

Techniques of magnetic measurements

It is a capital mistake to theorize before one has data.

Sir Arthur Conan Doyle,
Scandal in Bohemia

6.1 Introduction

Following the electronics revolution, particularly the production of integrated circuits and microcomputers, instrumentation for magnetic measurements has improved dramatically. Advances have included increases in sensitivity, speed of measurement, portability, availability and simplicity of operation. Magnetic susceptibility equipment is now commercially available at reasonable cost largely owing to the technology developed for the amateur metal-detecting market. Such commercial instruments are now more sensitive, reliable and accurate than many which were purpose built for use in palaeomagnetic research laboratories. Being battery operated the commercial instruments are not tied to the model conditions of the laboratory bench and are ideal for field surveys. Furthermore, their digital displays, electronic calibration and automatic push button zeroing enable reliable susceptibility measurements to be made at a rate of several per minute by inexperienced users. Apparatus for magnetic remanence measurements has similarly been greatly simplified in terms of convenience and speed of operation. These various electronic advances in instrumentation have been fundamental to the recent increase in scope of application of magnetic investigations to environmental projects and especially to the use of magnetic

measurements as a preliminary reconnaissance tool at the start of site investigations. Magnetic instrumentation has thus become attractive to many earth scientists and environmentalists and is no longer solely used by the palaeomagnetic specialists.

Not all magnetic instrumentation has been streamlined by commercial attention. So on entering a magnetic laboratory one may still be faced by a bewildering array of large coils, magnets, cables, furnaces, dewars and spinning shafts and be tempted to reflect that an apt synonym for palaeomagnetism is indeed palaeomagic. Nevertheless the bulk of the environmental magnetic measurements discussed in Chapters 8–16 have been made on just two types of instrument. These are the susceptibility bridge of Section 6.3.1 and the fluxgate magnetometer of Section 6.2.3. Both instruments are very simple, extremely quick to use and are to be found in most palaeomagnetic research laboratories. A third important piece of equipment is either a pulse discharge magnetiser (Section 6.6.2) or an electromagnet (Section 6.6.2). Electromagnets or pulse discharge units are needed for the generation of high magnetic fields such as those used in the study of isothermal remanence. Again such apparatus is relatively simple to use, and exceptionally fast in operation; hundreds of magnetically saturated samples can be produced in an hour.

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Table 6.1 Uses of instrumentation.

Subject of investigation	Magnetic property	Section	Example of application	Chapter	Instrumentation	Section
concentration	initial susceptibility	4.4	DSDP core 514	12	susceptibility	6.3
concentration	saturation magnetisation	4.3.2	N. Atlantic sediment cores	12	induced magnetisation	6.4
mineralogy	magnetite, haematite	3.2	sources of suspended sediments	9	remance/field generation	6.2/6.6
domain state	hysteresis, coercivity	4.6	Plynlimon bedload tracing experiment	9	induced magnetisation	6.4
viscosity	superparamagnetism	4.3.2	enhanced soils on archaeological sites	8	pulsed induction meter	6.7.3
temperature dependence	Curie temperature	4.8	atmospheric fallout in the English midlands	11	Curie balance	6.4.1
natural remanence	thermoremanence	4.3.1	Iceland lava flows	13	remance	6.2
multicomponent remanence	demagnetisation	2.5	Gass lake drying remanence	14	magnetic cleaning	6.5

There are a wide variety of experimental techniques available to the investigator of the magnetic properties of minerals. A comprehensive and detailed review of them all is beyond the scope of this chapter. The aim of the chapter is directed more towards indicating the range of methods, to outlining the most important physical principles upon which the instrumentation is based, discussing some experimental problems and limitations, and describing the main instruments featured in the later application (Chs 8–16). Table 6.1 outlines for a range of magnetic properties (a) the main magnetic instrumentation sections of the book connected with their measurements, (b) the associated introductory theory and (c) an example of their use.

The first group of instruments described are those which measure magnetic remanence (Section 6.2). The second group described have those in which an applied magnetic field is used to produce a magnetic signal. In low applied fields we have magnetic susceptibility (Section 6.3) and at high applied fields we have induced magnetisation (Section 6.4). A third group of instruments are used for magnetic cleaning and these are described in Section 6.5. Ancillary equipment for the production and reduction of magnetic fields is described in Section 6.6. The final Section (6.7) covers the major types of portable equipment which are used for field surveys.

6.2 Measurement of remanent magnetisation

The classic instrument capable of detecting weak remanent magnetisations is the astatic magnetometer

(Section 6.2.2). Considering its simplicity, it is a remarkably sensitive piece of apparatus (Blackett 1952). The fluxgate magnetometer (Section 6.2.3) is in general more sensitive and faster to use than the astatic magnetometer. Fluxgate magnetometers can also tolerate magnetically and vibrationally noisy working environments. They are available commercially, complete with an on-line microcomputer (Molyneux 1971) programmed to perform statistical calculations and spherical trigonometric calculations. Thus fluxgate magnetometers can be operated very easily and successfully, even in the field, without need for any specialised palaeomagnetic training. We have made extensive use of fluxgate magnetometers in our environmental investigations.

In principle, calibration of instruments for measuring remanent magnetisation is straightforward. A coil, of the same dimensions as the samples under investigation, carrying a d.c. current can be used to provide a known magnetic moment (measured in Am²) (Collinson 1983). In practise spinner magnetometers are difficult to calibrate and great care must be paid to sample shape and position.

6.2.1 Generator magnetometer

The remanent magnetisation of a sample can be measured by spinning it at a high rate in a coil (Johnson & McNish 1938). As the sample spins the field lines of its dipole moment cut the windings of the coil and induce an alternating electromotive force (e.m.f.) in accordance with Faraday's Law, just as occurs in the operation of a small dynamo. The amplitude of the induced electrical signal is proportional to the sample magnetic moment and its

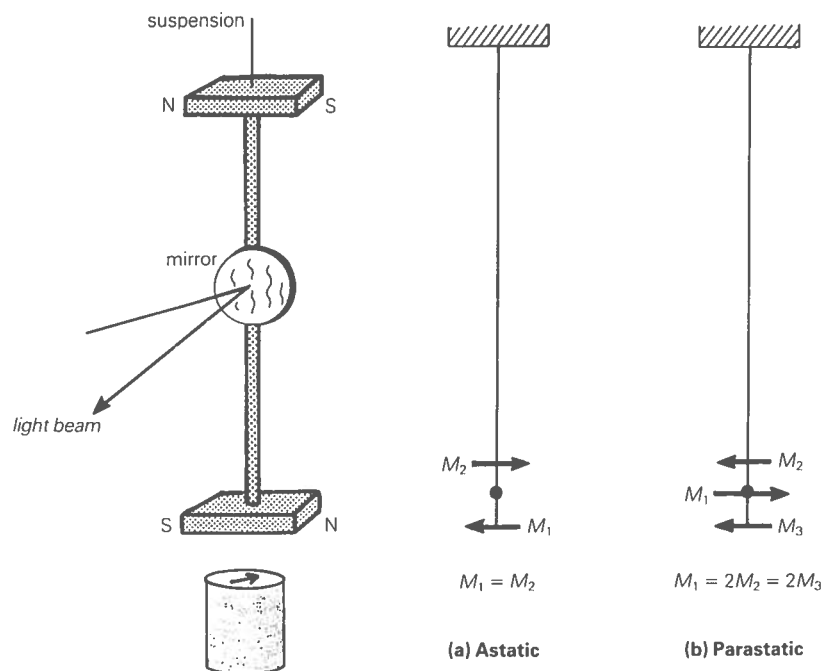


Figure 6.1 (a) Astatic suspension, two magnets with identical moments fixed antiparallel to each other on a rigid stem. An optical lever system is used to detect small rotations of the system caused by a sample placed beneath the lower magnet. (b) Parastatic suspension, central magnet has twice the moment of the upper and lower magnets.

phase is determined by the direction of the sample moment. Since only the component of sample magnetisation perpendicular to the pick-up coil axis contributes to the rotating magnetic field and to the alternating electric signal, it is necessary to spin a sample in more than one orientation in order to determine its remanence fully. In normal generator magnetometer operation a predetermined sequence of six different orientations is used for each sample. The main limiting factor in the generator method of remanence measurement is the accumulation of electrostatic charges on the sample holder. In addition there is the practical drawback of the break up of moderately fragile samples at the high rotation speeds.

6.2.2 Astatic and parastatic magnetometers

The astatic magnetometer is a very simple and reliable instrument. As it can be operated either in zero field or in a low magnetic field it can be used to measure low field magnetisation, initial susceptibility and anisotropy of susceptibility, as well as magnetic remanence. Despite the low cost involved in constructing an astatic magnetometer, its requirement of a magnetically quiet and vibration-free environment

has led to its replacement by other instruments in palaeomagnetic laboratories around the world.

The astatic magnetometer is based on the principle that the torque on a suspended magnet depends on the applied magnetic field. So the field associated with a magnetised sample can be detected by bringing the sample close to a suspended magnet and watching the magnet twist. The key to measuring the weak magnetisation of natural samples lies in making the magnetic suspension sensitive to magnetic field gradients, but at the same time insensitive to changes in magnetic field. This situation is achieved in the astatic magnetometer by using two magnets, each with the same magnetic moment, but aligned antiparallel to each other, as shown in Figure 6.1a.

Another sensitive arrangement of the magnet suspension (Fig. 6.1b) is the parastatic system (Thellier 1933). In this system (Fig. 6.1b) three magnets are used: the central magnet of moment $2M$ is balanced by two antiparallel magnets of moment M fixed above and below it. With both astatic and parastatic magnetometers a sequence of measurements is needed in order to find the total magnetic remanence of a sample. In some experimental arrangements as many as 32 measurements are needed per sample, each involving a different sample

position. However, recent advances which include spinning the sample near the magnet suspension, electronic monitoring of the optical system, and on-line data processing combined with the parastatic configuration, enable the remanence of fairly weak samples to be measured in a town environment at a rate of tens of samples per hour.

6.2.3 Fluxgate magnetometer

The sensitivity, flexibility and ease of operation of fluxgate magnetometers have made them the work horses of modern palaeomagnetism. A fluxgate probe is normally about 50 mm long and consists of a high permeability core, such as a strip of mu-metal, on which a primary and secondary coil have been wound to make a transformer (Fig. 6.2a). In operation an alternating current in the primary coil drives the core around its hysteresis loop in and out of saturation. Any direct magnetic field along the axis of the probe offsets the hysteresis loop (Fig. 6.2b). This distortion due to the applied field introduces asymmetry or even harmonics into the waveform (Fig. 6.2c) and can be detected in the output of the secondary coil. In routine operation a fluxgate probe can detect fields of 1 nT (1 γ) and in practice the noise level is so low that when measuring the remanence of natural samples the practical limit is generally set by the purity of the sample holder.

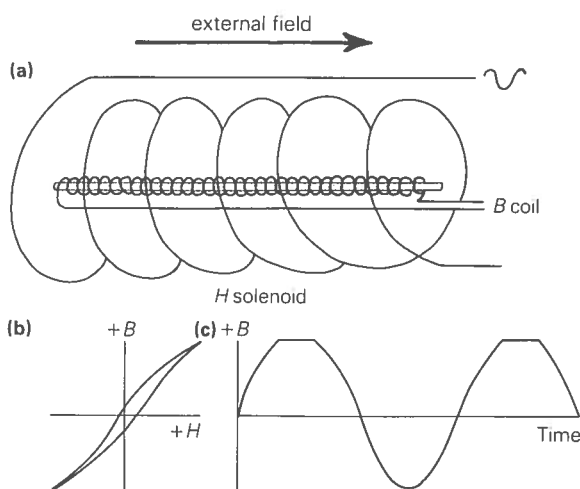


Figure 6.2 Schematic diagrams of: (a) primary and secondary windings of a fluxgate probe; (b) offset B - H loop; (c) asymmetrical output and saturation caused by an external magnetic field.

Instrument sensitivity can be increased and the effect of any sample inhomogeneity significantly reduced by using a double-probe gradiometer arrangement. The double-probe layout is essentially the same as that of the astatic magnetometer, antiparallel fluxgates being used rather than antiparallel magnets. Further sensitivity is produced by using a ring-shaped gradiometer probe, by spinning the sample and by shielding the fluxgate probe from the Earth's magnetic field. All these features are incorporated in the whole core magnetometer (Molyneux *et al.* 1972) of Figure 6.3. Spinning the sample provides a convenient method of taking numerous readings by triggering rapid computer sampling of the fluxgate output using a photocell device (Fig. 6.3). For routine measurements of conventional 10 ml volume specimens the sample is spun six times, i.e. about three mutually perpendicular axes in both upright and inverted positions. Using this procedure remanent magnetisations as low as 10^{-4} A m $^{-1}$ (10^{-7} G) can be measured in about five minutes. Fluxgate based equipment has been used for the great majority of lake sediment remanence measurements described in Chapter 14. It has also been used for many of the saturation remanence and coercivity measurements reported in Chapters 8–12. These strong laboratory remanences, however, can be measured in a few tens of seconds as one short spin is quite sufficient for each sample. Portable, battery-operated fluxgate magnetometers are now manufactured commercially by L. Molyneux with sensitivities, and speed and convenience of operation, equalling those of the older laboratory-based versions.

6.2.4 Superconducting magnetometer

The main advantages of cryogenic superconducting magnetometers (Goree & Fuller 1976) are their high sensitivities and fast response times. Figure 6.4b shows the usual layout of a superconducting magnetometer with a vertical access hole. The pick-up coils (Fig. 6.4a), flux transformer and SQUID (superconducting quantum interference device) sensor (Fig. 6.4c) are enclosed by a superconducting shield. The sample remains at room temperature and is lowered in to the magnetometer's sense region on a long plastic rod. Insertion of a sample into the superconducting magnetometer pick-up coil system initiates a direct current in its superconducting circuitry. This current

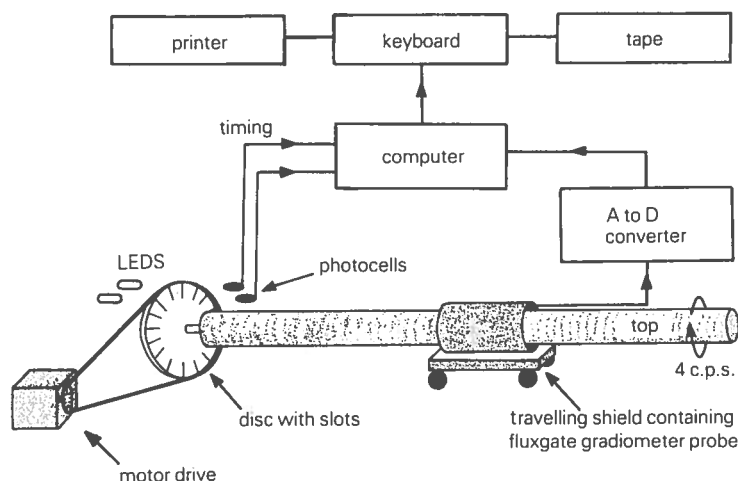


Figure 6.3 Schematic diagram of a computerised spinner magnetometer arranged for whole core declination and horizontal intensity measurements.

is fed via a flux transformer to another coil where it produces an amplified field which is detected by a SQUID. Measurement is independent of the rate at which the sample is inserted and, by using three mutually perpendicular pick-up coils, total remanent magnetisation can be found from one sample insertion.

SQUID sensors have been used to detect magnetic field fluctuations as low as 50 fT ($0.000\ 05\ \gamma$) (Fig. 15.3 and Section 15.5) and to measure remanent magnetisations of $5 \times 10^{-9}\ \text{A m}^2\ \text{kg}^{-1}$ ($5 \times 10^{-9}\ \text{G cm}^3\ \text{g}^{-1}$) (Collinson 1983) with a one second averaging time. They can also be used for several other types of measurement. For example, by trapping a low magnetic field in the same region, measurements of viscous magnetisation and measurements of initial susceptibility and its anisotropy have been made. The superconducting magnetometer has exceptional potential in the magnetic investigations of natural samples, but it is likely to remain a tool operated by the specialist owing to the high costs of its helium consumption.

6.3 Measurement of initial susceptibility

Magnetic susceptibility equipment is the simplest of all magnetic instrumentation to use. Measurement, using the a.c. method (Section 6.3.1), involves sliding a sample into the instrument and reading a meter or dial or else pressing a button for a teletype print out. A hundred samples can easily be processed on such equipment in less than an hour. Portable susceptibility

bridges are made with sensing heads of a variety of shapes and sizes, so that it is possible to measure the magnetic susceptibility of whole cores, soil profile faces and *in situ* bedrock as well as the more usual samples of 25 mm diameter rock cores or 10 ml volume plastic boxes. Several of the magnetometers, generally used for measuring magnetic remanence (Section 6.2), can be adapted to measure initial magnetic susceptibility (Section 6.3.2).

The change of susceptibility with temperature can be investigated with many of the instruments used for normal room temperature measurements, the only modification being to place a small non-magnetic furnace or dewar inside the sensing coils. The sample size which can be accommodated is, of course, considerably reduced and the instrument sensitivity lowered by about an order of magnitude.

6.3.1 The a.c. method

The most common method of measuring initial susceptibility involves the use of a balanced a.c. bridge circuit (Mooney 1952, Mooney & Bleifuss 1953, Aksenov & Lapin 1967, Molyneux & Thompson 1973). Bridge methods are in general very accurate and they are widely used for the measurement of small changes in inductance, capacitance or resistance. In susceptibility bridges the magnetising field is produced by a current-carrying solenoid, flat coil or Helmholtz coil pair. A balanced coaxial pick-up coil is used to detect the induced magnetisation. Insertion of a sample into the coil system alters its inductive balance and produces an out-of-balance signal, in the

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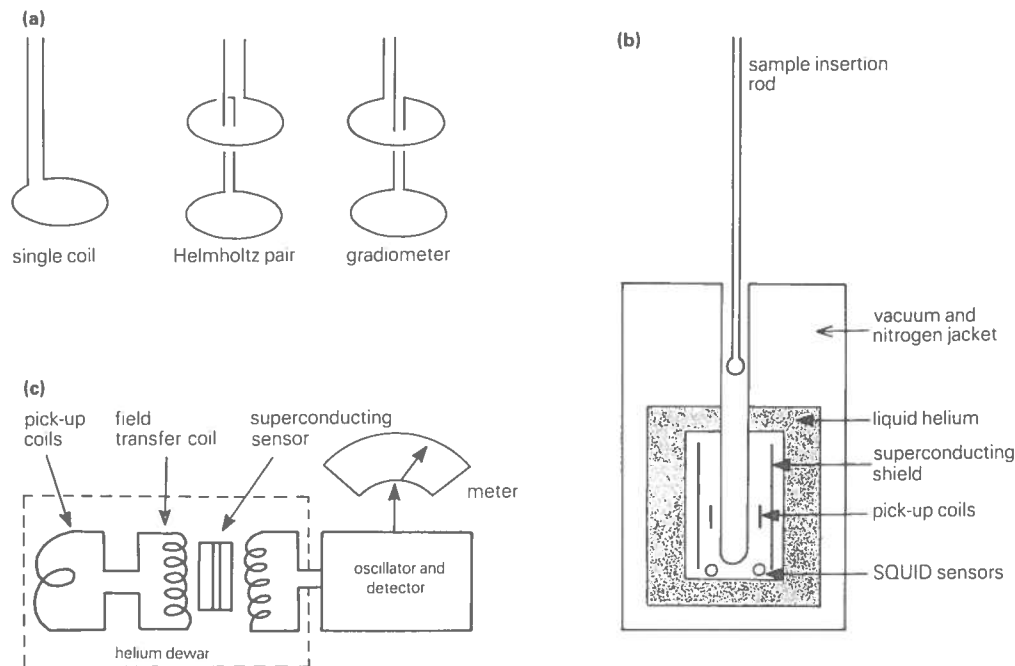


Figure 6.4 Superconducting magnetometer: (a) pick-up coil configurations; (b) general layout of vertical access magnetometer; (c) the heart of the magnetometer lies in its SQUID sensors which detect the d.c. current produced by the insertion of a sample into the pick-up coils.

pick-up coil, which is proportional to the total susceptibility of the sample. Depending on the type of a.c. bridge and coil arrangement used, the out-of-balance signal may be amplified and measured in millivolts, rectified and measured in microamperes or nulled using a low resistance high linearity potentiometer, for both the in-phase and quadrature (Section 6.3.4) components. The ultimate sensitivity of a.c. induction bridges is probably limited by the mechanical and thermal stability of the sensing coils.

An alternative approach is to use a balanced transformer circuit. Bruckshaw and Robertson (1948) used a double coaxial pick-up and Helmholtz pair. Likhite *et al.* (1965) and Radhakrishnamurty *et al.* (1968) employed a similar arrangement in constructing equipment which worked at different frequencies and could measure samples of different sizes. A further approach employed by Bartington (pers. comm.) is to detect the frequency change which is caused in a sharply tuned 'metal detector' oscillator circuit by the introduction of a sample (Lancaster 1966). Smit and Wijn (1954) in addition to describing four a.c. bridges for measuring the frequency dependence of both in-phase and quadrature susceptibility summarize

resonator and wave methods which can operate at substantially higher frequencies.

Modern a.c. instruments using peak magnetic field strengths of about 0.1 mT (1 Oe) at a frequency between 1 and 10 kHz have noise levels below 1×10^{-6} SI units (about 10^{-7} G Oe⁻¹). Natural susceptibilities vary from the weak negative susceptibility (diamagnetism) of unpolluted peat or of carbonate- and quartz-rich materials to the comparatively high susceptibility of 10^{-3} SI units (10^{-4} G Oe⁻¹) of basic igneous rocks. Susceptibility bridges are most easily calibrated by reference to paramagnetic salts such as copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) or ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) with susceptibilities of $7.4 \times 10^{-8} \text{ m}^3 \text{ kg}^{-1}$ ($5.9 \times 10^{-6} \text{ G Oe}^{-1} \text{ cm}^3 \text{ g}^{-1}$) and $1.4 \times 10^{-6} \text{ m}^3 \text{ kg}^{-1}$ ($115 \times 10^{-6} \text{ G Oe}^{-1} \text{ cm}^3 \text{ g}^{-1}$) respectively. Calibration samples should be of the same size and shape as the specimens under investigation.

6.3.2 The direct method

Astatic (Section 6.2.2), ballistic (Section 6.4.2) and superconducting (Section 6.2.4) magnetometers can be used to measure low field susceptibility.

Magnetisation measurements are made in the presence of a low direct field, rather than in the zero field condition of magnetometers used for remanence measurements. So both a remanent and an induced moment contribute to the magnetisation. The relative importance of these two moments can be found by reversing the orientation of the sample. The remanence rotates with the sample while the induced moment remains in the applied field direction. By calculating the sums and differences of the magnetisation for the two sample orientations the separate remanent and induced moments are found. The initial susceptibility is then given by the ratio of the induced moment to the applied field.

6.3.3 Anisotropy of initial susceptibility

It is possible to detect anisotropy of susceptibility using several types of instruments. However, as it is often necessary to measure 1% variations in susceptibility of samples with mean values around 10^{-8} SI units (10^{-7} G Oe $^{-1}$), instruments are needed which can measure susceptibility differences of 10^{-10} SI units (10^{-9} G Oe $^{-1}$). The most common methods of measuring anisotropy use a low field torque meter (Ising 1943, King & Rees 1962) or an alternating current bridge. Figure 6.5 illustrates the orthogonal coil

arrangements and instrument responses of the alternating current method.

The size and shape of sample can have significant effects on anisotropy measurements. It is not simple to calculate the most appropriate sample dimensions as they depend in a complex way on the geometry of the magnetic sensor. In general however, the sample shape should approximate that of a sphere. So cylinders should be used with length-to-diameter ratios of about 0.9. Cuboid, irregularly shaped samples and samples departing from the recommended dimensions by more than a few percent will produce erroneous results which will reflect the sample's shape rather than its magnetic fabric.

6.3.4 Quadrature and frequency-dependent susceptibility

The time delay between the application of a field and the full magnetisation response can be investigated through the measurement of quadrature susceptibility. A.C. susceptibility can be divided into 'in-phase' and quadrature ('out-of-phase') components. The more pronounced the lag in the magnetisation response the more important the quadrature susceptibility. The conventional bridge arrangements of Section 6.3.1 can be used to measure quadrature susceptibility (or permeability) if suitable phase detection circuitry is incorporated in the out-of-balance electrical monitoring system.

Another approach used in investigating magnetic relaxation phenomena is that of measuring susceptibility at different frequencies. The variation of susceptibility with frequency is known as the susceptibility spectrum. At low frequencies, magnetisation remains in phase with field, so the in-phase susceptibility has a value close to that of the direct, static susceptibility while the out-of-phase susceptibility is effectively zero (Galt 1952, Smit & Wijn 1959). However as the frequency is increased, relaxation effects become more important and the in-phase component, after a small rise (Snoek 1948), decreases steadily, while the out-of-phase component rises, peaks and then also falls back to zero. The peak out-of-phase susceptibility and the most rapid decline in in-phase susceptibility theoretically occur at the same frequency. The overall trend is for susceptibility to fall with increasing frequency of measurement.

Bhathal and Stacey (1969) noted that the initial susceptibility of multidomain magnetites only exhibited very small susceptibility changes of 0.3% per

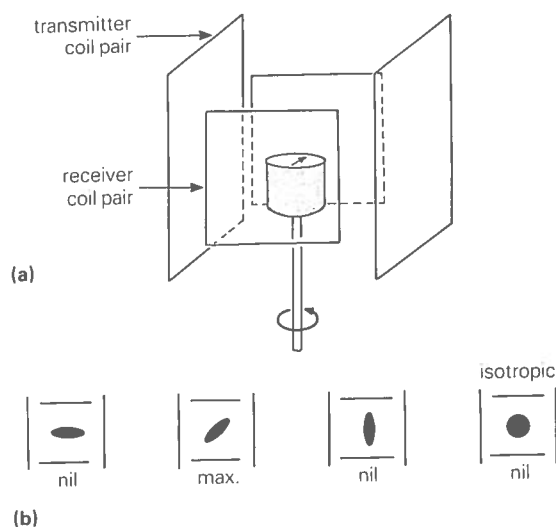


Figure 6.5 (a) Layout of orthogonal transmitter and receiver coils in anisotropy of susceptibility bridge. (b) The plan view diagrams schematically illustrate the coupling between the coils for three orientations of an anisotropic sample and for an isotropic sample.

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decade of frequency at measurement frequencies of around 1kHz. The much larger falls of susceptibility with increased measurement frequency and the high quadrature (out-of-phase-components) that are observed in certain natural samples are thus unlikely to result from the movement of domain walls in large multidomain grains. The dominant cause of high quadrature readings or of pronounced changes in susceptibility with frequency will instead be the viscous effects of ferri- and ferromagnetic grains lying close to the superparamagnetic/stable single domain boundary with relaxation times of around 10^{-4} seconds. The overall fall of susceptibility with increasing measurement frequency observed in natural samples can be accounted for by the magnetisation of grains becoming 'blocked in' as the superparamagnetic/stable single domain boundary shifts to smaller volumes as the frequency of measurement is raised.

By judicious selection of frequency it is possible to investigate usefully the susceptibility spectrum by making just two susceptibility measurements. The instrument of Bartington (Section 6.3.1.), which is the main bridge we have used in making the frequency dependent susceptibility measurements of Chapters 8–11 and 16, uses two frequencies of 1 and 10 kHz and a peak alternating strength of 3×10^{-4} T (3 Oe) in the discrimination oscillator circuit. Introduction of a sample into the detection coil creates a small frequency shift. The difference in shift at 1 and 10kHz is taken as a measure of frequency dependent susceptibility and is given the symbol χ_{fd} in later chapters. Using this equipment with natural samples, the range of values we have encountered for frequency dependent susceptibility expressed as a percentage of total susceptibility (χ_{fd}/χ) is between 0 and 24%. The maximum change in frequency dependent susceptibility for coarse multi-domain magnetite was less than 0.26%, in excellent agreement with the results of Bhathal and Stacey (1969); the highest frequency dependent differences were found for dusts from deflated soils (see Section 11.8 and Fig. 11.16).

6.4 Measurement of induced magnetisation

A very great number of different experimental arrangements have been devised for the measurement of the induced magnetisation of gases, liquids and solids. Measurements of induced magnetisation in medium to high strengths are rather more difficult to

carry out than measurements in low field strengths and quite sensitive equipment is needed in order to investigate the magnetisation properties of most natural materials. The need for sensitivity arises because the induced magnetisation of natural materials, although dominated by strongly magnetic ferrimagnetic crystals, is actually very weak on account of the low concentrations of ferrimagnetic crystals. Indeed the magnetisation of natural samples is often rather similar in strength to the weak magnetisation of paramagnetic substances.

Methods of measuring induced magnetisation may be divided into three main groups. These are:

- measurement of the dipole field of a magnetised sample,
- measurement of the force on a magnetised sample in a non-uniform magnetic field,
- measurement of induction by use of coils.

The force method forms the basis of the Curie balance (Section 6.4.1), an instrument which is widely used in palaeomagnetic laboratories. The induction method forms the basis of the ballistic magnetometer (Section 6.4.2) and the vibrating sample magnetometer

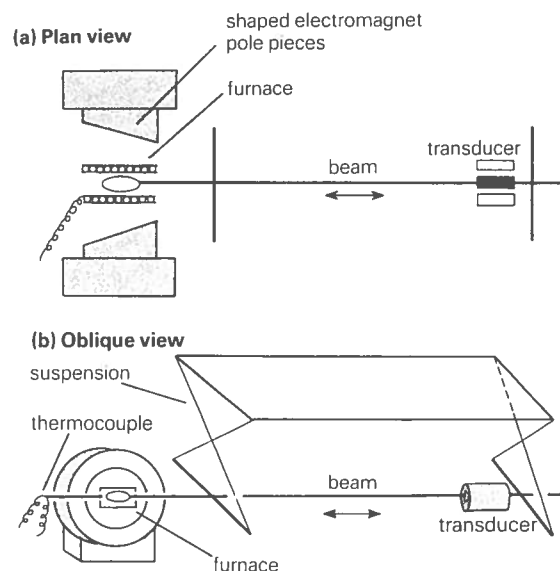


Figure 6.6 Schematic plan (a); and oblique (b) diagrams of a Weiss-Curie force balance. The sample is enclosed in a small evacuated silica capsule and is suspended between the shaped pole pieces of an electromagnet. It is restrained to move horizontally in a tube which can be heated or cooled by a small brass furnace/dewar. The magnetic force on the sample is detected by a transducer. Magnetic balances are the most common instruments that have been used for measuring the Curie temperatures of natural materials.

(Section 6.4.3). These two induction-based instruments are used for detailed investigation of the hysteresis properties of natural material.

6.4.1 The force method

Magnetic balances measure the force exerted on a sample placed in an inhomogeneous magnetic field. Many experimental arrangements, such as the Gouy (1889) method, use long, thin samples. Another arrangement, the Faraday–Curie method (Fig. 6.6), uses small, roughly spherical samples (Curie 1895, Foëx & Forrer 1926) and has been found very suitable for measuring natural materials in the form of small chips or powder pellets.

Magnetic balances can be used to determine acquisition of magnetisation curves, the saturation magnetisation and the hysteresis properties of ferromagnetic minerals and natural samples over a wide range of temperatures and atmospheres. They are routinely used in the estimation of Curie temperatures by recording the change of saturation magnetisation with temperature. Examples of saturation magnetisation versus temperature records of natural samples containing magnetite are shown in Figure 4.14. Construction of complete hysteresis curves using the

force method presents some experimental problems, also the work is somewhat time consuming so that other approaches to drawing complete hysteresis loops, such as using a vibrating sample magnetometer (6.4.3), are usually preferred.

6.4.2 Ballistic magnetometer

The basic instrument has great flexibility (West & Dunlop 1971, Nagata 1976). It has been used to determine the susceptibility of natural samples, as well as their minor hysteresis loops and their saturation magnetisation at both low and high temperatures. It is, however, somewhat limited in sensitivity and so has mainly been used in investigation of igneous rocks.

6.4.3 Vibrating sample magnetometer

The vibrating sample magnetometer (Foner 1959, Kobayashi & Fuller 1967) is the instrument most widely used in measuring the hysteresis properties of natural samples. It has a high sensitivity and the capacity for hysteresis measurements to be made over a wide range of temperatures. It operates on the

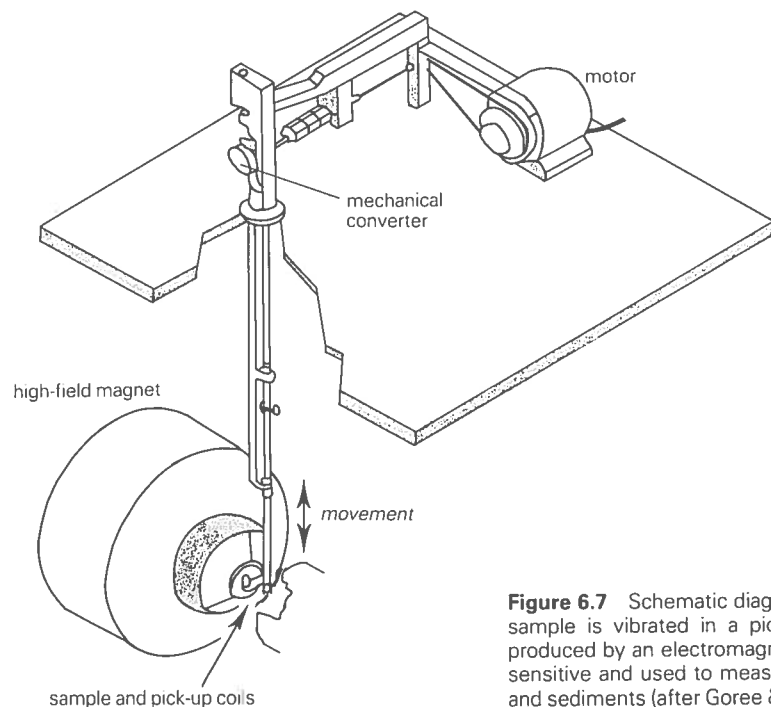


Figure 6.7 Schematic diagram of a vibrating sample magnetometer. The sample is vibrated in a pick-up coil array. A uniform magnetic field is produced by an electromagnet. Vibrating sample magnetometers are very sensitive and used to measure and plot out the hysteresis loops of rocks and sediments (after Goree & Fuller 1976).

induction principle, sample magnetisation being detected as an a.c. voltage in the pick-up coils (Fig. 6.7). The voltage, generated by vibrating a sample at a fixed frequency in the coil system, results from the flux change across the pick-up coils as the sample changes position. In the normal vibrating magnetometer configuration, depicted in Figure 6.7, the sample is vibrated perpendicular to the magnetic field direction.

Direct output can easily be arranged in the form of graphs of hysteresis loops. The magnetising field of the electromagnet is monitored by a Hall effect probe (Section 6.6.1) and the output fed to one channel of an x-y graph plotter. The magnetisation measured by the rectified output of the magnetometer pick-up coils is fed to the other channel. Hysteresis loops are traced out by varying the magnetic field. It takes some minutes to draw a complete hysteresis loop by smoothly cycling the magnetic field from its peak (saturating) value through zero to the opposite polarity peak and then back to the original value. So only two or three samples can be processed in an hour. Furthermore, extracting information from the graphical output can be rather time consuming, particularly when magnetic mixtures are being investigated.

Samples with saturation magnetisations greater than $10 \text{ mA m}^2 \text{ kg}^{-1}$ ($10 \text{ mG cm}^3 \text{ g}^{-1}$) such as igneous rocks or heat-enhanced stream bedload samples, cf Chapter 9, can be measured without difficulty on a vibrating sample magnetometer. The hysteresis properties of many sediments can be measured if care is taken with sample holder correction and long integration times are used.

Figures 4.12 and 16.5 illustrate the main magnetic parameters derived from vibrating sample magnetometer measurements and their uses in describing magnetic crystal assemblages. Such hysteresis diagrams and magnetic parameters provide the most comprehensive magnetic characterisation of natural materials which can be achieved without extravagant specialisation or excessive effort.

6.4.4 Alternating field method

A very neat and rapid method of obtaining hysteresis characteristics is the alternating magnetic field induction method (Bruckshaw & Rao 1950) in which hysteresis loops can be displayed directly on an oscilloscope screen (Likhite *et al.* 1965). The sample magnetisation is detected using the double-search coil

method of a.c. susceptibility bridges (Section 6.3.1). Magnetising fields of up to 0.4 T (4000 Oe) have been obtained by using an electromagnet with a high permeability core. The rather low sensitivity of the a.c. method has so far restricted its possible applications with natural materials to the examination of basic igneous rocks. The instrument is very appealing, however, on account of the speed with which it can be used. Another useful feature of the alternating field method is the ease with which low temperature investigations can be performed. The change of hysteresis properties with temperature can be monitored by simply dipping a sample in liquid nitrogen, placing it in the detection coil and then allowing it to warm up to room temperature. Large collections of basalts have been characterised magnetically using this technique. A practical application of such magnetic characterisation has been the preselection of basalts for more time-consuming studies such as palaeointensity determinations.

6.5 Magnetic cleaning techniques

As rocks or sediments have had a complex magnetic history it is necessary to apply demagnetisation techniques to separate out their various components of remanent magnetisation. Two methods routinely used for this task are alternating field demagnetisation (As 1967, Creer 1959) and thermal demagnetisation (Thellier 1938, 1966). The former is used for samples in which magnetite carries the remanent magnetisation, the latter for haematite-bearing samples and samples which are chemically stable at elevated temperatures. The principle of magnetic cleaning or partial demagnetisation is that the less stable components of the remanent magnetisation are selectively removed to leave the more stable components.

As detailed demagnetisation studies can be very time consuming, a common practice when dealing with collections of hundreds of samples is to investigate the demagnetisation properties of up to about 10% of the samples in detail. These pilot samples are chosen to be representative of the main collection and their demagnetisation results are used to decide on the most efficient method of isolating the different components of magnetisation in the collection.

6.5.1 Alternating field demagnetisation

Demagnetisation is accomplished by subjecting a sample to an alternating field which is gradually reduced to zero. The alternating field is produced by a coil in a tuned a.c. circuit, which is generally driven at mains frequency. The field is smoothly reduced from its peak value (Fig. 6.8) by a liquid rheostat or motor-driven voltage regulator. The effect on the sample is to remove a part of the remanence by magnetic grains with coercive forces lower than the strength of the peak applied magnetic field. The part of the remanence carried by grains with higher coercive forces remains unaltered.

Pilot samples are generally demagnetised at steps of 5 or 10 mT (50 or 100 Oe) up to about 100 mT (1000 Oe). Demagnetisation at each step is carried out along three mutually perpendicular axes or alternatively a tumbling device, which presents all axes of the sample to the alternating field, can be used. In practice it is essential to ensure that demagnetisation is carried out in zero field and also that the alternating field is free from both transients and asymmetry. Without these safeguards an anhysteretic remanence (ARM) may be grown which can mask the natural remanence and invalidate the cleaning process. As tumbling devices speed up the demagnetisation process, and randomise unwanted anhysteretic remanences they are expedient and widely used.

6.5.2 Thermal demagnetisation

There are two methods of carrying out thermal demagnetisations. One, called continuous thermal demagnetisation, involves monitoring the change in remanence of a sample as it is heated (Wilson 1962). The other method, called progressive thermal demagnetisation, consists of a number of partial demagnetisation steps using successively higher temperatures (Thellier 1938). At each step the

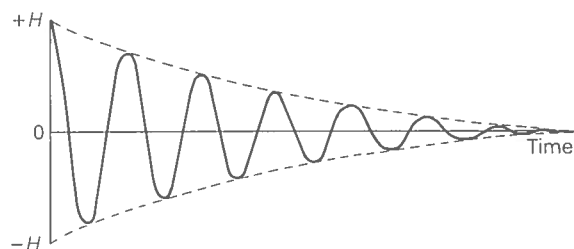


Figure 6.8 Decay with time of the waveform of the alternating field in a demagnetisation cycle.

sample's remanence is measured after having been heated and cooled in a zero magnetic field. With both thermal demagnetisation methods the components of remanence with low blocking temperatures are removed first thus revealing the higher stability portion of the remanence.

In the first method, a furnace is mounted within a magnetometer and the sample's remanence is measured while it is hot. In the second method, large separate furnaces, which can accommodate up to 50 samples, can be used. The progressive method is much faster than the continuous method when dealing with large collections. Temperature steps of 50 or 100 °C are commonly used for the treatment of pilot samples in the progressive method. Particular importance is attached to changes in remanence at the highest blocking temperatures. Samples may acquire a spurious TRM in the progressive method, when cooling down, unless the ambient field is strictly maintained at a low value $< 10 \text{ nT}$ ($< 10 \gamma$).

6.6 Magnetic fields

All experimental techniques in palaeomagnetic or mineral magnetic investigations involve the production, cancellation or detection of magnetic fields. The field strengths with which we are routinely interested vary over eleven orders of magnitude from 0.01 nT (0.01 γ), for the field associated with a weakly magnetised sample, to 2 T (20 000 Oe) for the field in the air gap of an electromagnet. So a variety of apparatus is needed to measure or produce fields of different strengths.

6.6.1 Measurement

The easiest method of measuring low magnetic fields, i.e. in the range 1 nT (1 γ) to 0.2 mT (2 Oe), is with the fluxgate probe (Section 6.2.3). The total intensity of the Earth's magnetic field can be measured very conveniently by the proton magnetometer. Stronger magnetic fields are measured using a Hall probe. Alternating magnetic fields can be most easily measured with a search coil method.

SEARCH COIL

The simplest way to measure a steady or alternating field in air is to use a search coil and an instrument for measuring a current pulse. A search coil consists of a number, N , of turns of wire on a small former of cross-

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sectional area A . When placed in a magnetic field, B_0 , the flux through it is NAB_0 . If the flux is changed by switching off the field, removing the coil or turning it so that it encloses no flux then an e.m.f. is produced across the coil which can be measured by a fluxmeter or a ballistic galvanometer. The flux change is NAB_0 ; so from a measure of the flux and the dimensions of the coil we have a measure of the field. If the coil is rotated in the field or if the coil is held stationary and the field is alternated then an alternating e.m.f. is induced in the coil. The amplitude of the alternating e.m.f. is a measure of the field intensity and is equal to $NAB_0\omega$, where ω is the frequency of alternation.

HALL EFFECT PROBE

Hall probes can be used to detect magnetic fields which vary in strength from 50 μT to 3 T (0.5 Oe to 30 000 Oe). Metals and semiconductors when carrying an electric current perpendicular to a magnetic field produce an e.m.f. in the third perpendicular direction proportional to the magnetic field. This behaviour is a Hall effect. Hall effect probes are very simple and very convenient to use.

PROTON PRECESSION

There are two methods of obtaining a measurement of the Earth's magnetic field from atomic behaviour. One is to expose a sample such as an alkali vapour to radiation and determine its resonance precession frequency with optically pumped sensors and the other is to determine the rate of free precession of a particle such as a proton. The free precession method, although not as sensitive as the resonance method, is widely used in surveying and observatory instruments as it is reliable, stable and absolute. It measures the total field intensity rather than just one component of a field. The proton precession detector head consists of a bottle of about 300 ml volume containing a liquid with a high concentration of protons (e.g. alcohol) surrounded by a coil. Before each measurement the bottle is subjected to a strong polarising field. This tends to align the protons which then precess when the field is removed. The frequency of precession, which is measured, depends only on the product of the gyromagnetic ratio of the proton (a well determined quantity) and the field intensity. The gyration of the protons dies away in a few seconds allowing field measurements to be rapidly repeated. Proton magnetometers can measure magnetic fields in the range 20–80 μT (20 000–80 000 γ) with an accuracy of

1 nT (1 γ). However, they cannot operate in field gradients above 500 nT m^{-1} (5 $\gamma \text{ cm}^{-1}$). Portable, lightweight battery-operated proton magnetometers are the most common instruments used in magnetic geophysical prospection.

6.6.2 Generation

All magnetic fields are generated in some way by the magnetic effects of electric currents. In the laboratory magnetic fields up to a few mT (10 Oe) in strength are most conveniently generated by current-carrying coils. Moderate laboratory fields of up to 2.5 T (25 kOe) can be supplied by iron-cored electromagnets and modest d.c. supplies of a few kilowatts power. Higher fields have rarely been used in the investigation of natural materials but if needed, superconducting magnets (Wilson 1983) can produce persistent d.c. fields up to 15 T (150 kOe), explosive pulse methods can be used to generate 50 T (500 kOe) fields for the order of a microsecond (Rubin and Wolff 1984), while the electromagnetic flux-compression method can yield 280 T (2.8 MOe) fields for magneto-optical or cyclotron resonance studies (Miura *et al.* 1979). Portable coil systems are produced commercially which can supply very uniform, repeatable short-duration fields of strengths up to 0.8 T (8000 Oe) using a pulsing discharge system similar to a flash light and are ideal for isothermal remanence studies. Mains-driven pulse discharge units can produce fields of up to 5 T (50 kOe) or even 10 T (100 kOe) in coils cooled to liquid nitrogen temperature (McCaig 1977).

COILS

Uniform direct fields of up to 1 mT (10 Oe) can be produced without any difficulty from circular coil windings in the form of a solenoid and a d.c. power supply or battery. The field inside a solenoid is equal to $\mu_0 Ni$ where N is the number of turns per metre and i the current in amps. The field is constant anywhere inside the solenoid but falls off sharply at its ends.

Helmholtz coils are also widely used in magnetic laboratories. These consist of a pair of circular coils arranged so that the spacing between them is equal to their radius, a . This Helmholtz configuration produces a remarkably uniform field. The field has an intensity of $0.9\mu_0 Ni/a$ at the midpoint and is uniform to 10% within a sphere of radius $0.1a$. Square coils can also be used to produce a similarly uniform field. They are arranged with a spacing equal to 0.544 times the length of one of their sides.

ELECTROMAGNETS

The principal parts of an electromagnet (Fig. 6.9c) are an iron yoke, iron-cobalt alloy pole pieces and two coils carrying direct current to build up and maintain a magnetic flux in the yoke. In most electromagnets the energising coils are placed close to the pole pieces as this produces a slightly larger field. The strength of the magnetic field which can be produced between the pole pieces greatly depends on the width of the gap between them. Consequently the pole piece gap is normally kept as small as possible. Flat-faced pole pieces with a truncated cone shape (Fig. 6.9a) are commonly used to concentrate the flux and produce strong uniform fields. Specially shaped pole pieces such as those shown in Figure 6.9b can be used to produce non-uniform fields. These shaped pole pieces are used with magnetic force balances where the rather unusual condition of a uniform product of the field and the field gradient is desirable.

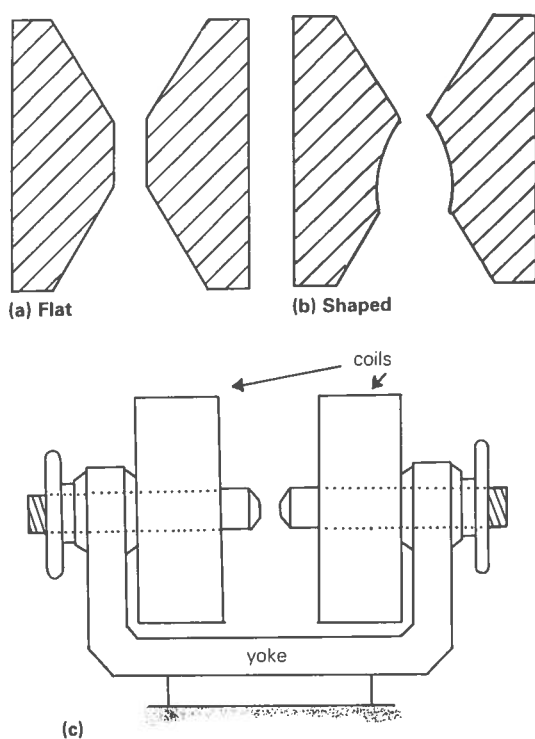


Figure 6.9 (a) Conical flat-faced pole tips of an electromagnet used to produce high uniform magnetic fields, for example in the vibrating sample magnetometer of Fig. 6.7. (b) Shaped pole pieces used to produce magnetic field gradients, e.g. in the magnetic force balance of Fig. 6.6. (c) Overall layout of an electromagnetic showing Y-shaped yoke and support, adjustable pole pieces and large exciting coil pair.

6.6.3 Shielding

The development of materials which can deflect magnetic fields has greatly simplified investigation of weak magnetic fields in town laboratories (Cohen 1967, 1970, Thomas 1968). Magnetic shields can screen the sensitive equipment needed for weak field measurement from spurious transient magnetic fields such as those produced by lifts and also from steady magnetic field gradients associated with stationary ferrous objects such as steel girders and pipes. Magnetic shields are made from high permeability materials for example the iron-nickel alloys of mu-metal and permalloy. The most efficient shielding is achieved by using a series of thin sheets rather than a single thick block. Triple-sheeted shields can be expected to reduce magnetic fields by a factor of 5000. The extremely high permeability of the shields can only be attained by annealing the alloy after it has been fabricated into its final shape. Straining of the alloys, for instance by a dent or a scratch, can significantly reduce their shielding ability. For this reason it is often prudent for the outer shield to be made of a somewhat lower permeability but higher strength material.

6.7 Portable instruments

A wide range of portable, battery-operated equipment has been developed for the geophysical prospector and the archaeological surveyor. One group of these portable instruments is classed as passive. Instruments in this class measure the magnitude of the Earth's magnetic field or one of its components. The proton magnetometer is the commonest instrument of this group. The fluxgate magnetometer is another common passive instrument which is often used to measure the gradient of the vertical magnetic field. The other main class of magnetic surveying instruments is made up of active equipment. Active instruments produce a magnetic field and then measure the response of the ground to the applied field. They are used to investigate near-surface features. The two main types of active instruments are those that operate on pulsed induction and on induction balance principles.

6.7.1 Magnetometers

Local anomalies in the Earth's magnetic field arise from changes in the remanent magnetisation and

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susceptibility of subsurface materials. The composition, shape, depth and orientation of the anomalous material all control the distortion of the Earth's magnetic field. Interpretation of total field magnetic anomalies is thus not a simple task, and often involves quite complicated mathematical modelling procedures. However, the width of a magnetic anomaly gives information about the depth of the source of the anomaly, and the magnitude gives an indication of the nature of the source. On archaeological sites large anomalies of up to 200 nT (200 γ) are associated with burnt materials which possess a remanent magnetisation, such as kilns (Aitken 1974). Smaller, but well defined anomalies, are found connected with soil features, such as silted-up pits and ditches. These are generally caused by the higher magnetic susceptibility of topsoil compared with its subsoil or bedrock and are typically between 5 and 100 nT (5 and 100 γ) in strength (see Section 8.10 and Fig. 8.7). Anomalies associated with igneous intrusions may be over 1000 nT (1000 γ) in amplitude.

When a magnetic survey is conducted, the spacing between observations and the height of the detector, above the ground surface, are adjusted to local conditions. A fairly standard arrangement, however, when looking for fairly shallow objects on an archaeological site would be a detector height of 0.3 m with a 0.5 or 1 m grid spacing.

During the time in which a survey is conducted the Earth's magnetic field will change in strength because of its diurnal variation. As these background diurnal changes are of the same order of magnitude as the strength of many anomalies, it is necessary either to operate a separate base station or to make repeated measurements at one locality. The survey readings can then be corrected for the background changes.

6.7.2 Gradiometers

The usual method of measuring gradients of the Earth's magnetic field is to measure the intensity of the field at two localities simultaneously. The gradient is given by the field difference divided by the distance between the measuring localities. Two fluxgate detectors mounted about 1 m apart on a staff form a lightweight instrument which is suitable for measuring the gradient of the vertical component of the Earth's magnetic field. A continuous digital output makes the equipment particularly suitable for rapid scanning.

Gradiometers react strongly to near-surface

features, as the lower detector responds much more vigorously to buried objects than the upper detector, while deep geological features affect both detectors equally. Consequently, the main application of portable gradiometers has been in the surveying of archaeological sites and the detection of the magnetic polarity of surface exposures of igneous rocks.

6.7.3 Pulsed induction

Portable pulsed induction instruments have been developed as very sensitive metal detectors (Colani 1966). They can detect magnetic materials as well as responding to metallic objects. Their extreme sensitivity enables them, for example, to detect differences in the thickness and magnetic content of topsoil. They operate by first transmitting a magnetic pulse into the ground for about 1 ms (Fig. 6.10). They then monitor the response of the ground to the pulse over the next few microseconds. A response is received from metallic objects because the primary magnetic

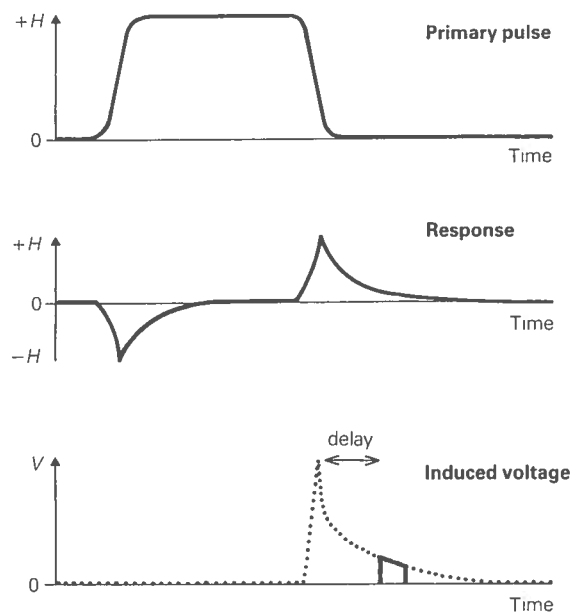


Figure 6.10 Operation of pulsed induction instrument. A primary magnetic pulse emitted by a transmitter loop induces eddy currents in metallic objects or magnetisation changes in viscous materials. These responses to the primary pulse changes increase rapidly but decay more slowly. The response initiated by the trailing edge of the primary produces an induced voltage in a receiver loop which is sampled and displayed a short time after the end of the main pulse (modified from Colani 1966).

pulse creates eddy currents in the metal. A changing secondary magnetic field is produced by the decaying eddy currents and this is picked up and integrated by the pulsed induction equipment. Superparamagnetic and viscous magnetic materials produce a similar response of a slowly decaying secondary magnetic field. Pulsed induction instruments thus respond strongly to metallic objects, but in their absence, measure magnetic viscosity.

Lightweight pulsed induction equipment consisting of a ground coil, handle and small electronics box is ideal for rapid scanning or surveying. Output is displayed on a meter mounted on the electronics box. On some soils, magnetic signals may be so strong that the coil has to be held a few centimetres above the ground level rather than directly on it. With pulsed induction equipment, as there is both a transmitted signal and a pick-up signal, sensitivity falls off as a sixth power of distance and so it is only possible to detect shallow objects no matter how strong the signal. The results of a pulsed induction survey of the magnetic content of topsoil of a Welsh lake catchment are described in Chapter 10.

6.7.4 Induction balance

The magnetic susceptibility of *in situ* natural materials can be measured using lightweight portable equipment based on the a.c. bridge method (6.3.1). This type of equipment is extensively used by amateurs interested in metal detecting (Lancaster 1966). A most useful facility of induction balance equipment is its ability to discriminate between magnetic materials and metallic objects through the phase of the out-of-balance signal. The principle of operation of the portable induction balance is the same as that of the a.c. susceptibility bridge (Section 6.3.1). The main difference between these pieces of equipment lies in the geometry of their transmitter and receiver coils. In the laboratory equipment, solenoids are used and the material under investigation is placed inside the coils. In the portable surveying equipment the material under investigation lies outside the coils. Elaborate arrangements of loops or flat coils have been designed to give both good sensitivity and precise source location in the surveying equipment.

As with all active instruments the sensing depth of the induction balance is fairly shallow. In the sandwich coil layout of 'metal detectors' the signal is dominated by material within a few centimetres of the

coil system. A rather different coil design, which has been commercially developed for pipe finding, can be used to sense objects down to 1 m in depth. In this type of induction balance equipment the transmitter and receiver coils are separated by about 1 m and are mounted at right angles to each other on the ends of a shaft.

Continuous digital readout combined with the facility for metal discrimination makes the induction balance a very convenient surveying tool. Sensitive, battery-operated kits have been recently designed to serve as both laboratory and surveying equipment and to accept a remarkably wide range of detector coils and probes.

6.8 A basic environmental magnetic kit

Magnetic instrumentation is constantly being improved. Over the past few years new developments have created the situation in which portable equipment, easy to operate and maintain, has become available commercially at realistic prices. Sensitive palaeomagnetic and rock magnetic equipment can now be reliably operated by inexperienced investigators with little or no background in physics or electronics.

Chapter 16 presents a case study of the application of magnetic measurements to one catchment area. The flow diagram of Figure 16.2 illustrates the sequence in which mineral magnetic and palaeomagnetic measurements can most efficiently be applied to the bedrock, soils, river sediments and estuarine cores of such a catchment. The complete Rhode River/Chesapeake Bay study of Chapter 16 has drawn on a wide range of magnetic instrumentation, but the major part of the study has been performed with the aid of just three magnetic instruments. These are (a) an a.c. magnetic susceptibility unit (Section 6.3.1), (b) a flux-gate magnetometer (Section 6.2.3) and (c) a pulse discharge magnetiser (Section 6.6.2). These three pieces of equipment can be purchased for less than the cost of the average family car and form the basis of an 'environmental magnetic kit'.

Battery-operated, portable susceptibility equipment, in addition to its routine laboratory rôle, can also be used with suitable accessory, plug-in coils for field prospecting, for whole core scanning and for frequency-dependent or quadrature susceptibility measurements. A susceptibility bridge is probably the

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most useful single piece of equipment for 'environmental' magnetic studies.

The fluxgate magnetometer has been refined into a highly sensitive, portable, battery-operated instrument which can be used for both palaeomagnetic natural remanence studies and for artificial remanence measurements in mineral magnetic studies. A fluxgate magnetometer costs around four times as much as a susceptibility bridge.

Portable pulse discharge magnetisers have only recently become available commercially. They routinely produce more repeatable, more accurate magnetisations than electromagnets and are simple to use and calibrate.

All three instruments in this basic kit can be easily transported. For example they can be taken as hand luggage on an aeroplane.

Looking to the future, one piece of equipment which it would be extremely useful to be able to add to this kit would be an instrument for measuring induced magnetisations. The equipment should, of course, be fully portable, very robust, totally reliable, foolproof in use and sensitive enough to determine the hysteresis characteristics of pre-nineteenth century peat samples (Section 11.5). If instrumental advances continue at the rate of the last ten years, and if applications for environmental magnetic studies continue to be found, such a dream instrument could soon become reality.

6.9 Summary

Advances in many branches of science can be linked with instrumental developments. Palaeomagnetism is an example of such a subject since the development of the astatic magnetometer system at the end of the nineteenth century permitted the earliest investigations of the natural remanence of rocks. A major step forwards took place in the 1950s when very sensitive astatic and parastatic systems were constructed around newly developed high coercivity, high remanence, permanent magnets. Computer control, especially of the fluxgate system, in the early 1970s greatly speeded the palaeomagnetic measuring

process. Further increases in sensitivity and speed of measurement have recently been achieved through the production of super conducting SQUID magnetometers. Accompanying each of these instrumental developments have been new possibilities of investigating additional rock types and consequent progress in both geomagnetic and geological aspects of palaeomagnetic research.

Other instrumentation for mineral magnetic research has not shown the spectacular advances of the palaeomagnetist's magnetometer, and so measurements of mineral magnetic parameters such as coercive force, Curie temperature and saturation magnetisation remain rather specialised. Magnetic susceptibility measurements have however become much easier and more sensitive through recent electronic developments. Mineral magnetic experiments in 'environmental studies' have thus tended to centre around susceptibility bridges and the palaeomagnetist's magnetometers, and the measurements of initial susceptibility, isothermal remanence, anhysteretic remanence and remanence coercivity.

Further reading

General book

Aitken 1974. *Physics and archaeology*.

Advanced books

Collinson 1983. *Methods in rock magnetism and palaeomagnetism*.

Collinson, Creer and Runcorn 1967. *Methods in palaeomagnetism*.

Nagata 1953. *Rock magnetism*.

General journal papers

Collinson 1975. Instruments and techniques in palaeomagnetism and rock magnetism.

Banerjee 1981. Experimental methods of rock magnetism and palaeomagnetism.

Magnetic minerals and environmental systems

Interdisciplinary research can result from two sorts of inquiry, one relating to common structures or mechanisms and the other to common methods.

Jean Piaget

Main Trends in Inter-Disciplinary Research

The major primary energy sources in the sun and in the Earth's interior ultimately drive all the systems of energy transformation and material flux which are the concern of environmental scientists. Whether we are studying the lithosphere, hydrosphere or atmosphere, or the cycles which involve the transfer of material between them, the movement of solid particles in some form or other is of major importance. The processes involved include entirely natural ones such as the major ocean circulation systems more or less unaffected by human activity, many others such as soil development and river erosion strongly modified by man's activity, and some, for example the particulate output associated with fossil-fuel combustion, which are entirely the result of modern technology.

7.1 Surface processes and magnetic minerals

The extent to which magnetic properties and the measurements used to characterise them are conservative within environmental systems depends on the nature of the processes to which the magnetic minerals have been subjected. Some of the most important effects are outlined below.

- (a) *Chemical transformations.* Of primary interest here are those which take place as a result of weathering, soil formation and sediment diagenesis. They may lead to the conversion of paramagnetic iron to ferri- or antiferromagnetic forms. They may equally bring about transformations between magnetic mineral types or lead to the conversion of ferri- or antiferromagnetic oxide compounds to paramagnetic forms. In relation to the present parameters they may therefore be either 'constructive', 'transformative' or 'destructive'.
- (b) *Physical comminution.* Physical weathering, erosion and transport by water or ice will often involve the comminution of material. Where this gives rise to reduced size, with or without changed shape in the magnetic minerals present, the magnetic parameters may be altered. Preliminary results from measurements of glacial drifts derived from homogeneous source areas suggest that they can be compared with the bedrock from which they were derived despite intervening comminution which has changed some grains from multidomain to stable and pseudo-stable single domain (Ch. 2).
- (c) *Transport and deposition.* Where material is

moved with neither chemical transformation nor comminution, the main processes mediating between sediment source and the point of deposition are often sorting mechanisms which will affect both the magnetic and non-magnetic fractions in the material. In consequence, the magnetic parameters of sediments may differ in important ways from those of the source material from which they have been derived without any chemical or physical change affecting the magnetic domains themselves. Bjorck *et al.* (1982) give examples of the relationships between particle size and magnetic susceptibility in several till and sediment samples and illustrate the way in which selective depletion or enrichment of given size ranges during transport and sedimentation will affect the measurements. The distinction between *particle size* and *magnetic grain size* is crucial. Although the two can obviously never be completely independent of each other, the relationship will rarely be simple and direct. For example fine-grained haematite abounds as an important constituent of the cement coating sand grains. Equally, coarse shale particles may include stable single-domain and superparamagnetic magnetite.

- (d) *Concentration and dilution.* Several processes will lead to either concentration or dilution of magnetic minerals without affecting their nature. For example primary magnetic minerals in the bedrock may be more persistent than other soil minerals during weathering and thus become concentrated in the upper part of the regolith. Conversely, the growth and accumulation of organic matter in soils, and peat and its deposition in lake sediment will dilute magnetic mineral concentrations, as will the deposition or precipitation of material such as calcium carbonate and diatom silica. Although where such processes of dilution and concentration are significant, they will give rise to variations in susceptibility and remanence values, they will not affect interparametric ratios.

7.2 Primary and secondary magnetic minerals

In practice, it is convenient to distinguish between primary and secondary magnetic minerals within the

context of the regolith, the weathering zone at the Earth's surface. *Primary* magnetic minerals we consider to be those present in the parent material upon which weathering and soil formation may be taking place, irrespective of whether the parent material is igneous, metamorphic or sedimentary bedrock, or drift. *Secondary* magnetic minerals are those formed from 'primary' iron by chemical processes or biogenic effects. In most situations the distinction is reasonably clear in theory though practical differentiation may often be more difficult. There are special cases where the distinction becomes blurred or breaks down, for example where previous weathering regimes have led to the formation of iron oxides in subsoil which now forms the basis for present day soil development (as in some weathered basalts) or where soil development is taking place on recent alluvium derived from both bedrock and topsoil sources.

7.3 Magnetic minerals and material flux

Iron compounds are among the most ubiquitous components of natural materials, forming some 2% of the Earth's crust. On the longest geological timescales the cycles and patterns of mountain building, denudation, geosyncline development and crustal displacement all involve large-scale movements of materials which include forms of magnetic iron oxide. Geological processes, such as sea-floor spreading, the eruption and flow of lavas, the dispersal of volcanic ashes and the transport of eroded sediments under the influences of water and wind, control the magnetic mineralogy of natural materials, sometimes by forming or transforming magnetic oxides through the influence of heat, hydration/dehydration or changes in E_H and pH, sometimes by merely transporting stable magnetic oxides to environments where they may persist unaltered for long periods in sediments and sedimentary rocks. The magnetic oxides in the crust together with paramagnetic forms of iron (which are potentially convertible to magnetic oxides by thermal or chemical action, both natural and anthropogenic) form the main primary source of the minerals considered in the present account (Fig. 7.1). The second primary source, the cosmic flux of extra-terrestrial magnetic particles, is significant only in the immediate vicinity of meteor impact sites and in certain marine environments where the flux from lithospheric sources is minimal, e.g. the central

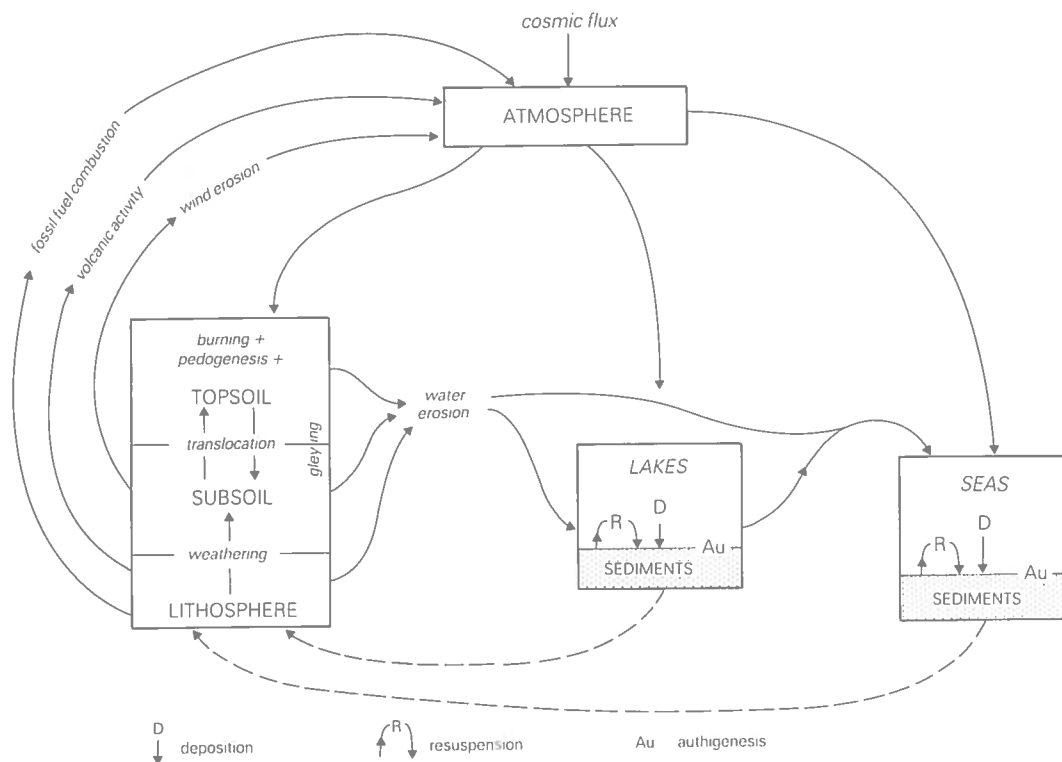


Figure 7.1 Schematic diagram of the cycle of magnetic minerals.

Pacific. Chapter 3 is concerned with the various magnetic minerals present in primary crustal and extraterrestrial materials.

As rock surfaces are exposed to atmospheric processes, to colonisation by plants and to resultant weathering and soil formation, the iron compounds present in bedrock may be subject to many processes of concentration, dilution and transformation. Secondary magnetic oxides may even be formed near the soil surface. Soil iron compounds are among the most abundant and most sensitive components of the soil system. Where chemically stable magnetic oxides are the end product of weathering and pedogenic processes, they may be diagnostic of soil horizon type or of weathering régime. The processes involved in the formation of secondary magnetic oxides and their effects in the soil are a major theme of Chapter 8.

The secondary magnetic oxides formed at or near the soil surface often differ in crystal form and size from the primary magnetic oxides present in the underlying substrate. Both primary and secondary

magnetic minerals are eroded from soils and substrates, and may become incorporated in river and lake sediments. Differences between primary and secondary magnetic minerals can thus form the basis for sediment source identification in rivers, lakes and estuaries (Chs 9, 10 & 15).

Primary iron compounds are transformed to magnetic oxides, not only by weathering and soil formation, but also through the combustion of fossil fuel by man. The burning of solid fuels in particular, whether in domestic or industrial appliances, generates large volumes of magnetic spherules which are an important component of fly-ash from solid fuel fired power stations. Other industrial processes such as metal smelting and steel manufacture are of major significance in the discharge of magnetic particulates into the environment. The magnetic characteristics of virtually every type of particulate emission from industrial and domestic fossil-fuel combustion as well as many other industrial processes, provides hitherto little used scope for the magnetic monitoring of

particulate pollution both atmospheric and marine (see Chs 11 & 12).

7.4 Natural remanence and mineral magnetic properties

In the following chapters we make use of two different but interrelated types of magnetic parameters. In Chapters 13 and 14 emphasis is placed on remanent magnetisation acquired in the Earth's magnetic field. Such remanence is called natural remanent magnetisation (NRM) whether formed as a result of cooling through the blocking temperature, crystal growth through the blocking volume or the deposition and 'fixing' of detrital particles. Chapters 8–12 are concerned with mineral magnetic properties. These are the magnetic characteristics of a substance which are an expression of the intrinsic magnetic properties of the constituent magnetic crystals and of their dispersion as fine particles within the substance. Such characteristics are generally measured by monitoring the signal induced by an instrumentally generated field, as in the case of magnetic susceptibility and magnetic hysteresis measurements. They may also involve monitoring the change in induced signal with temperature as in Curie temperature measurement.

Palaeomagnetic measurements of natural remanent magnetisation are important for the insight they give into the behaviour of the Earth's magnetic field through time. It is out of the results of natural remanence-based studies that geologists and geophysicists have established the magnetic polarity timescales of such enormous significance in the present theories of plate tectonics and sea-floor spreading. They are also a prime source of empirical insight into the nature of the dynamics of the Earth's fluid core.

In all palaeomagnetic measurements, the primary aim is to recapture the directional and intensity characteristics of the Earth's magnetic field for a given period and location. The signal retained in rocks or sediments will however reflect an assemblage of conditions including magnetic mineral types and concentrations, mechanisms of formation, depositional and post-depositional history and so forth. In order to compensate for these so that as much as possible may be learned about the primary concern, the Earth's magnetic field, many supplementary palaeomagnetic and mineral magnetic measurements

may be required in order to isolate and to some degree 'standardise' the palaeomagnetic signal.

From the above, we can see a distinction between magnetic properties which reflect ordering consequent on the existence of the Earth's magnetic field – natural remanent magnetisation – and magnetic properties which are solely a consequence of the magnetic crystal structures, grain sizes and shapes present. Although in practice the various magnetic properties can be closely connected as explained below. The five parts of Figure 7.2 diagrammatically illustrate the palaeomagnetic and mineral magnetic characteristics of some different natural substances.

- (a) The abundance of magnetic minerals in the Earth's crust varies from rock to rock. In some rocks the remanence of the magnetic minerals has been aligned with that of the Earth's magnetic field and can provide palaeomagnetic data.
- (b) Magnetic crystals are also found in the atmosphere. Dust particles containing magnetic minerals may have a magnetic remanence. However, the natural remanences of the various particles in any bulk atmospheric samples we investigated will not be aligned and so cannot be detected in such assemblages.
- (c) Atmospheric fallout and erosion and weathering of the Earth's crust contribute magnetic minerals to river systems. As with the atmospheric dusts, mineral magnetic studies can be performed on river sediments in order to investigate the concentration and types of assemblages of magnetic minerals contained in the sediments. Investigations of natural remanences again cannot be used because of the lack of alignment. Laboratory remanences, such as isothermal remanence are, however, readily usable in mineral magnetic studies of both river and atmospheric samples.
- (d) Recent lake and marine sediments are also derived from crustal erosion and atmospheric fallout. Their constituent magnetic particles may have natural remanences field during sediment formation. The alignment process may have been more efficient in some horizons producing a higher intensity of natural remanence (B versus G). In some horizons the remanence of the magnetic particles may not have been aligned (C and E). In other horizons

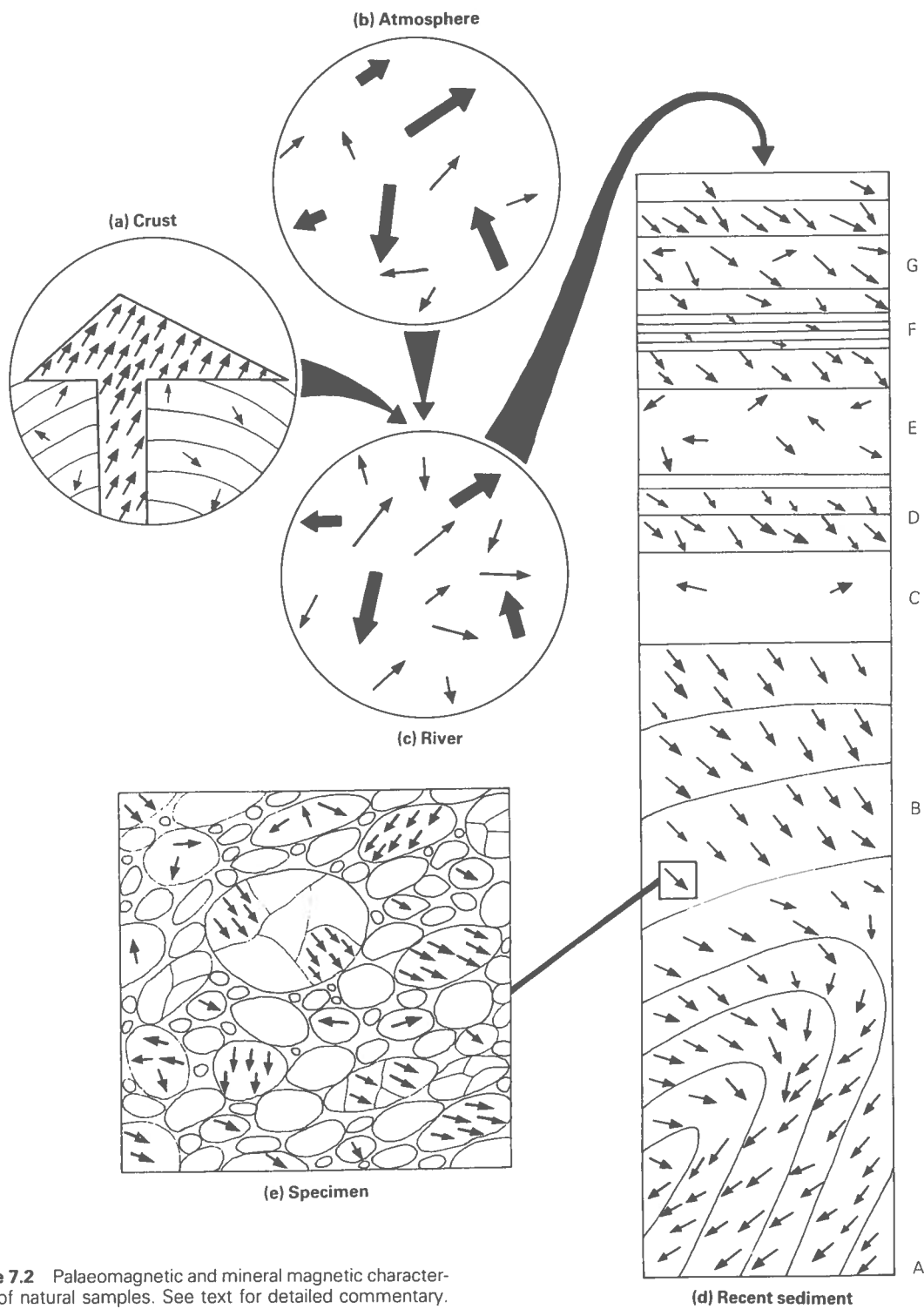


Figure 7.2 Palaeomagnetic and mineral magnetic characteristics of natural samples. See text for detailed commentary.

the remanence of the magnetic particles may be aligned but its direction may not coincide with that of the Earth's magnetic field (A). The types and concentrations of magnetic minerals may also vary from horizon to horizon (A to G).

- (e) Variations in magnetic properties are also found on a microscopic scale. For example a 10 ml specimen from a recent sediment core will be composed of a range of particle sizes and mineral types. Some of the particles may be magnetic. The composition and natural remanence of these particles will vary and the remanence may be aligned to a certain extent so the specimen as a whole has a natural remanence. This picture may be further complicated by some of the magnetic particles themselves being subdivided into magnetic and non-magnetic crystals and by the magnetic crystals being even further subdivided into magnetic domains.

Although, we will be mainly interested in variations in the magnetic properties of bulk samples on a macroscopic scale, we need to bear in mind the microscopic complexity when making environmental interpretations from mineral magnetic and palaeomagnetic measurements. Also, for our purposes it is useful to retain a clear distinction between palaeomagnetic studies and measurements (involved with the history of the Earth's magnetic field), and mineral magnetic studies and measurements (related to the intrinsic properties of magnetic minerals).

7.5 Sampling and measurement

Magnetic measurements demand their own approach to field sampling, and to sample storage, preparation and treatment. In the case of natural remanence measurements of sediments the main requirements are:

- (a) *Orientation.* A minimum requirement for inclination measurements alone is knowledge of which way is up. For relative declination measurements, constant azimuth must be established. For absolute declination, true azimuth must be known for each sample.
- (b) *Preservation of structure.* Samples should not be dried out or frozen at any stage between sampling and measurement. This applies equally well to

whole cores and to subsamples which should always be stored in a damp environment, e.g. in sealed plastic containers. The size of sample used will depend on the type of magnetometer available. 10 ml plastic cubes are ideal for use with fluxgate and parastatic magnetometers for 1' cylinder rock samples. Some superconducting magnetometers (Section 6.2.4) will only accept smaller samples.

- (c) *Isolation from stray magnetic fields.* Storage should be in magnetically screened environments wherever possible. This has the double advantage of isolating samples from contemporary fields which may induce artificial viscous remanences (Section 4.3.2) in the sample and establishing the samples in an environment where gradual decay of previously acquired viscous remanences will take place.

Samples for natural remanence measurement can be taken from free faces or from core tubes either split length ways or extruded from the base and many kinds of core samples provide material suitable for palaeomagnetic study. Alternative types of corer are described in a wide range of recent articles and reviews of which those by Hakanson and Jansson (1983), Goudie (1981), Barber (1976), Mackereth (1958, 1959), Davis and Doyle (1969) and Digerfeldt (1978) are especially useful.

Mineral magnetic measurements can be carried out on a very wide range of materials. Surfaces can be measured by magnetic susceptibility search loops and probes (Section 6.3.1). Here the main problem is ensuring constant geometry between surface and sensor for every location measured. Signals decline with distance following a power law, so spurious variations are easy to produce through careless field practice. Downhole susceptibility probe readings can be made using appropriate sensors from the Bartington Instruments range. Cores of rock, soil or sediment can be scanned for volume susceptibility provided, where necessary, they are retained in clean non-metallic core tubes or can be extruded into non-metallic liners without distortion and consequent volume variation (Section 10.3). A very wide range of mineral magnetic measurements can be carried out on subsamples of fresh, moist material provided it is not too loose or sloppy to retain physical coherence and magnetic moment after magnetisation or whilst spinning. Mass specific measurements on dried material pose no problems provided the material has

not been heated above *c.* 60 °C and the sample is packed so as to immobilise all particles. Filter paper residues are easily measured either by placing them in 10 ml plastic sample pots or laying them on special templates (Section 11.7). The special and subtle nature of magnetic contamination poses unexpected problems especially in artificial remanence measurements where the sample holder, filter paper or packing material is inevitably magnetised along with the sample. When dealing with weakly magnetic samples, all holders and packing materials should be screened by being washed then pre-magnetised and measured empty. Unsuitable pots and packing can then be rejected. Glass fibre filter paper is best avoided (Section 11.7) but if it is used, blanks must be pre-measured and values for them subtracted. With very weakly magnetic peat, the magnetic signal-to-noise ratio can be greatly improved simply by compression. Finally, the diamagnetic properties of sample holders and packing materials must be taken into account in susceptibility measurements of weak samples.

Although magnetic measurements are thus extraordinarily versatile the wide range of environments and applications considered and the rather divergent purposes and requirements of natural remanence as against mineral magnetic measurements, have led us to make special reference to sampling and measuring procedures where appropriate in each chapter.

7.6 Summary

It follows from the processes and relationships briefly described in this chapter that magnetic measurements are potentially of great environmental interest. The contribution of natural remanence measurements establishing chronologies of sedimentation may be vital to palaeoenvironmental studies in both marine and lacustrine environments. Coupled with rapid magnetically based core correlations, they greatly enhance our capacity for more reliable quantitative studies of material flux. In lake watershed ecosystems this approach is important especially where either the terrestrial or aquatic ecosystems are mineral-limited, or where changes in land use and vegetation have been associated with major shifts in material flux. Mineral magnetic parameters are valuable stratigraphic tools in palaeoenvironmental and palaeolimnological studies since they can be used alongside more time-consuming and often destructive analyses to give additional insights into sediment types and sources. At the same time, since the combustion-derived anthropogenic forms of magnetic minerals are often associated with processes releasing substances which are ecologically damaging to the environment (e.g. SO₂ and many heavy metals), magnetic monitoring of particulate pollution may also have important ecological implications.