

Full vector archaeomagnetic records from Anatolia between 2000 and 1400 BCE: implications for geomagnetic field models and the dating of fires in antiquity

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Abstract

Anatolia, as one of the busiest crossroads of ancient civilizations, provides an ideal platform for archaeomagnetic studies. Previous results from the Middle East have suggested the occurrence of a strong peak in geomagnetic intensity at ~1000 BCE associated with dramatic field strength variations that could require a radical rethinking of geodynamo theory. The behavior of the field in the centuries preceding this peak remains poorly constrained, however. Here we present the results of full-vector archaeomagnetic experiments performed on 18 sets of samples from three archaeological sites belonging to Assyrian Trade Colony and Hittite periods. Associated rock magnetic analyses showed that the major magnetic carrier is magnetite stable up to 700°C and the magnetic mineral assemblage is composed mostly of non-interacting PSD grains.

The directional results are compared with existing data and with the most recent global geomagnetic field models pfm9k.1b and SHA.DIF.14k. The directions are in remarkably good agreement with SHA.DIF.14k which is based on archaeomagnetic and lava flow data. Together with our earlier results from Anatolia, we triple the existing database of directions for the 700 year long period 2250-1550 BCE, over a large region from Greece to Azerbaijan, and from Moldavia/Ukraine to Egypt.

23 Three archaeointensity methods: thermal IZZI-Thellier, microwave Thellier and the multi-
24 specimen protocol (MSP) produced virtual axial dipole moment estimates ($9.2\text{--}11.1 \times$
25 10^{22}Am^2) that are somewhat higher than contemporaneous (regional and global) data and
26 model predictions suggesting that the field was already substantially stronger than today
27 more than 800 years prior to the reported peak. In addition to constraining geomagnetic
28 variability, our data also allows us to assign relative dates to inferred fire events in the
29 Assyrian Trade Colony Period sites. This allows us to conclude that the fire events at the
30 largest site, Kültepe, were not all contemporaneous with one another and with the
31 abandonment of the site as has been previously hypothesized.

32

33 **Keywords:** Archaeomagnetism, archaeointensity, palaeosecular variation, Hittite, Assyrian,

34 **Anatolia, Turkey**

35 1. Introduction

36 Over the past decade, evidence for a short-lived period of very high geomagnetic
37 intensities in the Levant rapidly accumulated (Ben-Yosef et al., 2008a; Ben-Yosef et al., 2009;
38 Ben-Yosef et al., 2008b; Gallet and al-Maqdissi, 2010; Gallet and Butterlin, 2014; Gallet et al.,
39 2014; Gallet et al., 2003; Gallet et al., 2006; Gallet and Le Goff, 2006; Gallet et al., 2008;
40 Genevey et al., 2003; Shaar et al., 2011). At least three studies present reliable
41 paleointensities that exceed twice the current field strength in this region ~1050-850 BCE.
42 The occurrence of the palaeointensity high, or '*archaeomagnetic jerk*' (Gallet et al., 2003)
43 sparked considerable debate: such geomagnetic features are not captured by even the most
44 recent geomagnetic models describing changes in the field (Nilsson et al., 2014; Pavón-
45 Carrasco et al., 2014). Moreover, it was recently shown that current geodynamo theory
46 cannot sustain the existence of this phenomenon (Livermore et al., 2014).

47 Most of the available data for this region is derived from archaeological artefacts, such as
48 shards, copper slag or fired mud-bricks. Their rock magnetic properties are generally
49 favourable, but such samples are often found un-oriented, so they do not provide
50 constraints on directional variations. Only in-situ materials like kilns or burnt mud-brick walls
51 provide the opportunity to obtain reliable palaeodirections; studies reporting these or full
52 vector descriptions of the field are scarce (e.g.: Bucha and Mellaart (1967); Saribudak and
53 Tarling (1993); Ertepinar et al., (2012)). Therefore, directional records for the Levant are
54 supported by less data than the palaeointensity curve for the past millennia – only 30% of
55 the data in GEOMAGIA50 includes directions. Directional data is particularly scarce ~2250-
56 1550 BCE, the Assyrian and Hittite periods in the Levant.

To further constrain the occurrence of the high palaeointensities -and to possibly elucidate their driving force- a full vector record of the geomagnetic field in this area for the past 5 millennia is indispensable. Here, we look at Anatolia as one of the busiest crossroads of ancient civilizations, an ideal platform for archaeomagnetic studies. Here, we present new data from two Assyrian and one Hittite period site. Our new data triples the available directional information for this particular time interval. Our palaeointensities are obtained by three different and independent methods: thermal IZZI-Thellier experiments, the Microwave Thellier technique, and the Multi-specimen protocol. The credibility of our findings is greatly enhanced if the results of (two of) these methods agree. We compare our results to the latest compilations and models of the field, pfm9k.1b (Nilsson et al., 2014) based on both sediment and igneous/archaeomagnetic data, and SHA.DIF.14k (Pavón-Carrasco et al., 2014) based on archaeomagnetic and lava flow data alone.

During our field campaigns we also sampled a number of (sub-)sites within Kültepe. The timing and character of the demise of this settlement has puzzled archaeologists for years. By comparing palaeodirections from different parts of the settlement to each-other and to the regional record in directions, we conclude that this settlement was destroyed (burned) not at once, but in various stages.

2. The Bronze Age in Anatolia

In the early second millennium BCE, Anatolia was in the form of city-states where Assyrian merchants came to trade textiles and metals. These merchants sometimes resided in Anatolia which gave the era its name: Assyrian Trade Colony Period. After the trading relations had started, a number of trading centers called *Karum* were established in the major cities of the time. This is also contemporaneous with the earliest writing to appear -

80 inscribed on clay cuneiform tablets. There are more than 20.000 tablets found in Kültepe,
81 ‘the trade capital of the period’, dating between 1970-1740 BCE. The richness of cultural
82 findings and extensive dendrochronology studies allowed the archaeologists to have a well
83 defined age constraint on the site. This unique combination made Kültepe the reference site
84 for dating the other archaeological sites. The Assyrian Trade Colony Period ended at ~1650
85 BCE with the emergence of Hittites in Anatolia (Fig. 1).

86 In ~1700 BCE, people of unknown origin migrated to Anatolia and united the city states
87 under one central authority, laying the foundations of the Hittite empire centered at
88 Hattusa, which is now a UNESCO World Heritage site. The domination of the Hittites lasted
89 for almost a thousand years and the empire reached its height in the 14th century BCE
90 controlling a large part of Anatolia, the northern Levant and Upper Mesopotamia. The reign
91 started as a kingdom (Old Kingdom, ca. 1650-1500 BCE), then became an Empire between
92 1400-1200 BCE. After 1180 BCE, the empire disintegrated into several independent city
93 states called Neo-Hittites and completely vanished ~8th century BCE. The historical
94 documentations of Hittites show a remarkable political and military power as well as a very
95 rich and long lasting culture (Sagona and Zimansky, 2009). The sampling is carried out on
96 three archaeological sites two of which are from the Assyrian Period (Kültepe and
97 Kalehöyük) and the third (Şapinuva) is from a Hittite Period site. A description of each
98 settlement is given in the appendix.

99 **3. Rock magnetic analyses and results**

100 Room temperature bulk magnetic susceptibilities and thermomagnetic curves were
101 determined for the identification of the magnetic carriers and thermal stability. Based on the
102 preliminary results from these experiments and the quality of the directional results, 9 sites

appeared to be suitable for archaeointensity measurements. For these, we additionally performed hysteresis loop, Isothermal Remanent Magnetization (IRM) acquisition and First Order Reversal Curve (FORC) diagram experiments (Roberts et al., 2000).

Low field bulk magnetic susceptibility. Samples were measured with a Kappabridge KLY-2. The results are homogeneous among different rock types and the values range 0.05-35.0*10⁻³SI. The results are used to calculate the Koenigsberger Ratio (Q_n) which is an appropriate measure to distinguish whether the samples carry a complete thermoremanent magnetization (TRM). All materials other than the majority of KA granites have Q_n value greater than 1 indicating that TRM strongly dominates, providing a positive stability test (Fig. 2a).

Thermomagnetic curves (Curie balance). Measurements were done using a modified horizontal translation type Curie balance that uses a cycling rather than a steady magnetizing field (Mullender et al., 1993). Field settings varied from 50-300mT to 270-300mT. Heating and cooling rates were 10°C/min and experiments were done in air. For all types of materials other than the KT ignimbrites, the heating and cooling curves are essentially reversible indicating that the magnetic minerals are stable until 700°C (Fig. 2b-g). The mud-brick samples from KA, KT and the granite (KA) and ignimbrite (KT) samples have a single Curie temperature (T_c) at ~580°C which is characteristic for magnetite (Fig. 2b-d). The mud-bricks of SPN also have a T_c at ~580°C, again pointing to the presence of magnetite as the main carrier, but there is an extra inflection point at ~350°C, which could point to some maghemite, or possibly titanomagnetite or Al-substituted magnetite (Dunlop and Özdemir, 1997) (Fig. 2e). The vitrified mud-bricks from KT12 exhibit an almost reversible curve which shows mainly paramagnetic contribution preventing the identification of any magnetic carrier (Fig. 2f). The curves from some ignimbrites of KT show a difference between heating

and cooling curves resulting in irreversible loss in magnetization up to 80% indicating major alteration (Fig. 2g).

Hysteresis loops and FORC diagrams. For the sites that looked promising for archaeointensity measurements, additional rock magnetic properties such as hysteresis loops, IRM acquisition curves and FORC diagrams were investigated, to assess domain state and magnetic stability. From each set 3-5 samples were measured with an alternating gradient force magnetometer (AGFM). After correction of the paramagnetic and diamagnetic contributions on the hysteresis loops (Fig. 3a) we derived the hysteresis loop parameters (H_c , M_s) while from the IRM acquisition curves (Fig. 3b) we derived the remanence parameters (H_{cr} , M_{sr}). From their ratio's we constructed a Day Plot (Day et al., 1977) to analyse the domain state of the samples. The results of all 29 measurements show that the samples contain only pseudo single domain (PSD) grains (Fig. 3c, Table A1). There is no indication of a high coercivity mineral since all the samples are saturated at or below 200 mT (Fig. 3b).

A FORC diagram is also useful to assess the domain state of magnetic minerals. It additionally gives information about the local interaction fields for an assemblage of magnetic particles (Roberts et al., 2000). Three diagrams from each type of building material are shown in figure 3d. The mud-bricks from KA have a symmetrical FORC diagram with a peak distribution centered close to the origin, showing a B_c slightly lower than derived from the hysteresis analysis, with a minor spread along the B_u axis which suggests the presence of small MD or PSD grains with minimal magnetostatic interaction. The FORC distribution of the mud-bricks and vitrified mud-bricks from KT have one closed inner contour with peak at $B_c=10\text{mT}$ and $B_c=20\text{mT}$, respectively, consistent with those determined from the

corresponding hysteresis loops. Both diagrams have a very narrow contour spreading along the ordinate indicating the assemblages are dominated by non-interacting PSD grains.

Based on the rock magnetic measurements, we decided that all selected sets are suitable for archaeointensity experiments.

4. Methods

To determine the characteristic remanent magnetization direction (ChRM) at least 8 specimens per site were demagnetised (Table 2a) thermally (TH) or with alternating field (AF). The demagnetization was performed with small AF or TH increments (at least 15 steps) up to a maximum of 100mT or 580°C. AF demagnetization is carried out after heating the samples to 150°C to remove possible high coercivity and low T_c minerals, or to remove possible stress in magnetite grains at low temperatures. The demagnetization results were interpreted via orthogonal projection diagrams (Zijderveld, 1967) using an eigenvector approach (Kirschvink, 1980), the mean directions of ChRMs were calculated according to Fisher (1953). The acceptance criteria for maximum angular deviation (MAD) of individual directions and the α_{95} of the means are taken as 10°, but values are typically much lower than that. Figure 4 and 5 show convincing examples of demagnetization diagrams for each type of material and the ChRM directions of each set.

For the paleointensity measurements, we adopted three protocols. We emphasized the TT experiments, together with a fair number of MW experiments. In addition, if specimens were still available, we added a small number of multi-specimen experiments (Dekkers and Böhnelt, 2006) corrected for domain state according to Fabian and Leonhardt (2010). Figure 6a shows representative examples of a successful and a failed measurement from each type of experiment. These three methods were also applied to a large set of volcanics from

Hawaii by De Groot et al. (2013) who concluded that the results were remarkably accurate if the results of two or more methods mutually agreed, testifying to the importance to not adhere to just one protocol.

4.1. Thermal IZZI-Thellier experiments (TT)

The experiments were performed using a laboratory field of 50-60 μ T and a temperature range of 20-530°C. The IZZI protocol (Tauxe and Staudigel, 2004) was used with field applied parallel to the NRM of the sample which enables the detection of multi-domain behaviour and benefits from the advantages of providing the opportunity to check the consistency of IZ and ZI steps and rendering an extra pTRM tail check unnecessary (Yu and Tauxe, 2005).

A custom built orientation tray was used to align each sample's NRM with the applied field direction, reducing the effects of anisotropy during TRM acquisition (Rogers et al., 1979). The results were interpreted using the NRM-TRM plots. The acceptance criteria, adopted from Coe (1978) and supplemented by those of Selkin and Tauxe (2000), are as follows:

1. For the linear fit:

- the number of points used for the best fit line (N) ≥ 5 ;

- the ratio of standard error of the slope to absolute value of the slope (β) < 0.1 ;

2. The NRM fraction (f) ≥ 0.4 , with an exception on specimen KT8_10 with $f=0.35$ where there is a sister specimen with $f>0.5$ and no evidence of curvature. We could not achieve $f\geq 0.7$ as recommended by Biggin (2010) because of thermo-chemical alteration occurring at higher temperatures, however, in ~80% of the measurements $f>0.5$ as suggested by Biggin and Thomas (2003);

3. Quality factor (q) > 5 , where most results are higher than 10;

196 4. For the pTRM checks:

197 - number of successful pTRM checks ≥ 3 ;

198 - the ratio of difference between the pTRM check and relevant TRM value to the
199 length of the selected NRM-TRM segment (DRAT) $< 10\%$.

200 In addition, the directional aspects such as the MAD and α were analysed by principle
201 component analysis and the upper limits are set to 10%.

202 *4.2. Microwave experiments (MW)*

203 The experiments on the mud-brick samples were performed using the IZZI protocol
204 (Tauxe and Staudigel, 2004) and a laboratory field ranging from 35-100 μT , applied at least 45
205 degrees from the NRM direction. Possible influence of anisotropy was checked for by
206 comparing the direction of the magnetization acquired with that of the applied field. In all
207 cases, no significant systematic offsets were observed suggesting that anisotropy was
208 negligible. For three specimens from the shards IZIZ protocol was used with laboratory field
209 parallel to the samples NRM (Aitken et al., 1988; Walton, 1979). In both protocols, to check
210 for possible influence of thermo-chemical alteration, pTRM checks were performed after
211 every two double-treatments. The same selection criteria were employed as in the TT
212 experiments.

213 *4.3. Multi-Specimen Method (MSP) corrected for domain state (DSC)*

214 To reduce the effect of non-ideal MD behavior and progressive alteration during TT
215 experiments, Dekkers and Böhnell (2006) proposed a method, the 'multi-specimen parallel
216 differential pTRM method', here referred to as MSP-DB. The idea behind the method is
217 simple: to overprint an ancient TRM with a laboratory pTRM induced at a temperature much
218 lower than the Curie temperature in a laboratory field applied in the same direction as the

219 TRM. The initial suggestion that this protocol was domain-state independent, however, did
220 not hold; Fabian and Leonhardt (2010) proposed an addition to the protocol to correct for
221 MD behavior. As a rule, we apply the domain-state corrected protocol, referred to as MSP-
222 DSC.

223 To conduct the MSP-DB and MSP-DSC measurements we used four specimens per site,
224 simply because there were insufficient specimens. For the MSP experiments, it is first
225 necessary to check for the absence of secondary magnetizations, and then to select a set-
226 temperature for the pTRM acquisition that is below the point where chemical alteration is
227 significant. To determine this temperature we relied on the a priori knowledge from the rock
228 magnetic experiments and the thermal demagnetization. The experiments were conducted
229 using thermal demagnetiser. To induce the pTRM parallel to the NRM, we used a specially
230 designed sample holder. Because of the limited amount of specimens we applied 4 steps.
231 The samples were heated at either 300 or 350°C. The MSP experiments were accepted if the
232 average progressive alteration, $\mathcal{E}_{alt} < 3\%$. When $\mathcal{E}_{alt} > 3\%$, the data point with the highest
233 alteration is omitted from the group. If the average alteration after the omission is less than
234 3%, the new best fit and its error envelopes is calculated based on three data points. For the
235 MSP-DCS protocol there is an additional requirement where, Δb , the difference between the
236 theoretical ($b=-1$) and the actual value of y-axis intercept of the best-fit line should be
237 smaller than 10%. If this requirement was not fulfilled, implying that the MSP-DSC protocol
238 did not properly correct for MD behaviour, we used the MSP-DB protocol provided that the
239 \mathcal{E}_{alt} is still less than 3%.

5. Results

5.1. ChRM Directions

Şapınuva. The samples that are collected from the fallen mud-bricks (SPN1) show single component magnetite magnetizations (Fig. 4a). There is a slight inflexion in the thermal decay curve at ~350°C. This observation is coherent with what was found in the Curie curves (Fig. 2) and could imply that there could be maghemite. Out of 14 specimens, 5 samples (two being sister samples) gave inconsistent directions, indicating that the mud-brick blocks from which they were sampled were displaced after burning. When these 5 samples are discarded, the distribution becomes clustered with $k > 100$ (Fig. 5, Table 2a).

Kalehöyük. The samples from KA produced good results from its mud-bricks (Fig. 4b, c) and less conclusive or no result from the granites (Fig. 4d). Clearly, the granites have not been fully heated. The set from KA1 is composed solely of mud-bricks. The demagnetization diagrams are single component with a minor overprint removed at low temperatures (Fig. 4b). The remanence is nearly completely removed at 580°C but not yet at 100 mT, which could indicate the presence of some maghemite. In the AF demagnetization diagrams, the percentage of remanence that is left after 100 mT is ~20% (Fig. 4c). From 8 cores measured, 7 gave successful results producing a well-defined ChRM with high k -value of 1066.

Sampling of KA2 was made on four blocks of granite and one block of mud-brick. Out of 20 samples measured, 12 belonging to two different granite blocks produced single component magnetization diagrams with random -likely original- directions indicating that they were not burnt at sufficiently high temperatures. The samples from the other two granite blocks have a low-temperature (LT) due to partial heating (Fig. 4d) up to some 350°C and a high-temperature (HT) randomly directed component, whereas the mud-brick samples

are single component. The ChRM analysis of these three blocks, where mud-bricks are in agreement with the LT component of granites, yields a cluster with $k > 100$.

The samples collected from KA3 have single component demagnetization diagrams with random groups of directions indicating that the granite blocks carry their original magnetizations and hence were not sufficiently burnt (Fig. 5). This is in line with the results from the calculation of Q_n where the majority of the granites have $Q_n < 1$ (Fig. 2a).

The set from KA4 is also composed of only granites collected from two different blocks from the foundation of a mud-brick wall where KA1 was taken. The demagnetization diagrams are generally single component but of low quality. In some samples, there is a slight inflexion in the decay curve $\sim 500^\circ\text{C}$ which can be interpreted as a second magnetic mineral. The fact that there is an inflexion in the decay curve at that temperature, but no obvious bending in the Curie curves suggests that the reason is insufficient burning rather than a second magnetic mineral (Fig. 2, 4). The ChRM of the set displays a poor cluster with $k = 64$, $\alpha_{95} = 6.1$ and there is significant disagreement between the directions obtained from KA1 and KA4. Considering the scattered distribution of KA4, the granite blocks may have slightly moved (Fig. A3) while the mud-brick wall (KA1) is more solid and better burnt. Therefore, the results from KA1 are considered to be more reliable and directions from KA4 are discarded from further analyses (Fig. 5, Table 2a).

Kültepe. The samples collected from Kültepe generally produced good results, especially from the mud-bricks. The sets that are composed solely of mud-brick (KT1, KT2 and KT3) show single component magnetite magnetization with a minor overprint that is completely removed at low temperatures (Fig. 4e). This is supported with the findings from the Curie curves where the mud-bricks display an ideal magnetite magnetization and the uniform

286 thermal decay (Fig. 2, 4e). These three sets have well-defined ChRMs with high k values
287 (200-600) and $\alpha_{95}<1.7$.

288 Out of 5 sets that are composed only of ignimbrites, 4 sets (KT6, KT7, KT10 and KT11)
289 have turned out to be not sufficiently burnt considering the single component
290 demagnetization diagrams with random directions (Fig. 5). The samples from the last
291 ignimbrite set, KT9, were either fully burnt providing a meaningful direction or sufficiently
292 heated to have a clear well-determined LT component in the demagnetization diagram that
293 we consider to represent a ChRM due to firing. This set is also of good quality with $k>300$,
294 $\alpha_{95}<2.5$.

295 There are 4 sets (KT4, KT5, KT8 and KT13) that are composed of both mud-bricks and
296 ignimbrites. The ignimbrite samples from these sets have either single or two component
297 demagnetization diagrams (Fig. 4f) whereas the mud-bricks are single component. The
298 directions obtained from these two different building materials (the LT component of
299 ignimbrites) are consistent within each set. Only 2-3 samples in each set were clear outliers
300 (ignimbrites, obviously not sufficiently heated) and therefore excluded.

301 There is one set that is composed fully of vitrified mudbricks (KT12). The
302 demagnetization diagrams are single component decaying uniformly straight to the origin
303 (Fig. 4g). The Curie curves represent an almost purely paramagnetic contribution (Fig. 2) and
304 did not allow identification of the magnetic carrier. The demagnetization diagrams, however,
305 show that the magnetization is fully removed at $\sim 500^{\circ}\text{C}$ pointing to Ti-poor magnetite. The
306 set displays a well-defined ChRM with $k=244$, $\alpha_{95}=1.3$ (Fig. 5, Table 2a).

307 Out of the 13 sets of samples from Kültepe, 9 are considered to be of good quality with
308 IGRF corrected declinations between 348.7° - 5.0° and inclinations between 41.4° - 56.0° .

5.2. *Archaeointensity results*

Archaeointensities were determined for 9 sets of samples (1 set of mud-bricks from KA, 1 set of vitrified mud-bricks from KT and 7 sets of mud-bricks from KT) where 5 were successful to yield a result in all three methods. Figure 6a shows an example of a successful and a failed measurement from each method. The plots of all measurements are presented in figures A5 and A6, results are reported in table 2b and detailed statistical parameters are given in table A2 and A3. For TT and MW measurements, no MD-type behaviour is observed in the NRM-TRM plots (except for one specimen from KT4) supporting the general findings from the rock magnetic analyses. The results from different protocols reasonably agree with each other, yet, except for KT8, the MSP results are systematically lower than the other two protocols (Table 2a, Fig. 6b). This discrepancy is the highest in KT3 (up to 30% with the MW). Out of 54 TT and MW measurements, 47 are appointed to be successful (Table A2). From 8 sets of MSP measurements, 7 were successful either with DSC or DB solution. No systematic differences were observed between the TT and MW results from the same sample sets. Since the cooling rate effect, if present, is expected to be enhanced in MW estimates and make them systematically higher than sister estimates using longer cooling times (Poletti et al., 2013), this agreement suggests that no cooling rate correction is required for the data as a whole.

The set from the mud-bricks of KA1 has a mean intensity value of 58.5 μ T from 3 TT (out of 3) and 1 MW (out of 2) measurements. The single successful MW measurement is in excellent agreement with the TT measurements where the result differs by 1.4 μ T from the TT average. The MSP results were rejected due to alteration.

From KT1, we made one TT measurement which has failed and one MW measurement with intensity value of 60.9 μ T in which the MSP-DB result (58.2 μ T) obtained from four data points is in line with the value within 5%.

The samples from KT2 and KT4 produced good quality TT results, however, failed in all MW experiments either due to noisy NRM-TRM plot, indestructible NRM or MD-curvature (KT2_3, KT2_4 and KT4_6, respectively, in figure A5). We were not able to perform the MSP method on KT2 because there were not enough samples, so we present an intensity value of 54.8 μ T for the set, based on two TT measurements. The MSP result from KT4 is of good quality with minor alteration and $\Delta b < 10\%$ allowing to opt for the domain corrected solution. The set has a mean intensity value of 54.7 \pm 4.3 μ T based on 5 TT and an MSP-DSC result derived from four points.

The entire TT (22 out of 22) and the majority of the MW measurements (13 out of 15) from the sets KT3 (mud-bricks and shards), KT5, KT8, KT13 (mud-bricks) and KT12 (vitrified mud-bricks) and have passed the selection criteria, producing high quality NRM-TRM plots. The TT and MW measurements from the mud-bricks of KT3 produced comparable results whereas the MSP result is approximately 30% lower than the average of these two methods. One specimen from the MW measurements from the set yielded a value that is too high to fit the population. Therefore, even though measurement meets the acceptance criteria it is considered to be an outlier and rejected from further analyses. The samples from KT5 produced two low and two high TT results in which the lower values are in line with the MSP-DSC result and the higher values are in agreement with the MW. The set has a mean intensity value of 51.5 \pm 7.2 μ T. The set KT8, among all the sets, has the most consistent results in both individual sample level and mean intensities obtained from three protocols. For the MSP measurement, although the average alteration is slightly higher than the

acceptance limit ($\mathcal{E}_{\text{alt}}=3.06\%$), we included the result for further analyses since the data points are perfectly linear and the result is in excellent agreement with the other two protocols. 7 TT and 3 MW measurements, and a MSP-DB solution from three data points produced an average intensity of $54.8\pm2.0\mu\text{T}$. The measurements from the vitrified mud-bricks of KT12 produced the highest intensity value with $62.3\pm4.8\mu\text{T}$ from 6 measurements (4 TT, 1 MW and 4 data points from MSP-DB). The set has exceptionally high f value ($f_{\text{ave}}=0.84$) compared to other sets (Table A2). The samples from KT13 produced well behaved NRM-TRM diagrams from TT, acceptable results from the MW method. The set has a mean intensity value of $53.9\pm5.1\mu\text{T}$ obtained from 3 TT, 2 MW and a MSP-DSC result.

6. Discussion

6.1. *ChRM directions*

To be able to compare our results with the existing Eastern Europe and Near & Middle East data from GEOMAGIA50 and the Turkish data (Ertepinar et al., 2012; Saribudak and Tarling, 1993; Sayın and Orbay, 2003), they were relocated to Kayseri. Then, all the data points are plotted against the existing data from GEOMAGIA50 and geomagnetic field models calculated at Kayseri (Fig. 7a, b). We use the latest models SHA.DIF.14k (Pavón-Carrasco et al., 2014) and pfm9k.1b (Nilsson et al., 2014); both models provide error envelopes. The pfm9k.1b model uses also sediment data, and is appreciably more smoothed and has a larger error envelope than SHA.DIF.14k (Figs. 7, 8).

A first observation is that the new model SHA.DIF.14k very well fits our earlier directional observations, including the large declination swing to nearly 20°E around 2000 BCE (Fig. 7a). This swing was not recorded by the CALS7k model (Korte and Constable, 2005) we then used. Nor is it recorded by the heavily smoothed pfm9k.1b model. Also all other directional

results from this earlier study fit better with the new model, for example the inclination values around 2500 BCE (Fig. 7b). Only the paleointensities (VADM) show a less perfect fit, and both our earlier data and the compiled Middle East data (Fig. 7c) are still higher than both models predict around 2600-2500 BCE.

With respect to our new data, the prediction of declination from the models agrees within error with the declination of the single direction from ~1350 BCE (**SPN1**) while the inclination value is lower (by more than 10°) than predicted. At this time interval SHA.DIF.14k shows a maximum in the inclination as high as 68°. The records from Greece (Tarling and Downey, 1990) and Turkey (Sayın and Orbay, 2003) around this period are on average 5° higher than our result. Because of the large error bar of **SPN1**, the result still falls within the range predicted by pfm9k.1b.

There are two directions from ~1775 BCE, **KT1** and **KA1**, both sites are reported to come from the same level (Kültepe Ib,) with a very well constrained age, both have high k values (300, 1066) and low α_{95} (1.7°, 1.8°). The prediction of SHA.DIF.14k is in perfect agreement with directions from **KT1**. The direction of **KA1** from the allegedly time equivalent level, however, does not fit within error with SHA.DIF.14, especially the inclination is 10° lower than predicted, while only the declination falls within the error envelope of pfm9k.1b. In this interval the Turkish data (Sayın and Orbay, 2003) show a large swing in declination, up to 25° to the east compared to the models. This shallow inclination of **KA1** can be explained by either an overprint of another fire event occurring in a later stage of the settlement, or a dip in inclination for the time period. This result is discussed in more detail in section 6.3.

The data sets from 1875±45 BCE (**KA2**, **KT2**, **KT3**, **KT4**, **KT5**, **KT8**, **KT9**, **KT13**) show a wide range of declination (344.0°-3.9°) and inclination (41.4°-57.0°) values, in a short time interval of ~90 years. The declination results are mostly consistent with pfm9k.1b, but with respect

to SHA.DIF.14k most declinations are significantly (0-12°) to the west of the prediction. The inclinations are partly within range and partly shallower (up to 9-15°) compared to the models, but consistent with the GEOMAGIA50 data from Greece, which admittedly are very few. Naturally, it is inevitable that the models are heavily smoothed and cannot adequately represent such rapid variations, but it is noteworthy to note that around 1875 BCE, SHA.DIF.14k shows a significant dip in inclination. The relative ages of these data points are discussed in more detail below.

The oldest site from Kültepe (**KT12**, ~2250 BCE) produced a high quality result but is poorly constrained in age. The directions fit well with both models, but the paleointensity does not (discussed below).

These new results -certainly if we can constrain them better in age- are very useful to improve the resolution of the models since there is lack of data for this time periods. Only 8 records are available in GEOMAGIA50 for the 700 year long period 1550-2250 BCE, from Greece to Azerbaijan, and from Moldavia/Ukraine to Egypt. Our 12 new directional records in this time interval plus the 3 results from Ertepinar et al. (2012) almost triple the database for this entire region. In addition, these high quality data sets contribute in terms of a better spatial distribution. This will reduce any bias (the local variations in the field) introduced by the few existing data sets, considering that the majority of the GEOMAGIA50 data is coming from Eastern Europe, some from the Near East (22%) and very few from the Middle East (only 2%).

6.2. Archaeointensities

All the archaeointensity values were converted into virtual axial dipole moments (VADM) and plotted along with the Middle East data (see introduction for references related to

Levant), Turkish data (Ertepinar et al., 2012), the new results from Tell Atchana (Hammond et al., 2015) and the global field models SHA.DIF.14k and pfm9k.1b (Fig. 7c). A summary of the palaeointensity results are in Table 2b, while all details can be found in Tables A2, A3.

The data sets from ~1775 BCE, **KA1** and **KT1** produced similar intensity values, higher than the prediction of both models and the majority of the Middle East data. Also the new results of Hammond et al. (2015) are in line with the generally lower intensities found in the Middle East and the models. The fact that at least two different methods involved in the acquisition of the results (TT+MW for **KA1** and MW+MSP for **KT1**) produce the same intensity however, gives faith that these higher intensities are reliable. There are several very similar intensities in this same period from Ben-Yosef et al. (2008a) and from GEOMAGIA50. Hence there seems to be short period of high intensities ~1775 BCE. Additional measurements from these levels would increase the reliability of the data points.

The intensity values of data sets from 1875±45 BCE (**KT2**, **KT3**, **KT4**, **KT5**, **KT8**, **KT13**) show a dispersion of <6%, the highest being $9.59 \cdot 10^{22}$, and the lowest $9.00 \cdot 10^{22}$. These palaeointensities are higher than the prediction of SHA.DIF.14k, GEOMAGIA50, Middle East and Hammond et al. (2015) data, although the Tell Atchana result at 1875 BCE is in line with our results.

The archaeointensity result ($10.89 \pm 0.84 \cdot 10^{22} \text{Am}^2$) from the data set from ~2250 BCE (**KT12**) is also significantly higher than what is predicted by the models and higher than the GEOMAGIA50 and the Middle East data. Considering the large age error, however, this high intensity could fit very well with the high intensity interval (2600-2450 BCE) found in both the Middle East and Ertepinar et al. (2012) data.

Our data from the period 2600-1750 BCE (including those of our earlier study) are always significantly higher than predicted by the SHA.DIF.14k model based on archaeomagnetic and

lava flow data, and generally higher than predicted by pfm9k.1b (Fig. 7c). Again, our new data point to the existence of short-lived periods with high intensities, as observed earlier in the Levant.

6.3. Relative chronology of fire events in the Assyrian Trade Colony

Period sites

Common true mean direction (CTMD) test developed by McFadden and McElhinny (1990) is applied to assess whether the fire events in Kültepe is a single big catastrophic event. The test is performed with Monte Carlo simulation for effectively applying the V_w statistic test (Watson, 1983). The angle (γ) between the means, and γ_c , the critical angle in the test is determined. If $\gamma < \gamma_c$ the test is positive and the distributions share a CTMD. The test is classified as A, B, C or indeterminate, depending on the value of γ_c . The sets KT4 & KT8 and KT5 & KT13 share a CTMD with classification A ($\gamma_c < 5^\circ$). The rest of the correlations are negative. Furthermore, the sites from Kalehöyük are also examined for their CTMD to compare if any of the fires in this settlement is contemporaneous with any of the fire events in Kültepe. The CTMD test of the site KA2 produced class B correlation with KT9 whereas KA1 and KT5 & KT13 share a CTMD with classification A. This latter result introduces a conflict since KT5 and KT13 are from Kültepe-level II group, and the age of KA1 is supposed to be time equivalent of Kültepe-level Ib. This disagreement is discussed in more detail in the following parts. Based on the results of the CTMD test of Kültepe-level II, the areal distribution of fires is plotted in figure 8a. As can be seen from the figure, KT2, KT3 and KT9 are local fires whereas KT4 & KT8 and KT5 & KT13 are larger scale fires. Therefore, we can conclude that the timing of fires in Kültepe are different and the site was not abandoned as

a result of a big catastrophic fire event as was also suggested by Sagona and Zimansky (2009).

To establish a relative chronology for the fire events in Kültepe-level II (KT2, KT3, KT4, KT5, KT8, KT9, KT13 from Kültepe and KA2 from Kalehöyük), we sorted the data based on their CTMD results and then, on easternmost to westernmost declination. This best reflects the trend in the SHA.DIF.14k model at this time interval (Fig. 8b). In this scenario, the oldest/youngest age -within the age errors- is assigned to the most westerly/easterly declination while the time span between each fire event is arbitrarily divided into equal time intervals of 10 years. The corresponding inclination values fairly agree with the trend of the model but in this scenario declinations are more westerly ~1850 BCE while inclinations are steeper ~1900 BCE. It seems that the model has not (yet) enough resolution to predict these larger swings in directions. These swings are however fully compatible with observations of secular variation over the past 3000 yr. In cooperation with our earlier data and the data points from Kültepe-level Ib the relative position of KA1 is also clarified where all three components are aligned on a reasonably smooth path. Therefore, since the CTMD test is conducted only including the sites from Kültepe-level II and based only on directions, the test result does not represent the whole picture, and the assigned age for the site should be accurate.

The VADM values are essentially in accordance with the trend of the curve from the SHA.DIF.14k model but systematically higher. Since the VADM values are similar, they cannot be used to put more constraints on the order of fires. The scenario presented here fits with a logical possible sequence of fire events at Kültepe, and the magnitude of geomagnetic field changes are similar to secular variation as observed today and fit within the given age limits. We do realise however that other scenarios are possible, and that the

time constraints within the given age uncertainty do not allow this or any other particular scenario to be robust. For example, in a scenario where the data sets are sorted on increasing inclinations based on the mild increasing trend in the model, results in more abrupt changes compared to the first scenario, and the declinations display erratic jumps of 5-15° within 10 year time intervals.

If we are to compare different scenarios, we favour the scenario where both declination and inclination change gradually and the abrupt and erratic changes in the directions in the other scenarios in such a short time interval are unlikely to occur. Our confidence in this preference has increased when gufm1 model (Jackson et al., 2000) is examined, which is constructed for the time interval of 1590-1990 CE, using the measurements from old ship logs, survey data and observatories. This model, although being extremely young compared to our data points, has a very high temporal resolution that can detect changes in time scale of years. Therefore, it sets an example how fast the directions can change in short periods of time.

7. Conclusions

This study concentrated on the characterization of the full vector magnetic field over Anatolia for Assyrian and Hittite periods. The rock magnetic properties are checked using the room temperature susceptibilities and Curie curves for the directional analyses and additional hysteresis parameters, IRM acquisition and FORC diagrams for the intensity experiments. The samples are found to be suitable for archaeomagnetic experiments.

The ChRMs obtained (12 out of 18) gave good quality results with $k > 100$ and $\alpha_{95} < 5$ (Table 2a). The remaining 6 sets are either displaced or not sufficiently burnt. Together with our

earlier results, we triple the amount of directional results in the period 2250-1550 BCE for the entire region.

The archaeointensity experiments were carried out on 9 sets of samples using three different methods: thermal IZZI-Thellier, microwave, and the multi-specimen technique and they produced comparable results (Table 2b). Yet, the majority of the MSP results are systematically lower than the other two protocols except in KT8. Out of these 9 sets, 5 were successful to yield a result in all three methods.

The results are compared with the existing data from the region and with the global geomagnetic field models pfm9k.1b and SHA.DIF.14k. It appears that pfm9k.1b is over smoothed and has a large error envelope that accommodates most of the data presented here, with the exception of some of the palaeointensity results. The SHA.DIF.14k model is remarkably consistent with the directional data of this and our earlier study. The palaeointensities we find however are invariably higher than the predictions of this model.

Finally, we assess the relative order of fire events in Kültepe with the help of the field models and the CTMD test and conclude that the timing of fire events are different and the abandonment of the site was not result of a catastrophic fire event.

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Figure Captions

Figure 1. Map showing the sampling locations (red circles) and the boundaries of Hittite and Assyrian Kingdoms. Green circles are the previously published data from Anatolia (Ertepinar et al., 2012). White circles refer to the locations of data points from GEOMAGIA50v2 (Donadini et al., 2006; Donadini et al., 2009; Korhonen et al., 2008) within a circle of ~1600 km from Kültepe (Kayseri province, 38.85°N and 35.63°E), the approximate center of Turkey – which is used as the reference point.

Figure 2. (a) The Koenigsberger ratio (Q_n) of remanent versus induced magnetization (Dunlop and Özdemir, 1997). The black lines show the Koenigsberger ratio isolines. For the materials other than granites of KA, the values cluster at $10 < Q_n < 100$ providing a positive stability test. (b – g) Representative Curie curves for different groups of magnetic composition or behaviour: (b) mud-brick and (c) granite sample from KA, (d) mud-brick sample from KT all showing ideal magnetite magnetization with a single Curie point at ~580°C; (e) mud-brick sample from SPN with T_c at ~580°C pointing to the presence of magnetite as the main magnetic carrier; the extra inflexion in both curves indicate a secondary carrier (at ~350°C) which could indicate possible presence of maghemite, titanomagnetite or Al substituted magnetite (Dunlop and Özdemir, 1997); (f) Vitrified mud-bricks from KT12 that exhibit an almost reversible Curie balance curve with strong paramagnetic contribution; (g) ignimbrite showing a major difference between heating and cooling curves resulting in irreversible loss in magnetization up to 80%.

Figure 3. (a) Hysteresis loops (displayed after the paramagnetic and diamagnetic correction on a mass-specific basis) and (b) IRM acquisition curves for three different material types: mud-bricks from KA, mud-bricks from KT and vitrified mud-bricks from KT; (c) Day Plot (Day

et al., 1977) showing all magnetic mineral assemblages are composed of PSD grains; (d) representative FORC diagrams for each type of material plotted with a smoothing factor (SF) of 3 for the mud-bricks of KA and KT and SF=4 for the vitrified mud-bricks of KT. The contour interval is taken as 10. The peak distributions are centered at +0, 10 and 20 mT respectively.

Figure 4. Representative examples of demagnetization diagrams from each type of material. Closed (open) symbols are the projection of the vector end-points on the horizontal (vertical) plane. The corresponding temperature (in °C) or the alternating field (in mT) values are shown. In parentheses the method used to demagnetize the sample. Normalized intensity decay plots are also shown on either side of the demagnetization diagram. (a) Single component magnetite magnetization from the mud-bricks of SPN; (b, c) Th/AF demagnetization diagram of single component magnetite magnetization from the mud-bricks of KA with possible contribution of maghemite; (d) two component (LT and HT) demagnetization diagram from the granites of KA; (e) Single component demagnetization diagram from the mud-bricks of KT; (f) single component and two component demagnetization diagrams from the ignimbrites of KT; (g) uniformly decaying single component demagnetization diagram from the vitrified mud-bricks of KT.

Figure 5. Equal area projections of the characteristic remanent magnetization direction of each set. The red circles are α_{95} cones of confidence. N is the number of samples, k is precision parameter, and D/I is the declination/inclination. Below are the rejected data sets due to low k or high α_{95} value.

Figure 6. (a) Representative examples of a successful and a failed measurement obtained from three different paleointensity methods. The NRM-TRM plots of a thermal IZZI-Thellier (TT) and a microwave (MW) experiment are shown with associated orthogonal vector plots in core coordinates. Solid red (open blue) symbols are horizontal (vertical) planes. Diagrams

are normalized to initial NRM intensity. The arrows represent the pTRM checks engaged in every two double-treatments. P/AP/SP stands for the applied field direction parallel/antiparallel/subperpendicular to the samples NRM. The relevant temperature steps for TT experiments are shown on the side of the data point. ThellierTool4.0 (Leonhardt et al., 2004) was used to plot the data. On the left an accepted 'domain corrected' solution (MSP-DSC) and a rejected 'parallel differential pTRM' solution (MSP-DB) of multi-specimen method are shown. (b) Comparison of the results from three different protocols. The site means are shown as histograms and the individual measurements are represented in diamonds and circles.

Figure 7. Comparison of (a) inclination and (b) declination results of this study (red) with the Eastern Europe and Near & Middle East archaeomagnetic data from GEOMAGIA50v2 (grey), the Turkish data (orange and blue), and the global geomagnetic field models pfm9k.1b and SHA.DIF.14k; (c) mean site VADM values of this study (red) plotted against the data from GEOMAGIA50v2 (grey), Middle East (pink circles, orange triangles and light green squares), Turkey data (blue circles and green diamonds) from Ertepinar et al. (2012) and Hammond et al. (2015), respectively, along with the two global geomagnetic field models. All data are recalculated to Kayseri (see caption to Fig. 1).

Figure 8. (a) Areal distribution of fire events shown on an aerial photograph of site Kültepe; (b) declination, inclination and VADM distribution of Kültepe level II data points (green dots) as sorted from the most westerly to the most easterly declination based on the westerly trend in the SHA.DIF.14k model for the period of ~2100-1850 BCE. The blue dots are data points from Kültepe-level Ib and the black dots are Turkey data from Ertepinar et al. (2012). The time intervals between sites in Kültepe-level II are arbitrarily taken as 10 years.

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