



ARCHAEOMAGNETIC INTENSITY OF CERAMIC SHERDS FROM TWO RHODIAN BYZANTINE CHURCHES: A PRELIMINARY INITIATIVE

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ABSTRACT

Archaeointensity results were obtained from ceramic sherds incorporated in the 'mortar' of two Rhodian Byzantine churches. Samples were analysed using a modified Thellier method with samples orientated so that the Natural Remanent Magnetization is at right angles to the applied laboratory field. Only a few samples showed high magnetic stability and an archaeointensity result was obtained from ceramic sherds (tilebrick) from the lower interior walls of the church (Afandou KATHOLIKI). Comparison with the Geomagnetic Field Variation plot indicated the probability density of possible dates between ~1000 to ~1500 AD. a date, broadly consistent with historical records.

KEYWORDS: Rhodian Byzantine Churches, Archaeointensity, Ceramic sherds, Modified Thellier method, Dating

INTRODUCTION

Archaeomagnetic studies have allowed for the determination of the palaeomagnetic secular variation (PSV) record of the past geomagnetic field and have been used as a means to establish absolute or relative dates for suitable archaeological materials including pottery sherds (Kovacheva *et al.*, 1998). When pottery is removed from a kiln, generally, only the archaeomagnetic intensity can be determined, as the orientation of the firing position is lost and the directional parameters (declination and inclination) cannot be determined. It is the small magnetic minerals within clay-based materials used for the manufacture of ceramics which record the strength and direction of the ambient magnetic field after they have cooled in-situ. Palaeo Secular Variation (PSV) curves for ancient magnetic field intensity, declination and inclination, have been established regionally from such archaeological and other natural materials, such as lavas and indicate the past behavior of the geomagnetic field. If the archaeointensity of suitable materials, such as pottery sherds from a particular archaeological context, is known, then, it is possible to compare such archaeointensity results with the established PSV curves and thus obtain a date. A preliminary study was made on thirteen samples of pottery sherds, taken from two Rhodian Byzantine churches: St. Nicolas Foundoukli, and Afandou Katholiki, both of uncertain age spanning between 10th to 17th c AD. Archaeointensity determinations were made on the sherds which had been incorporated in the wall mortar, using the Modified Thellier method, Kono and Ueno (1977), which uses single heating during treatments, however, some modifications were made to the sample preparation. As a prerequisite criterion for determination of ancient magnetic field strength, only samples which exhibited a very high magnetic stability to progressive thermal demagnetization (PTD) were used. PTD results used with principal component analyses showed that a single component of remanent magnetization remained up to ~500-550°C and Viscous Remanent Magnetization (VRM) was removed after the ~100°C treatment. Previous investigations initiated on Byzantine Churches by one of us (IL) include

Liritzis and Kovacheva (1992), Aitken *et al.*, (1989), and Liritzis (1989).

SAMPLES AND SAMPLING

Two Rhodian Byzantine churches were sampled: St. Nicolas Foundoukli, East wall (5 sherds, code NFE) and South wall (3 sherds, code NFS) of ~15th c AD (Achimastou-Potamianou, 2004) and Afandou Katholiki, (Orlandos, 1948, Sifopoulos 2004) Lower interior wall (3 sherds, code AKL) and Upper interior wall (2 sherds, code AKU).

For the church at Afandou Katholiki (Sifopoulos, 2004) according to tradition construction started ~10th c AD there was a temple dedicated to Demeter, built at that location in the 3rd. century BC. This was destroyed but in the 5th- 6th century AD, another building, a triparted palaeochristian Basilica was erected, possibly using some of the materials from the previously destroyed site. This in turn was destroyed by unknown causes in the 8th century AD. A new church was built upon the ruins of the palaeochristian church ~10th c. AD with well constructed Gothic arches but occupied only the mid-aisle area of the earlier construction. It bears wall paintings believed to be from the 14th. to 16th centuries AD or later 17th c AD. (Orlandos 1948) (Photos 1A, B).

METHODS

Treatment and Sample Preparation

Measurements were made using the MMTD (Magnetic Measurements Thermal Demagnetizer) and Molspin fluxgate magnetometer, (Molyneux, 1971). The essential principles of the single heating modified Thellier method (Kono and Ueno, 1977) are applied in this study with an additional step which allows a laboratory field Thermoremanent Magnetization (TRM) to be directed, exactly perpendicular to the stable Natural Remanent Magnetization (NRM), 'cleaned' of any VRM components. Kono and Ueno (1977) routinely removed VRM components using a 100 °C step in thermal demagnetization treatments. It is however, indeed preferable to experiment with samples of the same type as those to be used for archaeointensity deter-

minations, to establish the exact temperature level, required to remove these soft secondary

components before proceeding with ancient field intensity determinations.



Photo 1. View of present status of the two churches: (a) Aghios Nikolaos Foundoukli, (b) Afandou Katholiki

Technical note on sample preparation

'Directional' methods have been applied in Thellier-type treatments to obtain archaeo and palaeo-intensity results with a laboratory field applied in the same direction as the NRM with success in the use of ceramics obtained from Peru, China and Egypt (Shaw *et al.*, 1996, 1999) and lavas from Italy (Hill and Shaw, 2000).

This is carried out using the controlling program for microwave heating to allow the field coils around the microwave cavity to be programmed so that the laboratory field is applied in such a manner. In this study, a conventional modified Thellier method is used and the sample itself can be physically oriented, so that a laboratory can be applied (TRM) at 90° to the 'cleaned' NRM direction.

After the direction of the NRM vector of the sample is established, it can be oriented so that it is confined to the plane of the circular end plane of a standard one inch (2.54 cm.) 'palaeomagnetic' sample, in this case, a perspex or quartz open-ended cylinder.

Archaeological samples of burnt mud brick or pottery sherds are not drill cored and may be fairly irregular, so the sample is firstly cut to an approximate 1cm side cube. This regular shape may also have benefits in reducing magnetic anisotropy effects especially those related to shape anisotropy (O'Reilly, 1984).

This size of sample can fit into and be rotated within the standard cylinder. On a small mound

of magnetically clean plasticine placed on a bench, the cylinder is pushed on to it vertically and then carefully withdrawn, retaining the flat-surface plasticine onto which an arbitrary fiducial mark can be drawn. The 'cubed' pottery sample is then pushed gently into the plasticine in random orientation, making sure that it is well secured and at the approximate centre of the cylinder. The magnetic remanence is then measured and a note made of the inclination value.

The sample is then re-orientated by a small amount in any direction by accessing it from the open end of the cylinder before a second remanence measurement is made. The inclination value is checked to see if it has increased or decreased. This process is repeated until the inclination value becomes zero at which point, the NRM vector lies in the plane of the end face of the cylinder. This is necessary, so that when the cylinder is lying elongate in the furnace tray, a laboratory TRM (applied along the axis of the furnace) is directed at right angles to the NRM direction.

Next, the open end of the cylinder is filled with non-magnetic "Plaster of Paris" (gypsum) making sure no air bubbles remain and the filled end is smoothed off. The sample is allowed to dry after which the plasticine originally retaining the sample but now retained by the hardened plaster of paris, can be removed. Complete removal of the plasticine is essential so that there is no burning of any residual plasticine

during the subsequent elevated temperature treatments. The open end is then filled with plaster of paris and smoothed off, so that the sample is completely contained within the cylinder. When dry, an arbitrary fiducial mark can be made on the cylinder-end surface. The monitoring of the arbitrary declination direction (fiducial mark), after each heating step is important as it indicates any departure from the NRM - TRM plane.

As an additional check (pTRM; partial TRM), on the arbitrary declination value (which would indicated a departure of the measured vector from the NRM - TRM plane), a field free space (FFS) heating/cooling step can be made between 400°C to 550°C (before remanence is complete-

ly lost) depending on the magnetic constituents type and Curie temperature (T_c) of the sample. Maximum furnace temperatures up to 650°C or above 700°C can be used were cylinders are pyrex or quartz glass respectively (ie) for a sample containing hematite.

For ancient field strength determinations, a choice of laboratory TRM value, close to the ancient archaeomagnetic field intensity should be used so that the condition $F_{Ancient} / F_{lab.} = 1$ is satisfied (Aitken, 1981).

Experimental laboratory TRM fields of 31, 37, 49 and 57 μT were applied to different samples (Table 1). Only two samples AKL2 and 3 fulfilled the criterion $F_{Ancient}/F_{lab.} = 1$.

TABLE 1. Magnetic results from PTD and Modified Thellier archaeointensity determinations. NFE and NFS are codes for samples from St. Nicolas Foundoukli (East and South) exterior walls, respectively. AKU and AKL are codes for samples from Afandou Katholiki (Upper and Lower) interior walls, respectively.

Sample No.	No. vector end pts. used in regression	Magnetic Stability	Gradient	(F^L) Lab Field (μT)	(F^A) Uncorrected Ancient Field (μT)
NFS 1	4	Fair	1.4481	49	70.8
NFS 2	4	Good	1.4962	49	71.1
NFS 3	4	Good	1.4129	49	69.2
NFE 1	11	Good	1.5001	31	46.5
NFE 2	4	Poor	2.0214	31	62.7
NFE 3	8	Poor	2.4355	31	75.5
NFE 4	7	Poor	0.7362	49	36.1
NFE 5	10	Poor	0.8803	49	43.1
AKU 1	11	Fair	1.9628	37	72.6
AKU 2	11	Very good	1.6389	37	60.6
AKL 1	8	Fair	0.8301	57	47.3
AKL 2	12	Very good	0.9727	57	55.4
AKL 3	12	Very good	0.9382	57	53.5

	Corrected 9% \pm 3%	(F ^A /F ^D) -1
AKL 2	(F ^A) Mean	
AKL 3	(μT)	
	49.6 \pm 1.6	0.12 \pm .03

Demagnetization and modified Thellier method

Progressive thermal demagnetization (PTD) was carried out on test samples from each sherd at 50°C intervals (Fig.1) and the low field susceptibility measured after each heating cooling step to monitor for any thermally induced irreversible mineralogical changes. No significant changes were observed. The laboratory cooling period was 'forced' and generally about 2 hours down to room temperature. The shape of the demagnetizations curves together with principal component analyses indicates the stabilities of the NRM (Table 1, Fig. 1). VRM components were removed after the 100°C PTD step. Magnetic remanences appears to diminish to less than 10% at approximately 500 – 550°C possibly indicating that the magnetic carrier is magnetite or low titanium titanomagnetite (Fig.1).

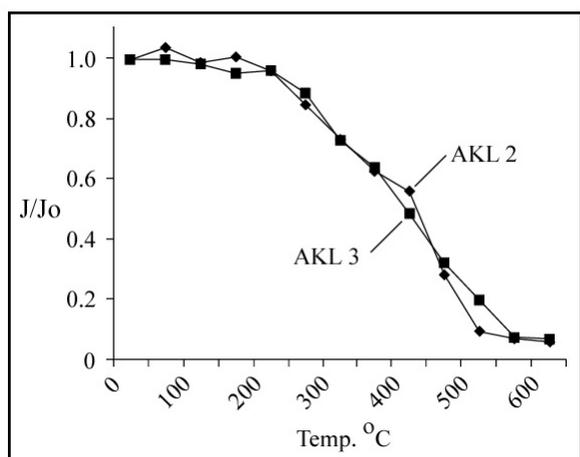


Figure 1. Progressive Thermal Demagnetization curves. Normalized decay of NRM with incremental increase in temperature for two sherd samples from Afandou Katholiki.

Other samples (intended for archaeomagnetic intensity determinations) were routinely thermally demagnetized at 100°C before sample preparation. For the determination of ancient magnetic intensity, single heating steps were made at steps of 25°C up to a maximum of 400°C with forced cooling. Low field susceptibility was monitored after each heating/cooling step and a non-magnetic field, heating/cooling step applied at higher temperature in order to check for consistency in the arbitrary declination of the sample. This is necessary to confirm that the magnetization has remained in the TRM-NRM plane. A

deviation of more than 2 degrees in the arbitrary declination value (ie) out of the plane is regarded as unacceptable.

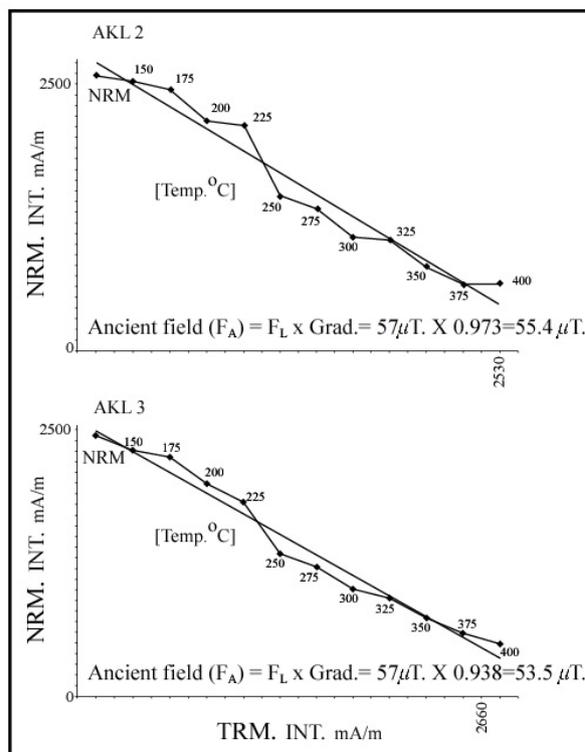


Figure 2. Archaeointensity values obtained from linear regression analyses (gradients), using the Modified Thellier method with samples' NRM oriented at 90° to the laboratory field (TRM).

RESULTS AND DISCUSSION

Only two samples, AKL 2 and 3 after receiving a laboratory field of 57 µT came close to satisfying the condition $F_{ancient}/F_{lab.} = 1$. The gradients obtained from the 'best fit' linear regressions are 0.97 and 0.94 respectively with calculated 'raw' archaeointensities of 55.4 and 53.5 µT. (mean $\approx 54.45 \pm 0.93$) (Table1, Fig.2).

Concerning the determination of archaeintensities and notwithstanding, any apparent mineralogical changes, Barbetti *et al*, (1977) noticed that Thellier-type measurements of ancient field intensity on specimens of clay and sediments which were apparently well-baked and oxidized in ancient times, often fail to give consistent results over part or even most of the blocking temperature spectrum. They suggest, that post-baking chemical alteration or "weathering" (when not preserved in a dry environment) leads to the presence of hydrated iron minerals

which are major cause of the non-ideal behavior in such materials. The behavior of hypothetical specimens containing either goethite or lepidocrocite can be predicted using simple models. The presence of such hydrated minerals may not be readily detected by methods other than Thellier type techniques and would result in estimates of ancient geomagnetic field strength that are systematically too low. A forced cooling rate at 30°C per minute using the MMTD caused samples to cool to room temperature in the laboratory over a short period (approximately 2 hours) as opposed to the presumed cooling period after a conflagration (or cooling kiln) which is more likely to be over a period of a few days or even longer. Furthermore, archaeointensity results may be affected by the rate at which the sample is cooling while the TRM is being acquired during the Thellier type experiment (Walton, 1982). This was predicted by Dodson and McClelland-Brown (1980) and shown experimentally by Fox and Aitken (1980) to be 6% to 12% too high. According to Chauvin *et al.*, (2000), for baked clays which contain single domain grains, as is the case in the burnt mud brick samples (Downey, 2011) and probably the magnetic carrier in the sherds in this study, the TRM acquisition after natural slow cooling can be higher by 5% to 12% than that acquired (after rapid laboratory cooling), producing an overestimate of the archaeointensity value.

However, the cooling rate effect is considered significant only if it is higher than the 'alteration factor' otherwise it is impossible to estimate whether the TRM acquisition capacity has changed because of the cooling rate effect or because of mineralogical changes (De Marco *et al.*, 2008). This alteration factor given as a percentage relates to changes in TRM acquired before and after heating in order to investigate if any there had been mineralogical alterations during the slow cooling rate stage. In this study, monitoring of low field susceptibility after each successive heating and cooling step indicated that there were no thermally induced mineralogical changes.

Dodson and McClelland-Brown (1980) showed that for a given group of grains, the effective blocking temperature is lower in the case of slow cooling and therefore the fractional

alignment that is blocked-in, is higher, both because of the reduced thermal agitation at the lower temperature and because of the intrinsic increase of activation energy. According to Fox and Aitken (1980) it would be desirable to use a laboratory cooling time of the same order as the presumed archaeological cooling time.

Anisotropy of magnetic susceptibility (AMS) is a reliable petro-fabric indicator as numerous studies have shown for laboratory and natural materials and there are many factors which control it. De Marco *et al.* (2008) report that the degree of magnetic anisotropy K_{max}/K_{min} may vary greatly from 6.5% to 58% but there is no correspondence of the anisotropy degree with the type of structure or the way it was constructed. It appears to depend on the type of material. For 'baked' clays (burnt mud brick) or pottery sherds as in this study, the anisotropy degree is generally lower than 12% but in some cases, correction factors of only 1.5% need be applied. Morales *et al.* (2006) report that in some TRM acquisitions, no anisotropy corrections are applied since the AMS is less than 1%.

In view of the varied arguments presented above and in the absence of specific cooling rate correction factor or anisotropy measurements for the samples used in this study, a reduction of the archaeointensity value obtained, between 6% and 12% as suggested by Fox and Aitken (1980) (ie) $9\% \pm 3\%$, has been applied. This reduces the archaeointensity value to $49.6 \mu T \pm 1.6 \mu T$. (Table 1).

This mean value for F^A is used in the formula and compared to the 'Geomagnetic Field Variation' plot, (Xanthakis and Liritzis, 1991) showing ($F^A/F^D - 1$) against Age (years), $\{F^D = 0.309 (4 - 3\cos^2L)^{1/2}\}$, F^A is the ancient geomagnetic field, F^D is the axial dipole field and L is the latitude of the site. The F^D value is calculated to be 0.443 Oe. (44.3 μT) for the sites Rhodes and ($F^A/F^D - 1$) = 0.12 ± 0.03 . (Table 1, Fig. 3). The intersection of this value on the Geomagnetic Field Variation plot, shows the probability density of possible dates between ~1000 to ~1500 AD. (which includes the High and Late Middle Ages).

Another, but entirely unlikely possible date (intersection) occurs between ~ 1700 to ~ 1500 BC and a much younger date at ~1750 AD. (Fig. 3). During the Medieval period, the geomagnetic

field intensity change with time was within narrow limits, thus making it difficult to determine an exact date (Liritzis, 1989; Aitken *et al.*, 1989).

The archaeointensity (49.6 μT) result is also compared directly to the SCHA.DI-00 palaeosecular variation curve obtained for Sofia (Bulgaria) (Pavon-Carrasco *et al.* 2008), which is ~ 6 degrees latitude north of Rhodes, where the archaeomagnetic intensity value is considered to be of similar value and gives an age ranging from $\sim 1420 - 1480$ AD.

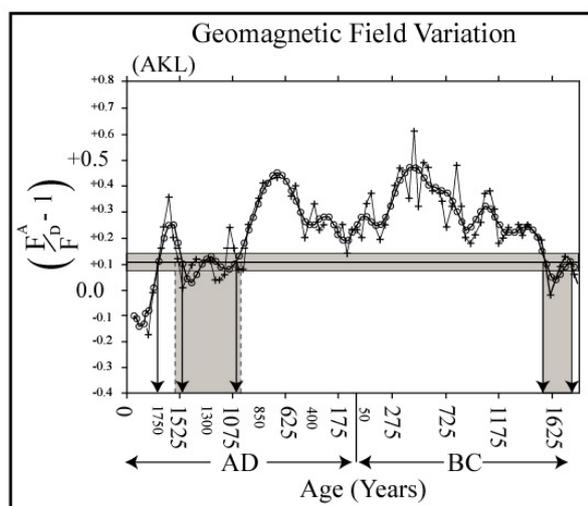


Figure 3. Geomagnetic Field Variation (after Xanthakis and Liritzis, 1991, modified). Where F^A is Geomagnetic Field and F^D is Axial Dipole Field, calculated from: $F^D = 0.309 (4 - 3 \cos^2 L)^{1/2}$: where L is site Latitude (36.4°N). $F^D = 0.443$. Intersection of reference curve shows probability density of possible dates with errors (grey shading).

The SCHA.DI.00 model was developed using the Spherical Cap Harmonic Analysis (SCHA) technique applied to five of the Bayesian Palaeosecular Variation Curves (Gallet *et al.*, 2002; Schnepf and Lanos, 2005; Marton and Ferencz,

2006; Gomez-Paccard *et al.*, 2006 and Zananeri *et al.*, 2007). The date is also consistent with the archaeointensity secular variation curve record obtained for Bulgaria by Kovacheva *et al.*, (1998), while it concurs with the evidence of intensity variations demonstrated earlier from Greek and Bulgarian churches during same period of interest (Liritzis and Kovacheva, 1992). The date is also consistent (~ 1500 AD.) with the most recent archaeomagnetic Bayesian SV curves for Greece (calculated at Athens), De Marco *et al.*, (2008).

CONCLUSION

The single heating modified Thellier method with an orthogonal sample preparation approach, which also reduces shape anisotropy effects, shows that, meaningful archaeointensity results can be obtained. The age range presented here ~ 1000 to ~ 1500 AD, from a limited number of pottery sherds, present in the mortar of the Rhodian Byzantine church walls, appears to be consistent with the incorporation of contemporary pottery (Middle Ages) (ie) during the most recent building phase.

However, older pot- sherds, re-used from previous destruction rubble, may also be present or may even have been imported from another region entirely. Pottery derived from further afield is a possibility but would have to come from some distance where the geomagnetic field intensity was significantly different.

Only preliminary results are presented here and a more comprehensive sampling programme, under progress, may reveal the presence of more ancient pot- sherds, as well as, better defining the ages of the Medieval sherds.

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