

# Aristotle University of Thessaloniki

# ARCHMAT Erasmus Mundus Joint Master in ARCHaeological MATerials Scienece

### ARCHEOMAGNETIC STUDY OF BAKED CLAYS FROM A NEOLITHIC SITE IN THESSALY, GREECE

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#### Abstract

The study conducted for this thesis was a classical archaeomagnetic study with the aim of obtaining archaeodirectional data to enrich the poorly covered Neolithic period in the Greek directional SVCs. Burnt clay samples, from the kiln complex at the Middle Neolithic site Koutroulou Magoula located in Thessaly, Greece, were studied. The grouping of the initial NRM measurements was satisfactory, thus indicating that the majority of the samples were burnt in situ. Stepwise thermal demagnetization was employed to isolate the ChRM of the samples, which in most cases revealed one characteristic component of magnetization. Rock magnetic measurements were also employed to access the mineralogical composition and stability of the sampled material. Specifically, thermomagnetic analysis and coercivity spectrum analysis (i.e., acquisition of isothermal remanent magnetization (IRM), indicated that the samples contained mainly low-coercivity magnetic minerals (such as magnetite or titanomagnetite). Some samples did contain a small portion of high-coercivity minerals as well, most likely haematite. Overall, the experimental procedures proved to be successful and the mean directions (declination – D, inclination – I, and confidence parameter -  $\alpha_{95}$ ) for the site were calculated: D [°]= 6.9; I [°] = 55.9; and  $\alpha$ 95 [°] = 4.5. From the mean directions calculated a final date was purposed for the site by comparing the obtained directions to the Balkan and Neolithic reference curves. Date interval (Balkan curve) = 5463 - 5200 BC; date interval (Neolithic curve) = 5488 - 5187 BC, both at a 95% confidence level. Both dating intervals are similar suggesting a reliable date was obtained.

**Keywords:** archaeomagnetism, archaeomagnetic dating, archaeodirections, burnt clay, Neolithic Period, Greece

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#### Introduction

Archaeomagnetism and paleomagnetism are specialized studies within the field of geosciences. They have a wide range of applications to address problems in geology, archaeology, and geophysics (Tarling 1983). For example, paleomagnetism has facilitated our understanding of the phenomena of continental drift, seafloor spreading, and plate tectonics, as well as providing clarity on the processes that allow continents to grow and mountains to form (Butler 1992). Beyond their applications in the hard sciences, magnetic studies have become a valuable tool for archaeologists. As many fields have become more interdisciplinary, so too has archaeology. This has led to the expansion of the discipline and the incorporation of various other fields of science into the archaeology toolbox further aiding our understanding of the past. As we will see, archaeomagnetism is a unique part of geoscience and its application within archaeology has aided the understanding of the geomagnetic field in antiquity as well as helped deconvolute archaeological chronologies.

Considering the overall goal of paleomagnetism and how it relates to archaeomagnetism, "the primary objective of paleomagnetic research is to obtain a record of past configurations of the geomagnetic field" (Butler 1992). Furthermore, the study of the geomagnetic field is unbounded in its temporal range as it can be studied over a range of time scales, including archaeological, geological, and even cosmic (Tarling 1983). Herein lies the distinction between paleomagnetism and archaeomagnetism. They don't differ in the principles that inform the techniques and research but rather the distinction reflects the purpose of the research. Archaeomagnetism, at its core, has the same primary objective as paleomagnetism of obtaining a record of the past geomagnetic field. However, archaeologists can employ archaeomagnetism as a dating technique for archaeological materials and sites, while the applications of paleomagnetism, as mentioned, are more firmly rooted in Earth and Geosciences. Thus, the simplest way to distinguish the two is that archaeomagnetic studies

are conducted within archaeological contexts, whereas paleomagnetic studies are longer-term (Tarling 1983).

If the overall goal of archaeomagnetism is to obtain a record of the past geomagnetic field through the determination of the geomagnetic field components so that this record can be employed to date archaeological materials, how, then, is it possible to identify the past geomagnetic field components using archaeological materials? First, it is based on two physical phenomena:

(i) The Earth spontaneously generates a magnetic field, which fluctuates in intensity and direction with time.

(ii) Under certain conditions naturally occurring magnetic minerals can become permanently magnetized [i.e., acquire a remanent magnetization] according to the magnetic field pertaining at that time.

(Linford 2004)

Magnetic minerals are present in most soils, clays, and even as trace elements in many types of rocks (Linford 2004). Thus, they are common in most types of archaeological materials, however the materials most utilized for archaeomagnetic investigation are clay artifacts and structures, such as kilns, ovens, bricks, tiles, and in general any kind of burnt clay (Aidona et al. 2010). If they have gone through a remanence inducing event, usually for archaeological materials this is by means of thermal processes, they can obtain and preserve a stable remanent magnetization that is parallel and proportional to the geomagnetic field at the time and place of the remanence inducing event (Linford 2004). Overall, the requirements for obtaining magnetic data from archaeological materials are as follows. The materials must:

- (i) contain magnetic minerals capable of carrying a stable remanent magnetization,
- (ii) have experienced a remanence-inducing event at some time in their history...,
- (iii)have remained undisturbed since acquiring the remanence so that its direction is still meaningful.

(Linford 2004)

However, the requirement of in situ materials is only necessary for obtaining directional data, as displaced material can be used for intensity determinations (Tarling 1983).

Moreover, by studying materials that preserve these components the data can be plotted to create secular variation curves (SVCs) if the materials have been dated by other independent methods, such as by reference to archaeological chronologies, or by radiocarbon or thermoluminescence dating (Aitken 1978). These curves allow of us to investigate and visualize the changes of the Earth's magnetic field through time. As well, for regions with reliable SVCs the magnetic data obtained from archaeological materials of unknown age can be used to date the material by comparing their magnetic data (direction and intensity) with secular variation curves for the same region (De Marco et al. 2014).

Regarding archaeomagnetic research in Greece the requirement of in situ material for the determination of the directions of the past geomagnetic field has resulted in a disparity between intensity data and directional data, with an abundance of intensity data and less directional data (Tema & Kondopoulou 2011). Further, most of the published data for the Greek directional SVCs corresponds only to the last 4 millennia with only 4 new data points available for the Neolithic period (figure 1) (Aidona and Kondopoulou 2012; Fanjat et al. 2013). (See De Marco 2007 & De Marco et al. 2014 for more detail on the archaeodirectional data set and SVCs available for Greece). So, due to poor coverage of the Neolithic period that currently exists in the Greek directional data, the goal of this thesis is to conduct a classic archaeomagnetic study of baked clays from Koutroulou Magoula, a Neolithic tell site in Thessaly, Greece. The aim is to obtain archaeodirectional data from the samples taken from the site, with the hope that the expected results will enrich the poorly covered Neolithic period in the Greek Directional Secular Variation Curves.



**Figure 1.** Directional Secular Variation Curves for Greece. Red stars indicate new data obtained after 2010, while the black dots correspond to the data used for the construction of the SVCs. (De Marco et al. 2014).

#### Chapter 1: History of archaeomagnetism

#### 1.1 History of magnetism and the development of the discipline

Archaeomagnetism is not a new discipline/technique; although, one could argue that its popularity and application as a tool for archaeologists has only been realized within recent decades. However, the history of magnetism as a science and the awareness of the magnetic properties of the Earth has a much longer history. Since ancient times people were aware, to some degree, of the magnetic properties of stones (Tarling 1983). For example, it is said that "the earliest observations on magnets are supposed to have been made by the Greek philosopher Thales in the sixth century B.C." (Merrill et al. 1996). As well, it is thought that the term "magnetism" comes from the city of Magnesia (An ancient Greek city founded at the beginning of the 8th and 9th centuries BC, located in what is now modern-day Turkey (Bingöl 2013), as the etymology of the word "magnet" is thought to refer to "the stone from Magnesia" (Buschow and de Boer 2003). As researchers and archaeologists within Greece, we tend to focus on this area of the world and orient our knowledge within that framework. But, if we look to other areas, we also see an awareness of magnetic properties early on.

For example, sites attributed to the Olmec civilization that emerged in the Americas in what is now modern-day Mexico around 1400 BC have revealed an early acquaintance with magnetism (Guimarães 2004). Specifically, "archaeologists have found Olmec objects made of iron ore, dating from the Early Formative period (1500 – 900 BC)" (Guimarães 2004). One artifact that stands out from the rest is a polished magnetic bar that dates between 1400 – 1000 BC (figure 2) (Guimarães 2004). It has been speculated that this bar might have been



**Figure 2.** Small polished magnetic bar of haematite with a carefully cut groove running down the length of the bar. Found in San Lorenzo (oldest known Olmec center located in modern day Mexico) and dated 1400 - 1000 BC. Scale in centimeters. (Carlson 1975).

part of a magnetic compass. Although its function is debated, these types of finds tell us that the Olmec people may have known the properties of magnetic ores early on and at the least they had the ability to work magnetic minerals, as they used magnetic iron ores to manufacture various objects for daily use (Guimarães 2004). Conversely, Needham (1962) states that the earliest form of the magnetic compass was invented in China as early as the second century BC (figure 3) (cited in Merrill et al. 1996). The evidence from China is less disputed and tells us that they had an awareness of magnetic directivity.



**Figure 3.** Drawing and reconstruction of what is thought to be the earliest form of a Chinese geomagnetic lodestone compass. The south pointing lodestone spoon is carved and balanced to allow it to rotate freely on a polished bronze plate. (Drawing and reconstruction were produced by Wang Chen-To. Images originally published in Needham 1962). (Cited in Carlson 1975).

Furthermore, it wasn't until the 12th century AD that the discovery of the magnetic compass reached Europe (Merrill et al. 1996). Despite its slow diffusion from China, in the centuries that followed its introduction and use in Europe new information was discovered surrounding the magnetic properties of the Earth as people started to investigate the Earth's magnetic field in greater detail. For example, William Gilbert, a physician to Queen Elizabeth I, conducted a series of experiments investigating "the variation in inclination over the surface of a piece of lodestone cut into the shape of a sphere" (Merrill et al. 1996). The result of this experiment led him to discover that the Earth acts like a giant magnet; this information along with the results of his other experiments investigating magnetism were published in 1600 in a treatise entitled De Magnete (Merrill et al. 1996).

Later, the first geomagnetic chart depicting the Earth's declination was drawn up by Edmund Halley and published in 1702, following the first scientific sea voyages that took place between 1698 and 1700 (Merrill et al. 1996). This first geomagnetic chart only referred to declination, as the first chart referring to inclination wasn't published until 1768, by Johan Carl Wilcke in Stockholm (Merrill et al. 1996). Because of these early investigations we have direct historical magnetic (directional) data going back to the Early Modern period, i.e., the beginning of the17th century (figure 4) (De Marco 2007).



**Figure 4.** Distribution of directional data through time. (a) Declinations 1600 - 1649. (b) Declinations 1650 - 1699. (c) Declinations 1700 - 1749. (d) Declinations 1750 - 1799. (e) Inclinations 1600 - 1699. (f) Inclinations 1700 - 1799. (g) All data 1800 - 1849. (h) All data 1850 - 1899. (Jackson et al. 2000).

Then, in the 1830s C.F. Gauss, a German mathematician, developed a method for measuring the absolute intensity of the magnetic field (Jackson et al. 2000; Merrill et al. 1996). Gauss was the first to represent the geomagnetic field in mathematical form (Merrill et al. 1996) From his contributions and the regular operation of magnetic observatories starting in the 1840s, we see the continuous observation of the total geomagnetic field from the mid 1800s onwards (De Marco 2007). Fast forwarding to today, there are many geomagnetic observatories active across the globe, "where continuous measurements of the geomagnetic field are made for long stretches of time with the best possible accuracy" (figure 5) (Gunnarsdóttir 2012).



**Figure 5**. Global distribution of geomagnetic observatories. (Cafarella et al. 2008).

Despite access to direct observations from historical records and geomagnetic observatories, the period this data represent is short when compared to the timescales observed in paleo and archaeomagnetic studies (Jackson et al. 2000). So, because historical data sets are restricted both temporally and spatially, paleo and archaeomagnetism studies through indirect measurements (i.e., through the study of materials that preserve the past values of the components of the geomagnetic field) can investigate the geomagnetic field at greater time scales and in wider regions than historical data sets cover. (For a more detailed account on the history of study see Tarling 1983 & Merrill et al. 1996).

#### **1.2** Current state of archaeomagnetic research globally

Compared to the long history of investigation into magnetism and the Earth's magnetic field, archaeomagnetism is a much newer development. But, relative to other scientific methods, specifically dating methods, archaeomagnetism can be considered the oldest geophysical-geochemical dating technique available to archaeologists (Tarling 1975). This is due in part to the first detailed studies of magnetic properties of volcanic rocks by Delesse (1849) and Melloni (1853), and later Folgerhaiter's (1894, 1895, 1899a, b) expansion of these studies (cited in Tarling 1983). Folgerhaiter concluded that "not only did the volcanic rocks acquire their magnetization on cooling, but also the direction of the acquired magnetization paralleled the geomagnetic field" (Tarling 1983). He then went on to also study bricks and pottery. He observed that the remanent magnetization of "vases buried for 2000 years was randomized, i.e. each vase had preserved its initial direction of magnetization for that period of time" and concluded that if there firing positions were known this could provide a record of the direction of the geomagnetic field (Tarling 1983; Merrill et al. 1996). Although these early realizations started magnetic studies of archaeological materials, it wasn't until the latter half of the 20th century when significant improvements were made "in the theoretical understanding of the processes of magnetization, particularly in rocks, and in instrumentation for measuring and isolating their magnetization," that archaeomagnetic studies became more widespread (Tarling 1975). Still despite decades of paleo and archaeomagnetic research there are many regions with no magnetic data or limited data. (See figure 6 for a compilation of archaeomagnetic data across the globe. For a more details on both paleo and archaeomagnetic data available globally see Korte et al. 2005). Thus, we continue to investigate to expand magnetic data sets both spatially and temporally.



**Figure 6.** Global complication of archaeomagnetic data. (a) Sites of archaeomagnetic directional data. (b) Sites of archaeomagnetic intensity data. (Left) The average locations of archaeomagnetic regions are plotted. (Right) Concentrations of the data are shown. (Korte et al. 2005).

Moreover, just in the last 1-2 decades the field of archaeomagnetism has made substantial steps forward both at regional scales and globally with an increase in the number studies being conducted (Kondopoulou et al. 2015). As a result, a considerable amount of new and more accurate directional and intensity data have been produced (Tema & Kondopoulou 2011). In turn, this has led to the establishment of Secular Variation Curves for many regions around the world; specifically in Europe there are SVCs available for France, the Iberian Peninsula, Germany, Austria, Italy, the United Kingdom, Hungary, Bulgaria, and Greece (De Marco 2007; Tema & Kondopoulou 2011).

Global databases have also been enriched by the uptake in the number of studies being conducted. While these large databases are mainly used for broader purposes, such as modeling the global features of the Earth's magnetic field, global models have an important role in any archaeomagnetic work; they can be used as a means of comparison for checking the accuracy of regional data. But global models do not replace well established regional data. So, although increased archaeomagnetic research has enriched all magnetic databases and has consequently led to the improvement of geomagnetic field models (GMF) at regional and global scales, because of the spatial and temporal variations of the geomagnetic field we cannot rely solely on already well established GMFs to extrapolate the data to regions with poor coverage. Rather, further research at regional levels is required to accurately model regional changes in the geomagnetic field and for the greatest applicability of archaeomagnetism as a dating technique.

Right now most of the SVCs available for Europe comprises mainly of directional data and cover only the last three millennia, apart from Bulgaria (De Marco 2007; Tema & Kondopoulou 2011). "The Bulgarian database is the richest in Europe, covering the last 8000 years" (Tema & Kondopoulou 2011). This is important, as we will see, because databases from areas surrounding Bulgaria, including Greece, can be enriched if combined with the data from Bulgaria (Kondopoulou et al. 2015; Tema & Kondopoulou 2011.) This can supplement lack of data in some instances or be used as a means of comparison, but again it doesn't replace well-constructed regional curves. Overall, the various European datasets, as well as "the Greek SVC [especially for archaeodirectional data] need still new data from specific periods where lack of adequate, well-dated, structures hampers further archaeomagnetic research" (Kondopoulou et al. 2015).

#### 1.3 Greek Data

Greece followed the developments within the field of archaeomagnetism as the first archaeomagnetic investigations in Greece took place in the 1960s. However, systematic investigations didn't start until the late 1970s. Since then, various studies have been carried out investigating both intensity and direction of the past geomagnetic field in Greece. (See De Marco 2007 & Tema Kondopoulou 2011for more details). But most studies utilized displaced materials, limiting them to archaeointensity determinations only. This has resulted in a

situation opposite to what we see in the rest of Europe. In general, Europe has a greater number of directional data compared to intensity data, Greece, on the other hand, has far more intensity data compared to directional data. (Tema & Kondopoulou 2011). Although, the early studies, in the latter half of the 20th century, provided a large quantity of data in many cases their reliability has been questioned as the data was "obtained by different specialists, using different techniques and methods" (Tema & Kondopoulou 2011). In contrast to these rocky and uneven beginnings, "since 2000 a considerable number of high quality archaeomagnetic data have been obtained in Greece" (Tema & Kondopoulou 2011).

Further, not only has continued work obtained new high quality data, but older data has been compiled and re-evaluated. The first case of this was Evans (2006) who published a systematic compellation of Greek directional data up to that date (cited in De Marco et al. 2014). Then, the PhD thesis of De Marco (2007) not only added new magnetic data from six sites in Greece, but also complied and re-examined previous intensity and directional data that was then used to construct secular variation curves for the Aegean region (figure 7). Also, De Marco et al. (2014) published an updated catalogue and SVC for Greek directional data. Overall, the extensive archaeomagnetic work in Greece, in the last two decades, has resulted in SVCs for the region that span the last 8000 years for intensity and the last 4500 years for direction (figure 8) (Kondopoulou et al. 2015).



**Figure 7.** (Top) Geographical distribution of archaeodirectional data complied to construct the directional SVCs for the Aegean area. (Bottom) Geographical distribution of archaeointensity data complied to construct the intensity SVC for the Aegean area. (De Marco 2007).



**Figure 8.** The Secular Variation Curves constructed for the Balkan peninsula (including Greece). (a) Declination, (b) inclination, and (c) intensity. All data points are reduced to Thessaloniki (40.60° N, 23.00° E). Although, these SVCs are not up to date they clearly show the disparity between Greek intensity and directional data, as well, the limited temporal range of the Greek directional data. (Tema and Kondopoulou 2011).

#### Chapter 2: Magnetism and the Earth's geomagnetic field

#### **2.1 Introduction**

Archaeomagnetic studies at first sight seem simple, but rather the reality is that magnetism is a complex phenomenon (Tarling 1983). As Butler remarked "generally students find developing an intuitive feel for magnetism and magnetic fields more difficult than for electrical phenomena" (Butler 1992). One reason for this is, we don't experience magnetic phenomena in a direct way like the way we experience the pull of the Earth's gravitational field for ourselves (Tauxe et al. 2018). Nevertheless, we are aware of the magnetic properties of the Earth and its materials. But awareness doesn't equal complete understanding. So, we continue to study the changes of Earth's magnetic field through time by examining materials that have been acted on by said field. This has led researchers to a better understanding of the dynamics of the Earth and has allowed for magnetic studies to be employed in other disciplines such as archaeology. Thus, extending the application of magnetic studies from merely an investigative approach into the dynamics of the Earth to now also as a tool that can be used for dating various materials in a variety of temporal contexts.

I won't go into depth on the theoretical aspects of magnetism as it is beyond the scope of this paper and not required for understanding the archaeomagnetic method and results. However, some of the basics need to be covered, even if at a superficial level, to orientate the results into the broader phenomenon that is magnetism. So, in this chapter I will briefly discuss the theory and concepts associated with magnetism and the Earth's magnetic field. This will include (1) some basic definitions of magnetic properties, (2) a description of the Earth's geomagnetic field with consideration given to its origin and how we understand/model it, (3) the nondipole components of the surface geomagnetic field, (4) the geomagnetic fields temporal (secular variations) and spatial variations, (5) the basics on magnetic materials, and (6) the types of magnetizations that can occur within these materials.

#### 2.2 Magnetism

Magnetism is one of the oldest sciences and is a unique feature of our Earth as it can be adequately measured through time, whereas other geophysical properties, such as seismicity, gravity, and electrical properties, cannot be (Tarling 1983). This is because, unlike magnetic properties, the others "are transient features that leave no clear trace of their previous values" (Tarling 1983). So, we know that the Earth's magnetic field leaves a trace, and, at the most basic level, we know that magnetism is the attractive and repulsive forces observed in various materials. But what is a magnetic field?

At the lowest level, the atomic scale, the spinning of an electron creates a magnetic field; this is because, "a magnetic field is produced by the movement of an electrical charge" (Tarling 1983). However, "magnetic fields are different from electrical fields in that there is no equivalent to an isolated electrical charge; there are only pairs of "opposite charges" – magnetic dipoles" (Tauxe et al. 2018). Therefore, because isolated magnetic charges, i.e., monopoles, do not exist a magnetic dipole is the smallest unit of magnetism (Butler 1992). Furthermore, here are the definitions of the basic properties associated with magnetic phenomena.

Magnetic Moment: "The Magnetic dipole moment or more simply the magnetic moment, M, can be defined by referring either to a pair of magnetic charges... or to a loop of electrical current" (Butler 1992). (For the sake of simplicity, I will refer to a pair). "For the pair of magnetic charges, the magnitude of charge is m, and an infinitesimal distance vector, I, separates the plus charge from the minus charge" (Butler 1992). Thus, we can define magnetic moment as: M = m I. This is the simplest way to define the smallest unit of a magnetic charge, i.e., a magnetic dipole. Although we can define these concepts mathematically, this multipole combination is just that, "more a mathematical convention rather than a physical

reality" (Butler 1992). Overall, magnetic charges don't exist as singular phenomena but are paired charges, and while our definitions of these concepts are more theoretical, we have mathematical conventions to define and describe magnetic phenomena.

- Magnetic Field: "Magnetic force field or magnetic field, H, in a region is defined as the force experienced by a unit positive magnetic charge placed in the region" (Butler 1992). There is no experiment that can be performed following the conditions of the definition. (Butler 1992). However, "an experiment that you can perform... is to observe the aligning torque on a magnetic dipole moment placed in a magnetic field" (Butler 1992). This is how we study the Earth's past geomagnetic field. We cannot directly measure its previous values, so we do this by analyzing magnetic materials that have been acted upon by the Earth's magnetic field. The simplest way to understand this concept is by observing a compass. "A magnetic moment that is free to rotate will align with the magnetic field" (Butler 1992). This is the underlying mechanism that allows a compass to work. Meaning, a compass needle, which has a magnetic moment, when allowed to freely rotate, the needle, i.e., the magnetic moment, will align with the horizontal component of the geomagnetic field, thus determining the magnetic azimuth, or in other words, it will point north. (Butler 1992). For the study of the past geomagnetic field, we are observing the past alignment of magnetic minerals with the geomagnetic field that are preserved within a material.
- Magnetic Intensity: As touched on earlier not only do magnetic phenomena have a directional component but also an intensity. The magnetic intensity, J, of a material can be defined as "the net magnetic dipole moment per unit volume" (Butler 1992). When considering magnetic materials, we see basically two types

of magnetizations occurring: induced magnetization and remanent magnetization (Butler 1992). "When a material is exposed to a magnetic field H, it acquires and induced magnetization, **J**i" (Butler 1992). "These quantities are related through the magnetic susceptibility,  $\chi$ : **J**i =  $\chi$  **H**;" where the induced magnetization of a material equals its magnetic susceptibility times the strength of the field it is being exposed to (Butler 1992). In other words, the strength of the magnetic field a material is exposed to and the level of susceptibility a material has of acquiring a magnetization both play a role in the resulting magnetic intensity of a material.

Magnetic Susceptibility: We can define magnetic susceptibility, χ, simply "as the magnetizability of a substance" (Butler 1992).

Although briefly covered here, these concepts are the pillars to understanding how magnetic processes work and are the basic properties of magnetic materials. The magnetic phenomenon that is vital to archaeomagnetic studies is the magnetization a material can acquire when exposed to an external magnetic field. More specifically, as mentioned, there are two main types of acquired magnetization, the one not covered above, remanent magnetization, "is a recording of past magnetic fields that have acted on a material" (Butler 1992). This type of magnetization is the focus of paleo and archaeomagnetic research. Later, we will see how materials can acquire and retain this type of magnetization that records both the intensity and direction of the past geomagnetic field and how we isolate and measure a materials remanent magnetization.

#### 2.3 The geomagnetic field

The most basic way we can visualize the Earth and its magnetic field is as a uniformly magnetized sphere or as a magnetic field similar to that produced by a bar magnet (figure 9) (Linford 2004; Thompson and Oldfield 1986). This idea that the geomagnetic field can be



**Figure 9.** Approximation showing that the Earth's magnetic field acts like a simple magnetic dipole. (Gunnarsdóttir 2012).

equated to a simple dipole magnetic field, as mentioned, was first recognized by William Gilbert in 1600. Later, Gauss, with his method for analyzing the geomagnetic field and from observations of the surface field he determined the Earth's magnetic field originated from within the Earth. (Thompson and Oldfield 1986). Today, it is still accepted that the Earth's geomagnetic field is internally generated. However, we now know the geomagnetic field is a bit more complicated. The geomagnetic field measured at the surface of the Earth is not produced by one source, i.e., an internally generated dipole field. On the contrary, the geomagnetic field of the Earth "is a superposition of magnetic fields generated by different sources" (Gunnarsdóttir 2012). The different sources include:

- The main field, generated in the Earth's fluid core by a geodynamo mechanism;
- The crustal field, generated by magnetized rocks in the Earth's crust;
- The external field, produced by electrical currents flowing in the ionosphere and in the magnetosphere, owing to the interaction of the solar

electromagnetic radiation and the solar wind with the Earth's magnetic field;

• The magnetic field resulting from an electromagnetic induction process generated by electrical currents induced in the crust and the upper mantle by the external magnetic field time variations.

(Lanza and Meloni 2006)

The internally generated field is the most dominate one as it accounts for 90% of the surface field (Butler 1992). But collectively these various sources contribute to the total geomagnetic field that we observe at the surface of the Earth.

However, I want to focus back on the main field by discussing its origins and how it is sustained. The origins of the Earth's magnetic field (i.e., the main field) and its sustainability over time is a complex matter that is still a topic of discussion. Nevertheless, the best working theory we have today is that of the dynamo theory that was first developed in the 1940s and 1950s (Thompson and Oldfield 1986). Basically, it is the idea that the magnetic field is associated to the free electron circulation within the convection currents in the outer core of the Earth, which consists of molten iron and nickel (Linford 2004). The energy source that drives these convection currents "is thought to be either the radioactive decay of elements in the Earth's core... or the gravitational energy released by the sinking of heavy material in the outer core" (Thompson and Oldfield 1986). Despite the source of the driving energy "the resulting thermal or compositional instabilities lead to the formation of convection currents," this in turn creates a self-sustaining dynamo within the outer core of the Earth (Thompson & Oldfield 1986). Thus far, the basic mechanisms of how the Earth's magnetic field is generated have been covered and that in the simplest form we can visualize the Earth's magnetic field as a bar magnet. But it is necessary to model the Earth's magnetic field further to understand the magnetic measurements taken at the surface.

#### 2.3.1 The geocentric axial dipole model

"The study of the geomagnetic field requires some model for use in analyzing paleomagnetic [or archaeomagnetic] results, so that measurements from different parts of the world may be compared" (McElhinny and Mcfadden 2000). The model used to roughly represent the morphology of the geomagnetic field is that of a geocentric axial dipole (GAD) field model (figure 10) (Butler 1992). It is a simple model that "corresponds to the field of a



**Figure 10.** Model of a geocentric axial dipole. The magnetic dipole (**M**) is located in the center of the Earth and is aligned with the rotational axis. The magnetic field directions as would be produced by a GAD are schematically shown. (Redrawn from Butler 1992).

geocentric dipole directed along the rotational axis," meaning that the "geomagnetic and geographic axes [or poles] coincide as do the geomagnetic and geographic equators" (McElhinny 1973). The GAD represents an idealized version of the Earth's geomagnetic field, but while a simplified version it still has a base in reality; as the variations of the Earth's magnetic field "*when averaged over periods of several thousands of years*" conform to this simplified model (McElhinny 1973). This in turn supports the hypothesis that at the most basic level the Earth's magnetic field is a geocentric axial dipolar field (Butler 1992). Thus, it can be said that over long periods of time the geomagnetic field conforms to this simplified model but a snapshot of the field at any moment in time would show that the Earth's magnetic field is more complex and deviates from the simple configuration presented with the GAD model (Butler 1992).

To understand these deviations, an adjusted model is necessary to best represent the magnetic field at any given moment. This adjusted model is an inclined geocentric dipole model, where the geocentric dipole is inclined to the rotational axis of the Earth (Butler 1992). The inclined model that best fits the geomagnetic field today is one where the geocentric dipole "has an angle of [about] 11.5° with the rotation axis" (figure 11) (Butler



1992). This inclined model accounts for about 90% of the surface geomagnetic field; in some locations it perfectly describes the surface field while in other areas, even the best-fitting dipole model falls sort, where up to 20% of the surface field cannot be accounted for (Butler 1992). This tells us that although much of the surface field can be attributed to the Earth's internally generated dipole field, "the amount remaining is significant" (Butler 1992). This is because, while dominantly dipolar, the geomagnetic field observed at the surface is a combination of various components, including a nondipolar component that seems to be the major contributing factor to the differences seen at some locations to that of the inclined geocentric axial dipole model (Butler 1992).

#### 2.3.2 Nondipole field

To understand the contributing sources to the Earth's geomagnetic field the bestfitting dipole field can be subtracted from the surface field, what remains is called the nondipole field (Butler 1992). As mentioned, the magnetic field observed at the surface is a combination of various sources, but those mentioned in section 2.3, such as the crustal field and the external field only contribute a very small portion to the total surface field. However, one source that contributes a more significant amount to the total field is the nondipole field. It is a field that is distributed unevenly over the surface of the Earth with six or seven continental sized features that dominate it (Figure 12) (Butler 1992; De Marco 2007). It is



**Figure 12.** The morphology of the nondipole geomagnetic field from 1945. The arrows represent the magnitude and direction of the horizontal component of the nondipole field; contours indicate vertical intensity of the nondipole field. (For more detail see Butler 1992). This is simply to show the variation present in the nondipole field to understand how the nondipole field contributes to intensity and directional variations spatially. (Butler 1992).

suggested that the nondipole field originates from the "fluid eddy currents in the outer core near the interface with the overlying solid mantle" (figure 13) (Butler 1992). Furthermore, the



**Figure 13.** Depiction of the Earth's main dipolar field. The eddy currents are shown near the core/mantle boundary which are thought to create the nondipole field. (Linford 2004).

nondipole features are dynamic as they exhibit growth, decay, and motion (Butler 1992). So, just as the dipole portion of the field changes in direction and intensity, the nondipole component also affects the temporal and spatial variations we observe at the surface of the Earth. Overall, we can say that the geomagnetic secular variation we observe is a result of two contributing phenomena: "(1) nondipole changes dominating the shorter periods and (2) changes in the dipolar field with longer periods" (Butler 1992). Furthermore, we have methods for separating these two components to see their changes through time independently of one another. However, studying them separately is unnecessary in most archaeomagnetic work. What we must keep in mind is that the magnetic field at the Earth's surface, what we analyze through archaeological materials, is a combination of various sources, with the dipole and nondipole fields making up the majority.

#### 2.3.3 Secular variation

Secular variation has been mentioned briefly thus far, but this topic requires further discussion as it is the focus of most magnetic studies, either directly for studies set out to

build secular variation curves for various regions, or indirectly in cases where secular variation curves are used to date materials and sites. To reiterate, changes in the direction and intensity of the surface geomagnetic field with time constitute what we call geomagnetic secular variation (Butler 1992). Patterns of secular variation tend to be similar over subcontinental regions but vary significantly from continent to continent (figure 14) (Butler



**Figure 14.** Secular variation of London, Paris, Rome, and Boston plotted from historical records. The comparison between these four locations show that secular variation patterns are similar over subcontinental regions. Paris and London have very similar directions. Although, Rome deviates from both, its pattern is still similar. Boston, on the other hand, has a completely different pattern. Thus, supporting the statement that from continent-to-continent secular variation patterns are very different. (Aitken 1978).

1992). As mentioned above, variations in the dipole field dominate changes over longer periods while the nondipole field seems to be the driving force behind the changes observed in the geomagnetic field over shorter time periods. We can get a general idea about the dipole changes by looking at the position of the north geomagnetic pole through time (figure 15).



**Figure 15.** Positions of the north geomagnetic pole over the past 2000 years. The square is the mean geomagnetic pole position over this period. At some points it has seemed that directional changes are cyclical however, as I hope this representation shows, directional changes can be described as a random walk around the mean direction. (Butler 1992).

Further, to visualize temporal changes, magnetic data is plotted to create geomagnetic secular variation curves (SVCs). As mentioned, we have direct observations of the variations of the geomagnetic field beginning from the Early Modern period in Europe. But, because historical data is limited both temporally and spatially we cannot rely solely on it to understand secular variations. First, because "the time range needed to effectively model secular variation is greater than that provided by historical observations alone" (De Marco 2007). Second, because the spatial variation seen across the global seem to reflect "the size of the nondipole sources of the geomagnetic field within the Earth's core;" the construction of separate secular variation curves for different regions is required (Butler 1992; De Marco 2007). Thus, to extend our data of the Earth's magnetic field before the 17th century and into different regions not observed in historical data, we can employ archaeomagnetic measurements of archaeological features and artifacts as well as palaeomagnetic measurements of geological samples, such as sediments or lava flows, to observe greater time scales and wider regions (De Marco 2007).

Furthermore, the need for separate secular variation curves from different regions due to the spatial variations that are present at the surface of the earth has led to discussions on what the optimal size is for archaeomagnetic regions. Meaning over what distances does magnetic data show similar patterns, and at what point will we see directional scattering, because directional scattering "will introduce significant errors into the compiled secular variation record" (De Marco 2007). To determine this, we must consider how we compare data from different sites within a specific region. We cannot simply compare data acquired from different sites without any transformation of the data. On the contrary, a specific method must be employed to relocate the data points from a specified region to a singular reference point (Tema & Kondopoulou 2011). "In archaeomagnetism, the most commonly used method for relocating remanence vectors to a reference point is the relocation via pole method" (Tema & Kondopoulou 2011). Thus, to understand at what distances magnetic data starts to become dissimilar Casas & Incoronato (2007) conducted an analysis on the distribution of the relocation errors for both direction and intensity (cited in Tema & Kondopoulou 2011). They "showed that in Europe, errors are restricted to about 0.25°/100 km [for direction] and 100-200 nT/100 km [for intensity]" (Tema & Kondopoulou 2011). So, the consensus, at least for Europe, is that SVCs can be constructed using data from areas with a radius less than 1000 km, without introducing any significant errors (De Marco 2007; Tema & Kondopoulou 2011). Based off the 1000km parameter, the generally tendency seems to be the construction of separate curves for different countries. There are exceptions (due to size of countries or depending on where the central reference point is located which could allow data from multiple courtiers to be combined, as seen with the SVCs constructed for the Balkan Peninsula), but, generally, this method tends to work well in most cases.

Overall, observing and analyzing the secular variations of the Earth's magnetic field is the method we have at our disposal today for investigating the processes and dynamics

associated to the Earth's geomagnetic field (De Marco et al. 2014). At the same time, such information "can be used to date archaeological material of unknown age by comparing their archaeomagnetic direction and/or intensity with the SV reference curves for a certain region" (De Marco et al. 2014). The accuracy of the archaeomagnetic dating method is dependent on many variables. However, the key feature for the success of this dating method is the reliability of the reference SV curve used. "In order to construct a reliable SV curve, a significant number of well-dated archaeomagnetic data, homogeneously distributed over time and coming from small geographic regions, is necessary" (De Marco et al. 2014). So, the first and foremost goal of any archaeomagnetic work is either the construction or use of appropriate and reliable secular variation curves.

#### 2.4 Data Presentation

It has been stated that a magnetic field has both a direction and an intensity, this makes a magnetic field a vector field (Tauxe et al. 2018). Thus, as any magnetic field is a vector field, so too is the Earth's geomagnetic field. In order to completely define the geomagnetic field, or any magnetic field, three components are required: declination inclination and intensity (figure 16) (Thompson and Oldfield 1986). In figure 16 F represents the total field vector, and the intensity of the vector is given by its length (Butler 1992; De Marco 2007). The directions (declination and inclination) of the vector "are normally expressed in terms of polar co-ordinates" (Tarling 1983). Declination (D) is the horizontal component of the direction of magnetization and is measured in degrees clockwise from geographic North (Tauxe et al. 2018; Tarling 1983). Inclination (I) is the vertical component of the direction from the horizontal plane); "it is positive downward and



**Figure 16.** The components of a magnetic field (Declination - D, Inclination – I, and the total field vector (intensity) - F) represented in relation to both polar and cartesian coordinates. (Merrill et al. 1996).

ranges from +90° for straight down and -90° for straight up" (Tauxe et al. 2018). The measurement of magnetic vectors is usually in reference to cartesian coordinates (x, y, and z), however, they can be transformed so that vector measured reflects its geographic position. Most programs that run the instruments for measuring magnetic vectors, or the program used to process magnetic data can provide a vectors geographic position if the geographic orientation of the material being measured is input into the program without the operator having to transform the data themselves.

Overall, in paleo or archaeomagnetic studies such as this present thesis, we are observing and measuring the surface geomagnetic field and how it has acted on materials in the past. We do this by examining the remanent magnetization present in various materials to ascertain the characteristic components of the geomagnetic field vector at the surface of the Earth at the time the materials in question were created (or in other words underwent a remanence inducing event). So, then, the question arises of what types of materials can preserve this information?

#### **2.5 Magnetic materials**

#### **2.5.1 Diamagnetic and paramagnetic materials**

At the atomic level all materials can be considered magnetic (Tarling 1983). However, not all can acquire a remanent magnetization. So, while in a general sense all materials are magnetic, we can further subdivide them into various categories based on their atomic structures and in turn how they react to an applied magnetic field (Tarling 1983). First considering the materials that can't acquire a remanent magnetization: diamagnetic and paramagnetic materials (Tarling 1983).

Diamagnetic substances are those whose electron shells are full (Tarling 1983). Because they are full "the electron spin motions are paired and cancel each other out" (Thompson and Oldfield 1986). Thus, the spinning of electrons in diamagnetic materials do not create a magnetic moment within the materials, meaning that in the absence of an applied magnetic field they are nonmagnetic (Thompson and Oldfield 1986). However, when placed in a magnetic field they will acquire a very weak induced magnetization in the opposite direction of the applied field; but once they are removed the magnetization will be lost (Tarling 1983; Thompson and Oldfield 1986).

Paramagnetic substances, on the other hand, are substances whose outer electron shells are incomplete (Tarling 1983). This means that "each atom has a magnetic moment due to the uncompensated electron spins" (Tarling 1983). When these substances are placed within a magnetic field, their magnetic moments will tend to align in the same direction as the applied field (Tarling 1983; Thompson & Oldfield 1986). Thus, paramagnetic materials, unlike diamagnetic substances can acquire an induced magnetization that is parallel to the applied field (Butler 1992). On the other hand, like diamagnetic materials, paramagnetic materials in the absence of an applied field are nonmagnetic, but for a different reason. It is because at any temperature above absolute zero the thermal energy causes the atomic
magnetic moments of paramagnetic materials to oscillate rapidly and randomly resulting in the random orientation of the electron spins (Butler 1992). Thus, in most real-world conditions and in the absence of an applied magnetic field the atomic magnetic moments in paramagnetic materials will be equally distributed in all directions resulting in no overall magnetization (Butler 1992).

To summarize, paramagnetic and diamagnetic materials can acquire an induced magnetization when exposed to an external magnetic field, but their magnetization reduces to zero when they are removed from such a field. Thus, they are nonmagnetic in the absence of an applied field. Conversely, with regards to archaeomagnetic research, we are interested in the type of magnetic materials that can acquire a remanent (or permanent) magnetization.

## **2.5.2 Ferromagnetic materials**

There are materials that in the absence of an applied field possess a magnetization (Tauxe et al. 2018). This type of response is termed remanent or spontaneous magnetization; the phenomenon is also loosely termed ferromagnetism (Tauxe et al. 2018). Like paramagnetic materials, ferromagnetic materials possess atoms with unpaired electrons in their outer shells. However, unlike paramagnetic materials where there are no interactions between adjacent atomic magnetic moments the opposite is true for ferromagnetic materials. Ferromagnetic materials possess strong interactions between neighboring atomic spins and these strong interactions result in their ability to acquire a remanent magnetization (Butler 1992; Tauxe et al. 2018). It is this fundamental property of ferromagnetic materials that makes them the focus of archaeomagnetic studies as the remanent magnetization they can acquire records the components (direction and intensity) of an applied magnetic field (Butler 1992). This is because after removal from the magnetic field their magnetization does not return to zero, but under the right circumstances the material can retain a record of the applied field (Butler 1992). Moreover, the remanent magnetization that they can acquire is

stable and, in a sense, "fossilizes" the direction and intensity of the magnetic field within the material at the time it underwent a remanence induing event (e.g. when it was heated to high temperatures and subsequently cooled) (Aidona et al. 2010). (This process will be covered in more detail in the following section).

Further, ferromagnetic materials can be divided into three categories based on the type of spin alignment (also referred to as exchange coupling) their unpaired electrons exhibit (Butler 1992; Tauxe 2003). The three categories are: ferromagnetism, antiferromagnetism, and ferrimagnetism (Tauxe 2003). The term ferromagnetic can be used generally to refer to all three categories, but is used specifically to refer "to solids with parallel coupling of adjacent atomic magnetic moments" (figure 17a) (Butler 1992). Because of this parallel coupling they can "attain a very strong spontaneous magnetization, even in the absence of an external magnetic field" (Tarling 1983). The other two categories refer to materials that exhibit antiparallel coupling of atomic magnetic moments. This antiparallel magnetization "can be visualized as two separate magnetic lattices, each of which is magnetized in the opposite direction to the other" (Tarling 1983). Specifically, antiferromagnetic refers to materials where "the magnetization of the two lattices is exactly balanced," this results in no external magnetization and in turn no spontaneous magnetization (figure 17b) (Tarling 1983). On the other hand, ferrimagnetic refers to materials where "the two magnetic lattices are not exactly equal, so they have a spontaneous magnetization in the same way as ferromagnetic materials" (figure 17c) (Tarling 1983). However, the resulting magnetization is much weaker than that of ferromagnetic materials, as it is dependent on the difference between the two lattices (Tarling 1983).



**Figure 17.** Types of spin alignment/exchange coupling for a) ferromagnetic, b) antiferromagnetic, and c) ferrimagnetic materials. (Butler 1992).

Generally, materials that are referred to as "magnetic" are either ferromagnetic or ferrimagnetic (Tarling 1983). But there are obvious exceptions to this as one magnetic mineral that we encounter very often in archaeological contexts, hematite (a canted antiferromagnetic mineral), can acquire a remanent magnetization (Butler 1992, Tauxe et al. 2018). While mainly the materials of interest in paleo and archaeomagnetic studies are either ferro or ferrimagnetic, even antiferromagnetic materials due to specific circumstances (such as imperfect spin alignment due to canting, lattice defects, or impurities (figure 18)) can give rise to a weak magnetic moment (Tarling 1983; Thompson and Oldfield 1986). This exemplifies the complexity of magnetic materials. But the theory of spin alignment that is



**Figure 18.** Types of antiferromagnetic spin alignment: a) basic antiferromagnetism, b) spin-canted antiferromagnetism, c) defect antiferromagnetism. (Tauxe 2003).

important to know, state by Tauxe (2002), is "in crystals of [magnetic] minerals, quantum mechanical exchange interactions between neighboring atoms force all unpaired electron magnetic moments to align, resulting in a net spontaneous magnetization" (cited in Linford

2004). More specifically, within magnetic minerals spin alignments will occur in regions of the crystal know as magnetic domains (Linford 2004).

However, for the sake of simplicity, I will not go further in depth on this topic, but will simply leave the discussion at the overarching principle that all ferromagnetic materials have the ability to acquire a remanent magnetization. Although, it should be noted, how these materials acquire and retain a magnetization is a more complicated phenomenon then described here, as it deals with the complex crystalline structures and atomic interactions of these materials. (For more detail on ferromagnetism see Butler 1992 and Tarling 1983). Overall, it is important to at least understand the basics of magnetic mineralogy because "an essential part of every paleomagnetic [or archaeomagnetic] study is a discussion of what is carrying the magnetic remanence and how rocks [or other materials] got magnetized" (Tauxe et al. 2018). Thus, some more basics on the properties of magnetic minerals will be covered in the next section, but in reference to how they acquire a remanent magnetization.

# 2.6 Remanent magnetization

### 2.6.1 NRM

There are different types of processes that can result in a ferromagnetic material acquiring a remanent magnetization, but before delving into the specifics of these processes I would like to discuss in what state we find magnetic materials in because as archaeologists we work backwards in time. We find sites and artifacts after they are created, used, and subsequently buried. Same with the archaeological materials that are used for archaeomagnetic studies. We find and study them after they have gone through a remanence inducing event and have subsequently been buried for hundreds to thousands of years. So, we term the remanent magnetization present in a sample when it is procured from a site as natural remanent magnetization (NRM). In a sense, the NRM of a material can be equated to

the life history of an artifact, but in terms of its magnetization. Meaning that it is a record of all the magnetic processes that have acted upon and left a trace in the object throughout its history. More specifically, the NRM is all the remanent magnetizations present in a sample that is naturally occurring before any laboratory treatment or influence (Butler 1992; Tarling 1983).

Moreover, NRM is usually a combination of more than one type of magnetization; a primary and secondary NRM, which together total the natural remanent magnetization of a sample (Butler 1992). For rock samples the primary NRM is the remanent magnetization that is acquired during rock formation and is the component of interest for paleomagnetic investigations (Butler 1992). "*Secondary NRM* components can be acquired subsequent to rock formation and can alter or obscure primary NRM" (Butler 1992). It is virtually the same for archaeological materials. For example, the primary NRM for a baked clay structure such as a kiln would be the remanent magnetization acquired during the last firing of the kiln, or for a ceramic object it would be the magnetization acquired when it was fired during its manufacture (Tarling 1983). The secondary NRM would be the any subsequent events after the structure or object's last firing, including events during its deposition where it might have acquired additional magnetizations (Tarling 1983).

First and foremost, we are interested in the primary NRM, otherwise termed the characteristic remanent magnetization (ChRM), as it is the component we try to isolate in our sampled materials because it is the ChRM that gives us the values of the components of the Earth's geomagnetic field at a specific moment in time. There are many different types of remanent magnetizations a material can acquire (table 1). But primary NRM can be associated to one of three forms: thermoremanent magnetization, chemical remanent magnetization, detrital remanent magnetization (Butler 1992).

Table 1.	Types	of remanent	magnetizations.	(Tarling	1983).
				(	

Remanent type of magnetization	Abbreviation	Characteristic or definition			
Naturally occurring magnetizations Natural	NRM	Summation of all components of specimen remanence acquired by natural processes.			
Thermal	TRM	Acquired by cooling over a range of temperatures starting at or below the Curie temperature.			
Depositional/detrital	DRM	Acquired by the physical rotation of magnetic particles during deposition as a sediment.			
Post-depositional	PDRM	Acquired by sediments after deposition but prior to metamorphism or weathering; usually a combination of physical rotation of interstitial particles and chemical changes as sediments consolidate.			
Chemical	CRM	Acquired as a magnetic mineral nucleates and grows in a magnetic field.			
Shear	SRM	Acquired mostly by unconsolidated sediments when subjected to shear.			
Laboratory-induced magnetization Isothermal	IRM	Acquired by magnetic particles in steady magnetic field in a few seconds; the field is generally strong. This can also be acquired naturally in lightning strikes.			
Anhysteretic	ARM	Acquired when a ferromagnetic particle subjected simultaneously to alter- nating and direct magnetic fields.			
Rotational	RRM	Acquired by a specimen rotating within an alternating magnetic field. ) no d.c.			
Gyromagnetic	GRM	Acquired by a specimen in an alternating magnetic field without rotation. ) field			
Shock	SRM 🖊	Acquired by magnetic particles when shocked by impact. This can also occur naturally such as during meteoritic impacts.			

All 'natural' magnetizations can be duplicated, to varying extents, in the laboratory, and some 'laboratory' magnetizations may also occur in nature. In general, acronyms can be misleading, e.g. SRM has two meanings and their use is avoided in this book.

#### 2.6.2 Thermoremanent magnetization

As has already been pointed out, the primary magnetization we are trying to isolate in archaeological materials through laboratory analysis is most often a thermoremanent magnetization (TRM), thus the following discussion will solely focus on this form. A TRM is acquired when a ferromagnetic material cools "from above its Curie temperature (T<sub>C</sub>) in the presence of a magnetic field" (Butler 1992). To understand why this happens some characteristic properties of magnetic minerals need to be addressed.

Moreover, each ferromagnetic mineral can acquire a maximum magnetization, that is referred to as saturation magnetization,  $J_s$  (Butler 1992). The "saturation magnetization decreases with increasing temperature, becoming zero at the Curie temperature" (Butler 1992). Thus, above the  $T_c$  all substances behave paramagnetically, meaning they don't possess a magnetization as all there atomic magnetic moments are randomly orientated at these high temperatures (Tarling 1983). This is because "interatomic distance increases during thermal expansion" which results in the strength of exchange coupling and saturation magnetization to decrease (Butler 1992). Specifically at the Curie temperature of a given mineral "interatomic distances have increased to the point at which exchange coupling is destroyed" and so at high temperature ferromagnetic materials behave like paramagnetic materials where their atomic magnetic moments are independent from one another (Butler 1992). However, upon cooling they will regain their strong atomic interactions (i.e., exchange coupling) (Butler 1992). It is upon cooling that the magnetic minerals will align (i.e., acquire a remanent magnetization) with the ambient magnetic field (Figure 19). More specifically it is at the blocking temperature of the material that the TRM becomes locked into the material (Merrill 1996; Linford 2006). Otherwise, meaning that once a material cools past its blocking temperature, its magnetic moments are fixed and thus it retains a stable TRM that is parallel and proportional to the orientation and intensity of the magnetic field applied during cooling (Tauxe 2003; Tema et al. 2015). The blocking temperature "will depend on the precise mineralogical composition of the substance as well as its crystalline organization" (Linford 2006). All these properties are characteristic of a specific mineral as they depend on the atomic and crystal structures of the mineral. (See table 2 for an overview of some of the characteristic properties of various magnetic minerals). Furthermore, because we know the characteristic properties for magnetic minerals, we can use this knowledge to assess the magnetic mineralogy of a given sample.



**Figure 19.** Depiction of the acquisition of a thermoremanent magnetization. a) The magnetic grains/domains within the sample are initially magnetized in random direction that cancel each other out. b) and c) When a sample is heated the magnetic domains demagnetize when the temperature exceeds the materials blocking temperature. d) and e) upon cooling the magnetic domains within the material become magnetized again but in a direction that is close to the ambient field, which results in a net magnetization within the material that is parallel and proportional to the ambient field. (Linford 2006).

Mineral		$T_{\rm c} \left( T_{\rm n} \right) (^{\circ}{\rm C})$	J <sub>s</sub> (kA m <sup>-1</sup> )	<i>B</i> <sub>c</sub> (T)	
Oxides					
Magnetite	Fe <sub>3</sub> O <sub>4</sub>	575	480	0.3	
Maghemite	γ-Fe <sub>2</sub> O <sub>3</sub>	590 - 675	380	0.3	
Hematite	$\alpha$ -Fe <sub>2</sub> O <sub>3</sub>	675	2.5	1–5	
Goethite	α-FeOOH	60 - 130	2	>5	
Sulfides					
Pyrrhotite	FeS <sub>1+x</sub>	≤320	80	0.5–1	
Greigite	Fe <sub>3</sub> S <sub>4</sub>	330	125	0.03	

**Table 2.** Properties of common magnetic minerals.  $T_c(T_n) = Curie$  (Néel) Temperature;  $J_s =$  saturation magnetization;  $B_c =$  maximum coercivity. (Lanza and Meloni 2006).

#### 2.6.3 Secondary NRM

As mentioned, NRM is not only a primary (thermoremanent) magnetization, but can contain other magnetizations (secondary NRM) that can obscure the primary NRM. "Secondary NRM can result from chemical changes affecting ferromagnetic minerals [(CRM)], exposure to nearby lightning strikes [(naturally acquired IRM)], or long-term exposure to the geomagnetic field subsequent to rock formation [or subsequent to acquisition of TRM]" (Butler 1992). Like in geological conditions, "any chemical changes in archaeological materials that result in the growth of new magnetic minerals will result in their acquisition of a chemical remanence" (Tarling 1983). But despite the ability of archaeological materials to acquire a CRM it is rarely encountered. Tarling (1983) remarked, "the number of objects that come into this category are few and none have been investigated". As well, lightning strikes could contribute to the total NRM, but the most prevalent form of secondary NRM encountered in archaeological materials is the latter of the three mentioned, which is termed viscous remanent magnetization (VRM).

## 2.6.4 Viscous Remanent magnetization

VRM "is a remanent magnetization that is gradually acquired during exposure to weak magnetic fields" (Butler 1992). Furthermore, "all ferromagnetic particles left lying in the Earth's magnetic field will acquire a viscous remanent magnetization... that depends on the grain size and composition of the material, and on the duration and strength of the geomagnetic field" (Tarling 1983). Compared to geological materials, the VRM in archaeological materials is likely to be small due to the shorter duration of exposure to the geomagnetic field (Tarling 1983). However, even though small, the viscous components present in archaeological materials can still obscure or distort the primary component.

Moreover, VRM can be considered as a low stability component whereas the primary magnetization of a material is a high stability component. Because secondary components are of low stability, they can be easily removed in a laboratory setting, thus allowing for the isolation of the ChRM and the determination of the components of the past geomagnetic field. In the next chapter I will discuss the techniques employed to remove low stability components of magnetization in a sample in order to isolate its ChRM, as well as the techniques employed to assess the magnetic minerals carrying the remanent magnetization in a given sample.

# **Chapter 3: Archaeomagnetic method**

## **3.1 Principles of the archaeomagnetic method**

To reiterate, archaeomagnetism is a sub-discipline of geophysics that studies the magnetic remanence of archaeological materials for the purposes of studying the variations of the past geomagnetic field and for dating archaeological materials. (Thompson and Oldfield 1986). More specifically, through laboratory investigation it is possible to determine the components of the Earth's past geomagnetic field, both its direction and intensity, from archaeological materials. This is possible because of two physical phenomena. First, the intensity and direction of Earth's geomagnetic field fluctuates over time, and second, magnetic minerals can acquire a permanent (i.e., remanent) magnetization that preserves the past configuration the geomagnetic field. Because most archaeological materials (mainly clay artifacts and structures) contain magnetic minerals when they are heated to high temperatures and subsequently cooled in the presence of an ambient field, in this case the Earth's geomagnetic field, they will acquire a thermal remanent magnetization (TRM) that parallel and proportional to the geomagnetic field at that specific time and place (Tema et al. 2015). If

not heated again this magnetization will be fossilized within the material and "it would take a magnetic field of the strength of the earth some thousands, millions or even billions of years to change the magnetic alignment already acquired by [the] ferromagnetic particles" within the material (Tarling 1975).

Moreover, as clay artifacts are abundant in most archaeological context there are many opportunities to retrieve magnetic data. However, rarely, if ever, do we find clay artifacts in the last stage of production (i.e., in the kiln where they cooled after firing) and so the requirement of in situ material for directional determinations limits the materials that can be utilized. Consequently, this has lead researchers mainly to the remains of furnaces and kilns that have been used for pottery, lime, glass, and metal production for the investigation of the directional components of the past geomagnetic field (De Marco 2007). This is because these structures are typically made from, clay, tile, or bricks, all of which contain magnetic minerals (De Marco 2007). Another characteristic of these features that make them suitable targets for magnetic studies is that during their operation they reach temperatures above 700°C, which is above the Curie temperature of all remanence carrying minerals (De Marco 2007). But the archaeomagnetic method is not restricted to high fired materials. The remains of burnt clay from houses, soils, and floors, as well as, from domestic hearths and ovens can also be studied (De Marco 2007). These features tend to possess a weaker magnetization and the clay will be of lower quality, as they usually have not been exposed to such high temperatures; however, this does not prohibit the determination of their characteristic remanent magnetization (Aidona & Kondopoulou 2012; De Marco 2007). So, although the requirement of in situ material limits the materials available for directional determinations, there is still great potential within archaeological sites to investigate both the direction and intensity of the past geomagnetic field.

Overall, the archaeomagnetic method aims to isolate and identify the characteristic (primary) remanent magnetization in a material in order to determine its direction and intensity. Furthermore, when archaeological structures and artifacts have "been dated by reference to archaeological chronology, or by radiocarbon or thermoluminescence dating, there is the opportunity to obtain otherwise irretrievable geophysical information" (Aitken 1978). Meaning that if the archaeomagnetic method is applied to dated materials that carry a remanent magnetization it is possible to determine the past geomagnetic field components at that time. Thus, magnetic data from structures and artifacts that have been dated by other independent methods can be used to build secular variation curves. Once specific regions have well-established SVCs, covering archaeological interesting periods, magnetic data from materials of unknown age can be dated by comparison to the specific regional curves (Aitken 1978; De Marco et al. 2014).

#### **3.2** Archaeomagnetic fieldwork (sampling) and sample preparation

The first step in any archaeomagnetic study is to take samples of the material or structures from the site in question. Because sampling is required, the archaeomagnetic method can be considered semi-destructive, as after analysis the samples are left intact for further study (Artioli 2010). There are exceptions to this because the method involves various analytical techniques, some more destructive than others (e.g. demagnetization techniques, for isolating the ChRM, limits possible further measurements). Generally speaking, the archaeomagnetic method is semi-destructive as the samples are not fully consumed during measurement. Moreover, sampling is necessary because the methods for analyzing the ChRM to determine the direction and intensity of the past geomagnetic field from clay materials "requires precise control that is difficult to achieve successfully in the field" (Tarling 1983).

#### 3.2.1 Sampling and orientation

Samples are separately oriented pieces of baked clay from a structure or structures within an archaeological site. Specimens on the other hand, "are pieces of samples prepared to appropriate dimensions for measurement" (Butler 1992). Further it is common to prepare multiple specimens from an individual sample, while it is not a requirement, doing so provides additional checks on the homogeneity of the samples and the results of the experimental procedures (Butler 1992). A generalized sampling scheme is shown in figure 20.



**Figure 20.** Hierarchical chart showing a generalized sampling scheme for archaeomagnetic studies. Samples can be taken from various locations across an archaeological site, from a specific context within the site, or from a single feature (such as a kiln). Multiple samples are taken (the quantity differs from case to case but generally the minimum is 6 to 8). Each sample is them prepared in the laboratory into multiple specimens. (Adapted from the generalized paleomagnetic sampling scheme present in Butler (1992)).

Further, the sampling of archaeological materials for archaeomagnetic studies is done by trained professionals, i.e., specialists in the field of archaeomagnetism (De Marco 2007). Before sampling takes place the archaeomagnetist first gathers some preliminary information. This includes assessing the suitability of the material for archaeomagnetic study that is to be sample (Linford 2006). See table 3 for an overview of factors that influence the suitability. Along with the assessment of overall suitability, the context of the samples is documented, which includes a plan of the site and structure(s) to be sampled, location information of the site/structures (i.e., the geographical coordinates), and photographs and

All features	Is the feature still in the same position as it was when it acquired its remanence? This is a fundamental requirement of the archaeodirectional method. The feature should be inspected for crack that might indicate that it has moved or been disturbed since firing/deposition.	
	In the absence of cracking, has the feature slumped or lost its structural integrity?	
	If there is evidence for slumping (see Part 1, The mean remanent direction) can the feature be assumed to have been level originally?	
	Is it possible to estimate the direction and degree of movement (strike and dip)?	
	Is the feature free of bioturbation, eg tree roots, mole activity?	
	At a smaller scale, is the material comprising the feature still well consolidated? For instance, loose sand or soil may become friable and individual particles may have moved realigning their stored magnetic field directions.	
	Is the feature likely to have been in close proximity to ferrous material when it acquired its remanence, eg an iron-smelting furnace that cooled with slag left inside it?	
	This can distort the magnetic remanence recorded in the feature.	
Thermoremanent features	Has a suitably intense firing event taken place? (Usually determined by changes in the coloration of the fabric or magnetic susceptibility measurements.)	
	If the feature is composed of clay, has it been baked hard?	
Depositional remanent features	Is it likely that the sediment has settled out of solution in low-energy conditions so that a DRM can form?	

Table 3. Factors influencing the suitability of a feature for archaeomagnetic analysis. (Linford 2006).

drawings documenting sample locations (De Marco 2007, Linford 2006). Overall, many aspects regarding the nature and state of preservation of the materials or structures to be sampled needs to be considered for the archaeomagnetist to follow an optimal sampling scheme. Once the preliminary work of assessing and documenting the site and structures to be sampled is done, the actual sampling can take place.

Sampling procedures are varied due to the diversity of paleo and archaeomagnetic investigations (Tauxe et al. 2018). However, an essential requirement of any sampling method is that an accurate orientation is taken of each in situ sample (Aitken 1978). This is because the laboratory measurements will be made with respect to the specimen coordinate axes (Cartesian coordinates x,y,z – this will be further explained in the next section) that are determined by the orientation in field (Butler 1992). So, although no standard convention exists the overarching similarity across sampling methods is that "all orientation schemes are designed to provide an unambiguous in situ geographic orientation for each sample" (Butler

1992). Furthermore, the most employed sampling methods are either samples cored with a portable drill or block samples (Butler 1992). Block samples tend to be more widely used in archaeomagnetic studies. (The following discussion will focus on this type of sampling, giving a general view of the method used for the collection of samples used for this thesis. See butler 1992 and Tarling 1983 for more details regarding other methods.)

To take a block sample of baked clay an appropriately sized block (determined by the archaeomagnetist and archaeologist) is isolated from the surrounding material using knives, chisels, etc., but left intact at the base (Aitken 1978). Before it is completely removed its orientation must be determined. First a flat surface must be realized on the sample, this can be done by gluing a small Plexiglass plate to the surface, where a direction arrow can be placed (De Marco 2007; Linford 2004). Methods of orientation are also varied; however, archaeological samples are regularly oriented relative to true north (azimuth) and the local horizontal plane (dip). Moreover, if the attached plate is not horizontal, then an inclinometer is used to determine the dip of the surfaces slope (Linford 2006). See Parameter P2 (figure 21) for details on what the dip angle is. Then "an arrow must be marked onto it denoting an accurately established reference direction" (Linford 2006). In this case the reference direction is the azimuth (i.e. an arbitrary strike that is measured in degrees clockwise from true north, see Parameter P3 in figure 21), and can determined using either a sun compass or a gyrotheodolite (Linford 2004). Magnetic compasses can be used in some cases, but care should be taken when they are used because surrounding magnetic materials can affect a magnetic compass, thus resulting in an inaccurate reference direction (Linford 2006). Once the orientation is recorded and marked on the sample it can be fully removed from its context and transported to the laboratory.



Figure 21. Orientation parameters. (AGICO 2018).

### **3.3.2 Sample preparation**

When working with baked clays the first step is to consolidate the samples, this is required because such samples are usually quite fragile. Thus, to ensure that they do not fragment during sample preparation or during measurement they are treated with a consolidant, such as polyvinyl acetate in acetone or sodium silicate (Linford 2004). This is either done by submerging the whole sample in the consolidant (see De Marco 2007) or simply by pouring the solution onto the sample until it is visibly saturated. Once the samples are sufficiently consolidated, they are ready to be prepared into specimens.

The samples collected need to be sub-sampled because most instruments for measuring magnetic remanence (magnetometers) have strict requirements on the size and shape of samples that they can accommodate. Most instruments either accommodate small cylindrical or cubed samples. The magnetometer available at Aristotle University of Thessaloniki (AGICO JR-6) can accommodate cylindrical samples 2.5 cm in diameter and about 2 cm in length. To achieve these measurement parameters the block samples need to be drilled into cores and the cores cut to the appropriate length.

Before drilling the samples are first plastered into larger blocks, using Plexiglass frame molds and a mixture of gypsum plaster and water. These larger blocks allow for more control when drilling, less chance of fragmentation, as well as, allowing for as many cores to be taken from the material as possible. Further, to achieve this the samples are placed into the frame molds with the oriented surface at the top. The liquid plaster is then poured around the blocks and the top surface (oriented surface) is left exposed. While the plaster is still wet the samples are leveled within the plaster. Once the plastered samples are completely dry the field arrow is replaced by a laboratory arrow that is transcribed as a grid across the whole oriented surface. This is to ensure that when drilled into cores the sample's orientation will not be lost. The field arrow is the orientated direction marked on the sample in the field, but

"when the samples are prepared into specimens for measurement, the field arrow is often replaced by a *lab arrow* which is frequently in some other direction" (Tauxe et al. 2018). Further, it is by reference to the field arrow that we determine the sample coordinate system which is based off Cartesian coordinates (Figure 22a). This is important because it is by these coordinate axes that the specimens will be measured.



**Figure 22.** Sample coordinate system a), and orientation conventions for a cylindrical specimen b) and for a cube specimen c). a) represents a block sample where the field arrow is aligned to represent the y-axis and then in the laboratory it is replaced by a laboratory arrow the represents the x-axis, thus the laboratory arrow is a perpendicular arrow to the field arrow and points to the top of the sample if the field arrow is pointing to the right. a) (De Marco 2007). b) & c) (Tauxe et al. 2018).

The samples are then drilled into cores. At this stage the laboratory arrow is transcribed down the length of the cores in the same direction as the laboratory arrow on the original oriented surface. The direction into the outcrop in figure 22b represents the laboratory arrow drawn at this stage; this can also be visualized in figure 22c as the red arrow. Next, each core is cut into as many specimens as possible of appropriate length. Their orientation (laboratory) arrow is only on the length of the cylindrical specimens at this stage, but it needs to be transferred back to the top of each specimen (the surface that pertains to the original oriented surface). Once this is done each specimen is given a sub-sample code that is written on the specimen itself; this code denotes which sample the specimen belongs to and if multiple specimens were cut from the same core. If done correctly each specimen will look exactly like figure 22b. It will have a laboratory arrow on the top surface (representing the xaxis) as well as have the x-axis denoted down the length of the cylinder.

#### **3.4 Laboratory measurements**

The archaeomagnetic method is not simply the employment of one analytical technique that results in the determination of the past geomagnetic field components, but is a combination of techniques and measurements that together result in our ability to retrieve magnetic data from archaeological materials. As well, the methods vary depending on the purpose of the research, such that different methods are required to obtain either directional or intensity data. Furthermore, not only do we want to isolate the characteristic remanent magnetization of our samples, but the method also involves rock magnetic measurements so that we can assess the mineralogical composition and stability of our samples. In next sections, I will briefly cover the methodology employed for obtaining directional magnetic data from archaeological materials.

# 3.4.1 Initial measurement of NRM

The first measurement preformed in the laboratory is an initial measurement of the total remanent magnetization (NRM) present within the specimens; this is before any other treatment or measurement of the material is conducted. This is an important step because it gives the researcher a base measurement of the remanent magnetization the sample material possesses. Further, from these initial measurements it is possible to get an idea of possible secondary components obscuring the ChRM of the samples (Butler 1992). For archaeological materials, it is not expected that large secondary components (VRM) will be present. However, the distribution of initial NRM measurements give an idea on whether the samples were in situ. It also provides an opportunity to select the best possible specimens to be thermally demagnetized (i.e., those that are most likely to have a thermoremanent magnetization, as well as those most likely to provide accurate directions).

### **3.4.2 Rock magnetic measurements**

Apart from isolating the ChRM of the sample, an important part of paleo- and archaeomagnetic studies is an assessment of the magnetic minerals that carry the remanent magnetization (Aidona and Kondopoulou 2012). For paleomagnetic studies the identification the magnetic minerals present within rock samples "can help guide the design of partial demagnetization experiments and the interpretation of the results" (Butler 1992). However, for archaeomagnetic studies on clay materials we know more or less the magnetic minerals that might be present within our samples. Thus, the goal of rock magnetic measurements in this instance is to evaluate first if there are indeed magnetic minerals present that are capable of possessing a primary magnetization and second to evaluate the suitability/stability of the mineralogical composition of materials for obtaining reliable archaeomagnetic results (Aidona and Kondopoulou 2012).

The overall challenge of rock magnetic measurements "is to associate a particular component of NRM (identification from partial demagnetization) with a particular ferromagnetic mineral" (Butler 1992). This can help determine if the ChRM isolated within a sample is indeed the primary NRM. However, this determination is not always possible. In most cases what we hope to gain from rock magnetic measurements is not the specific identification of the magnetic minerals present within a sample, but an idea of the types present and the stability of those present so that we can say with confidence the directions determined from the ChRM that was isolated are accurate.

Moreover, "there are three families of techniques used to identify ferromagnetic minerals: (1) microscopy techniques including optical microscopy, electron microprobe and SEM; (2) determination of Curie temperature; and (3) coercivity spectrum analysis" (Butler 1992). With regards to the first group, the time and cost (particularly for the employment of SEM) associated with microscopy techniques make them out of reach for most studies.

Although these techniques are arguably more accurate in identifying specific minerals, no one technique is perfect; for example, optical microscopy and SEM have difficulties identify ferromagnetic minerals when they are in low concentration and just generally due to the small size of their grains (Butler 1992). So, while SEM "can provide pivotal information in particular cases," the latter two techniques are just as capable of providing a valid assessment of the overall mineralogical composition of various materials (Butler 1992).

Furthermore, because we know the characteristic properties of magnetic minerals, we can employ various techniques to determine the points or limits of these properties in order to determine which magnetic minerals are present within a sample. First, we can determine the curie temperature of the magnetic minerals present within a material from strong-field thermomagnetic analysis, where the magnetization of a sample exposed to a strong magnetic field (100mT) is monitored while temperature is increased (Butler 1992). Or more simply from thermomagnetic analysis, where the sample is heated to a high temperature (700°C) and then cooled to room temperature all while the magnetic susceptibility of the material is monitored. Both experiments allow for the determination of the curie point and give an overall idea of the behavior of the material during heating. From this we can infer which magnetic minerals are most likely present within a given sample.

Coercivity spectrum analysis (also termed acquisition of IRM) can also give us information on magnetic minerology. The analysis consists of inducing an isothermal remanent magnetization (IRM) in the sample by exposing it to a magnetizing field and then measuring the resulting IRM (Butler 1992). This procedure is repeated increasing the strength of the magnetizing field incrementally (Butler 1992). It allows us to identify at what point a sample becomes magnetically saturated. Further, because saturation magnetization is known for the various ferromagnetic minerals, the results allow us to determine if the sample consists of low- or high-coercivity magnetic minerals based on the point (at what magnetic

field strength) they become magnetically saturated. Overall, assessing the magnetic mineralogy of the sample, even if we cannot determine for certain the specific magnetic mineral (or combination of minerals) within a sample, these assessments provide valuable information that helps assess the reliability of the results of archaeomagnetic analyses.

#### **3.4.3 Isolation of the characteristic component of NRM**

The NRM in most cases, as has already been discussed, is usually a combination of more than one magnetic remanence: a primary NRM and a secondary NRM. It is the characteristic component of the NRM (primary NRM) that we try to isolate in the laboratory. Although relatively small, the secondary NRM within archaeological samples is typically a viscous remanent magnetization. The VRM, as well as most forms of secondary NRM, can be referred to as a low-stability component and can be easily removed from the sample, whereas the primary components of NRM (for archaeological materials this would be the thermal remanent magnetization) are more resistant and can be referred to as high-stability components (Butler 1992). Moreover, we employ demagnetization procedures in order to remove any low stability components from our sample and in turn to isolate the characteristic remanence so that it can then be measured, and its direction determined. Although, the secondary components in archaeological materials are small, we still need to employ methods that remove secondary components. This is to ensure that the directions obtained from the samples are accurate representations of the ChRM of the samples and in turn an accurate representation of the components of the geomagnetic field in the past.

There are three different demagnetization techniques that can be employed: alternating-field demagnetization, thermal demagnetization, and chemical demagnetization. This discussion will focus on thermal demagnetization, as this was the technique employed for this thesis. (See butler 1992 for details on the other two techniques). Specifically, thermal demagnetization "involves heating a specimen to an elevated temperature ( $T_{demag}$ ) below the

Curie temperature of the constituent ferromagnetic minerals, then cooling to room temperature in a zero magnetic field" (Butler 1992). This procedure results in any magnetic grains with blocking temperatures below the ( $T_{demag}$ ) to acquire a remanent magnetization that is equal to zero (i.e., there magnetizations will be randomized), thus in turn "erasing the NRM carried by these grains" (Butler 1992). When the NRM of the specimen is measured after the thermal demagnetization procedure, the grains with blocking temperatures above the  $T_{demag}$  are the ones contributed to the measured direction.

However, this method raises an important question; "what is the appropriate demagnetization level (T<sub>demag</sub>) for isolating the ChRM" (Butler 1992)? In most cases we can't be certain which demagnetization level will be appropriate for a specific specimen or group of specimens and so to overcome these uncertainties, a progressive thermal demagnetization technique can be employed. The procedure for this technique "is to sequentially demagnetize a specimen at progressively higher levels, measuring the remaining NRM following each demagnetization" (Butler 1992). The temperature steps for this procedure "are distributed between ambient temperature and the highest Curie temperature" (i.e., until the specimen(s) are fully demagnetized) (Butler 1992). The typical strategy utilizes temperature increases from 50 to 100°C for each step at lower temperatures and smaller temperature increases, in some cases only increasing by 5°C from one step to the next, when within 100°C of the Curie temperature (Butler 1992). The result of this technique "is a set of measurements of NRM remaining after increasing demagnetization levels" (Butler 1992). From these sets of measurements the directional components of the ChRM of each specimen can be determined, as well as the overall mean directions for the site from which the samples were collected.

### **3.5 Archaeomagnetic dating**

"Dating of archaeological remains is essential in archaeological research, in order to place in chronological order findings and civilizations...and often the contribution of a scientific dating technique is necessary" (Tema et al. 2015). Today there are multiple scientific dating methods that are available to archaeology, such as radiocarbon, obsidian hydration, dendrochronological, archaeomagnetic, and luminescence dating. "Each one of these dating techniques however has its own advantages and limitations... [and so] when possible, the combination of more dating techniques together with the available archaeological evidence may offer the best approach for obtaining a more precise chronological framework for an archaeological site" (Tema et al. 2015).

Furthermore, as mentioned briefly already, archaeomagnetic dating is the oldest geophysical-geochemical technique available to date archaeological materials (Tarling 1975). However, as we have seen archaeomagnetic research was not widespread until the latter half of the 20<sup>th</sup> century. While its slow start can be attributed to various reasons, once improvements came in our understanding of the geomagnetic field of the Earth and in technology and instrumentation the technique still didn't gain popularity like other dating techniques have over the last few decades. Even today, discussions surrounding archaeometry rarely mention archaeomagnetism as a technique available to archaeologists. This may be because the gap between the two contributing disciplines, archaeology and geophysics, has still not quite been fully bridged. Generally, when we think of dating methods in archaeology we either think of relative dating methods, such as ceramic sequencing (typology dating), or the absolute dating methods of radiocarbon and thermoluminescence. There are valid reasons for why we immediately turn to these methods, such that they have provided reliable results within the field of archaeology for decades. Moreover, we can pinpoint why archaeomagnetic dating has not gained widespread use like these methods.

In the 1970s when archaeomagnetism was gaining more widespread application, its employment as a dating technique was still very limited. At this time it was still "a difficult technique because most archaeological materials contain complex magnetizations, only one of which may be related to a precise, dateable time," and the theory and techniques for accurately isolating the characteristic component were still in their infancy (Tarling 1975). Furthermore, the archaeomagnetic dating method consist of comparing "measured directions of unknown age with dated variations of the geomagnetic field" (Tarling 1975). This also further complicates this dating method. Whereas other dating methods, such as radiocarbon dating, can by employed independently from any previous study on the materials the archaeomagnetic dating method is a circular method. (Tarling 1975). Meaning "the geomagnetic variations themselves can only be defined by previous archaeomagnetic observations which have been dated by other techniques, such as by  $C^{14}$  dating, thereby compounding the errors in both techniques" (Tarling 1975).

More specifically, the technique is reliant on materials that have been magnetized at a specific time in the past and that have retained this magnetization, so that when measured in the present the obtained direction and intensity of their magnetization can provide a record of the past geomagnetic field at that specific time (Tarling 1975). Further, if we have a dated record of the variations of the past geomagnetic field (i.e., reliable secular variation curves), only then can we use this record to date archaeological materials (Tarling 1975). Now a days there are reliable secular variation curves for several regions around the world that allow archaeomagnetic dating to be employed with great success. However, as is evident with the purpose of this thesis there is still a lack of adequate archaeomagnetic data for various regions and periods that do not yet allow for the utilization of archaeomagnetic dating in many cases.

# Chapter 4: Archaeomagnetic Investigation of baked clay from the Neolithic site of Koutroulou Magoula in Thessaly, Greece

# 4.1 Introduction

To accomplish the goal of gaining more data points for the directional secular variation curves for Greece, baked clay from the Neolithic site Koutroulou Magoula was studied. As mentioned previously, for the study and identification of the directional components of the Earth's geomagnetic field in situ material is need. This condition is the main reason we see more intensity data as opposed to directional data for Greece. Although pottery is ubiquitous in the Greek environment and makes up the majority of site assemblages it is never found still in production. While this is not an issue for studies of intensity, to obtain directional data we need to turn to other materials, such as sediments, kilns, or baked clay. Neolithic sites, generally, present excellent conditions that facilitate archaeomagnetic investigations, especially when it comes to investigating archaeomagnetic directions and the need for in situ material.

More specifically, the characteristics of Neolithic settlements in Greece present ideal conditions for archaeomagnetic research because during the Neolithic period in SE Europe populations engaged in the deliberate social practice of house burning (Aidona & Kondopoulou 2012). This practice lasted for about 1500 years during the Neolithic period and can be seen throughout the Balkan peninsula, thus resulting in an abundance of burnt material that can be studied (Aidona & Kondopoulou 2012). Furthermore, "it has been found that clay constitutes 90% of the construction material" in these Neolithic contexts, as well not one site has been recorded in the region with completely unburnt remains (Aidona & Kondopoulou 2012). Also, the spatial distribution of Neolithic occupational contexts is an attractive characteristic for archaeomagnetic studies. This is because, in Greece, Neolithic sites generally have shorter periods of occupation and tend to have horizontal rather than

vertical extensions (Aidona and Kondopoulou 2012). This feature allows for a certain level of confidence when sampling Neolithic sites for archaeomagnetic studies, as it is more likely that different samples from various structures from the same site will all correspond to the same occupation level, or at least be relatively close in date. Overall, the Neolithic period in the greater Balkan region, including Greece, consists of a considerable quantity of potential sites that exhibit ideal conditions for archaeomagnetic studies. (Aidona & Kondopoulou 2012). But despite their potential only a few archaeomagnetic studies have been conducted for these contexts. Thus, the goal of this thesis is to take advantage of the conditions of the Koutroulou Magoula, to obtain the much-needed archaeodirectional data for the Neolithic period, to enrich the Greek directional SVCs.

# 4.2 Site description

Koutroulou Magoula, is a Middle Neolithic tell site located near the southwestern edge of the Thessalian Plain (figure 23) (Hamilakis et al. 2017). Systematic archaeological work at this site started in 2001, directed by Kyparissi-Apostolika, with various seasons



**Figure 23.** Location of the Koutroulou Magoula in central Greece on the southwestern boundary of the Thessalian plain. Right (Hamilakis et al. 2012). Left (https://blogs.brown.edu/koutrouloumagoula/the-site/)

continuing until 2009 (Hamilakis et al. 2017). "Formally since 2010 work at the site has continued as part of the Koutroulou Magoula Archaeology and Archaeological Ethnography Project" (Hamilakis et al. 2017). The project is a collaborative effort between the Greek Archaeological Service (the Ephorate of Antiquities of Phthiotida and Evrytania and the Ephorate of Palaeoanthropology and Speleology) and the University of South Hampton (under the auspices of the British School at Athens); co-directed by Kyparissi-Apostolika and Hamilakis (Hamilakis et al. 2017). (See <a href="https://blogs.brown.edu/koutrouloumagoula/">https://blogs.brown.edu/koutrouloumagoula/</a> for more details on the research goals of the project).

Moreover, Koutroulou Magoula is part of the vast panorama of Neolithic settlements/sites in Greece (Aidona and Kondopoulou 2012; Hamilakis et al. 2017). Today, there are more one thousand Neolithic sites that have been discovered and/or partly excavated (Aidona and Kondopoulou 2012). Specifically, Koutroulou Magoula is one of many Neolithic tells in central Greece (figure 24) (Hamilakis et al. 2017). However, the Koutroulou Magoula stand out from the rest in the region as it is much larger than the typical size



**Figure 24.** Distribution of Neolithic sites in central Greece. (Hamilakis et al. 2017).

observed of tell sites in Greece. It is 206 m long, 182 m wide, and stands 6.6 m above the surround landscape (Hamilakis et al. 2017). The overall extent of cultural activity covers an area of ca. 3.7 ha, whereas other tell sites in Greece are rarely over 2 ha in area (Hamilakis et al. 2017).

Moreover, excavation has revealed various architectural remains at the top of the tell as well as on the eastern slope that, based on conventional pottery chronology, date to the Middle Neolithic (Hamilakis et al. 2017; Ifantidis 2021). However, half of the context of this large site has been lost due to agricultural activity (figure 25) (Hamilakis et al. 2017). This is a shame as survey work revealed that that there was more extensive activity in the western part of the site when compared to the eastern part (Hamilakis et al. 2017). Despite this the site still offers rich cultural contexts, "characterized by unique elaboration and preservation" (Hamilakis et al. 2017). Further, the majority of the work over the years has been concentrated on investigating the top of the site, but in 2017 the excavation was extended on to the eastern slope, where three trenches were opened (figure 26) (Bennet 2018).



**Figure 25.** Topography of Koutroulou Magoula. The leveling of the western half, due to agricultural activity, is visible here. (Hamilakis et al. 2017).



**Figure 26.** Plan of the tell showing the trenches excavated at the top of the site and the location of the three trenches ( $\pm 15$ , X16,  $\Psi 21$ ) opened on the eastern slope during the 2017 season. (Bennet 2018).

Moreover, the site has provided a rich array of artifacts. However, the main interest for this thesis is in the architectural remains and building phases, along with their state of preservation and proposed dates as the proposed date will be used to date our archaeomagnetic results. First, I will discuss the buildings that have been unearthed at the top of the tell as these contexts have been radiocarbon dated.

At the top of the tell a large habitation site has been revealed, which includes two rectangular buildings and several others partially uncovered (figure 27) (Hamilakis et al. 2017). Building 1, unearthed during the first campaign, shows two of three building phases that seem to have been built atop one another (figure 28) (Hamilakis et al. 2017). Thus, one of the goals of the new project was to clarify the chronological association between various features, such as the phases of building 1 (Hamilakis et al. 2017). The various building phases



**Figure 27.** Site plan of the main excavation area at the top of the koutroulou Magoula (progress made by the end of the 2015 excavation season). (Hamilakis et al. 2017).



Figure 28. The architectural phases of Building 1. (Left) Hamilakis et al. 2012. (Right) Hamilakis et al. 2017.

identified, not just in building 1, but throughout the site exhibit different construction techniques (see Hamilakis et al. 2017 for details). Building 2's construction was revealed to consist of stone foundations that seem to have been covered by upright clay slabs (figure 29) (Hamilakis et al. 2017). As well "during excavation extensive layers of burned clay were



Figure 29. Building 2, showing the clay slab that cover the stone foundations of the walls. (Hamilakis et al. 2017).noted on the top of the floor and the walls" (Hamilakis et al. 2017). The southern wall of this room is missing, and it is suggested by the archaeologists that this might suggest "a non-

habitational space, possibly communal in nature;" while the extensive burning suggests it may have been deliberately destroyed by fire (Hamilakis et al. 2017). Thus far, the cultural layers reach 2.5 m deep, however cores taken in 2012 revealed that the cultural layers extend at least 5 meters below the surface of the tell (Hamilakis et al. 2017).

The depth of the cultural layers and evidence of various building phases seem to suggest a longer occupation of the of the site, in contrast to the general trend seen of Neolithic settlements. However, the project carried out AMS radiocarbon dating to help clarify the dates of the main (Neolithic) occupational phase at the site, as there is also evidence for its use as a burial site in the Late Bronze Age and in medieval times. Moreover, seven samples "were dated by AMS in the Centre for Isotope Research of the University of Groningen" (Hamilakis et al. 2017). All samples, excluding the sample that came from a disturbed context (Sample #GrA-60924), where there was evidence of bronze age activity, gave dates within the first two centuries of the sixth millennium B.C. (table 4) (Hamilakis et al. 2017). What the archaeologists marked as interesting from these results was that the

Table 4. AMS dates from the Koutroulou Magoula (Hamilakis et al. 2017).

Lab Nr	Location	Date of Unearthing	Depth below Surface (cm)	<sup>14</sup> C Age (B.P.)	Calibrated Date (cal B.C.)	Probabilities (%)
GrA-60924	Trench H3 (Extension), NK285, Context 048/01, base of tholos tomb	9/26/2012	55.0	$3745 \pm 40$	2206–2045 2286–2032	68.2 95.4
GrA-60921	Trench Ø3, Context 36/23	9/17/2010	84.0	6930 ± 45	5869–5743 5967–5723	68.2 95.4
GrA-60916	Trench $\Theta$ 2, Pass IB Square $\Gamma$ 46, A. $\Delta$ .2, Orientation: 130 cm north x 280 cm east	11/7/2005	63.0	6960 ± 45	5894–5776 5977–5738	68.2 95.4
GrA-60918	Trench H, Pass IA, Square A38, OM κ.π. 288γ	5/30/2002	128.3	$6990 \pm 45$	5976–5812 5984–5764	68.2 95.4
GrA-60919	Trench Ø4, Context 721/073	9/22/2011	18.0	704 <b>0</b> ± 45	5986–5891 6014–5814	68.2 95.4
GrA-61065	Trench @3, Context 008/02	9/9/2009	74.0	$7050 \pm 45$	5987–5899 6016–5841	68.2 95.4
GrA-60912	Trench @1, Boring KTLC-1	9/2012	500.0	7095 ± 45	6020–5916 6055–5891	68.2 95.4

average time difference between the deepest sample taken from a depth of 5 m (Sample #GrA-60912) to the sample taken from only 20 cm (Sample #GrA-60919) from the surface is only 150 – 170 years (Hamilakis et al. 2017). Thus, the overall depth of the site does not reflect a long period of occupation, on the contrary in fits the generally model seen where most Neolithic sites do not exhibit evidence of long periods of occupation. Furthermore, regarding the AMS dates and the dates suggested by pottery typology, there is disagreement between the two methods. The dates suggested by pottery typology places the site a few centuries later than the AMS dates. The archaeologists suggested that this disparity does not suggest a miscalculation on the part of the radiocarbon dating method, but rather an issue in the conventional dating (pottery-typological dating) for the Greek Neolithic.

Now turning to was uncovered on the eastern slope; I will focus on trench  $\pm 15$ , as this is where the samples for this thesis were collected from. Moreover, initial investigations of trench  $\pm 15$  "revealed rich and extensive activity, including complete ceramic vessels (rare on the site), a concentration of quern stones and a possible hearth" (Bennet 2018). Continued work in this area in 2018 and 2019 clarified that in fact the possible hearth, Feature 2 (figure 30) uncovered in 2017, were the remains of a pottery kiln (Bennet 2020). Specifically in



Figure 30. Feature 2 – Ceramic kiln located in trench E15. (Bennet 2019a).

2019, extensive excavation of Feature 2 revealed the kiln's floor and the fill removed to expose it, although heavily burned clay, was rich in cultural material (Bennet 2020). Its floor and parts of its wall were very well preserved, and the excavators were able to fully explore the extent of the feature concluding that it was indeed a kiln of a rounded trapezoid shape, measuring about 1m across with its opening to the south (Bennet 2019a; Bennet 2020). At the kilns southeastern corner, a circular fired object was found suggesting a possible platform or working surface (Bennet 2020). The excavators suggest that there may have been other kilns/ovens in the southern half of the space, but they were not able to clearly identify them

(Bennet 2020). However, the overall conclusion is that the area contained a kiln complex (Bennet 2020). The work on the eastern slop is not fully published yet, so there is a lack of data available regarding the dating of the area. Thus, I am going off what the AMS radiocarbon dates from the contexts on the top of the tell revealed. Further, because the sample from various depths at the top of the site are close in date and the fact that the overall Neolithic occupation of the site in the reports is given a date of the middle Neolithic, I fell it is valid to assume, at this moment in time, that the Neolithic contexts on the slope fall within the first two centuries of the 6<sup>th</sup> millennium (i.e., the date range provide by AMS radiocarbon dating).

### 4.3 Field work (sampling)

The archaeomagnetic sampling of the material from the site was conducted by Elina Aidona PhD. All together 9 block samples of baked/burnt clay were taken from various locations within Trench  $\pm 15$ , the kiln complex on the eastern slope. Each sample was separately oriented in reference to true north (Azimuth) and the local horizontal plane (dip), following the procedures described in section 3.2.1. It's clear why this context was chosen for archaeomagnetic analysis as the whole area is constructed from clay. Further, due to the use of the space as a kiln complex and an abundance of burnt material encountered during the excavation, trench  $\pm 15$  seems to have been sufficiently burned in the past. Thus, providing ideal circumstances for archaeomagnetic investigation. Figures 31 and 32 show the whole trench and figure 33 shows the individual samples taken from the area.



Figure 31. Trench  $\Xi 15$  from the south.



Figure 32. Trench  $\pm 15$  from the northwest.


**Figure 33.** Individual sampling locations. a) sample #KOU1. b) sample #KOU2. c) sample #KOU3. d) sample #KOU4. e) sample #KOU6. f) sample #KOU7.



**Figure 33 cont.** g) sample #KOU8. h) sample #KOU9. i) imaging showing the location of sample #KOU9 on the southern edge of the kiln (Feature 2).

# 4.4 Sample preparation

The sample were prepared in the geology laboratory at Aristotle University of Thessaloniki, by the author. Out of the 9 samples that were collected from the site, 8 were prepared into specimens for measurement (figure 34). Samples #Kou 6 was left out due to



Figure 34. All eight block samples before sample preparation.

poor quality. Each sample was first consolidated using a stone hardener. Then, each sample was plastered (with a mixture of powdered gypsum and water) using Plexiglass frame molds (figure 35). After the plaster had fully dried the laboratory arrow was transcribed across the



**Figure 35.** Process employed for plastering the block samples. a) The sample was placed in the Plexiglass mold with the oriented surface facing up and the field arrow point to the right (representing the y-axis of the sample coordinate system). b) The plaster was then poured and the sampled was leveled in the plaster. c) Once the plaster thickened slightly it was removed from the mold and left out to dry.

whole surface of each sample, in reference to the field orientations, following the method outlined in section 3.3.2 (figure 36).



**Figure 36.** Procedure for replacing the field arrow with a laboratory arrow. a) Before the orientation plate (field orientation) was removed from the sample, using a ruler a parallel line to the field arrow was drawn across the top of the block. b) Then the Plexiglass plate was removed (although not shown here) and again using a ruler perpendicular lines were drawn across the whole surface of the sample. These perpendicular lines represent the laboratory arrow and the x-axis of the sample coordinate system, so they need a direction as well. If the field arrow points to the right, then the laboratory arrow will point to the top of the block. c) The final result will look like this.

Once this was complete each sample was carefully drilled, perpendicular to the

oriented surface, into cores (figure 37). After all possible cores were drilled, the laboratory

arrow was transcribed down the length of the cores (figure 38). Each core was then cut into as

many specimens measuring 2 cm in length, using a wet circular saw. Once again, the

laboratory arrow was transcribed back to the top surface (the surface that corresponded to the

original oriented surface) of each core and each was given a specific specimen number/code



**Figure 37.** Drilling the cores. Using a diamond core drill, the cores were drilled perpendicular to the oriented surface. This process is time consuming as the slower the cores are drilled the less chance there is that the cores will be broken during this process.



**Figure 38.** Images showing the laboratory arrow (x-axis) on the surface of the cores and transcribed down the length of the cores after drilling. It is important to follow the direction of the laboratory arrow on the top surface so that the orientation has the right position and direction down the length of the core.

that indicated from which sample they came from and if they came from the same core

(figure 39). Overall, from 8 samples a total of 49 specimens were prepared.



**Figure 39.** Process of drawing the final lab arrow and labeling of the specimens. (Bottom) Images shows the complete specimens that are ready for analysis.

### 4.5 Methodology – Laboratory techniques/measurements

During sample collection of in situ material the archaeomagnetist takes great precaution in assessing the material, to ensure that the best quality samples are taken. Meaning that they assess the state of preservation of the material, carefully watching for signs of displacement. However, the most careful sampling methods, doesn't ensure that the samples retrieved will be in situ. Thus, the first measurements taken in the laboratory when investigating directional data is to determine if the sample were in fact in situ. This is done by measuring the NRM of all the specimens and comparing the results. Therefore, the NRM all specimens (n=49) were measured using an AGICO JR-6 spinner magnetometer. These results were accessed before moving forward with the rock magnetic measurements and demagnetization procedures.

To access the magnetic minerology of the samples two analyses were preformed: thermomagnetic analysis and coercivity spectrum analysis (acquisition of IRM). More specifically, the high-temperature behavior of magnetic susceptibility (thermomagnetic analysis) for the determination of the Curie points of the magnetic minerals present in the material was preformed, using a Bartington susceptibility meter with a furnace attached, on six samples (a small portion from the original block samples were powdered to perform this measurement). Changes in their magnetic susceptibility were recorded continuously from room temperature up to 700°C and back down. As well, to identify at what point the samples become magnetically saturated, which provided further information on the composition of magnetic minerals in the sample, coercivity spectrum analysis was conducted. This allows for the determination of either low- or high-coercivity magnetic components within a material. For this analysis an isothermal remanent magnetization (IRM) was imparted on six specimens with an impulse magnetizer (ASC scientific model IM-10-30) to a maximum field of 1200 mT or 2500 mT depending on the specimen. This was done incrementally and after

each step the magnetic remanence (IRM) of each specimen was measured with the AGICO JR-6 spinner magnetometer, thus allowing for stepwise acquisition curves of IRM to be obtained for each specimen.

Furthermore, for this thesis, progressive thermal demagnetization was employed using a MMTD-80 (Magnetic Measurements, UK) furnace. From the initial NRM measurements of the 49 specimens 20 specimens were selected for thermal demagnetization. The heating strategy employed was composed of 12 temperature steps, from 100°C to 580°C. The remanent magnetization and magnetic susceptibility of each specimen was measured after each step with the AGICO JR-6 spinner magnetometer and the Bartington susceptibility meter. Measurements on susceptibility during thermal demagnetization procedures provide another check on the magnetic stability of the specimens, specifically regarding the behavior of the individual specimens subject to thermal demagnetization. This is necessary because although the other rock magnetic measurements give an overall idea regarding the magnetic minerology of the samples that is helpful in accessing the reliability of the results, the specific material analyzed for those measurements are not the specimens that are then subject to the thermal demagnetization procedure.

## **Chapter 5: Results**

### 5.1 NRM results

The majority of the NRM directions of all the specimens grouped satisfactorily (figure 40). Although some outliers were present, the results suggest that the samples were in fact in situ. However, to investigate the deviations from the overall grouping of some of the specimens, three specimens from the outliers were thermally demagnetized. This was to see if there would be any improvement in their directions as there may be secondary NRM

components obscuring their characteristic remanent magnetization (These results will be discussed later on).



**Figure 40.** Stereographic projection of the NRM distribution of all 49 specimens. Dots represent specimen measurements (full dots correspond to positive inclinations and open dots indicate negative inclinations). Dots circled in red are the outliers that were rejected. Arrows indicate the outliers that were subjected to thermal demagnetization. The mean NRM directions (excluding the outliers, n=38) are D [°] = 4.0, and I[°] =59.6; with  $\alpha_{95}$  [°] = 4.0.

### **5.2 Rock magnetic results**

## 5.2.1 Thermomagnetic analysis

For the determination of the Curie points and to see if any mineralogical changes occur during heating of the samples (i.e., analysis of their thermochemical stability), thermomagnetic analysis was conducted on 6 samples. Samples #KOU1 and #KOU2 were not subject to this analysis as all specimens from both samples were rejected after thermal demagnetization and thus not part of the calculation of the mean directions for the site. Overall, as seen in figure 41, the thermomagnetic curves show good reversibility indicating that there are no significant changes in mineralogy at these temperatures. The Curie points were determined using the second derivative method and vary between 550°C and 610°C (table 5).



**Figure 41.** Thermomagnetic curves showing the variation of magnetic susceptibility with temperature. (a) KOU 3, (b) KOU 4, (c) KOU 5, (d) KOU 7, (e) KOU 8, and (f) KOU 9.

Table 5. Curie points of each sample determined from thermomagnetic analysis.

Sample	KOU 3	KOU 4	KOU5	KOU 7	KOU 8	KOU 9
Curie Point	608°C	584°C	593°C	554°C	568°C	562°C

#### 5.2.2 Coercivity spectrum analysis results

To further understand elements of the mineralogy of the samples, specifically if they contain low- or high-coercivity magnetic minerals IRM acquisition curves were obtain for representative specimens from 6 out of the 8 samples (Figure 42). Overall, the analysis showed that the majority of magnetization in all the specimens was acquired in low fields (up to 300 mT), thus suggesting a prevalence of low-coercivity magnetic minerals within the sampled material. Specifically, the IRM curves for the specimens from samples KOU2, KOU3, and KOU5 (Figure 42a, b, c) show a dominance of low-coercivity magnetic minerals, as all three specimens have acquired a large percentage of their magnetization in fields up 300 mT. On the other hand, the IRM curves for the other three specimens from samples KOU7, KOU8, and KOU9 (Figure 42d, e, f) suggest the presence of both low- and highcoercivity magnetic minerals as they rapidly acquire a majority of there IRM up to 300mT, but then this is followed by a gradual acquisition of additional IRM. These sample were still not fully saturated at a field strength of 2.5T. But, because of the limitation of the impulse magnetizer (the coils available in the laboratory for the instrument were only capable of reaching a maximum field strength of 2.5T) it was not possible to fully saturate these specimens. However, it is still possible to assess the presence of low- versus high-coercivity minerals at these limits. Overall, since they acquired the majority of their magnetization in low fields (up to 300 mT) the majority of magnetic minerals present in these samples are lowcoercivity components, but a small portion of high-coercivity components is also present, as they were not fully statured at high fields (up to 2500 mT). Furthermore, there seems to be two distinct groups (Group1 = KOU2, KOU3, and KOU5; Group2 = KOU7, KOU8, and



KOU9) when looking at the overall shape of the plots suggesting that at least in terms of coercivity the mineralogical compositions of the samples in each group are very similar.

**Figure 42.** IRM acquisition curves. (a) KOU 2-1; (b) KOU 3-3a; (c) KOU 5-2b; (d) KOU 7-1b; (e) KOU 8-3a; (f) KOU 9-3.

# **5.3 Archaeodirectional results**

Based off the initial NRM measurements 20 specimens (consisting of 2 to 3 specimens from each sample) were selected for thermal demagnetization, including three specimens from the outliers. First, no abnormal behavior in magnetic susceptibility was observed as the sample were heated during the demagnetization procedure (figure 43). Further, as seen in figure 4,3 there are only small changes in magnetic susceptibility throughout the experiment, thus suggesting excellent magnetic stability of the specimens that were thermally demagnetized.



**Figure 43.** Variation of magnetic susceptibility of all 20 specimens during thermal demagnetization procedure.

Thermal demagnetization for the majority of the specimens revealed the presence of one characteristic component (Figure 44). In cases where a secondary viscous component could be recognized, it was removed at low temperatures. However, thermal demagnetization did not improve the direction of the outliers (figure 45). Similarly, all specimens from sample KOU 1 and KOU2 (and one specimen from KOU 8) were not consistent with the overall grouping of the characteristic remanence obtained after thermal demagnetization, and so they were excluded from the calculation of the mean directions reducing the number of successful specimens to 14. Further, all specimens were totally demagnetized up to 580°C.



**Figure 44.** Zijderveld diagrams (left), stereographic plots (center), and thermal demagnetization curves of one specimen from each sample. a) KOU1-1A, b) KOU2-2A, c) KOU3-1A.



Figure 44 cont. d) KOU4-1, e) KOU5-1A, 7) KOU7-1A.



Figure 44 cont. g) KOU8-3B, and h) KOU9-1.



**Figure 45.** Stereographic projection showing the distribution of the ChRM of the 20 specimens thermally demagnetized. Those circled in red indicate the specimens that were rejected for the final calculation of the mean directions. The arrows indicate the specimens that were outliers after the initial NRM measurements. As can be seen here thermal demagnetization did not improve their directions.

The directions of the characteristic remanent magnetization (ChRM) of each specimen were calculated by preforming principal component analysis (Butler 1992) and was done within Remasoft software. The ChRM of each specimen was determined using at least 5 demagnetization steps, and the obtained directions yielded small maximum angular deviation (MAD) values lower than 3° in all cases (except for the rejected specimens which in some cases had much larger MAD values) (table 6). Overall, from the ChRM of 14 specimens, that represent 6 samples, the mean directions for the site were obtained and are present in figure 46 and in table 7.

Specimen	<b>D</b> [°]	I [°]	MAD
KOU1-1A*	36.6	-79.1	2.7
KOU1-2A*	191.6	-49.6	1.6
KOU1-2B*	334.2	37.4	2.8
KOU2-2A*	285.3	11.1	7.9
KOU2-3*	315.8	56.5	1.7
KOU3-1A	14.2	60.1	1.7
KOU3-1B	17.5	58.4	2.7
KOU3-2A	12.7	56.1	1.3
KOU4-1	4.5	52.7	1.1
KOU4-2B	25.8	48.6	0.7
KOU4-3B	26	50.8	0.7
KOU5-1A	8.8	53.2	2.5
KOU5-2A	350.3	53.8	1.3
KOU7-1A	358.5	53.8	0.8
KOU7-2A	346.6	57.9	0.8
KOU7-3A	351	55.1	1.3
KOU8-1A	12.5	45.7	2.8
KOU8-3B*	333.2	8.9	4
KOU9-1	355.5	60.6	1.2
KOU9-2	6	67.5	1.8

**Table 6.** Directions of the ChRM of each specimen determined after thermal demagnetization using principal component analysis. Asterisks (\*) indicate specimens that were reject from the final mean directions determined for the site.



**Figure 46.** Stereographic projection showing the distribution of the ChRM for the specimens (n=14) used to calculate the mean directional components. The mean values are  $D [^{\circ}] = 6.9$  and  $I [^{\circ}] = 55.9$ .

**Table 7.** Mean ChRM direction for the studied site. N – number of specimens contributing to the calculation of the direction; D, I – averaged declination and inclination of the site;  $\alpha_{95}$  – semi-angle of the confidence's cone; k – precision parameter.

Site	N	<i>D</i> [°]	<i>I</i> [°]	α <sub>95</sub> [°]	k
Koutroulou Magoula	14	6.9	55.9	4.5	79.11

### **Chapter 6: Discussion**

### 6.1 Overview of results

Based on the rock magnetic measurements we observe that the magnetic mineralogy of the studied samples exhibits good stability overall. Further, based on the determination of the Curie point and the fact that all specimens were fully demagnetized at 580°C the sample most likely contain magnetite-like magnetic minerals. The IRM acquisition curves also suggest a presence of magnetite. Although we cannot be certain of the specific minerals carrying the ChRM based on these experiments alone, we can infer on the most likely minerals present. Moreover, the characteristic patterns observed in the IRM acquisition curves suggest that the majority of the magnetic minerals within the sample are of lowcoercivity type, either magnetite or titanomagnetite. The specimens that were not fully saturated up to 2.5T seem to suggest a typical mixture of low- and high-coercivity magnetic minerals. In these cases, these corresponding specimens also contained a majority of lowcoercivity minerals, but they do possess a small portion of high-coercivity minerals. The mixture is most likely composed of mainly magnetite (or titanomagnetite) with a small percentage of hematite.

Furthermore, thermal demagnetization proved to be successful for the majority of the specimens. The rejected specimens after thermal demagnetization (all specimens from samples KOU1, KOU2, and the one specimen from KOU8) could have various reasons for which a characteristic remanence failed to be determined. For example, the negative values for the inclination observed in some of the specimens is not a result that would be found in archaeological contexts. Such negative inclinations cannot be observed for the periods covered by archaeological findings of the last 8Kyrs, so it is evident that there is an error of some type here. For example, in one case (KOU1-2A) it seemed as though the directions were accurate just that they were inverted. Overall, for samples KOU 1 and KOU 2 the variability in the results after thermal demagnetization, and the complete rejection of each sample may suggest a possible error in sampling or an error during sample preparation. However, the most likely cause is that they were probably not sufficiently heated to high enough temperatures in the past. This is also probably the case for sample KOU 8, where all but one specimen was rejected after the initial NRM measurements and thermal demagnetization. During sample preparation of KOU8, specifically during the drilling process, the cores were very fragile and only half of the cores drilled came out intact. Thus, I would say that the results of KOU 8, or lack thereof favorable result, may be due to difficulties during sample preparation, or there could have been portions of the sample well

fired while other parts were not exposed to high temperatures. However, despite the unfavorable results observed in some of the samples, the remaining samples/specimens showed favorable results. From the ChRM of the remaining specimens the calculation of the ChRM mean directions for the site was possible. Overall, the mean directions for the site were calculated from the ChRM of 14 specimens that represent 6 samples.

Furthermore, the purpose of this thesis was to obtain the directional data from the studied material so that the results could then be included into the Greek directional SVCs. However, as mentioned in order to be able to construct, or in this case add to, a secular variation curve the contexts from which the studied material comes from must be dated first by other methods. So, while the directional data were achieved from the samples, on further examination of the archaeological information for the site it was clear that a specific date could not be confidently given to the context of the samples. Moreover, the Neolithic occupation of the site has been given an overall date based on pottery chronology as Middle Neolithic (ca. 5800- 5300 BC), whereas the AMS radiocarbon dating from the contexts at the top of the site date these contexts to the first two centuries of the 6<sup>th</sup> millennium (ca. 6000 – 5800 BC). However, the samples studied for this thesis come from the recently unearthed contexts on the eastern slope of the site. While these contexts do belong to the main Neolithic occupation of the site, they have only recently been excavated and there is not enough published data pertaining to these contexts for a date to be confidently suggested. So, it was decided that archaeomagnetic dating would be conducted on the studied samples.

### **6.2** Archaeomagnetic dating

The archaeomagnetic dating of the samples from Koutroulou Magoula was preformed using the 'Archaeo\_dating' Matlab tool by Pavon-Carrasco et al. (2011). The dating was based on the Balkan reference curve (Tema and Kondopoulou 2011) and the Directional Neolithic curve (Carrancho et al. 2013). The dating is performed independently for Declination and Inclination values calculating the probability density of the two components. In the end of the procedure a combined probability density is proposed as a final dating of the site. (See figures 47 and 48).



**Figure 47.** Archaeomagnetic dating of the site using the Balkan reference curve. The dating interval obtained separately for declination and inclination, are combined to calculate the dating interval at 95% probability level.



**Figure 48.** Archaeomagnetic dating of the site using the Neolithic reference curve. The dating interval obtained separately for declination and inclination, are combined to calculate the dating interval at 95% probability level.

Furthermore, the combination of declination and inclination probability densities in reference to both the Balkan and Neolithic reference curves give similar date ranges for the site (Figure 47 and 48). The obtained date interval for the site based on the Balkan reference curve is 5463 – 5200 BC, and the date interval obtained from the Neolithic reference curve is 5488 – 5187 BC both at a 95% confidence level. While these results show good agreement in the dating intervals obtained, the archaeomagnetic dating does not correspond to either the AMS radiocarbon dating or the archaeological dating of the site. AMS radiocarbon dating places the site within the first two centuries of the 6<sup>th</sup> millennium BC in the transitional period between the Early and Middle Neolithic, whereas the pottery typology places the Neolithic occupation of site a few centuries later, within the Middle Neolithic period. Overall, the archaeologists, based on the AMS dates of samples from various depths within the cultural

layers, suggest that the Neolithic occupation of the site was short-lived (Hamilakis et al. 2017). However, the archaeomagnetic dating suggests a later date than both the AMS and pottery typology dating. The archaeomagnetic dating places the contexts of the samples closer to the transitional period between the Middle Neolithic (ca. 5800 - 5300 BC) and the Late Neolithic 1 (ca. 5300 - 4800 BC).

However, the archaeomagnetic dating results are not in complete conflict with the other dates as the context of the samples, the eastern slope of the site, has not been radiocarbon dated. Also, it has only been recently excavated and so further information on the archaeological chronology of this area could possibly agree with the archaeomagnetic dating. Although each dating method for the site has given different dates, I do not believe one method invalidates the others. At the moment, it seems that the Neolithic occupation of the site lasted longer than previously believed. One piece of evidence from the excavation that supports the later date of the contexts on the eastern slope is a spear point made of honey chert found in trench  $\pm 14$  (the area directly next to the kiln complex, trench  $\pm 15$ , where the samples are from). Initial reports suggest it is probably a Late Neolithic spear point, most likely originating from present-day Bulgaria (Bennet 2019b). I don't want to speculate too much, as I don't know enough about the site and Neolithic sites in general, but the fact that it is a larger tell site than typically observed seems to support the idea of a longer occupation. As well, we must remember that the event we are dating through archaeomagnetic analysis is either the last firing within the kiln complex or a fire destruction phase/abandonment of the area. So, the archaeomagnetic dating most likely represents the end date of the Neolithic occupation of the site.

## Conclusion

The objective of this thesis was to obtain archaeomagnetic directional data from the Neolithic tell site Koutroulou Magoula to provide additional data for the Neolithic period in Greece for the purpose of enriching the Greek directional SVCs. The study was successful as we were able to isolate and determine the directional components of the past geomagnetic field within the studied materials and in turn, we were able obtain the mean directions for the samples and the site. The obtained mean directions proved to be reliable throughout the study. More specifically the samples showed good magnetic stability, one characteristic component of magnetization was successfully determined for the majority of the specimens subject to thermally demagnetization, and the confidence  $[\alpha 95]$  and precision [k] parameters for the grouping of the obtain directions of the specimens were within the limits of what is considered trustworthy values. Furthermore, based on the successful results of the experimental procedures the archaeomagnetic dating intervals obtained from the comparison of the mean directions of the site to both the Balkan and Neolithic reference curves are promising. More specifically, the fact that both curves provided similar dates, both at a 95% confidence interval, further suggests that the final archaeomagnetic dating of the samples/site are trustworthy.

Although, the archaeomagnetic dating does not match up with the radiocarbon dates obtained for the contexts located at the top of the tell (the proposed archaeomagnetic dates place the site about 400 years later), the radiocarbon dates do not invalidate our results. First, because the radiocarbon dating and archaeomagnetic dating were conducted in separate locations and second, the archaeomagnetic date obtained most likely represents last phase of the area (either its last use or its destruction). As well, the archaeological evidence seems to supports a later date. The general archaeological chronology suggested for the site (Middle Neolithic ca. 5800 – 5300 BC) is closer to our proposed date. Similarly, the spear point found

on the eastern slope, that was suggested to date to the late Neolithic period, further supports the accuracy of the archaeomagnetic date. Overall, we were able to provide another method of dating for site that I hope will help provide a more precise chronological framework for the main Neolithic occupation of Koutroulou Magoula.

#### **Further research**

Although, a date independent from the archaeomagnetic method could not be given to the sampled materials, once a precise archaeological chronology is obtained for the contexts on the eastern slope of the site or further radiometric dating is conducted for the context of our samples the archaeodirections obtain during this study can then be included into the Greek directional SVCs. Moreover, the success of this study is a step in the right direction for the overall goal of achieving more archaeodirectional data that covers the Greek Neolithic period. Also the successful results have showed that there is future potential in obtaining accurate archaeomagnetic data specifically at Koutroulou Magoula and more genrally from other Neolithic sites in Greece. Regarding the specific samples utilized for this study, the next step would be to conduct an archaeomagnetic intensity study on the same samples, so that the total geomagnetic field vector for the site can be define. This would increase the accuracy of the archaeomagnetic date obtained for the site and eventually provide additional data for the Greek SVCs.

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