable dating solutions at 95% confidence level (Fig. 5). Four different dating intervals were obtained: 2256–2143 BC; 2061–1888 BC; 1651–1520 BC and 1081–1000 BC. The first two intervals can be discarded because they are inconsistent with the Bronze Age context for this unit. The last one is within the bounds of possibility and with the largest statistical probability but is out of the radiocarbon date range (1510–1410 yr BC) by more than three centuries. The one which best agrees with the radiocarbon dating is the 1651–1520 BC

interval, although slightly sooner than indicated by radiocarbon date. In any case it is consistent with a Middle-Late Bronze Age for the MIR103 unit.

The archaeomagnetic dating reported does not improve the accuracy of radiocarbon dating for this case study although both are archaeologically consistent. Beyond that, thanks to the recently developed European PSV curve (Carrancho et al., 2013), is already possible to date with archaeomagnetism *in situ* burnt archaeological materials from Western Europe for these older periods. Much remains to be done in order to improve and extend back in time the archaeomagnetic dating technique but the potential of these materials for both geophysical and archaeological purposes is indisputable.

4. Case 2: estimating heating temperatures

4.1. Background

Determining the temperature at which a burnt archaeological remain was heated in the past is a topic of interest for archaeologists. This information is interesting because on the one hand, it may help to reconstruct the technological conditions under which a combustion structure was carried out. On the other hand, it has also geochronological implications since other dating methods (e.g., thermoluminescence or TL) require a minimum heating temperature in the materials to be dated in order to obtain reliable results (e.g., Mercier et al., 1995). Therefore, determining this information with other techniques is archaeologically valuable.

Magnetic methods are not new to this aim and different approaches have been proposed. Linford and Platzman (2004) proposed a method to estimate heating temperatures applying a linear unmixing model based on the correlation observed between the maximum exposure temperature recorded in experimentally burnt sediments and the hysteresis properties. Hrouda et al. (2003) used progressive susceptibility *versus* temperature measurements as palaeotemperature indicator quantifying magnetomineralogical changes induced during laboratory heating. In essence, this approach does not differ substantially from that proposed by Spassov and Hus (2006). These authors performed several rock magnetic analyses on Roman kiln samples and tested the results with a thermal conductivity model. In both cases, the basic assumption relies on the fact that if a sample was heated in the past to a given temperature, it should not show mineralogical alterations when heated again until that temperature under similar conditions in the laboratory. A similar approach using the reversibility of thermomagnetic curves combined with other rock magnetic measurements and petrographic and dielectric analyses was tested on prehistoric potsherds from Venezuela by Rada Torres et al. (2011).

Here we use an alternative approach based on the stepwise thermal demagnetization of the NRM of oriented samples. Many prehistoric fireplaces do not reach temperatures high enough (600–700 °C) to acquire a full TRM. In theory, any archaeological material heated to temperatures below the Curie temperature (T_C) of the ferromagnetic mineral present (e.g., magnetite T_C : 580 °C; Dunlop and Özdemir, 1997) is able to record the Earth's magnetic field direction on cooling through the acquisition of a partial thermal remanent magnetization (pTRM). Regarding the carbonaceous facies of burnt anthropogenic cave sediments, this pTRM will partially reset the original magnetization recorded by the substrate which is supposed to be a depositional remanent magnetization or DRM recorded before any previous heating. Thus, two components of magnetization should be distinguished and progressive TH demagnetization of the NRM can be used to isolate both

components. The highest temperature step at which the low temperature component is still present defines the last heating temperature.

Progressive TH demagnetization has been widely used in volcanic studies to distinguish emplacement mechanisms and the temperature of emplacement of pyroclastic flows, lithic clasts and other volcanic products (e.g., Kent et al., 1981; Bardot and McClelland, 2000; Cioni et al., 2004; McClelland et al., 2004; Porreca et al., 2007). Its application to various archaeological materials of different age has also been investigated (e.g., Gose, 2000; Brown et al., 2009; Herries, 2009). However, to our knowledge, this method has not been yet tested in burnt anthropogenic cave sediments.

4.2. Results and discussion

Fig. 6(a–f) illustrates representative examples of TH demagnetization diagrams of the NRM from carbonaceous samples from different Holocene burning events from El Mirador, Portalón and El Mirón Cave (Spain; Fig. 1). These burning events have previously been studied for archaeomagnetic purposes (Carrancho et al., 2013). However, the objective here is to illustrate how the identification of pTRMs permits the estimation of the last heating temperature in the carbonaceous facies of these fires.

After removing a low temperature component probably of viscous origin (<150–200 °C), an intermediate component of normal polarity between 200–250 °C and 400–450 °C is systematically observed (Fig. 6a–f). Finally, a high temperature (HT) component can also be distinguished between about 400–600 °C. The estimation of the ancient heating temperature is based on identifying the maximum unblocking temperature ($\max T_{UB}$) of the pTRM. This is at about 400–450 °C where the intermediate magnetization component switches the direction (highlighted with grey ellipses in Fig. 6).

The fact that the intermediate magnetization component (pTRM) lies along the Earth's magnetic field direction showing normal polarity is the basic principle of this technique. The HT component is of normal polarity and predates the pTRM component. It represents the Earth's magnetic field direction originally recorded by the archaeological surface during its formation and on which subsequently the burning took place. The remanence associated to the HT component would be detrital (DRM). During the heating, a portion of its original remanence with unblocking temperatures (T_{UBS}) less than or equal to the maximum temperature heating underwent by the carbonaceous facies (ca. 400–450 °C; Fig. 6) was replaced by the pTRM acquired on cooling. Under the proviso that the materials remains undisturbed (*in situ*) after burning, the progressive TH demagnetization of the NRM may yield the heating temperature. Occasionally we have observed that the HT component is randomly oriented (e.g., Fig. 6f). This might be explained if before heating, the substrate was for whatever reason reworked (e.g., some kind of intentional preparation of the surface). Such reworking would necessarily be produced before heating because the pTRM direction is northward and again showing $\max T_{UB}$ between 400–450 °C. We are currently trying to reproduce this effect experimentally in order to verify this hypothesis.

The palaeomagnetic estimation of the heating temperature in carbonaceous samples requires that the intermediate magnetization component is of thermal origin. If it results from another mechanism of magnetization such as viscous remanent magnetization (VRM) or chemical remanent magnetization (CRM) the temperature assessment may be erroneous. In “geologically” young materials like these (~5–2 ky BC) and considering that the main carrier is PSD magnetite, the intermediate component with $\max T_{UB}$ of about 400–450 °C is highly unlikely to be due to a viscous

overprint. That is not compatible with the time-temperature nomograms for magnetite (Pullaiah et al., 1975). The possibility of a CRM is more difficult to prove because it can be derived from the formation of a new magnetic phase or the growth or shape change of a pre-existing one (Dunlop and Özdemir, 1997). These are preliminary data and further experiments are being carrying out in order to verify it. It should be noted, however, that Q_n ratio values of all carbonaceous samples but one (Fig. 6f), are over unity suggesting a pTRM origin of the magnetization. This has been tested with partial thermomagnetic curve experiments as we outline below.

Partial thermomagnetic curves of a carbonaceous sample adjacent to the specimen P2-01 (Fig. 6b) were carried out in order to study its thermomagnetic reversibility following Hrouda's et al. (2003) method to estimate palaeotemperatures. A complete thermomagnetic curve of this sample is irreversible (heating and cooling cycles do not coincide) when heated up to 700 °C (Fig. 7a). In order to study at which temperature step magnetic alteration begins, partial thermomagnetic runs were carried out on another (sister) powdered sample (~350 mg) in 50 °C incremental steps from 200 °C to 550 °C (Fig. 7b–h). As expected, the heating and cooling cycles exhibit high reversibility until 400–450 °C (Fig. 7b–f). However, when the sample is heated in the laboratory over 500 °C (Fig. 7g–h), the magnetization during the cooling cycle considerably increases because mineralogical transformations take place (formation of secondary magnetite). Consequently, the sample loses its thermomagnetic reversibility. This alteration can be quantitatively estimated as $A_{30} = 100 (j_{30} - J_{30})/J_{30}$, where j_{30} and J_{30} are the magnetization on the cooling and heating curves at 30 °C, respectively (Hrouda et al., 2003). In this case study, the alteration starts at 450–500 °C, reaching a maximum at 550 °C (Fig. 7i). These results agree well with the maximum T_{UB} temperatures determined for the carbonaceous sample P2-01 (Fig. 6b) and are a solid indication that the intermediate magnetization component is a pTRM.

As far as the ashes are concerned, these most likely reached temperatures over 600–700 °C, which has been shown in our previous studies (Carrancho et al., 2009, 2012, 2013). Ashes from this type of fire are characterized by high NRM intensities (one order of magnitude higher than carbonaceous or more), Q_n ratios > 1, stable and univectorial NRM demagnetization diagrams and full reversibility in thermomagnetic curves. The thermomagnetic curves of the ashes shown in Fig. 3a–b (case study 1) are a good example of this kind of behaviour. This is logical since ashes are the last residue of combustion and the underlying carbonaceous facies represents the fire-altered topsoil on which the fire was performed. The carbonaceous facies does not differ substantially from the “black layer” studied by Mallol et al. (2013) in Middle Palaeolithic and experimental fires. In essence, both are blackened layers rich in charcoal remnants and organic matter. Our heating temperatures determined with paleomagnetic analyses are very similar to those reported by Mallol et al. (2013) from a series of actualistic fire experiments. Canti and Linford (2000) also reported temperatures of around 400 °C on the substrate beneath ashes exceeding 800 °C and Carrancho and Villalain (2011) and Calvo-Rathert et al. (2012) monitored temperatures of around 350 °C in the peripheral surface of an experimental fire. More dramatic colour changes could be seen depending on the original mineral composition and burning conditions. According to Mallol et al. (2013), the duration of heating and the amount of fuel used seem to be less important factors in the formation and preservation of this blackened layer as is the presence of organic matter. Indeed, burning of organic matter is necessary to promote the formation of magnetite under prevailing reducing conditions (Carrancho et al., 2009). In any case, the palaeomagnetic evidence presented here indicates that this facies systematically underwent heating temperatures up to 400–450 °C.

The application of this method differs depending on the nature of the archaeological material studied. In contrast to sediments as studied here, rocks commonly located around archaeological fireplaces have their previous (geological) magnetization. In such a case, an eventual pTRM should also record normal polarity if the rocks are *in situ*. However, HT component should exhibit a random direction corresponding to the original remanence acquired during the rock's genesis. Good examples of this are published using experimental and archaeological materials (e.g., Gose, 2000; Herries, 2009). The usefulness of the palaeomagnetic method for determining heating temperatures in burnt anthropogenic cave sediments is certainly of high value for the archaeologists.

5. Case study 3: assessment of post-depositional processes

5.1. Background

Identifying potential syn/post-depositional processes in archaeological cave fires and evaluating their degree of alteration is relevant because if these processes are severe enough, there are significant implications for the cultural interpretation of a site. Depending on the degree of alteration, these processes can cause displacement or dispersion of artefacts within the stratigraphy over distances of millimetres to centimetres or even meters. Other effects involve fragmentation of bone and lithic remains, mixing of burnt and natural sedimentary components and in the most extreme cases, the complete homogenization of the sediment. The implications of these processes are not only cultural but also chronological. Some authors have noted the importance of collecting samples for thermoluminescence (TL), optical stimulated luminescence (OSL) and electron spin resonance (ESR) dating from undisturbed areas showing the least evidence of mineralogical change (e.g., Mercier et al., 1995; Bateman et al., 2007). The measurements of the radiation dose-rates can be seriously affected and not accurately reflect the dose-rates prevailing in the past. It is easy to understand the significant consequences derived from the correct assessment of the degree of alteration caused by these processes in terms of establishing a reliable age determination.

Regardless of whether the responsible agent is anthropogenic, biogenic or geogenic (see Goldberg and Sherwood, 2006 for a good synthesis), syn/post-depositional processes in cave fires can be generally grouped as physical and/or chemical. The latter imply mineralogical changes and diagenesis in general. Particularly, ash diagenesis from archaeological cave fires has been extensively studied over recent years with diverse techniques such as soil micromorphology, Fourier transform Infrared spectrometry (FTIR), geochemistry or scanning electron microscopy, among others (e.g., Bull and Goldberg, 1985; Weiner et al., 1993, 2002; Karkanas, 2010). Particularly interesting are some studies carried out on Middle Palaeolithic sites establishing a diachronic sequence of diagenetic alteration of calcite, the major component of wood ashes (e.g., Schiegl et al., 1996; Weiner et al., 1993, 2002). However, burnt anthropogenic cave sediments (and combustion features in general) are susceptible not only to diagenesis but also to reworking. That is, mechanical disturbances of the burnt sedimentary facies. Mechanical reworking of cave fires has been traditionally addressed through simple macroscopic or field observations. The absence of some of the facies composing these fires (rubefied sediment, charcoal and ashes), absence of their lateral continuity or mixing of burnt and unburnt material are the main criteria used. Recently, Mentzer (2014) detailed a comprehensive description of the main features characteristic of reworked combustion structures both at macro and microscale. The palaeomagnetic technique has been recently proposed to evaluate mechanical post-depositional processes in archaeological cave fires (Carrancho et al., 2012). This case

study aims to test the reliability of the method determining to what extent the mechanical reworking might have affected a partially bioturbated Late Holocene burning event from El Portalón Cave (Burgos, Spain; Fig. 8).

5.2. Results and discussion

Representative examples of NRM demagnetization diagrams corresponding to ashes from different parts of the structure are shown in Fig. 8. Thermal demagnetization of a carbonaceous specimen from this event is shown in Fig. 6a (P3-16; Fig. 8) and whose characteristics are reported in Section 3.2.2 (case study 2).

The NRM demagnetization diagrams of specimens to the right side of the burrow (Fig. 8a–b) exhibit an anomalous and unstable directional behaviour. Q_n ratio values are not greater than 1 and initial magnetization intensities (NRM_0) are one order of magnitude lower than those from pure white ashes. On the contrary, NRM demagnetization plots to the left of the burrow (Fig. 8c–d) are defined by a stable single palaeomagnetic component, around 10 times more magnetic than carbonaceous samples, displaying high Q_n ratio values and reproducible directions among them. The main magnetic carrier is a low-coercivity mineral as the normalized decay intensity plots indicate. According to thermomagnetic curves this mineral is low-Ti titanomagnetite or partially maghaemitized magnetite with Curie temperatures of around 580 °C–600 °C (Fig. 9a–c). Maghaemite might be responsible of the inflection observed at about 310 °C in Fig. 9b, although it could also be due to change of grid structure.

Even when these structures were partially affected by bioturbation, it is still possible to evaluate whether mechanical reworking extends beyond the visual alteration originally observed in the field in order to exclude those samples for calculating the mean archaeomagnetic direction. The quality selection criteria established by Carrancho et al. (2013) to obtain a reliable mean direction in these fires are related to the following factors: (i) a good preservation of the structure (presence of all the sedimentary facies for each burning event, meaning ashes over underlying carbonaceous facies), (ii) the intensity of the burning with regard to the quantity of fuel employed (ash thickness) and (iii) an efficient record of the magnetization (Koenigsberger ratio values greater than 1 and a majority of demagnetization diagrams with univectorial NRM among the ashes).

The results in this study are very similar to those reported by Carrancho et al. (2012) where the magnetic behaviour of two different burning events from El Mirador cave (one strongly bioturbated and other apparently *in situ*) was analysed and compared. It is evident that samples showing anomalous magnetic behaviour were reworked by the effect of bioturbation. However, the interesting fact as this case shows is that adjoining areas to the

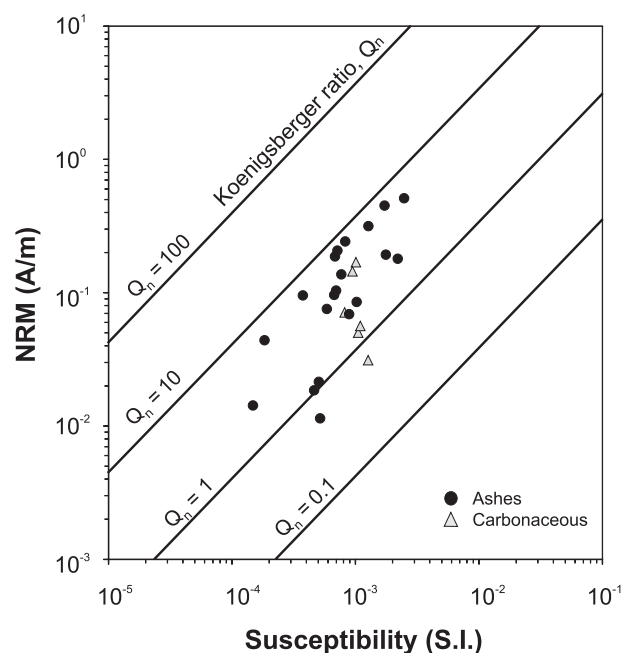


Fig. 10. Natural remanent magnetization (NRM) vs. bulk magnetic susceptibility (S.I.) showing lines of constant Koenigsberger ratio (Q_n) between 0.1 and 100 for the P3 burning event samples (see legend).

bioturbation may also suffer from reworking and in many cases this effect cannot be easily distinguished in the field. Although in this case it did not imply movement of archaeological remains in the stratigraphy (fumiers are usually not rich in archaeological materials), special care must be taken during the excavation of these fires. Archaeostratigraphic 3D projections of coordinated artefacts (e.g., pottery, lithic remains) can be particularly useful for a proper archaeological interpretation.

From the magnetic point of view, a useful parameter with regard to TRM preservation is the Q_n ratio. Koenigsberger values for this collection are between 1 and 7.3 (Fig. 10) whereas two out of three samples with values < 1 correspond to ashes from the reworked side (e.g., Fig. 8b). The other is a carbonaceous sample. On the basis of these results, the relationship between the *in situ* nature of the structure and the preservation of the TRM is obvious. Mechanical reworking promotes the disorganization of the magnetic moments of the ferromagnetic grains reducing the remanence but maintaining the bulk magnetic susceptibility. As this parameter does not depend on the orientation of the magnetic grains (excluding the anisotropy), the direct consequence is that the TRM is lost and Q_n values become considerably reduced. Moreover, the

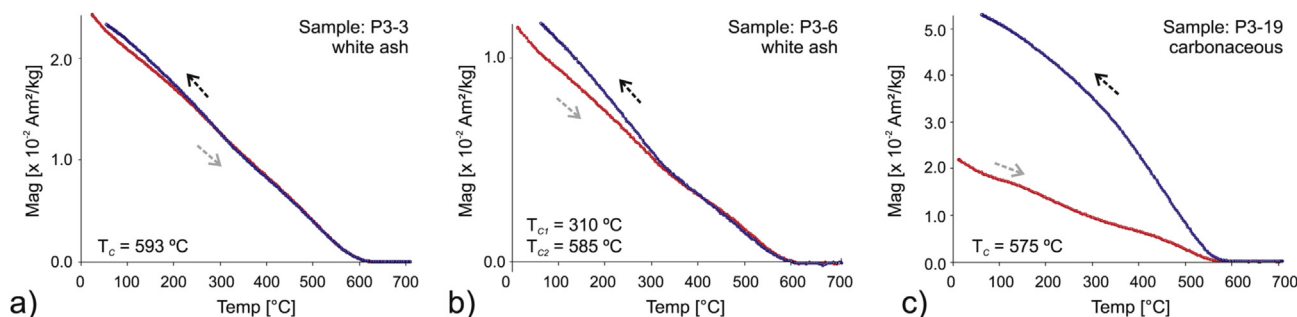


Fig. 9. (a–c) Representative thermomagnetic curves (magnetization vs. temperature) of two ashes and a carbonaceous sample from P3 burning event (El Portalón Cave). Symbols are as in Fig. 3.

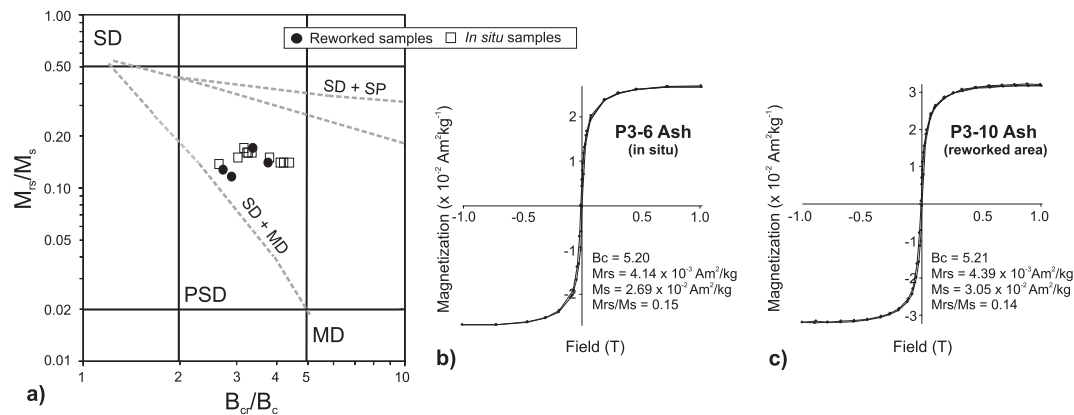


Fig. 11. (a) Day-Dunlop logarithmic plot (M_{rs}/M_s vs. H_{cr}/H_c) of representative *in situ* and reworked ash samples from P3 burning event (Fig. 8). The dashed lines represent mixing curves taken from Dunlop (2002) for mixtures of single-domain (SD) with multidomain (MD) or superparamagnetic (SP) magnetite particles. (b–c) Two representative hysteresis loops of an *in situ* and a reworked ash, respectively, showing main hysteresis parameters.

multicomponent NRM structure of reworked samples is also indicative of alteration along with lower magnetization values. Carrancho et al. (2012) have described the importance of combining these analyses with macroscopic field observations such as determining the lateral continuity of the facies, absence of sedimentary mixtures, etc.

No significant differences in terms of magnetic composition or domain state variation are observed between *in situ* and reworked ash samples from the rock magnetic experiments carried out. The backfield ratios obtained oscillates between 15.79 and 22.94 mT without distinctive differences between both types of samples. The hysteresis ratios obtained range from $0.116 < M_{rs}/M_s < 0.170$ and $2.645 < B_{cr}/B_c < 4.380$ (Fig. 11a), indicating a pseudo-single domain (PSD) state for the magnetite grains, which suggests that the granulometric distribution of both the *in situ* and reworked ashes is quite similar. This homogeneity in magnetic properties can also be observed in the representative hysteresis loops shown in Fig. 11(b–c) and similar results were reported in analogous studies (Carrancho et al., 2009, 2012; Kapper et al., 2014a,b).

Summarizing, it is of primary importance for archaeomagnetic dating purposes to determine the *in situ* nature of a cave fire if only directional analyses are carried out. Magnetic orientation for archaeointensity determinations is not indispensable, although the material cannot be disaggregated. For archaeologists, the concept of “*in situ*” does not necessary mean the same as for archaeomagnetists. The latter look for burnt materials that preserve exactly the same position as they had when cooled. Any post-depositional movement, no matter how minimal, may have significant effects in the archaeomagnetic results. Archaeologists usually consider that a combustion feature remains *in situ* as long as artefacts or sediments do not experience significant stratigraphic movements which may compromise the cultural interpretation of the record. Using the above guidelines and when possible combining this information with that provided by other disciplines (e.g., micromorphology and FTIR) is the best way to infer the primary or secondary position of an archaeological combustion feature.

6. Conclusions

Three applications of archaeo- and rock magnetism to the study of burnt anthropogenic cave sediments have been reported in the following case studies: (i) archaeomagnetic dating; (ii) estimating palaeotemperatures and (iii) evaluating post-depositional processes.

Case study 1: A mean archaeomagnetic direction was obtained from a burning event at El Mirador Cave. Its comparison with the directional European SV curve yielded several dating intervals. According to archaeological evidence, the most likely date of the last burning was 1651–1520 yr BC (95% of confidence), slightly older than an independent radiocarbon date from this unit but both are archaeologically consistent. The agreement of the two dating methods reveals the potential of anthropogenic burnt cave sediments as geomagnetic field recorders as well as the possibility to be dated by archaeomagnetism. These data represent the first archaeomagnetic dating obtained in this type of materials.

Case study 2: Stepwise thermal demagnetization of the NRM of oriented carbocaneous samples is a useful method to estimate the last heating temperature. These samples show an intermediate palaeomagnetic component of normal polarity that we interpret as a pTRM with maximum unblocking temperatures of 400–450 °C, representing the last heating temperature. These temperatures agree well with those obtained from partial thermomagnetic analyses.

Case study 3: The archaeomagnetic analysis of a burning event partially bioturbated allowed to obtain a comparative characterization of the magnetic behaviour of *in situ* samples against reworked samples. The latter showed low NRM intensities (at least one order of magnitude), Q_n ratios < 1 and multicomponent nature of NRM along with anomalous directions. Mechanical reworking extends beyond the deformation which one can visually identify in the field. Therefore, special care must be taken when excavating these features in order to interpret correctly the primary position of the materials.

As a concluding remark, archaeomagnetic analyses on burnt anthropogenic cave sediments have a great potential not only from the geophysical point of view (reconstructing directional and/or intensity changes of geomagnetic field in the past) but also for archaeological purposes. We encourage our colleagues to work on this type of materials promoting multidisciplinary collaboration.

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