Methods in Rock Magnetism and Palaeomagnetism

Techniques and instrumentation

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Preface

During the last 30 years the study of the magnetic properties of rocks and minerals has substantially contributed to several fields of science. Perhaps the best known and most significant advances have resulted from the study of palaeomagnetism, which led to quantitative confirmation of continental drift and polar wandering through interpretation of the direction of remanent magnetism observed in rocks of different ages from different continents. Palaeomagnetism has also, through observations of reversals of magnetization, ancient secular variation and ancient field intensities provided data relevant to the origin of the geomagnetic field, and other investigations have contributed significantly to large-scale and local geological studies, the dating of archaeological events and artefacts and more recently to lunar and meteoritic studies. Rock and mineral magnetism has proved to be an interesting study in its own right through the complex magnetic properties and interactions observed in the iron–titanium oxide and iron sulphide minerals, as well as contributing to our understanding of remanent magnetism and magnetization processes in rocks.

Simultaneous with the development of these studies has been the development of instruments and techniques for the wide range of investigations involved.

The need for reliable measurements of the remanent magnetization of more weakly magnetized rocks has resulted in the development of sensitive magnetometers and improvements in the techniques for the removal of unwanted magnetizations. Much effort has been expended in improving methods for deriving ancient magnetic field intensities from terrestrial and extraterrestrial rocks and in devising instruments and techniques for identifying and characterizing magnetic minerals. Along with these developments have been those concerning new methods of presentation of palaeomagnetic data and of techniques for deriving the most reliable estimate of ancient magnetic field directions and pole positions.

Details of many of these instruments and techniques are in the literature but scattered rather widely, and others remain unpublished yet are deserving of wider knowledge. It is the purpose of this book to bring together for easy reference the range of instruments and techniques currently in use, and also to describe some of those used in the past and now less used but which may still be of interest and of use for particular investigations. It is hoped that the book will be useful both to those already working in the subject and to those with either a geological or physical background embarking on studies of any of the many aspects of the magnetism of rocks.

Although equipment for routine palaeomagnetic and rock-magnetic studies is now commercially available, information is given in the book to enable such equipment to be built in the laboratory and also more specialized equipment for particular studies. The emphasis is on the basic principles of the various instruments and techniques. Signal processing and the use of computers and microprocessors is not described in detail as information on these aspects of instrumentation is available from other sources.

Workers in palaeomagnetism and rock magnetism have been among the most reluctant converts to SI units, but an increasing number are now using them. In the author's opinion the use of c.g.s. is now no longer justified and SI units are used in the book. A table of conversion factors between c.g.s. and SI units is given in Appendix 1, together with some notes on the SI units used in the book.

I am grateful to many of my colleagues both in this country and abroad for discussion and information on various topics. Particular thanks are due to M. J. Gross, F. J. Lowes, L. Molyneux, J. H. Parry, W. O'Reilly, J. S. Rathore, P. V. Sharma, A. de Sa, A. Stephenson, M. Stupavsky and D. H. Tarling. I am grateful to Mrs L. Boon, Miss S. Bell, Miss A. Codling and Mrs L. Whiteford for jointly typing the manuscript and to Mrs D. Cooper for drawing the diagrams.

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Chapter One

Rock magnetism and magnetic minerals

1.1 Introduction

Rock magnetism is the term commonly applied to the study of the magnetic properties of rocks and minerals, how these properties depend on factors such as grain size and shape, temperature and pressure, and the origin and characteristics of the different types of remanent magnetizations which rocks and magnetic minerals can acquire. Although all minerals possess some magnetic properties, even if only paramagnetic or diamagnetic, the term 'magnetic minerals' is used here only for those minerals which are capable of carrying remanent magnetism.

Rock magnetic investigations and techniques are basically an extension to rocks and minerals of studies of the classical magnetic materials, namely iron and other transition elements and their alloys. However, metallic iron is only very rarely encountered in terrestrial rocks (although it is the dominant magnetic mineral in lunar samples and some meteorites), and it is the ferrimagnetic and antiferromagnetic iron–titanium oxide minerals which are of chief concern in and add interest and complexity to rock magnetism. In addition, composite minerals of the titanomagnetite and titanohaematite series, in which there is a gradation in or discontinuous change of magnetic properties with composition, and the presence of particles covering a wide range of grain sizes associated with superparamagnetic, single domain and multidomain behaviour are other factors which contribute a variety of interesting phenomena.

Although rock magnetism is an interesting and informative study in its own right, many investigations are directed towards identifying the mineral(s) carrying the natural remanent magnetization (NRM) of rocks and towards a better understanding of the origin of the NRM and its reliability as a palaeomagnetic indicator.

The magnetic mineral content of rocks is typically only 1–5 % by weight and it may not be easy to separate them out in a pure form for investigation, particularly if they are fine grained. An alternative approach pursued by several workers is the investigation of laboratory-prepared minerals, notably the titanomagnetite series and haematite. One result of this work has been to emphasize the dependence of magnetic properties on the presence of small amounts of impurities, non-stoichiometry and, particularly in the case of haematite, the mode of formation of the mineral.

The magnetic properties of rocks, including their NRM, can also often provide information on their formation and history and on the constituent minerals and their physical state. For example, the temperature to which the country rock is raised during intrusion of a nearby dyke or sill can be estimated from the blocking temperature of the PTRM (partial thermoremanent magnetization) acquired, and magnetite can be detected in rocks by magnetic measurements at a level (0.01–0.001 %) well below that detectable by conventional methods such as X-ray diffraction or optical reflectance. These and other applications, including determination of the intensity of the ancient geomagnetic field, are described later in this book.

A branch of rock magnetism of potential practical use is the effect of pressure on magnetic properties as a possible indicator for earthquake prediction. The principle is the surface observation of changes in the magnetic anomaly pattern due to a build-up of stress in the underlying rocks (Stacey, 1964). Depending on the Königsberger ratio of the rocks (Section 10.2), changes in either initial susceptibility or remanent magnetism could be the cause of a changed anomaly pattern. In a similar way, changes in induced or permanent magnetism of crustal rocks arising from temperature and pressure changes due to movement of magma have been proposed as an indicator for predicting volcanic eruptions (Stacey, Barr and Robson, 1965).

Although the study of the magnetism of meteorites has been mainly concerned with their NRM and its origin, they are of some interest for rock magnetism because of the contribution of iron and iron–nickel (as well as magnetite) to their magnetism. An understanding of the magnetic properties of the former two materials and their variation with temperature is clearly necessary for a further understanding of meteoritic NRM and its implications.

A recent development in rock magnetism is the investigation of the magnetic properties of lunar rocks and dust returned by the *Apollo* missions of 1969–72 and the unmanned Russian *Luna* landing and sample return programme. The presence of iron (or dilute nickel–iron) in a wide range of grain sizes as the dominant magnetic mineral, uncertainty as to the origin of the iron in some rocks and of some of the rocks themselves (the breccias) and the limited amount of material available for investigation all combine to make lunar rock magnetism a fascinating and demanding study. For an overview of the field the reader is referred to the review by Fuller (1974), and for more detailed accounts to the proceedings of the annual lunar science conferences, published as supplements of *Geochimica et Cosmochimica Acta.*

Some of the instruments and techniques employed in rock magnetism were developed from those used in classical magnetic studies and some have been developed specifically for use in rock magnetism. In the former category are various types of Faraday balance and vibration magnetometer for the measurement of induced magnetization, high field susceptibility and Curie points, while the phenomena of rotational hysteresis and piezomagnetism have been mainly studied only in rocks and minerals and have required the development of new instrumentation for their successful investigation. The need for this stems partly from the greater sensitivity often required for measurements on rock samples compared with other magnetic minerals, either because of the weak magnetic properties of many rocks or the availability of only restricted amounts of separated minerals.

1.2 Magnetic minerals

In this section a brief account is given of the minerals of chief interest in the rock magnetism and palaeomagnetism of terrestrial and extraterrestrial material. It is not intended as a comprehensive description of the minerals and their properties, but rather as a guide to the occurrence and abundance of magnetic and other minerals of interest in rocks, their most important magnetic properties, and other properties relevant to magnetic studies. The references given are intended to guide the reader to the literature, where more detailed information and accounts of relevant research are available. Useful general references to the mineralogy of rock magnetism are Nicholls (1955), Nagata (1961) and O'Reilly (1976).

For those who are not familiar with them, the terms used to describe magnetic properties are defined in Section 3.1.

1.2.1 Iron and nickel–iron

(*a*) *Iron*

Native iron is rare in terrestrial rocks, but is the most important magnetic constituent of many meteorites and all lunar rocks. In these materials it occurs either in the pure form or with a varying nickel content. A terrestrial occurrence in some Newfoundland ophiolites is reported by Deutsch, Rao, Laurent and Seguin (1972), based on studies of their magnetic properties.

Iron exists in at least two allotropic forms, one of which is the body-centred cubic structure (α-iron), stable at room temperature and up to 910°C. The Curie point of iron is 778°C, above which it is paramagnetic, and between 778° and 910°C it is known as β-iron although there is no structural change from the α-form. Above 910°C the structure is a face-centred cubic lattice (γ-iron) (Adcock and Bristow, 1935). The relative density of iron is 7.85, and its electrical conductivity is $1.0 \times 10^3\,\Omega^{-1}\,\mathrm{m}^{-1}$.

Iron is the classic ferromagnetic material, in which neighbouring atomic moments are aligned parallel to one another by strong exchange forces. Its spontaneous (saturation) magnetization is 218 $\mathrm{A\,m^2\,kg^{-1}}$. The intrinsic susceptibility is very high ($\sim 10^{-2}\,\mathrm{m^3\,kg^{-1}}$) but the effective initial susceptibility of an assembly of non-interacting grains (as occurs in lunar rocks or stony meteorites) is governed by their demagnetizing factor and is typically $\sim 5 \times 10^{-4}\,\mathrm{m^3\,kg^{-1}}$ (Section 2.3).

Iron is magnetically anisotropic with easy and hard directions of magnetization along the (100) (cube edge) and (111) (cube diagonal) respectively. The coercive force of large multidomain grains is very low (1–5 mT), but is much higher in single domain and small multidomain particles. Néel (1949) derived theoretical values for the superparamagnetic–single domain and single domain–multidomain boundaries in spherical grains of 160 Å (0.016 μm) and 320 Å (0.032 μm) respectively, but Butler and Banerjee (1975a) and Wasilewski (1981) suggest there is no stable single-domain size range for spherical grains, but may be for elongated grains.

The magnetism of iron and its alloys and their many industrial applications have been comprehensively documented by Bozorth (1951) and in the electrical engineering literature, in particular in the *Transactions of the Institute of Electrical and Electronic Engineers* (*Magnetics*). Research into the magnetic properties of finely divided iron has been promoted through the recent interest in the magnetism of lunar rocks and dust returned by the American *Apollo* and Russian *Luna* missions. These materials contain grains of iron (or dilute nickel–iron) up to ~1 mm in diameter which often span the superparamagnetic to multidomain range and carry remanent magnetism with a wide range of stabilities. The grain-size distribution of iron in lunar dust has been examined through magnetic studies by Stephenson (1971b), Dunlop *et al.* (1973) and Gose and Carnes (1973).

Studies of laboratory-prepared analogues of lunar iron are described by Pearce, Hoye, Strangway, Walker and Taylor (1976). The reader is also referred to papers in the proceedings of the lunar science conferences, published annually from 1970, in which results of lunar magnetic studies are reported, and to the review by Fuller (1974).

(*b*) *Nickel–iron*

Indigenous nickel–iron alloys are found only very rarely on Earth, but are an important constituent of some meteorites and often contribute the major part of their magnetic properties. Nickel is ferromagnetic with a saturation magnetization of 57 $\mathrm{A\,m^2\,kg^{-1}}$ and a Curie point of 357°C. α-Iron can hold up to ~ 20 % of nickel (which has a face-centred cubic structure) in the lattice, and this form of nickel–iron in meteorites, with typically 5–10 % of nickel, is known as kamacite: nickel–iron with a higher proportion of nickel is termed taenite, which has the face-centred cubic structure of γ-iron. Plessite is an intimate mixture of kamacite and taenite crystallites which occurs in some meteorites.

The nickel–iron content of meteorites is typically ~100 % in the irons, ~50 % in the stony irons, 5–25 % in the chondrites and ~1 % or less in the achondrites. Of the irons, the octahedrites, with an average 8% Ni, contain kamacite and subsidiary taenite and show the characteristic triangular Widmannstätten pattern (intersecting plates of kamacite with taenite margins) on etching a polished surface: the hexahedrites, with typically 5% Ni contain kamacite only and show a system of lines (Neumann lines) on etching. The cobalt content of meteoritic metal is usually 5–15% of the nickel content. Among the comprehensive texts and reviews on meteorites, that of Wasson (1974) contains a summary of their magnetic properties.

The magnetic properties of nickel–iron show a marked discontinuity at ~27% (atomic) nickel content, at which composition the alloy essentially loses its ferromagnetic properties. The apparent Curie point of the alloys also varies in a complex way with nickel content, but there is a distinction to be made here between a true Curie point and the $\alpha \rightarrow \gamma$ transition temperature, above which alloys with 27% Ni are paramagnetic. The effect of increasing nickel content in iron is to steadily lower the transition temperature (910°C in pure iron) until at a nickel content of ~4–5% it is just below the iron Curie point of 778°C (Pickles and Sucksmith, 1940). Thus, although alloys in the range 5–27% Ni lose their ferromagnetic properties at lower temperatures (e.g. 705°C and 625°C at 9% and 16% Ni respectively) these are not true Curie points. Among other features, the $\alpha \rightarrow \gamma$ transition in these alloys is not thermally reversible, i.e. on cooling, the $\gamma \rightarrow \alpha$ transition occurs at a lower temperature. With a nickel content in the range ~30–50% there is a true Curie point in γ-phase taenite (Hoselitz and Sucksmith, 1943).

Because of the above effects and the wide range of grain size of the nickel iron in meteorites, considerable variation in their magnetic properties is observed. As expected, the remanent magnetism of the irons is of low stability because of the coarsely crystalline nature of the metal. Some of the chondrites show a rather stable remanence carried by kamacite, for example the Cook and Farrington meteorites (Stacey, Lovering and Parry, 1961) and the Al Rais meteorite (Watson, Larson, Herndon and Rowe, 1975): these chondrites also exhibit the thermal hysteresis associated with the $\gamma \rightarrow \alpha$ transition in kamacite. The stability of the remanence suggests the presence of very fine particles or perhaps a microstructure in the nickel–iron. Stacey *et al.* (1961) point out that if the meteorites acquired their magnetic remanence by cooling in an ambient field, then this remanence may in fact be a type of CRM (chemical remanent magnetization) rather than TRM (thermoremanent magnetization) if it was acquired at the $\gamma \rightarrow \alpha$ transition rather than by cooling through a true Curie point or blocking temperature. Such a magnetization process may also have implications for determinations of the field intensity in which the meteorite remanence was acquired.

1.2.2 The titanomagnetite series

This important series of magnetic minerals consists of solid solutions or intergrowths of different compositions of the end members, magnetite

(Fe_3O_4) and ulvöspinel (Fe_2TiO_4). Magnetite-rich to approximately equimolecular compositions are important in palaeomagnetism as the carriers of NRM in a wide range of igneous rocks, some sedimentary rocks (e.g. limestones) and varved clays and lake and sea-bottom sediments, and magnetite is the magnetic constituent of some meteorites.

(*a*) *Magnetite* (Fe_3O_4)

Magnetite is a cubic mineral with the inverse spinel structure, i.e. the cations occupy two different lattices, A and B, in the crystal, with Fe^{2+} and Fe^{3+} ions in the latter and Fe^{3+} only in the former. In the normal spinel structure, divalent cations are on one lattice and trivalent cations on the other. In magnetite there are two cations on B sites for each one on A and the atomic moments are oppositely directed. Thus the unit cell has a net magnetic moment: this is ferrimagnetism, of which magnetite is one of the best-known examples.

Magnetite is a dark black mineral with a theoretical relative density of 5.20, although natural samples lie in the range 5.16–5.22. It is optically isotropic in polished section, with a reflectivity of $\sim 21\%$ in air: the electrical conductivity of natural magnetite is very variable, but is commonly in the range $\sim 10^2$–$10^4\ \Omega^{-1}\ m^{-1}$ (Parkhomenko, 1967).

With the exception of iron, magnetite has the strongest magnetic properties, with a saturation magnetization variously quoted in the range 90–93 $A\ m^2\ kg^{-1}$: its Curie point is 578°C. It is magnetically anisotropic with easy and hard directions of magnetization along (111) and (100) respectively. Based on the theory of Néel (1955), the single domain–multidomain boundary for spherical grains is at about 280 Å diameter: this is close to the value reported for the superparamagnetic boundary (Stacey, 1963; Dunlop, 1973a), i.e. the diameter below which particles are rendered magnetically unstable through thermal agitation (Section 2.4). Thus the direct transition from multidomain to superparamagnetic behaviour in equidimensional magnetite particles appears to be a possibility, and stable NRM may be carried by elongated grains or pseudo-single-domain grains (Stacey, 1963; Butler and Banerjee, 1975b). The intrinsic susceptibility of magnetite is very high ($\sim 10^{-2}\ m^3\ kg^{-1}$) but the effective initial susceptibility of an assembly of non-interacting grains (i.e. as in a rock sample), as with iron, is governed by their demagnetizing factor and is typically $\sim 5.0 \times 10^{-4}\ m^3\ kg^{-1}$ (Section 2.3).

The coercive force of multidomain magnetite is low, usually in the range 2–20 mT, and the coercivity of remanence usually lies in the range 10–50 mT. Both these properties are particle-size dependent, their magnitude increasing with decreasing size (Stacey, 1963; Parry, 1965; Dunlop, 1973b). Saturation magnetization is achieved in fields of 50–150 mT with a saturation remanence of typically 1–20 $A\ m^2\ kg^{-1}$, again dependent on grain size.

Between about −145°C and −155°C magnetite undergoes a structural change from the cubic to orthorhombic form (the Verwey transition), and marked changes in mechanical and electrical properties occur (Verwey and Haayman, 1941). This transition occurs in the same temperature range in which K_1, the first magnetocrystalline anisotropy constant, passes through zero when magnetite is cooled from room temperature, and it is likely that the two phenomena are connected (O'Reilly, 1976).

(*b*) *Ulvöspinel* (Fe_2TiO_4)

Ulvöspinel (sometimes known as ulvite) is the other end member of the titanomagnetite series. Like magnetite it has the inverse spinel structure, but the Fe^{3+} cations on the A and B sites in magnetite are replaced by Fe^{2+} and Ti^{4+} respectively. Thus, since Ti^{4+} contributes no magnetic moment, and the Fe^{2+} cations on each lattice are oppositely directed, ulvöspinel is weakly ferrimagnetic with a Néel temperature (the temperature above which the atomic ordering is destroyed) of 120 K (-153°C). It is paramagnetic at room temperature, with a theoretical susceptibility of $\sim 1 \times 10^{-6}\ \mathrm{m^3\,kg^{-1}}$, based on its ferrous iron content.

Ulvöspinel is rare in terrestrial rocks, occurring chiefly as exsolution blebs or lamellae in some magnetite ores: there is some magnetic evidence of its occurrence in lunar rocks (Runcorn, Collinson, O'Reilly, Stephenson, Battey, Manson and Readman, 1971). Its name is taken from Södra Ulvön, in northern Sweden, where a magnetite ore containing an estimated 52% of discrete ulvöspinel is found (Deer, Howie and Zussman, 1962). It has an X-ray relative density of 4.7–4.8, and can be synthesized by sintering an intimate mixture of the Fe_2O_3 and TiO_2, or produced from a melt (Hauptman and Stephenson, 1968). It is optically isotropic, with a reflectivity slightly less than that of magnetite. Ulvöspinel oxidizes to ilmenite and magnetite: the ilmenite lamellae are parallel to (111) in the magnetite, whereas those of ulvöspinel in magnetite ores are parallel to (100) (Ramdohr, 1969).

(*c*) *Titanomagnetites* ($x\mathrm{Fe_2TiO_4}\cdot(1-x)\mathrm{Fe_3O_4}$)

Complete solid solution of magnetite and ulvöspinel only occurs above 600°C and below this temperature a varying degree of exsolution takes place, resulting in a structure of intergrown lamellae except in a restricted range of compositions near $x = 0$ and $x = 1.0$ (Basta, 1960).

There is a steady gradation of properties with change in bulk composition. The Curie temperature decreases almost linearly with increasing x, from 578°C for $x = 0$ to -153°C for ulvöspinel ($x = 1$). At $x \simeq 0.80$, the Curie point is at room temperature. Other magnetic properties behave in a similar but less linear fashion, e.g. saturation magnetization and saturation remanence. Although measurements have not been reported, initial susceptibility may be expected to change with x in a rather complex way. With high magnetite content, susceptibility will be governed by the particle demagnetizing factor (Section 2.3), and be essentially constant. As x increases, the intrinsic susceptibility of a particle decreases and the demagnetizing effect assumes less