Archaeological applications of naturally occurring nanomagnets

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Abstract. The ubiquitous presence of iron minerals within the soils and sediments forming archaeological sites can often provide a valuable record of past human activity. These records are formed through the alteration of weakly magnetic minerals to fine grained iron oxides, such as magnetite or maghaemite, that leave an almost indelible magnetic “finger print” on the landscape. Archaeologists have exploited these magnetic records at a variety of levels from geophysical survey to reveal the location of a site, to determining how old a particular excavated feature may be through archaeomagnetic dating. More recent studies have investigated the process of magnetic enhancement through the often complex interaction of pedogenic, microbial and anthropogenic mechanisms and pathways. This research has revealed many unique magnetic signatures within archaeological sediments that may help to identify a range of significant environmental conditions, such as the effects of climate change or the deliberate use of fire. This paper aims to provide an overview of how the techniques of environmental magnetism may be applied to the analysis of archaeological remains. Both field based geophysical prospecting and the measurement of magnetic properties from samples recovered during excavation will be considered. The interpretation of the resulting magnetic measurements will also be addressed through the use of an unmixing algorithm applied to hysteresis data.

1. Introduction
Iron minerals, mainly very fine grained, nano scale oxides and oxy-hydroxides, are ubiquitous within the soils and sediments that have developed over the majority of outcropping geology. Despite existing in relatively low concentrations (generally less than 1% of soil mass by weight) these iron minerals are often responsible for determining both the distinctive colour of many soils and, more importantly, their magnetic properties. These vary widely between the differing species of iron minerals with the ferrimagnetic oxides magnetite and maghaemite demonstrating the strongest magnetic properties. In addition, many of the more weakly magnetic iron minerals are extremely sensitive to environmental change and will be readily transformed to more strongly magnetic forms by comparatively subtle reduction and oxidation mechanisms.

The sensitivity of iron minerals to environmental change occurs over a wide range of scales from the regional response to climate change, demonstrated through paleoclimate records [1], to the microscopic influence of soil bacteria [2]. Whilst these changes may occur as part of the natural pedogenesis of soil it is clear that anthropogenic activity, both today and in antiquity, provides an additional moderation, often at a highly local scale, to the enhancement and concentration of magnetic iron oxides within the soil [3]. Perhaps more surprising is the persistence of these alterations within
the buried soil horizons and sediments that constitute the archaeological record and the ability of modern scientific techniques to interpret these magnetic “finger prints” left by the actions of our ancestors in the past [4] [5].

This paper aims to provide a short review of the potential offered by the geomagnetic to archaeological science by considering the processes and mechanisms that influence the anthropogenic alteration of iron minerals and the instrumentation and methodology required to record and interpret these records. Brief case studies will be used to illustrate the application of these techniques within three main areas of research: the geophysical location of archaeological activity through magnetic survey at both an inter and intra site level, the physical dating of archaeological events through the secular variation of the earth’s magnetic field and, finally, the reconstruction of environmental conditions from the formation of diagnostic magnetic minerals.

2. Alteration of magnetic minerals and magnetisation processes

Figure 1 provides a simplified summary of the main chemical pathways and reaction temperatures required to alter weakly magnetic iron minerals commonly found in the soil, for example lepidocrocite (γFeOOH), goethite (αFeOOH) and haematite (αFe₂O₃), to far stronger ferrimagnetic forms, such as magnetite (Fe₃O₄) and maghaemite (γFe₂O₃). These reactions generally follow the initial reduction and subsequent oxidation of the precursor minerals at relatively modest temperatures, conditions that may occur inorganically through, for example, the action of natural forest fires [6] [7].

![Diagram showing pathways for the formation of highly magnetic iron oxides from weakly magnetic precursor minerals, commonly found in the soil. Many of the conditions necessary for these processes (generally altering the pH and Ph of the soil) may be controlled either directly or indirectly by (micro) biological mechanisms, classed as either boundary organised mineralization (BOM) for the intracellular production of minerals or biologically induced mineralization (BIM) for extra cellular production.](image-url)
Similar chemical alterations may also be induced organically, through biological moderation due to soil bacteria at ambient soil temperatures. This may occur either within the highly controlled cell structure of the organism (boundary organised mineralization or BOM) or due to the extra cellular export of metabolic products resulting in mineral formation with other ions within the environment (biologically induced mineralization or BIM). BOM type bacteria generally thrive within a micro-aerobic environment and produce distinctive crystals of magnetite (and under certain conditions the iron sulphide greigite, Fe₃S₄) often displaying a highly regular morphology and limited size range. The presence of such diagnostic “magneto-fossils” are evident within many geological contexts and may be readily compared to the mineralization processes witnessed in current, live cultures of bacteria [8].

In contrast, many BIM type bacteria prefer anaerobic conditions and utilise Fe ions as a means of electron exchange during respiration. Due to the uncontrolled, extra cellular nature of the BIM mineralization process the resulting iron minerals are often very fine grained, covering a wide crystal size range, that confuses the distinction between inorganic and organic alteration pathways. In general, all bacterial activity requires close association with organic matter to provide an energy source and it is certainly common to find that the magnetic properties of the organic-rich, biologically active topsoil are enhanced compared to the underlying subsoil. In addition, the inorganic formation of mixed valency magnetite through the hydrolysis of lepidocrocite, goethite or haematite is mediated by the concentration of Fe²⁺ available to precipitate the more highly magnetic mineral. The supply of Fe²⁺ ions is obtained by the bacterial reduction of soil iron oxides [9] and it is likely that the increased Fe reducing capacity of soils after the addition of organic matter is due to increase microbial activity.

Figure 2. The preferential alignment of magnetic particles with the earth’s magnetic field produces an enhanced permanent magnetisation. A thermoremanent magnetisation (a) will develop when sufficient thermal energy is available to overcome the unblocking energy of individual magnetic grains (represented by red arrows) that will rotate to a new orientation more closely parallel to the ambient field (blue arrows). Detrital remanence (b) occurs when fine grained magnetic material settles from suspension and particles physically rotate to align with the ambient field. Newly formed magnetic minerals may also develop a chemical remanence (c) as the crystal precipitates from solution in the presence of an ambient field.

Whether derived from organic or inorganic pathways magnetic minerals within the soil may undergo a further process of alignment along the direction of any ambient magnetic field, usually the current direction of the earth’s magnetic field (figure 2). For example, any magnetic particle provided with sufficient thermal energy to overcome its blocking energy will become free to adopt a new direction of magnetisation along the crystallographic axis most closely aligned to the external magnetic field. On removal of the thermal energy this new direction of magnetisation will become blocked leading to the production of a permanent thermoremanent magnetisation shared by all the surrounding particles subjected to the same heating event (e.g. [10]). Alternative means for the orientation of magnetic particles may occur during the precipitation from solution of newly formed magnetic minerals, to produce a chemical remanence, or through the physical rotation of small, water-
borne particles settling from suspension to produce a detrital remanence. The magnitude of these remanent magnetisations may often equal, or in the case of thermoremanent magnetisation, exceed the induced magnetic component due to the magnetic susceptibility of the material within the earth’s magnetic field.

2.1. Anthropogenic modification – magnetic archaeology

For magnetic techniques to be of use to archaeologists there must, of course, be a direct link between anthropogenic activity in antiquity and the processes resulting in the alteration or concentration of magnetic minerals in the soil. The production of ferrous metal artefacts, such as swords or shield bosses, would provide an obvious highly magnetic target, although in practise the location of such objects is often the preserve of metal detectors. However, many semi-industrial archaeological processes, such as metal working or pottery production, do indeed leave a distinctive magnetic “finger print” behind on the landscape due to either the high temperature alteration of the iron minerals within the fabric of the kiln itself or due to the distribution of waste products, such as the fuel ash and associated burnt soil.

![Figure 3. Formation of a magnetic anomaly at a theoretical site (a) with a thin layer of moderately magnetic topsoil 0.2m thick overlying a less magnetic subsoil. Creation of a 1m deep ‘V’ shaped ditch cut feature (b) assumes the admixture of the excavated soil on the surface that subsequently silts back into the ditch (c). The final magnetic (gradiometer) anomaly (d) has been calculated for the buried ditch assuming an additional 0.2m of topsoil has accumulated over the original ground surface and an instrument sensor height of 0.2m (the approximate direction of the earth’s magnetic field is also shown and was assumed to have a magnitude of 50000 nT). No additional mineral alteration or alignment of the silted particles to form a detrital remanent magnetisation is assumed during the formation of the cut feature, but could certainly be evident where stable single domain magnetic particles are present.](image-url)

There is also considerable evidence that more subtle occupation activity has left a distinctive magnetic trace on the landscape, for example land clearance through deliberate burning or repetitive use of domestic fires. Even the concentration of organic matter, for example within the decayed timber of a post-hole structure, may encourage preferential colonisation by bacteria resulting in localised biomineralization [11]. Magnetic models also demonstrate that simply disturbing the existing magneto-stratigraphy at a site by constructing a bank and ditch cut feature will produce a weak, but detectable, magnetic anomaly (figure 3). The magnetic record produced by archaeological activity can be divided into three categories, related to the precise nature of the activity and the information that each record contains:
2.1.1. Magnetic survey. Geophysical survey provides an important means to aid the location and interpretation of archaeological remains. Magnetic methods, based on measuring the local variation of the earth’s magnetic field over an archaeological site, are both rapid and cost effective to deploy. Under favourable conditions the recorded magnetic anomalies can reveal a wide range of buried archaeological features including burnt remains (e.g. hearths, pottery kilns and metal-working activity), ditches, pits and even very subtle features such as the response due to an individual post-hole setting.

2.1.2. Archaeomagnetic dating. Naturally occurring processes of magnetisation can result in the alignment of individual magnetic grains along the orientation of the earth’s magnetic field. As the earth’s magnetic field has constantly changed direction, both over geological and archaeological time scales, the determination of a “frozen” direction of magnetisation within an archaeological feature can provide means of physical data when compared to a suitable reference curve. To be successful, the acquisition of magnetisation should be related to a single, relatively rapid event, for example the final firing of a pottery kiln, and the archaeological structure must survive in tact without any physical reorientation. In practise, burnt features such as hearths or pottery kilns often provide a suitable source of samples for archaeomagnetic dating, although the detrital remanence formed within sediments may also be used.

2.1.3. Environmental archaeology. The formation and survival of iron minerals is highly sensitive to the local soil conditions and, as such, the presence or absence of specific minerals may act as important environmental markers. For example, the iron sulphide greigite oxidises rapidly and although rarely found within soil is indicative of an anoxic formation environment [12]. Specific processes may also produce a characteristic assemblage of altered magnetic minerals, such as the deliberate use of fire that often results in an increased concentration of very fine grained superparamagnetic particles (e.g. magnetite or magaemite with a grain size <0.03 µm). The varying magnetic properties of different types of granite has even been successfully used as a means of determining the provenance of materials and the likely quarry source of Roman building columns [13].

3. Instrumentation and methodology
Suitable instrumentation for archaeological applications varies mainly between in situ field based measurements, either of the magnetic properties of archaeological features or the indirect local influence they have on the earth’s magnetic field, and laboratory based analysis of recovered samples. Field based instruments have often been based on vertical gradient fluxgate magnetometers, due to the stability and low power consumption of the solid state sensors (figure 4(a)). These hand-held instruments may be used to rapidly acquire measurements of the variation of the earth’s magnetic field (of total magnitude ~50,000nT in the U.K.) over an archaeological site, typically in a range of between 1 and 10 nT, collecting data over a rectangular grid with a sample interval of 0.25m x 1.0m. Both the sample interval and sensitivity of these fluxgate instruments (~0.1 nT) allows a wide range of archaeological features to be detected on sites developed over favourable soil conditions. Where a greater degree of detail is required, or weaker magnetic anomalies due to the archaeological features are expected (for example due to a greater depth of burial) then a more highly sensitive optically pumped caesium vapour magnetometer system may be deployed. These instruments offer a potentially higher level of sensitivity (~0.01 to 0.001 nT) that benefits from application at a denser sample interval, typically 0.1m x 0.5m, but at the expense of more cumbersome equipment and a more substantial power supply. To obtain the best results from such highly sensitive magnetometers it is necessary to customise the design of the instrument through mounting the individual sensors on a non-magnetic cart to separate them from sources of magnetic interference, such as the batteries and control electronics (figures 4(b) and 4(c)).
Figure 4. Field magnetometers used for magnetic surveys of archaeological remains. Hand held vertical fluxgate gradiometers (a) offer the advantage of light weight and rapid data acquisition. More sensitive caesium vapour sensors are cumbersome when carried manually (b) but may be greatly improved by mounting on a non-magnetic cart system (c) to separate the sensors from the batteries and control electronics.

Measurement of the direct magnetic properties of archaeological materials usually begins with determination of magnetic susceptibility in a low-field AC bridge. Either in situ field measurements or laboratory analysis of recovered samples may be used and driving fields within the bridge are kept to a level that will not overwrite any significant directional magnetisation that may be present within samples recovered for archaeomagnetic dating. Laboratory measurement at two, or more, frequencies of driving field allows the frequency dependence of the sample to be determined (e.g. at 470Hz and 4700Hz for the Bartington MS2 susceptibility meter). This is related to the grain-size distribution of magnetic particles within the sample near the superparamagnetic threshold where, as the measurement...
frequency increases, there is insufficient time for individual grains to obtain sufficient thermal energy to unblock and align with the rapidly changing AC driving field.

Orientated samples, collected for archaeomagnetic dating, may be measured by a range of spinner-fluxgate or SQUID based laboratory magnetometers [14]. For optimum sensitivity these magnetometers should be isolated from ambient magnetic fields and repeat measurements then made on each sample in a variety of physical orientations to determine the recorded direction of any remanent magnetisation present. Results may be improved through the gradual demagnetisation and remeasurement of samples to isolate stable remanence directions from more spurious overprints affecting more easily realigned magnetic particles.

More detailed analysis of the magnetic properties of archaeological material may be made on specially prepared subsamples on a range of laboratory instrumentation. Typically, this involves the laboratory induced magnetisation of a sample in a range of applied fields to determine the hysteresis properties that may provide an indication of the grain-size distribution and magnetic mineral types present (e.g. [15] [16] [17]). Automated instrumentation for such measurements allows the application of pulse fields to 5T and sensitivity to extremely weak induced magnetisations produced by the low concentrations magnetic minerals found in some archaeological samples.

The variation of magnetic properties with temperature provides a further insight into the grain size distribution and mineral assemblage present. Low temperature measurements of induced magnetisation, typically over a range from 10 to 300° K, can reveal the unblocking temperatures of fine grained material that is superparamagnetic at ambient temperatures and certain mineral types exhibit characteristic thermomagnetic anomalies, such as the Verwey transition of magnetite at ~120° K [18]. High temperature measurements, from 300 to 1000° K, are equally informative and may reveal the presence of a particular mineral through a diagnostic Curie temperature. However, archaeological sediments often contain a low concentration of iron oxides within an organic rich matrix and at such high temperatures it often proves difficult to suppress the chemical alteration of the original constituent mineral assemblage. Some of these alterations may provide indirect evidence for the presence of specific minerals, for example the inversion of maghaemite to haematite above 550° K can provide the only indication of this mineral that does not otherwise display a distinct Curie temperature [19] [20].

![Figure 5. Schematic of a magnetic particle extraction system for separating the low concentration of fine grained magnetic particles in an archaeological sample from the non-magnetic sediment matrix.](image)

Following the application of a suite of magnetic measurements individual samples may be selected for further sample processing to extract the low concentration of magnetic minerals from non-
magnetic matrix [21]. The efficiency of the magnetic extraction is dependent upon the individual sample under analysis and may require pumping the sediment slurry past a high-gradient magnetic field for several days (figure 5). The resulting magnetic extract is usually proves sufficient to prepare a sample for imaging with a transmission electron microscope, that can be used to reveal characteristic crystal morphologies and identify mineralogy from X-ray analysis. Attempts to apply Mössbauer analysis often provides mixed results due to either the low concentration of iron minerals within the original bulk samples or the difficulty of obtaining a sufficient quantity of extracted material. Some notable success has been gained with suitable archaeological samples, particularly when the Mössbauer experiments are conducted at low temperatures and high applied fields [22].

4. Case studies

4.1. Magnetic survey at Stanton Drew
The village of Stanton Drew, near the city of Bristol in the south west of England, has long been known as a megalithic site composed of three stone circles. The largest of these, the Great Circle, has a diameter of approximately 100m and was the subject of a routine magnetic survey conducted by English Heritage to assist with management issues at the site [23]. For this initial survey data was acquired with a Geoscan FM36 fluxgate gradiometer (figure 4(a)) at a sample interval of 0.25m x 1.0m and the results are presented as a greyscale image, with areas of enhanced magnetic response shown in lighter tones (figure 6). Despite a degree of modern interference, such as the extreme response of the wire fences to the north of the plot, the survey has revealed some subtle archaeological detail including a broad anomaly due to the henge ditch surrounding the monument and what appear to be a completely unknown series of 9 concentric inner circles.

Figure 6. Greytone image of the initial fluxgate gradiometer survey conducted over the location of the Great Circle at Stanton Drew.

To improve the resolution of this very exciting discovery a test survey was conducted to compare the response of the fluxgate gradiometer to a more highly sensitive Scintrex SM4 caesium vapour
magnetometer, in this case operated as a hand held vertical gradiometer (figure 4(B)). The original fluxgate gradiometer survey was also repeated (figure 7(a)) to match both the lower operating height (figure 7(b)) and the increased sample density (figure 7(c)) used for the caesium instrument (figure 7(d)). The results demonstrated the greater resolution of the high sensitivity instrument and when applied to the whole of the Great Circle confirmed both the presence of the concentric circles and that these were in turn composed from a series of discrete pit-type magnetic anomalies (figure 7(d)).

![Figure 7](image)

**Figure 7.** Results from subsequent geophysical survey over a test area at Stanton Drew comparing the original survey (a) to repeat surveys conducted to the fluxgate gradiometer with both lowered sensors (b) and a higher 0.25m x 0.5m sample density (c) to match the data from the high sensitivity caesium vapour magnetometer (d). Wider survey of the interior of the stone circle improved the resolution of the concentric anomalies and suggested construction from a series of discrete, pit-type responses possibly representing the location of former timber uprights.

It seems highly likely that the individual magnetic anomalies are due to the remains of single timber uprights, originally forming a giant wooden temple. Such sites are rare but it seems reasonable
to suggest a prehistoric timber structure at Stanton Drew, predating the later stone circles of a scale, of a complexity worthy of comparison with more highly recognised monuments at Avebury and Stonehenge itself. Limited excavation of the site has been proposed both to test the archaeological hypothesis and to recover samples from the magnetic anomalies to determine the source of the enhanced magnetic response. The magnitude of the recorded anomalies are comparatively weak, so whilst high temperature alteration of the surrounding soil through the destruction of the timber monument by fire remains a possibility a more subtle magnetisation process, perhaps from bacterial biomineralization, can not be dismissed.

Figure 8. Caesium magnetometer total field data from the survey of the Headlands Iron Age enclosure near Marlborough, Wilts. The location of the main enclosure ditch is shown by a strong, ovoid anomaly separated by the modern field division that largely follows the C10th tithing boundary. The interior of the enclosure shows extensive evidence of occupation activity and there is some suggestion that the outer ditch has been recut during an earlier phase of the monument. Entrances are apparent to the north and south where the later tithing boundary passed through the monument. Much weaker magnetic anomalies are found away from the intensive occupation centred on the enclosure and a group of discrete subtle responses may well represent the location of an extensive timber building.
4.2. Magnetic survey at the Headlands Enclosure

To improve the resolution of the caesium vapour magnetometer system further four specially modified total field sensors were mounted on a non-magnetic, wheeled cart (Figure 4(c)). This allows magnetic components, such as the batteries, instrument electronics and data logger consoles, to be removed to either end of the cart away from the magnetometer sensors positioned immediately below the central axle. Mounting the sensors on a rigid platform also reduces positional errors and artefacts in the data due to the gait of the operator when using a hand-held instrument.

This system was used to great effect at the site of the Iron Age ‘Headlands Enclosure’ in Wiltshire, that was first identified through aerial photography as a slightly ovoid ditch encompassing an area of ~1.5ha with a possible entrance and apparent “antennae” ditches facing to the NW. The enclosure is also bisected by an historic C10th tithing boundary that is still evident in the modern land division between the two arable fields. The use of the cart mounted magnetometer system allowed a total area of approximately 5ha to be surveyed and the circuit of the Headlands enclosure ditch has now been recorded as a largely continuous response with evidence for possible entrances (figure 8). A broad, curvilinear ditch-type response passes through the enclosure parallel to the modern field boundary is almost certainly the response to the tithing boundary, noted in the aerial photographic record. Whilst the magnetic response in the vicinity of the enclosure is well defined, with total field anomalies over the ditches exceeding the local average of the earth’s magnetic field by 40nT in places, the use of a high sensitivity caesium magnetometer has proved particularly successful for recording more subtle magnetic responses at the Headlands Enclosure site, revealing a wealth of anomalies to complement both the aerial photographic data and the previous, partial geophysical survey of the monument.

4.3. Bacterial magnetism

Archaeological excavations at the village of Yarnton, near Oxford, UK, revealed a wealth of settlement activity including what appeared to be a significant boundary ditch, providing a division in the landscape, scaled by overlying deposits of river alluvium, found on the modern floodplain (Figure 9). A concentration of prehistoric funerary activity, focused around a Neolithic enclosure, had already been revealed, suggesting a physical division of the ritual landscape from domestic settlement and occupation that may well have been marked by the proposed boundary ditch. Preliminary dating of this feature, required to interpret its relationship with the surrounding archaeological landscape, was based on the recovery of abraded Bronze Age pottery sherds that appeared to have been weathered within the contemporary topsoil for some time before the later boundary ditch was established. Subsequently, an organic rich layer identified at the base of the ditch provided sufficient material for
radiocarbon dating. Analysis revealed that this material also had significantly enhanced magnetic properties in comparison to the surrounding substrate [24].

![Graphs and images](image)

Figure 10. (a) Hysteresis loop of a representative sample from the organic rich basal layer of the boundary ditch. The broken line shows the initial hysteresis loop prior to correction for the paramagnetic slope. (b) Variation of low temperature magnetisation given in a 2.5 T field at 20°C K following cooling from 300°C K to 20°C K in zero field conditions (ZFC, solid line) and then an applied field of 2.5 T (FC, broken line). The decay of low-temperature magnetisation was made in zero field conditions on warming from 20°C K to 300°C K. The first derivative of the magnetisation curve, \( \frac{dM}{dT} \), is also shown to enhance the inflection at ~120°C K concurrent with the Verwey phase transition of magnetite. (c) Transmission electron micrograph of the magnetic particles extracted from the studied sediment. A bacterial origin for the magnetite is proposed due to the distinctive size range, morphology and absence of metal impurities.

Orientated samples recovered from the organic layer revealed that the sediments had a detrital remanent magnetisation, possibly recording the direction of the Earth's magnetic field at the time of soil formation. This direction could then be compared against the known secular variation of the field in the U.K. to suggest an archaeomagnetic date [25] [26], in this case formed between 205 and 90 BC (figure 9(b)) and proved in good agreement with the radiocarbon determinations from two samples of the organic layer.
Further magnetic measurements were conducted on the samples to determine the nature of the remanent carrying component, including hysteresis and low temperature experiments. Both the shape and coercivity of the hysteresis loops (figure 10(a)) suggest a single domain remanence carrier, possibly either the iron oxide magnetite or the iron sulphide greigite, highly similar to the results reported from other studies of bacterial magnetosomes [27]. The presence of magnetite is further inferred from the results of low temperature remanence experiments, that reveals the presence of a diagnostic Verwey transition at approximately 120° K in the first derivative of the magnetisation curve (figure 10(b)). Other inflections at ~60° K and ~270° K showing a loss of remanence, may possibly correlate with the presence of ilmenite (FeTiO₃) [28] and hematite [29].

Further analysis of the remanence carrier was conducted by preparing magnetic extracts yielded strong evidence for the presence of bacterial magnetosomes. Transmission electron microscopy (TEM) analysis of the magnetic extract (figure 10(c)) revealed the presence of distinctively shaped crystals, including both hexagonal and ‘bullet-shaped’ examples, falling within a narrow size range between ~30 to 75nm. This highly controlled morphology, together with energy dispersive X-ray analysis of individual crystals, which showed only Fe and O with no metal compositional impurities, suggesting formation within a highly controlled intracellular environment [30]. In contrast, the inorganic precipitation of magnetite during pedogenesis produces considerably finer grained particles (<30nm), of indistinct morphology and frequent substitution of Fe within the crystal lattice with other common soil elements, such as either Al or Ti [31].

The TEM analysis also revealed the presence of the iron sulphide, greigite, which together with the presence of magnetite magnetosomes is indicative of a micro-aerobic environment. Other environmental factors recognised as important for the proliferation of magnetotactic bacteria are the availability of organic matter, iron and sulphide, conditions that were all likely to be met at the base of the boundary ditch. The accumulation of magnetotactic bacteria would rapidly increase whilst conditions within the sediment layer remained favourable and as each generation of bacteria die their magnetosomes would become aligned with the ambient direction of the earth’s magnetic field.

In this case the magnetic bacteria have apparently accumulated quite rapidly, within a period of approximately 100 years during the late Iron Age, when conditions at this site were uniquely favourable. This concurs with a period when the climate in southern Britain was moving towards warmer, wetter conditions promoting the onset of floodplain conditions along low lying river valleys. The magnetic bacteria are therefore acting as a biomarker for local environmental change as the water levels within this ditch began to rise. This concurs with the archaeological record at the site that shows a gradual shift of occupation activity away from the modern floodplain from the Iron Age / Roman transition.

4.4. Magnetic ghosts

The location of inhumations remains a challenge for geophysical techniques due to the comparatively small size of the grave cut and the often subtle physical contrast between the buried body and the surrounding body. Graves may even be difficult to identify during excavation where acidic soils can lead to the complete dissolution of skeletal remains [32]. However, recent research demonstrates that human remains may be closely associated with a “shroud” of enhanced magnetic material in the soil surrounding the burial [33]. Figure 11 shows the results from a magnetic susceptibility survey conducted over a partially excavated Anglo-Saxon grave at Lakenheath, Suffolk, with the location of the subsequently revealed skeletal remains superimposed over the image.

The source of the enhanced magnetisation is not entirely clear, although the human body contains approximately 3 to 4g of iron and sufficient organic matter to promote extensive bacterial activity possibly resulting in the production of biogenic magnetite. Magnetic particles extracted from samples of the Lakenheath grave cut certainly indicate the presence of magnetite during low temperature experiments (figure 12(a)). However, transmission electron micrographs of the same material (figure 12(b)) are inconclusive and the X-ray analysis shows strong Fe and O peaks together with significant
quantities of Al, Si, C, S, Ti and Cu. This may suggest a possible mixture of both pedogenic and biogenic sources for the enhanced magnetisation.

**Figure 11.** Results of a topsoil magnetic susceptibility survey conducted over grave number 0589, part of the Anglo-Saxon graveyard excavated at Lakenheath, Suffolk. The location of the subsequently excavated skeletal remains revealed in grave 0589 are also shown superimposed over the interpolated false-colour image of the topsoil magnetic susceptibility data.

**Figure 12.** (a) Variation of low temperature magnetisation for a sample of the grave cut at Lakenheath. The first derivative of the magnetisation curve shows evidence for the Verwey transition of magnetite. (b) Transmission electron micrographs of magnetic particles extracted from the grave cut showing a conglomeration of fine-grained magnetic minerals together with some non-magnetic matrix minerals such as quartz crystals. Results from the X-ray dispersive analysis from a distribution of particles is shown inset.
Figure 13. Isothermal remanent magnetisation acquisition curves and hysteresis loops from the controlled laboratory heating of subsamples of the three soil types recovered at the Yarnton site.

4.5. Unmixing hysteresis data to estimate firing temperatures

Archaeological samples rarely contain a single, dominant magnetic phase but are composed of a mixture of minerals covering a wide grain size range. This complicates the interpretation of hysteresis measurements and often invalidates the classification of such data by comparison with the behaviour of well defined standards, such as the Day plot of the ratios $H_{CR}/H_C$ vs $M_{RS}/M_S$ [15]. However, both hysteresis loops and isothermal remanent magnetisation curves contain considerable information regarding the mixture of magnetic minerals present that may be revealed through more detailed analysis.

Figure 13 shows the magnetic properties of three soil types (a clay rich soil, a sandy loam and a sand and gravel substrate) that were divided into subsamples and subjected to controlled overnight laboratory heating to temperatures from 100 to 500°C. The normalised data shows clear distinctions between the soil types subjected to different heating regimes, for example the isothermal remanent magnetization curves for samples of the clay rich soil heated above 300°C. The hysteresis loops show more subtle variation, such as the gradual dominance of lower coercivity minerals in the sand and gravel samples with increasing temperature, reflected by the alteration of the hysteresis loops from a two component “wasp-waisted” shape to a single component curve [34] [35].

It is proposed that the variation of hysteresis properties with temperature demonstrated by figure 13 may be used as a means of estimating the approximate firing temperature of archaeological sediments recovered from the same site [36]. In this case it is assumed that the hysteresis behaviour of individual magnetic components may be summed linearly to approximate the shape of the magnetisation curve or hysteresis loop of a mixed sample [37]. When the likely range of magnetic behaviour can be estimated through a known end-member data set, such as laboratory heated soil samples, an unmixing algorithm may be applied to interpret the magnetic properties of archaeological sediments.
\[
\begin{pmatrix}
    a_1, a_2, a_3, \ldots, a_m
\end{pmatrix} = \begin{pmatrix}
    x_{1,1} & x_{1,2} & x_{1,3} & \ldots & x_{1,n} \\
    x_{2,1} & x_{2,2} & x_{2,3} & \ldots & x_{2,n} \\
    x_{3,1} & x_{3,2} & x_{3,3} & \ldots & x_{3,n} \\
    \vdots & \vdots & \vdots & \ddots & \vdots \\
    x_{m,1} & x_{m,2} & x_{m,3} & \ldots & x_{m,n}
\end{pmatrix} = \left( \sum_{i=1}^{m} a_i x_{i,1}, \sum_{i=1}^{m} a_i x_{i,2}, \sum_{i=1}^{m} a_i x_{i,3}, \ldots, \sum_{i=1}^{m} a_i x_{i,n} \right)
\]

### Solution \times \text{End Members} = \text{Model}

Where the single row solution matrix, \((a_1, a_2, a_3, \ldots, a_m)\), represents the normalised proportion of each end member, \(x_{i,1}, x_{i,2}, x_{i,3}, \ldots, x_{i,n}\), used to calculate the theoretical model data for comparison with the magnetisation curve or hysteresis loop under investigation. The goal of the unmixing algorithm is to find a solution that produces a model matching the unknown experimental data under the constraints that each value \(a_i\) to \(a_m\) of the proposed solution must be \(\geq 0\) and the sum \(a_1 + a_2 + \ldots + a_m = 1\).

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**Figure 14.** Application of the temperature unmixing model to a selection of samples recovered from archaeological features during the excavations at Yarnton unmixed against the laboratory heated end member data from soils collected from the same site (figure 13). For each sample the measured isothermal remanent magnetisation and hysteresis data is shown superimposed over that calculated by the unmixing model where the quality of fit is indicated by the least means squared regression value, \(R^2\). The distribution of end members selected by the model to match the experimental data is shown in the underlying bar charts together with an estimate of the equivalent exposure temperature.

Figure 14 shows the results of the unmixing algorithm applied to a selection of archaeological sediments where magnetic alteration through burning was expected. A comparison between the model data and the experimental measurements is shown and distribution of end-members in the model is shown as a bar graph separate temperature components. An estimated exposure temperature for each sample is also given based on the weighted average of the constituent end-members. It is clear from the results of the unmixing algorithm that the magnetic properties of sediments recovered from obviously burnt archaeological features are best described by a model dominated by high temperature end-members heated above 400° C. However, it is of interest to note that samples taken from both pit and post-hole features may also contain a significant component of burnt material, perhaps through either the destruction phase of the original timber building or the deliberate burial of ash within a domestic waste pit.
5. Conclusions
Whilst the traditional tool of the archaeologist may still be considered to be the trowel, it is clear that
the naturally occurring nanomagnets can provide considerable assistance when examining human
activity in the past. Like the trowel, geophysical survey using magnetic methods can reveal a wealth of
significant remains and offers the advantage of examining entire archaeological landscapes beyond the
confines of an individual site. Where suitable features are revealed, archaeomagnetic dating provides a
useful complement to other physical dating techniques, such as radiometric methods, and may provide
important local chronologies to interpret prolonged activity at production sites [38]. Understanding the
origin of archaeological magnetisation is also of relevance in determining environmental conditions
and change over archaeological time scales and, perhaps, in revealing the function of specific features
related to high-temperature processes.

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