# 6. Application of Mineral Magnetism in Atlantic Scotland Archaeology 1: Techniques, Magnetic Enhancement and the Identification of Fuel Sources

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*Natural and archaeological deposits tend to contain mixtures of magnetic grains of differing 1) concentrations, 2) domain states (linked to grain size) and 3) mineralogies. Many laboratory based magnetic measurements are available to study these attributes. Fire ash is a significant component in the formation of archaeological deposits in Atlantic Scotland and its magnetic enhancement as a result of heating means it is easily identified and traced. Here several magnetic techniques were developed from which different types of fuel ash could be identified. The techniques were successfully applied to archaeological samples from the Western and Northern Isles of Scotland. The results show a uniformity in the use of well-humified peat as the major fuel source at seven sites on Lewis, whereas considerable variability in fuel types was observed at Cladh Hallan, South Uist and Old Scatness Broch, Shetland.*

## INTRODUCTION

Magnetic susceptibility has become a routine way of analysing archaeological deposits both in-situ and in the laboratory (e.g. Tite 1972; Yates 1988; Clark 1990; Crowther and Barker 1995; Batt and Dockrill 1998; Peters *et al.* 2000). The aim of the present paper is to incorporate susceptibility measurements into a wider range of possible magnetic measurements to gain further insight into the magnetic make-up (in terms of concentration, domain state and mineralogy) of archaeological deposits. The particular focus here is on the role of fire ash within the archaeological record and how detailed magnetic studies can be employed to trace fire ash and investigate possible fuel sources. Further details of the background described below can be found in Thompson and Oldfield (1986), Clark (1990), Dearing (1994), Maher and Thompson (1999) and Walden *et al.* (1999).

## MAGNETISM

Magnetic properties are influenced by 1) the concentration of magnetic grains, 2) their mineralogy and 3) domain state. Usually natural and archaeological deposits contain complicated mixtures of all three attributes. Unravelling the different influences can be problematic, however the range of measurements and examples of applications described below highlight the usefulness of laboratory based magnetic measurements in archaeology.

### Theory

The magnetic measurements described here can be explained in terms of the phenomenon of hysteresis. If an originally unmagnetised ferromagnet is placed in a small magnetic field it acquires a magnetisation. If the field is subsequently removed the magnetisation is lost. However, beyond a certain field, on removal from that field, the magnetisation does not return to zero, and a remanent magnetisation has been acquired. The change in magnetisation lags behind the change in field. This phenomenon is known as magnetic hysteresis. As an increasingly larger field is applied the magnetisation increases, until a point is reached when an increase in the applied field produces no increase in magnetisation. Saturation has then been reached. Measurements can be made when the applied magnetic field has been removed from the samples or alternatively in the presence of a magnetic field

## Domain state

Very small magnetic grains exhibit superparamagnetic (spm) characteristics in which thermal energies are similar to magnetic energies. As magnetic grains increase in size, their magnetic energies dominate over the thermal energies. Within these grains are regions called domains, which are magnetised uniformly in one direction. Grains that are slightly larger than spm size show a uniform direction of magnetisation. These grains contain only one domain and are referred to as single-domain (sd). As the grain size increases the various magnetic energies compete to produce an overall lower total energy. As a result of this competitive process domains form, separated by domain (or Bloch) walls. Grains slightly larger than sd grains are referred to as pseudo-single-domain (psd). Grains that contain ten or more domains are referred to as true multi-domain (md) grains. The grain sizes at which the boundaries between the different domain states occur vary for different minerals and in some cases the exact grain sizes at which the boundaries occur are still uncertain.

The different domain states (spm, sd, psd, md) exhibit different magnetic characteristics. In the presence of a magnetic field the magnetisation of spm grains can become aligned in the direction of the field and a magnetisation is observed. On removal of the field the thermal energies re-orientate the magnetisation and no remanent magnetisation is observed. In comparison sd grains have a very stable magnetisation. In the presence of an applied field the domain walls within md grains move to allow the magnetisation to be orientated in the direction of the field. On removal of the field the domains reform with different orientations until a minimum in the total energy is found. This minimum will not be that of the original unmagnetised state, some of the domains will still be orientated in the direction of the field, hence a weak remanent magnetisation is observed. Overall it is easy to magnetise and demagnetise md grains, and hard to alter the magnetisation of sd grains. Psd grains show a continual variation between the sd and md characteristics. Spm and md grains are referred to as being magnetically 'soft', whereas sd grains are magnetically 'hard'.

## Mineralogy

The magnetic mineralogy of soils is complicated and not fully understood! Many factors control the nature and distribution of magnetic grains within soils including parent material, climate, biological action, drainage, relief, time, atmospheric fall-out and fire (Maher 1986). Fire can alter the magnetic signature in two ways; (i) heating of substrates and (ii) the ashing of fuels such as peat. The main magnetic minerals found within soils are the iron oxides and oxyhydroxides, which include maghaemite, magnetite, haematite and goethite. Other doped (e.g. with titanium, aluminium or manganese) forms of these minerals forming solid solution series can also be present in significant quantities. These minerals can exist within soils as discrete particles, as aggregated concretions, or as very fine-grained material coating other grain or void surfaces (Taylor *et al.* 1983; Maher 1986).

Magnetite ($Fe_3O_4$) has a cubic inverse spinel structure and is ferrimagnetic. It has the highest magnetisation of all magnetic minerals and is magnetically soft. Haematite ($\alpha$-$Fe_2O_3$) forms in the corrundum structure and is basically anti-ferromagnetic. However due to spin-canting a small net magnetisation is observed. In comparison to magnetite it is a very weak magnetic mineral and is magnetically hard. Maghaemite ($\gamma$-$Fe_2O_3$) has the same composition as haematite, but the cubic structure of magnetite. It is very difficult to distinguish between maghaemite and magnetite. Goethite ($\alpha$-FeOOH) forms in an orthorhombic structure. It is only weakly magnetic, but is magnetically very hard. It is uncertain whether magnetite or maghaemite is the most abundant ferri-magnet within soils and although magnetite/maghaemite dominate the magnetic signal, haematite and goethite are more abundant. Further general details on magnetic mineralogy can be found in Thompson and Oldfield (1986).

## MAGNETIC MEASUREMENTS

There are two main types of magnetic measurements; those measured in a magnetic field and those measured after removal from an applied field. Susceptibilities are measured in the presence of a magnetic field, whereas remanent magnetisations are measured after removal from an applied magnetic field.

### Susceptibilities

Magnetic susceptibility is a measure of how easily a material can be magnetised. The samples are placed in a small alternating magnetic field of about 0.1mT peak (comparable to the Earth's magnetic field of 0.06mT), and the response to the magnetic field (which depends upon the nature and concentration of the magnetic grains) is displayed on the meter. The most commonly used susceptibility bridge is the Bartington MS2 system (Dearing 1994). There are several approaches to measuring susceptibility incorporating differences in temperatures over which the measurements are made and also in the a.c. frequency. The main ways are:

### Specific susceptibility

Specific susceptibility ($\chi$) is the easiest and most fre-quently measured magnetic parameter. $\chi$ is defined to be the ratio of the induced low frequency magnetisation to the applied magnetic field and on a mass specific basis is measured in units of $m^3kg^{-1}$. $\chi$ gives a rough indication of

magnetic concentration. It can be used as a ratio with other magnetic parameters to study domain state and mineralogy variations. It can also be plotted on its own as a function of depth to allow comparison between samples in a profile, or alternatively plotted two dimensionally on a grid.

*Frequency dependent susceptibility*
Measurement of susceptibility at different frequencies can be used to detect the presence of spm grains, which are common in archaeological samples. Using the Bartington susceptibility bridge measurements are made at 0.47 and 4.7 kHz. Samples containing spm grains will have proportionally lower values of susceptibility at high frequency (hf) compared to low frequency (lf). To allow direct comparison between samples the parameter frequency dependent susceptibility, $\kappa_{fd}$, is calculated, and expressed as a percentage, as follows;

$$\kappa_{fd} = ( \; (lf - hf) \; / \; lf \; ) \times 100$$

Dearing (1994) has classified $\kappa_{fd}$ values into bands; low values of <2% are interpreted as containing virtually no spm grains, probably <10%. Medium $\kappa_{fd}$ values of 2 – 10% are a mixture of spm and larger grains. High $\kappa_{fd}$ values of 10 – 14% are virtually all spm grains, probably >75%. Very high $\kappa_{fd}$ values of >14% are interpreted as either erroneous measurements, anisotropy, weak sample or metal contamination.

*Variation of susceptibility with high temperature*
Monitoring the variation of magnetic susceptibility with increasing temperature (up to 700°C) provides insight into the magnetic mineralogy and to some extent domain state. As magnetic minerals are heated their thermal energies begin to dominate over their magnetic energies and at a specific temperature, their Curie Temperature ($T_c$), the minerals effectively loose their magnetisation and a drop in susceptibility is observed. Heating the samples up to 700°C may cause chemical alteration, thus unlike the other measurements described here, high temperature susceptibility measurements can be sample destructive. However, the resulting heating and cooling curves are good for comparison between samples and comparing pre- and post-heating susceptibility values can give an indication of whether or not a sample has previously been heated (an increase in susceptibility after heating suggests that there are components within the sample which have not previously been heated to elevated temperatures). The $T_c$s of the common magnetic minerals magnetite and haematite are known to be 580°C and 675°C respectively and can be easily identified from the high temperature susceptibility curves. Impurities in the minerals can alter the $T_c$s e.g. the substitution of titanium in magnetite reduces $T_c$, in fact the $T_c$ of titanomagnetites range from −153 to 580°C.

## Remanent magnetisations

On removal from a susceptibility bridge, the small magnetisation induced in a sample during measuring is lost. However if a large enough magnetic field is applied to sd, psd or md grains, a magnetisation can be retained after removal of the field. Grains of different mineralogies or domain states can display varying responses to the application of magnetic fields. The responses can be highlighted by applying the magnetic fields in different ways or over a range of field strengths or temperatures. Two of the most commonly measured remanent magnetisations, isothermal and anhysteretic, are described below, in addition to low temperature thermo-remanence magnetisations.

*Isothermal remanent magnetisations*
Isothermal remanent magnetisations (IRMs) are the simplest type of remanent magnetisation. On a mass specific basis they are measured in units of $Am^2kg^{-1}$. They are grown by placing a sample in a direct field and, after removing the field, measuring the remaining magnetisation. Here a pulse magnetiser and electromagnets were used to grow IRMs in fields of 60mT and 1T respectively and they were measured using a fluxgate spinner magnetometer. The maximum IRM attained by a sample is referred to as the saturation IRM (SIRM) and gives an indication of magnetic concentration. Usually a field of 1T is sufficient to saturate samples (with the exception of samples containing haematite and goethite), thus here we refer to the IRM grown in a field of 1T as SIRM. Mass specific SIRM allows a direct comparison between samples based on concentration. Mineralogical and domain state information can be obtained by studying the variation of IRMs with changing field values. IRM acquisition curves are obtained by growing IRMs in successively higher fields. Normalisation of the IRM acquisition data by SIRM allows direct comparison between samples. Here IRM acquisition curves are represented by the single magnetic ratio of $IRM_{60mT}/SIRM$. Further IRM curves can be measured by either direct field demagnetisation of SIRM (often referred to as back IRMs) or alternating field demagnetisation of SIRM. The shape of the curves can be characteristic of particular minerals (cf. Peters and Thompson 1998). For each magnetic mineral it is easier to magnetise md rather than sd grains (spm grains do not contribute to remanent magnetisations), thus the ratio $IRM_{60mT}/SIRM$ will be higher for md compared to sd grains.

*Anhysteretic remanent magnetisations*
Anhysteretic remanent magnetisations (ARMs) are more complicated than IRMs. Although the ARM measurements are described here after IRMs, if a suite of magnetic measurements are to be carried out, it is good practice to measure ARMs before IRMs. ARMs are grown by superimposing a small direct field onto a larger, smoothly

decreasing, alternating field. Here ARMs have been grown using an adapted Molyneux AF demagnetiser (0.1mT direct, 99mT peak alternating) and measured using a fluxgate spinner magnetometer. The resulting ARM is referred to as the saturation ARM (SARM). ARMs can not be grown in high stability magnetic minerals such as haematite and goethite as the magnetic fields used here (limited by the equipment) are too small. Thus SARM gives an indication of the concentration of the soft magnetic minerals e.g. magnetite or maghaemite. Further information regarding the domain state and mineralogy can be obtained by step-wise alternating field demagnetisation of SARM. Here the ratio ARM $_{demag\ 40mT}$ / SARM was selected to represent the demagnetisation data. It is easier to demagnetise md than sd grains, thus ARM $_{demag\ 40mT}$ / SARM ratios will be lower for md than sd grains. (To allow direct comparison between ARM values acquired in different direct fields it is recommended that the parameter $\chi_{ARM}$ (ARM normalised by the direct field value) is used.)

*Low temperature remanences*
Low temperature magnetic measurements can give further information on concentration, mineralogy and domain state. In particular the Verwey transition in magnetite, where a drop in magnetisation with cooling is observed at 150K and the Morin transition in haematite, where an increase in magnetisation with cooling at ~263K is observed, thus allowing identification of these two minerals. Spm grains can be recognised by their blocking characteristics. Here low temperature thermo-remanence curves were carried out using a MPMS$_2$ superconducting SQUID magnetometer. Using the MPMS$_2$ supercon-ducting magnet a magnetic remanence was imparted in the samples using a field of 1T at 300K. The samples were then cooled in a zero field to 20K while the remanent magnetisation (M$_R$) was monitored (cf. Linford 2000; Peters *et al.*, 2002).

**Additional measurements**

For completeness other types of measurements which have successfully been applied to archaeological samples include low temperature susceptibility measurements and hysteresis loops (e.g. Peters 1995; Peters and Thompson 1999). Low temperature susceptibilities are measured by cooling a sample in liquid nitrogen and monitoring the variation in susceptibility as the sample is allowed to warm back to room temperature. The measurements can be carried out using the Bartington MS2 bridge with the sensor used to monitor susceptibilities at high temperatures. Similar to the low temperature remanence measurements, the Verwey and Morin transitions and spm characteristics can be recognised in the resulting susceptibility curves. Hysteresis loops can provide inform-ation on concentration, domain state and mineralogy (cf. Peters 1995). They can be measured using several currently available instruments including vibrating sample magnetometers, alternating gradient magnet-ometers and alternating field translation balances. Hysteresis loops can provide a lot of information on the magnetic make-up of samples and their application to archaeology, in particular the study of fire ash, is currently being explored.

**Sampling and measuring procedures**

Two types of sampling strategies can be employed in the mineral magnetic study of archaeological deposits; (i) spot sampling, where one sample per context is measured (archived samples can be used) and (ii) detailed sampling in the form of profiles (e.g. Peters *et al.* 2000) or grids (e.g. Smith *et al.* 2001). For a general assessment of samples it is good procedure to carry out the magnetic measurements on bulk samples dried and sieved at 2mm. For specific studies it may be necessary to measure different grain size fractions e.g. in the identification of fuel sources the <63µm grain size fraction is measured. For the room temperature measurements of susceptibility, ARMs and IRMs samples are tightly packed into 2cm cylindrical plastic pots; typically ~15g of sample is required to fill the pot. Smaller subsamples are loosely packed into a silica glass sample holder for high temperature susceptibility measurements and very small samples (~60mg) are put into gel capsules for the low temperature remanence measurements. The order in which measurements are generally carried out is room temperature susceptibility, then ARMs followed by IRMs. Two subsamples can then be taken from the pots for the temperature measurements. Two measurements of each of susceptibilities, ARMs and IRMs can be carried out on a single sample in 5–10 minutes. High temperature susceptibility measurements take 90 minutes for a heating and cooling cycle per sample and a low temperature remanence curve takes three hours. Further general details regarding magnetic measuring procedures and analysis of magnetic data can be found in Thompson and Oldfield 1986 and Walden *et al.* 1999.

TRACING FIRE ASH

The strong magnetic signal commonly associated with archaeological deposits is mainly due to fire ash (although metalworking residues can also produce a strong magnetic signal e.g. Dewar *et al.* 2002). Fire ash is a significant component in site formation in Atlantic Scotland and it can be found within many context types; abandoned hearths, trampled into floor surfaces, dumped on middens or alternatively used to amend soil in field systems. Tracing fire ash can help to link features within a site (cf. at Galson; Peters *et al.* 2000), aid in the understanding of site formation, human exploitation of natural resources, burnt ecofact taphonomy (Church and Peters, this volume)
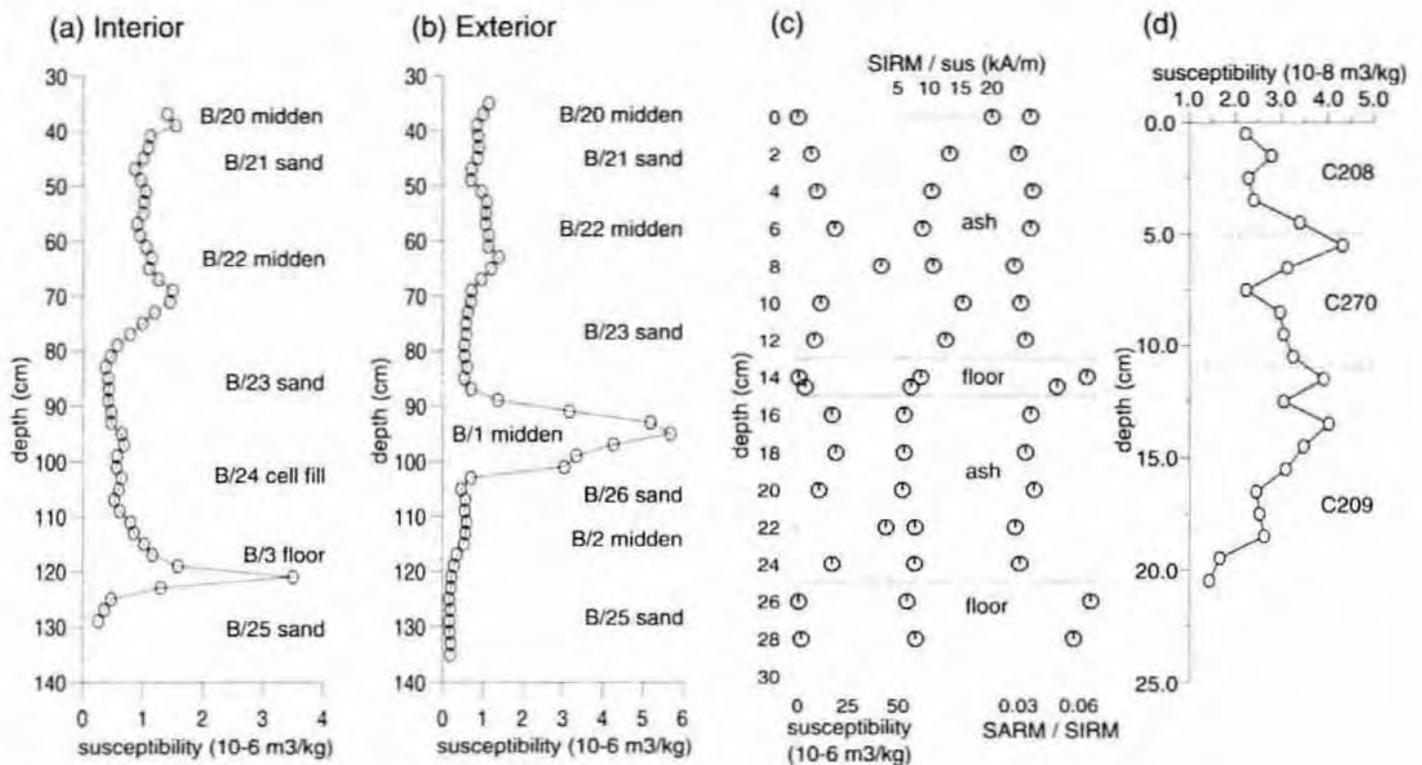
Fig. 1. Profiles of magnetic data. (a) and (b) are profiles of $\chi$ ($10^{-6}m^3kg^{-1}$) for the interior (profile MS1) and exterior (profile MS2) respectively from structure B at Galson. (c) displays profiles of the magnetic parameters $\chi$ ($10^{-6}m^3kg^{-1}$), SIRM/$\chi$ (kAm$^{-1}$) and SARM/SIRM for the Cladh Hallan hearth. (d) is $\chi$ ($10^{-8}m^3kg^{-1}$) for the rig profile from Guinnerso, in which C208 is peaty turf and upper fibrous peat, C270 is gritty, peaty silt, forming anthropogenically enhanced soil within the rig and C209 is fibrous-upper peat gradually changing to well-humified peat.

and help reconstruct house activity areas (Smith *et al.* 2001). In this section we will explore different ways of displaying magnetic data in relation to assessing the overall magnetic make-up of deposits and differences between context types.

The data presented here are from the sites of Galson and Guinnerso on the Isle of Lewis and from Cladh Hallan on South Uist. At Galson coastal erosion has revealed two major levels comprising of a number of Iron Age burial cists (Neighbour *et al.* 2000) overlain by domestic structures with associated middens and palaeosols of Iron Age to Medieval date (Neighbour and Church 2001). Samples were collected from the upper level for the purposes of mineral magnetic studies (Peters *et al.* 2000). Two sampling strategies were employed; initially twelve spot samples were collected from specific features e.g. middens, a floor and a hearth. Subsequently detailed vertical profiles, at 2cm intervals were taken through the interior of structure B (profile MS1), the exterior of the same structure (profile MS2) and a hearth in structure C (profile MS3). Calibrated radiocarbon dates from an external midden (context B/1) of structure B date its accumulation to the 10th century AD, whilst radiocarbon dates from the floor level (context B/3) from structure B, places its occupation in the 11th to 12th centuries AD. Therefore it seems likely that structure B was built into

a slightly earlier midden (B/1). A single radiocarbon date from the hearth in structure C placed the occupation in the 5th to 6th centuries AD, the late Iron Age of the region. At the multi-period site of Guinnerso (Church and Gilmour 1999), situated within extensive moorland, a detailed (1cm intervals) vertical profile was taken through a relic field system rig associated with 15th-17th century radiocarbon dated late Medieval activity, to investigate any amendment as a result of the addition of fire ash. At Cladh Hallan (Marshall *et al.* 2000) a detailed (2cm intervals) vertical profile was taken through a series of hearths located in the center of a circular dwelling (House 401) of late Bronze / early Iron Age (Peters and Batt 2002). The measurements were carried out on bulk (<2mm) samples.

Figure 1 displays four profiles of magnetic data from Galson (a and b), Cladh Hallan (c) and Guinnerso (d). Figures 1(a) and 1(b) are profiles from the interior and exterior of structure B at Galson. The magnetic parameter $\chi$ has been plotted in both profiles. $\chi$ gives a rough indication of magnetic concentration and here we will assume that it is proportional to the amount of fire ash. We see that the floor level within the structure (context B/3) and one of the middens from the exterior (context B/1) have significantly higher magnetic concentrations than the other midden, cell fill and sand contexts. The

high $\chi$ values suggest that ash from the hearth in the structure has been trampled onto the floor surface. Comparing the $\chi$ values of the floor (B/3) and the external midden (B/1), we estimate that a similar proportion of ash was deposited on both. The variation in $\chi$ between midden B/1 and the other three middens in the profile reflects differing formation processes and the amount of ash dumped. (It is interesting to note the correlation in the increase in $\chi$ over the boundary of contexts B/23 and B/22 in both the interior and exterior of the structure giving an example of how magnetic data can be used to correlate between different sections of a site.)

In Figure 1(c) three magnetic parameters ($\chi$, SIRM/$\chi$ and SARM/SIRM) have been plotted as a function of depth for the series of ash build-ups and floor levels within the central hearth at Cladh Hallan. We see that the floor levels are characterised by low $\chi$ values compared to the ash layers i.e. lower magnetic concentration. The floor levels are also characterised by high SARM / SIRM ratios compared to the ash layers. Comparing the two ash layers, the upper ash build-up has higher SIRM / $\chi$ values than the lower one. SIRM / $\chi$ can reflect differences in mineralogy and/or domain state. These differences could be a result of different fuel types / burning procedures producing different magnetic signatures.

In Figure 1(a), (b) and (c) we have seen how contexts containing fire ash components can be identified by their high $\chi$ values. Figure 1(d) shows the $\chi$ values of the rig profile from Guinnerso. Note that the units in (a)-(c) are $10^{-6} m^3 kg^{-1}$, whereas in (d) they are $10^{-8} m^3 kg^{-1}$. If fire ash had been used to amend the soil within the rig, we would expect to see a large increase in the $\chi$ values of context C270, the anthropogenically enhanced soil. As no increase is observed we can conclude that fire ash, in this instance, was not used to amend the soil. Bulk sampling and soil micromorphology confirmed that the only addition to the soil was small pieces of Lewisian gneiss, the local basement rock.

Figure 2 displays an alternative way of plotting magnetic data by using biplots. The data presented is listed in Table 1 and was measured on fifteen samples from the site of Galson, including examples of hearth, floor, midden, sand, cell fill and till samples. In the four biplots of Figure 2, the hearth samples and the till sample are seen to be the extremes. The hearth samples are characterised by high $\chi$ and SARM values (high magnetic concentration), low $ARM_{demag\ 40mT}$ / SARM, low SIRM / $\chi$ and high $IRM_{60mT}$ / SIRM ratios (magnetically soft) and low SARM / $\chi$ and SARM / SIRM values. In comparison the till sample has a low magnetic concentration and is magnetically hard. The high SARM / $\chi$ and SARM / SIRM values, in the case of the till sample, are likely to be due to differences in mineralogy / domain state. (Dalan and Banerjee 1998 have used a combination of ARM and $\chi$ to investigate grain size variations in relation to archaeological soils.) The midden, floor, cell fill and

sand samples display a range of magnetic characteristics. One of the sand samples (from context B/25 at the base of the profile cf. Figure 1) displays characteristics approaching those of the till, emphasising it is natural in origin. The two floor samples and one of the midden samples display characteristics approaching those of the hearths, highlighting the ash component within the samples, but also showing that there are non-ash components contributing to the overall magnetic signal. The remainder of the midden and sand samples group together, with characteristics suggesting no significant ash component in the midden samples, yet some form of anthropogenic input to the sands as they are different from the till and basal sand. The cell fill displays similar characteristics to these midden and sand samples.

## SOURCING FIRE ASH

### Identification of fuel types

The aim is to use the magnetic signature of experimentally produced fire ash of different fuel types to identify fuels used in prehistory. The outcome of heating experiments are dependent on many variables; peak temperature, rate of heating and insertion temperature, length of time at peak temperature, rate of cooling, heating and cooling atmospheres and crucible size (Oldfield *et al.* 1981). Replication of the magnetic properties of fire ash recovered from archaeological sites has not been achieved by heating experiments carried out in the laboratory (Peters unpublished data). Here the development of magnetic techniques for the identification of prehistoric fuel usage has been based on directly replicating domestic prehistoric burning procedures in a field-based situation thus reducing the need to consider the heating variables listed above. Three three-sided domestic hearths, based on excavated evidence from the late Iron Age site of Bostadh, in Great Bernera, Lewis (Neighbour and Burgess 1997) were reconstructed at Calanais Farm, Lewis. Following controlled and repeated burning of different fuel types (well-humified peat (whp), fibrous-upper peat (fup), peat turf (pt) and wood(wd)) mineral magnetic measurements were carried out on bulk (<2mm) and sieved (<63µm) fire ash residues. Several techniques have been developed based on the magnetic data to discriminate between the ash of the different fuel types (Peters *et al.* 2001; Peters *et al.* 2002). Figure 3 summarises the techniques. In Figure 3(a) $\chi$ and $\kappa_{fd}$ have been plotted for the twenty three sieved ash samples. We observe complete discrimination between the four fuel types in the biplot. In order to make use of all the room temperature susceptibility, ARM and IRM data recorded for the ash samples, stepwise discriminant analysis was carried out on the data for both the bulk and sieved samples. Figure 3(b) displays the resulting biplot. We observe good discrimination of the wood and well-humified peat ash
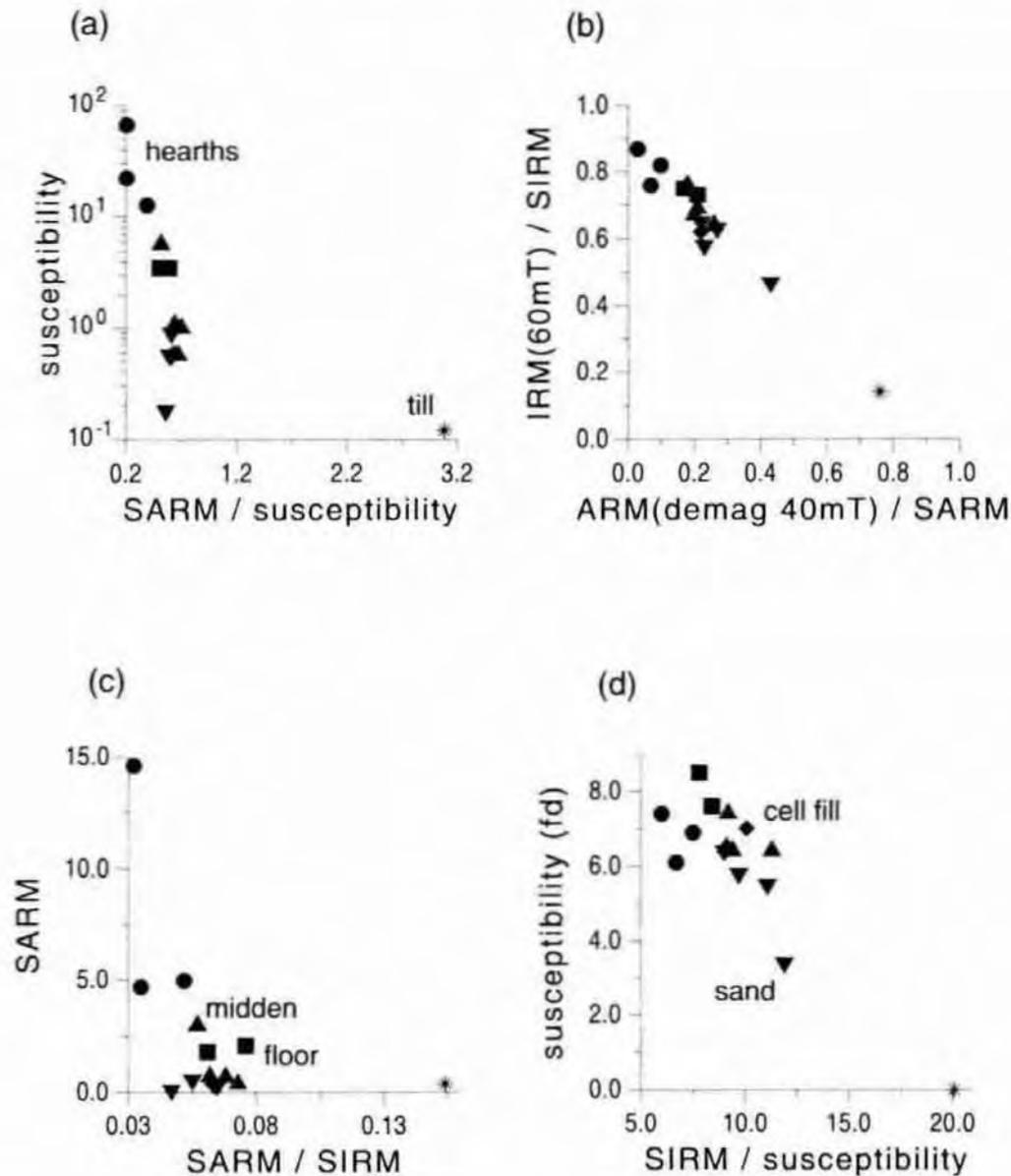
(a)

(b)

(c)

(d)

Fig. 2. Biplots of magnetic parameters for fifteen samples from the site of Galson, Isle of Lewis. In all four biplots the hearth samples are represented by circles, floor samples by squares, midden samples by triangles, sand samples by inverted triangles, cell fill by a rhombus and till by an asterisk. Units are listed in Table 1.

and some overlap between the fibrous-upper peat and peat turf ash. The third magnetic technique is monitoring the variation of susceptibility with temperature up to 700 °C (Figures 3(c) and (d)). The fibrous-upper peat and peat turf ash display a single magnetic component with a drop in magnetic susceptibility at ~ 600 °C e.g. Figure 3(d). In comparison the well-humified peat and wood ash display one, sometimes two, magnetic components, characterised by drops in susceptibility at ~ 330 and/or ~ 550 °C e.g. Figure 3 (c). The final magnetic technique is monitoring the variation in magnetic remanence as a sample is cooled from room temperature to 20K (M_R − T curves). Figure 3(e) displays a curve characteristic of well-humified peat / wood ash. These samples show an increase in remanence with cooling, attributed to the blocking characteristics of spm grains. In comparison, Figure 3(f), which is characteristic of fibrous-upper peat

and peat turf ash, decreases with cooling, displaying a smooth Verwey transition characteristic of small stable grains.

**Applications to archaeological samples**

The fuel type identification techniques can be applied to archaeological samples. Due to the mixed nature of archaeological deposits, samples were sieved to 63 μm, isolating the ash component from sand and other large clasts (Peters et al. 2001).

The samples described in this section are from seven sites (Dun Bharabhat, An Dunan, Cnip, Guinnerso, Loch na Beirgh, Bostadh and Galson) on the Isle of Lewis, Cladh Hallan on South Uist and Old Scatness Broch on Shetland. The sites from Lewis are described in more detail in Church and Peters (this volume). Old Scatness,
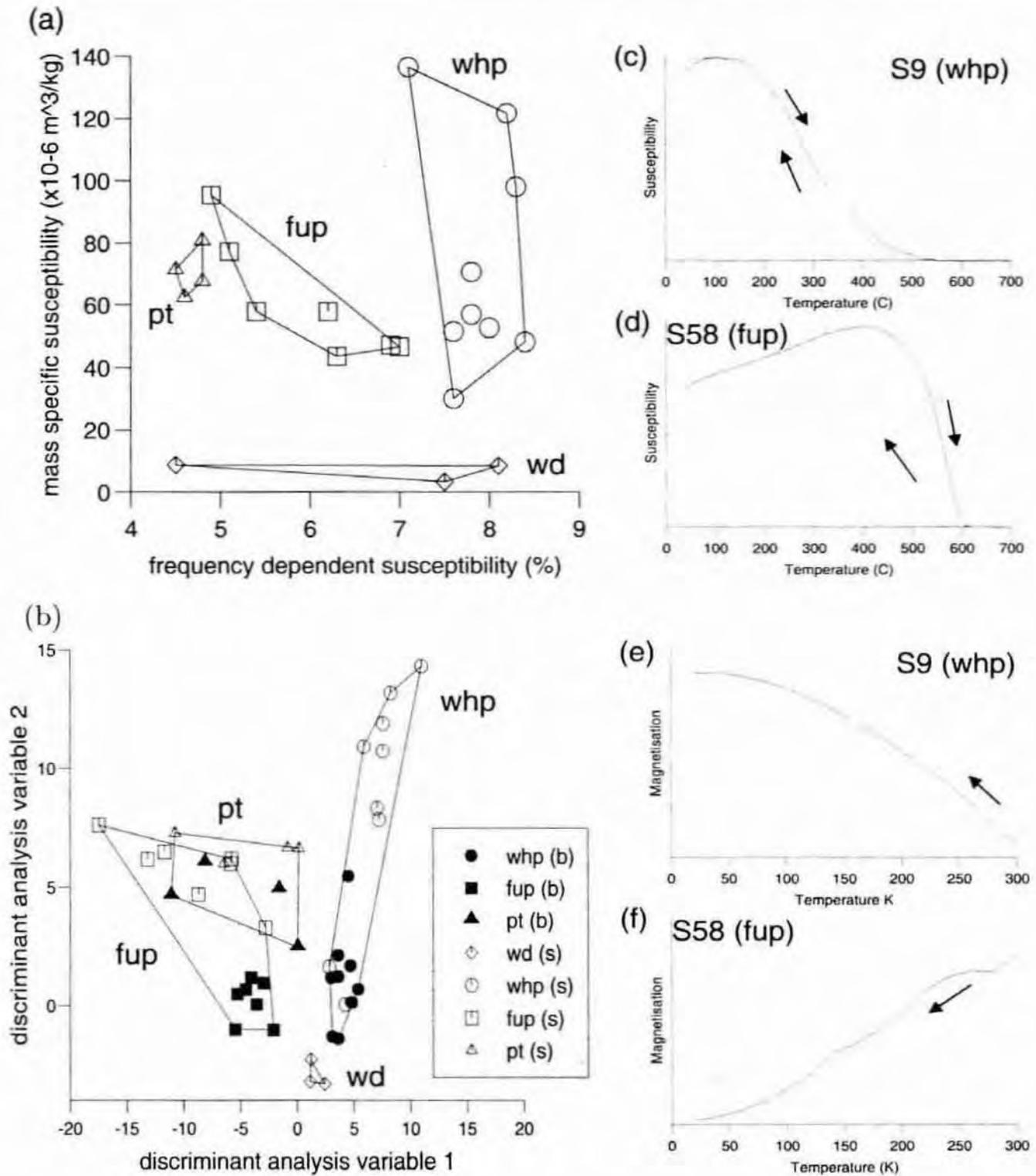
Fig. 3. Techniques for discriminating the modern fuel ash residues. (a) is the biplot of $\chi$ versus $\kappa_{fd}$ for the 23 sieved ash samples (Peters et al. 2002). (b) is the discriminant analysis biplot for the bulk (b) and sieved (s) ash samples (Peters et al. 2001). (c) and (d) are the high temperature susceptibility curves for the well-humified peat sample S9 and the fibrous-upper peat sample S58, respectively. (e) and (f) are the $M_R - T$ curves for the samples S9 and S58 respectively.

Shetland, is a multi-period settlement mound consisting of a broch surrounded by circular features, cellular structures and wheelhouses, together with Pictish, Norse and Post-Medieval settlement (Nicholson and Dockrill 1998; Dockrill and Batt this volume).

Figure 4 displays the result of applying the dis-

criminant analysis biplot technique to samples from the nine sites. Figure 4 (a) shows the results from the seven Lewisian sites. A range of hearth and ash spread samples have been studied from the sites. The samples from Guinnerso have been divided into those from the Iron Age and those from Medieval dates. The predominant

*Table 1. Examples of mineral magnetic data for fifteen samples from the site of Galson, Isle of Lewis. A40A is defined as the ratio $ARM_{demag\ 40mT}$ /SARM, 160S is $IRM_{60mT}$ /SIRM, SISU is SIRM/$\chi$ SASI is SARM/SIRM and SASU is SARM/$\chi$.*

| Sample | $\chi$ $10^{-6}m^3kg^{-1}$ | $\kappa_{fd}$ -% | SARM mAm²kg⁻¹ | SIRM mAm²kg⁻¹ | A40A | 160S | SISU kAm⁻¹ | SASI | SASU kAm⁻¹ |
|---|---|---|---|---|---|---|---|---|---|
| Hearth B/4 (GA11) | 12.73 | 6.9 | 4.99 | 95.7 | 0.10 | 0.82 | 7.5 | 0.052 | 0.39 |
| Hearth (MS3–6) | 68.0 | 6.1 | 14.6 | 458.9 | 0.03 | 0.87 | 6.7 | 0.032 | 0.21 |
| Hearth (MS3–16) | 22.3 | 7.4 | 4.70 | 135.1 | 0.07 | 0.76 | 6.0 | 0.035 | 0.21 |
| Floor B/3 (MS1–43) | 3.50 | 7.6 | 1.80 | 29.6 | 0.21 | 0.73 | 8.4 | 0.061 | 0.51 |
| Floor B/3 (GA8) | 3.50 | 8.5 | 2.08 | 27.2 | 0.17 | 0.75 | 7.8 | 0.076 | 0.59 |
| Midden B/1 (MS2–31) | 5.69 | 7.4 | 2.97 | 52.4 | 0.18 | 0.76 | 9.2 | 0.057 | 0.52 |
| Midden B/2 (MS2–40) | 0.57 | 6.5 | 0.38 | 5.21 | 0.26 | 0.64 | 9.1 | 0.073 | 0.67 |
| Midden B/20 (MS2—2) | 1.00 | 6.4 | 0.70 | 11.3 | 0.21 | 0.69 | 11.3 | 0.062 | 0.70 |
| Midden B/22 (MS2–11) | 1.08 | 6.4 | 0.69 | 10.2 | 0.20 | 0.67 | 9.4 | 0.068 | 0.64 |
| Sand B/21 (MS2–5) | 0.90 | 5.5 | 0.55 | 9.97 | 0.22 | 0.65 | 11.1 | 0.055 | 0.61 |
| Sand B/23 (MS2–21) | 0.58 | 6.4 | 0.34 | 5.20 | 0.23 | 0.58 | 9.0 | 0.065 | 0.59 |
| Sand B/25 (MS2–49) | 0.18 | 3.4 | 0.10 | 2.14 | 0.43 | 0.47 | 11.9 | 0.047 | 0.56 |
| Sand B/26 (MS2–37) | 0.57 | 5.8 | 0.35 | 5.51 | 0.27 | 0.63 | 9.7 | 0.064 | 0.61 |
| Cell Fill B/24 (MS1–35) | 0.60 | 7.0 | 0.38 | 6.06 | 0.22 | 0.62 | 10.1 | 0.063 | 0.63 |
| Till | 0.12 | 0.0 | 0.37 | 2.41 | 0.76 | 0.14 | 20.1 | 0.154 | 3.08 |

fuel source used in Lewis throughout the time span of the studied sites is well-humified peat. Some evidence of wood burning (e.g. at Guinnerso; Peters *et al.* 2001) and the use of peat turf / fibrous-upper peat (e.g. at Dun Bharabhat; Church and Peters 2000) is also observed. The samples plotting to the right of the well-humified peat envelope may indicate a very localised trend in fuel source type from the Bhaltos Peninsula (the sites of Dun Bharabhat, Cnip and Loch na Beirgh). In Figure 4(b) the results are shown for the upper and lower ash sections (cf. Figure 1) from Cladh Hallan. We observe a marked change in fuel type between the two sections. The lower section is predominantly well-humified peat ash, whereas the upper section has a more mixed nature trending towards peat turf / fibrous-upper peat ash. Figure 4(c)

displays results from the site of Old Scatness Broch. The samples selected are from two horizontal profiles taken through fuel-ash rich middens infilling a central piered roundhouse (Structure 12), of 10m in internal diameter. Five contexts were sampled. The results in Figure 4(c) show a very 'mixed' use of fuel sources, with no apparent correlation to context.

Figure 5 displays the high temperature susceptibility curves for selected samples from Lewis, Cladh Hallan and Old Scatness Broch. Figures 5(a) and (b) are samples from Bostadh and Guinnerso respectively. Both curves display characteristics of well-humified peat / wood ash. In Figure 5(c) the profile, with depth, of the high temperature susceptibility curves for the twelve ash samples from Cladh Hallan is shown. There is a clear
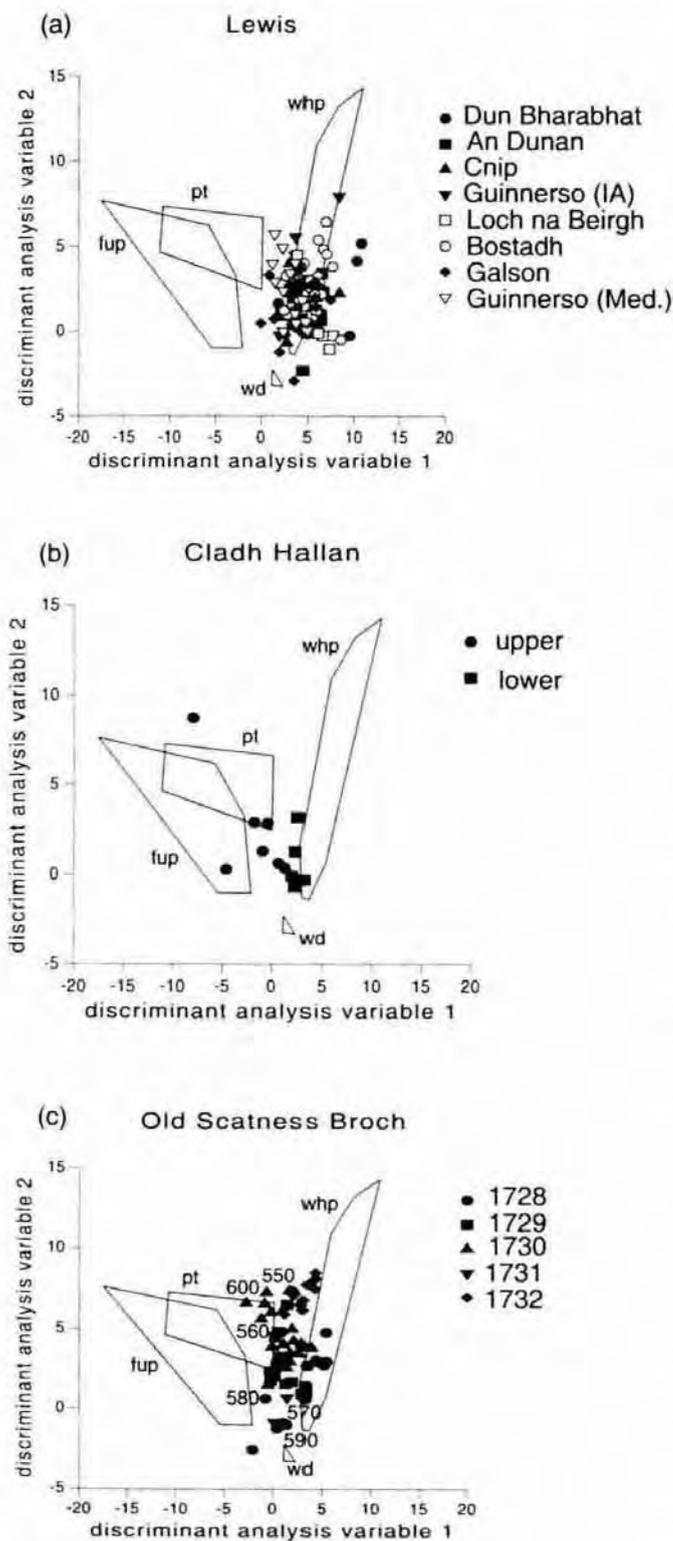
(a)

Lewis



(b)

Cladh Hallan



(c)

Old Scatness Broch



*Fig. 4. Application of the discriminant analysis biplot of Peters et al. (2001) to samples from (a) Lewis, (b) Cladh Hallan and (c) Old Scatness Broch. In (b) 'upper' and 'lower' refer to the two ash sections in Fig. 1(c). In (c) the numbers 1728 – 1732 are the context numbers and 550 – 600 are $T_c$ values in °C.*

difference in the curves between the upper and lower sections. The lower section curves, with the drops in susceptibility at ~330°C and almost total loss of

susceptibility by 550°C are characteristic of well-humified peat / wood ash. In comparison the upper curves, with loss of susceptibility approaching 600°C are characteristic of peat turf / fibrous-upper peat ash. Figures 5(d) and (e) are samples 10278 (context 1732) and 10334 (context 1728) showing the range of observed curves from Old Scatness Broch. All the 50+ Old Scatness Broch samples for which high temperature susceptibility measurements were taken display drops in susceptibility at temperatures greater than or equal to 550°C. A few of the $T_c$ temperatures have been indicated on the discriminant analysis biplot in Figure 4(c). We would expect to see the higher $T_c$s plotting in the peat turf / fibrous-upper peat ash region and the lower $T_c$s plotting in the well-humified peat / wood region, but no such correlation is observed. These results suggest that the fuel source(s) used at Old Scatness Broch does not correspond to one used in the experimental burning at Calanais i.e. fire ash from different peat localities may produce different magnetic signals.

**Modelling fuel ash components**

The low temperature thermo-remanence curves (Figures 3(e) and (f)) display differences between the fuel ash types fibrous-upper peat / peat turf and well-humified peat / wood. Chemical alteration does not occur in the low temperature measurements, unlike the high temperature susceptibility measurements, therefore using the specific low temperature remanence data of the modern ash residues it should be possible to quantify two fuel ash components within archaeological samples. An umixing algorithm was developed to quantify fuel ash components (Peters *et al.*, 2002). The low temperature curves of the 23 modern ash residues were used as end-members in the unmixing. After regression the end-member coefficients were summed to give overall totals of fibrous-upper peat / peat turf ash and well-humified peat / wood ash within the archaeological samples. Figure 6 displays four examples of the $M_R$ – T measured and modelled curves. In Figure 6(a), context 744 from Bostadh, and Figure 6(c), sample S11 from Cladh Hallan, we observe that both contain 100% well-humified peat / wood ash. The other Cladh Hallan sample, S5, Figure 6(d), shows an increase in remanence with cooling to a sharp peak and then drop in remanence to 20K. The increase is characteristic of well-humified peat / wood ash, whereas the decrease is characteristic of fibrous-upper peat / peat turf ash. The unmixing algorithm has found a best fit of 84% well-humified peat / wood ash and 16% fibrous-upper peat / peat turf ash. The final example, Figure 6(b), sample 10334 from Old Scatness Broch has been estimated by the unmixing algorithm to be composed of 50% of both fuel components.

Magnetic unmixing techniques have been successfully applied in many environmental situations e.g. in the study of river catchments (e.g. Peters 1995). This preliminary attempt at unmixing fuel ash components using magnetic
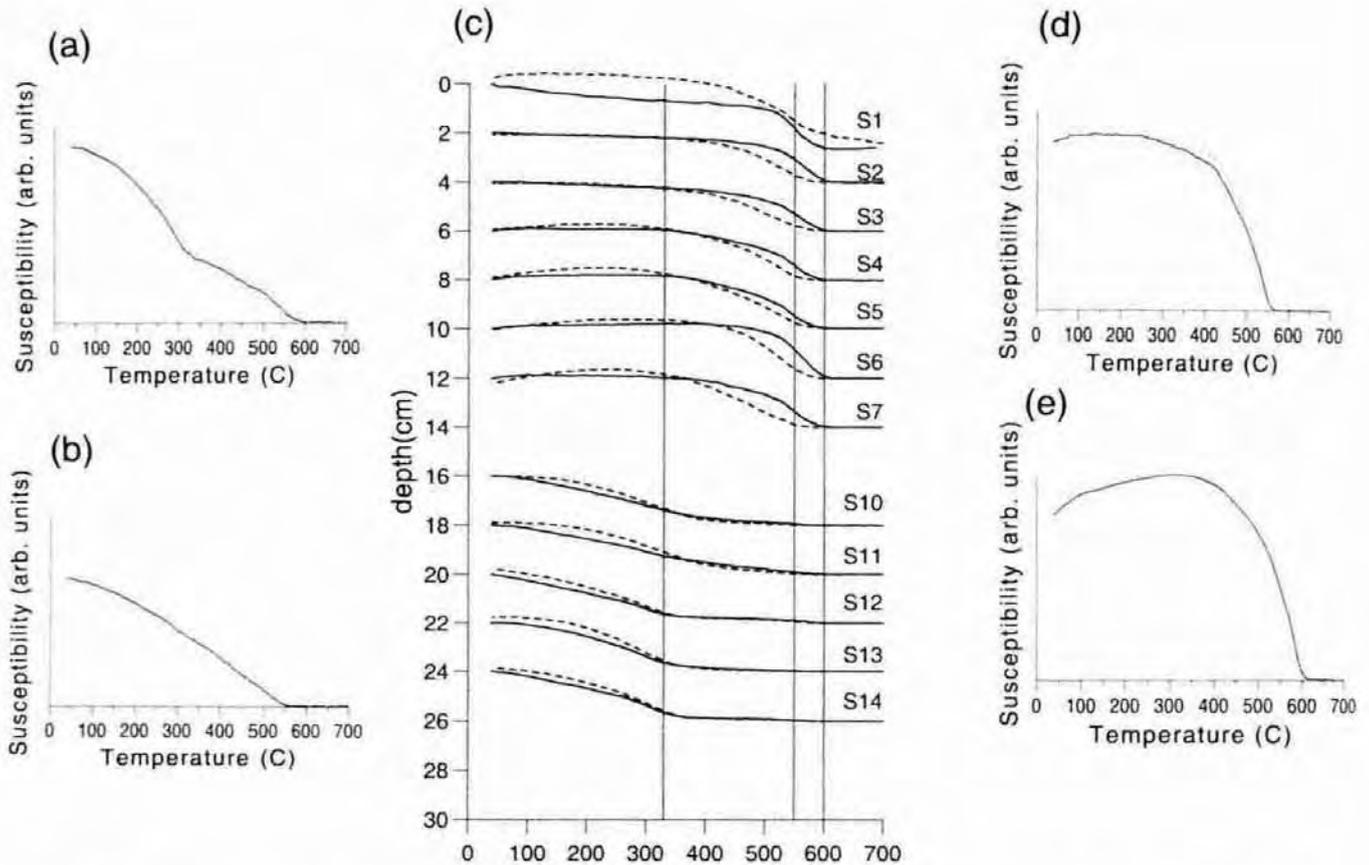
Fig. 5. *High temperature susceptibility curves for (a) Context 744 from Bostadh, (b) Guinnerso hearth sample 311(b), (c) profile of the ash samples from Cladh Hallan, (d) sample 10278 (context 1732) from Old Scatness Broch and (e) sample 10334 (context 1728) from Old Scatness Broch. The heating curves are indicated by solid lines and the cooling curves by dotted/dashed lines.*

data has given an indication of the potential for the development of magnetic unmixing techniques within archaeology.

CONCLUSIONS

–  A wide range of laboratory based magnetic measurements are available, which are applicable within the study of archaeological samples.
–  Fire ash, a significant component in site formation, can easily be identified and traced using magnetic data.
–  Measurement of modern ash residues of known fuel types has allowed the development of techniques from which fuel type components can be identified and quantified within archaeological samples.
–  Well-humified peat was found to be the major fuel source at seven sites on Lewis, whereas considerable variability in fuel use was observed at Cladh Hallan, South Uist and Old Scatness Broch, Shetland.

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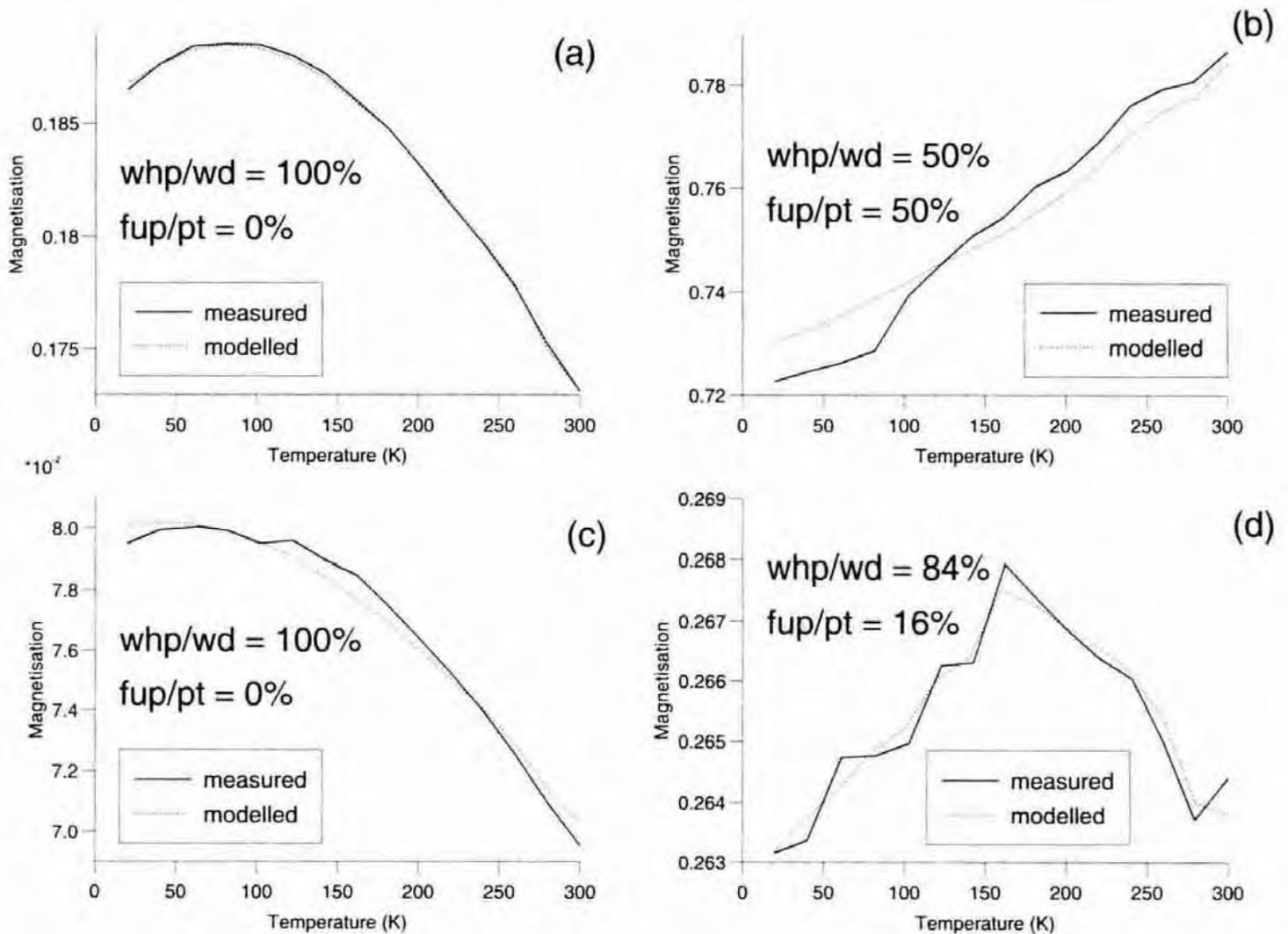Church, M. J. and Peters, C. (2000) Sedimentary analysis of soil

Fig. 6. Four examples of the $M_R$-T observations and models calculated by the unmixing algorithm. $M_R$ is in $Am^2kg^{-1}$. (a) is context 744 from Bostadh. (b) is Old Scatness Broch sample 10334. (c) is Cladh Hallan sample S11. (d) is Cladh Hallan sample S5.

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