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Bachelor of Geology

# VARIATIONS OF THE GEOMAGNETIC FIELD'S INTENSITY DURING THE MIDDLE AND LATE BRONZE ERA

MSc Thesis

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#### ELISAVET-GEORGIA T. KYRIAKIDOU Bachelor of Geology

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#### SUMMARY

The combination of geophysical methods with archaeology has grown to become a very important research tool, used in studies both of geological and archaeological impact. A branch of the geophysical methods which combines geomagnetism with archaeology is called *Archaeomagnetism* and contributes to the study and the dating of materials which, at some point of their history, have been burned and cooled down. These materials need to contain magnetic minerals, inside of which is stored information about the changes of the magnetic field of the Earth through years. This is also a very important aspect of archaeomagnetism.

The following research is the analytical archaeomagnetic study of samples from two different areas in Greece, Archontiko in Central Macedonia and the Phryctoriae in Pediada of Crete. For a better comprehension of this thesis, the most important terms are given, along with the basic definitions about the magnetic field of the Earth. Further information and the basic principles of the archaeomagnetic method are explained, such as the types of magnetic minerals and how each one stores the magnetic properties of the geomagnetic field at the time of their last heating and cooling. A very important aspect for an archaeomagnetic study is the secular variation curves, that depict the changes of the geomagnetic field of a chosen area through time. Even though there are several archaeomagnetic studies taking place in Greece, there are still parts of the Greek secular variation curve that require more studies for completion.

Some basic archaeological information is given, regarding the origins and the usage of the samples, which are ceramic fragments from the area of Archontiko used as house utilities and baked clays from the Cretan Phryctoriae, which are used as communication system. Samples from both areas have previously been dated through other methods, radiometric and archaeological, which placed them in the Late Bronze Era.

A brief description about the methodology and the measurements of the conducted experiments is following. Those are three rock magnetic experiments (IRM, Thermomagnetic Curves and Hysteresis Loops) and the palaeointensity experiment (Thellier-Thellier). The resulting diagrams are cited, along with a quick interpretation, followed by a more detailed discussion on each experiment and diagram separately, in the last chapter. The rock magnetic experiments provided details about the magnetic consistency of the samples, while the palaeointensity experiment gave information about the geomagnetic field's properties, at the moment when these samples were last burned and cooled down, as it was "stored" within the samples. The results from the palaeointensity experiment were added in the Greek SVC, which was then compared to some neighboring SVCs.

Ψηφιακή συλλογή Βιβλιοθήκη



#### ΠΕΡΙΛΗΨΗ

Ο συνδυασμός των γεωφυσικών μεθόδων με την αρχαιολογία έχει εξελιχθεί σε ένα πολύ σημαντικό ερευνητικό εργαλείο, το οποίο χρησιμοποιείται τόσο στη γεωλογική όσο και στην αρχαιολογική έρευνα. Ο κλάδος της γεωφυσικής που χρησιμοποιεί τον γεωμαγνητισμό στην αρχαιολογική μελέτη ονομάζεται *Αρχαιομαγνητισμός* και συμβάλει στη μελέτη και χρονολόγηση υλικών, τα οποία κάποια στιγμή κάηκαν και έπειτα ψύχθηκαν. Αυτά τα υλικά πρέπει να περιέχουν στη σύσταση τους μαγνητικά ορυκτά, μέσα στα οποία αποθηκεύονται οι πληροφορίες σχετικά με τις αλλαγές του μαγνητικού πεδίου της Γης ανά τους αιώνες. Αυτή είναι και μια πολύ σημαντική πλευρά του αρχαιομαγνητισμού.

Η παρούσα έρευνα αφορά στην αρχαιομαγνητική μελέτη δειγμάτων από δυο διαφορετικές περιοχές της Ελλάδας, από το Αρχοντικό στη Κεντρική Μακεδονία και από τις Φρυκτωρίες στη Πεδιάδα της Κρήτης. Με σκοπό την καλύτερη κατανόηση της εργασίας, δίνονται κάποιες σημαντικές γνώσεις θεωρίας, μαζί με τους βασικούς ορισμούς σχετικά με το μαγνητικό πεδίο της Γης. Εξηγούνται περισσότερες πληροφορίες και οι βασικές αρχές της αρχαιομαγνητικής μεθόδου, όπως τα είδη των μαγνητικών ορυκτών και τον τρόπο με τον οποίο το καθένα αποθηκεύει τη μαγνητική πληροφορία κατά τη διάρκεια της καύσης και ψύξης του. Στην αρχαιομαγνητική έρευνα, πολύ σημαντικές είναι οι Καμπύλες Αιώνιας Μεταβολής, που απεικονίζουν τις αλλαγές του γεωμαγνητικού πεδίου σε μια επιλεγόμενη περιοχή με το πέρασμα του χρόνου. Αν και έχουν πραγματοποιηθεί πολλές αρχαιομαγνητικές έρευνες στον ελληνικό χώρο, υπάρχουν πολλά τμήματα της ελληνικής καμπύλης τα οποία χρειάζονται περισσότερα στοιχεία για να δώσουν μια πιο ολοκληρωμένη εικόνα.

Ακολουθούν κάποιες βασικές πληροφορίες σχετικά με την αρχαιολογική έρευνα, σχετικά με την προέλευση και τη χρήση των δειγμάτων, τα οποία είναι θραύσματα αγγείων από

την περιοχή του Αρχοντικού, που χρησιμοποιούνταν ως χρηστικά σκεύη, και ψημένη άργιλος από τις Φρυκτωρίες της Κρήτης, οι οποίες αποτελούσαν ένα ιδιαίτερο σύστημα επικοινωνίας. Δείγματα και από τις δυο περιοχές έχουν προηγουμένως χρονολογηθεί μέσω διαφόρων μεθόδων και τοποθετήθηκαν στην Ύστερη Εποχή του Χαλκού.

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Ακολουθεί μια σύντομη περιγραφή σχετικά με τη μεθοδολογία και τις μετρήσεις των πειραμάτων. Αρχικά διεξήχθησαν πειράματα μαγνητικής ορυκτολογίας (Ισοθερμικής Παραμένουσας Μαγνήτισης, Θερμομαγνητικές Καμπύλες και Βρόγχοι Υστέρησης) και ακολούθησε το πείραμα παλαιοέντασης (Thellier-Thellier). Στην συνέχεια παραθέτονται τα διαγράμματα που προέκυψαν, μαζί με μια σύντομη ερμηνεία, και το τελευταίο κεφάλαιο αποτελείται μια πιο λεπτομερή συζήτηση, σχετικά με τα αποτελέσματα κάθε πειράματος χωριστά. Από τα πειράματα μαγνητικής ορυκτολογίας, μελετήθηκαν και υπολογίστηκαν οι μαγνητικές ιδιότητες των δειγμάτων, ενώ το πείραμα της παλαιοέντασης παρείχε πληροφορίες όσον αφορά στην ένταση του μαγνητικού πεδίου της Γης που επικρατούσε εκείνη την εποχή και αποθηκεύτηκε μέσα στα δείγματα, τη στιγμή που αυτά κάηκαν και ψύχθηκαν για τελευταία φορά. Τα αποτελέσματα του πειράματος της παλαιοέντασης προστέθηκαν στην ελληνική καμπύλη αιώνιας μεταβολής (SVC), η οποία μετά συγκρίθηκε με άλλες γειτονικές καμπύλες.



#### INTRODUCTION

It is very common for scientists of all disciplines to combine different types of knowledge, means and expertise, throughout the years. This assists in achieving the best possible results for an experiment or research, while new fields are formed and named. Such a discipline is archaeomagnetism, which combines and uses physical and geophysical methods in the field of archaeology. These methods provide us with important information about the structure of the sample's origin and the parameters of the magnetic field that were recorded in it during its last heating and subsequent cooling. Additionally, it helps scientists to collect the needed information and understand the evolution of the Earth's magnetic field throughout times.

The present dissertation focuses on the conduction of magnetic experiments on samples taken from two different areas of Greece, from Central Macedonia and Crete. The most important parameter for these experiments is the *remanent magnetisation*, that has been recorded in previously burned materials, such as the baked clays used for the experiments of this thesis. The purpose of this procedure is to find details about the geomagnetic field's state, at the moment the samples were burned and cooled for the last time.

At the beginning of this thesis, a few details about the magnetic field of the Earth are given, to present a clearer background as to what we based our research methods on. It is then explained how baked clay materials get magnetised, since this is a major factor of the research. The types of magnetisation an element can acquire are also mentioned.

Then, more information about the archaeomagnetic method are presented, from the method's basic principles, to the way samples are collected and the required characteristics of these samples. The most important part is how the remanent magnetisation is captured inside the sample during its last cooldown, after a high

temperature heating, and aims to the creation of the Secular Variation Curves, which indicate the magnetic field's changes through time. The second chapter closes with a state -of the art- for the archaeomagnetic studies in Greece.

Ψηφιακή συλλογή Βιβλιοθήκη

The next chapter deals with the areas from which the samples were retrieved including both archaeological and some geographical information, that can help with the conclusions in the end. These areas are Archontiko, a village inhabited until today near Giannitsa, and central Crete, where a very interesting communication system was found, which used big structures, called Phryctoriae.

Continuing, details about the way the samples were processed and the experiments that were conducted for the purposes of this thesis, are explained. In total, five magnetic experiments took place, four of which are rock magnetism experiments and the fifth being the Thellier experiment.

Lastly, the results for each area are presented, explained and checked to see if they are in agreement with each other. The successful correlation between them is giving a more certain final conclusion. BIβλιοθήκη GEOΦΡΑΣΤΟΣ" Chapter 1: The Magnetic Field of the Earth A.Π.Θ

The *magnetic field of the Earth* is the space around Earth where the magnetic forces are applied. The study of the magnetic field is very valuable, since it helps us to explore the activity taking place in the deeper layers of our planet (such as the movement in Earth's core), not only in recent years, but also through the geological past of the Earth. In addition, on a more profitable area, the geomagnetic elements assist in finding mineral deposits and oilfields.

Magnetism, the force linked to the magnetic field, was observed as a property in the early historic years, since we find it being mentioned in Greek scripts, dated at the 6<sup>th</sup> BC, while the first magnetic compass was created more than 1000 years ago, in China.

### 1.1 The Magnetic Field and Magnetic Elements of the Earth

The strength or magnetic intensity of a magnet is expressed by the **magnetic moment**. The result of the magnetic moment per unit of volume or weight (A/m or Am<sup>2</sup>/kg respectively) on an object or material is known as **magnetisation**.

Not all materials are affected by the magnetic forces when placed inside the magnetic field. The objects on which the magnetic forces apply are called **magnetic materials**. The **magnetic value** (also referred to as **magnitude**), **m**, is analogue to the magnetic forces and can be positive or negative. To change the direction of the magnetisation, a new magnetic field has to be applied, known as **coercivity**.

In addition, the magnetic material shows a directional variation of the magnetic properties, based on the magnetic forces that apply on it and also on its own structure. This phenomenon is called **magnetic anisotropy**.

There are regions in a magnetic material, where walls are formed to separate the parallel magnetic moments of the atoms, minimising its potential magnetic energy. These regions

are called **magnetic domains**. There are two types of domain behavior, depending on the mineral and size of the crystal. The **single domain behavior** appears when the entire grain is magnetised in the same direction, while when the mineral consists of many domains magnetised in various directions, we deal with **multidomain behavior**. Some grains with single domain behavior, but who are also a bit too large to be single domain grains, are called **pseudo-single domain**. **Grains** are the sum of crystals that together create formations of various shapes and in macroscopic sizes. A crystalline mineral usually consists of many such crystals, whose atoms are placed on the same grid.

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As mentioned before, different magnetic materials acquire different magnetisation in a magnetic field. The value that expresses this ability is the **magnetic susceptibility** and, like most of the magnetic properties, it also shows directional anisotropy.

When the magnetic field passes from a material through another with different magnetic properties, it refracts in a manner similar to that of light rays. The **magnetic refraction** is the deviation of its direction from one material to the other.

When a magnetic moment is measured while in an inducing magnetic field, then the magnetisation is called **induced magnetisation**. It consists of the **remanent magnetisation** and the **transient magnetisation**. When the magnetic field is removed, the remanent magnetisation remains, while the transient magnetisation is nullified.

The source of the Earth's magnetic field existence is still a subject of debate. It is accepted, however, that it is closely linked to the outer core, a layer composed mostly of nickel and iron, 3000m beneath the surface of our planet. Generally, it is believed that the geomagnetic field is produced by the movement of the free electrons in that layer, while the rotating motion of the Earth, the gravity and the thermodynamics turn it to a dynamo that preserves itself.

The Earth's magnetic field is mainly represented by a dipole field, similar to that of a common magnet. The magnet's axis passes through the center of the Earth and so it is known as a geocentric axial dipole (*figure 1.1 a*). Regardless of this, the two poles do not completely coincide with the geographical north and south poles, since there is a small deviation of 11.5° from the rotational axis (*figure 1.1 b*). This places the geomagnetic north pole in Canada and the south in Antarctica and needs to be taken into account when using a magnetic compass.

Ψηφιακή συλλογή Βιβλιοθήκη



*Figure 1.1:* Visualisation of Earth's dipole magnetic field. (a) The electric circular movements in the outer core (red area) produce the geomagnetic field is generated, while the intermittent lines path the way of the field currents from the south to the north pole (Linford, 2004). (b) Comparison of the geographical and magnetic poles and equators, with the inclination between them. (Butler, 1992) (Both figures from DeMarco, 2007)

The deviation, of course, also applies on the geomagnetic equator in relation to the geographical equator.

As shown in *figure 1.1*, the geomagnetic field is depicted in circular lines that move from the south to the north pole. Although they begin with a direction parallel to the Earth's surface, their path moves away from or into the ground as they reach the magnetic poles. The orientation of the geomagnetic vector, on any single spot of the Earth's exterior layer, is defined basically by two characteristics, the direction and the intensity, both in correlation with the geographic poles (*figure 1.2*). The direction is described by two angles. The first is the **Declination (D)**, which represents the angle between the geographic north pole and the horizontal part of the geomagnetic vector and the second is the **Inclination (I)**, the angle between the horizontal component and the whole field vectors and is positive when the z-axis is downwards.

Ψηφιακή συλλογή Βιβλιοθήκη



**Figure 1.2**: Visual presentation of the magnetic field components, on the cartesian coordinate system (X, Y, Z). (H): Vector of Horizontal Intensity | (Z): Vector of Vertical Intensity | (F): Total Field Intensity | (I): Inclination | (D): Declination The axi correspond to (X): North | (Y): East | (Z): Down

Vector norm represents the **Intensity of the field (F)** and the size of the magnetisation that the magnetic minerals will obtain. In addition, it shows the power with which the needle of a compass will be aligned accordingly to the magnetic poles. The intensity is

measured in micro-Tesla ( $\mu$ T), as established in the International System of Units (SI). These seven parameters (*figure 1.2*) are called **geomagnetic elements**.

Ψηφιακή συλλογή

In a simplified way, it was mentioned that the Earth's magnetic field is a dipole field. In reality though, this is a bit more complicated, since there are also more limited, nondipole magnetic fields that add to the main magnetic field. These fields are causing changes to the inclination, declination and magnitude through space and often interact with each other. They can be generated by a variety of sources, triggered by movement, expansion and decay.

There are two different types of sources, the internal and the external. From the total measurements, the **internal sources** take over the most part, with the most important one being the *Main Magnetic Field*, that reflects on more than 90% of the measured field. It is related to the outer core and is described by the Secular Variation Curves. Another internal source is the *Anomaly Field*, which comes from the rocks in the planet's crust, corresponding to 1-2% of the total field. Lastly, around 1% is caused by the **external sources**, the small, more temporary fields, generated in the ionised upper parts of the atmosphere, due to the flow of electric currents there. To provide the complete picture of the actual geomagnetic field, all these parameters have to be taken into account.

Even though the geomagnetic field changes through time, the main magnetic field stays practically stable for a period of a few years' time. Its intensity is calculated as the average of the geomagnetic field's intensity for a certain amount of time, measured by several stations.



*Figure 1.3:* World Declination Map for 2010 (image from <u>http://www.geomag.bgs.ac.uk/data\_service/services.htm</u>)

Each region and country have their own geomagnetic models, but it is still possible for global geomagnetic models, known as **magnetic reference field models**, to be created. These magnetic field models depict the alterations of the geomagnetic field's global distribution and variations with time, providing separate information for both the dipole and non-dipole part of the field, always in accordance with a global spherical, harmonical analysis. The most well-known magnetic reference field models are the **International Geomagnetic Reference Model (IGRF)** and the **World Magnetic Model (WMM)** *(figure 1.3)*.

#### 1.2 Magnetisation of Raw Material

Generally, the minerals that have the ability to be magnetised are found all around us, depending on the conditions their magnetisation can be permanent. According to their

ability to get magnetised, the materials are divided in **three main categories**: diamagnetic, paramagnetic and ferromagnetic.

Ψηφιακή συλλογή Βιβλιοθήκη

A **magnetic field H** is produced by an electric current, and furthermore, the orbit of the electrons around the nucleus and themselves. When a material is exposed to a magnetic field, it acquires an induced magnetisation M<sub>I</sub>, that is expressed as:

$$M_l = \chi H$$

where  $\chi$  is known as **magnetic susceptibility** and represents the level of a material's magnetisation after it is exposed to a magnetic field. This function applies in cases where the M<sub>I</sub> is parallel to H. For anisotropic material the susceptibility  $\chi$  is a 3x3 matrix.

In **diamagnetic material**, the M<sub>I</sub> is small and it nullifies when the magnetic field it has been exposed to is removed and the susceptibility is small, negative and practically unrelated to the temperature. Generally, all materials can act as diamagnetic, since they get triggered when an external magnetic field applies on the orbit of an electron. So, the magnetic moment adjusts to oppose this applied field, by changing. Minerals and materials that often show diamagnetism are quartz, calcite, water etc.

Some solids show magnetic moments on an atomic level, even without an applied magnetic field. The solids with such ability are called **paramagmetic**. Their atoms have a random orientation for their spin, while the atomic moments are positioned in such a way that the produced magnetisation is zero. As the material is exposed to a field H, the spins are placed parallel to it and into lines, the magnetic energy decreases and the obtained magnetisation is the product of the field and the paramagnetic susceptibility. When the field is removed, the magnetisation nullifies again. Some of the most common materials to show paramagnetic behaviour are the pyroxenes, the amphiboles, olivine, biotite and the pyrite and siderite.



Figure 1.4: (a) Magnetisation, J, versus magnetising field, H, for a diamagnetic substance. Magnetic susceptibility, χ, is a negative constant. (b) J versus H for a paramagnetic substance. Magnetic susceptibility, χ, is a positive constant. (c) J versus H for a ferromagnetic substance. The path of magnetisation exhibits hysteresis (is irreversible), and magnetic susceptibility, χ, is not a simple constant (Butler, 2004)

There are some solids that, when there is no field applying on them, they produce one themselves. They are known as **ferromagnetic solids** and this type of activity is called **ferromagnetism**.

Thanks to the remanent magnetisation, these solids show atomic magnetic moments that have intense interactions with the other nearby atomic moments. This is opposite to the paramagnetic atomic spins that do not interact with each other and the magnetisation of ferromagnetic solids can be much larger even in the same magnetic fields.

The ferromagnetics are very important for palaeomagnetic studies, as they can "store" the magnetic field properties and even the direction of a magnetic field. The expression of the magnetic susceptibility is more complicated than for the other categories of solids, because of the **hysteresis loops**, which are the way the magnetisation, M, connects to the applied field, H.



**Figure 1.5**: Alignment in the Ferromagnetic minerals a) ferromagnetic | b-c) antiferromagnetic | d) ferrimagnetic (Figure from Tauxe, 2005)

There are three types of ferromagnetic minerals, based on their crystal structure which affects their spin alignment and therefor the total magnetic energy:

- I. ferromagnetism (sensu stricto)
- II. antiferromagnetism:
- III. ferrimagnetism

The atomic spins in the **ferromagnetic** solids are aligned parallel to their neighboring spins. When all atomic moments are parallel, the exchange energy is minimum, while the Ms is stronger (e.g., pure iron). On the contrary, in the antiferromagnetism and ferrimagnetism, the spins are aligned in layers, but each layer is anti-parallel to the other *(figure 1.5)*.

More specifically, in **antiferromagnetic** material, sometimes each layer cancels the other having equivalent magnetic moments, resulting in  $M_s=0$  (e.g., ilmenite). In other cases, their spins are not perfectly aligned, having a small inclination of a few degrees, or showing some small structural defects, giving space for the appearance of a weak net moment (e.g., hematite or fine-grained hematite respectively). The moment from which the antiferromagnetic solid's atomic placement is distorted and turns into a paramagnetic, is known as the **Néel temperature** (TN).

The most important category though is the **ferrimagnetic** materials, where uneven magnetic moments align in anti-parallel layers, hence producing a net magnetic moment, M<sub>s</sub>, with direction towards the stronger layer (e.g., magnetite). They can react to even weaker magnetic fields, recording them into their magnetic domains.

The minerals more often found in the samples subjected to archaeomagnetic studies are the oxides of iron, with magnetite being of major importance, followed by maghemite and hematite. The oxyhydroxides of iron and the ferrosulfides are also common. The colour of clays is affected by the environment in which the iron-oxide grains are being heated. They are red if the environment was oxidising or greyscale if it was antioxidant.

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**Figure 1.6:** Mineralogic composition of ferromagnetic minerals. In this diagram are the names and the chemical consistency of the most important FeTi-oxides. The arrows indicate the direction of oxide increasement and the lines represent the series of titanomagnetite and titanohematite (Figure from Tauxe, 2005).

A mineral of high interest is **magnetite** ( $Fe_3O_4$ ), a dark-coloured mineral, with saturation magnetisation at 50-150 mT. Its frequent appearance in samples and strong magnetic properties have established it as the most important ferromagnetic mineral. It is anisotropic and the magnitude of coercivity increases inversely to its size and Curie Temperature.

Another important ferromagnetic mineral is **maghemite** ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>), which turns into hematite when the temperature surpasses the 250-300 °C (*e.g., Tauxe, 200*5). Because of

this, the Curie temperature ranges between 590 to 675 °C. It is created because of magnetite oxidation in lower temperatures, as product of erosions and sometimes from lepidocrocite after it loses its water.

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Lastly, an antiferromagnetic mineral which is useful in such a research is **hematite** ( $\alpha$ -**Fe**<sub>2</sub>**O**<sub>3</sub>**).** It often co-exists, in both igneous and metamorphic rocks, with the strongly magnetised titanomagnetite, making hematite's contribution in the rock's magnetic properties insignificant. Nonetheless, it has a high coercivity and from ferromagnetic, it becomes paramagnetic once its Néel temperature of 750 °C is surpassed.

Minerals	Chemical Formula	Curie/ <u>Néel</u> Temperatures (°C)	Coercivity (mT)
Magnetite	Fe₃O₄	575-585	10-50
Maghemite	γ-Fe₂O₃	~600	-
Hematite	α-Fe₂O₃	675	>1000
Titanomagnetite 60%	xFe₃O₄(1-x)	150	8
Pyrrhotite	Fe <sub>7</sub> S₃	~320	100-500
Greigite	Fe₃S₄	~333	60-100
Ilmenite	FeTiO₃	-233	-

**Table 1.1:** Table with some important ferromagnetic minerals, their chemical formulas, the Curie/Neel temperature and their typical coercivities (based on the Table 2.1 of DeMarco, 2007)

Other minerals with palaeomagnetic interest are titanomagnetites, with a Curie temperature ranging from -150 to 580°C, pyrrhotite with a Curie temperature at around 333°C and greigite, with a Curie temperature at 320 °C.

The mineral with the lowest coercivity out of these is the titanomagnetite, while hematite has the highest. Their coercivity is heavily affected by the size of their grains and their concentration of Ti (*Table 1.1*).

The **Natural Remanent Magnetisation (NRM)**, is the remanent magnetisation present in a sample prior to laboratory treatment. NRM typically is composed of one or more types of magnetisation depending on the history of the sample. The NRM component acquired during the rock formation is termed "primary". However, secondary NRM components can be acquired subsequently to rock formation and can alter or obscure primary NRM. The NRM is then the vector sum of all the naturally acquired components of magnetisation.

Βιβλιοθήκη

1.3 Types of Magnetisation

The primary NRM appears in three types of magnetisation: the **Thermoremanent Magnetisation (TRM)**, the **Chemical Remanent Magnetisation (CRM)** and the **Detrital Remanent Magnetisation (DRM)**. The secondary NRM can arise from ferromagnetic minerals affected by alterations of chemical nature, from strikes of lightning in close range and, in the long run, because the geomagnetic field is affected by rock creation processes.

When a sample is reaching a temperature higher than its Curie or Neel temperatures and then cools down to room temperature in the presence of a magnetic field, then the remanent magnetisation it obtains is called **Total Thermo-remanent Magnetisation** (**tTRM** or **TRM**). The remanent magnetisation acquired from temperatures lower than the Curie to the temperature of its environment, is called **Partial Thermo-remanent Magnetisation (pTRM)**.

The remanent magnetisation that is acquired by a magnetic mineral, as it crystalised inside a magnetic field in temperatures below their Curie, is known as **Chemical Remanent Magnetisation (CRM)**.

In the cases when the magnetic particles align, sediments in an air/water sediment interface obtain **Depositional (Detrital) Remanent Magnetisation (DRM)**. Even though

they cannot move, due to the sediments above them, their remanent magnetisation often changes over geological timescales, because of the geological activities (cryoturbation, bioturbation etc).

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Apart from the primary types of magnetisation there are also many secondary. If a sample is placed inside a constant or stable magnetic field and then this field is removed, the magnetisation measured afterwards is the **Isothermal Remanent Magnetisation (IRM)**.

There are cases when the depositional remanent magnetisation is not stored in for a while after the sediment is deposited or when chemical changes affect the magnetisation. Then the remanent magnetisation sediments acquires the **Post-depositional Remanent Magnetisation (pDRM)**, which appears right after their deposition and is preserved on the long run during the geological years.

In cases of exposure to weaker magnetic fields, the spontaneous remanent magnetisation acquired by the sample is the **Viscous Remanent Magnetisation (VRM)**. It is an unstable type of remanent magnetisation, with blocking temperatures lower than 200°C for the magnetic domains, giving space for changes to take place in the magnetic properties even in room temperature. This makes it particularly unstable, with a timescale from a few minutes to even centuries, and also unwanted.

Lastly, if a less steady magnetic field's effect is removed from the measured remanent magnetisation, the result is the **Characteristic Remanent Magnetisation (ChRM).** It is a very important remanent magnetisation, since it reflects the Earth's clean magnetic field during the cooling of a burned material. The measure of the ChRM directions, from individual samples, is expressed by the **precision parameter K**, indicating the point distribution on a sphere's surface. The distribution and the K parameter are inversely proportional.

There are two important temperatures for Archaeomagnetism. The first is the **Curie-Temperature**, **T**<sub>c</sub>, the temperature at which a material turns from ferromagnetic to paramagnetic during heating (and vice-versa for the cooling process). This happens because the more thermally fluctuated the atomic magnetic moments of a mineral are, the weaker the magnetisation becomes, until it nullifies at the Curie temperature. At this point the thermal fluctuations are so intense, that any magnetic order disappears and more heating will not make any notable difference.

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*Figure 1.7:* Normalised saturation magnetisation to temperature, for hematite and magnetite (figure from Butler, 1992)

The second temperature is the -previously mentioned-, **Néel-temperature**  $T_N$ , the temperature at which the ferrimagnetic and anti-ferromagnetic materials are antiparallel and beyond which the material becomes paramagnetic.

BIβλιοθήκη GEOΦΡΑΣΤΟΣ" Chapter 2: The Archaeomagnetic Method A.Π.Θ

With the help of Archaeomagnetism, the scientists aim to understand and trace the changes of the geomagnetic field throughout the prehistoric and historic years, for a chosen area (*Aitken, 1978*). This requires the discovery of structures and materials, that at some point had reached high temperatures, consist of magnetic materials and it is possible to date them.

As a method, it is used to create and establish the Secular Variation Curves. The SVC of each area is unique and shows the changes of the geomagnetic field through time, over this specific area.

The most essential progress of the field began in the 1930s, when Professor E. Thellier started using baked clays in measurements (*Thellier, 1938*). This progress, now known as *"Thellier-Thellier Experiment"* (*Thellier & Thellier, 1959*), was adopted as a reliable method during the 1960s and it has spread worldwide.

#### 2.1 Basic Principles and Important Definitions of Archaeomagnetism

The **Archaeomagnetic method** is a very important method, mainly used to study and understand the changes of the magnetic field of the Earth through the years and aims at the creation and update of the SVC of each country and/or region.

The **samples** in the archaeomagnetic research are magnetised archaeological material, last fired in the historic or prehistoric years. These samples are preferably taken from **insitu** structures, which means from structures found in their primary positions. In the laboratory, smaller pieces known as **specimens**, are cut out of the samples. These specimens are either cylindrical or cubical in form, having been retrieved with the use of drills or saws respectively. In the end, an average is calculated for the measured value of each sample's specimens, reducing, this way, the effect of any possible error and problematic measurements, providing more accurate final results.

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What is measured in these samples is the **remanent magnetisation** they have acquired during the last time they were burned and cooled down. The intensity of Earth's past magnetic field, estimated from the remanent magnetisation of the archaeological material, is called **archaeointensity**.

In the laboratory, it is possible to use a known, artificial magnetic field and calculate accurately the geomagnetic field in the past through reheating experiments. The measurements can be really precise in cases where the material acquired their natural remanent magnetisation as thermoremanent magnetisation.

During the archaeointensity experiment, a second partial thermoremanence is given to the sample every second heating. This extra step is called **pTRM check test** and is used as a quality check test, aiming in recognising any possible chemical distortions that might have occurred during the previous steps of the experiment. It has to be applied in any temperature between the lowest and highest of the experiment.

Based on the mineralogical composition and crystalline structure of a sample, there is a temperature at which, during cooling, the TRM is stored in the sample and "freezes". That temperature is known as **blocking temperature**, while the temperature at which the remanence is lost is called **unblocking temperature**. These two temperatures do not have to be necessarily equal.

To isolate a blocking temperature or the high coercivity component of a sample and remove the less stable ones from them, the sample goes through **partial demagnetisation**. This is possible by exposing the sample to a new magnetic field (AF demagnetisation), by heating it to a specific temperature in an oven (thermal demagnetisation) or by exposing it to radiation from low-power microwaves (microwave demagnetisation).

The initial **magnetic susceptibility** of a sample is widely used for the archaeomagnetic measurements and methods. An exterior field shows a magnified effect around superparamagnetic (SP) grains, because they show higher susceptibility than an SD grain of equal size.

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The **anisotropy of magnetic susceptibility (AMS)** should not be disregarded, since it can give much information about the magnetic phases and structure, as each mineral gets magnetised easier along different axes during different phases.

The monitoring of the magnetic susceptibility helps to identify the magnetic elements of a sample through the Curie temperature. A small amount is taken and crushed. Then, through a controlled procedure, the material is heated up to 700°C and left to cool down. This way a curve is produced on which it is possible to note the Curie temperature and other phase changes. When this curve is reversible, it means that there were no changes in the mineralogy of the sample while conducting the experiment. The sample's primary susceptibility is called **high temperature magnetic susceptibility**.

The basis of archaeomagnetic research lies on **two main principles**. The first is the variations of the geomagnetic field's direction and intensity on a global scale and over time and the results of this principle is shown with the creation of the Secular Variation Curves. The second is how the geomagnetic field's information can be stored inside the archaeological material, when the material is burnt and cooled down. The archaeomagnetic method itself is based on this second principle, measuring this stored information and using it as a means to understand the changes of the geomagnetic field through time.

Archaeomagnetism can be used as a dating method for material found in archaeological sites, giving them an **archaeomagnetic dating**. This can be achieved by comparing the magnetisation measured from the respective material, with the variations of the past

geomagnetic field, using the Secular Variation Curves of the area in which the elements were found.

#### 2.2 The Secular Variation Curves (SVC)

Ψηφιακή συλλογή Βιβλιοθήκη

As it was mentioned above, the geomagnetic field's parameters, such as the direction and magnitude, vary through space and time. This means that, not only does it change over the centuries, decades and even years, but it also differs from one area to another.

This led to the creation of the Secular Variation Curves. These curves are different for each area and region and represent the changes of the magnetic field that took place during the course of time between 1-105 years.

Even though the last 4 centuries many direct observations of the secular variations were conducted, an important quantity of information has been acquired from historical studies. Unfortunately, they are limited in both time and space, and the details needed to create the SVC of a region exceed those provided by the historical recordings. This requires the use of other methods, such as palaeomagnetic measurements of clay-based geological formations, studying of archaeological features with the use of archaeomagnetism, or sometimes the combination of these. This way, it is possible to indirectly study the magnetic field of the Earth from older geological periods.

Of course, there is often a correlation between the magnetic observations used for the different secular variation models, since each model depends heavily on the amount of data and the spatial spread they have. This means that it is often necessary to take into account all the available data from the archaeomagnetic and palaeomagnetic measurements not only from the area of interest, but also from a wider region or even globally.

So far, Europe provides the majority of archaeointensity information, with a highly scattered distribution of the intensity measurements (*figure 2.1*). This means there is still a respectful amount of data to be obtained in order to better understand the Earth's magnetic field and its changes throughout time.

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*Figure 2.1:* The archaeomagnetic sites in Europe (white diamonds from Arneitz et al.2017; the other marks and the updated map from Schnepp E. et al., 2020).

There seems to be a pattern that connects the secular variation of the regions on a subcontinental scale, but it changes greatly between continents. This proves how important the smaller, non-dipole sources of geomagnetic field that exist inside the Earth's core are *(Butler, 1992).* It, also, creates another problem, as new studies on these non-dipole sources have to take place and more databases with information on the secular variation caused by these sources for each region is needed (*Tarling, 1983*).

Based on these findings and the respective studies, to avoid errors related to extensive directional spread, it has been decided that the archaeomagnetic records are stable over areas of approximately 1000 km in diameter (*Noël & Batt, 1990; Batt, 1997; Sternberg, 1997*).

#### 2.3 The Archaeomagnetic Method and Dating

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What is actually measured with the archaeomagnetic method does not represent the moment *when* the structure was built, but the *last time* it got fired and cooled (kilns and old clay-based kitchenware) or was deposited (volcanic lavas). In other words, the method can be used to study objects and material that have been through a remanence-inducing event, that caused it to reach temperatures above its Curie temperature.

These archaeological artifacts are usually made out of material that contains magnetic minerals, such as clay, tile or bricks. At some occasions, even burnt structures like houses and soils can be studied, although they show a weaker magnetisation. The most common objects used in archaeomagnetic measurements are clay vessels, as a plethora of them can be found in most archaeological sites. They come from different time spans and it is often easy to have an archaeological dating estimation.

It should always be kept in mind though, that every time they get fired, they lose their previous record of magnetisation, unless the last firing incident took place in lower temperatures. In that case, information about the geomagnetic field during both heatings can be stored inside the material.

The archaeomagnetic method can also be used as a dating method and can be used even for materials that have been moved from their primary position (bricks, tiles etc.). Although the undisturbed, in-situ structures provide many more magnetic related information of directional nature, those moved can be used for intensity related studies.

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The direction and the intensity of the magnetic field recorded in the artifacts can be compared (usually statistically) to the already established SVCs of the area to which the archaeological site belongs. This not only helps in finding the date of the object's last heating, but also in comparing it to the other structures of the site on various levels (age of creation, last use etc) *(Evans, 1994)*. This is based on the fact that materials found in the same area, that also have been magnetised at the same time, are expected to have recorded similar magnetic properties. The more objects are studied and the database is enlarged, the more accurate and reliable the method will get.

To use archaeomagnetic dating more effectively and avoid any possible errors, it is very important to create more detailed secular variation curves for as many regions as possible (*Pavón-Carrasco et al., 2014*). By studying many magnetic materials from all around a specific region, it is possible to map the changes from which the geomagnetic field went through in that area. It is necessary for the results to agree not only with each other, but also with the already established curve for the region of interest.

Sometimes, the rate of the geomagnetic field's alterations is high. This can be both positive and negative, as on one hand the changes in a small time-window can provide a conclusion with more precision in the dating. On the other hand, the constant change of the geomagnetic field can give similar characteristics to various points through time, making it hard to place a sample that shows these repetitive characteristics on the correct point.

Like in all studies, the best conclusions are those that have been checked by more than one dating methods. So, another way to achieve better precision, is to have a relative dating based on the archaeological studies (coins, historical sources etc.), or even with the help of other dating techniques (such as the 14C method). This way, results are crosschecked and there is archaeological information to support the date determined through the archaeomagnetic method.

In addition, a comparison of different areas' results can bring us one step closer to understanding better how the magnetic field is generated, as the SVCs complete a bigger picture on the geomagnetic field.

Taking all these aspects into account and after a long run of research, some countries succeeded to obtain very well-established secular variation curves, with the Bulgarian SVC being one of the most complete for Europe, but also about worldwide, providing information about the last 8 millennia (*Kovacheva et al., 2014*).

Despite the importance of the archaeomagnetic studies, it is not always easy to obtain the required samples, as in some countries, including Greece, permissions for such research are difficult to obtain, while in others, the structures that can be used are of little value due to the lack of other historical information.

#### 2.4 Archaeomagnetic Studies in Greece

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The archaeomagnetic studies in Europe have gained more importance in the last few decades, with such studies in the Balkans beginning in the 60s (*Kovacheva, 1969*). In Greece, the first archaeomagnetic studies on pottery took place during the 80's (*Thomas, 1981; Walton, 1984; Aitken et al., 1989*), while the first Greek archaeomagnetic data from kilns were published earlier, in 1963 (*Belshé et al., 1963*). There is more information about archaeointensity than directional data and the age coverage is wide, ranging from Neolithic Era (*Aidona and Kondopoulou 2012; Fanjat et al., 2013*), to the Byzantine (*Spatharas 2005; Evans 2006; De Marco 2007; Kondopoulou et al., 2015; Aidona et al., 2018, Genevey et al., 2018*) period.

Generally, the Greek prehistoric data, were almost solely taken from ceramics and pottery, since they can be found in most sites in plethora and can provide very accurate results. They also show distinct archaeological features, making a cross-check dating easier.

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The Greek directional SVC has been recently updated regarding the last 4500 years (*De Marco et al. 2014*), but it is not yet complete, showing gaps during some eras. The first updated intensity curve was by DeMarco (*De Marco et al. 2008*), but since then new high-quality data have been presented, in order to fill the existing gaps of the Greek curve (*figure 2.2*).



Figure 2.2: Greek Intensity SV Curve, as of 2020

Even though many archaeomagnetic studies were performed in Greece, the measurements so far have not given enough results to cover the whole Greek SVC. The periods with few or no available archaeomagnetic data are met in the later Neolithic era, at around 4400-3400 BC, and then the second in the Bronze Age and more specifically the middle Bronze Age of Europe, from 2300-1600 BC.

Despite the fact that the rich archaeological material found in the Balkans can help scientists construct a full geomagnetic field vector with continuity through the whole millennia, due to these period gaps, some data are considered to be questionable and
might be considered biased (*e.g.Pavón-Carrasco et al., 2014*). Thankfully, the majority are reliable enough, especially from the most recent studies, which provided high quality results (*e.g. Aidona et al., 2018*).

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In many cases, especially those where other dating methods fail, the Balkan SVCs are used to date baked clay structures of uncertain age. For the best outcome, there are comparisons between the results of different dating methods, like thermoluminescence or radiocarbon data and archeological methods (*Aidona and Kondopoulou, 2012; Kondopoulou et al., 2015; Tema et al., 2015*). Chapter 3: The Archaeological Sites

Ceramics are the most common type of the archaeological findings in all archaeological sites of Macedonia. The clay vessels are very important in understanding the past, providing us with information referring to everyday life and habits of the ancient inhabitants. This way, we can piece together a more complete image about their society, their culture and the conditions under which they were living. For this reason, the discovery of intact and wholesome ceramic vessels and artifacts is significant and valuable.

In this present study we present archaeomagnetic results from two archaeological sites in Greece. A short description of the sites is given below.

#### 3.1 Archontiko

The village Archontiko is an archaeological settlement, between the regions of Ancient Pella and Giannitsa (Central Macedonia). The settlement was dated with various methods. The dating given by the archaeologists was around 2300-1400B.C., while the radiometric dating of 21 specimens gave a shorter period of dating between 2300-1600B.C.

The settlement's habitability was constant and this is also verified by the more recent part of the village of Archontiko, which is still populated.

The main studies on the area began in 1992 and are still ongoing. The research was initiated by archaeologists of the corresponding department of AUTH (ex. *Papaeuthumiou-Papanthimou et al., 1998*) and additional research was conducted by members of the Geology Department of Aristotle University of Thessaloniki (AUTH) (*Syrides et al., 2009*). The main aim of the research is the restoration of the old settlement.

Based on the more recent findings, there seems to be a sequence of different, successive stratigraphic horizons, separating three phases in the evolution of the settlement. The

main phase of the site dates from the end of the Early Bronze Age to the beginning of the Middle Bronze Age. Since the information about continuous habitation in ancient Macedonia during those periods are only few, the value of this settlement is even greater. With the use of radiometric dating, the main phase was placed between 2300-1900B.C (*Papaefthumiou-Papanthimou et al., 2004*).

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Figure 3.1: The site of Archontiko (Deliopoulos, 2014)

At the top on the area's hill, a second, newer phase was discovered. The artifacts that were found were not that many, but it seemed to be quite unique and were decorated with carved geometrical motifs and the use of a white-coloured paste. The second phase belongs to the Late Bronze Age.

Lastly, on the eastern part of the archaeological site, the first and oldest phase from the end of the Early Bronze Age appears. There seems to be a destruction layer, which was caused by a fire. There were many artifacts found on the floors of the preserved houses, some of them kept intact. What makes the research in this site even more important are the many intact ceramics found. From vessels and clay-pots, to storing cases and ovens, more than ¾ of the findings are coming from houses of the first two horizons.

These artifacts were categorised in 6 types based on their usage:

1. Those used as a simple kitchenware

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- 2. The ovens, most of which have a handle, making them portable (*figure 3.2*)
- 3. The artifacts associated with burials and
- 4. The vessels used to burn herbs and spices for ceremonial purposes
- 5. The circulars of smaller and larger size
- 6. The ceramics with more than one morphological criterion

Between the many vessels that were used for cooking, light, heating etc., some elliptic, ceramic structures, with 0.7-1m height were found. While their use was in discussion, samples were taken, from both the bottom and the walls of the structures and were studied with the help of many methods, such as spectroscopy, light and scanning electron microscopy, X-ray diffraction, but the most important one was the pyrotechnic method.



*Figure 3.2*: Clay vessel similar to the ones used for this study's measurements, from the archaeological site of Archontiko (image from <u>https://www.archaiologia.gr/blog/issue/</u>)

This way, it was concluded that most of these structures were used for cooking, in a manner similar to today's ovens. They would raise the temperature high, at around 400-500°C and after 30 minutes they would stop the heating. The structure preserved the temperature high at around 200°C, giving time for the food to be cooked. Sometimes, lower temperatures could be used to dry fruit and vegetables, so they could be stored.

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The material which was used in the walls of these ceramics was clay, with some rounded pieces of feldspars, carbonised plants and shell fragments. The floor of the structure was made of a material similar to that of the walls, with an additional, insulating layer of invertebrates (mainly of the genus Ostrea). The structure's chemical substance is non-calcareous, but contains sulfur, due to the organic materials, and some phosphorous. Therefore, it was concluded that it came from some river that used to be close by, and which has now dried out.

Generally, many similarities were found in the vessels of horizons II and III. The colour of the clay and the baking layers on their walls indicate firing conditions that were not completely controlled, so they were probably baked in open holes, or wide-open fires. Although similar firing layers were studied in some of the vessels of horizon I, comparing the horizons II and III, there is an equal number of vessels based on clay colour, and therefore based on the type of firing they went through. Furthermore, there were some differences, like how in horizon III there were more closed, small-grained and open largergrained vessels than in the horizon III.

There is one last horizon (IV) to be studied, which seems to be as well preserved as the rest. It consists of both building structures and old tombs.

Finding completely intact ceramics is not that common, so the ceramics found here will play a major role in understanding the lifestyle back then. In 2012, by using the statistical method of Bayesian Inference to analyse the results of the radiometric studies, a more collected dating of the horizons of Archontiko was achieved. The dates are shown in the table below (*Table 3.1*).

Period	Horizon	Dating
LBE	I	1516-1414 B.C.
EBE	II	1923-1877 В.С.
		2085-1903 B.C.
	IV	2130-2087 B.C.

**Table 3.1:** The resulted dating from the Bayesian Analysis model LBE: Late Bronze Era | EBE: Early Bronze Era (Μανιάτης Γ., 2014)

Our samples all belong to the main phase and more specifically Horizon II (ARC2, ARC3, ARC5, ARC6, ARC7, ARC8 and ARC9) and Horizon III (ARC1 and ARC4).

# 3.2 Cretan Phryctoriae

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During an archaeological excavation in Crete in the area of Pediada, between 1982-1989, archaeologist Nikos Panagiotakis discerned an old, unique system of communication with the use of fire. It was not rare for fire to be used to exchange messages between two areas, since it had already been used in the region where Syria stands today and also in ancient Greece during the Minoan times, especially from 1900 to 1700B.C.

The number of structures, found in the area during surface ordeal survey, is large. Due to the topographic characteristics and the growth of the region, a communication system was needed. The created system was based on the exchange of fire signals and covered more than 800 square kilometers, between the palaces of Mallia and Knossos, reaching the area of today's Heraklion.

The fire was lit on big, manmade, tower-like structures, that also served as observatories and were named Phryctoriae ( $\Phi \rho \nu \kappa \tau o \rho (\epsilon \varsigma)$ , from the ancient Greek word for torch ( $\phi \rho \dot{\nu} \kappa \tau o \varsigma$ ). They were usually built upon remote and unapproachable locations, like hills and cliffs and were positioned in a way that made it easier to guard the roads and passages in all the province and coasts.

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Often, between the Phryctoriae there was one or more smaller observatories, named Soros (*figure 3.3*), which would rebroadcast the messages from one main Phryctoria to the other. In case of bad weather, when the communication through fire and smoke was not possible, the distance between them could be covered on foot, by a runner.



Figure 3.3: Image of Soros t'Amygdaliou (figure from Panagiotakis et al, 2013)

This system of communication was established, not only for direct exchange of information between the cities, but also for recognising approaching enemy troops. The fire would also pose as a sign of power, scaring sometimes the enemy and thus protecting the city.

For the purpose of dating the Phryctoriae, in addition to the archaeological information, luminescence method was used. Since both of these methods draw their information from the sample's last heating event, the comparison of their results is quite accurate, although so far studies that combine these two methods have been limited (*Aidona et al., 2013a, b*).

The results of the luminescence dating of Phryctoriae, provided a mean age at 1606 BC  $\pm$  170 years. More specifically, samples from two different Phryctoriae were used for the luminescence dating. The first, the SKA samples, were placed at around 1521 ( $\pm$ 177) B.C. and the second, the SX samples, were placed at around 1691 ( $\pm$ 162) B.C.

These same samples were used for the experiments conducted for the purpose of this thesis.

The samples with code names SKA belong to Soros Kamberi and are all baked clays. The samples with code names SX came from Soros Chartis, with the samples SX1, SX2, SX5 and SX6 being samples of baked clays, while the samples SX3 and SX4 were sherds.

Both Soros are located close to the village Sambas.

# Chapter 4: Methodology and Measurements

The methodology for an archaeomagnetic research depends on its initial purpose. Generally, there are many experiments based on the methods of archaeomagnetism, that can be done to determine a sample's magnetic properties and also its ability to acquire TRM.

The most common ones are based on a sample's mineralogy are the *rock magnetic* experiments and the ones studying the archaeointensity, like the *"Thellier-Thellier"* experiment (*Thellier & Thellier, 1959*), which was used for the purpose of this thesis too.

Since each sample can usually be used only for one of these experiments, it is essential to collect many extra samples for every rock magnetic experiment that has to be done, in order to understand and study the magnetic properties more thoroughly.

#### 4.1 Preparation of Samples

Ψηφιακή συλλογή Βιβλιοθήκη

ΤΖΑΦΦ

The first step that needs to be done is to prepare the samples. For both the Thellier-Thellier experiment and the rock magnetic experiments specimens in the form of cylindrical cores were used.

At the beginning, we started with nine different clay pieces from Archontiko (*figure 4.1*) and ten different samples from Phryctoriae, with each of these samples being numbered.

The first step was to plaster all samples *(figure 4.2),* so that later it would be possible for specimens to be retrieved, without the risk of them breaking. The specimens were cut with the use of a drill the next day, as the plaster was left overnight to dry.

To get as many specimens as possible and, therefore, achieve the best precision in the measurements ahead, we cut as many cores as possible from each sample.

In the end, we had 28 specimens from Archontiko and 25 specimens from Phryctoriae.



Figure 4.1: The 10 samples from Archontiko in their initial form.



Figure 4.2: The plastered samples from Archontiko

It is very important to keep track at each sample that is being processed and name the specimens correctly. Each specimen's name shows from which area and which sample it came from (*figure 4.3*)



Figure 4.3: The cores from the first 6 samples of Archontiko (ARC), after the use of the drill

With the samples in the appropriate form, the actual experiments could begin. Before that, the NRM of all cores was measured and saved.

# 4.2 Rock Magnetic Measurements

The rock magnetic experiments are based on the mineralogic consistency of the samples we have. The magnetic material records the properties of the geomagnetic field at the time of the last cooldown they went through after a heating.

The rock magnetic experiments conducted for the purpose of this thesis are:

- The Isothermal Remanent Magnetisation (IRM)
- The Thermomagnetic Measurement (K-T curves)
- The Hysteresis Loops

For this study's sites, samples were retrieved both from Archontiko and from Phryctoriae.

# 4.2.1 Isothermal Remanent Magnetisation Measurement

Ψηφιακή συλλογή Βιβλιοθήκη

**Α.Π.Θ** 

When a material is exposed to a strong magnetic field, it acquires a magnetisation known as Isothermal Remanent Magnetisation (IRM). This ability is often used in laboratory experiments, in order to distinguish the mineralogy of a sample. The magnetic saturation of each magnetic mineral differs and depends on their ability to get magnetised in either low or high magnetic fields.

Generally, the samples that saturate in magnetic fields of 300-500mT, belong to softfraction magnetic minerals (like magnetite). Samples with hematite and goethite acquire their saturation at much higher fields (up to 3T), and belong to the hard-fraction magnetic minerals.



Figure 4.4: ASC Scientific IM-10-30 Impulse Magnetizer, Bartington Instruments

At first, the samples are exposed to increasing magnetic fields, each time using a slightly stronger than the previous one, starting with a relatively weak magnetic field (stepwise magnetisation). Each time the exposure lasts a short period of time and the acquired

remanent magnetisation is measured, before the next field is applied. The process ends when the sample reaches its magnetisation saturation point (usually at around 1200 Oe).

For the IRM measurements, the instrument used in our laboratory was the ASC Scientific, model IM-10-30 Impulse Magnetiser *(figure 4.4)*.

### 4.2.2 Thermomagnetic Measurement

Ψηφιακή συλλογή Βιβλιοθήκη

The thermomagnetic measurements have as a main goal to produce curves, that represent the monitoring of the magnetic susceptibility, with the application of increasing temperature and subsequent cooling.

At some point, the magnetic susceptibility will decrease relatively abruptly. Those temperatures are the Curie temperatures, which are characteristic for each mineral and can be used to identify the main ferromagnetic minerals the burnt sample contains and which have enhanced the magnetic behavior of the sample.

While going through the experiment, when the maximum temperature we imparted to the sample (~700°C) is reached, the applied temperature will begin to decrease, in an attempt to get a reverse curve. If this second curve is similar to the first one, it means that there were no important chemical changes during the heating and the sample is stable mineralogy-wise, giving the green light to proceed to the archaeointensity measurements.

The samples, though, can often show more than one Curie temperatures and give often non-reversible curves, go through chemical changes or even have distribution of grains with different sizes, producing curves that are more difficult to be interpreted.

The most stable results come from tiles, bricks and clays that have been heated in high temperatures, while material like burnt soil and plasters provide us with more unstable results. The most common magnetic mineral found in archaeological samples is magnetite, while hematite is rarer. If during the experiment the magnetic susceptibility suddenly decreases in the lower temperatures, then its grains have possibly gone through slow oxidation. When the decrease happens in higher temperatures, the oxidation is probably higher.



Figure 4.5: Models MS2WFP of Furnace (left) and Power Supply Unix (right), by Bartington Instruments

To measure the thermomagnetic curves, the instruments used were MS2WFP Furnace and the MS2WFP Power Supply Unix, by Bartington Instruments (*figure 4.5*).

#### 4.2.3 Hysteresis Loops

Ψηφιακή συλλογή Βιβλιοθήκη

The ferromagnetic minerals are valuable to the palaeo- and archaeomagnetic research, because they possess the ability to record the properties of the magnetic field they are exposed to. When this field is removed, the magnetisation is not lost completely, but instead stays recorded in the material. The variation of the magnetisation M with the applied field H is called **hysteresis loop**. The shape of the resulting curve taken by a hysteresis study heavily depends on the type of the ferromagnetic minerals and the size and nature of the grains.



Figure 4.6: Hysteresis Loop and its parameters (figure from Tauxe, 2005)

When the magnetic field that applies on the ferromagnetic minerals is increased, then the magnetisation will also increase until it reaches its point of **saturation magnetisation Ms**. It will not surpass that value though, no matter how strong the field keeps getting afterwards.

When the magnetic field that acts on ferromagnetic material is removed, the smaller the grains of the material, the larger will be the ratio of **remanent magnetisation Mrs**, to the saturation magnetisation (*Mrs/Ms*).

It is also possible to change the direction of the magnetisation to the opposite, by exposing the material to a magnetic field with opposite direction from the one it already possesses. The required opposite magnetic field to zero the magnetisation is known as **coercive force Hc**. At some point, if this reverse magnetisation is strong enough, it can even cancel the initial remanence. This is known as **remanent coercivity Hrc**. Their ratio (*Hrc/Hc*) is higher when the grains are larger.

If the opposite field is applied with even more power, then the saturation magnetisation takes a negative value -Ms.

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Then, a positive magnetic field has to be used again, so that the remanent magnetisation will become zero and lastly go back to the initial positive saturation. This is how a hysteresis loop closes and is complete (*figure 4.6*).

Since at the Curie temperature, which is unique for each mineral, the saturation magnetisation is nullified, the ferromagnetic material is possible to have a remanent magnetisation M<sub>r</sub>, caused by the magnetic field that had previously acted on it.

This experiment was conducted with the help of a VSM, a Vibrating-Sample Magnetometer.

#### 4.3 Archaeointensity Measurement (Thellier-Thellier Experiment)

There are a few experiments with the use of which it is possible to measure the palaeointensity and one of the most common is the "Thellier-Thellier" method. It is based on a series of a complete cycle of heatings and cooldowns of the samples, on various, constantly increasing temperatures.

For the experiment, an oven with zero internal magnetic field is required and used for every step. Throughout the whole process, while the samples are in the oven, they are exposed to the same controlled magnetic field, which applies on their Z axis (*figure 4.7*). For every temperature the samples are being heated, let to cooldown and measured twice, in both normal and the reverse field. This is in accordance with partial thermoremanent magnetisation's "*Law of Additivity*", which states that the sum of all independent pTRMs will result in the total TRM. This field also has to be as close as possible to the intensity of the geomagnetic field at the time the samples were last burnt. This is why an archaeological or other type of dating of the samples is important.



**Figure 4.7:** The oven used for the experiment was the Magnetic Measurements Thermal Demagnetiser (Serial Number 142), distributed by Magnetic Measurements. On top of it is the TTi EL301 power supply, by Thurlby Thandar Instruments, providing the steady magnetic field for the whole experiment as the samples were burnt and cooled down.

Usually, the difference between the first steps is around 50°C, while as moving towards higher temperatures, the change is smaller, reaching 5-10°C if needed. The aim is to reach the Curie temperature of the samples, after carrying out the necessary temperature steps.

The first step is the measurement of the Natural Remanent Magnetisation possessed by the samples and then moving on to its slow replacement by the new pTRMs, produced by each cycle (*figure 4.8*).

It is necessary to perform a check, known as "pTRM". Every two or three temperature steps, a check takes place by repeating a previous, lower temperature step. This is to confirm that no chemical reactions happened at the time of the earlier steps and the samples are still capable of gaining a pTRM.



*Figure 4.8*: The Molspin Limited spinner, used to measure the magnetisation of the samples, after each step of burning-cooling.

The experiment is heavily based on the hypothesis of pTRMs' "Law of Independence", which expresses how the obtained partial thermal remanence differs for every pair of consecutive temperatures.

As the measurements of each step continue, an NRM-TRM plot is created for each sample. This plot shows the connection between the NRM component left in the sample after every heating and the pTRM acquired at each step (*figure 4.9*).

The higher the quality of an NRM-TRM plot, the more stable the remanent magnetisation of a sample is and is also believed that the blocking and unblocking both happen at the same temperature. This last hypothesis is known as *"Law of Reciprocity"*. However, it does not apply for the more complicated grains (*Dunlop & Özdemir, 1997*) and these samples are usually counted out, since the initial TRM will not be separated correctly, eventually bringing errors to the result.



Figure 4.9: Linear, array plot from a random sample of this study (SKAB1)

In addition to that, many more things have to be checked and corrected if necessary. The anisotropy is a factor that needs to be considered during such an experiment and should be calculated for each one of the samples. Even though for the study of clays, anisotropy is not a very decisive factor (*Kovacheva et al., 1989*), it is of crucial importance for the study of ceramics and pottery (*Gomez-Paccard et al., 2006*)

When a sample loses 70% of its magnetisation, it is heated, aligned to its different axis (X+, X-, Y+, Y-, Z+), cooled and then measured. This way they can be used for the check process.

Also, a seemingly small difference in the parameters under which the cooling effect takes place, could lead to an erroneous TRM measurement. For example, a slower cooling can

give a higher TRM. This means that a track on the cooling rate has to be kept, to avoid a more biased result and aiming to the highest accuracy.

Ψηφιακή συλλογή Βιβλιοθήκη

The last cooling of the material happened in environmental conditions taking a much longer time than the experiment in the lab. So, after the last heating of the samples, they are left in the oven to cooldown slowly, for 24-48 hours. This way, any errors from the quick, laboratory cooling can get corrected (cooling-rate effect).



### 5.1 Rock Magnetic Experiment

As mentioned before, three different rock magnetic experiments took place for the purpose of studying this thesis' samples. There were five or more specimens used for these experiments and here are the results of each process and experiment.

# 5.1.1 Isothermal Remanent Magnetisation

When a strong magnetic field applies on a sample, the magnetisation it acquires in room temperature is the IRM. Depending on the saturation point of the sample, it's consistency in magnetic minerals can be identified.

In the experimental re-enactment of this process in the laboratory, we used a magnetic field that reached the 1200mT. Five samples were selected. Two were from Archontiko and three from the Phryctoriae. Apart from their NRM state, they were exposed to 13 different field intensities, from 30mT to 1200mT. It is visible from the diagrams that this also seems to be their saturation point.



#### Archontiko:



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Both the diagrams from Archontiko, show similar behaviour of the samples and the coexistence of many different magnetic minerals in their consistency (*figure 5.1*).



#### Phryctoriae:

Ψηφιακή συλλογή Βιβλιοθήκη

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Figure 5.2: The IRM acquisition of Phryctoriae samples in relation to the applied magnetic field

The diagrams of Phryctoriae show high diversity from one another and a variety of magnetic minerals in their consistency *(figure 5.2)*.



One very helpful factor that can be used for magnetic mineral studies is their Curie temperature. Through the Thermomagnetic experiment, we can find where that temperature stands for each sample, thus defining its mineralogy.

For this thesis, the temperatures used for the thermomagnetic experiment were from  $50^{\circ}$ C to  $700^{\circ}$ C.





Figure 5.3: The thermomagnetic diagrams for the Archontiko samples

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The changes between the two curves of each sample (heating with red and cooling with blue), indicate possible chemical changes of the magnetic minerals during the samples' heating and cooling.

Ψηφιακή συλλογή Βιβλιοθήκη



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Figure 5.4: The thermomagnetic diagrams for the Phryctoriae samples

Both set of samples show some differences between the susceptibility during the heating and the cooling process (*figure 5.3 and 5.4*). Even though in some cases this could mean that some exterior factor changes the environment under which the experiment took place, most curve diversities show that there were probably some chemical changes in the sample 's mineralogical consistency, resulting in the creation of new magnetic minerals.



For the hysteresis loops, six different samples were examined, two from Archontiko (ARC1 and ARC6) and four from Phryctoriae (SX1, SX2, SKAA and SKAC). The magnetic field used was from -1.2T to 1.2T.

ARC1







Figure 5.5: Hysteresis loops for the samples of Archontiko

For the ARC1 and ARC6 samples, the coercive force  $H_c$  is estimated at 15 kA/m and the remanent magnetisation is 0.1 Am<sup>2</sup>/kg (*figure 5.5*).



Figure 5.6: Hysteresis loops for the SKA samples of Phryctoriae

For the SKAA and SKAC samples, the coercive force  $H_c$  seems to have a small diversity, estimated at 8 and 9 kA/m respectively. The remanent magnetisation is 0.07 Am<sup>2</sup>/kg for both *(figure 5.6).* 



Figure 5.7: Hysteresis loops for the SX samples of Phryctoriae

The SX1 and SX2 samples display very different behaviours, with the SX1 having an estimated coercive force  $H_c$  at 6 kA/m, while for the SX2 it is estimated at 12 kA/m. The remanent magnetisation is 0.07 Am<sup>2</sup>/kg for both (*figure 5.7*).

At first look, the results indicate different types of material not only between areas, but in some cases even between the samples from the same area of interest too.

# 5.2 Palaeointensity Measurement (Thellier Experiment)

Βιβλιοθήκη

Α.Π.Θ

The biggest experiment from the ones conducted for the purpose of this thesis, is the Thellier-Thellier experiment.

The first and lowest temperature in which the samples were baked, was 100°C, while the highest some samples reached was 570°C. At the beginning, the temperature was raised by a step of 50°C for each heating, until it reached the 400°C, where the step difference dropped to 40°C, 25°C and finally 20°C. Every two temperatures, a check measurement was taken (pTRM test).

There were four points where the samples lost 70% or more of their magnetisation, where the anisotropy measurements were taken. These temperatures were 440°C, 480°C, 505°C and 530°C.

As soon as the magnetisation of the samples was close to the beginning of the two axes, a 24 hour-long cooling process followed, at 510°C for some samples and at 555°C for the rest.

The laboratory magnetic field used for an experiment has to be close to the expected archaeointensity value. Since the expected value as given through the GMF models was 50-55  $\mu$ T, the magnetic field that was used in this experiment was 55 $\mu$ T.

After the measurement of the samples, the results taken were converted in a form that could be processed through the RenArMag\_v3511 software. With the use of this software, the different magnetic values were calculated based on the measurements of each step, the anisotropy correction and the cooling process. The samples for which these corrections were unsuccessful were rejected.

The linear NRM-TRM diagram of each sample had to be determined for at least 5 points. Also, the values for the Maximum Angle of Deviation (MAD) and the Deviation Angle (Dang) had to be lower or equal to 10 (*Gomez-Paccard et al., 2006*).

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In the anisotropy correction, the correction factor had to be bigger than the alteration factor. For the samples that met this requirement, the archaeointensity correction was applied. For a 24-hours cooling process, this correction is expected to be lower than 10% and the alteration factor is expected to be lower than 5% (*Gomez-Paccard et al., 2006*).





Figure 5.9: ARC6-2

The samples from Archontiko give less linear results and needed higher temperatures to reach the values needed for the anisotropy step and then saturation. Many samples had to be rejected, but the ones that gave trustworthy results seem to be in agreement with each other.



Figure 5.10: SKAA2



Figure 5.11: SX4-2

The samples from Phryctoriae show a better continuity in total, providing us with a clearer final image. For most, it took less steps in total to reach the saturation point needed for the anisotropy step and the last cooling process.

The arrays and diagrams of all samples are shown in the Appendices and were all created with the use of the freeware applications *"ThellierTool"* and *"RenArMag\_v3511"*.



After the completion of the experiments and analysis of the results, it is now possible to reach some conclusions, including a variety of information provided by the experiments. In the end, the results from each experiment were compared to each other, in this way a much more complete pattern was obtained.

# 6.1 Rock Magnetic Analysis

As mentioned in previous chapters, three different rock magnetic experiments were conducted in total, on samples from all studied areas. These experiments mostly provided information about the consistency and mineralogy of the samples.

An earlier archaeomagnetic study conducted on the last phase of Archontiko settlement (Late Bronze Age, c.1450BC; *Tema et al.,2012*) provided several rock magnetic results which are of interest if compared to the present ones. In spite of the 600-700 years separating the two phases, similarities are striking in all common experiments such as IRM acquisition and thermomagnetic analysis. In a subsequent publication (*Kondopoulou et al.,2017*) several additional experiments such as hysteresis curves and FORC diagrams on the same material allowed to look in detail in the magnetic characterisation of different layers within a single specimen. This procedure revealed important differences in the distribution of magnetic minerals within only 1.5 cm of thickness.

# 6.1.1 Isothermal Remanent Magnetisation

Through the IRM experiment, it is possible to determine a number of parameters on the magnetic mineralogical consistency of the samples. In total, five samples were studied with this method. Two samples came from Archontiko (ARC3-1 and ARC6-4) and three samples came from the Phryctoriae (SKAC1b, SKAE2a and SX2-1)

The two samples from Archontiko (ARC) show similar behaviour during the experiment. They seem to saturate in lower fields (around 300 mT), indicating the presence of low coercivity magnetic minerals (e.g., magnetite).

Unlike Archontiko, the diagrams of the samples from Phryctoriae, displayed a few differences when compared to each other. Samples SKAC1b and SX2-1 contain probably a mixture of magnetite and a small quantity of hematite, as the saturation is not complete until 1.2T while the sample SKAE2 seem to include mostly magnetite, as it is completely saturated.

In total, the main magnetic carrier in all samples is magnetite and other minerals of this same category, with only two samples showing evidence for the existence of hematite.

#### 6.1.2 Thermomagnetic Curves

Ψηφιακή συλλογή

The most important information acquired through the thermomagnetic measurements is the Curie point of a sample and this helps in the process of understanding a sample's consistency. Furthermore, this analysis allows to observe possible mineralogical transformations resulting from the heating-cooling process.

All samples from the Archontiko (ARC1 to ARC9) were studied through the thermomagnetic experiment, while from Phryctoriae only seven were examined (SKAB, SKAC, SKAE and SX1, SX2, SX4, SX6). The resulting diagrams placed the Curie temperatures between 580-600°C for most samples.

In the Archontiko samples' diagrams, the Curie temperature ranged between 570-600°C, indicating the presence of magnetite and maghemite in all of them. None of the samples gave diagrams that were completely reversible, while for all samples the heating line is above the cooling line, which means that there was no obvious creation of new minerals during the experiment.
While the Archontiko samples produced diagrams similar to each other, that was not the case for the Phryctoriae samples. The SKA and SX samples showed a bigger diversity when compared to each other. Their Curie temperatures were ranging between 530-590°C, which indicates the presence of magnetite in their consistency, with titanomagnetite or maghemite as the main magnetic carrier.

In contrast to the ARC samples, the Phryctoriae did have two samples with diagrams that showed good reversibility, the SKAB and the SX2. The heating line was above the cooling line for all the samples, with the only exception being the sample SKAE. This means that during the heating procedure of the SKAE sample, new minerals (most likely maghemite) were created.

The results of the thermomagnetic measurements and the IRM measurements seem to agree, both indicating big concentrations of magnetite, verifying each other's reliability.

## 6.1.3 Hysteresis Loops

Ψηφιακή συλλογή Βιβλιοθήκη

The diagrams of the hysteresis loops provide information about the type of magnetic domain behavior the magnetic material has and therefore the grain size of the samples.

Before the interpretation of the hysteresis loops, it would have been very helpful knowing the concentration of magnetic minerals inside the studied clays, so that a more detailed and verified conclusion could be taken. Unfortunately, this concentration was too small in the study's samples and was impossible to measure it, even with the use of more sensitive methods, such as X-Ray diffraction. Therefore, the results rely solely on the hysteresis loops diagrams.

For Archontiko, two samples were studied, ARC1 and ARC6. For the ARC1 sample, the magnetisation appears at 0.6Am<sup>2</sup>/kg, while for the ARC6 it is a little lower, at 0.45Am<sup>2</sup>/kg. Both of them have a coercive field of 15KA/m. Their curves seem to be relatively smooth,

so the clay probably consists of relatively clean magnetite. In case there is also a quantity of titanomagnetite in the samples, their titanium content is pretty low. Most likely, they both are of single domain or pseudo-single domain behaviour.

Ψηφιακή συλλογή Βιβλιοθήκη

From the Phryctoriae sites, four were examined and had their Hysteresis Loops' diagrams studied. These samples are SKAA, SKAC, SX1 and SX2.

The SKA samples displayed some interesting results. The SKAC sample shows a hysteresis loop that widens at intermediate applied fields (0.2T), while it is narrow at low fields. This can be explained by nucleation type procedures of magnetic domains in titanomagnetites grains or the presence of hematite, which often displays a peculiar and complicated behaviour. In both cases, the magnetic minerals are of multi-domain type. The SKAA sample provided a similar image to the SKAC, its behaviour being also that of a multi-domain sample.

Lastly, the SX samples displayed a great diversity between them. The SX1 gave a very slim loop and a saturation magnetisation of 0. 55 Am<sup>2</sup>/kg, compared to the wider loop the SX2 produced, with a 0.7 Am<sup>2</sup>/kg saturation magnetisation. The behaviour of SX1 is probably multi-domain, while this is hard to tell for SX2.

About the differences between the SX samples, there are two possible explanations. One would be that, even though the samples come from the same formation (the same Phryctoria), they were made out of different material. In big formations such as Phryctoriae, it was not rare for new material to be used to fix any damages caused by the usage and the passing of time.

Another explanation for this diversity could be that, since Phryctoriae had a larger surface and the fire was lit in its center, the samples come from two positions of different distances from the fire. One could have been from the center of the Phryctoria, where the fire stood, while the other could be from the sides or around the center, generally being heated in lower temperatures.

In conclusion, the Archontiko samples are of single or pseudo-single domain behaviour, while Phryctoriae are of multi-domain behaviour.

# 6.2 Archaeointensity Interpretation

Ψηφιακή συλλογή Βιβλιοθήκη

The longest experiment that was conducted for the purpose of this thesis was the Archaeointensity experiment (Thellier-Thellier).

In total, 41 specimens from 21 fragments were studied and measured. The success rate was 73.2%, since 30 of those specimens gave good quality measurements, while 11 were rejected for not meeting the required criteria.

While still in the early first steps of the experiment, some specimens showed inconsistencies and did not seem to belong to a linear array and instead were scattered on the plot. These specimens were rejected in the earlier stages of the measurement and did not reach the anisotropy steps.

After the final measurements were completed, the values of each specimen were analysed to make sure that all met the required criteria, previously mentioned in chapter 5.

Those criteria imposed the values for the Maximum Angle of Deviation (MAD) and the Deviation Angle (Dang) of each specimen, to be lower or equal to 10. Also, after the calculation of the F-cooling, the correction factor should be lower than 10%, while the alteration factor was expected to be lower than 5% (*Gomez-Paccard et al., 2006*).

The specimens that would not agree to these criteria, were also rejected (Table 6.1).

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	<u>-61-(</u>	) <sub>P</sub>	<u>AT</u>	0	Σ										
Y	1. Th	ήμα		ας				Mad						ΣF	F
Ľ	Sample	lab	(°C)	n	f	g	q	(°)	Dang	STD	Fraw	ΣF	F@	@	cooling
03	ARC1-1	55	200-530	11	0.86	0.86	11.9	8.39	3.67	0.06	52.70	3.28	56.18	5.27	50.38
	ARC1-2	55	200-550	6	0.45	0.76	3.9	7.69	7.09	0.09	52.46	4.61	48.00	2.99	42.03
	ARC1-3	55	150-570	6	0.94	0.89	12.2	6.62	3.8	0.07	55.05	3.80	55.18	3.58	50.65
	ARC2-1	55	200-530	9	0.79	0.84	18.3	5.31	9.31	0.04	48.75	1.77	49.99	1.88	45.46
	ARC2-3	55	250-505	7	0.63	0.81	37.9	5.41	8.85	0.01	56.22	0.76	57.73	0.94	53.77
	ARC3-2	55	350-530	5	0.45	0.74	16.1	6.65	9.8	0.02	51.70	1.07	47.08	2.42	41.93
	ARC4-3	55	0-550	12	0.91	0.84	19.4	9.72	4.71	0.04	49.69	1.95	50.04	2.08	45.64
	ARC4-4	55	200-530	9	0.71	0.79	18.3	14.49	6.11	0.03	49.11	1.51	43.91	2.50	41.72
	ARC5-1	55	250-480	6	0.61	0.79	14.2	5.02	9.97	0.03	58.09	1.97	57.07	2.27	51.64
	ARC6-1	55	0-570	12	0.7	0.86	19.8	7.59	1.83	0.03	57.85	1.76	61.33	2.09	58.43
	ARC6-2	55	300-570	9	0.65	0.85	18.6	8.82	1.27	0.03	56.58	1.69	65.49	1.52	56.92
	ARC6-3	55	300-530	7	0.51	0.73	10.3	13.25	1.82	0.04	50.54	1.83	54.90	3.35	52.67
	SKAA1	55	200-505	8	0.75	0.83	11.1	6.96	1.71	0.06	49.87	2.80	53.56	3.01	48.43
	SKAA2	55	0-505	11	0.9	0.89	29.1	9.06	2.23	0.03	48.61	1.34	55.10	1.60	50.41
	SKAB1	55	100-550	12	0.84	0.89	36.5	5.6	2.14	0.02	53.15	1.09	56.98	1.16	51.03
	SKAC1a	55	250-505	7	0.57	0.81	9.89	7.39	2.18	0.05	41.91	1.94	46.95	2.11	43.16
	SKAC2	55	200-480	7	0.59	0.82	10.2	3.71	2.7	0.05	41.41	1.98	41.53	1.94	36.47
	SKAC3a	55	200-505	8	0.7	0.85	25.1	6.56	0.98	0.02	39.05	0.92	45.40	1.47	42.30
	SKAC3b	55	200-505	9	0.75	0.86	38.7	6.84	4.57	0.02	43.84	0.73	45.10	0.65	41.56
	SKAC4a	55	250-505	7	0.57	0.81	11.7	6.26	2.31	0.04	42.35	1.69	46.80	2.18	41.53
	SKAC4b	55	250-505	7	0.73	0.83	28.6	5.45	1.19	0.02	39.34	0.83	39.38	0.78	39.38
	SKAD1	55	0-505	7	0.46	0.81	6.14	9.98	6.69	0.06	42.48	2.58	36.61	1.69	41.48
	SKAD3	55	0-505	11	0.83	0.87	23.9	4.98	4.26	0.03	46.40	1.40	46.56	1.38	46.56
	SKAE1	55	100-505	10	0.92	0.71	50.4	3.32	1.35	0.01	64.33	0.84	64.32	0.83	61.25
	SKAE2b	55	100-505	10	0.9	0.69	24.9	5.62	0.79	0.02	61.89	1.54	62.39	1.11	56.34
	SX1-1	55	200-400	5	0.47	0.72	10.3	6	10.7	0.03	<mark>41.88</mark>	1.38	<u>56.43</u>	1.96	52.18
	SX2-2a	55	150-505	9	0.74	0.85	12.7	2.2	0.42	0.05	50.42	2.48	64.29	2.93	55.86
	SX2-2b	55	150-505	9	0.77	0.85	8.3	2.99	1.92	0.08	47.45	3.75	47.10	3.54	42.54
	SX4-1	55	100-550	12	0.88	0.84	24.8	4.54	0.79	0.03	53.24	1.60	53.22	1.60	52.11
	SX4-2	55	440-550	5	0.65	0.75	9.59	4.13	1.92	0.05	53.31	2.68	53.35	2.70	51.96
	SX5-1	55	200-505	7	0.65	0.81	12.9	5.33	1.67	0.04	58.17	2.39	58.17	2.39	52.91

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Table 6.1: The results and values for each specimen of each sample. The yellow lines indicate samples that got rejected, once all measurements were complete, because they would not meet the required criteria.
 Sample: Sample's Code Name | Flab: Magnetic Field use in Laboratory | Tmin-Tmax: Temperatures of Successive Points in Linear Segment | n: Number of Successive Points in Linear Segment | f: Fraction of NRM | g: Gap Factor | q: Quality Factor | Mad: Maximum Angle of Deviation | Dang: Deviation Angle | STD: Standard Deviation | Fraw: Intensity Measured | ΣF: Intensity Deviation | F@: Intensity after adding the Anisotropy | ΣF@: Deviation of Intensity after adding the Anisotropy | Fcooling: Intensity after Adding the Cooling Measurement

After the measurement of all specimens, the average for each sample was calculated, along with the respective STD (Table 6.2). The specimen taken from the Archontiko

belonged to six different samples, with one sample being completely rejected in the end (ARC3). For Phryctoriae the total samples were nine, with one being rejected after the

	Flab				Mad							
Specimen	()	f	g	q	(°)	Dang	STD	Fraw	ΣF	F@	ΣF@	Fcooling
ARC1	55	0.75	0.84	9.33	7.5667	4.85333	0.07	53.40	3.90	53.12	3.95	50.51
ARC2	55	0.63	0.81	37.9	5.41	8.85	0.01	56.22	0.76	53.86	0.94	49.61
ARC3	55	0.45	0.74	16.1	6.65	9.8	0.02	51.70	1.07	47.08	2.42	
ARC4	55	0.81	0.81	18.9	12.105	5.41	0.03	49.40	1.73	46.98	2.29	43.68
ARC5	55	0.61	0.79	14.2	5.02	9.97	0.03	58.09	1.97	57.07	2.27	51.64
ARC6	55	0.62	0.81	16.2	9.8867	1.64	0.03	54.99	1.76	60.57	2.32	56.01
SKAA	55	0.82	0.86	20.1	8.01	1.97	0.04	49.24	2.07	54.33	2.31	49.42
SKAB	55	0.84	0.89	36.5	5.6	2.14	0.02	53.15	1.09	56.98	1.16	51.03
SKAC	55	0.65	0.83	20.7	6.035	2.32167	0.03	41.32	1.35	44.19	1.52	40.73
SKAD	55	0.65	0.84	15	7.48	5.475	0.05	44.44	1.99	41.59	1.54	46.56
SKAE	55	0.91	0.7	37.7	4.47	1.07	0.02	63.11	1.19	63.36	0.97	58.80
SX1	55	0.47	0.72	10.3	6	10.71	0.03	41.88	1.38	56.43	1.96	
SX2	55	0.75	0.85	10.5	2.595	1.17	0.06	48.94	3.12	55.70	3.24	49.20
SX4	55	0.76	0.79	17.2	4.335	1.355	0.04	53.28	2.14	53.29	2.15	52.03
SX5	55	0.65	0.81	12.9	5.33	1.67	0.04	58.17	2.39	58.17	2.39	52.91

final examination of the results (SX1).

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*Table 6.2*: The average values for each sample, based on the individual measurements of each specimen.

The final and most important part of the experiment, was the calculation of the average intensity F for each horizon and area.

	Geographical Coordinates	Samples	F Average (μT)
ARC		ARCZ ARCS ARCS	52.42
Horizon II	40 70%4 22 47%5	Anez, Anes, Anes	STD: 3.27
ARC Horizon III	40.79 N 22.47 L	ARC1, ARC4	47.10
			STD: 4.83
SKA		ΔΙΙ ΣΚΔ	49.31
5114	25 71°N 75 78°E		STD: 6.60
sx	55.24 N 25.26 L	All SX	51.38
37			STD: 1.94

Table 6.3: The average intensity F for each group of samples, based on the area they were discovered and their archaeological dating

Based on the archaeological findings, the ceramic fragments from Archontiko that were examined belonged to two different phases. The samples ARC1 and ARC4 gave an average F=47.10  $\mu$ T, both representing Horizon III, while the samples ARC2, ARC5 and ARC6 represented Horizon II and gave an average F=52.42  $\mu$ T. The Phryctoriae samples also provided two different intensities, the SKA samples giving an average F=49.31  $\mu$ T and the SX samples giving an average of F=51.38  $\mu$ T (*Table 6.3*).

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Ceramics from Archontiko's last phase were previously studied for archaeointensity as well as for their magnetic characteristics (*Tema et al, 2012; Kondopoulou et al.,2017*) and a comparison of these results could provide some interesting features. Once again, the samples' behaviour during the Thellier experiment is almost identical: the successful fragments from the *Tema et al.,2012* study are N=3 (with n>/2) and another N=3 with n=1. Here we calculated N=4(n>/2) and N=1 with n=1. The success rate was exactly the same than in our present study, varying between 25-75%. In a detailed cross-section of some fragments from these previous studies, a pronounced inhomogeneity of their magnetic content was observed. Also, the firing temperatures as calculated through XRD experiments varied from low (550 °C) to medium/high ones (750-800 °C). From this observation we can safely deduce that the clays used in both phases were of the same origin and, moreover, the firing techniques did not differ much. Therefore, there is ground to support a continuous technology in the preparation of ceramics within the whole period between 2000-1500B.C.

Many methods have been used for the purpose of dating samples from the areas of Archontiko and the Phryctoriae (*Table 6.4*). By using the most reliable dates provided through these methods, it is possible to position the resulting average intensities on the Greek SVC.

Sample	F	F average	ΣF	Archeological Dating	Radiometric	Luminescence
ARC1	50.5	47.4	4.00	Late Bronze Era	2005 1002 DC	
ARC4	43.7	47.1	4.83	(Horizon III)	2003-1903 BC	
ARC2	49.6	52.4	3.27		1923-1877 BC	

N N	GE	<sup>νηφιακ</sup> βιβλ	ιή συλλογή ΙΟθήκΓ ΡΑΣΤ	ΟΣ'	%		
3	ARC5	51.6	Γεωλονί	ac	2300-1900 BC		
X	ARC6	56	Π.Θ		(Horizon II)		
0	SKAA	49.4					
	SKAB	51				1493 (±224) BC	1531 (1177)
	SKAC	40.7	49.3	6.6		1549 (±273) BC	1521 (±177) BC
	SKAD	46.6			Late Bronze Era		
	SKAE	58.8			1900-1700 BC		
	SX2	49.2				1713 ±259	1001 (1102)
	SX4	52	51.4	1.94			1691 (±162) BC
	SX5	52.9					20

Table 6.4: Dating of the samples through other technics

The archaeological dating of Archontiko placed phase II between 2300-1900 BC and phase III in the Late Bronze Era, while the first radiometric dating provided an estimated dating of 2300-1600 BC for the same samples. With the use of Bayesian Distribution, Horizon II of Archontiko was dated between 1923-1877 BC and Horizon III was estimated to be between 2085-1903 BC Since the early radiometric dates showed high STD, the most acceptable dates are the dates calculated with the help of the Bayesian Model (*Maniatis, 2012*).

The Phryctoriae are believed by archaeologists to be from the Late Bronze Era, between 1900-1700 BC A luminescence study on these specimens, estimated an average dating at 1521 BC for the SKA samples and an average dating at 1691 BC for the SX samples, but with high STD. Since the archaeologists believe that the structures cannot be younger than 1700 BC, the archaeological dating is accepted as the most reliable one, while the OSL dating is taken into account only as a confirmation.

Placing the results on the Greek SVC, they seem to agree with the estimated curve created on the basis of the previous archaeomagnetic studies that were conducted in Greece so far and especially close to some of the highest quality data from after 2008 for the same period (SKS point) *(figure 6.1)*.



*Figure 6.1:* The Greek intensity SVC, updated with the newly studied samples. ARC1-Archontiko Horizon III | ARC2-Archontiko Horizon II | SKA/SX-Phryctoriae

Sometimes, environmental studies on the area of interest for the respective time periods can also provide valuable information that can help in the explanation of archaeomagnetic results. Such a study on the palaeogeographical changes in the area of Archontiko was conducted in 2009 (*Syrides G. et al. 2009*). It was concluded that at around ~2000 B.C., a paleoenvironmental change took place and the waterline of a near lake came the closest to Archontiko. This rapid change can possibly explain a change in the magnetic values, limited in this specific area.

Apart from the Greek SVC, to verify the trustworthiness of the results, it is helpful to compare the new results to other SVCs from neighboring countries, that have been updated in the very recent years.

A good reference SVC for the results could be the Bulgarian SVC. For this comparison, the intensities of the Greek samples are relocated from Thessaloniki to Sofia and are compared to other samples that represent the Bulgarian SVC for the years of interest (*Kovacheva M., et al., 2014*) (*Table 6.5*).

Ψηφιακή συλλογή Βιβλιοθήκη "ΘΕΟΦΡΑΣΤΟΣ"									
Martin	Sample	ογίας Age	F Average	Relocated Intensity (μT)					
0	ARC1	2085-1903 BC	47.10	48.11					
	ARC2	1923-1877 BC	52.42	53.55					
	SKA	1000 1700 BC	49.31	53.74					
	SX	1900-1100 BC	51.38	55.99					

Table 6.5: Greek Archaeomagnetic samples with relocated intensity from Thessaloniki to Sofia

The intensity of the Bulgarian samples that belong to sites dated within 2350-1500 B.C., is from 49.49 to 68.40 (*Table 6.6*).

Sample	Age	Intensity (µT)
84	1500 BC (1600-1400 BC)	68.40
321	1775 BC (1800-1750 BC)	60.57
85	1800 BC (1900-1700 BC)	60.21
77	2025 BC (2100-1950 BC)	63.30
79	2350 BC (2400-2300 BC)	53.87
78	2383 BC (2559-2204 BC)	49.49

**Table 6.6:** Archaeomagnetic samples from Bulgarian database for the years of interest (*Kovacheva M. et al., 2014*) The intensities from the SVC of Bulgaria for the Bronze Era seem to be a little higher than the Greek ones (*figure 6.2*). Despite this deviation, the fluctuation of the intensity values is similar in both cases, indicating an increasing trend from 2000 BC to 1700 BC. It should be mentioned here, that for the construction of the Bulgarian curve in this specific time interval (2000-1700 BC) only three available intensity data were used as it seen in *Table 6.6*. Therefore, the curve for this period is not so reliable



*Figure 6.2:* Comparison of the results of this study with the Bulgarian curve (Kovacheva et al., 2014). All intensity values are relocated to Sofia coordinates.

Another comparison could be made with the data base from Eastern Mediterranean countries (Turkey, Cyprus, Israel). In a recent compilation by *Ertepinar et al., (2020)* all data from the above countries are plotted and compared with the SHA.DIF.14k geomagnetic model. In order to compare our results with these databases we calculate the VDAM values as it shown in *Table 6.7*.

	Age	F Average	VDAM (ZAm2)
ARC1	2085-1903 BC	47.10	80.7
ARC2	1923-1877 BC	52.42	89.8
SKA	1000 1700 PC	49.31	90.2
SX	1900-1700 BC	51.38	94

Table 6.7: Conversion of each average intensity to VADM



**Figure 6.3**: Site mean VADMs of potsherds (red diamonds) and mud-bricks (red circles) along with the data from Geomagia50.v3.2 (gray), the Middle East, Cyprus, Georgia (orange), and Turkey (blue and pink) and the global field model SHA.DIF.14k (green), sets with one successful measurement (dark green, transparent). (Ertepinar et al. 2019) This study's data are the blue squares.

Placing the converted intensities on the intensity SVC model SHA.DIF.14k the Greek points seem to be in a good agreement with the model's trend for the years during the Bronze Era *(figure 6.3)*, and especially the results from Archontiko are positioned very close to the curve. The Phryctoriae results fall on a spot where there are not many measurements, so the comparison is a little less clear. Regardless, there is an agreement between our results and the SVC, therefore the results seem to be quite trustworthy.

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Finally, the obtained results from this study have a great contribution on our effort to fill the gaps on the Greek SVC, as they correspond to a time span that, so far, has not been sufficiently studied neither for Greece, nor for the Balkans. Hopefully, the new intensity data which are still in progress, will providing us a chance to cover the space on the SVC even further.



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**APPENDICES** 

Thellier-Thellier Results for Samples:

























Figure B1: SKAA1



Figure B2: SKAA2

### 101



Figure B3: SKAB1

### 102



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Figure B12: SKAE1





Figure C1: SX2-2A





Figure C3: SX4-1











Figure D3: ARC3-2



## 121



Figure D5: ARC4-1







Figure D7: ARC5-2





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Figure D10: ARC8-1



Figure D11: ARC8-2



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