HIGH-TEMPERATURE MAGNETIC SUSCEPTIBILITY BRIDGE

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HIGH-TEMPERATURE MAGNETIC SUSCEPTIBILITY BRIDGE

by

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ABSTRACT

A low-field (0.31 Oersted peak) a.c. susceptibility bridge has been built. Volume susceptibilities as low as $7 \times 10^{-8}$ emu/cc are detectable.

The bridge was calibrated over the range $10^{-6} - 10^{-2}$ emu/cc using two independent methods, namely (1) paramagnetic salts, and (2) a 40-turn coil. The two methods gave very good agreement.

A furnace, capable of attaining 800°C, was added to the bridge, so that susceptibility could be measured as a function of temperature. The furnace was made of non-magnetic Chromel-A heater wire and wound non-inductively.

Errors in room temperature susceptibility readings range from 2% for $k \sim 1 \times 10^{-2}$ emu/cc to 20% for $k \sim 1 \times 10^{-7}$ emu/cc. At present, room temperature susceptibilities of less than $4 \times 10^{-5}$ emu/cc cannot be measured to better than 20% as a function of temperature due to more serious restrictions during heating.

Possible improvements in the equipment capable of reducing these errors, as well as alterations to extend the usefulness of the apparatus, are discussed.

Specimens from several different rock types including lava flows, ignimbrites, red sandstones and dykes were subjected to high temperature susceptibility measurements in air. Results from these measurements generally confirm the results obtained from studies of Curie points and other rock magnetic properties by various authors.
CHAPTER 1

INTRODUCTION

1.1 Magnetic Susceptibility

If a magnetic field, \( \vec{H} \), induces a magnetic moment per unit volume, \( \vec{\mathbf{j}} \), in a material, then these quantities are related by the equation

\[
\vec{\mathbf{j}} = k \vec{H}
\]

(1.1)

where \( k \) is called the magnetic susceptibility of the material in the case of paramagnetic materials. For ferromagnetic materials \( k = k(H) \) and in general \( k \) is constant only over the initial portion of the magnetization curve. This initial (or "reversible") susceptibility, \( k_0 \), is defined as

\[
k_0 = \frac{d\mathbf{j}}{dH} \bigg|_{H=0}
\]

(1.2)

where the absence of vectors indicates that the magnetization is parallel to the applied field, i.e. perfect isotropy is assumed. Any further increase in the applied field produces an irreversible magnetization.

The magnitude of susceptibility depends on the type of magnetism, and is generally in the ranges:

- \( k \approx -10^{-6} \) emu/cc for diamagnetic materials
- \( k \approx +10^{-6} \rightarrow 10^{-4} \) emu/cc for paramagnetic materials
- \( k \approx 1 \rightarrow 10^5 \) emu/cc for ferromagnetic materials.

It should be noted that, in equation (1.2), \( k_0 \) is the true volume susceptibility and \( H \) is an external applied field. However, the magnetic field acting in a ferromagnetic material may not be the same.
as the applied field. In practice, the internal or effective field, $\hat{H}_{\text{eff}}$, is less than the applied field and is given as

$$\hat{H}_{\text{eff}} = \hat{H}_0 - N\hat{J}$$

(1.3)

where $\hat{H}_0$ is the applied field and $N$ is the demagnetizing factor.

The value of $N$ depends on the shape of the ferromagnetic material. For a spherical ferromagnetic grain the value is $4\pi/3$. The apparent susceptibility, $k_a$, can then be written as

$$k_a = \frac{k_0}{1 + Nk_0}$$

(1.4)

In natural rocks the susceptibility depends on a variety of factors, among them the composition of the actual rock, the shape and distribution of the ferromagnetic minerals, as well as possibly magnetic grain interaction and sample shape.

Important factors other than those mentioned above are:

(a) **Magnetic Field Strength**

It has been found that the susceptibility of ferromagnetic materials increases with an increase in the applied field up to a point (typically ~150 Oe for magnetite-bearing rocks) and then decreases again. This behaviour is expected from $dJ/dH$ on the virgin curve of a typical hysteresis loop (Strangway, 1967).

Worldwide measurements of paleointensities suggest that the amplitude of the Earth's magnetic field has not changed substantially over most of geological history (Tarling, 1971). Thus, any rocks which are used will have come under the influence of this field, and therefore it is advantageous to measure susceptibility in applied fields of the
order of magnitude of the present field. In such low fields susceptibility can be considered to be nearly, but not completely, independent of the field (Pandit, 1967).

(b) State of Magnetization

Contradictory evidence has been published on the effect of the remanent magnetization of a sample on its susceptibility. However, it is found that, generally, the susceptibility decreases with increasing remanent magnetization (Strangway, 1958, 1967).

(c) Grain Size

For a given material the susceptibility tends to increase with increasing grain size. This is especially important for diameters less than 50 microns and the rate of increase of $k$ is greatest when this size approaches a value determined by the transition from single-domain to multi-domain structure, since single-domain grains are magnetically hard and so tend to have low susceptibilities. Multi-domain grains are easier to magnetize since it is easier to move domain walls than to rotate magnetization vectors.

(d) Stress

Theory confirmed by experiments (See summary by Breiner, 1967) shows that susceptibility decreases with increasing stress in the direction of the applied stress. This susceptibility change is nearly linear with stress. Its magnitude depends on the rock type, but it is usually in the range of 1 - 3% in susceptibility per 100 kg/cm$^2$ stress. For stresses applied perpendicular to the axis of measurement, the results quoted by Breiner were inconsistent.
(e) Temperature

The changes in susceptibility produced by changes in the temperature depend on the type of material used. For diamagnetic materials there is no temperature dependence, whereas paramagnetic substances obey the Curie law $\chi = C/T$, where $\chi$ is the susceptibility per mol, $T$ is the absolute temperature and $C$ is a constant. Ferromagnetic materials are strongly temperature dependent. For single crystals, the susceptibility generally decreases with increasing temperature until the Curie temperature, $T_c$. Above $T_c$ the materials are paramagnetic, obeying the Curie-Weiss law $\chi = C'/(T-T_c)$ where $C'$ is a constant.

Since rocks are much more complex than a single crystal, the susceptibility variation as a function of temperature fits no simple expression. It has been found, however, that susceptibility generally increases on heating, occasionally by a large factor, before falling off sharply at the Curie temperature. Generally, if this increase is reversible, it will be due to the 'Hopkinson Effect' (Nagata, 1961 p.143).

1.2 Importance of Susceptibility Measurements

Both in geophysical prospecting and in its application to geological phenomena such as sea-floor spreading, knowledge of the initial susceptibility is necessary in the interpretation of anomalies in the Earth's magnetic field. Since susceptibility is field dependent, measurements should be made in fields on the order of the Earth's field, as pointed out previously. Since the origin of these anomalies may be as deep as some tens of kilometers below the surface, knowledge of the behaviour of the material as a function of temperature is of great importance.
Measuring susceptibility at high temperatures is an alternate way of obtaining a good estimate of Curie points (Chapter 3). In fact, the sharp decrease in susceptibility just below $T_c$ may result in more accurate values of $T_c$ than are obtained at high fields (Nagata, 1961). Since susceptibility depends on the mineral content of a specimen, any chemical transformation due to heating (Chapter 3) will at once be noticed.

The initial susceptibility is important to paleomagnetism because it enters the Königsberger ratio, $Q_n$, defined as

$$Q_n = \frac{J_n}{k_0 H}$$

which is the ratio of the natural remanent magnetization (NRM), $J_n$, to the induced magnetization, and has been found to be a useful gauge of stability of the NRM. For ferromagnetic rocks, this ratio commonly lies between 1 and 10, but may lie outside these limits. For rocks of the same general composition, $Q_n$ generally tends to decrease with increasing age; being frequently less than 1 for rocks of pre-Tertiary age, even in the case of igneous rocks.

1.3 Instruments for Measuring Susceptibility

Methods of measuring susceptibility developed by various workers are summarized in Table 1.1, along with figures for their attainable sensitivity. The classical instruments (e.g. balances by Kelvin, Curie and Gouy) were designed for high-field measurements of the weak paramagnetism or diamagnetism in various materials, but all methods listed are applicable to powdered or whole rock. The sensitivity requirements for measurements with ferromagnetic rocks tend to be as high as
Table 1.1 Some established methods of measuring magnetic susceptibility and anisotropy of susceptibility

<table>
<thead>
<tr>
<th>Method</th>
<th>Developed By</th>
<th>Field H (Oe)</th>
<th>Temperature (°C)</th>
<th>Correlation with k</th>
<th>Sensitivity (emu/cc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Balance</td>
<td>Kelvin (1890)</td>
<td>$10^3$ to $10^4$</td>
<td>-268 to 1000</td>
<td>Apparent loss of mass</td>
<td>$10^{-7}$</td>
</tr>
<tr>
<td></td>
<td>Curie (1903)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Gouy (1889)</td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>High</td>
<td>Measurement of electric charge</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Room temp.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1 to 10^3</td>
<td>To 800</td>
</tr>
<tr>
<td>Ballistic</td>
<td>Chevallier (1925)</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>Stschodro (1927)</td>
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</tr>
<tr>
<td></td>
<td>Nagata (1940)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mutual</td>
<td>Mooney (1952)</td>
<td>Low</td>
<td>Room temp.</td>
<td>Imbalanced resistance</td>
<td>$3 \times 10^{-6}$</td>
</tr>
<tr>
<td>Inductance</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bridge</td>
<td>A.C. Transformer</td>
<td>$\sim$1</td>
<td>Room temp.</td>
<td>Unbalanced signal</td>
<td>$\sim 10^{-8}$</td>
</tr>
<tr>
<td></td>
<td>Bridge Graham (1964)</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Inductance</td>
<td>Bruckshaw &amp;</td>
<td>$\sim$0.5</td>
<td>Room temp.</td>
<td>Differential voltage measured by a potentiometer</td>
<td>$1 \times 10^{-5}$</td>
</tr>
<tr>
<td></td>
<td>Robertson (1948)</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Inductance</td>
<td>Radhakrishnamurty &amp;</td>
<td>0.5</td>
<td>To 700</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sahasrabudhe (1965)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D.C. Astatic</td>
<td>Blackett (1952)</td>
<td>Low</td>
<td>Room temp.</td>
<td>Deflection</td>
<td>Varies with magnetometer sensitivity</td>
</tr>
<tr>
<td>Magnetometer</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(Anisotropy)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Spinner</td>
<td>Noltimier (1967)</td>
<td>To 70</td>
<td>Room temp.</td>
<td>Voltage</td>
<td>$10^{-8}$</td>
</tr>
<tr>
<td>Magnetometer</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(Anisotropy)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Torsion</td>
<td>Stone (1962)</td>
<td>To 40</td>
<td>Room temp.</td>
<td>Deflection</td>
<td>$5 \times 10^{-8}$</td>
</tr>
<tr>
<td>Balance</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(Anisotropy)</td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>
those for purely paramagnetic materials, since the large susceptibility of the ferromagnetic materials in the rock is effectively reduced by their dissemination in a comparatively non-magnetic groundmass, and because of the desirability of making measurements in low fields (Section 1.2).

The methods used to measure susceptibility of rocks (Table 1.1) have been reviewed in Collinson et al (1967) and by Pandit (1967). Subsequent to these reviews, some new susceptibility instruments have been reported in the literature. These include:

(a) Pandit's (1967) a.c. inductance bridge. This was built according to the design by Bruckshaw and Robertson (1948), in which the detector is a concentric double coil arrangement connected in series opposition with a third coil consisting of a small number of turns being used as a fine balance. This bridge is capable of a high signal-noise ratio, but proved to be difficult to balance, so that in practice, low-field (0.5 Oe) volume susceptibilities less than $5 \times 10^{-5}$ emu/cc cannot be reliably measured with it.

(b) Christie and Symons' (1969) solenoid transformer bridge, which will be further discussed in Section 2.1, is capable of detecting volume susceptibilities as low as $5 \times 10^{-8}$ emu/cc in a field of 0.3 Oe.

(c) Stephenson and de Sa's (1970) a.c. bridge, in which a furnace capable of reaching 700°C was incorporated, has a noise level equivalent to $1 \times 10^{-6}$ emu/cc in a field of 2.5 Oe.

1.4 Objective

The aim of this investigation was to build an instrument for
measuring susceptibility of rock specimens in low inducing fields. The instrument had to be sensitive enough to measure weakly magnetic rocks (10^{-7} - 10^{-5} \text{ emu/cc}). It had to include a furnace to allow the measurement of susceptibility as a function of temperatures as high as 800°C.

The objective also included the measurement of temperature-dependence of susceptibility on a selection of rock specimens from different coastal areas across the North Atlantic. The purpose of this was two-fold:

1. To test the reliability of the instrument; and
2. To provide an important additional magnetic parameter for these rocks, which had previously been subjected to different kinds of magnetic measurements by other members of the geophysics group at Memorial University.
their instrument, while retaining high sensitivity. Furthermore, the stability of the system with respect to drift was satisfactory if certain precautions were taken as described later.

2.2 The Susceptibility Bridge

The theory behind the bridge is simple. It is made up of two, nearly identical, balanced assemblies, each consisting of a pair of Helmholtz coils coaxial with a pick-up coil. Fig. 2.1 shows the relative positions of the two assemblies. The two pick-up coils are subjected to the a.c. field produced by their respective Helmholtz coils. Since these fields are equal, and the pick-up coils are connected in series opposition, there is no signal from them until a magnetic sample is placed in one of the pick-up coils. The net emf output is due to the induced magnetization, which is a measure of the susceptibility of the sample. The actual construction of the bridge is more complex because of the difficulty in producing identical assemblies.

2.3 The Bridge Construction

A Princeton Applied Research (PAR) HR-8 oscillator and lock-in amplifier provides a 1000 Hz signal that is used to excite the two sets of Helmholtz coils, H1 and H2 (Fig. 2.2), which are connected in series with a 0.038 µF capacitor for maximum current flow. The fields produced induce a voltage in the two pick-up coils, P1 and P2, which are connected in series opposition with capacitors C2, C3, C4 across P2. These capacitors are used to resonate the pick-up system. Since it is extremely difficult to build identical coils, an additional winding, H3,
P: Pre-Amplifier

H1, H2: Helmholtz Coils - 1000 turns - No. 22 Copper Wire
(25 layers of 40 turns each)

H3: Counterwound Coils - 50 turns - No. 22 Copper Wire

P1, P2: Pick-up Coils - 1000 turns - No. 28 Copper Wire

C1: 0.038 \mu F, C2: 0.001-0.01 \mu F, C3: 0.0001-0.001 \mu F, C4: 15-350PF
(Air Capacitor)

R1: 1200 \Omega, R2: 500 \Omega, R3: 10 \Omega

Figure 2.2 Wiring Diagram for Susceptibility Unit
is added to one of the two assemblies. It is used to adjust the field slightly, and resistors R1 - R3 are used for fine balancing of the conductive component. The net output from the pick-up system is fed into a modified PAR Type B pre-amplifier.

This pre-amplifier has a transformer input for low input impedances, with an initial gain of 100. The transformer connections were changed to reduce the gain to approximately 52, as this resulted in fewer long- and short-period drifts. The signal then passes through a linear amplifier, a narrow-band amplifier, a phase-sensitive detector, and a d.c. voltmeter. This voltage is simultaneously displayed on a Hewlett-Packard (Model 3430A) digital voltmeter and on a Hewlett-Packard (Model 7100B) chart recorder.

2.3.1 The Inducing System

Since the bridge consists of two almost identical transformers, only one will be described. Fig. 2.3 shows a cross-section of the transformer. A uniform alternating magnetic field is produced by the pair of Helmholtz coils. Using the equation in Naqata (1961, p. 72), it can be shown that the maximum deviation from a uniform field over the space occupied by a cylindrical specimen of diameter 2.22 cm and height 2.54 cm is less than 1%. Theoretically, the peak field produced by each pair of Helmholtz coils for a peak current of 1.9 mA is 0.30 Oe, and measurement with a small search coil gives a value of 0.31 Oe.

2.3.2 The Pick-Up System

The pick-up coil (Fig. 2.3) rests on three screws which are...
Figure 2.3  Helmholtz and Pick-Up Coil Assembly
used to center it. The inside dimension of the coil had to be made large enough to accommodate a furnace and cooling assembly designed to take cylindrical rock specimens of 2.22 cm diameter. In the absence of the furnace, there is enough room to accommodate 3.5 cm cubes or 5.0 cm diameter cylinders, increasing the sensitivity by a factor of about four, and facilitating measurement of the anisotropy of susceptibility.

For the same current used previously, the field of the Helmholtz coils produces a peak voltage of 0.79 volt in each pick-up coil. In order to measure susceptibilities of the order of $10^{-7}$ emu/cc, it was necessary for the differential voltage between the two pick-up coils to be seven orders of magnitude less than the voltage of each coil.

2.4 The Heating System

In order to measure susceptibility of rocks as a function of temperature, it was necessary to build a furnace that could heat specimens up to $800^\circ$C. The furnace, which had to be non-inductive and non-magnetic, was built as described below.

Thirty-four turns of size 22 Chromel-A heating wire were wound non-inductively, i.e. seventeen doubled back, on a quartz tube (Fig. 2.4; fewer than the actual number of turns are shown). The wire was cemented to the tube with a thin layer of high-temperature ceramic. This was necessary to avoid any slipping of the wires which would cause a short-circuit.

The furnace was then placed inside a second quartz tube separated from it by a layer of magnesium oxide to insulate the furnace and to hold it in place. The insulation is quite effective; it takes 15 - 20% more current to attain a given temperature without it.
Figure 2.4 Furnace
This assembly is placed inside a third glass tube. Water is circulated in the space between the second and third tubes to provide cooling.

To measure a specimen, it is placed on a quartz sample cradle (Fig. 2.4) which is then lowered into the furnace tube. There it rests upon a quartz pedestal in the center of the pick-up coil.

At the beginning of an experiment a thermocouple is lowered into the tube and rests on top of the rock. In this way the surface-temperature of the rock, rather than the temperature of the air, is measured. The thermocouple is connected to a Leeds and Northrup temperature potentiometer.

Since knowledge of the temperature distribution inside the furnace is essential, the thermocouple was lowered to the bottom of the furnace, and after the furnace had attained an equilibrium temperature, the thermocouple was slowly raised and the temperature measured at height intervals of the order of $\frac{1}{2}$ cm. The results of these measurements, obtained at two different mean temperatures (Fig. 2.5), indicate that the maximum temperature difference in the volume occupied by a standard specimen is $15^\circ$C. This difference probably is always further reduced during actual heating and cooling of a specimen, since thermal conduction within the rock would tend to decrease the temperature gradient.

The furnace was used in only one of the transformer assemblies. This arrangement frees the second assembly for future use in anisotropy or low-temperature measurements. An identical furnace in each assembly (with the second furnace left empty) might have reduced some of the difficulties due to non-symmetry described in the next section.
Figure 2.5 Temperature Distribution along Furnace Axis
2.5 The Cooling System

Since the transformer assemblies are very temperature-sensitive, especially if only one of them is affected by the heat treatment, several possibilities for cooling the furnace were explored:

(1) Air at room temperature, forced between the second and third glass tube, was not very effective in removing the heat produced by the furnace.

(2) An attempt was made to cool the furnace by using tap water. This was tested with the furnace switched off. Since the tap water was much below room temperature and since only one of the two assemblies was cooled, the output of the bridge changed drastically. This drifting in the output voltage continued and no equilibrium was established after more than an hour's run. Using two cooling systems in parallel, one for each assembly, might have achieved a satisfactory result, but it was not attempted.

(3) A further alternative was a closed system. This was attempted using two 3-gallon containers of water. This method was satisfactory except that the water temperature rose about 20°C over the 1½ hours required to heat a specimen. To eliminate this problem, the cooling system was expanded, using an 80-gallon capacity plastic tub as the main reservoir. The water was pumped from there into a large, plastic-coated wooden tray of dimensions 105 cm x 93 cm x 13 cm, from which it flowed through the system (Fig. 2.6). The flow-rate is adjustable. Due to the large volume of water available, the temperature rose less than a degree Centigrade even when the furnace was in continuous use.
Figure 2.6 Susceptibility Unit Showing the Cooling System

A - Wooden Tray  B - Water Tub  C - Furnace
Since the water in the reservoir was about 2°C colder than were the assemblies, it cooled the one containing the furnace, causing a small drift in the differential output voltage. However, the bridge achieved equilibrium after about 20 minutes and the balance was not affected any further. This method of cooling the furnace was finally adopted, since it gave the most satisfactory results.

2.6 Calibration

Several possible ways exist to calibrate a susceptibility bridge.

One of the most widely used methods appears to be the one where the susceptibility of several specimens, usually rocks also carrying a remanence, is determined using an astatic magnetometer (Pandit, 1967). The specimens, ranging over several orders of magnitude, generally $10^{-4} - 10^{-2}$ emu/cc, are then measured in the bridge and the calibration is achieved in that manner.

A second method is to use paramagnetic salts (Collinson et al, 1963). This method is useful for all ranges up to about $2 \times 10^{-4}$ emu/cc. It was used in the present calibration to give values in the lower ranges and to confirm the calibration procedure adopted below.

A third method was also employed. It uses the principle of modelling induced magnetization of a sample by passing an a.c. current through a coil. A single turn of wire produces a magnetic dipole moment, $\hat{m}$, which is given as

$$\hat{m} = \frac{i}{10} \, dS$$  (2.1)

where $i$ is the current in amperes and $dS$ is the mean cross-sectional area.
of the loop in cm$^2$.

The magnitude of the total dipole moment/unit volume, $J$, for a coil of $N$ turns, height $h$ cm and radius $r$ cm, is

$$J = \frac{N}{10} \frac{\pi r^2}{h} = \frac{Ni}{10h}$$  \hspace{1cm} (2.2)

Since the susceptibility is $k = \frac{J}{H}$, where $H$ is the applied field along the axis of the coil, then, assuming that $H$ is parallel to $J$,

$$k = \frac{Ni}{10hH}$$  \hspace{1cm} (2.3)

Forty turns of number 32 copper wire were wound evenly on a lucite rod of diameter 2.22 cm and height 2.54 cm. The dimensions were chosen so that the coil would closely resemble the shape of a standard specimen.

This coil was connected to a Hewlett-Packard (Model 6824A) Power Supply-Amplifier, which was operated in the Amplifier mode. It was fed from the PAR HR-8 oscillator and was therefore operating at the same frequency as the bridge. The current passing through the coil was determined by measuring the voltage drop across a standardized resistor using a Philips (Model 2403) Voltmeter.

The bridge was then calibrated by measuring the current, converted into volume susceptibility, and the corresponding voltage from the pick-up coils. Fig. 2.7 shows the calibration curve.

Three salts were prepared to confirm this calibration. They were (Collinson et al., 1963):

1) Manganese Sulphate  \hspace{1cm} (x = 6.46 \times 10^{-5} \text{ emu/g})
2) Cobalt Nitrate \hspace{1cm} (x = 3.31 \times 10^{-5} \text{ emu/g})
3) Nickel Nitrate \hspace{1cm} (x = 1.36 \times 10^{-5} \text{ emu/g})
Figure 2.7 Calibration Curve

- Standard Coil
- 1, 2, 3 Standard Salts 

see text
The salts were powdered and then placed in containers having the same inside dimensions as the calibration coil. If the volume of the containers and the mass of the salts are known, the susceptibility/unit volume of the salts may be calculated. The three points are shown in Fig. 2.7 and confirm the coil calibration.

Since a number of specimens were less than 2.54 cm in height, a correction factor had to be applied to the bridge output readings. This factor was obtained by shortening the calibration coil and recalibrating. This was achieved by 10 successive removals of 2 turns each from the 40-turn coil and performing mini-calibrations at each stage. This procedure resulted in the correction curve (Fig. 2.8) for samples of height from 1.27 cm to 2.54 cm.

For any specimen in this height range, the output voltage of the system has to be multiplied by the correction factor for the corresponding height. This corrected voltage is then used to determine the susceptibility of the specimen.

The bridge calibration indicates a linear relationship between susceptibility and voltage output. The minimum detectable signal, which is 1 μV, corresponds to a susceptibility of 7 × 10⁻⁸ emu/cc. This high sensitivity makes the bridge suitable for measuring most rocks useful for paleomagnetic studies as well as for measuring susceptibility anisotropy of the relatively more magnetic rocks.

2.7 Errors

Uncertainties in the susceptibility measurements may be broken into two main categories - those present in
Figure 2.8 Correction of Susceptibility for Sample Height
(A) the room-temperature measurements, and
(B) the high-temperature measurements.

There are several sources of errors which contribute to (A). Some of these are minor errors like:

(i) The error involved in the calculation of the specimen height. By making several height measurements, this error can be reduced significantly.

(ii) Axial positioning of the specimen. The error involved here is minimal, since the specimen always rests on the pedestal. Even as much as a 1 mm positioning error along the axis causes only a 0.5% error in susceptibility, and the error resulting from a lateral offset of the same amount would be negligible.

(iii) Short-term drift. This can be greatly reduced by taking 'zero' readings before and after the specimen has been measured, as well as by repeat measurements. The measurement may be made with one of the longer time constants available on the HR-8, in order to reduce short-term fluctuations.

(iv) Errors in determining the inducing magnetic field were small since accurate electronic measuring instruments were used.

(v) Frequency drift. The frequency of the inducing field fluctuates by ±0.1% over short periods and even less, when short periods are averaged, over periods of hours.

The two major sources of errors are in (vi) the measurement of the output voltage of the system and in (vii) the susceptibility determi-
(vi) The lock-in amplifier (PAR) has a gain accuracy, after calibration, of 1% on the range calibrated and 2% on all other ranges. This accuracy was confirmed by switching ranges and measuring the same output on the different ranges. Since the voltage readings are displayed on a digital voltmeter, there is no error involved in reading the meter scale on the amplifier.

(vii) The error in the susceptibility calibration is difficult to estimate. Both $N$ and $h$ in equation (2.3) contain negligible errors. The error in $i$ will certainly be less than 1% in the three higher ranges ($i = 0.002$ ma to 2 ma) and better than 5% in the lower range.

Errors in the susceptibility determination, using the salts, arise from errors in the measurements of the internal volume of the containers and the weight of the salts. The volume is determined to better than 1% and errors in weighing are negligible since they were performed on a Sartorius (Model 2600) single-pan balance with an accuracy of better than 0.05%. Impurities in the salts (the manufacturer states a purity of greater than 97.5%) may represent the largest error in this determination.

Besides the above-mentioned errors there are two additional sources of error in the high temperature measurements:

(viii) Long-term drifting of the bridge output, of a period of approximately one hour, represents a major problem for weak rocks. The 'zero' measurement is taken at the start and at the end of the run, which generally takes two to three hours. Any change in this 'zero' is interpolated linearly over the run. However, it was found that the 'zero' would oscillate about this
line with a period of about one hour, and that the amplitude of the oscil-
lation represents a susceptibility variation of about $\pm 7 \times 10^{-6}$ emu/cc.

(ix) A second limitation placed on high-temperature measurements
is due to the effect of heating the furnace, as the wiring may not be per-
fectly non-inductive or may contain small ferromagnetic traces. This
effect was observed during an empty-furnace run and found to be appreciable
only for rocks in the range of $10^{-5}$ emu/cc and less. Since the effect is
reproducible to about 10% for all temperatures up to 800°C, these empty-
furnace readings are subtracted from the total output.

The effects due to (viii) and (ix) represent a susceptibility
of about $4 \times 10^{-5}$ emu/cc at high temperatures and thus place a lower limit
on the susceptibility which may be measured reliably as a function of
temperature.

The expected errors in the determination of susceptibility
may be summarized as follows:

<table>
<thead>
<tr>
<th>Susceptibility (emu/cc)</th>
<th>Total Expected Error in Bridge Measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>$&gt; 1 \times 10^{-2}$</td>
<td>2</td>
</tr>
<tr>
<td>$&gt; 1 \times 10^{-3}$</td>
<td>3</td>
</tr>
<tr>
<td>$&gt; 1 \times 10^{-4}$</td>
<td>5</td>
</tr>
<tr>
<td>$&gt; 1 \times 10^{-5}$</td>
<td>7 at $4 \times 10^{-5}$ emu/cc</td>
</tr>
<tr>
<td>$&gt; 1 \times 10^{-6}$</td>
<td>10</td>
</tr>
<tr>
<td>$&gt; 1 \times 10^{-7}$</td>
<td>20% at $4 \times 10^{-5}$ emu/cc</td>
</tr>
<tr>
<td></td>
<td>Bridge should not be used</td>
</tr>
<tr>
<td></td>
<td>in these ranges</td>
</tr>
</tbody>
</table>
Errors in the temperature determination are two-fold:

(i) Uncertainties in the thermocouple voltage output. Since this was checked against a standardized thermocouple, the error is less than 1°C.

(ii) The furnace temperature distribution. The maximum temperature variation over the volume occupied by a specimen is 15°C at high temperatures and may thus be taken as the maximum error in the temperature determination. Because of the reduction of this temperature gradient by the thermal conductivity of rock specimens, an estimate of the maximum error in any quoted high-temperature value on the average is about ±10°C.
3.1 Introduction

The thermal dependence of susceptibility has been investigated for several different rock types. To determine consistency and reproducibility of results for these rocks, at least three specimens were processed from each of the different areas. Heating of the specimens, which was carried out in air, proceeded until their susceptibility dropped to nearly zero. The temperature was raised an additional 50 - 70°C and maintained at that level for several minutes before the specimens were slowly cooled. The susceptibility-vs.-temperature curves were generally recorded on a Hewlett-Packard (Model 7100B) strip-chart recorder. One of the typical runs is shown in Fig. 3.1. The zero level is interpolated between the start and the end of the run. The output is measured from this level and, after correction for specimen height, is converted into susceptibility from the calibration curve and is replotted. The rates of heating and cooling may be observed from the figure and correspond to about 100°C per 15 minutes. This rate was chosen after subjecting some specimens from the Wabana red sandstones (Section 3.6) to varying rates of heating. It was found that specimens would explode if heated more rapidly. Unfortunately further experimentation concerning the rate of heating was not carried out until after the conclusion of the present investigation. These experiments showed that the rate of heating could be increased substantially without breaking specimens other than those from Wabana.
Figure 3.1 Recording of Susceptibility Run for Cloud Mountain
However, as the rate is increased, a question on thermal lag in a specimen arises. Specimens from several localities were reheated, reaching 625°C in about 20 minutes compared with the previous 90 minutes required to reach the same temperature. The apparent Curie temperatures obtained for the fast heating were between 8°C and 15°C above those obtained using the slow heating. This increase was attributed to thermal lag, which was greater in the case of the fast heating. This difference in observed Curie temperatures can be eliminated by heating the specimen rapidly to just below its Curie temperature, leaving it at this temperature for several minutes to reduce the thermal gradient in the specimen, and then heating the specimen more slowly until just above the Curie point. This procedure, apart from greatly reducing thermal lag, allows measurements to be made much more quickly than by slow heating over the whole temperature range.

When the heating and cooling curves of Figs. 3.2 - 3.8 were plotted, it was found that for all the specimens the steep portion of the cooling curves was between 15°C and 40°C below the steep portion of the heating curves. To determine the cause of this discrepancy, a small hole was drilled axially to the center of one of the specimens from Henley Harbour (Fig. 3.2) and the thermocouple lowered into this hole. When the heating and cooling curves were plotted, there was no difference between the two curves. Hence the reason for this apparent phase lag between the original heating and cooling curves must be due to the fact that during heating the thermocouple reads above, and during cooling it reads below, the average specimen temperature. This procedure was adopted for at least one specimen from each site, and the proper correction made on the diagrams for all other specimens.
On each curve the susceptibility, $k$, is expressed in electromagnetic units (emu/cc) as obtained from the calibration curve in Section 2.6 and is the apparent rather than the true susceptibility (Section 1.1).

3.2 Specimens from: Henley Harbour

Several specimens from a single lava flow near Henley Harbour on the south coast of Labrador were subjected to susceptibility measurements. The specimens are fine-grained black basalts of presumed Lower Cambrian age. Murthy (1967) measured the natural remanence (NRM) intensities of 20 samples (155 specimens) and obtained values in the range $0.13 - 2.5 \times 10^{-3}$ emu/cc. The NRM intensities of the three fresh specimens chosen for the present study varied between $0.92 - 1.23 \times 10^{-3}$ emu/cc, which gave $Q_n$-values (equation 1.5) between $0.41 - 0.65$. $Q_n$-values less than unity are not unusual in such old rocks, even those of volcanic origin, because of the relative long time available for randomizing domain alignments contributing to the remanence. However, these low $Q_n$-values may indicate the significant presence of an unstable remanence component in these rocks. Murthy inferred such a component in his specimens from the fact that the observed mean NRM direction was not very different from that of an axial dipole field computed for present time. He confirms the presence of some instability in the otherwise quite stable NRM of these rocks by alternating field (AF) demagnetization.

Murthy (1967) further suggests that "... the relative inhomogeneity in the magnetization may be a reflection of partial chemical alteration in the rock". This suggestion may give a clue to the hump found in the susceptibility heating curves (Fig. 3.2), whereas it is absent in the cooling curves. In all three cases, this hump is superimposed upon a
Figure 3.2 Temperature Dependence of Volume Susceptibility for Specimens from Henley Harbour, Labrador.
gradual increase in susceptibility on heating to near the Curie point, 
\( T_c \), followed by a sharp susceptibility drop, giving \( T_c = 577^\circ \text{C} \). This
temperature corresponds to the Curie point of magnetite. The fact that 
the hump did not reappear during cooling suggests that the chemical 
change which produced it was irreversible.

Pandit (1967) measured room temperature susceptibilities of 
some Henley Harbour specimens and obtained average values of \( 3.7 \times 10^{-3} \) 
and \( 3.8 \times 10^{-3} \) emu/cc for samples HH24 and HH26 respectively. Considering 
the inhomogeneity of these rocks, these values compare well with the 
present single-specimen values of \( 4.4 \times 10^{-3} \) and \( 4.5 \times 10^{-3} \) emu/cc.

Somayajulu (1969) thermally demagnetized several Henley 
Harbour specimens. The intensities decreased sharply to \( 350^\circ \text{C} \), remaining 
early constant between \( 350 - 450^\circ \text{C} \) and increased again above \( 450^\circ \text{C} \). The 
temperature range \( 350 - 450^\circ \text{C} \) is marked by a fairly sharp drop in suscep-
tibility which may indicate removal of a magnetic component in the 
specimens. Somayajulu suggests that the stable component (with \( T_c \) greater 
than \( 500^\circ \text{C} \)) may be due to magnetite or titanomagnetite. This seems to be 
confirmed by the susceptibility curves.

3.3 Table Head

Table Head is located about 15 km north-east of Henley Harbour, 
still on the Labrador coast. There appears to be only one basalt flow 
present, which is possibly the same flow as at Henley Harbour and hence 
is also of presumed Lower Cambrian age (Murthy, 1967). The NRM intensities 
of the specimens measured by Murthy had a fairly wide spread, about 
\( 0.33 - 6.2 \times 10^{-3} \) emu/cc; the majority of the Table Head specimens being
more magnetic than those from Henley Harbour. This is certainly confirmed by the susceptibility measurements (Fig. 3.3). Pandit (1967) obtained a value of susceptibility of $10 \times 10^{-3}$ emu/cc as the average of three specimens from TH6, compared with $11.5 \times 10^{-3}$ emu/cc for the single specimen TH6C2 in Fig. 3.3. The higher susceptibilities for Table Head compared with Henley Harbour probably are due to a greater percentage of magnetite in the Table Head specimens. The $Q_n$-values are below 1, with TH6C2 giving a value of 0.23.

Except for their larger NRM- and k-values, the Table Head basalts show very similar behaviour to the Henley Harbour basalts; the susceptibility curves (Fig. 3.3) also give an indication of a hump, though it is not as pronounced as in the Henley Harbour basalts. The mean Curie temperature for the three specimens is about $571^\circ$C, compared with $577^\circ$C for Henley Harbour. The cooling curves for specimens TH20B and TH16bI are fairly typical for basalts and their shape points to the removal or destruction of an intermediate-temperature ($300 - 450^\circ$C, approx.) magnetic component. TH6C2 shows an anomalous behaviour, particularly in its cooling curve where the usual "Hopkinson" peak is replaced by a gradual increase in k on cooling from $500 - 400^\circ$C. To test this feature, the specimen was taken through a second heating-cooling cycle which gave reversible curves close to the cooling curve of Fig. 3.3. The much lower susceptibility value after cooling may indicate that a large percentage of the magnetite may have changed to some other mineral such as hematite. Hematite had been found associated with skeletal titanomagnetite in a polished section from sample TH6 analyzed by Dr. N. D. Watkins, then at Florida State University, who comments (Somayajulu, 1969 p.179): "This is an unusual example of coexisting low and high oxidation states, respectively."
Figure 3.3 Temperature Dependence of Volume Susceptibility for Specimens from Table Head, Labrador
Stepwise thermal demagnetization (Somayajulu, 1969) of several Table Head basalt samples showed results similar to Henley Harbour basalts, except that in the former case the intensities did not fall off as sharply with increasing temperature. This is probably correlated with the much smaller hump on the Table Head heating curves, and suggests greater stability on the part of the Table Head samples.

3.4 Cloud Mountain

There is only a single lava flow exposed on Cloud Mountain near Roddickton on the Northern Peninsula of Newfoundland. It is believed to be of Lower Cambrian age and may have erupted about the same time as Henley Harbour and Table Head. Some measurements on this flow are reported in Somayajulu (1969). The intensities of the specimens he measured range from $1 \times 10^{-3}$ to $7 \times 10^{-3}$ emu/cc and the $Q_h$-values for the three specimens in Fig. 3.4 range from about 1.5 to 4. These values are significantly higher than either the Table Head or the Henley Harbour values.

The susceptibility-vs.-temperature curves (Fig. 3.4) look very similar to those from Henley Harbour and Table Head. There is again an indication of a hump, but not as pronounced as in the Henley Harbour basalts. The Cloud Mountain basalts have a very noticeable rise just before falling off very steeply towards the Curie point close to 570°C. This is a typical feature in susceptibility curves for basalts (Radhakrishnamurty and Likhite, 1970). The Curie point is similar to that of both Henley Harbour and Table Head values and is just below that of pure magnetite. Polished sections of a specimen examined by Dr. N. D. Watkins (See section 3.2) (Somayajulu, 1969 p.179) showed the presence of titanomagnetite containing exsolution lamellae.
Figure 3.4 Temperature Dependence of Volume Susceptibility for Specimens from Cloud Mountain, Newfoundland
In summary, the measurements of temperature dependence of susceptibility for the three basalt localities, Cloud Mountain, Table Head and Henley Harbour have shown considerable similarities in their thermomagnetic behaviour. This lends support to the view of geologists (Clifford, 1965; Strong and Williams, 1972), that all three basalts originated under similar conditions during Lower Cambrian times.

3.5 Killary Harbour

The three specimens that were measured came from the lower-most two of six bands of ignimbrites (welded tuff) from outcrops of mid-Jurassic age near Killary Harbour, Eire (Murthy and Deutsch, 1971). Magnetic results from a preliminary collection of these rocks have been reported by Somayajulu (1969) and Deutsch and Somayajulu (1970). As in the case of the specimens from the sites discussed previously (Sections 3.2 - 3.4) much of the magnetic material was in the highest state of oxidation (N. D. Watkins, reported in Somayajulu, 1969), pseudobrookite and hematite frequently being found associated with titanomagnetite. NRM intensities ranging from $0.01 \times 10^{-3}$ to $6 \times 10^{-3}$ emu/cc probably indicate wide variations in ferromagnetic mineral content between different samples. The three specimens in Fig. 3.5 had $Q_n$-values between 1 and 6, indicating a stable remanence which confirmed the results of the detailed AF and thermal demagnetizations and of a positive fold test reported by the above authors.

During thermal demagnetization in air most of the remanence was lost below 600°C, leaving less than 20% above this temperature. This suggested that the main magnetic component in these specimens was magnetite or titanomagnetite and that the high-Curie point component may have been
Figure 3.5 Temperature Dependence of Volume Susceptibility for Specimens from Killary Harbour, Ireland
hematite. This would be consistent with the above-mentioned results from polished sections.

The susceptibility curves (Fig. 3.5) confirm the above conclusions, giving a mean value for the main Curie point of 567°C. Room temperature susceptibility values of several specimens not shown in Fig. 3.5 cover a range of over two orders of magnitude. This is consistent with the large variations of NRM intensities. The heating curves show irregularities in the intermediate temperature range similar in magnitude to those from Table Head and Cloud Mountain. The cooling curves are very similar to those obtained for all three previous localities (Sections 3.2 - 3.4), indicating a removal or destruction of some of the magnetic material, although in the case of KH51 only, the hump observed during heating reappears on cooling at a somewhat lower temperature; this might be due to a low-temperature titanomagnetite component. The susceptibility curves did not fall completely to zero, which may confirm the presence of some hematite in the specimens.

Contrary to the specimens in Sections 3.2 - 3.4, the susceptibility curves for Killary Harbour specimens do not show prominent "Hopkinson" peaks, nor do they resemble the typical "single-domain (SD)"-type curve of Radhakrishnamury and Likhite (1970), which are reversible and fall off much more gradually towards the Curie point than do those in Fig. 3.5. However, six Killary Harbour polished sections examined by Dr. G. S. Murthy (private communication) showed that the main ferromagnetic mineral in these ignimbrites is magnetite or titanomagnetite, present in grains of size less than 5 microns, i.e. a significant part of the magnetization may be carried in single domains, and the relatively high Qn-values would bear this out. A final conclusion regarding single- versus multi-domain state in the present specimens cannot be made on the basis of the results in Fig. 3.5.
Wabana is one of several red sandstone formations of Lower Ordovician age on Bell Island, Newfoundland. The rock is an oolitic iron ore, believed to be of primary origin. The major minerals contained in these rocks are hematite ($\alpha$-Fe$_2$O$_3$), chamosite ($\left(\text{Fe}_4\text{Al}_2\right)\left(\text{Si}_2\text{Al}_2\right)\text{O}_{10}(\text{OH})_8$) and siderite (FeCO$_3$), all of which contain iron, although only hematite is ferromagnetic at atmospheric temperatures and is believed to be the only significant carrier of the NRM. Rao (1970) has made a detailed thermomagnetic study of these rocks. The NRM intensities of his specimens ranged from $3.5 \times 10^{-5}$ to $10 \times 10^{-5}$ emu/cc, and stepwise thermal demagnetization showed a general increase in the intensity of magnetization, measured at room temperature, when the specimens had been heated to temperatures close to their Curie point; this was followed by a steep decrease after further heating to 685°C. Heating above 690°C and then cooling produced an intensity increase of one to two orders of magnitude.

High-field susceptibility measurements on ten Bell Island specimens, carried out in air using a Curie point balance built by L. G. Kristjansson (Deutsch et al., 1971), had shown a gradual increase in magnetic moment ($M$) up to 600°C and then a sharp drop near 685°C. However, $M$ never dropped to zero, and upon cooling these specimens from 685°C or higher temperatures, $M$ was observed to increase by one to two orders of magnitude, similar to the case of thermal demagnetization. This behaviour is also exhibited by the low-field susceptibility curves shown in Fig. 3.6. The susceptibility remains almost constant to about 550 - 600°C, then increases rapidly up to about 640°C, with a subsequent drop corresponding to a mean Curie temperature of about 670°C, confirming that the
Figure 3.6 Temperature Dependence of Volume Susceptibility for Specimens from Bell Island, Newfoundland
mineral is probably hematite. Magnetite, if at all present, could be available in only very small amounts since no drop in susceptibility was observed near its Curie temperature. Cooling of the specimens to 500°C resulted in enormous increases in susceptibility. Further cooling to room temperature decreased this susceptibility by about 20%. The large increase suggests a physical or chemical change in the magnetic mineral present. Additional susceptibility runs on a series of fresh specimens showed that this change takes place over a temperature range from about 570 – 700°C. These temperature values were determined by several consecutive reheatings of a specimen to successively higher temperatures. This procedure showed that, for maximum temperatures less than about 570°C, the heating curves were reversible on cooling, whereas above 570°C, the susceptibility values, obtained on cooling, increases sharply with increasing maximum temperature to which the specimen had been heated.

To test the stability of the new mineral that seems to have been produced by the initial heating, each specimen in Fig. 3.6 was subjected to a reheating. The curves obtained are shown in Fig. 3.7. Three interesting features are noticeable:

(1) The susceptibility generally increases to about 520°C before dropping off sharply to near zero at about 570°C.

(2) There is a second rise in susceptibility to about 640°C followed by a drop indicating a second Curie point at 670°C. This latter rise is the same as the one shown in the original heating and is certainly due to hematite. Comparison of these high-temperature rises in Figs. 3.6 and 3.7 shows that a major part of the original hematite is still present, indicating that the enormous susceptibility increase is not mainly due to
Figure 3.7 Temperature Dependence of Volume Susceptibility for Specimens from Bell Island, Newfoundland (Reheating).
any alteration of the hematite. Rao (1970) carried out a second and a third heating-cooling cycle in air, measuring a powdered Wabana specimen with the Curie point balance. All three curves had Curie points between 610°C and 620°C, and the third heating-cooling cycle was reversible with the second cooling curve. Some additional specimens used in the present analysis, but not shown in Fig. 3.7, also exhibited high Curie points, ranging from about 580 - 615°C, i.e. a fairly broad spectrum of Curie points is involved. Since heating of whole specimens in the present apparatus was much slower than that of powders using the Curie balance, an investigation into the effect of a change in the rate of heating and cooling of specimens may clear up some of these inconsistencies.

(3) The second cooling curves show a significant reduction in the susceptibility at 520°C as compared with the original cooling curves. A third heating and cooling (not shown in Fig. 3.7) showed an even further reduction in the susceptibility at that temperature. However, in all cases the room-temperature susceptibility remains essentially unchanged from its value after the initial cooling, even at the end of the third heating-cooling cycle. Several more heatings of a given specimen may be necessary to stabilize the susceptibility curves.

It is not clear what causes the large increase in both the low-field and high-field susceptibility curves. In discussing his results, Rao (1970) suggests that the new material may be maghemite (γ-Fe₂O₃), which is strongly ferrimagnetic. The presence in the rock of considerable siderite could have played a significant part in producing such maghemite, though the former is paramagnetic. Because of the metastable behaviour of maghemite, its Curie point has not been definitely established, but a
$T_c$-value in the neighbourhood of 620°C is possible (See summary in Rao, 1970, p.147). The conclusion regarding maghemite can be neither verified nor disputed by the results obtained during the present investigation.

3.7 Indian Harbour

Several specimens from a Precambrian swarm of gabbroic dykes near Indian Harbour on the Labrador coast were measured. From one of these dykes, Grasty et al. (1969) had obtained a whole-rock age of 2080 ± 42 m.y., but they believe that this date might be anomalously high, possibly through argon addition. The NRM intensities varied between $0.03 \times 10^{-3}$ and $9.5 \times 10^{-3}$ emu/cc, although most specimens were in the range of $1 - 2 \times 10^{-3}$ emu/cc (Murthy, private communication). The results of AF and thermal demagnetization obtained by Murthy and Deutsch (1972) indicated high stability of these specimens. For the three specimens chosen in Fig. 3.8, the $Q_n$-values lie between 0.55 and 0.75, which is relatively high for such old rocks.

The high-temperature susceptibility curves (Fig. 3.8) for L6a and L10A are nearly reversible. The curves differ from the usual curves for basalts in that the susceptibility did not increase just below the Curie point, but rather the susceptibility decreases at first slightly and then rapidly towards the Curie point. This is similar to the trend of the susceptibility curves for single-domain (SD) rock as discussed by Radhakrishnamurty and Likhite (1970). A predominantly single-domain behaviour of the Indian Harbour specimens would be compatible with the observed high stability of the NRM, but further study, particularly of polished sections, is needed. The simple trends of the three heating curves
Figure 3.8 Temperature Dependence of Volume Susceptibility for Specimens from Indian Harbour, Labrador
in Fig. 3.8 indicate that the measured susceptibility is due to a single component of titanomagnetite with a mean Curie point of 557°C. Similar simple trends and $T_c$-values had been obtained from the thermal demagnetizations (Murthy and Deutsch, 1972). Specimen L7b showed a slightly different behaviour from the other two specimens during both its heating and its cooling portions. Possible changes of grain-size distribution in L7b during heating may have been responsible for the irreversible trend observed on cooling.
CHAPTER 4

SUMMARY AND SUGGESTIONS

4.1 Summary

An a.c. magnetic susceptibility bridge has been built similar in design to that of Christie and Symons (1969), which is designed for room temperature only. The present instrument was enlarged to permit the measurement of susceptibility as a function of temperature; or to measure larger specimens in the absence of the furnace. The bridge consists of two nearly identical Helmholtz coil and pick-up coil assemblies. The Helmholtz coils produce a peak field of 0.31 Oe at 1000 Hz and induce a corresponding voltage of 0.79 volt in each pick-up coil. Once the system is nearly balanced, there is a 'zero' output; a magnetic specimen placed into the field of one assembly causes the system to be unbalanced. The difference in the output is proportional to the magnetic susceptibility.

The instrument is very sensitive; an output of 1 μV, which is just detectable, being equivalent to a volume susceptibility of $7 \times 10^{-8}$ emu/cc.

The bridge was calibrated using two independent methods:
(a) Paramagnetic salts were used in the range of $10^{-5} - 10^{-4}$ emu/cc, and
(b) a 40-turn coil was used to calibrate the bridge over the ranges from $10^{-6} - 10^{-2}$ emu/cc.

A correction curve for specimens departing from standard height was obtained. This increases the versatility of the instrument, permitting specimens of heights 1.27 cm to 2.54 cm to be measured.
Chromel-A heater wire was used to build a non-inductive, non-magnetic furnace capable of producing temperatures as high as 800°C. Circulating water provided effective cooling for the system.

At room temperature about one specimen can be measured per minute. When measurements are made as a function of temperature, a heating and cooling cycle will take about one and a half hours.

Errors in the susceptibility values are mainly due to short- and long-term fluctuations in the bridge, and to errors in the voltmeter and in the calibration of the bridge. Estimated total errors in a room-temperature measurement range from 2% for \( k > 1 \times 10^{-2} \) emu/cc to 20% for \( k > 1 \times 10^{-7} \) emu/cc. Since these errors are for single measurements and since systematic contributions to them are minor or absent, the error may be reduced significantly by performing repeat measurements.

Errors in the temperature determination are mainly due to the furnace temperature distribution and amount to about ± 10°C.

The susceptibility of specimens from half a dozen localities bordering the North Atlantic was measured in air as a function of temperature.

It was found that Lower Paleozoic specimens from Henley Harbour and Table Head, Labrador, and Cloud Mountain, northern Newfoundland, exhibited very similar behaviour. The heating curves for all three areas showed the presence of some alteration in the ferromagnetic material, and the cooling curves indicated the destruction of a ferromagnetic component. The specimens were cooled from above their Curie points, which were all in the range 570 - 580°C, close to that of magnetite.

The susceptibility curves for Ordovician ignimbrites from Ireland showed large variations in magnetic mineral content between speci-
mens with the main component being magnetite or titanomagnetite. Hematite may also be present since the susceptibility did not drop completely to zero even at 625°C.

Oolitic sandstone of Ordovician age from Bell Island indicated hematite as being the main magnetic component with a Curie temperature of about 670°C. The specimens exhibited extraordinary behaviour upon cooling, producing susceptibility increases of from one to two orders of magnitude. The newly formed mineral seems to have a fairly broad range of Curie points (570 – 620°C) and is possibly maghemite. Reheating the specimens shows that most of the original hematite is still present.

Two of the three Precambrian gabbroic dyke specimens from Indian Harbour, Labrador, suggest a simple, predominantly single-domain structure, which is consistent with their observed highly stable NRM. The Curie points had a mean value of 557°C, indicating titanomagnetite as the main ferromagnetic component.

4.2 Suggestions

Since one of the major problems in measuring high-temperature susceptibility is the long-term drift in the zero, steps should be taken to eliminate this problem.

One possible way is to use a much shorter time to complete a heating-cooling cycle than was the practice in the present investigation.

A second way would be to redesign the furnace in such a way that the specimen could be removed from its center while remaining at a given temperature. This would require increasing the length of the furnace-winding at least three-fold. The removal of the specimen would permit
checking the zero at any time. Presently, this can only be accomplished by completely removing the specimen from the furnace, consequently causing a large drop in the specimen temperature before the specimen can be recentered in the furnace.

Due to the high sensitivity of the bridge it will be possible to investigate some interesting relationships, such as:

- Remanent magnetization and susceptibility.
- Thermal dependence of coercivity and susceptibility.
- Variation of susceptibility with $H$ in very low a.c. fields.
- The empty assembly may be used to measure the anisotropy of susceptibility on larger than normal specimens.

Some other improvements of the apparatus would consist of additions to the equipment. For example, the bridge could be used for measuring low-temperature susceptibilities if the furnace were replaced with a properly designed cooling assembly.

Another change in design of the furnace would permit high temperature measurements in vacuum. This is especially important to reduce effects due to oxidation.
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