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Archaeo-directional and -intensity data from burnt structures at the Thracian site of Halka Bunar (Bulgaria): The effect of magnetic mineralogy, temperature and atmosphere of heating in antiquity

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Abstract

Archaeomagnetic results are presented from a series of burnt structures at the Thracian site of Halka Bunar. Archaeointensity and archaeodirectional studies were undertaken on three kilns from a pottery production complex. This has been dated to the late 4th and early 3rd century B.C. (325–280 B.C.) based on coins found associated with the kilns [Tonkova, M., 2003. Newly discovered Thracian Centre of the Early Hellenistic Age at the Spring "Halka Bunar" in the Land of C. Gorno Belevo. Annuary of the Institute of Archaeology with Museum. Bulgarian Academy Sci. 2, 148–196 (in Bulgarian)]. This data provides a new point for the Bulgarian archaeomagnetic curve (Dec: 348.70 ± 5.79, Inc: 62.20 ± 2.70, and Fa: 77.23 ± 2.17 μT). The kilns are thought to have been used for producing different types of pottery in a range of heating atmospheres and at different temperatures. Therefore, special attention was paid to the magnetic mineralogy of the samples and its effect on the palaeodata. Kiln 3, orange clay samples were dominated by fine to ultra-fine grained single domain and superparamagnetic magnetite, with a small proportion of haematite. The samples were heated in a high temperature oxidising environment. Kiln 2 was probably used to make grey ware pottery. The samples are light grey and were dominated by stable single domain magnetite formed by high temperature heating in a more reducing environment. Kiln 4, mottled samples consisted of a variable mineralogy showing characteristics of both Kiln 2 and Kiln 3 samples. It was probably used to make traditional, mottled, Thracian ware pottery and was heated to lower temperatures in a mixed environment of heating. Samples heated in an oxidising environment gave more reliable Thellier results than samples heated in a reducing environment in antiquity, as the latter altered heavily on re-heating. A fourth kiln and a destruction feature from different trenches than the kiln complex were also investigated to establish their age. Archaeodirectional data was not recoverable from these two structures due to post-burning disturbance. The mean archaeointensity from Kiln 5 (mean 78.0 ± 1.7 μT) is consistent with that from the main kiln complex (mean 77.23 ± 2.17 μT) and is therefore considered to be contemporary. It was probably not used to make pottery. The destruction feature records much lower archaeointensity values (mean 65.1 ± 1.1 μT). When this value is compared to the existing reference points of the Bulgarian database it suggests this feature is younger than the kilns (250–140 B.C.). Multiple age use of the site is therefore confirmed with a main period of occupation in the late 4th and early 3rd century B.C. and another phase of occupation in the mid 3rd to mid 2nd century B.C.
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Keywords: Archaeomagnetism; Palaeosecular variation; Thellier experiments; Mineral magnetism; Bulgaria; Pottery kilns

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1. Introduction

Archaeomagnetism, as an interdisciplinary branch of geophysics, aims to recover information about past secular variations of the Earth's geomagnetic field elements (both palaeodirectional [declination—D, inclination—I] and palaeointensity data), for a given territory (Aitken, 1978). When an adequate reference curve exists archaeomagnetic data can be used to date archaeological sites and burnt structures (Aitken, 1978; Lanos et al., 1999). The ability to date structures depends on the recovery of data from well dated burnt material and structures from archaeological sites. However, such reference curves also provide primary information concerning the origin and history of the Earth's geomagnetic field. In some instances a collection of primary geomagnetic field data can be undertaken from well dated structures on an archaeological site, while archaeomagnetic dating can be conducted on other less well dated structures from the same or adjacent sites or trenches. This is especially useful if stratigraphic correlation is unknown, or uncertain between archaeological excavations at the same site. Because the Balkan Peninsula is extremely rich in archaeological sites it is an ideal area for the success of such kind of investigation. Consequently, Bulgaria has one of the longest and most complete archaeomagnetic curves in the world (Kovacheva, 1997; Kovacheva et al., 1998). However, there are particular periods where little data exists due to the occurrence of fewer well dated archaeological sites. The main aim of archaeomagnetic research in Bulgaria is to try and infill these gaps in the sequence to make future dating of archaeological sites more accurate. One such period is the last Millennium B.C. to which the site investigated in this study (Halka Bunar) is dated.

Halka Bunar is located close to the town of Chirpan, around 50 km northeast of Plovdiv in south central Bulgaria. Three potentially different activity specific sectors have so far been identified: (1) manufacturing; (2) residential; and (3) cult. One interpretation of Halka Bunar is that it is a sanctuary of a Hellenised type and it is presumed that a chthonic cult was worshipped there (Tonkova, 2003). The most important feature of the site is the discovery of a unique pottery production centre in trench three of the first sector. This is the first documented evidence of a pottery centre consisting of multiple kilns in pre-Roman Thrace (Tonkova, 2003). It is also the first evidence of the systematic organisation of pottery production in an Early Hellenistic setting (Tonkova, 2003). The pottery complex consists of a series of four kilns arranged together on a working platform (Fig. 1). The stratigraphic relationship of the kilns and the fact that more than one kiln uses the same pit features suggests they are contemporary. The kilns are thought to have been used for making various types of pottery including higher quality grey ware, red ware and lower quality traditional Thracian ware pottery (Herries and Kovacheva, 2007). A fifth kiln (Kiln 5) comes from trench 11, to the west of this group. Its exact association to either the cult sector or the pottery kilns (Kilns 1–4) has not been completely determined but it is thought to be potentially younger than the pottery complex. The residential sector (trench 11) contained part of a burnt wall referred to as the 'destruction feature' (DF).

The research aims at the site were:

1. Recover archaeodirectional (AD) and archaeointensity (AI) data from the well dated kiln complex (Kilns 1–4) at Halka Bunar.
2. To compare the archaeomagnetic data (AI and AD) from the kiln complex with other burnt structures (DF and Kiln 5) to assess their age.
3. To investigate the reliability of viscous cleaning versus full alternating field and thermal cleaning of NRM.
4. To investigate the effect of varying magnetic mineralogies, caused by different environments and temperatures of heating in antiquity on AI data.

2. Sampling

A series of 34 independently oriented hand samples of baked clay were removed from five different features at the site (Fig. 1). Four of these broke during sampling. Only three of the four kilns (Kilns 2–4) from the pottery complex were sampled. This was due to the disturbance of the final structure (Kiln 1) by robber trenches that lead to the sites discovery. Samples were taken from the main grate (on which the pottery was rested) of Kilns 2 (samples HB16-17 and HB32–34) and 4 (samples HB01-10) and from the lower grate (in the fuel chamber) of Kiln 3 (samples HB11–15). Kiln 5 (HB27 to HB31) and the DF (HB19, 22, 23, 25, and 26) were also sampled. In the laboratory the samples were set in blocks of plaster and cut into 8 cm^3 cubes while preserving the field orientation made by both a magnetic and sun compass. Declination was corrected on the basis of these measurements.

Between 6 and 20 sub-sample cubes (specimens) were cut from each hand sample depending on its size. Careful note was taken of which 2 cm level each specimen was removed from as they may have experienced different temperatures of heating in antiquity. Specimens from depth may not have been heated sufficiently in antiquity to 'lock in' a strong, stable, ancient remanence.

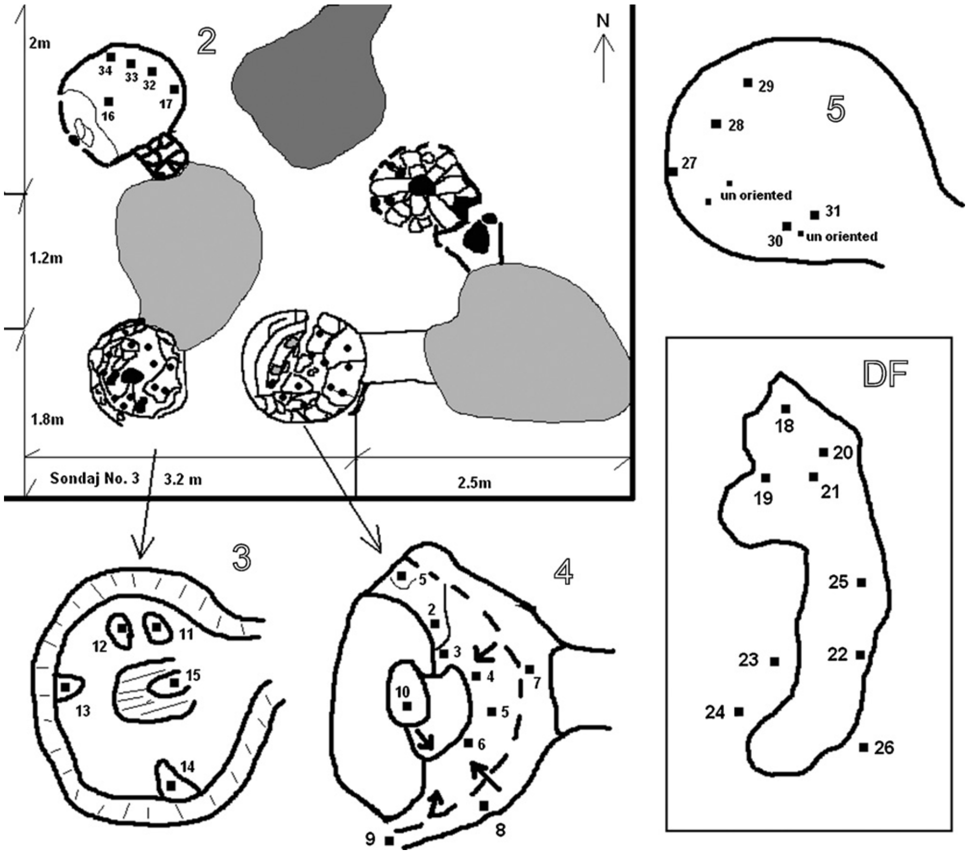

Fig. 1. Samples from the five features including the main kiln complex (Kilns 2, 3 and 4), Kiln 5 and the destruction feature (DF) at Halka Bunar. Kiln 1 was not sampled due to its destruction by treasure hunter's excavations at the site before excavation.

In some cases (Kilns 3 and 4) the samples were thick enough to remove 3 or 4 different 2 cm levels, while in some cases only 1 was possible (Kilns 2, 5). The samples from the different structures have a wide range of colour variation. Samples from Kilns 2 and 5 are light to dark grey, those from Kiln 3 and the DF are orange and those from Kiln 4 were dark grey to brown. However, in most cases the colour of samples from a single structure is relatively consistent, as are specimens from the same sample. The exceptions are samples from Kiln 4, which show a more mottled colouration and sample HB15 from Kiln 3, which is grey rather than orange. This is because HB15 came from the central supporting pillar of the kiln rather than from the lower grate of the fuel chamber, like the other Kiln 3 samples.

3. Experimental methods

Thermal demagnetisation was undertaken using a Magnetic Measurements (Ltd.) thermal demagnetiser at the University of Liverpool Geomagnetism Laboratory (ULGL). Alternating field demagnetisation was undertaken at the Bulgarian Academy of Science Laboratory in Sofia (BAS) on a Molspin (Ltd.) tumbling demagnetiser. Remanence measurements (Natural Remanent Magnetisation (NRM) and Isothermal Remanent Magnetisation (IRM)) were undertaken on a Molpsin (Ltd.) Minispin magnetometer at BAS and ULGL. Stepwise thermal demagnetisation at BAS was undertaken in a home made shielded kiln with a residual field of less than 5 nT inside the cooling chamber. IRMs were induced in a pulse magnetiser (Max field = 2 T; Min Field = 0.23 T).

A series of mineral magnetic tests were conducted on sister specimens to those used for the AI determination. The entire collection was first subjected to an evaluation of the specimens' magnetic viscosity, applying the zero field method (Banerjee, 1981). The specimens' initial NRM was first measured and the specimens placed into μ-metal boxes for 3 weeks. After this period the specimens were removed from the boxes and their stable, viscous free NRM (NRMst) was measured and the viscosity coefficient $\{S_v\% = [(\mathrm{NRM} - \mathrm{NRMst})/\mathrm{NRM}] \times 100\}$ was calculated.

Bulk magnetic susceptibility (X, $\times 10^{-6}\,m^3\,kg^{-1}$) measurements were undertaken on a KLY-2 Kappabridge at BAS, where the high temperature behaviour of magnetic susceptibility was performed using a CS-23 attachment (AGICO, Brno). Frequency dependence of magnetic susceptibility (X_{FD}%) and low temperature magnetic susceptibility (K_{LT}) were performed on a Bartington (Ltd.) MS2 system at ULGL.

IRM acquisition curves, backfields, hysteresis loops and thermomagentic curves were undertaken in air on a Magnetic Measurements (Ltd.) Variable Field Translation Balance (VFTB) at ULGL. Data was analysed using the RockMagAnalyzer 1.0 software written by Leonhardt (2006). Lowrie-Fuller tests (Lowrie and Fuller, 1971) were initially undertaken on a number of specimens followed by three-axes IRM (3IRM) tests (as per Lowrie, 1990). This involves inducing three different strengths of IRM (using 0.23, 0.46 and 2 T magnetic fields) along three different axes of a specimen. The specimen was then thermally demagnetised in set temperature steps. At each step the IRM left in each axis is measured.

Saturation isothermal remanent magnetisation tests (SIRM test) similar to those undertaken by Van Velzen and Zijderveld (1992) and Jordanova (1996) were used to look at the alteration of the specimens during laboratory heating. An initial 2 T IRM is induced in the specimen. A new 2 T IRM is induced after each thermal step and both the SIRM remaining after each thermal demagnetisation step (SIRMleft) and the newly induced 2 T SIRM (SIRM) imparted after this measurement are measured. Magnetic susceptibility (K) is also monitored. A comparison is made between the 3IRM (thermal demagnetisation of three-axes IRMs in the sister specimens) and the SIRMleft and this should identify specimens where new magnetic minerals are formed. The 3IRM demagnetisation curve shows the original mineralogy. By imparting a new 2 T SIRM after each heating step, magnetisation will be induced into any new minerals created by the previous thermal demagnetisation. K, SIRM and SIRMleft will be affected by any new minerals created. When the thermal decay of 3IRM and SIRMleft is identical the test is positive and when a substantial difference is observed it is negative. Negative SIRM tests are usually associated with unsuccessful Thellier experiments and vice versa (Jordanova et al., 1997, 2003). AF demagnetisation of SIRM(2T) was additionally performed after heating to certain temperatures during the SIRM test to look at changes in coercivity more closely.

Classic-style Thellier experiments (Thellier and Thellier, 1959) were performed in the BAS Vitosha Mountain laboratory. Specimens were heated in a non-magnetic kiln in the presence of the Earth's field, which is fairly stable at the site. The natural laboratory field was measured daily during the experiment with a fluxgate magnetometer (mean laboratory intensity [F_{lab}] = 37 μT, inclination [I_{lab}] = 70° and declination [D_{lab}] = 0°). Specimens were heated and measured twice, with an 180° vertical rotation of the specimen so that the field was directly opposite during each heating. pTRM checks were conducted after heating to 200 °C (120 °C heating), 300 °C (150 °C heating), 420 °C (320 °C heating) and 460 °C (320 °C heating). The specimen's alteration was monitored by measuring K between each heating step along with pTRM checks (8% acceptance criteria for the upper limit of pTRM checks) and remanence stability monitoring. Remanence measurements for this experiment were made using a computerised astatic magnetometer with an optical feedback system (Boroc Geomagnetic Laboratory, Russia).

4. Results and discussion

4.1. Mineral magnetism

Mineral magnetic data for selected samples is shown in Table 1. All the samples have mean S_v% of <10, while 26% of the samples have a mean S_v% less than 1, 66% less than 2 and 87% less than 3 (Table 1). The Koeningsberger ratio (Q = NRM/($K \times H$)), where K is the magnetic susceptibility and H is the strength of the ambient geomagnetic field (Dunlop and Ozdemir, 1997), was also calculated. All values of Q are greater than 1, with around 30% of the total number of samples having Q between 1 and 5, 50% between 5 and 10, and 20% >10 (Table 1). Such high Q values suggest a stable thermoremanent origin of NRM for all samples from the collection. The remaining data indicates that the Halka Bunar samples can be separated into two main mineralogical groups:

Group 1 samples consist of orange coloured samples from Kiln 3 (HB11-14) and the DF. Hysteresis and Lowrie Fuller tests (Figs. 2a and 3) indicate that Group 1 samples are dominated by low coercivity ferrimagnetic minerals. This is confirmed by alternating field demagnetisation of NRM (Fig. 9). The samples fall in the PSD grain size region of the Day Plot (Day et al., 1977; Fig. 4a). The samples lie in the region indicating an SP and SD mixture of grains as per Dunlop (2002). High X_{FD}% (mean 11.53%; Fig. 4b) and low RS (K_{LT} at −196 °C/K_{LT} at +25 °C) ratios also suggest that the samples are dominated by grains ranging across the superparamagnetic (SP) to single domain (SD) grain sized boundary. There is no evidence for MD grains,

Table 1
Archaeomagnetic and mineral magnetic data for Halka Bunar

Sample	Dec (°)	Inc (°)	α95	Mean X	No	Mean S_v%	Mean X_{FD}%	Q	T_c (°C)	H_{cr}	H_c	M_s	M_{rs}	M_{rs}/M_s	H_{cr}/H_c	B-ratio	RS	PS	RS/PS
Kiln 4																			
HB-01	343.50	57.10	1.10	3.01	8	1.53	6.53	2.44	539	42.14	16.40	135.31	42.42	0.31	2.57	0.94	0.76	0.77	0.99
HB-02	346.80	55.40	0.90	3.66	8	0.96	5.91	8.96	500		54.83	341.47	191.51				0.61	0.62	0.98
HB-03	*324.60*	*58.90*	*1.10*	4.28	*6*	1.20	5.02	8.05	560	68.83	37.87	507.28	252.74	0.50	1.82	0.96	0.74	0.75	0.99
HB-04	*316.10*	*57.30*	*0.80*	4.61	*12*	0.91	5.64	6.80	521		40.00	509.59	241.25				0.56	0.58	0.97
HB-05	342.40	68.50	0.80	5.59	11	1.30	7.09	6.02	580	80.84	42.71	422.01	207.32	0.49	1.89	0.96	0.63	0.70	0.90
HB-06	*333.00*	*48.20*	*2.60*	3.95	*5*	1.00	6.01	8.86	569	90.21	54.43	271.00	156.77	0.58	1.66	0.96	0.68	0.69	0.99
HB-07	*77.50*	*4.60*	*0.70*	3.72	*10*	0.86	7.14	3.23	538		19.61	262.26	94.93				0.78	0.79	0.99
HB-08	*298.40*	*71.50*	*0.90*	8.66	*7*	0.76	7.11	5.79	591	67.79	27.28	297.74	123.23	0.41	2.48	0.93	0.72	0.72	1.00
HB-09	355.20	60.40	0.60	3.70	12	1.01	5.62	7.50	566		39.05	543.14	272.10				0.78	0.79	0.99
HB-10	348.60	63.40	0.80	2.17	11	1.54	7.33	3.68	583	72.49	31.08	188.12	87.44	0.46	2.33	0.94	0.85	0.86	0.99
Mean	347.50	61.00	5.40	4.34	5	1.11	6.34	6.13		70.38	36.33	347.79	166.97			0.95	0.71		0.98
Kiln 3																			
HB-11	*354.60*	*42.10*	*1.40*	11.95	*7*	9.01	14.49	17.92	565	30.28	10.68	240.50	68.39	0.28	2.84	0.87	0.63	0.64	0.98
HB-12	348.20	61.00	0.70	14.08	11	1.89	10.79	14.98	592	37.97	11.52	256.19	74.29	0.29	3.30	0.85	0.60	0.70	0.86
HB-13	341.00	66.60	1.70	7.60	8	1.41	9.52	17.83	542		13.23	43.76	11.07				1.12	1.12	1.00
HB-14	344.80	61.90	0.80	14.96	11	1.76	11.45	12.90	564	26.06	8.35	349.73	94.37	0.27	3.12	0.84	0.55	0.68	0.81
HB-15	353.90	65.60	0.90	3.40	11	2.22	11.39	10.00	580	39.35	12.07	25.62	4.64	0.18	3.26	0.88	0.67	0.69	0.97
Mean	347.00	63.90	4.10	10.40	4	3.26	11.53	14.73		33.42	11.17	183.16	50.55			0.86	0.61		0.91
Kiln 2																			
HB-16	359.00	63.50	1.30	1.63	7	1.72	6.18	1.90	540	40.18	13.07	61.41	14.46	0.24	3.07	0.97	0.73	0.74	0.99
HB-17	346.10	56.30	2.20	2.84	7	1.82	5.75	5.63	520		27.03	150.57	62.76				0.57	0.59	0.97
HB-32	*41.20*	*65.30*	*2.60*	2.41	*6*	2.86	7.18	4.26	520		33.61	215.18	106.92				0.58	0.63	0.92
HB-33	*8.40*	*46.50*	*1.80*	2.14	*6*	1.78	*5.36*	7.22	520		44.48	175.60	93.88				0.59	0.59	1.00
HB-34	355.30	65.50	0.60	6.39	8	1.07	4.71	3.20	540	38.23	18.59	37.79	10.18	0.27	2.06	0.98	0.78	0.78	1.00
Mean	352.90	61.90	8.90	3.08	3	1.85	5.84	4.44		39.21	27.36	128.11	57.64			0.97	0.65		0.97
Site mean	348.70	62.20	2.70		12														
DF																			
HB-19	22.70	41.20	0.80	6.23.	5	1.79	9.74	7.30	626	33.37	9.66	107.83	26.39	0.24	3.45	0.77	0.78	0.85	0.92
HB-22	345.20	35.50	2.10	17.95	7	1.66	10.65	9.40	500	22.86	7.22	214.85	54.34	0.25	3.17	0.81	0.38	0.41	0.93
HB-23	300.80	64.40	1.30	14.88	5	2.29	12.50	14.40	520	24.41	7.95	331.87	92.72	0.28	3.07	0.78	0.33	0.36	0.92
HB-25	354.50	52.60	1.70	11.96	5	2.41	10.50	13.40	520	23.00	7.53	334.36	91.41	0.27	3.05	0.71	0.53	0.58	0.91
HB-26	324.30	37.50	1.40	6.29	4	3.81	11.10	6.50	580	25.87	7.56	110.19	26.42	0.24	3.42	0.75	0.51	0.53	0.96
Mean				10.22	5	2.39	10.90	10.20		25.90	7.98	219.82	58.26			0.76	0.51		0.93
Kiln 5																			
HB-27	324.30	43.80	1.60	10.25	6	1.07	5.49	5.19	600	56.71	27.79	95.39	42.01	0.44	2.04	1.01	0.73	0.74	0.99
HB-28	25.70	69.20	1.40	3.26	6	1.41	4.56	6.00	580	67.36	31.57	194.81	89.85	0.46	2.13	0.97	0.57	0.59	0.97

Table 1 (*Continued*)

Sample	Dec (°)	Inc (°)	$\alpha95$	Mean X	No	Mean S_v%	Mean X_{FD}%	Q	T_c (°C)	H_{cr}	H_c	M_s	M_{rs}	M_{rs}/M_s	H_{cr}/H_c	B-ratio	RS	PS	RS/PS
HB-29	49.50	−60.20	0.70	3.72	10	2.17	4.81	3.61	538	67.36	23.82	490.77	192.23	0.39	2.83	0.97	0.58	0.63	0.92
HB-30	209.60	36.10	2.20	8.16	3	4.16	6.50	4.75	610	48.20	20.17	174.39	61.41	0.35	2.39	0.99	0.59	0.59	1.00
HB-31	323.40	−27.60	2.90	5.62	6	3.13	4.60	4.37	540	46.98	25.54	557.31	259.35	0.47	1.84	0.97	0.78	0.78	1.00
Mean				6.20	6	2.39	5.19	4.78		57.32	25.78	302.53	128.97			0.98	0.65		0.97

Dec = declination, Inc = inclination, $\alpha95$ is a precision parameter (1 standard deviation), X = magnetic susceptibility (10^{-6} m^3 kg^{-1}), No = number of specimens, S_v% = viscosity coefficient, X_{FD}% = frequency dependence of magnetic susceptibility, Q = Koeningsberger ratio, T_c = Curie point; H_{cr} = coercivity of remanence, H_c = coercivity, M_s = saturation magnetisation and M_{rs} = saturation remanent magnetisation (hysteresis parameters 10^{-3} A m^2/kg); M_s/M_{rs} and H_{cr}/H_c = Day Plot ratios; B-ratio = SIRM/IRM$_{300\,mT}$, RS = K_{LT} −196°C/K^{LT} +25 °C, PS = K_{LT} −150 °C/K^{LT} +25 °C, RS/PS gives an indication of K_{LT} tail and change at isotropic point of magnetite. Mean values conducted using Fisher (1953). Italicised samples are not taken in the calculation ChRM.

which can show similar coercivty behaviour to these grain sizes. These samples also have the highest Q, S_v% and X values (Fig. 4b) for the site. Thermomagnetic curves show that magnetite is the dominant remanence carrying mineral (T_c, Table 1; Fig. 5a) and this is confirmed by thermal demagnetisation of NRM (Fig. 9). However, lower RS/PS ratios suggest that the samples do not have a low temperature magnetic susceptibility tail, indicative of maghaemite. Some Curie points above 600 °C are noted for surface specimens that also suggest the presence of maghaemite that is thermally stable to high temperatures (>600 °C; see discussion below). The orange colouration of the specimens suggests that haematite also exist, probably as a fine-grained amorphous pigment mineral. A lack of complete saturation of IRM by 300 mT (B-ratio [SIRM/IRM$_{300\,mT}$]; Table 1) suggests small amounts of coarser hematite also occurs.

Group 2 samples consist of light grey to brown samples from Kiln 4 (HB03, 05, 06, 08 and 09), Kiln 3 (HB15) and Kiln 5 (HB27, 28 and 31). Hysteresis and Lowrie Fuller test data (Figs. 2b and 3) indicate that Group 2 samples are dominated by high coercivity stable single domain (SSD) ferrimagnetic minerals. This is further confirmed by alternating field demagnetisation of NRM (Fig. 9). They fall in the SD and PSD region of the Day Plot (as per Dunlop, 2002). Magnetite is indicated by thermomagnetic curves (Fig. 5b). Thermal demagnetisation of NRM indicates that magnetite is the main remanence carrier (Fig. 9). A much lower proportion of fine-grained SD to SP grains is indicated by lower X_{FD}% values (mean 5.19–6.34%) and consequently lower X (Fig. 4b) and S_v% values (Table 1). There is little to no evidence for haematite as the B-ratio is closer to 1 for most samples.

The remaining samples (Group 3) come from Kilns 2, 4 and 5 and consist of dark grey to brown samples. Group 3 samples show a variation of mineral magnetic behaviours that suggests a mineralogical character intermediate between the Group 1 samples (Kiln 3, DF) and Group 2 samples (Kilns 4 and 5). The samples are dominated by SSD magnetite, as in the Group 2 samples. However, some samples have a slightly higher proportion of fine-grained SD grains and SP grains and some samples show evidence for haematite, while others do not. They plot in the intermediate region on the Day Plot (Fig. 4a).

Curie points of between 600 and 619 °C are recorded for some specimens from Kiln 3, Kiln 5 and the DF (Fig. 5c). These higher Curie temperatures are close to the Curie temperature of 610 °C estimated for maghaemite by De Boer and Dekkers (1996) and Özdemir and Banerjee (1984) report a natural

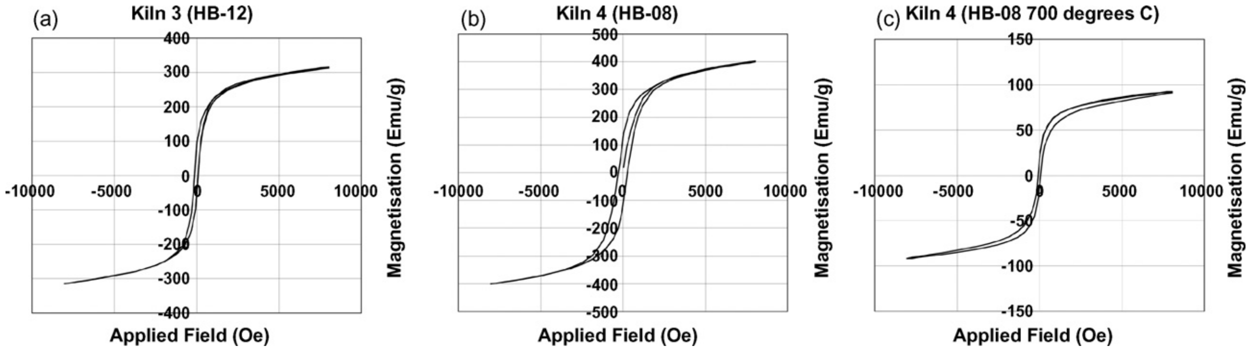

Fig. 2. Hysteresis loops for: (a) Group 1, lower coercivity specimen from a heavily oxidising atmosphere from Kiln 3 (HB12), (b) a Group 2 higher coercivty specimen from a more reducing atmosphere of heating in Kiln 4 (HB08) and (c) wasp waisted loop from the same sample after heating to 700 °C.

maghaemite stable to between 600 and 640 °C. However, maghaemite can be stable to inversion up to temperatures of 900 °C in special circumstances (Sartoratto et al., 2007). This can be caused by the maghaemite being of a larger grain size (Sidhu, 1988) and this idea is consistent with courser grained magnetite curves obtained by De Boer and Dekkers (1996) for natural maghaemite. Aluminium can also stabilise the inversion process (Schwertmann and Fechter, 1982) and heating in a reducing environment can stabilise inversion up to 700 °C (Van Oorschot and Dekkers, 1999). Moreover, maghaemite can be inverted at much lower temperatures (>∼330 °C) and at temperatures similar to magnetite.

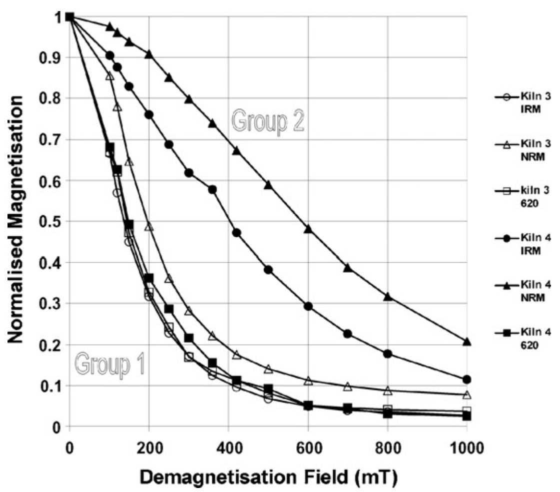

Fig. 3. Alternating field demagnetisation of NRM and IRM (2 T) from Lowrie and Fuller (1971) tests for a Group 2, grey, reduced specimen (Kiln 4) and a Group 1, red, oxidised specimen (Kiln 3). Demagnetisation of 2 T SIRM after the 620 °C heating step during the SIRM tests are also shown. No change in coercivity occurs for the Group 1 oxidised specimen. Coercivity lowers with heating in the Group 2 reduced specimen until it is similar to the oxidised Group 1 specimen after heating to 620 °C.

Therefore, the temperature of inversion of maghaemite is critically dependent on impurities, the formation process of maghaemite, grain size and shape, and the atmosphere of heating. An unequivocal identification of maghaemite in natural samples is therefore difficult.

The Halka Bunar specimens show a range of behaviours suggestive of maghaemite. The thermomagnetic curves are similar to the coarse grained maghaemite of De Boer and Dekkers (1996). IRM acquisition curves also share the same characteristics as those of De Boer and Dekkers (1996) and saturate at fields of 150 mT. This is slightly higher than suggested for the pure coarse-grained maghaemite of De Boer and Dekkers (1996) but its association in the Halka Bunar clay with other minerals such as magnetite may suggest that the maghaemite has formed by processes of low temperature oxidisation and maghematisation of exterior grain surfaces. Low temperature magnetic susceptibility measurements of many specimens that fall in the Group 1 mineralogy show less evidence for a low temperature tail, with lower RS/PS ratios (Table 1). This pattern is often seen with heavily oxidised specimens and again points to maghaematisation. In many cases these specimens have either a higher T_c (>600 °C) or a lower T_c (∼500 °C) than specimens with a mineralogy suggestive of purer magnetite.

If of primary origin, formed during heating reactions, the presence of stable maghaemite in specimens suggests the specimens have not been heated to temperatures where inversion would occur. Both Özdemir and Banerjee (1982) and De Boer and Dekkers (1996) suggest that the complete transformation of naturally occurring maghaemite to haematite takes place around 650–660 °C and so it might appear that the specimens with maghaemite have been heated to temperatures lower than this. The Curie curves for these specimens are not completely reversible and show a 20% drop

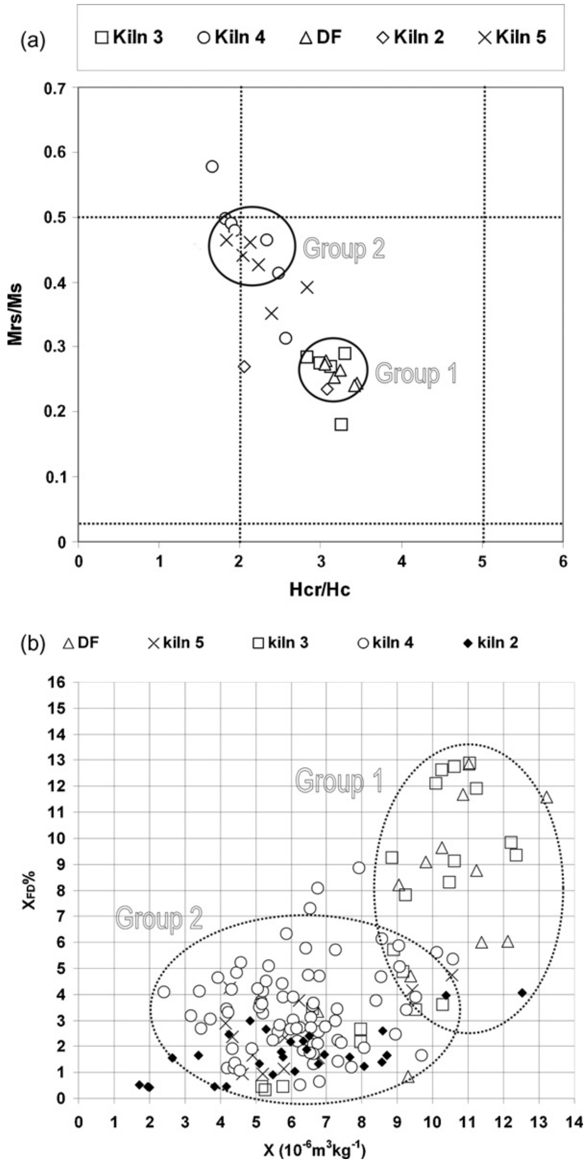

Fig. 4. (a) Hysteresis data plotted on a Day Plot (Day et al., 1977) using the criteria of Dunlop (2002); (b) a comparison of $X_{\mathrm{FD}}\%$ and X. Both plots show the relationship of the two main mineral magnetic groups (1 and 2) at Halka Bunar.

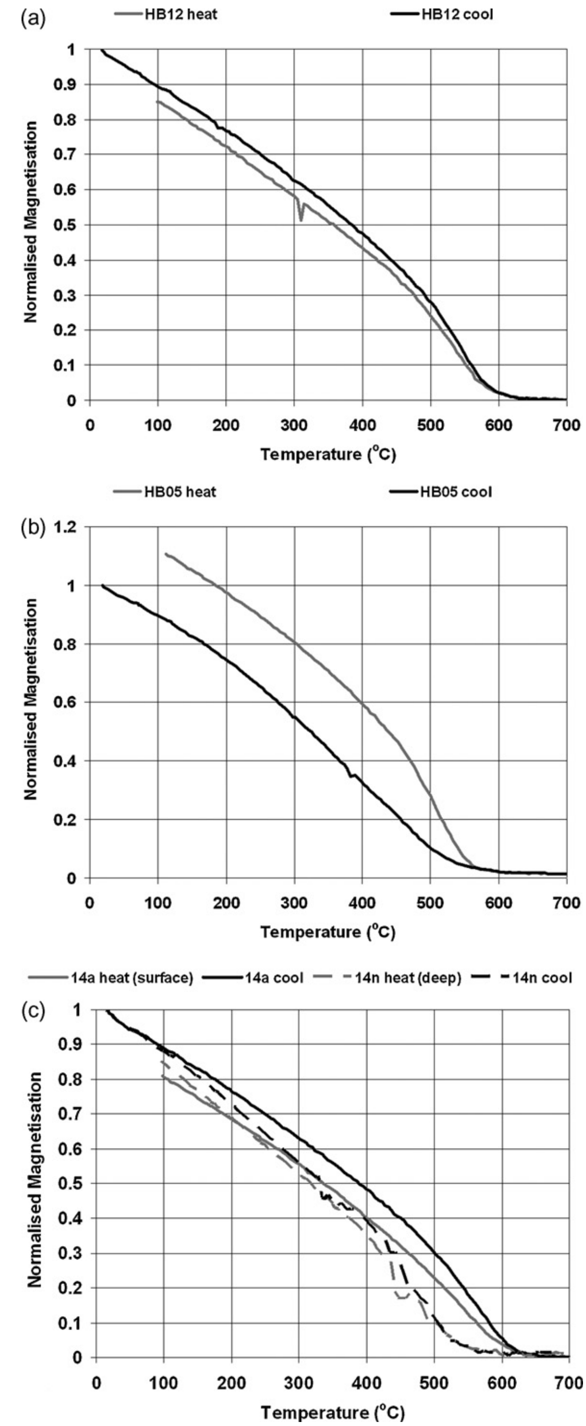

Fig. 5. Thermomagentic curves for: (a) Group 1 mineralogy specimens showing reversible curves; (b) Group 2 mineralogy specimens showing non-reversible curves; and (c) a comparison between surface specimens showing the presence of maghaemite that is thermally stable to temperatures above 600 °C (full lines) and a sample from depth in the same sample and indicating magnetite (dotted line). Cooling curve is the shorter, lighter line.

in magnetisation after heating, which further supports this assessment. The occurrence of maghaemite in the Kiln 3 and DF samples makes sense as these samples were heated in heavily oxidising conditions, as borne out by their mineralogy. In some cases the presence of maghaemite is also noted in surface specimens from some of the structures. This may suggest that these structures were left abandoned and exposed to the air (weathered) for some period after their last use and most likely suggests a secondary, post-heating formation of

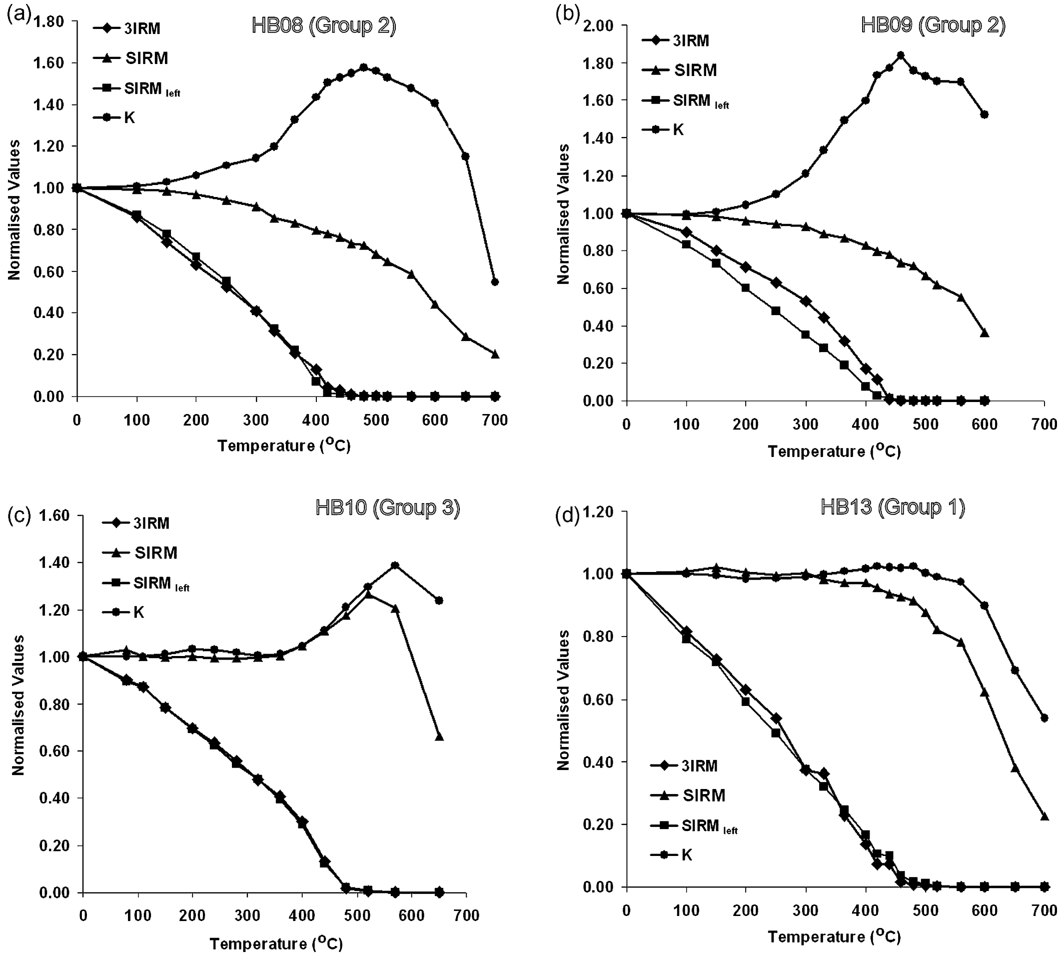

Fig. 6. SIRM heating alteration tests for Halka Bunar specimens. SIRM = 2 T induced IRM at each thermal step and SIRMleft during thermal demagnetisation, K = magnetic susceptibility, thermal demagentisation of 3IRM = 2 T induced IRM only once before thermal treatment (undertaken on a sister specimen). SIRM heating alteration tests for specimens H09 (negative), and HB08, 10 and 13 (positive).

maghaemite. For this reason, samples with this mineralogy were not preferentially used for Thellier analysis.

In review, Group 1 samples have a low coercivity mineralogy consisting of a mixture of SP and SD magnetite and maghaemite (both primary and secondary). Group 2 samples have a mineralogy suggestive of SSD magnetite with less SP grains and no haematite or maghaemite present. Group 3 shows a range of behaviours from both Group 1 and Group 2 samples.

4.2. Heating alteration tests

Variation in magnetic mineralogy and its subsequent alteration on heating has a profound effect on the suitability and reliability of different specimens for AI analysis using the Thellier method. Three forms of heating alteration experiments were performed: (1) SIRM tests (described previously; Fig. 6); (2) X and X_{FD}% measurements undertaken at each heating step during SIRM tests (Fig. 7); and (3) Thermomagnetic curves (Fig. 5).

All three types of heating alteration test indicate that there is little or no alteration in the magnetic mineralogy of Group 1 specimens. There is no change in coercivity (Fig. 3), X or X_{FD}% (Fig. 7) and they have reversible thermomagnetic curves (Fig. 5a). This is thought to be due to the fact that these specimens were heated in a heavily oxidising environment during antiquity. The specimens have therefore reached a thermomagnetic state that is stable in oxidised laboratory heating, as in the Thellier experiment, up to temperatures of ~620–640 °C. Heating in antiquity caused the formation of large amounts of fine to ultra-fine grained SD and SP magnetite, maghaemite and haematite, as indicated by mineralogical analyses of Group 1 specimens. Above 640 °C, more alteration occurs when haematite is formed in greater quantities.

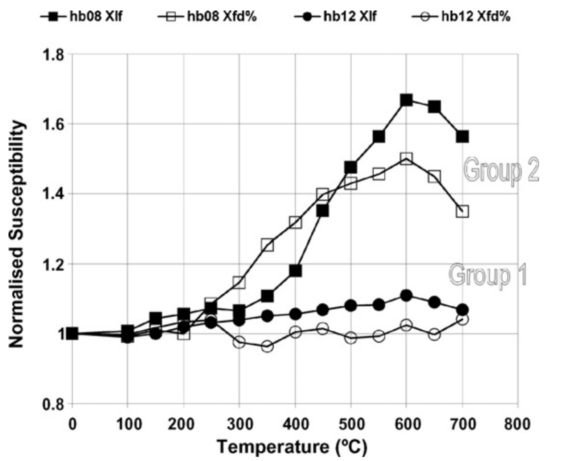

Fig. 7. Variation in room temperature low frequency magnetic susceptibility (X) and frequency dependence of magnetic susceptibility (X_{FD}%) on heating for Group 1 (Kiln 3, orange, oxidised) and Group 2 (Kiln 4, grey, reduced) specimens.

Due to variation in the temperature and atmosphere of heating in antiquity the mineralogy of Groups 2 and 3 alters at different temperatures on oxidised re-heating in the laboratory. A reducing atmosphere causes the formation of SSD magnetite and inhibits the production of haematite and the formation of SP grains until much higher temperatures (Coey et al., 1979; Van Klinken, 2001). The Group 2 and 3 specimens have a mineraology consistent with this (lower X_{FD}%, X and higher coercivity; Table 1). The mineralogy of Group 2 is thought to be due to two processes. In some specimens (Kiln 2) the presence of a high temperature reducing atmosphere during heating caused the formation of a greater proportion of SP grains than for specimens heated at a lower temperature in a reducing atmosphere, where SP grain formation is inhibited. In the case of other specimens (Kiln 4), a lower temperature of heating in a mixed atmosphere caused the formation of some SP grains but not the same quantity of SSD grains as specimens heated to a higher temperature in the same atmosphere. As such, a similar mineralogy is created and they display mineralogical characteristics of both other groups.

The mineralogy of both Group 2 and 3 specimens becomes identical to the mineralogy of the Group 1 specimens after heating to 620 °C and so the base clay mineralogy is thought to have been the same for all features before heating in antiquity. Based on the assumption that the initial clay was the same before heating in different heating environments the mineral magnetic variability will reflect variation in the firing temperature and the ambient atmosphere during heating being reducing, oxidising or a mixture (Ossipov, 1978).

In both groups, thermomagnetic curves are not reversible as new, more magnetic material is created (Fig. 5). Hysteresis analyses of specimens after heating to 700 °C indicate wasp-waisted behaviour indicating that low coercivity ferrimagnetic minerals and anti-ferromagnetic haematite have been formed. As coercivity (Fig. 3) and SIRM decrease during SIRM tests (Fig. 6a and b), X and X_{FD}% increase (Fig. 7). This is due to the formation of ultra-fine SP to SD boundary ferrimagnetic grains, the former of which do not contribute to SIRM. Therefore, the $SIRM_{left}$ and SIRM would decrease with the formation of these non IRM contributing secondary phases and the unblocking of small primary remanence carrying ferromagnetic minerals. At higher temperatures SP grains are altered and courser grained haematite is formed causing SIRM, X and X_{FD}% to decrease dramatically (Fig. 6), while high field coercivity increases during alternating field demagnetisation of SIRM (Fig. 3).

In conclusion, the magnetic mineralogy of the specimens suggests that the majority of samples from the main kiln complex have been well heated in antiquity and the kilns were probably used on multiple occasions. Archaeological data supports this as Kiln 2 was partially rebuilt. This is also borne out by high Q values. The heating alteration tests indicate that the Group 1 mineralogy is the best suited to Thellier analysis, consisting of thermally stable low coercivity magnetite that was formed by oxidised high temperature heating. In contrast, specimens from Group 2 are likely to be the least reliable for Thellier analysis despite the SSD mineralogy of the specimens. The mineralogy of the specimens indicates that they probably underwent a lower temperature of heating in a more mixed environment. They alter much more on heating with the formation of new ferrimagnetic minerals, mainly in the fine SD to SP grain size. The SIRM tests indicate that Group 3 specimens alter at higher temperatures than Group 2 (heated in a mixed environment at lower temperatures). This suggests that they have been heated to a high temperature in a reducing atmosphere. They would be better for Thellier analysis than the Group 2 specimens.

4.3. Archaeointensity data

Classic style Thellier (Thellier and Thellier, 1959) AI experiments were applied on 33 specimens taken from different parts of the five structures and are shown in Table 2. A range of suitable and non-suitable specimens were chosen for the experiment to test the mineralogical and heating alteration interpretations. The AI data presented were not subjected to the magnetic anisotropy

Table 2
Archaeointensity data for the five features at Halka Bunar

Samples	Fa (μT)	*S* (μT)	Temperature interval (°C)	SIRM test
Kiln 4				
hb02v	76.5	7.4	0–430	
hb03j	75.1	1.9	20–400	
hb03d	75.7	7.1	20–460	
hb04v	77.3	7.3	20–430	
hb05d	X	X	X	Negative
hb06v	73.4	5.5	20–460	
hb07d	76.3	1.4	0–360	
hb07g	82	3.1	20–400	
hb08v	X	X	X	Positive
hb09z	X	X	X	Negative
hb10d	73.4	3.6	20–460	
hb10e	77.3	3.6	20–430	Positive
Mean	76.3 ± 2.1			
Kiln 3				
Hb11z	76.3	4.6	20–430	Negative
Hb12l	78.6	2.1	20–460	
Hb12v	78.8	2.8	100–460	
Hb13e	80	3.4	120–520	Positive
Hb14a	79.7	2.1	150–520	Negative
Hb14m	78.1	2.2	20–520	Negative
Hb15b	76.6	3	20–430	Positive
Hb15j	76.8	2.5	20–460	Positive
Mean	78.3 ± 1.2			
Kiln 2				
Hb16l	82.5	4	20–430	Positive
Hb17z	74.3	2.2	20–400	
Hb32v	X	X	X	
Hb34g	76.9	8.5	100–360	Positive
Mean	76.2 ± 3.4			
Pott. cent. mean	77.23 ± 2.17			
Oven 5				
Hb28g	76.9	3.7	20–360	Positive
Hb29d	72.6	5	20–320	Positive
Hb29i	77.8	7.4	20–320	Positive
Hb29z	78.8	1.7	0–430	Positive
Hb31d	X	X	X positive	Positive
Mean	78.0 ± 1.7			
DF				
Hb19e	67.6	2.1	20–460	
Hb22j	64.1	1.2	20–430	Positive
Hb23g	65.2	1.1	120–460	
Hb25e	65.4	2.7	150–320	Positive
Mean	65.1 ± 1.1			

X, denotes that a specimens's results were rejected due to high magnetomineralogical alteration during the Thellier experiment. The means are weighted averages (Kovacheva and Kanarchev, 1986). The last column gives the results from the SIRM test (two more positive tests exist for Kiln 5: HB26, 27).

correction because it has been shown (Kovacheva et al., in preparation) that it is insignificant for such kind of material (baked clay from prehistoric kilns). We also expect that the cooling rate effect should be negligent for the size of the specimen used (8 cm^3) when conducting Thellier experiments with a natural cooling cycle (2–4 h) and without a fan. Thus, we consider that the cooling rate was essentially the same in both antiquity and the laboratory. Specimens showing big magnetomineralogical changes during heating were rejected if an AI determination could not be made before major mineralogical alteration occurred or were not used for the experiment entirely.

Specimens from Kiln 3 were sampled from the lower grate, within the lower fuel chamber and so have been very well heated and provide reliable results with a stable remanence up to between 460 and 520 °C (Fig. 8a), despite their low coercivity (Fig. 2a). Specimens from Kiln 4 were sampled from the upper grate, on which the pottery was rested during firing. Mineralogy and heating alteration tests indicated that Kiln 4 was possibly heated to lower temperatures than the other kilns, in a more mixed heating environment. This is confirmed in the Thellier experiments with alteration and loss of stable remanence at much lower temperatures during heating (Fig. 8b). Some specimens from Kiln 4 provide reliable results, while others do not (Fig. 8c). This is due to the mixed heating and atmosphere variation within the kiln and the high degree of mineral magnetic variability of the clays (Group 1 mineralogy). Specimens from Kilns 5 and Kiln 2 again provide varying reliability for Thellier experiments due to the change in heating atmospheres from antiquity to the laboratory (Fig. 8d and e).

All specimens from the DF provide reliable results. The Aria plots (Nagata et al., 1963; Fig. 8f) for these specimens are stable to between 320 and 430 °C and suggest a lower temperature of heating than for the kilns. This may also be in part due to the single and random heating nature of the event. Despite the low temperature of heating in antiquity these specimens provide some of the most consistent AI results from the site (Table 2). This indicates that specimens heated in a heavily oxidising environment gave the most reliable Thellier results (Fig. 8a and f) despite the temperature of heating that they have experienced. This is because both sets of oxidised specimens from the DF and Kiln 3 (Group 1 mineralogy) had reached an optimal mineralogy with thermomagnetic stability. Similar conclusions have been reported by Spassov and Hus (2006).

The AI results indicate that in most cases mineralogical analysis and heating temperature tests provide a reliable indicator of the success of particular specimens to Thellier experiments. However, the SIRM test as per Jordanova (1996) appears less reliable. In some cases this method works, as documented by Jordanova et al. (1997, 2003). For example, Group 2 sample HB10 gave a reliable Thellier result and has a positive SIRM tests and Group 3 sample HB05 gave a non-reliable Thellier result and had a negative SIRM test. However, for many other samples (e.g. HB08, HB11, HB14, HB31) a comparison of 3IRM and $SIRM_{left}$ does not appear to correlate well with more in-depth mineralogical analysis of heated samples. A comparison of $SIRM_{left}$ and 3IRM for sample HB08 suggest a positive SIRM test, despite changes in SIRM and mineralogy very similar to negative SIRM sample HB09. For some samples a comparison of $SIRM_{left}$ and 3IRM alone does not show if a specimen will be successful or not during Thellier experiments. This is most likely due to heterogeneity (more of a problem in reduced specimens) between sister specimens used for SIRM and 3IRM analysis. A comparison of all four parameters in the SIRM test must be evaluated to decide if a positive or negative result occurs for this test, especially when comparing specimens with varying base mineralogies. Overall, samples which have an increase in X and a rapid lowering in SIRM (Fig. 6a and b; HB08 and HB09) produce unreliable Thellier results and those that do not alter so heavily on heating produce reliable Thellier results (Fig. 6c and d: HB10 and HB13). A simple comparison of changes in X appears to be the best method for determining alteration and weather a sample will be successful or not.

In conclusion, the main reason for specimen failure during AI analysis was magnetomineralogical alteration due to a change in the atmosphere of heating, from a more reducing atmosphere in antiquity to a heavily oxidising atmosphere during the Thellier experiment. The behaviour of specimens during Thellier experiments is therefore dependent on the temperature and atmosphere of heating that the clay has experienced, which is a function of the location of sampling within the kilns and the use of the kilns during antiquity.

4.4. Archaeodirectional data

The zero field, magnetically cleaned NRM (NRMst; viscosity test described above) was used as the final calculated mean direction for the majority of samples from the site (Table 1). Representative samples of the collection were both thermally demagnetised and alternating field demagnetised to aid as a check that the NRMst value was a true representation of the primary direction of the sample (Table 3). A mixture of zero field

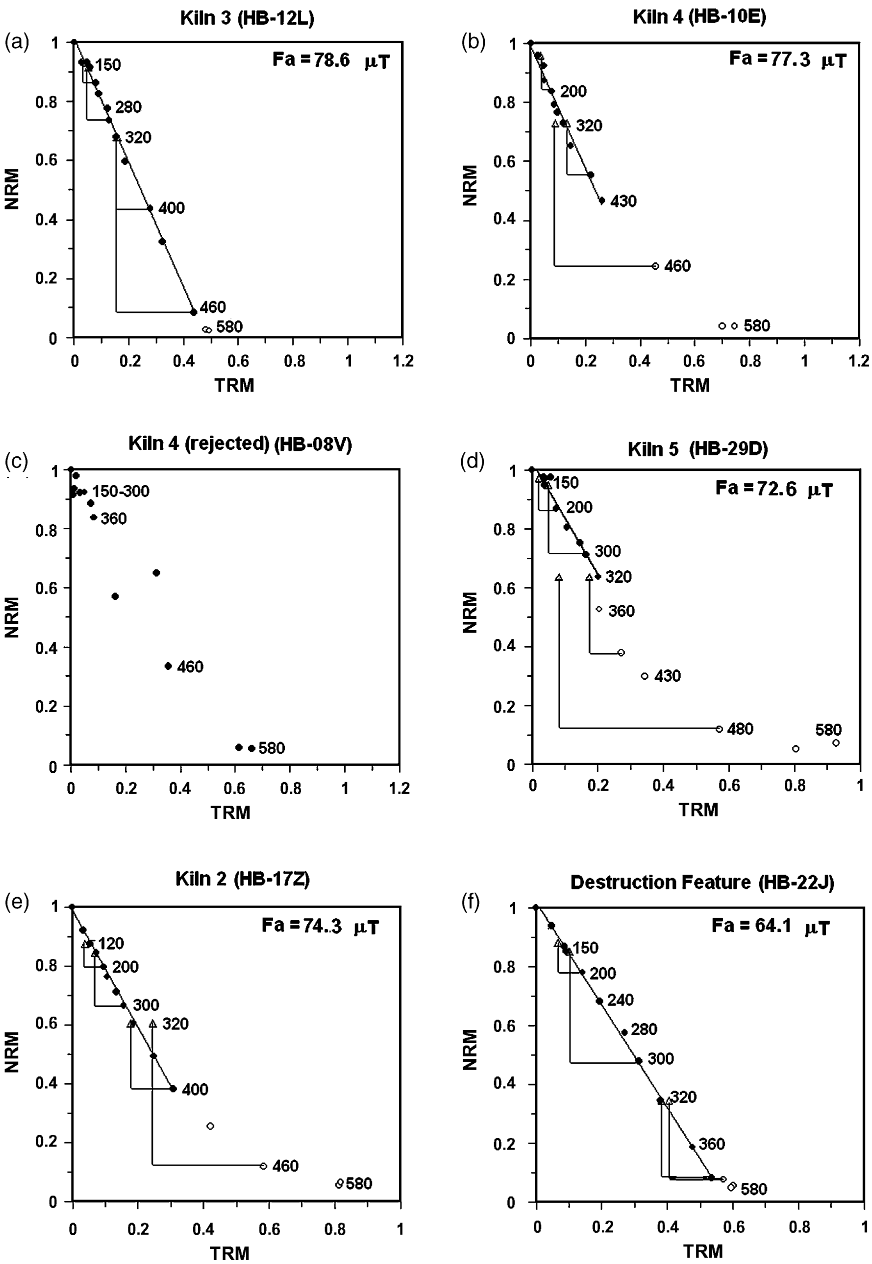

Fig. 8. Examples of Arai plots from Thellier Archaeointensity experiments for the different features at Halka Bunar: (a) Group 1 sample from Kiln 3; (b) Group 2 sample from Kiln 4; (c) Group 1 sample from the destruction feature; (d) Group 1 sample from Kiln 5; (e) failed Group 2 sample from Kiln 4; and (f) Group 3 sample from Kiln 2.

(NRMst; method described above), alternating field and thermally cleaned NRM directions were used in calculating the final sample means using Fisher (1953) statistics. Characteristic Remanent Magnetisations (ChRM) were calculated for the specimens using principal component analysis (Kirschvink, 1980).

All the viscous cleaned specimens show a strong single component direction of remanence during both alternating field and thermal demagnetisation (Fig. 9a). A small viscous component is seen in the thermal demagnetisation spectra because the specimens were transported to the LUGL to perform these experiments after the viscous cleaning method had been undertaken at BAS. As such they will have picked up a small viscous remanence during transport. Greater variation is seen between the results obtained for thermally cleaned

Table 3
A comparison of raw results obtained from the three kilns (2–4) used to provide the site mean using the viscous cleaning method (where NRM is the raw NRM and NRM_{st} is the viscous cleaned NRM) vs. the alternating field (AFD) and thermal demagnetisation (THD) methods

	CP Dec	CP Inc	ZP Dec	ZP Inc	MAD	NRM Dec	NRM Inc	NRM_{st} Dec	NRM_{st} Inc
AFD									
Kiln 3									
14v	337.2	61.5	335.4	61.1	1.4	338.3	62.6	337.9	62.2
13g	338.9	67.6	337.1	68.1	0.8	337.6	68.3	338.4	68.1
11a	348.5	43.5	348.8	43.2	0.4	349	44.1	349.9	43.7
15g	348.8	64.2	350.9	64.4	0.8	353.6	67	349	65
Mean	343.4	59.2	343	59.2		344.6	60.5	343.8	59.8
Kiln 4									
05g	339	68.7	339	68.4	0.6	338.1	68.9	336.8	69.1
08j	295.1	71	291.9	71.9	1.5	296.3	70.9	294.9	70.9
09d	350.3	61.4	350.5	61.4	0.6	350.5	61.6	350.2	61.4
10v	342.8	62	345	62.8	1.5	345	62.4	344	63
Mean	331.8	65.8	331.6	66.1		332.5	66	331.5	66.1
Kiln 2									
16d	354.1	65.8	355	65.3	0.6	354.9	66.1	355.9	65.8
16j	352.2	63.9	355	63.9	0.7	354.2	64.3	353.6	64
32g	41.3	64.4	42.3	64.9	0.7	39.3	64.6	40.6	64.8
34a	349.8	65.9	354.5	66.2	1.5	351.5	65.3	352	65.3
Mean	274.4	65	276.7	65.1		275	65.1	275.5	65
THD									
Kiln 3									
11d	349.4	40.7	348.6	39.3	1.8	350.7	43.2	350.7	41.4
12a	345.1	59.3	345.2	59.7	2.9	345.2	53.4	345.2	56.4
13v	343.3	65.9	339.3	67.7	3.7	342	66.1	343.2	65.9
14b	339.4	60.2	341.3	58.5	2.4	338.2	61.6	337.7	61.6
15d	356.2	63.4	356.8	63.4	4	350	65.6	350.2	65.4
Mean	346.7	57.9	346.2	57.7		345.2	58	345.4	58.1
Kiln 4									
01l	336.6	56.3	333.4	55.6	3.2	337	60.1	337.5	60
02k	343.1	53.3	343.9	53.2	0.6	347.2	55.5	347.5	55
03b	327.9	58.5	330.1	60.4	1.6	318.8	60.9	319.5	60.6
04d	314.1	60.7	314.8	60.2	1.4	310.9	58.7	310.2	58.9
05k	344.7	63.6	342.8	63.6	0.6	340.2	68	339.6	67.7
06g	329	45.8	333.4	49.9	4.3	329.8	46.5	329.7	45.9
07j	68	0.9	68.7	2.8	5.6	72.3	5.2	71.8	5
08z	302	72.5	305	75.3	1.8	292.7	71.4	290.6	71.5
09j	352	55.7	350.9	54.9	1	352.1	59.5	351.9	59.3
10m	351.6	56.5	352.8	54.3	1.4	347.8	61.9	346.6	61.5
Mean	306.9	52.4	307.6	53		304.9	54.8	304.5	54.5
Kiln 2									
16b	359	61.1	359.9	63	1.7	358.2	63.5	357.7	63.6
17a	346.5	53.9	346.8	53.5	2.4	345	54.7	345.6	54.7
32e	29.2	60.8	47.1	63.3	1.5	31.7	63	32.2	64.2
33v	1.4	49.3	0.8	50	2.5	4.3	50.5	4.1	50.2
34v	348.1	62.7	349.1	64.6	3.9	350.2	66	350.7	65.9
Mean	216.8	59.6	220.7	58.9		217.9	61.5	218.1	61.7

Values have been calculated using both stereoplot analysis (CP-Dec, CP-Inc) and Zijdeveld plots (ZP-Dec, ZP-Inc). MAD = mean angle of deviation and shows the reliability of the data.

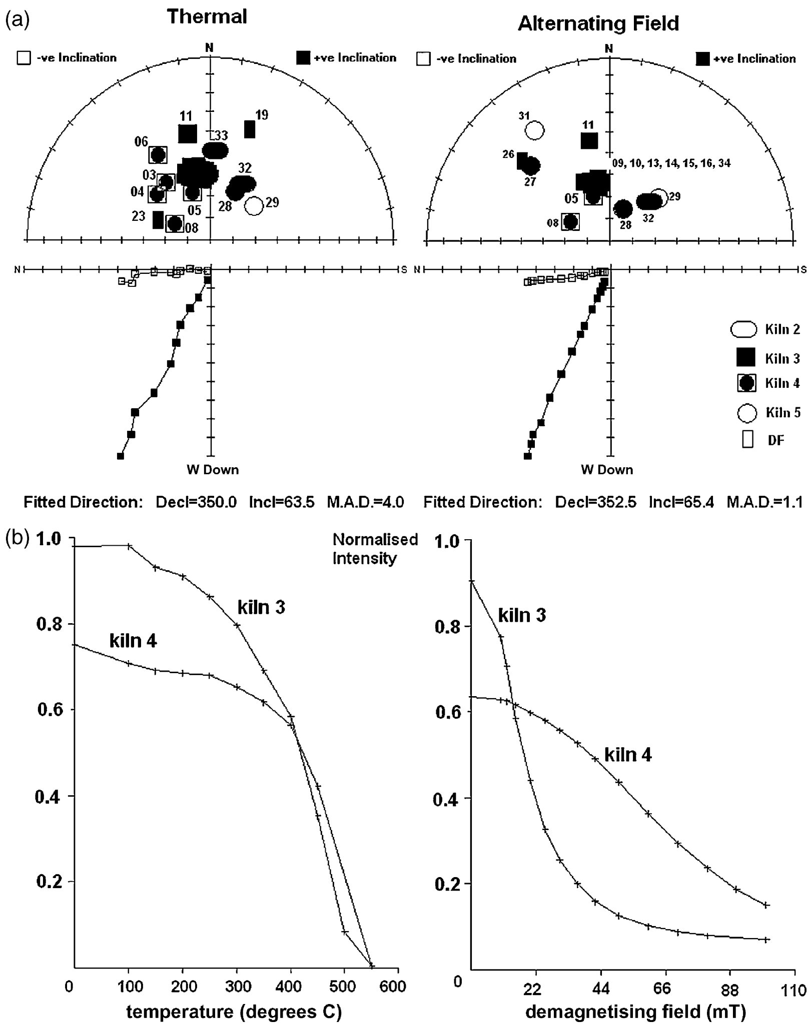

Fig. 9. (a) Comparison between thermal and alternating field demagnetisation for the estimation of archaeodirection from the five features at Halka Bunar; (b) Zijderveld plots showing that the samples have a stable single component of magnetisation using alterating field and thermal demagentisation; and (c) a comparison between intensity spectra for low coercivity, Group 1 specimens from Kiln 3 and higher coercivity, Group 2 specimens from Kiln 4.

specimens and the NRM_{st} than between the alternating field cleaned specimens and NRM_{st}. Least variation is seen for Kiln 3 orange specimens which have a thermally stable, low coercivity magnetic mineralogy and the greatest variability is seen for specimens that have a high coercivity magnetic mineralogy and alter heavily on heating, as shown by changes in magnetic susceptibility parameters (Fig. 7). In contrast, the viscous cleaned AD value is unaffected by the variation in ancient heating atmosphere, as long as the ancient temperature of heating was high enough to lock in the remanence.

The variations seen between alternating field cleaned values and viscous cleaned NRM (NRM_{st}) are not greater than the overall variability seen for sister specimens from the same block sample or in some cases the variability in interpretation by using a stereo-plot versus a Zijderveld plot. Good agreement is seen in the means for each kiln and so NRM_{st} is seen as a true indicator of field direction as long as comparative full demagnetisations are undertaken for representative samples to show that the NRM consists of a single component. For specimens that have formed in a reducing atmosphere, alternating field demagnetisation is preferable over thermal demagnetisation.

When the cleaned directions are compared (Table 1) on a stereo-plot (Fig. 9b) it can be seen that a core grouping of directions plot with a mean direction of 349.2°/+63°. All of the samples from the DF and Kiln 5 and some samples from Kilns 2–4 plot as a scatter around this central point. This suggests that the burnt clay from the destruction feature and Kiln 5 are not *in situ* as they do not show independent groupings of their own, as would be expected if the samples were burnt during a different time period. Excavation also showed partial destruction of Kiln 2, which was later rebuilt. The mineralogy of samples from Kiln 3 and 2 are the same as other samples from the kilns that plot in the mean direction. The Kiln 3 samples come from the lower grate, which consists of a series of pre-moulded and fired clay logs arranged in a circle. Such features are highly susceptible to movement during post depositional processes such as burial. As such the scattering of the majority of samples are considered to be due to post-depositional movement rather than due to a low intensity of heating that would not have locked in the remanence. This may not be the case for samples from Kiln 4, where a high degree of mineralogical variation occurs.

5. Archaeomagnetic age assessments

Archaeologically, two phases of life can be distinguished in the region of the kilns; one phase contemporary with the use of the kiln complex and a second following their abandonment. During the second phase the terrain changed its functions and became a part of the cult sector of the settlement. The site as a whole is thought to date to the Early Hellenistic Age. Based on coins bearing Parion, Seuthes III, Lysimachus, Kassander and Demetrios Poliorketes, the main occupation period of the site, and the period of use of the kiln complex, is thought to be centred on the last quarter of the 4th to the second decade of the 3rd century B.C. (325–280 B.C.; Tonkova, 2003). The upper boundary is determined by the hemidrahm of Parion as well as by the vessels type "lopas" and the lower by the coin of Demetrius Polircetes (Tonkova, 2003). It is contemporaneous with the flourishing of the Odrysian capital Seuthopolis that is located 60 km north of the site and is thought to end around 279 B.C. (Tonkova, 2003) when there was a number of Celtic invasions into Bulgaria. As the fossil remanence is a record of the Earth's geomagnetic field at the time of last firing of the kilns it is a *terminus ante quem* for the kilns use and so is likely to date towards the end of this period, ca. 280 B.C.

Bulgarian palaeosecular variation data for the 3rd and 4th century B.C. (Table 4) show a negative declination variation, between 343° and 357°, and positive inclination variation, between 61.75° and 68.7°. The site mean for the main kiln complex at Halka Bunar is calculated at 349.2°/63° and so the data is entirely consistent with the archaeological date. However, AD data show little time dependence due to the small number of samples from this period and would therefore not be ideal for defining an age range. Positive declinations are not seen until the 8th century B.C. and 5th century A.D. and inclination is equally variable, with lower values not seen until the 2nd or 3rd millennium B.C. and 2nd century A.D. (see Kovacheva, 1997). AI values from Kilns 2, 3, 4 are consistent with values of 76.2, 78.3 and 76.3 μT (Table 2), respectively. The mean AI of 77.23 ± 2.2 is almost identical to the site of Gomolava (320–240 B.C.) and with a mean age of 280 B.C. Combined with the AD data the AI data suggests that the kilns most likely date to ca., 280 B.C., as suggested by the archaeological evidence.

Table 4
A new data point from Halka Bunar compared to palaeosecular variation data for the period from 0 to 500 B.C. from Bulgaria (Kovacheva, 1997).

Site	Dec. (°)	Inc. (°)	Fa (μT)	Low age	High age
Karanovo	X	60.79	70.35	0	100
Chatalka	X	64.3	X	−100	0
Burgas	X	X	69	−200	−100
Isperih	354.66	67.36	65.11	−170	−140
Gomolava	357	63.7	66.44	−250	−150
Sborjanovo	347	65		−250	−240
Isperih	352.34	63.71	71.63	−260	−240
Vetren	348.5	64	71.72	−280	−170
Gomolava	X	X	77.05	−320	−240
HB KILNS	349.2	63	77.23	*−325*	*−280*
Vetren	352.77	68.51	87.73	−300	−280
Sevtopolis	X	68.7	80.22	−325	−275
Isperih	344.25	61.75	82.99	−350	−320
Lazar Stanevo	X	X	79.9	−420	−380
Vetren	343.03	59.61	X	−430	−380
Kardzhali	X	X	75.14	−560	−360
Bugojno	357.77	63.63	X	−550	−500

Dec. = declination [°], Inc. = inclination [°] and Fa. = intensity [μT] of the Earth's magnetic field. Positive values are A.D. and negative values B.C.

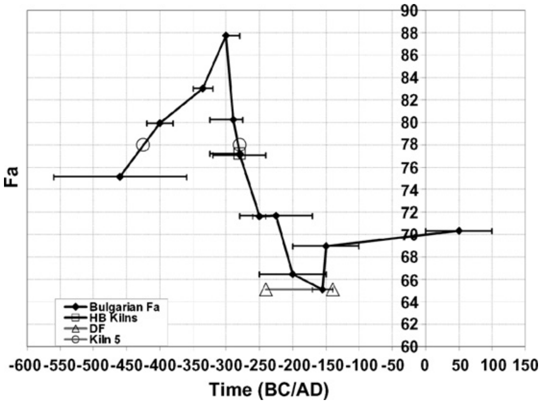

Fig. 10. The Halka Bunar Archaeointensity data plotted against the Bulagrian Archaeointensity (Fa) curve. The AI (square: 77.23 ± 2.17 μT) for the main kiln complex at Halka Bunar has been incorporated into the curve. The AI of Kiln 5 (circle: 78.0 ± 1.7 μT) suggests that it is most likely contemporary with the kiln complex, although it could equally data to the 5th century B.C. The AI of the DF (triangle: 65.1 ± 1.1 μT) suggests that it most likely dates to between 250 and 140 B.C.

The mean AI and AD data for these three kilns will be used to form a reference point for the Bulgarian Archaeomagnetic Database (see Kovacheva, 1997) because the data came from well dated archaeological features. This provides a vital new point for this low resolution period.

In contrast, the AI of the Earth's magnetic field is quite distinct for the 3rd to 4th centuries B.C. in Bulgaria (Table 4; Fig. 10) and provides a more reliable means of age assessment during this time period despite the resolution of the data. As the ages of Kiln 5 and the DF were unknown and no AD data was recovered from the structures, archaeomagnetic dating was attempted using only the AI data. The mean AI for Kiln 5 is 78.00 ± 1.7, which is consistent with the main kiln complex at ca. 280 B.C. The only other period that the kiln could date to is the late 5th C. B.C., and this seems highly unlikely. There is some suggestion from stratigraphic analysis that the DF is likely younger than the main kiln complex. The DF has much lower mean AI values of 65.1 ± 1.1 μT, which indicates this to be the case, most likely dating to between 250 and 140 B.C. (Table 4 and Fig. 10). The archaeological *terminus ante quem* has been based on a coin of Lisimah (306–281 B.C.; Tonkova) being found in association with the kilns.

The mineralogy and Thellier results of the DF suggest a single, low temperature heating in antiquity in an uncontrolled oxidising environment. The DF may therefore have been destroyed towards the end of the sites use and suggests the site was occupied for a longer period than expected. The suggestion of multi-phase activity at the site is borne out in the archaeomagnetic data with a main period of occupation in the late 4th and early 3rd century B.C. and another phase of occupation in the mid 3rd to mid 2nd century B.C. The change in occupation at the site may have occurred around 279 B.C. when there were a number of Celtic invasions and settlement into Bulgaria. Further archaeomagnetic data is needed for the Bulgarian database to better constrain the second phase of occupation at Halka Bunar.

6. Conclusions

(1) Viscously cleaned NRMs are a true indicator of field direction as long as a single component of magnetisation exists in the sample. For samples that have been heated in a reducing atmosphere in antiquity, alternating field demagnetisation is preferable over thermal demagnetisation due to alteration.
(2) Variation in both archaeodirectional and archaeointensity data is seen due to collapse and destruction of kilns.
(3) Variation in archaeointensity data is also seen due to differences in the magnetic mineralogy of the clays studied. This relates to variation in temperature and atmosphere of firing in antiquity.
(4) The data suggests that colour can be an important indicator of the temperature and atmosphere of heating in antiquity, and therefore the potential reliability of the samples for AI analysis.
(5) The occurrence of post-heating maghaemite in some surface samples indicates that detailed mineralogical analysis is preferable before undertaking Thellier analysis.
(6) The suggestion of multi-phase activity at the site is borne out in the archaeomagnetic data.
(7) Archaeodirectional and archaeointensity data from three contemporary kilns (Kilns 2–4) were presented that provided a valuable new archaeomagnetic reference point for the Bulgarian database in the late 4th and early 3rd century B.C. (325–280 B.C.), a period of low resolution.
(8) An analysis of archaeointensity data suggests that Kiln 5 is contemporary with the main kiln complex.
(9) An analysis of archaeointensity data suggests that the destruction feature belongs to a later phase of occupation and is optimally dated to between 250 and 140 B.C. and is most likely mid-late 3rd century B.C. in date.

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