Archaeological calibration of remagnetized volcanic rocks from pottery firing kilns in Cuentepec, Morelos, Mexico

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RESUMEN

Investigaciones etnoarqueológicas en Cuentepec incluyen experimentos durante la producción de cerámica, de donde es posible extraer conocimientos sociales a partir de la aplicación de técnicas arqueométricas. En este caso, el experimento trata sobre la confiabilidad de técnicas de fechamiento en arqueología. En Cuentepec, se usan pequeños hornos a cielo abierto para la fabricación de cerámica (comales de barro). Se tomaron muestras de roca volcánica que conformaban los hornos para verificar la confiabilidad de la dirección magnética registrada por las mismas y compararla con datos del Observatorio Geomagnético de Teoloyucan localizado cerca a la ciudad de México. Con el objeto de medir sus propiedades magnéticas se perforaron/obtuvieron en el laboratorio 47 núcleos pertenecientes a ocho muestras de bloque orientadas. Las curvas continuas de susceptibilidad magnética con altas temperaturas resultaron en muchos casos razonablemente reversibles, con puntos de Curie sugiriendo titanomagnetita de rica a pobre en titanio. Los parámetros de histéresis indican que todas las muestras caen en la región de tamaño de grano pseudo-dominio-simple, indicando probablemente una mezcla de granos multidominio más una cantidad significativa de granos de dominio simple. Las curvas de adquisición de magnetización remanente isotermal fueron muy similares para casi todas las muestras. La saturación se alcanzó en campos moderados del orden de 100-120 mT, lo cual indica algunas espinelas como portadores de la remanencia. Concluimos que las muestras obtenidas de la parte interna de los bloques que forman los hornos, las más cercanas al fuego, guardan los registros más confiables del campo geomagnético. Esto significa que el calor producido por el fuego probablemente sólo remagnetizó las partes internas de los bloques.

PALABRAS CLAVE: Experimento arqueomagnético, Cuentepec, México.

ABSTRACT

Ethnoarchaeological research at the site of Cuentepec, Mexico includes experimental pottery dating in which social knowledge is obtained from archaeometric techniques. At Cuentepec, open kilns are used for firing pottery. Samples from volcanic rocks in the kilns were taken to verify the reliability of the magnetic direction in these rocks as compared with data from Teoloyucan Geomagnetic Observatory, near Mexico City. In the laboratory, forty-seven cores from eight hand oriented rock samples were drilled. Continuous susceptibility measurements at high temperature yield in most cases reasonably reversible curves with Curie points ranging from Ti-rich to Ti-poor titanomagnetite. The ratios of hysteresis parameters indicate that all samples fall in the pseudo-single domain grain size region, probably indicating a mixture of multidomain plus a significant amount of single domain grains. Isothermal remanent magnetization acquisition curves were very similar for nearly all samples. Saturation is reached in moderate fields of the order of 100-120 mT, which points to some spinels as remanence carriers. We conclude that the samples obtained from the inner part of the blocks in the kilns closer to the fire kept the most reliable records of the geomagnetic field.

KEY WORDS: Arquaeomagnetic experiment, Cuentepec, Mexico.

INTRODUCTION

Archaeomagnetism as a dating technique has been successfully used at archaeological sites over the past three decades. This technique is based on the principle that magnetic minerals included in clay-lined archaeological features, such as kilns or floors, will preserve the existing direction and strength of the Earth's magnetic field as they are heated to high temperatures. Unless the kiln or fireplace has been reheated to higher temperatures that direction will be "locked in" and can be measured in the laboratory.

Archaeomagnetism is useful as an application of paleomagnetic methods to archaeological problems, because the magnetic field changes through time. By measuring the original geomagnetic field strength and direction in

archaeological materials, we are able to determine the most probable date of last firing of clay-lined fireplaces or similar artifacts.

Interest in the magnetic properties of pottery firing kilns in Mesoamerica was developed during the 1970's. In Mexico, archaeomagnetic measurements were done by Wolfman (1973) and Urrutia (1975). Wolfman (1973, 1990) developed an improved paleosecular variation curve at the Paleomagnetism Laboratory at the National University of Mexico (UNAM).

Our calibration experiments are related to estimating the reliability of dating methods in archaeology. At Cuentepec, open kilns are still being used for firing pottery. Samples of volcanic rocks from the kilns were taken to test the consistency of estimates of the magnetic direction and intensity recorded in these rocks as compared with data from the Teoloyucan Geomagnetic Observatory near Mexico City.

ARCHAEOMAGNETIC DATA

Historic observations of the geomagnetic field offer a direct record of secular variation, but these observations are limited in space and time (e.g. Malin and Bullard, 1981). Fired clay is commonly found at archaeological sites around the world. They can provide snapshots of the local geomagnetic field at specific points in time (Lund, 1996). Fired, stationary clay features such as kilns and ovens may provide accurate paleomagnetic records due to the properties of the magnetic minerals present. When clay-rich features are heated close to or above the Curie temperatures (magnetite = 580 °C, hematite = 680°C) and then allowed to cool to ambient temperatures, they acquire a thermal remanent magnetization (TRM) which is similar to the geomagnetic field at the time. They will retain this magnetization unless reheated to a high temperature, at which point a new magnetization will be acquired. The magnetization that is measured in the laboratory is assumed to have been acquired when the feature was last fired, which in turn is related to a particular cultural event (e.g., firing).

Archaeomagnetic data must be calibrated by independent dating techniques before they can be used in reconstructions. This process involves potential sources of error that could impact the precision dating (Tarling and Dobson, 1995; Wolfman, 1991). Great care must be taken to ensure that an event dated by independent means matches the event recorded by the archaeomagnetic feature, i.e., the date when the feature was last fired. Chronometric dating sources such as historic documents (Lengyel and Eighmy, 2001), dendrochronology (LaBelle and Eighmy, 1997), radiocarbon (Kean *et al.*, 1997), thermoluminescence (Becker *et al.*, 1994), or artifact seriation (Sternberg, 1982)

are typically combined with archaeological inferences to generate an independent estimate for the target event.

SITE AND SAMPLING

Cuentepec is a small town located ~105 km south of Mexico City (18° 51' north; 99° 20' west; Figures 1A and 1B). Eight volcanic rocks from two different kilns were removed from their fixed position after measuring and marking their orientation and securing with plaster (Figure 1C). After placing these block samples in a horizontal position, 47 cylindrical cores were taken in the lab normally to the horizontal plane (see small circles in Figure 1C), using an electric drill. All cores were marked to preserve their original orientation.

TECHNIQUES AND METHODOLOGY

An archaeomagnetic sample collected from an archaeological feature typically consists of between six and twelve oriented cubic specimens (Eighmy, 1991). We collected blocks from which we got the specimens. The data collected from each specimen are averaged to obtain the mean values for the entire archaeological feature. By averaging the data from at least five specimens, we may be able to correct for errors from differences in mineralogy, weathering, and firing times and temperatures within the feature, as well as for errors in specimen orientation made during sampling (Tarling and Dobson, 1995). The archaeomagnetic data recovered from an archaeological feature are typically described in terms of five parameters: declination (D), inclination (I), precision (k), angle of confidence (alpha95), and number of specimens (N).

Declination and inclination are directly measured from the specimens and averaged over the feature. Directional data describe the ambient magnetic field at the time when the feature was last fired. Precision and angle of confidence are Fisher statistical parameters that describe the dispersion of the specimen directional data and the spherical confidence limits, respectively (Fisher, 1953). The fifth parameter, N, is the number of specimens collected from the feature that contributed data to its mean D and I values. Occasionally, one or more specimens may contain data that are judged to be outliers. Thus, N may not be the same as the number of specimens collected. When discussing virtual geomagnetic poles (VGP, poles calculated for one single site), the declination, inclination and alpha95 parameters are replaced by pole longitude, pole latitude, and A95 (the radius of the 95% confidence circle around the calculated pole).

As in any standard paleomagnetic study, the coercivity spectra, magnetic stability and vectorial composition of the magnetization are analyzed. Zijderveld vectorial plots, representing the demagnetization processes, and a principal

component analysis of these vectors, have been used to determine the directions of the components of magnetization and the characteristic or primary direction (Table 1). Usually, cross-correlating the mean site declination and inclination with the paleosecular variation curve of the region (Le Goff *et al.*, 2002), a date may be determined. Often more than one crossing point is obtained for a sample site.

Continuous susceptibility measurements (i.e., susceptibility vs. high temperature curves) were obtained using a Highmoor instrument to identify the ferromagnetic minerals in the samples from their Curie temperatures. This can help guide the design of partial demagnetization experiments and the interpretation of results. The challenge

consists in associating a particular component of remanence with a particular ferromagnetic mineral, thus finding whether a characteristic remanence is primary or secondary. One sample from each site was heated up to 650°C at a heating rate of 20°C/min and then cooled at the same rate.

Small chips of rocks were used for measurement of magnetic hysteresis parameters with an 'AGFM-MicroMag' alternating gradient force magnetometer. Associated IRM acquisition and DC back-field demagnetization curves were also acquired with this instrument, to determine the magnetic mineral type from its magnetic stability and coercivity spectra. Hysteresis measurements at room temperature were performed on one sample from each block up to 1.2T. The
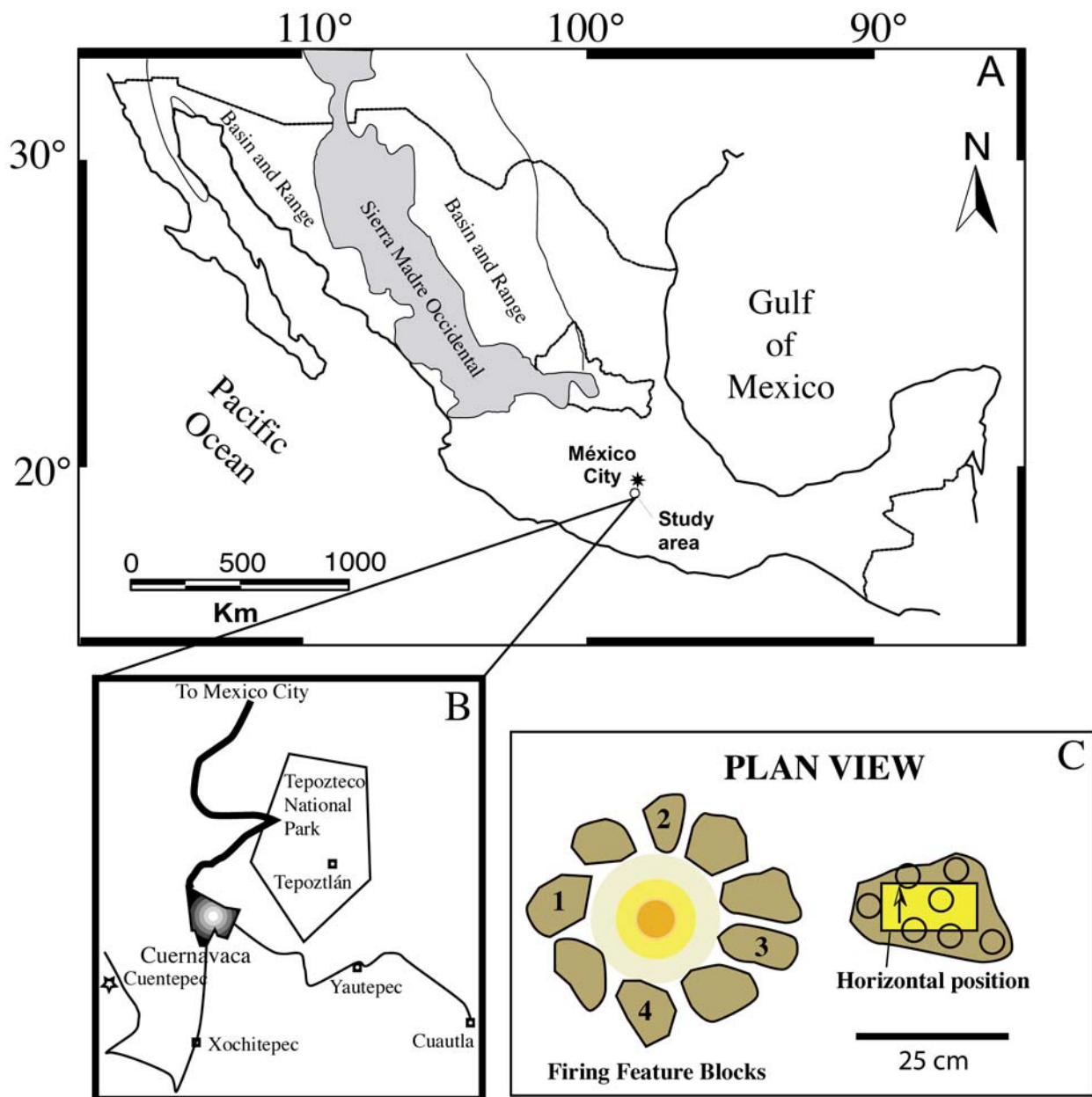

Fig. 1. A and B). Location of the studied area (see the star); and C) sampling blocks procedure.

Table 1

Paleodirectional results from volcanic rocks: N: Number of treated samples, n: number of samples used for calculation, Dec: Mean Declination, Inc: Mean Inclination, k and α_{95}: Precision parameter and radius of confidence cone

Sample	N/n	D	I	K	α_{95}
Firing feature 1					
Block	14/3	330.3	46.0	14.0	25.5
Block 2	6/5	357.2	60.8	103.6	7.6
Block 3	5/4	17.2	42.5	704.6	3.5
Block 4	4/0	----	----		
Mean		**354.9**	**51.8**	**19**	**19.4**
Firing feature 2					
Block 5	5/4	28.6	1.8	13.5	25.9
Block 6	7/6	5.8	14.9	6.4	28.7
Block 7	5/4	272.5	-70.4	131.3	8.0
Block 8	6/4	16.1	36.3	77.1	10.5

saturation remanent magnetization (J_{rs}), the saturation magnetization (J_s), and the coercive force (H_c) were calculated after correcting for the paramagnetic contribution. The coercivity of remanence (H_{cr}) was determined by applying a progressively increasing backfield after saturation.

The intensity and direction of natural remanent magnetization (NRM) were measured with a JR5 spinner magnetometer. The coercivity spectrum, stability and vectorial composition of NRM for every sample were investigated by step-wise alternating field (AF) demagnetization. AF demagnetization was carried out in 8-12 steps up to maximum fields of 100 mT using a Schonstedt AF demagnetizer in the triaxial stationary mode.

RESULTS

One curve corresponding to each block from the two kilns is shown in Figure 2. From the continuous susceptibility measurements, Curie temperature was determined by the methos of Prévot *et al.* (1983). In all cases, the presence of Ti-poor (blocks 4, 5 and 7) to Ti-rich titanomagnetites (blocks 1, 2, 3 and 8) was found. Some samples show evidence of two almost reversible ferrimagnetic phases during heating and cooling (blocks 1, 2, 3, 6 and 8). The lower temperature magnetic phases range between 110-150°C and between 250-400°C, and the higher ones are about 490°C and above 600°C. These latter temperatures could correspond to magnetite and hematite resulting from the heating process, respectively.

Typical hysteresis plots are shown in Figure 3. The curves are quite symmetrical in all cases. Judging from the

ratios of hysteresis parameters (Table 2, H_{cr}/H_c ranges between 1.30 and 4.61 and M_{rs}/M_s varies from 0.070 to 0.235), it seems that most samples fall in the pseudo-single domain (PSD) grain size region (Day *et al.*, 1977; Figure 4, right). This may suggest a mixture of multidomain (MD) plus a significant amount of single domain (SD) grains. Isothermal remanence (IRM) acquisition curves were very similar for all samples (Figure 5). Saturation is reached in moderate fields of the order of 100-120 mT, which points to spinels as remanence carriers. However, there are one (two) blocks in kiln 1 (2) that show IRM curves that do not reach saturation, suggesting the presence of high coercivity minerals, most probably hematite.

Some of the samples are characterized by simple univectorial component and most of them by two-component plots: one probably of viscous remanent magnetization origin can be removed at the first demagnetization steps (soft magnetic phase) and the second corresponds to the characteristic magnetization (Figure 6). The characteristic direction (ChNRM) for each sample was calculated from the vector plots and corresponds to the vector component going through the origin. Samples for which the vector plots did not pass through the origin were not used for the calculation of site means. Site-mean directions were calculated by vector addition giving unit weight to sample directions. Fisher statistics was used to estimate dispersion parameters. The overall-mean direction was calculated giving unit weight to site-mean directions. Site-mean directions and overall-mean direction, and associated statistical parameters are summarized in Table 1.

Site-mean inclinations vary within a wide range, from 1.8° to 60.8° (no negative value), and site-mean declinations
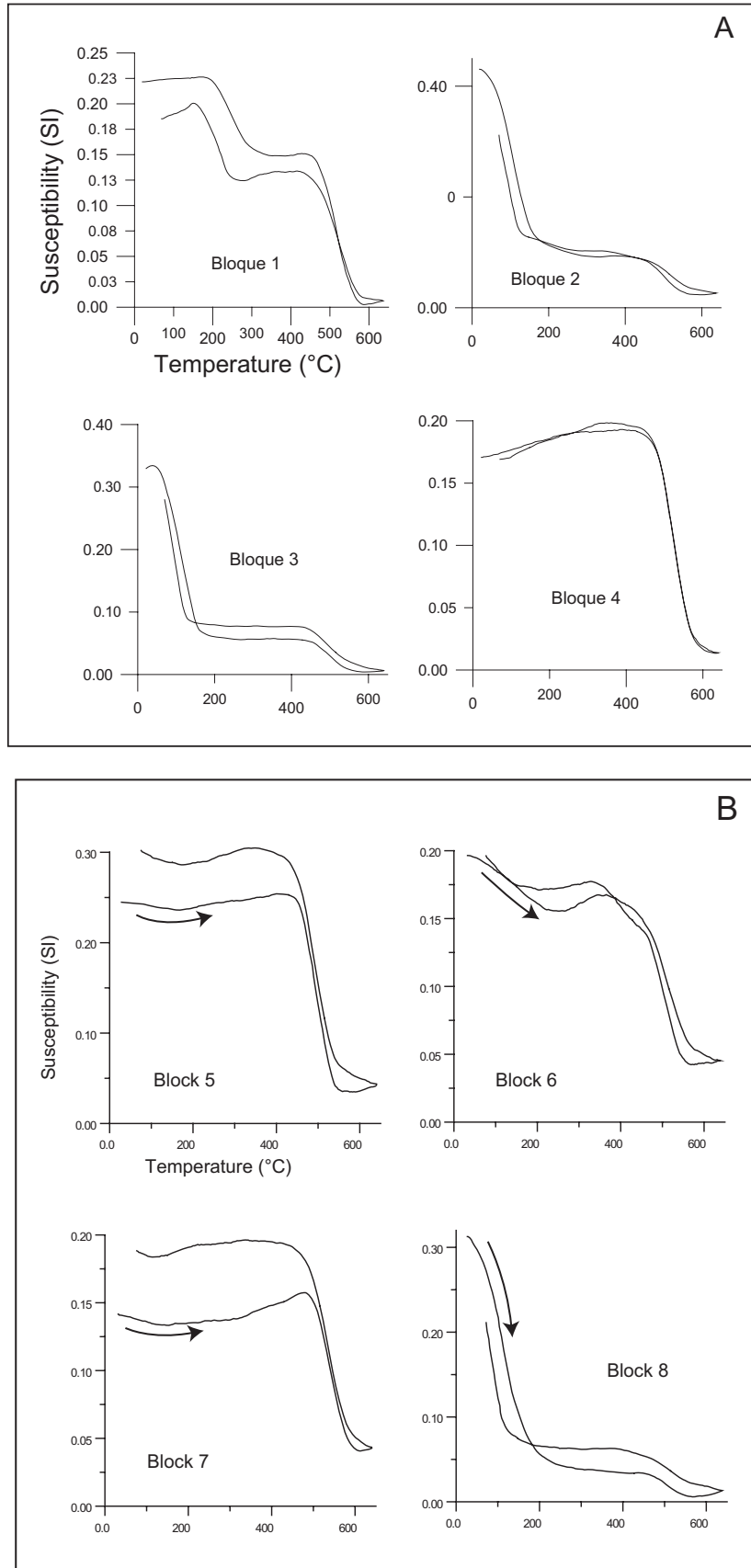
Fig. 2. Continuous susceptibility curves versus high temperature. A) Firing feature number 1. B) Firing feature number 2.
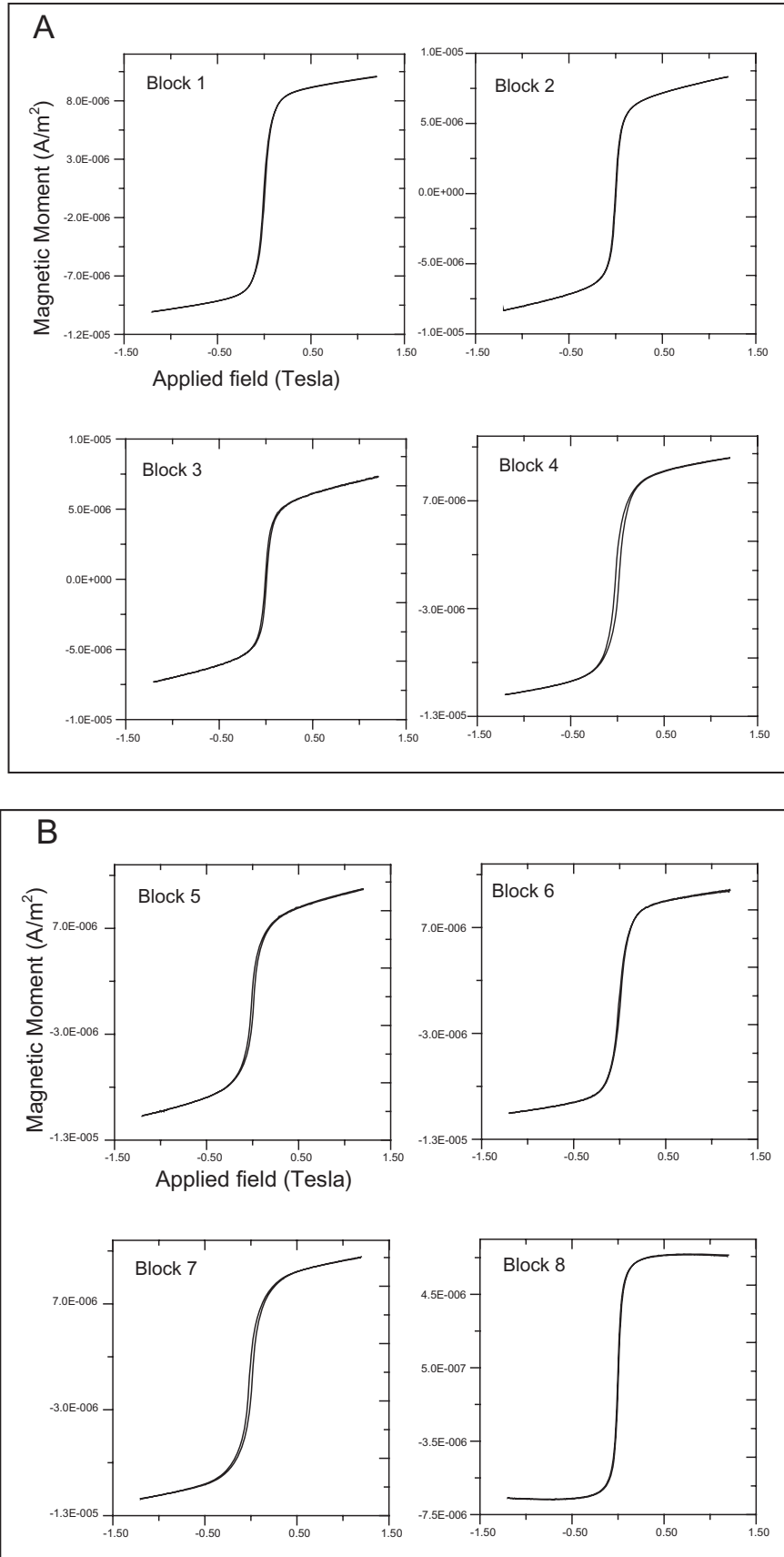
Fig. 3. Examples of hysteresis curves measured with a MicroMag system. A) Firing feature number 1. B) Firing feature number 2.

Table 2

Magnetic hysteresis parameters

Block	Mr(nAm2)	Ms(nAm2)	Mr/Ms	Hcr(mT)	Hc(mT)	Hcr/Hc
1	729	8550	0.085	9.09	5.72	1.30
2	450	6440	0.07	5.23	3.84	1.36
3	679	5340	0.127	11.6	7.19	1.61
4	2610	11100	0.235	27.5	18.6	1.48
5	1320	7920	0.166	17.4	11.0	1.58
6	740	8920	0.083	19.5	7.05	2.77
7	1750	9160	0.191	28.3	14.7	1.93
8	453	6590	0.069	14.0	3.04	4.61

Note: Mr/Ms and Hcr/Hc, hysteresis parameters measured in small chip rocks.

vary from 330.3° to 28.6° (omitting the extreme value 272.5) (Table 1). The overall-mean direction estimated for kiln 1 is: Dec=354.9°, Inc=51.8°, k=19, and α_{95} =19.4°. That for kiln 2 could not be determined because of an anomalous high dispersion. Overall and site-mean directions are distributed away from the dipolar direction (Id = 35.3°) and closer to the corresponding direction (I_g = 47.1°) estimated from the Teoloyucan Geomagnetic Observatory.

The within-site (kiln) angular dispersion of NRM and ChNRM directions is much higher than expected for volcanic rocks used for kilns with univectorial remanences.

DISCUSSION AND CONCLUSION

In some samples, the continuous curves of low-field susceptibility vs. high temperature indicate the presence of
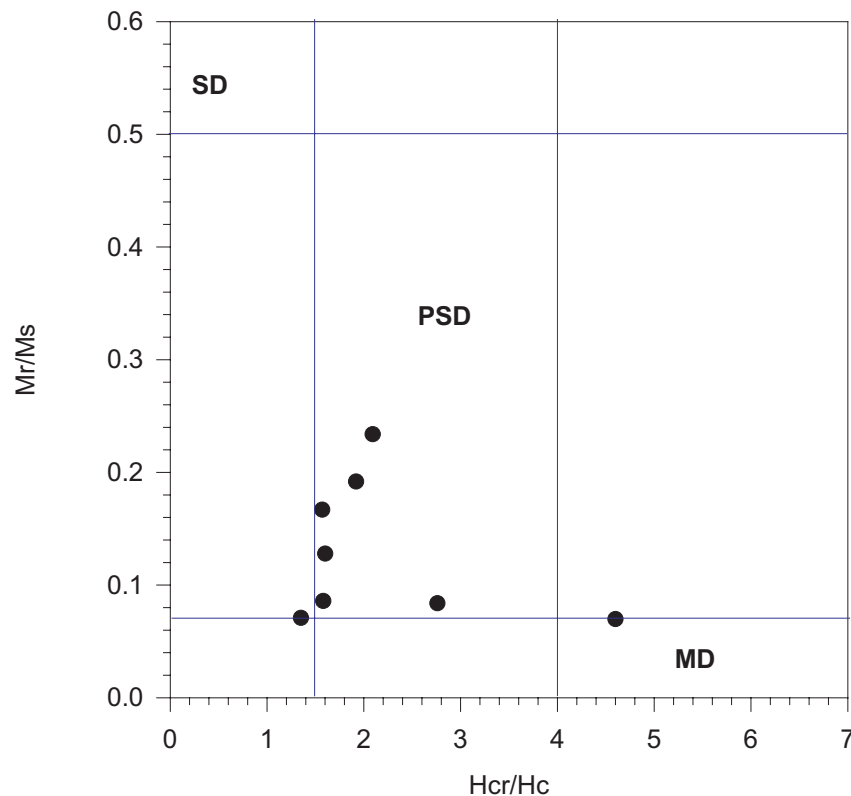

Fig. 4. Magnetic hysteresis parameter ratios plotted in a Day diagram (Day *et al.*, 1977).

a single ferromagnetic phase with a Curie point compatible with nearly pure magnetite (Figure 2, block 4, 5 and 6). Sample from block 1 shows evidences of two ferrimagnetic phases during heating. The lower Curie point ranges from 250° to 400°C, and the highest one is about 580°C. The cooling curve shows similar phases. Blocks 2, 3 and 8 show a rapid decay of the heating curve at 100°-150 °C and another at about 580°C, suggesting maghemite transformed to magnetite. The cooling curve shows also both phases.

The reversibility of cooling and heating curves indicates whether the magnetic minerals are stable or if they change their mineralogical phase due to increase in temperature (i.e. oxidation in air).

In general, the heating and cooling behavior could correspond to small pseudo-single-domain to multidomain magnetic grains (Dunlop and Ozdemir, 1997). The susceptibility values for higher Curie temperatures (blocks 5, 6 and 7) are useful to define the possible contribution of hematite over magnetite.

Analysis of hysteresis parameter ratios indicates that almost all samples fall in the PSD grain size region (Figure 4; Day *et al.*, 1977).

The IRM curves (Figure 5) also show that the blocks are composed by a mixture of titanomagnetite and titanohematite with varying Ti content.

The volcanic rocks that form the base of kiln number 1 have an overall-mean inclination (51.8°) that is about 5° higher than estimated from the Teoloyucan Geomagnetic Observatory (47.1°) or from the 1945 and 1950 IGRF models (Urrutia-Fucugauchi and Campos-Enríquez, 1993). The mean declination for the same firing feature is 354.9°, which (plus 7° of magnetic declination) gives D = 2°, lower than the expected value of 7°. In searching for an explanation, several factors have been considered: (a) instrumental and human error during sampling and measurement, (b) movement of blocks, (c) partial reheating of the blocks below the magnetic blocking temperature spectra during hand-made manufacture of 'comales'. The first and second factors related to the blocks and their relative position within the kiln are discarded because of the great care taken during sampling and measurement.

Vector plots (Figure 5) do not show any significant effects of secondary magnetization components that could affect the inclination/declination values.

The site-mean inclinations/declinations present a wide range. Note that not all blocks show high inclinations. In summary, the characteristic magnetic directions determined

in this study seem to be of primary TRM origin. This is supported by the thermomagnetic investigations which show that the remanence is carried in many cases by Ti-poor titanomagnetite, resulting from oxy-exsolution of original titanomagnetite during the last heating process, indicating thermoremanent origin of primary magnetization. Moreover, unblocking temperature spectra and relatively high coercivity point to 'small' pseudo-single domain magnetic structure grains as responsible for remanent magnetization. Single-component (including small VRM component), linear demagnetization plots were observed in most cases. The paleomagnetic record is, however, predominantly characterized by higher than expected site-mean inclinations as compared with data from the Teoloyucan Geomagnetic Observatory and estimates based on International Geomagnetic Reference Field 1945 and 1990 models.
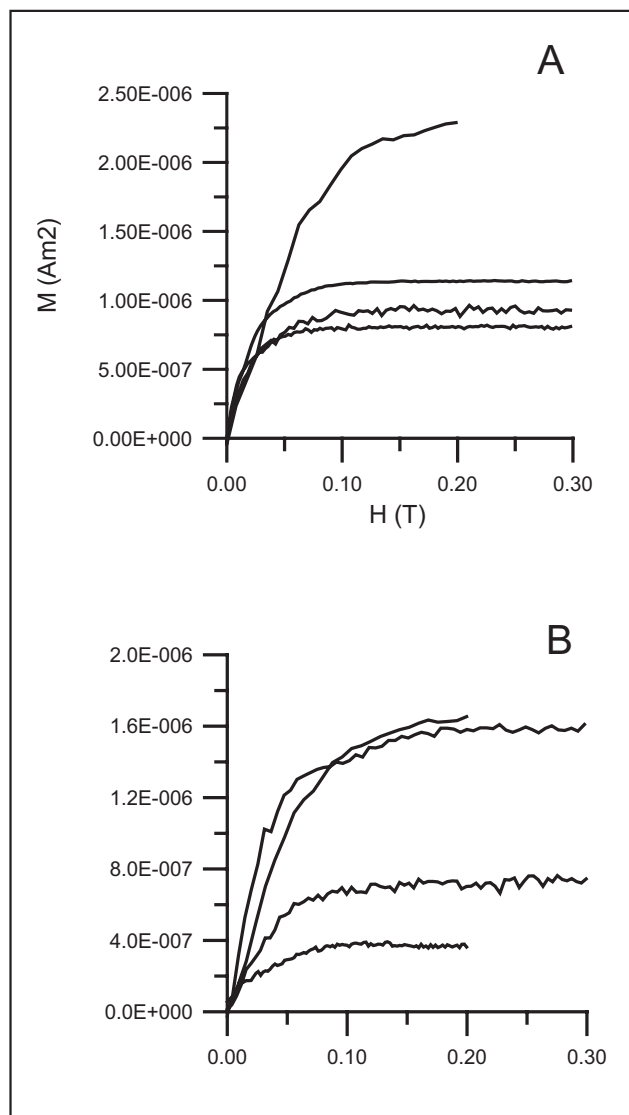

Fig. 5. IRM acquisition curves from small rock-chips: A) Firing feature 1, and B) Firing feature 2.
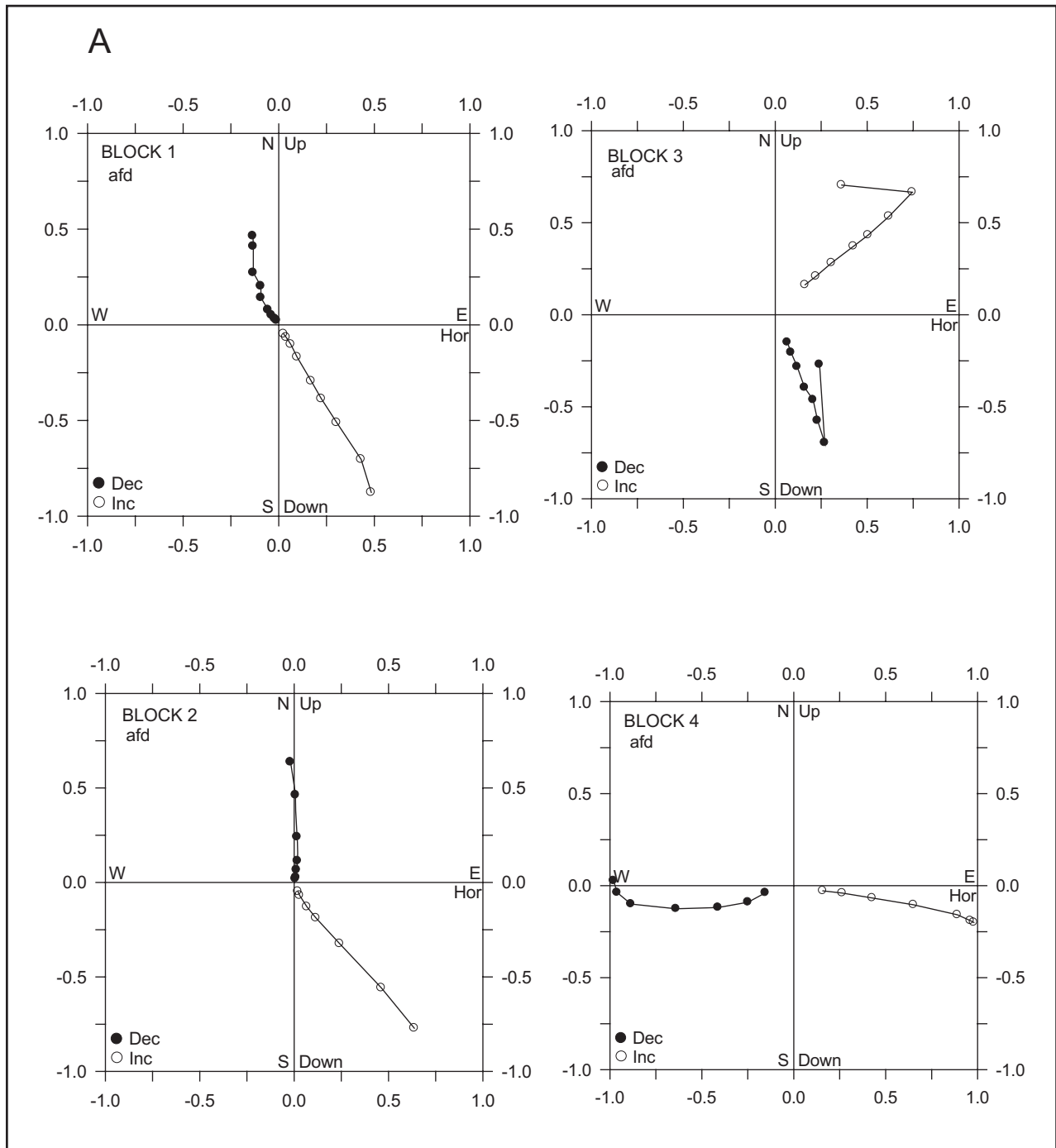
Fig. 6. Orthogonal vector plots of stepwise alternating field demagnetization (stratigraphic coordinates). The numbers refer either to peak alternating fields in mT. • – projections into the horizontal plane, x – projections into the vertical plane. A) Firing feature number 1. B) Firing feature number 2.

Analyses of paleomagnetic directions indicate that they are dispersed at the within-block level, probably due to the different degree of heat varying from the internal (major) to the external (minor) face of the block respect to the center of the fire (Figure 1). This means that the date for the maximum temperature (equivalent to 800 °C) was not hot enough to remagnetize the entire blocks around the kiln.

Some blocks facing the inside of a kiln suggest a close approximation to the expected direction of the present geomagnetic field, but other specimens differ largely from this. For example, block 4 has a very large dispersion (making impossible determining mean direction); and block 7 shows a very deviated declination and negative inclination (Table 1, Figure 6). We conclude that samples obtained from
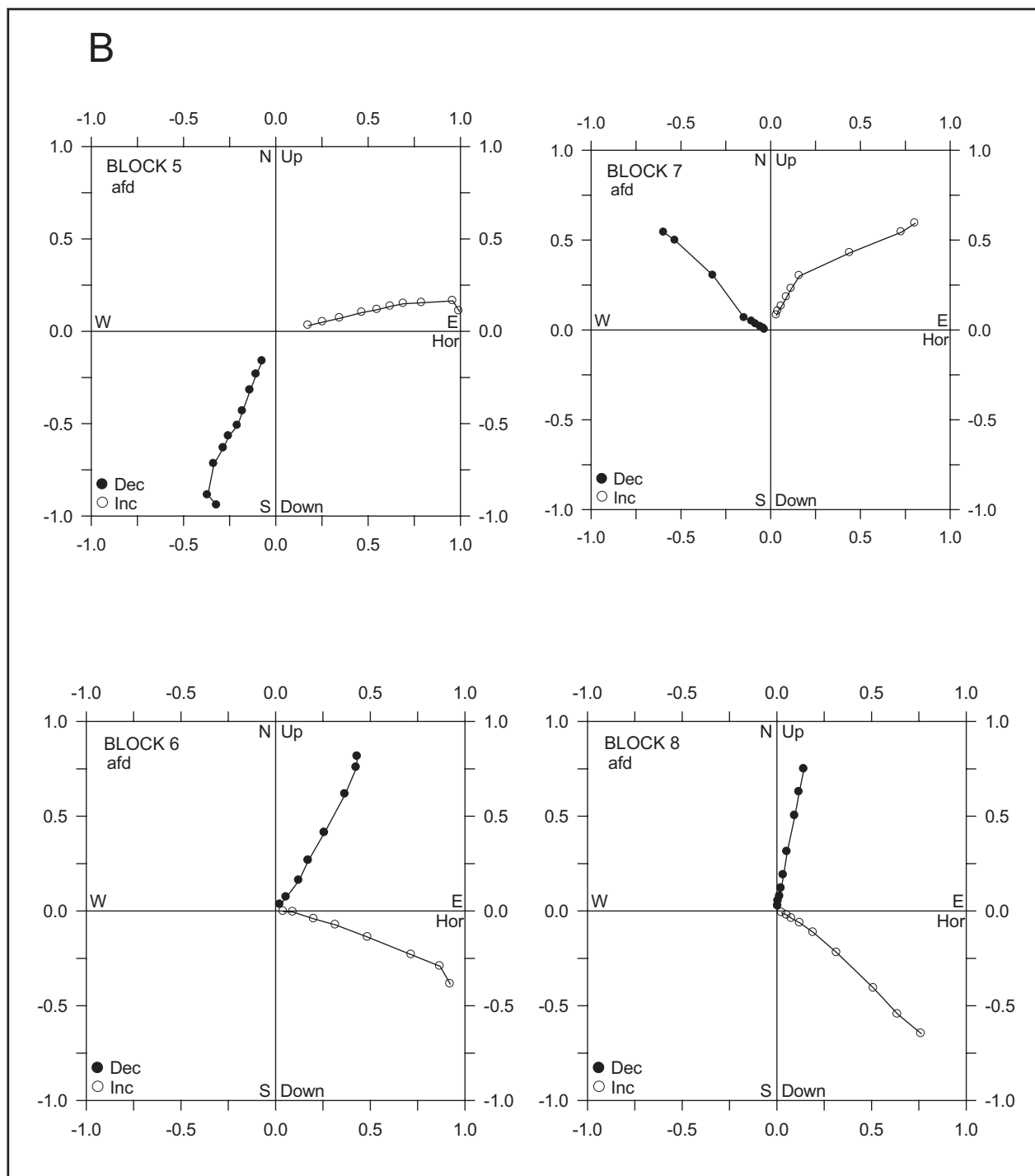
Fig. 6. Continued.

the inside-looking part of the blocks in the kiln facing the fire, present the most reliable records of the geomagnetic field, suggesting that the heat produced by the fire may have remagnetized a few centimeters in the inner part of the block. The other samples may have a mixture of primary magnetic remanence plus effects of partial reheating or alterations produced by natural or man-made events, such as oxidation, minor movements, etc.

A further paleomagnetic experiment may be necessary to test the influence of the relative position of cores/blocks regarding position with respect to the fire.

ACKNOWLEDGMENTS

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