A THERMOMAGNETIC ANALYSIS OF ROCKS (20-700°C): THE DESIGN AND CONSTRUCTION OF MEASURING APPARATUS, AND ITS APPLICATION TO ROCKS FROM THE WHITESHELL AREA

A thesis
Presented to
The Faculty of Graduate Studies
University of Manitoba

In Partial Fulfillment
of the Requirements for the Degree
Master of Science, Geophysics

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by
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October 1970

ABSTRACT

As a preliminary study, relating to the regional magnetic anomalies in Manitoba and Northwestern Ontario, a reconnaissance sampling survey has been made over the flank of a regional anomaly in the Whiteshell area of Southeastern Manitoba.

Thermomagnetic curves and Curie transition temperatures have been determined for 70 samples over the 14 mile traverse. The Curie temperatures are closely grouped about a mean of 573°C. It is considered improbable that the magnetic minerals found at the surface are directly related to a possible Curie level at the bases of the regional magnetic anomalies.

The design and construction of the recording thermomagnetic balance is described. A commercial electrobalance is coupled with an air-cored coil system and a furnace to provide sub-saturation thermomagnetic curves between 20° and 700°C., in air. The heating and cooling cycle may be completed in about one hour.

Indications are that rapid heating to 610°C. produces no significant changes in Curie temperatures, but heating to 700°C. may cause a decrease in Curie point, for the magnetic phases present in these rocks. This latter effect is considered not to be due to the presence of air.

ACKNOWLEDGEMENTS

The writer wishes to express his many thanks to Dr. D. H. Hall of the Department of Earth Sciences, University of Manitoba, for his supervision and for his advice and encouragement during the period of this study.

Thanks are due to Dr. A. C. Turnock for his help and advice on materials for the furnace and temperature measurements.

The author wishes to thank Mr. R. Fanset for his invaluable assistance during the collection of samples, also Miss I. Berta and Miss J. Lee for preparing and measuring some of the samples.

The author is grateful to Mr. M. Coey who kindly provided samples of synthetic magnetite.

This study would not have been possible without the contributions to construction of the equipment from Messrs. Trider, Gould, Grassler, White, Griffiths, Healey and Hildebrand.

The generous help provided by Messrs. Coyle and Rennie of Delro Ltd. during difficult times in the drilling programme, is much appreciated.

This work could not have been achieved without the encouragement and moral support of my wife.

The field work and construction were made possible by support from the National Research Council and the Geological Survey of Canada. The author also acknowledges the receipt of a Student Assistantship in the Department of Earth Sciences.

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CHAPTER I

INTRODUCTION

The present study is part of a continuing program of geophysical investigation into crustal structure in the Canadian Shield, being conducted by the Department of Earth Sciences, University of Manitoba.

Quantitative interpretations have been made of regional magnetic anomalies in North Western Ontario and Manitoba (Hall 1968, McGrath and Hall 1969, Bhattacharyya and Morley 1965). It is apparent that these anomaly systems are closely related to crustal structure, and the concept of crustal magnetic units has been used (Hall 1968). Pakiser and Zeitz (1965) have briefly considered the possibilities of regional anomalies, with respect to crustal structure, in the U.S.A.

Studies in rock magnetism have indicated a close connection between the magnetic properties of rocks and their chemical and physical history (Nagata 1961). Investigation of the magnetic properties may be used therefore as an aid to disclosing at least some parts of the physical and chemical history of the crust, and thereby its present structure. However, magnetic properties are controlled by a number of physical, chemical and petrological factors and although in many cases the boundaries of magnetic units may coincide with lithologic boundaries, they need not necessarily do so.

Interpretations of some of the regional magnetic anomalies in the Canadian Shield indicate that the magnetic units which give rise to the anomalies lie in the upper crust. The lower boundaries have been interpreted by Bhattacharyya and Morley (1965) as representing the mean depth of the Curie point geotherm, and by Hall (1968) as being related to the intermediate seismic discontinuity. The mean depth of the lower boundaries of the anomalies discussed is about 20 km.

The rocks at 20 km. depth are not directly accessible for laboratory analysis, and one can only sample surface rocks in the relevant areas, in order to study the problems of the Curie isotherm and of the origins of the magnetic anomalies. Analyses made on surface rocks will assist in the construction of a petrological model of the crust which would account for the magnetizations of the magnetic units.

The object of the present work has been to make a preliminary reconnaissance survey of Curie temperatures of surface rocks in the vicinity of a regional magnetic anomaly in South Eastern Manitoba (McGrath and Hall 1969).

Determinations of Curie temperatures will assist one in deciding whether rocks of the magnetic type found at the surface could be responsible for the magnetic anomalies with bases at about 20 km. However, the rocks found at the surface may continue at depth, but with significant changes in their magnetic minerals, such that the Curie level differs from that estimated from surface measurements. On the other hand, a significant change in overall rock type with changes in magnetic minerals might occur at about this depth.

Estimates of temperatures in the Canadian Shield have been given by Birch (1955), who, under different assumptions, suggests that at 20 km. depth, the temperature might be as low as 230°C or as high as 440°C. Such estimates are, by their nature, very approximate.

No means of determining thermomagnetic curves were available at the beginning of this study, and part of this thesis describes the design and construction of equipment for this purpose.

CHAPTER II

MAGNETISM IN ROCKS

Magnetism in matter

It will be useful to outline some relevant features of magnetic properties of matter in general, prior to a discussion of magnetism in minerals and rocks. When matter is placed in a magnetic field, it experiences a magnetization, the nature of which is dependent on the material. We define a magnetic susceptibility, as the ratio of the magnetization, to the applied field.

Diamagnetism is a property of all matter, and is present only when an external field is applied. Temperature does not enter explicitly into the diamagnetic susceptibility, which is always negative.

Paramagnetism (positive susceptibility) is found where the total spins of the atoms are not zero, i.e. the atoms have magnetic moments. Magnetization results from the orientation of the magnetic moments in an applied field while thermal disorder resists the tendency of the field to orient the moments. The temperature dependence of the susceptibility x of normal paramagnets is given, to a first approximation, by the Curie Law.

$$x = \frac{C}{T}$$

where C is the Curie constant, and T is the temperature.

Discussions of diamagnetism and paramagnetism may be found, for example, in Morrish (1965).

A small number of materials exhibit ferromagnetism, which is characterized by a spontaneous magnetization in the absence of an applied field. It is now well established that there exist small domains, each spontaneously magnetized, the magnetization of the whole specimen being the vector sum of the magnetizations of the domains. Since the directions need not be parallel, the resultant magnetization may be zero. Application of a field changes the domain arrangement, and hence an appreciable magnetization appears.

Ferromagnetism is brought about by the strong interactions between uncompensated spins in neighbouring atoms, such that the spins become aligned in a parallel manner. Other forms of strong coupling occur. The phenomenon of antiferromagnetism in its simplest form consists of two sub-lattices, one of whose spins tends to be antiparallel to those of the other (with no net spontaneous magnetization). In the case of ferrimagnetism, an asymmetry exists either in the crystallographic positions of the sub-lattices, or in the presence of ions of differing magnetic moments in the sub-lattices, or both. There is a net spontaneous magnetization in the case of ferrimagnetism.

The spontaneous magnetization is a function of temperature, and becomes zero at the Curie temperature.

Rock magnetism

The magnetic properties of rocks are the results of the effects of the minerals which comprise them. Four kinds of magnetic effect are found in rocks: diamagnetism, paramagnetism, ferrimagnetism and antiferromagnetism. Minerals which are composed of ions which have no permanent magnetic moments are diamagnetic, whereas those which contain ions such as Fe^{2+}, Fe^{3+}, Mn^{2+}, may be paramagnetic, ferrimagnetic or antiferromagnetic. If there is no effective interaction between the magnetic ions, the mineral is paramagnetic, while if strong interactions exist, the mineral may be ferrimagnetic or antiferromagnetic. Thus in the magnetic sense, rocks are comprised of a number of paramagnetic, ferrimagnetic and antiferromagnetic grains in a diamagnetic matrix.

The only significant magnetic ions in rocks are Fe^{2+}, Fe^{3+} and Mn^{2+}, although others may be present in small amounts in some minerals. The other common ions Si^{4+}, Al^{3+}, Mg^{2+}, Ca^{2+}, Na^{+}, K^{+}, Ti^{4+}, P^{5+} have no permanent magnetic moment. Nagata (1966) lists some of the more common paramagnetic minerals: olivines, pyroxenes, garnets, biotite, amphiboles, cordierites.

Ferrimagnetic minerals in rocks may be divided into two small groups: certain metallic oxides and certain metallic sulphides. Ferrimagnetic oxides such as magnetite, titanomagnetites, titanomaghemites, hemoilmenites are found in the majority of rocks. Ferrimagnetic sulphides, pyrrhotites of varying compositions, are much more restricted in occurrence. To illustrate the relative abundance of the ferrimagnetic oxides, Akimoto (1957) considers that the ferrimagnetism of over 99% of Japanese igneous rocks at atmospheric temperature comes from that of the titanomagnetites, and the remaining 1% or less originates in the ferrimagnetic portion of the hematite-ilmenite series.

Certain end members of the above solid solution series of oxides are antiferromagnetic: namely hematite, ilmenite, ulvospinel (at low temperatures); although hematite and ilmenite, to a lesser extent, show a weak parasitic ferromagnetism which may be due to lack of perfect antiparallelism of vectors (Stacey 1963).

The ferrimagnetic minerals possess a strong spontaneous magnetization and are the most significant in rock magnetism. The spontaneous magnetization is dependent on temperature in a manner which varies with the composition of the mineral. Its magnitude at a particular temperature is characteristic of the mineral, as is the temperature at which it becomes zero, i.e. the Curie temperature.

An outline of the theory of ferrimagnetism

The theory of ferrimagnetism was first discussed by Néel (1948), (reviewed by Smart 1955). Néel considered in some detail the magnetic properties of simple inverse ferrites. These have the inverse spinel crystal structure, where the lattice has per unit molecule one tetrahedral A site surrounded by 4 oxygen atoms, and two octahedral B sites surrounded by 6 oxygen atoms. The ferrites may have the formula MFe_2O_4, where M is a divalent ion. The inverse spinels, of which magnetite Fe_3O_4 is one, have an Fe^{3+} ion on the A site, with an M ion on one B site and an Fe^{3+} ion on the other. The interactions between A and B sites are strong and negative, and the A-A and B-B interactions are weak, causing antiparallel alignment of spins, with an interdependence between the spontaneous magnetizations σ_A and σ_B of the two sub-lattices.

Néel's theory of ferrimagnetism, in terms of the modified molecular field theory, gives the relations for the spontaneous magnetizations, i.e. when the external field is zero, (adapted from Stacey 1963, Smart 1955).

$$\frac{\sigma_A}{\sigma_{A_0}} = B_S\left(\frac{\gamma_{BA}\sigma_B + \gamma_{AA}\sigma_A}{kT}\right)$$

$$\frac{\sigma_B}{\sigma_{B_0}} = B_S\left(\frac{\gamma_{AB}\sigma_A + \gamma_{BB}\sigma_B}{kT}\right)$$

where σ_{A_0} and σ_{B_0} represent values at complete alignment, γ_{AB} , γ_{BA} , γ_{AA} , γ_{BB}

represent the interactions, k is Boltzmann's constant and B_s is the Brillouin function appropriate to a total electron spin 's' per cation:

$$B_s(a) = \frac{2s+1}{2s} \coth\left(\frac{2s+1}{2s} a\right) - \frac{1}{2s} \coth\left(\frac{a}{2s}\right)$$

Since γ_{AB}, γ_{BA} are negative and equal, σ_A and σ_B are oppositely directed and the net magnetization is $(\sigma_B - \sigma_A)$. In the case of magnetite, the theory predicts a value of 4 Bohr magnetons per molecule, or 92 emu/gm. which agrees well with experiment (Nagata 1961).

The reciprocal susceptibility at high temperatures is given by:

$$\frac{1}{\chi} = \frac{T^2 - C\gamma_{AB}(\lambda\alpha+\mu\beta)T+\lambda\mu C^2\gamma_{AB}^2(\alpha\beta-1)}{C[T-\lambda\mu C\gamma_{AB}(2+\alpha+\beta)]} \qquad (2.1)$$

where T is absolute temperature

N is a number of magnetic ions per unit volume

λ is the fraction of magnetic ions on A sites

$\mu = 1-\lambda$ is fraction of magnetic ions on B sites

C is Curie constant for N magnetic ions

$$\alpha = \frac{\gamma_{AA}}{\gamma_{AB}} \quad , \quad \beta = \frac{\gamma_{BB}}{\gamma_{AB}}$$

The $\frac{1}{x}$ - T curve is a hyperbola (Smart 1955).
The numerator in equation(2.1) may be factored into

$$(T - T_C)(T - T'_C)$$

$$T_C = \frac{1}{2} C\,\gamma_{AB}\left(\lambda\alpha + \mu\beta - [(\lambda\alpha - \mu\beta)^2 + 4\lambda\mu]^{1/2}\right)$$

$$T'_C = \frac{1}{2} C\,\gamma_{AB}\left(\lambda\alpha + \mu\beta + [(\lambda\alpha - \mu\beta)^2 + 4\lambda\mu]^{1/2}\right)$$

At T = Tc, the susceptibility becomes infinite, which on the molecular field theory indicates a Curie point. (T'c represents the Curie point for $\gamma_{AB} > 0$, parallel alignment of spins). The above theory considered only one type of magnetic ion. If more than one type is present, the general principles and results remain the same, although the analysis is more difficult.

Techniques have been discussed by Néel (1948), Smart (1955), Morrish (1965), by which the spontaneous magnetization as a function of temperature may be obtained by graphical and numerical methods.

Further developments have been noted by, among others, Morrish (1965), Belov (1965). For example, Belov discusses the region near the Curie transition in terms of the thermodynamic theory introduced by Landau and Lifshitz.

He has found that in both simple and mixed ferrites, the magnetization curves near the Curie point obey quite well the same thermodynamic equation used for ferromagnetic substances, namely

$$\alpha_1 + \beta_1\sigma^2 = \frac{H}{\sigma}$$

where σ is the resultant magnetization of the sub-lattices of the ferrite, H is the applied field, α_1 and β_1 are thermodynamic coefficients, dependent on pressure and temperature. α_1 and β_1 are related to an ordering parameter η such that when $\alpha_1 = 0$, $\eta = 0$ ($\beta_1 > 0$ at all temperatures). Since the Curie transition is one from order, $\eta = 1$, to disorder, $\eta = 0$, a determination of $\alpha_1(T)$ will define the Curie temperature. $\alpha_1 < 0$, $T < T_C$; $\alpha_1 = 0$, $T = T_C$; $\alpha_1 > 0$, $T > T_c$.

In the strict sense, the Curie temperature is only applicable in the absence of an external field. When a field is applied, the Curie transition occurs through a range of temperatures.

It is found in general that the temperature dependence of magnetization exhibits a "tail",(fig.1). It is proposed by Belov (1965), for example, that the "tail" is probably due to fluctuations of the chemical composition through the volume of the specimen, or to inhomogeneities in the lattice structure. He quotes results which suggest that the "tail" is shorter for more homogeneous materials.

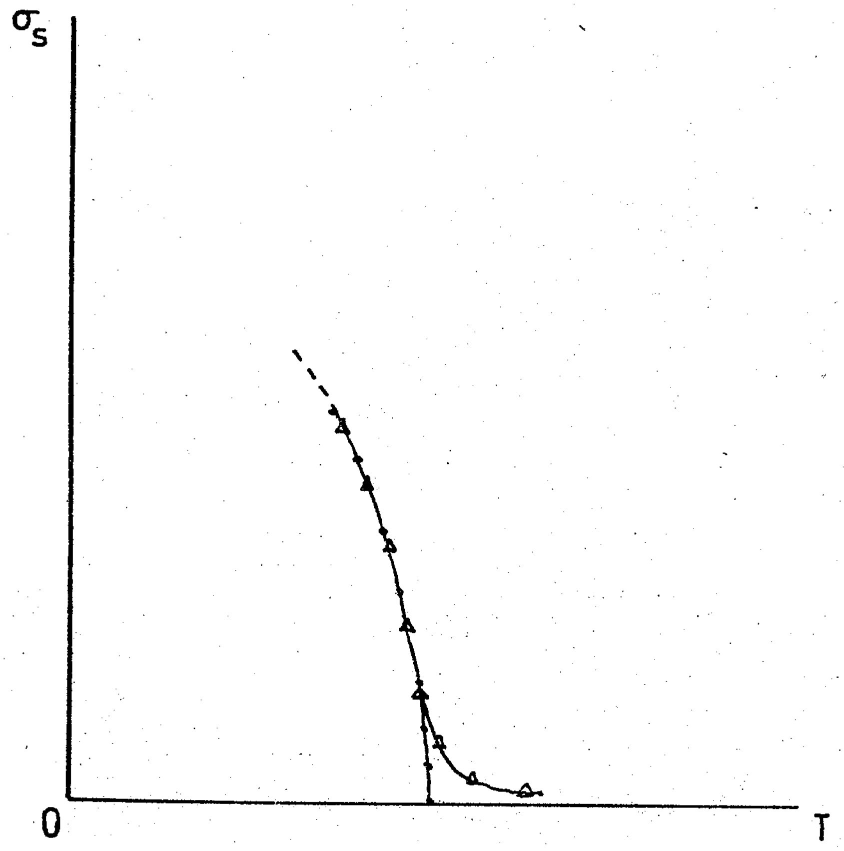


Fig. 1. Temperature Dependence of the Spontaneous Magnetization of Ferrites.

An idealized representation of the dependence of the spontaneous magnetization σ_s on temperature T, as determined by two methods:

• method of thermodynamic coefficients

△ method of lines of equal magnetization.

From a practical standpoint, one has to reach a decision as to what to take as the Curie temperature. In the absence of a field the Curie temperature has greatest significance and methods are available by which one may extrapolate from measurements in an external field to the situation at zero field (e.g. Belov 1965, Williams 1966).

However, in methods in which great precision is not required, or not possible, empirical definitions of the Curie point have arisen. A commonly used definition has been to take the point of inflexion of the measured curve to represent the Curie point (e.g. Bozorth 1951, Selwood 1956). Another definition used is to let the point of maximum concave curvature represent the transition point. (Ade-Hall et al. 1965). Neither of these is precise, and in some situations may be far from the true transition point (e.g. in certain ferromagnetic alloys, Belov 1965). However, for the present purposes, as a working definition, the point of inflexion on the measured thermomagnetic curve will be considered to represent the Curie point.

CHAPTER III

THERMOMAGNETIC ANALYSIS

Geological and geophysical significance

Thermomagnetic analysis in its broadest sense refers to any experiment which measures the change of a magnetic parameter as a function of temperature (Wasilewski 1969). In the restricted sense, in which it is generally used in rock magnetism, it means the measurement of the magnetization of a specimen over a range of temperatures, which usually includes the Curie point.

The results of studies in rock magnetism have numerous applications. Information has been gained regarding the past history of the geomagnetic field, of the evolution of crustal features such as the continents and ocean basins, and on a smaller scale, the development of the crust within the continents.

Many of these studies have involved measurements of the natural remanent magnetizations which are assumed to carry information relating to the past histories of the rocks. Thermomagnetic analyses may be applied to the rocks to test for stability of the magnetic minerals.

Thermomagnetic analyses, in the restricted sense, may be used in identifying magnetic minerals, since the Curie temperature, and also the saturation magnetization, are characteristic properties of a mineral.

In the present study thermomagnetic analysis is used to obtain information which will lead, with other information, to a knowledge of deep crustal structure.

Determination of thermomagnetic curves

A number of experimental methods exist which can be used for determining Curie temperatures, some of which give complete thermomagnetic curves. Some methods involve the measurement of a force in a non-uniform magnetic field, while others measure changes in electrical or thermal properties. Descriptions of methods may be found, for example, in Bates (1963), Collinson et al. (1967).

A method which has been commonly used in rock and mineral magnetism has been the Curie-Chevenau type of balance, (Larochelle 1961), where the sample is mounted on a beam suspended by a torsion fibre, and subjected to a force due to a non-uniform magnetic field. The measurement of the force has been made by noting the deflection of the beam, or by nulling the deflection by adjusting the torsion head. Variation of temperature will result in a thermomagnetic curve.

Disadvantages of this method are that the torsion fibre is very fragile and unpredictable in life time, and that the non-uniform field has to be known over a large volume, or alternatively, that continual readjustment of the torsion head is needed. An advantage is that the dead weight of the sample is not significant.

Translation balances of the null deflection type, with several fibres, may be used but again difficulties are likely to arise with the suspension system.

With the advent of sensitive and accurate electrobalances, the measurement of a vertical force became a relatively minor problem. Since the electrobalances use the null deflection method of balancing, no large region of known non-uniform field is needed; the sample does not move, and is held in position automatically. A disadvantage is that the dead weight of the sample now enters in, but arrangements can be made to allow for this.

A system using an electrobalance may be easily modified for determinations of other magnetic parameters such as coercive force, and remanence, which are not possible with most other arrangements.

The other methods, e.g. Vollstadt (1968), de Sa (1968), Petersen (1967), involving thermal capacity, electrical properties, or changes in characteristics of transducers with changes in susceptibility were generally discounted on account of the larger amounts of material needed as samples and/or because their application was restricted only to Curie point determination. None was as potentially versatile as a magnetic balance method.

The method chosen in the present study has therefore been a form of Faraday method using a commercial electrobalance to measure the force produced on a sample in a non-uniform field.

Theory of method

The force $\underline{F}$ on a magnetic dipole $\underline{m}$ in an applied magnetic field $\underline{H}$ is given by

$$\underline{F} = \nabla(\underline{m}.\underline{H})$$

to a first approximation (Cheston 1964).

For a body of volume v, the force may be written:

$$\underline{F} = \int_v \nabla(\underline{J}.\underline{H})\,dv$$

where $\underline{J}$ is the intensity of magnetization of the body.

In the case of a ferromagnetic substance, $\underline{J}$ is a complicated function of $\underline{H}$,

$\underline{J} = \underline{J}_o + \underline{J}(\underline{H})$

where $\underline{J}_o$ represents any remanent magnetization.

For polycrystalline materials, assumed isotropic,

$$\underline{F} = \int_v \nabla\left[\int_o^H J\,dH\right]dv$$

$$= \int_v \nabla\left[HJ - \int_o^H H\frac{\partial J}{\partial H}\,dH\right]dv \ .$$

For the z component of the force,

$$F_z = \int_v \left[J\frac{\partial H}{\partial z} + H\frac{\partial J}{\partial z} - \frac{\partial}{\partial z}\int_o^H H\frac{\partial J}{\partial H}\,dH\right]dv \ . \qquad (3.1)$$

Thus, the force is a non-linear function of the field and magnetization.

If we consider a paramagnetic substance where $J = xH$, then $\frac{\partial J}{\partial H} = x$, $\frac{\partial J}{\partial z} = x\frac{\partial H}{\partial z}$ and

$$F_z = xHv\frac{\partial H}{\partial z}$$

for $H\frac{\partial H}{\partial z}$ constant over v .

If, for a ferromagnetic substance, saturation occurs, then

$$\frac{\partial J}{\partial z} \longrightarrow 0 \quad , \quad \int_0^H H\frac{\partial J}{\partial H}dH \longrightarrow \text{constant in } z \, .$$

Thus, at saturation, the relation

$$F_z = Jv\frac{\partial H}{\partial z}$$

is approached, for $H\frac{\partial H}{\partial z}$ constant over v .

This provides a means of determining J_S, the saturation magnetization.

In the present work, saturation is not achieved and so equation (3.1) applies. It is seen that the force gives a measure of the magnetization but the exact relationship depends on factors related to the ferromagnetic structure.

In the foregoing, no account has been taken of demagnetizing fields. These reduce the effective field to $H - DJ$, where D is the demagnetizing coefficient. In the present work no precise knowledge of D is possible, and no allowance will be considered. The effects will undoubtedly be different for different grains in a given rock sample.

The sample is to be placed on the axis of symmetry of the field system; thus the 'x' and 'y' (horizontal) force components are minimized. The magnetic field and force measurement directions are both vertical, ('z' axis).

Sub-saturation fields are used here for two reasons: firstly a coil system was available in part; secondly, Curie temperatures are rather more sharply indicated at lower fields.

CHAPTER IV

DESCRIPTION OF EQUIPMENT

Electrobalance

The force measuring device in the present equipment is a commercially available electrobalance (Cahn Company, Model R.G.). This unit operates on the null balance principle, using a photoelectrically operated feedback system. A change in electromagnetic force equal to the change in test force is exerted in order to restore the balance beam to the null position. The gain of the feedback loop is high, and the restoring action is fast enough for the beam to be considered motionless for present purposes. This lack of deflection is particularly suitable in this application. The sensitivity is better than 1 microgram force.

One can consider a typical rock as having a volume susceptibility of 10^{-4} or more. As an estimate of the force exerted on a sample 0.1 c.c. in volume, consider the sample in a field of 200 oersteds, the gradient being 20 oersteds per centimetre.

Since, following earlier notation,

$$F \simeq x v H \frac{\partial H}{\partial z} ,$$

the force is found to be 0.4 milligrams. The balance is capable therefore of being used with samples of even lower susceptibility.

The limits of usability lie not with the balance itself, but rather with its mounting.

The beam mechanism is provided with a housing, which has a sliding door at the front for loading purposes. This housing is rigidly mounted on a plywood platform, which is bolted to a framework of angle iron on a movable base. The balance platform may be adjusted in height relative to the iron framework.

The angle iron frame has three lower platforms. The uppermost of these carries the coil systems which provide the magnetic field; the next one supports the D.C. supply units, the thermocouple cold junction, and plumbing for the water cooled furnace; the lowest level is for a furnace controller.

The electrobalance controls and the Texas Instruments "Serviriter II" strip chart recorder are mounted on a separate table to eliminate vibrations and mechanical feedback, which were found to cause problems. Also, during a run, one may wish to operate switches to change scales and ranges; this is more easily accomplished with a separate support.

Magnetic field system

The force to be measured is produced by a non-uniform magnetic field. In systems that have been built previously, (e.g. Doell and Cox, 1967, Schwarz, 1968), the more usual method of providing such a field has been to use a commercially available iron cored electromagnet, with specially shaped pole pieces.

Such a system has several advantages, but also some disadvantages. On the credit side, high fields (several kilo-oersteds) may be readily achieved and the units are ready-built. On the other hand, such units are expensive; they require very sturdy mountings, as their weights are great; water cooling is needed (though this is not too great a problem); because of remanence in the iron core, separate bucking coils and supplies are needed to null the field. Several special pole pieces are also required for different gradients, and these have to be changed manually.

The advantage of using high fields is that saturation of the magnetic material may be more nearly achieved, but it has been suggested (Carmichael 1965) that use of strong fields may tend to overemphasize high susceptibility materials and mask weaker substances which might be present.

In the present study there is no intention of measuring saturation magnetization (though at a later stage such determinations may prove useful). The present measurements are made on whole rock samples, not on separated ferromagnetic grains, and so saturation magnetization has less significance quantitatively. Thus very high fields are not essential.

As the measurements are made while heating in air, aerodynamic forces will play an important role, and due allowance must be made. This is done by measuring the dead weight of the sample at intervals, and from the difference between the readings for magnetic field on and off a measure of the magnetization is obtained. To do this, of course, the magnetic field must be nulled - this is possible more simply with an aircored coil system.

The Department possessed at the outset a Helmholtz coil system which had previously been used as part of a demagnetization apparatus on a spinner magnetometer (Hall, personal communication). The coil provides a reasonably uniform field over a volume of several cubic centimetres, up to about 400 oersteds. In the present equipment, this coil system provides the uniform magnetizing field. A second coil system has been designed and constructed to fit inside the Helmholtz system. This system consists of two identical solenoidal coils, mounted end to end on the same former.

The coils are in series electrical connection, with the current flowing such that the magnetic fields oppose each other. There exists along the axis of the coils a point X at which the total field strength is zero but the field gradient is a maximum, and reasonably uniform in the vicinity. The coils were designed to provide as large a gradient as possible, within the space available. The maximum gradient which can be produced is about 140 oersteds/cm., corresponding to a current of about 6 amperes. Much smaller gradients have normally been used in practice.

By placing the sample at the point X the magnetizing field and the gradient field may be adjusted independently simply by varying the respective currents.

A feature of this coil system is its flexibility. By interchanging connections to the coils the "Helmholtz" and "gradient" systems may exchange roles, the former providing now a small gradient, the latter a region of uniform solenoidal field. In this arrangement, fields up to about 1200 oersteds have been achieved for short periods of time.

A pulsing device is used to switch on and off the currents to both coil systems at intervals. This enables the dead weight to be recorded as well as the magnetic force, and also reduces the heat generated in the coils. The pulser provides a choice of "fields normally on" or "fields normally off", and the "mark/space" ratio of pulses is governed by a motor driven cam, which may be changed if required.

The power supplies for the coil systems are identical. Each consists of a variable transformer feeding a full-wave bridge rectifier followed by a 1,000 microfarad capacitor in parallel with the coils. The ripple voltage across the coils is of the order of 300 millivolts, which is acceptable for present purposes. Meter readouts of output voltages and currents are provided.

Sample suspension

The sample in a quartz bucket is suspended from the balance by means of the regular nichrome stirrup and two quartz fibres. The fibres have hooks at each end, one fibre extending from the bottom of the balance stirrup to a point a few centimetres above the coil units; the second is suspended from the first and carries the quartz bucket at its lower end. This arrangement provides for easy loading procedures.

The suspension is enclosed in a Vycor glass tube during operation, to shield it from air currents.

Furnace

The furnace is positioned inside the coil units, symmetrically about the sample position, and the Vycor tube fits inside the heating element. The 22 gauge alumel wire heating element is 21 feet in length, wound non-inductively, and is mounted inside an alundum former 6 inches in length. The element is set in fused alumina cement. On the outside of the alundum former is a 6 foot length of 36 gauge platinum wire, non-inductively wound; this is the temperature sensor for a furnace controller.

End plates of transite fit in the alundum and these also hold in position an outer casing consisting of a 2 inch I.D. copper pipe 6 inches in length, on the outside of which is soldered coiled 1/8 inch O.D. copper tubing for water cooling. Between the alundum and the copper pipe is calcined magnesium oxide.

Water cooling is used in order that the furnace may have low thermal inertia, and yet have a safe outer surface temperature. This latter is important since the coils surround the furnace. A low thermal inertia allows for rapid heating and cooling rates. The maximum permitted power dissipation is about 550 watts. the maximum temperature attainable in the furnace is 790°C.

Temperature measurement

Temperature measurement is by means of a platinum - platinum 13% rhodium thermocouple, which is embedded in a small piece of dolomite and positioned 0.5 cm. below the sample. The purpose of the dolomite is to provide a medium which will have thermal properties akin to those of the sample, so that the thermocouple will register a temperature which should be close to that which exists in the sample. The magnetic effect of the dolomite is considered to be negligible.

The thermocouple is supported by an alundum tube mounted in the base of the Vycor tube. The cold junction extends below the Vycor tube into a vacuum flask containing ice. Copper extension leads from the cold junction are taken to one channel of the strip chart recorder. The input circuit of the recorder has an 11 step attenuator which converts the range of the recorder to 10 millivolts for the thermocouple. Although the attenuator does not provide a truly potentiometric input at 10 mv., the error introduced in measuring the EMF is less than 0.01%, since the input resistance is of the order of 100,000 ohms.

Calibration procedures

Calibration of the thermocouple system was performed using the ice point and boiling point of water, and the fusion temperatures of certain inorganic salts, up to 770°C.

The calibration points were: ice point 0°C; boiling point of pure water 100°C; fusion of sodium hydroxide 318°C; fusion of potassium dichromate 392°C; fusion of lithium chloride 613°C; fusion of potassium chloride 770°C. (The temperatures were taken from the Handbook of Chemistry and Physics 1967).

The hot junction was placed in the salt and a cooling curve recorded on the chart recorder. This procedure was repeated at least three times. The plateau on the curve registered the fusion/freezing temperature.

Interpolation was performed using the above fixed points and the standard scale for platinum - platinum 13% rhodium thermocouples. (Handbook of Chemistry and Physics 1967).

The accuracy of calibration is limited by the accuracy of the recorder and the readability of the chart. The chart can be read to 0.01mv under optimum conditions, which represents approximately one degree.

The accuracy of the recorder is quoted as ±0.025mv. The internal calibration of the temperature channel on the recorder was checked using a standard reference EMF, and found to be well within this limit.

The spread of the several millivolt readings for each of the fixed points amounted to about ±0.01mv., giving an accuracy in degrees of about ±1. Temperature measurements are considered accurate to within ±3 degrees.

This is the temperature recorded by the thermocouple. Further tests were made to check the validity of the assumption that this is the temperature of the sample.

A nickel sample was used and the Curie point determined from the thermomagnetic curve. Within the accuracy of the measurement, the observed value agreed with the published value of 358°C (e.g. Bates 1963).

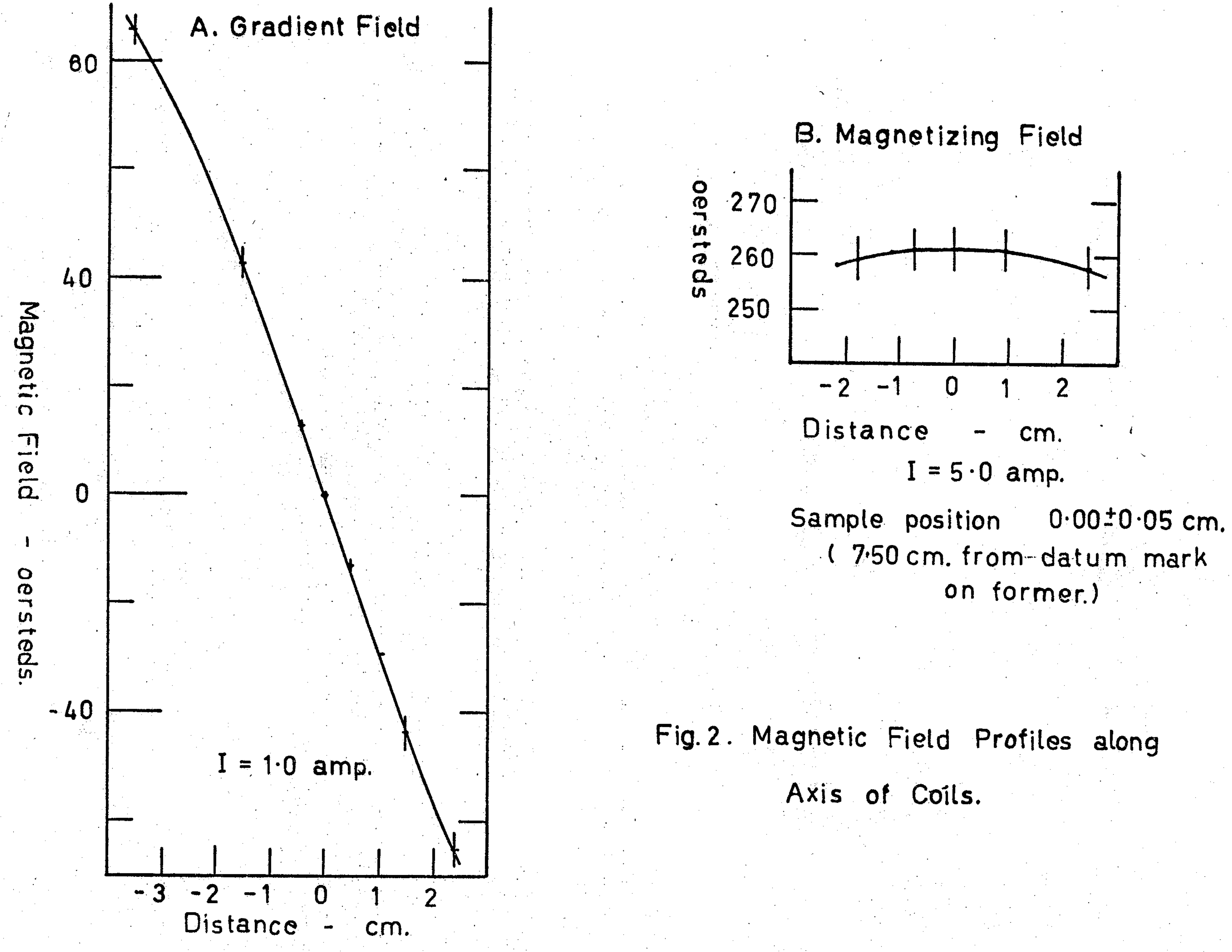
A further check was made using a sample of synthetic magnetite. A Curie temperature of 581°C was obtained on each of three runs with the same sample. This is in close agreement with the highest value quoted for natural magnetite, 583°C, by Chevallier et al (1954). Nagata (1961) gives 578°C for the Curie point for magnetite, and other values below 583°C are recorded in the literature. It is possible that the mineral samples investigated were not pure.

The magnetic fields were measured and calibrated against current values, using a Bell 110 Gaussmeter, with a Hall effect probe which was inserted into the coils, the Vycor tube and furnace being in place. Vertical profiles for both coil systems were obtained. A calibration of the magnetizing field at the sample position was made, and an approximate measure of the gradient in the region was taken. The absolute accuracy of the magnetizing field calibration is estimated at ±5%, but the repeatability of field settings is better than ±4 oersteds in the 200 oersted region.

Fig. 2 and 3 show the profiles and the field calibration.

The calibration procedures for the electrobalance and the chart recorder are standard and are described in detail in the respective manuals.

The literature provided with the electrobalance indicates that the mechanism is not affected by external magnetic fields. However, it has been found that a zero shift occurs on application of the magnetizing field. The differential force calibration of the balance is not affected. Since allowance has to be made for the effects of the magnetic field on the quartz suspension, the further correction of the zero shift presents no



I = 5·0 amp.

Sample position 0·00±0·05 cm.
(7·50 cm. from datum mark on former.)

Fig. 2. Magnetic Field Profiles along Axis of Coils.

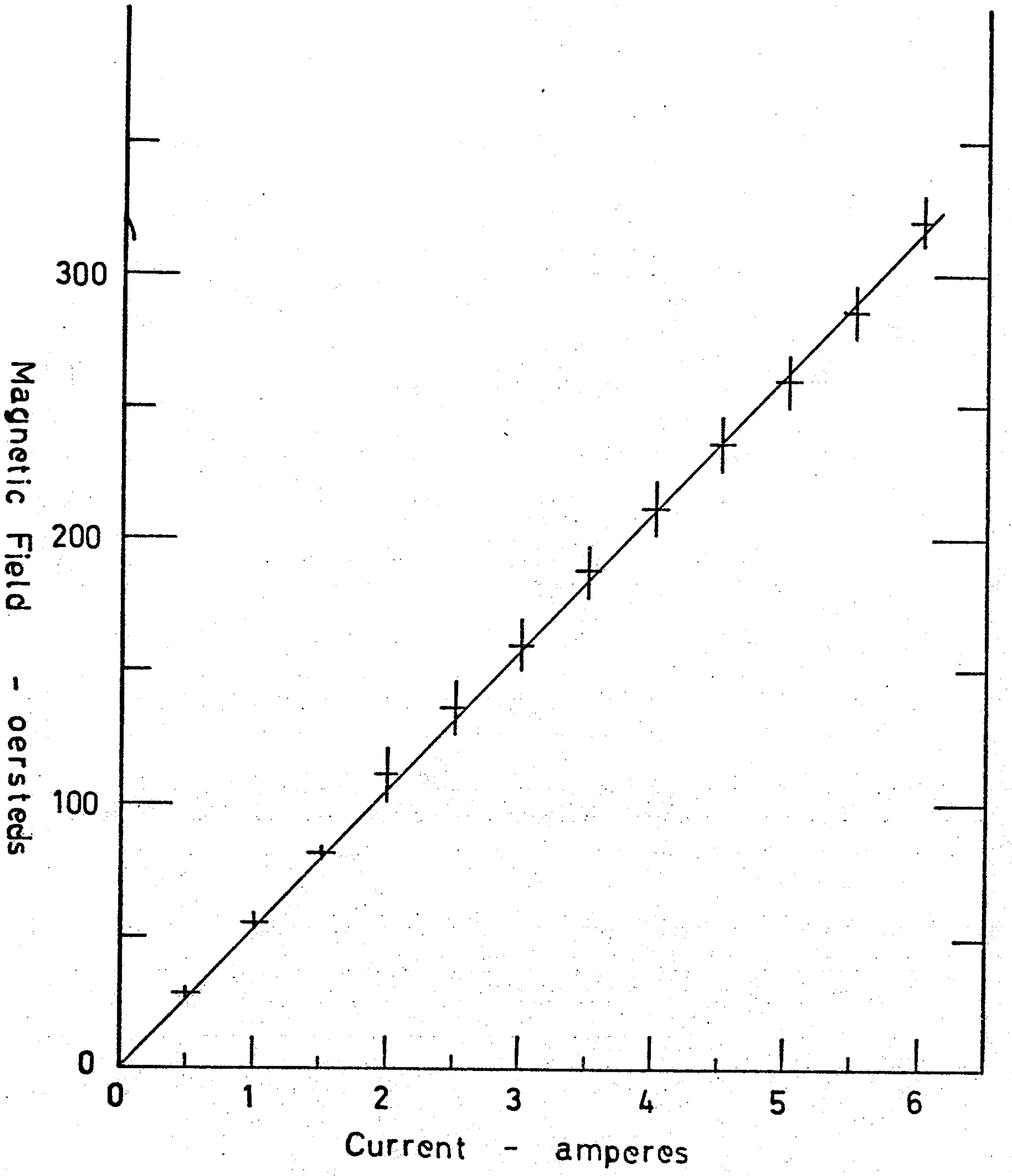


Fig. 3. Magnetizing Field Coils Calibration.

additional problem.

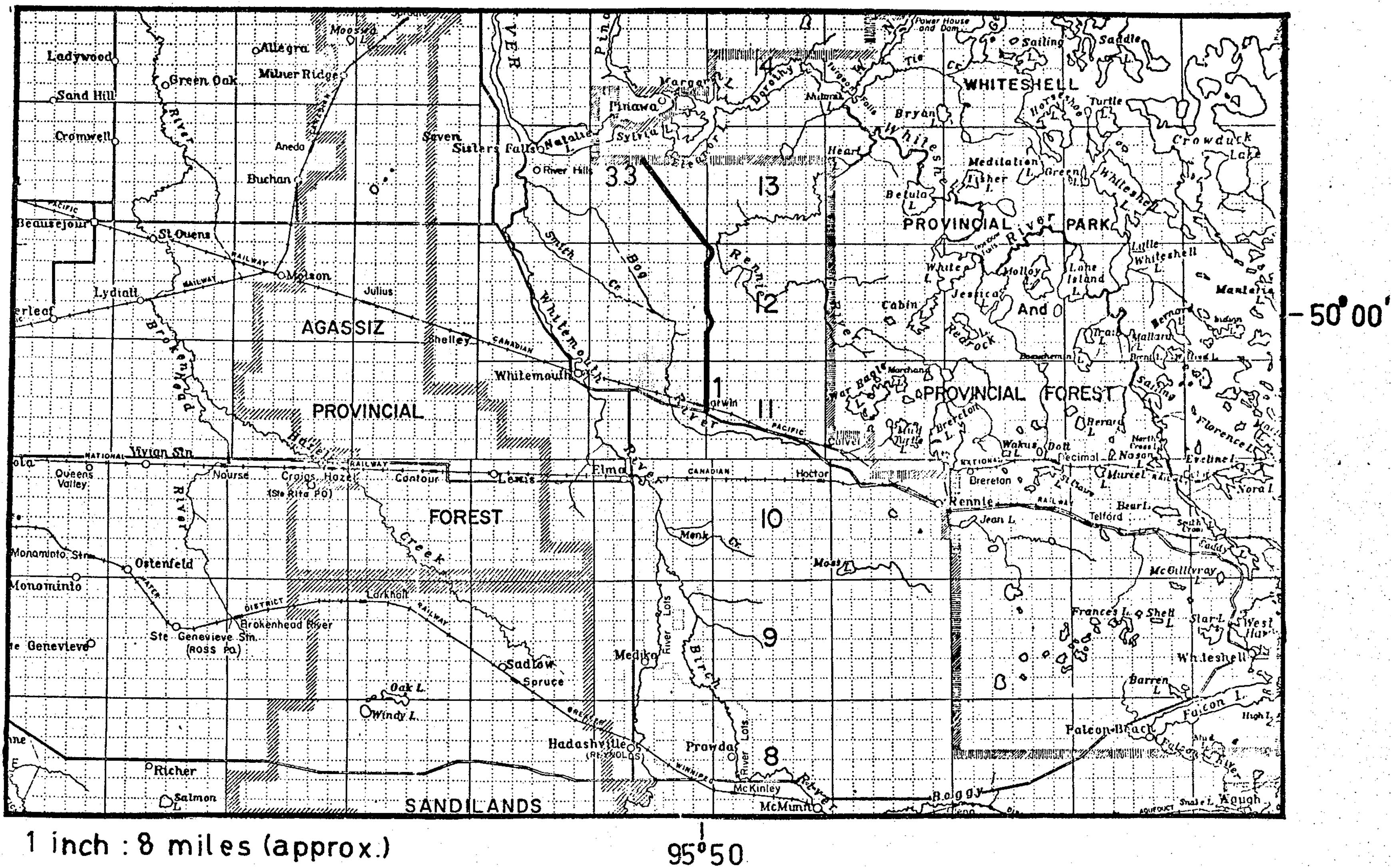
The furnace element is non-inductively wound, and carries alternating current, but checks were made for any magnetic force effects on the sample. No force was detectable under the conditions of operation.

CHAPTER V

GEOLOGY AND SAMPLING

The area presently under consideration is in a zone of regional anomalies of high relief in and around the Whiteshell Provincial Park, Manitoba. A traverse has been made over the flank of one of the anomalies, as a reconnaissance, preliminary to further more extensive surface investigations of the whole area. The actual location of the traverse is shown on the maps in figs. 4 and 5 . It follows a dirt road along the boundary between Ranges 12 and 13 northward from Provincial Highway 44, with two slight deviations, to intersect the boundary between Townships 12 and 13. From this point, the road, and traverse, trend N34°W to meet Provincial Road 307, 100 yards west of the Whiteshell Park Gate. The length of the road is 14.6 miles.

In this part of the Precambrian Shield, the relief is low, amounting to no more than 20 or 30 feet between hills and intervening valleys. Most of the low lying areas are filled with swamps, with an extensive swamp about one mile in width south of the boundary between Townships 12 and 13. South of this swamp, for a further 1.8 miles, no outcrop is visible close to the road. In the southern section of the traverse outcrops are quite frequent.



Traverse

Fig. 4 . Location Map.

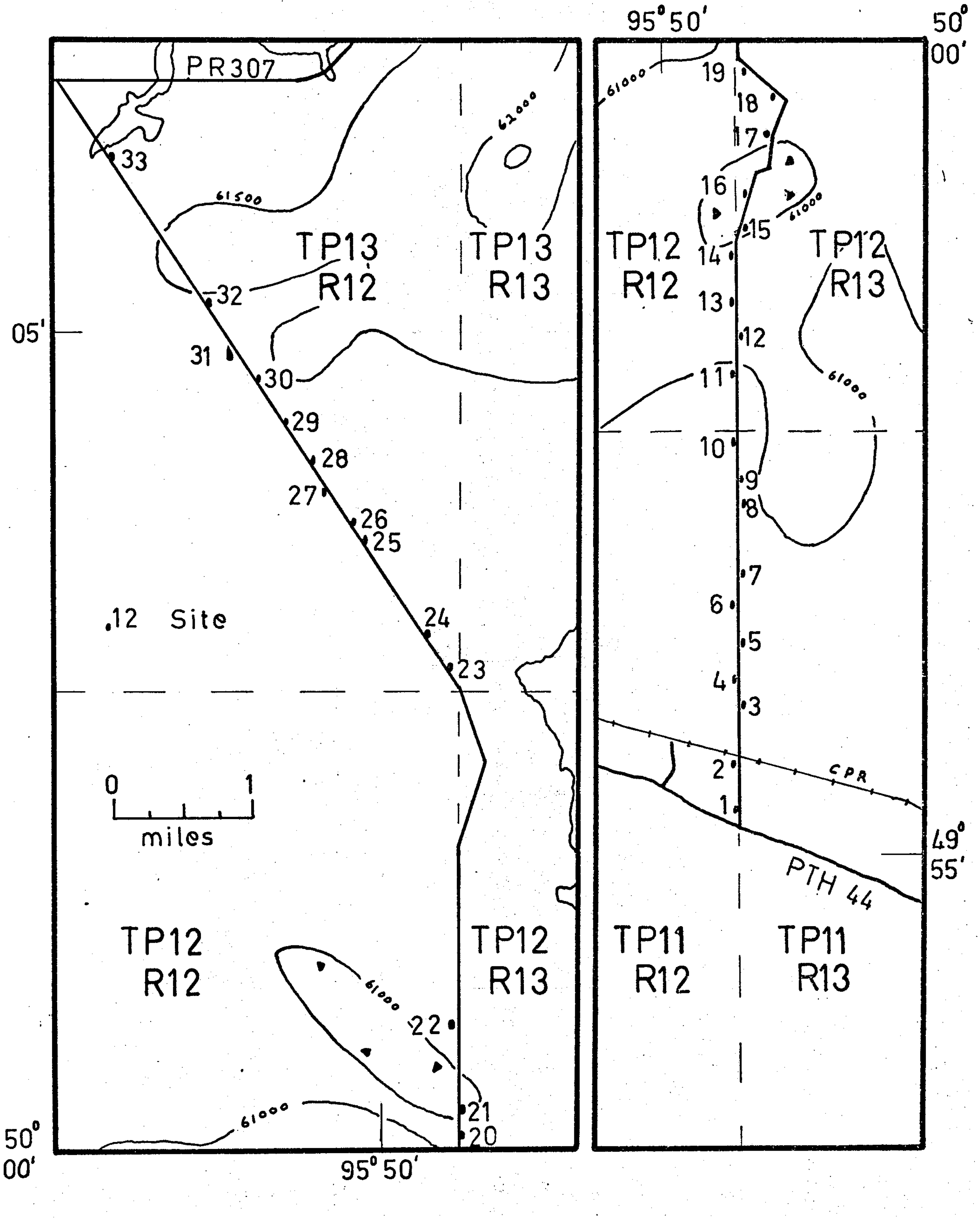


Fig. 5. Location Maps indicating Sampling Sites.

(From GSC Aeromagnetic Series Maps 4068G, 4069G 1966.)

This part of the Shield is a portion of the Superior geological province. No detailed geological reports are available on this localized area, although more detailed work has been done to the north and east. Wright (1938) in his summary states that the bedrock in the south eastern district of Manitoba is divisible into two major groups:
1) sedimentary and volcanic strata; 2) intrusives ranging from peridotite to granite. He adds that the areas of sedimentary and volcanic rocks are comparatively small, the most widespread rocks being granite and granite-gneiss.

The rocks presently under consideration are mainly granitic in character, with many crosscutting pegmatite dykes. In the more northern parts, more mafic intrusions and inclusions become common, and the structures of the rocks indicate much mixing of the rock types.

The textures range from medium grained to very coarse and pegmatitic. There are abundant quartz and potassium feldspar, with biotite and some plagioclase, the relative amounts varying from site to site. An important accessory is a strongly magnetic mineral, which is often visible with the unaided eye in drill cores and hand-specimens. In some areas the rocks are slightly foliated, but elsewhere appear massive.

Oriented drill core samples were taken at sites spaced approximately at ¼ to ½ mile intervals over a distance of about 14 miles, with the exception of a stretch of 2.8 miles, previously mentioned, and a section of about 1.2 miles near the northern end, where no outcrop was visible. The sites are indicated in fig. 5. At least two samples were taken at each site, chosen to be as representative of the outcrop as could be ascertained from a superficial examination prior to drilling. The drill cores were about 5 inches in length, on average.

CHAPTER VI

MEASUREMENT TECHNIQUES

Procedure

Preliminary susceptibility measurements were made on the cores, using a Geophysical Specialties MS_3 susceptibility bridge. Slices were then taken from the cores and placed in a vacuum desiccator at about 100°C for 3 or 4 days. This was considered advisable since early tests with non-desiccated samples showed appreciable weight decrease during the early stages of heating. There is also a possibility of erroneous temperature readings if any moisture is present.

Small chips were taken from the slices for use in the equipment described in Chapter IV. The mass of each test sample, usually 2 or 3 small chips, was within the range 200-300 milligrams.

The measurements were made with a field strength of 210 ± 10 oersteds, the field gradient being adjusted to a suitable value for each sample. The effective milligram range of the recorder was also adjusted for each sample.

It has been found that over several hours of continuous operation, the temperature rise in the magnetization field coils is excessive. To reduce the heat dissipation the pulser for the coils was arranged in the "field normally off" position, until about 100 to 50 degrees below a Curie point. The frequency is about one pulse per 100 seconds, each pulse lasting about 15 seconds.

As a Curie Point is approached, evidenced by a slight reduction in force, the pulser is switched to "field normally on" and this mode is retained until the temperature returns to well below the Curie point.

Thus, at lower temperatures, a dead weight curve is recorded, with the magnetic force measured at intervals, while at higher temperatures the magnetic force is continuously monitored, with the dead weight checked at intervals. With a less intensive series of measurements, the coils may be operated in the "fields normally on" mode at all times. Sufficient cooling will occur during breaks in operation.

The average rate of heating was of the order of 20 degrees per minute. The range of temperatures used for the present group of samples was from about 20°C to 610°C, with a few samples being heated to near 700°C. On cooling, the samples were removed at a little below 100°C, since further cooling time was considered excessive.

Preparation time for each sample was about 10 minutes, and each run to 610°C and back occupied approximately 65-70 minutes.

The furnace was controlled manually to provide an approximately linear temperature increase. Manual control entailed adjusting a variable transformer at about 5 minute intervals. Checks on coil currents were also made; slight adjustments were found necessary only at very infrequent intervals.

When sensitive milligram ranges had to be used noticeable changes in apparent dead weight were found. These were due to aerodynamic forces caused by convection currents occurring in the Vycor tube, and also due to actual changes in weight of the sample through oxidation and/or vapour loss.

In many cases, these changes caused the need for repositioning of the trace on the chart. This could be done readily during a run by adjusting the calibrated Mass Dial on the balance control unit.

Frequent checks were made of the temperature channel zero and also of the balance calibration. It was also found advisable to measure the force on the suspension separately for each sample, as a precaution against changes which might otherwise go undetedted. The force on the suspension is subtracted from the total force recorded to give the force on the sample.

From the chart produced by the recorder, temperature and force data were extracted, and plots of "magnetic force ratio" against temperature were made. ("Magnetic force ratio" equals force at t°C/force at 20°C). In some cases slight extrapolation back to 20°C was necessary.

Comments on techniques

A number of investigators have had misgivings concerning the heating of samples in air, during thermomagnetic analyses. It has been suggested that in some cases, at least, the results may not truly represent the unheated material, especially where slow heating was used.

Ade-Hall et al. (1965) have considered the problem of chemical change during heating, and conclude that no significant changes occur if the heating is sufficiently rapid. They suggest less than 20 minutes for heating to 650°C. This is possible in the present equipment, but the reliability of temperature determination decreases somewhat. A 30 minute heating time is found to give adequate temperature accuracy.

Second chips from several samples, whose Curie Points had previously been determined at this latter rate, were subjected to a determination at a faster initial heating rate, slowing near the Curie Point to allow more accurate temperature measurement. The time taken to reach 630°C was about 13 minutes. No significant differences in Curie Point were found on comparing the two determinations on each sample.

This helps to justify the presently used technique, for the present series of samples, but does not guarantee the technique for other series, particularly rocks containing titanium-rich magnetites. The work of Ozima and Larson (1970) suggests that changes may occur with titanium-rich titanomagnetites; however in their discussions, the rates of heating were not disclosed. Further tests would be advisable in these cases. Wasilewski (1969) has also considered alterations during thermomagnetic analyses and presents a number of factors which should be noted when evaluating thermomagnetic data.

Lowrie and Fuller (1969) discuss some effects of annealing at medium and high temperatures on magnetites. Their results indicate a net decrease in Curie temperature with annealing. Although not stated explicitly, their results suggest that the effect may become significant at about 500°C. Their high temperature anneals each lasted one hour, during which time, from their results, slight reductions in Curie temperatures occured. In the present measurements, the samples were above 500°C for from 15 minutes to 30 minutes approximately, depending on the maximum temperature used. If the effects described by Lowrie and Fuller occurred with the present samples, then slight differences in Curie points as shown by heating and cooling curves might be detectable. If slower heating and cooling rates were used. and higher maximum temperatures, it would be expected that the effects would be more prominent.

A synthetic magnetite sample was subjected to a thermomagnetic analysis, firstly to obtain a value for the Curie temperature of magnetite, and secondly to investigate any changes which might occur during the heating in air. The sample was heated to 700°C, cooled, twice reheated to 620°C and cooled, reheated to 670°C and finally cooled. The total time spent over 450°C was about 60 minutes. No change of Curie temperature was found, although evidence of inversion of a small amount of maghemite was found on the initial heating cycle, and slight reddening of the powder was observed at the end. Thus it may be expected that very slight changes might occur to near pure magnetite phases, but that the Curie temperature would be unaltered.

CHAPTER VII

INTERPRETATION

As the samples were rock chips, not separated mineral grains, the thermomagnetic curves apply to the whole mineral assemblage in the rock, not only to the magnetic minerals. This should be borne in mind in discussions of the general curves.

The Curie transitions are unaffected by the presence of the non-ferrimagnetic grains, but the net force on the sample at any temperature is a result of the combined effects of diamagnetism, paramagnetism and ferrimagnetism in the mineral aggregate. In some of the samples tested, where ferrimagnetic grains were few, the paramagnetic or diamagnetic effects were prominent.

Abrupt changes of slope in the curves may be interpreted as evidence of Curie transitions (Chevallier et al. 1954). Figure 6 indicates the types of thermomagnetic curves to be expected in an analysis of this kind. Another possible interpretation of fairly sharp changes in slope is that phase changes or chemical alterations may have occurred at some characteristic temperature. An example is the inversion of maghemite to hematite.

From the thermomagnetic curves, the possible Curie transition temperatures were estimated, using the empirical definition discussed in Chapter II, whereby the

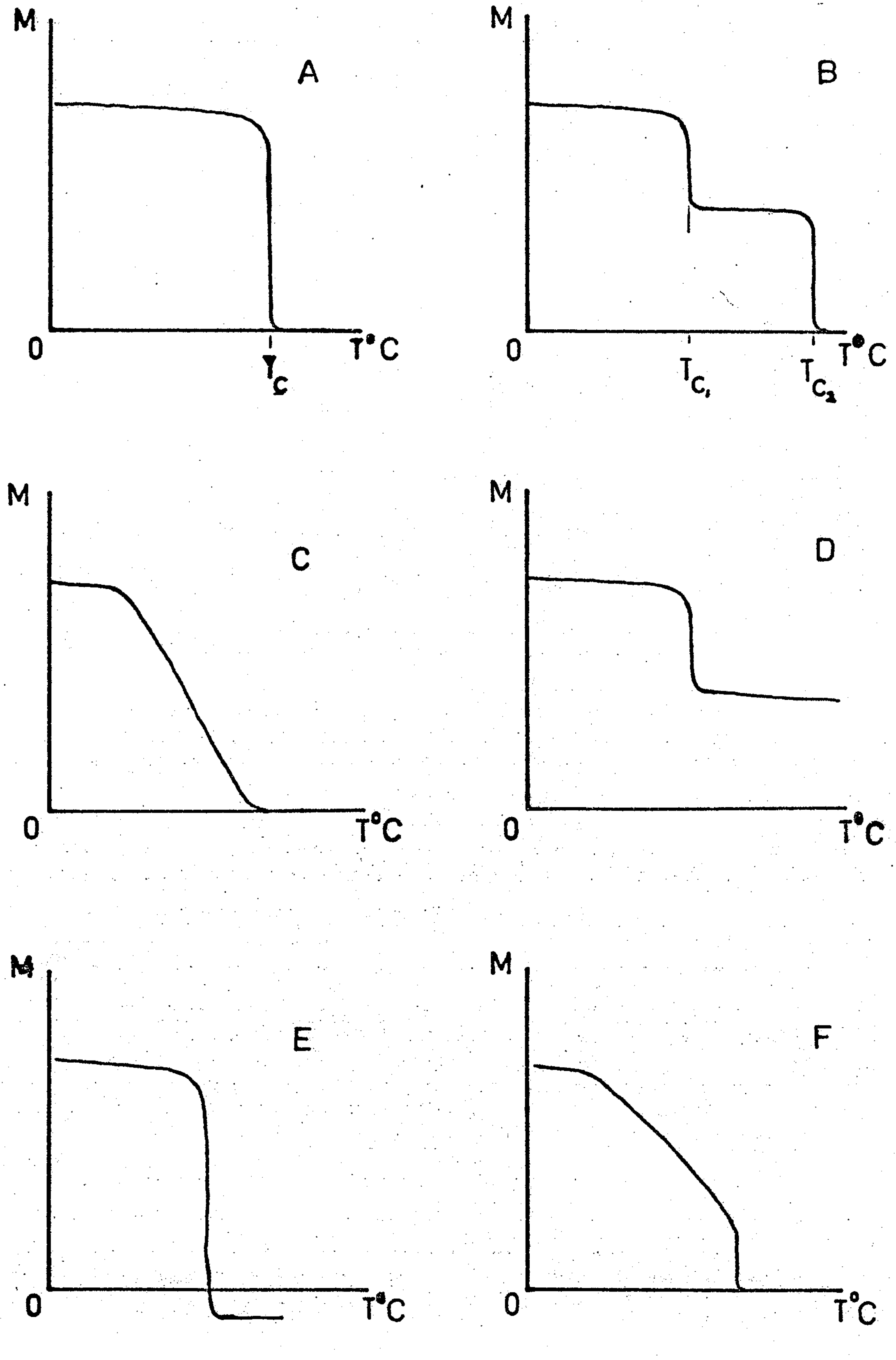


Fig. 6. Thermomagnetic Curves. See p.48 for captions.

Fig. 6. Thermomagnetic Curves.

A. Single Curie point; homogeneous magnetic phase.

B. Two Curie points; two magnetic phases.

C. Range of Curie points; solid solutions of continuously varying composition.

D. Single Curie point; homogeneous magnetic phase in paramagnetic matrix.

E. Single Curie point; homogeneous magnetic phase in diamagnetic matrix.

F. Range of Curie points, with highest one predominant; solid solution of continuously varying composition, with larger concentration of high Curie point end-member.

point of inflexion is used to represent the Curie point. The Curie temperatures of the samples and average susceptibilities of the cores are listed in Table 1. It can be seen from the table that the temperatures are close to one another, and to the Curie temperature of pure magnetite (583°).

Figure 7 shows the Curie temperatures measured for the samples. With three exceptions, it is seen that for a given site the Curie temperatures are within 3 degrees of each other. The accuracy of the determinations cannot be considered to be better than ±4 degrees. Although it appears from the plot that there might be a slight decrease in Curie temperature towards the south, the amount is less than the estimated errors and should not be considered significant in the absence of more precise checks and determinations, and more extensive sampling.

Any systematic trend which might have occurred during the period of measurements was eliminated by taking samples in a random sequence from storage.

In view of the Curie temperatures reported in Table 1, it would be reasonable to conclude that the ferrimagnetic phases present were largely magnetite in composition, but with some small amounts of other material in solid solution. The most likely materials would be ulvospinel Fe_2TiO_4 or ilmenite $FeTiO_3$ (Nagata 1966). The molecular amounts of

TABLE 1

SAMPLE DATA

Sample	Curie Temperature °C	Average Susceptibility 10^{-6} cgsu.
1A	570	1300
1B	569	1220
2A	570	2140
2B	572	900
2C	-	157
2D	571	1050
3A	570	1000
3B	-	-(low)
3C	570	1500
4A	570	740
4B	572	530
4C	-	25
5A	-	20
5B	-	150
5C	570	1200
5D	568	1410
5E	569	1720
6A	569	1080
6B	565	1470
6C	570	910
7A	569	1450
7B	571	1230
8A	573	3030
8B	573	2540

TABLE 1. (continued)

Sample	Curie Temperature °C	Average Susceptibility 10^{-6} cgsu.
9A	574	1170
9B	575	600
10A	572	1290
10B	573	1130
11A	573	1040
11B	570	960
12A	573	(900)
12B	574	1680
12C	-	450
13A	576	4670
13B	570	670
14A	574	1200
14B	577	(680)
15A	575	1800
15B	573	6530
16A	573	1420
16B	575	1050
17A	571	800
17B	573	695
18A	572	1880
18B	575	2700
19A	571	706
19B	572	4500
20A	573	870
20B	572	1510
21A	575	1270

TABLE 1. (continued)

Sample	Curie Temperature °C	Average Susceptibility 10^{-6} cgsu.
21B	-	56
21C	571	710
22A	574	1590
22B	573	1440
23A	576	1340
23B	577	1300
24A	572	1060
24B	575	1000
25A	572	3350
25B	573	3100
26A	574	885
26B	575	1460
27A	573	1340
27B	571	2100
28A	574	680
28B	576	980
29A	574	980
29B	572	856
30A	574	1560
30B	572	1560
31A	571	3830
31B	573	1990
32A	575	2060
32B	575 +(100 → 620)	90
33A	574	1260
33B	573	4070

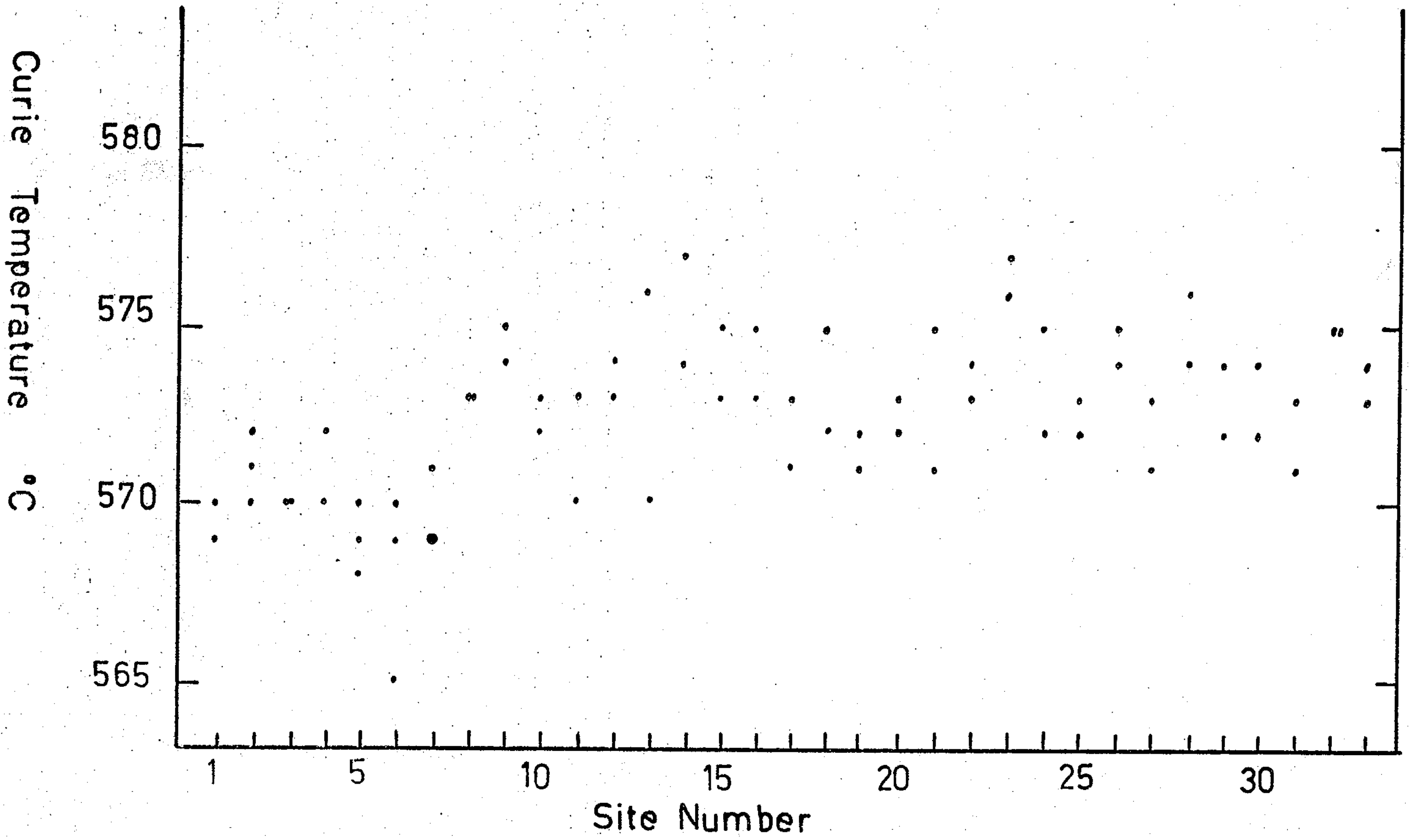


Fig 7. Curie Temperatures vs. Site Numbers.

these titanium minerals would be less than 4%, following the work of Akimoto (1957), Chevallier et al (1954).

The proposition that little titanium is present in solid solution does not eliminate the possibility of its presence in exsolution structures within the titanomagnetite grains, nor does it discount the existence of separate ilmenite grains within the host rock.

A further possibility, which cannot be discounted on the basis of the present measurements, is that the ferrimagnetic phase might be titanium-rich maghemite (Larson et al. 1969). According to these workers, titanium-rich maghemites decompose at temperatures higher than those reached in the present heating experiments. They found that the saturation magnetization curves were reproducible on cooling with Curie temperatures in the 500°C - 600°C region. Nagata (1966) discusses titanomagentites and titanomaghemites, and gives their variations in Curie temperature, saturation moment and lattice constant with composition. Thus further measurements and observations are necessary to ascertain the composition of the ferrimagnetic phase in these samples.

A few of the thermomagnetic curves show a slight gradual decrease at temperature below 200°C. However, this is at the limit of resolution of the system and cannot be considered as representing another magnetic phase.

One thermomagnetic curve is of particular interest, fig. 14. The sample, 32B, was taken from an outcrop where numerous mafic inclusions were present. The sample is from one of these inclusions. The curve shows a fairly sharp Curie point at 575°C, but in addition, there is an almost linear decrease in magnetization from a little above room temperature. The curve suggests the existence of a magnetic phase with Curie temperature near 150°C, and in addition phases with a range of compositions, and therefore Curie points, up to about 620°C. However, the initial susceptibility of the sample is less than 10^{-4}cgsu., and the quantities of magnetic minerals present are very small.

The general shapes of the curves are in fairly close agreement with those of the synthetic magnetite samples, figures 8 - 15. Deviations are most probably due to differences in grain sizes; small amounts of other magnetic phases of continuously varying compositions may also cause slight deviations. The differences between the "horizontal" parts of the curves in general may be attributed to thermoremanence effects.

In most cases, the Curie temperatures on the heating and cooling curves agreed, within the accuracy of measurement. In two cases, samples 15A and 23B, the difference was too great to be accounted for in this way. It is probable that in these cases some slight alteration took place. For sample 15A the decrease was about 9 degrees. For sample

23B the decrease was about 5 degrees, and the maximum temperature for both was 700°C. Other samples, 3A, 31B, 32B and 33B, were heated to near 700°C. With the exception of 32B, which is a special case, no significant changes occurred. For 32B, the measurements suggest a slight change in the shape of the "tail" above 575°C.

From the above observations, one may deduce that brief heating to 700°C may initiate significant changes in the Curie temperature, and therefore composition, of the magnetic phases. Further work is needed to verify this. The effect did not occur with all samples heated to 700°C and this may indicate differences in the natures of the minerals present. Furthermore, as a decrease in Curie temperature was observed, it is unlikely that oxidation due to heating in air was the cause, since oxidation results in an increase in Curie temperature (Nagata 1966). This may be an instance of the effects noted by Lowrie and Fuller (1969), see Chapter VI.

The average initial susceptibilities of the cores are listed in Table 1. There is a wide range of values, from 20×10^{-6} to 6530×10^{-6} cgsu. No distinct pattern of values along the traverse has evolved. In fact, there is often a wide range of susceptibilities at a given site. The values quoted in Table 1 are simple averages of the two susceptibilities measured by inserting each end of the core into the sample holder of the bridge in turn. In some cases, the two readings differed widely. It is evident that the distribution of the ferrimagnetic minerals is not uniform, and may be banded. Closer investigations of the rocks and the minerals are necessary before any definite conclusions may be reached.

Figures 8 - 15. Examples of Thermomagnetic Curves.

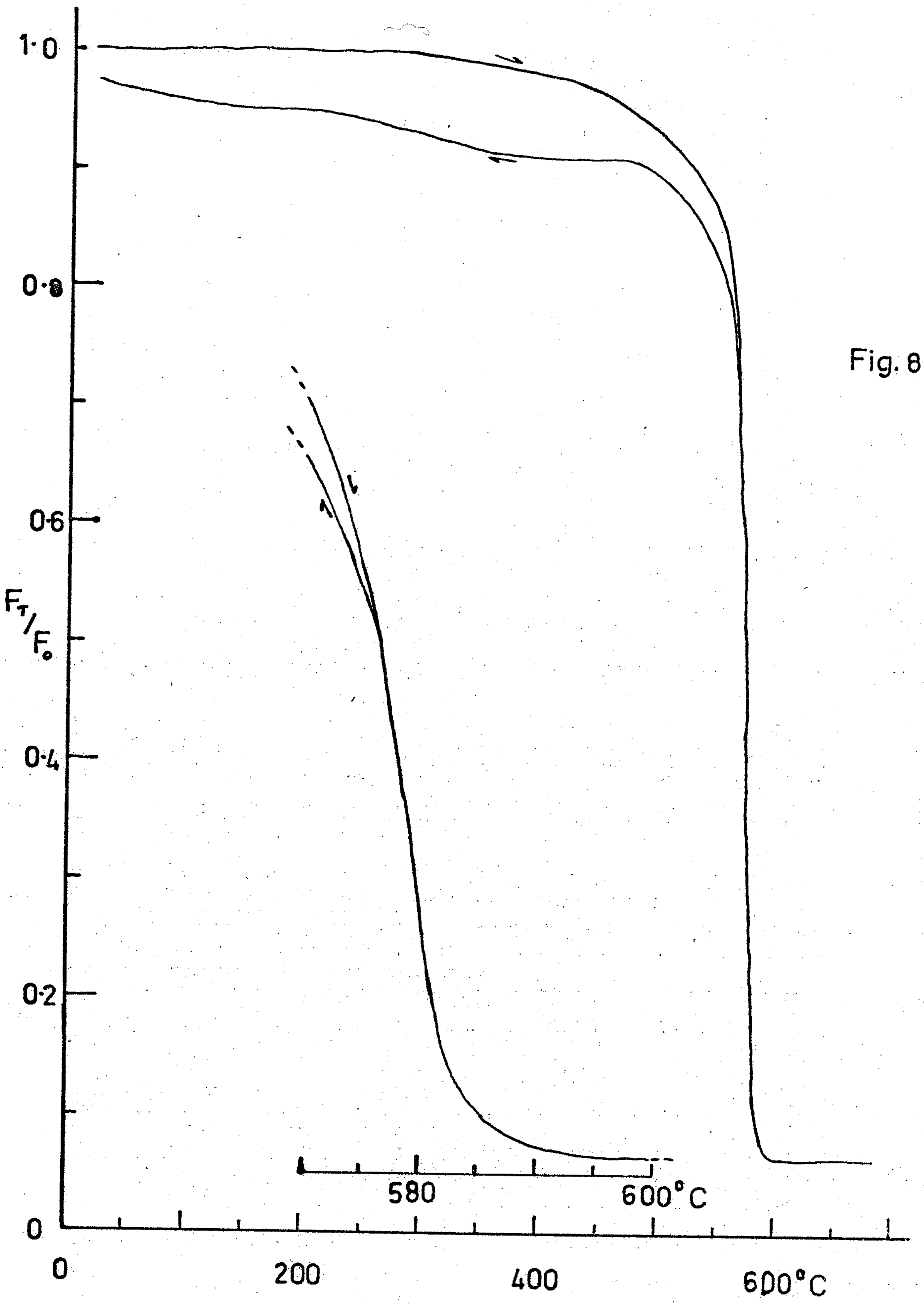


Fig. 8

Synthetic Magnetite T_c 581 ± 4° C

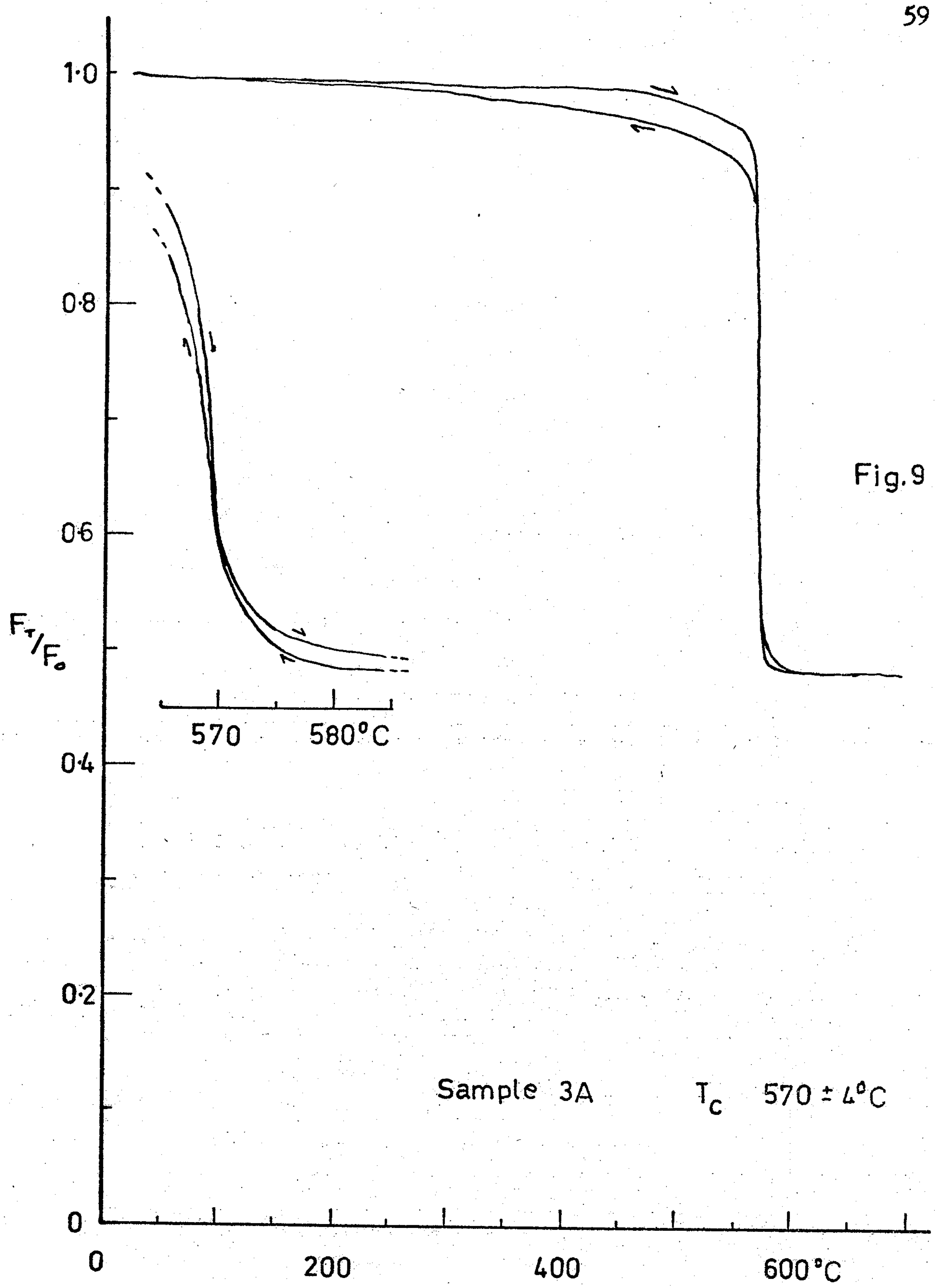


Fig. 9

Sample 3A T_c 570 ± 4°C

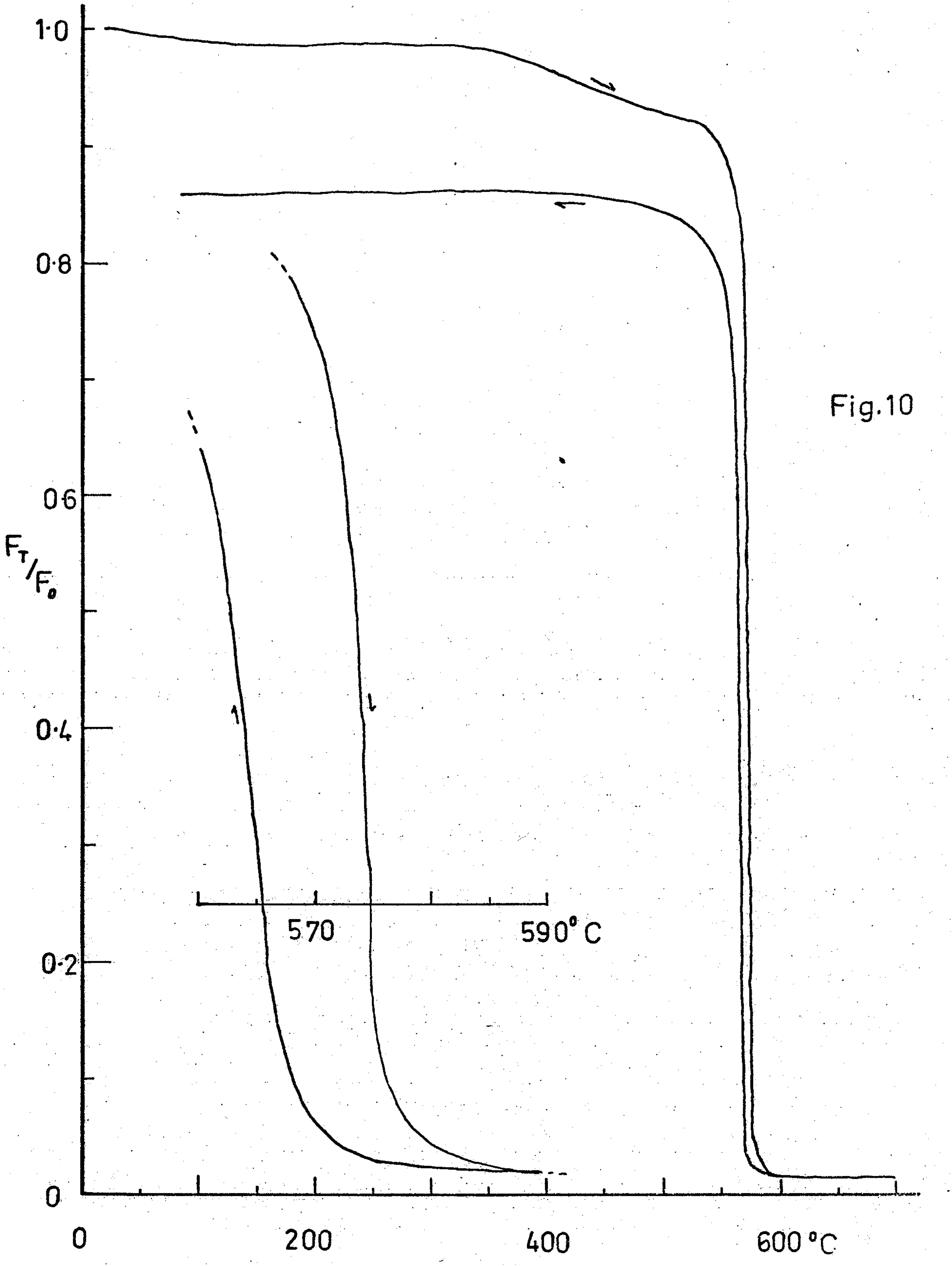


Fig.10

Sample 15A T_c (heating) 575 ± 4°C

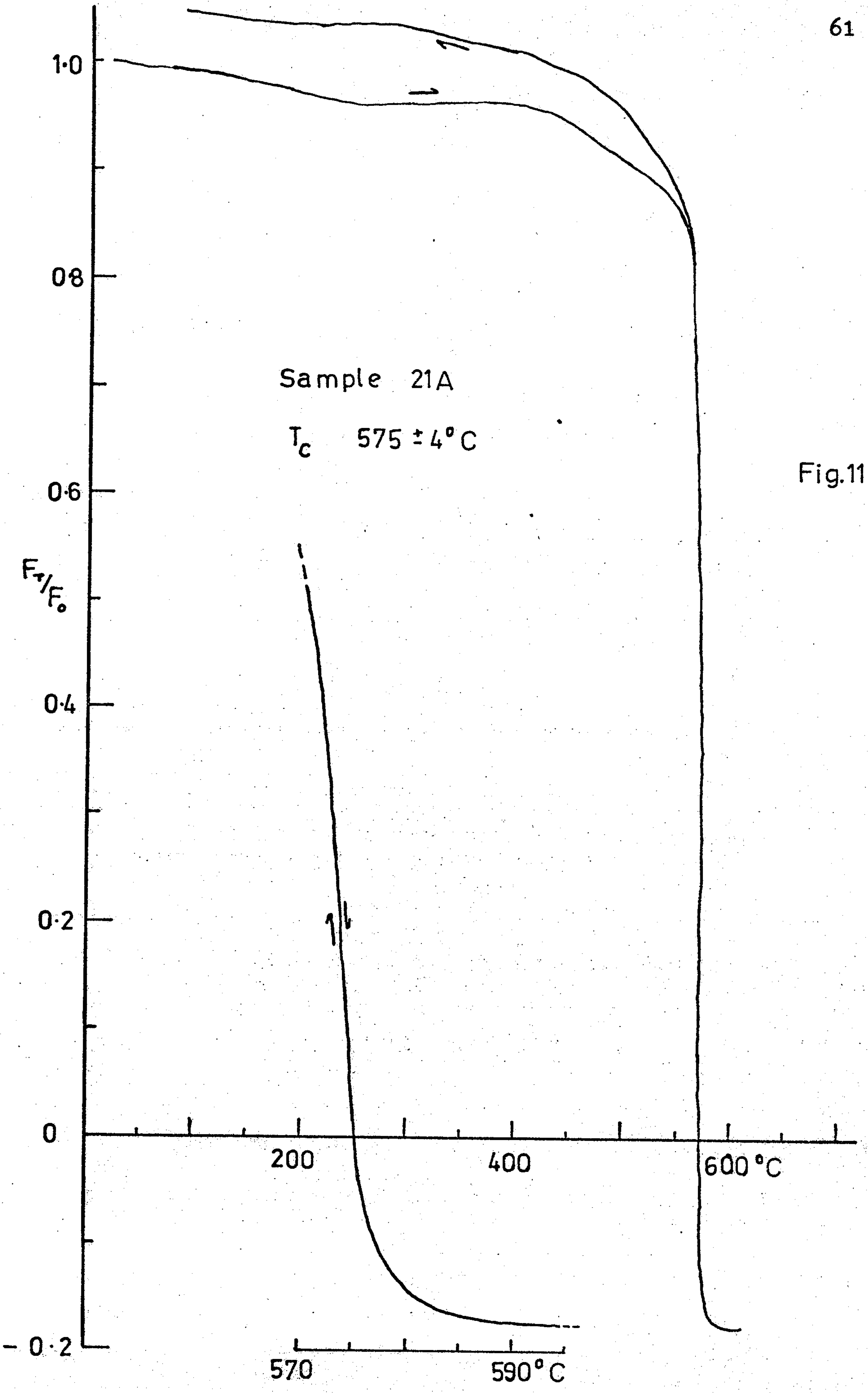

1·0
0·8
0·6
0·4
0·2
0
- 0·2
F_T/F_0
Sample 21A
T_c 575 ± 4°C
200
400
600 °C
570
590°C

Fig.11

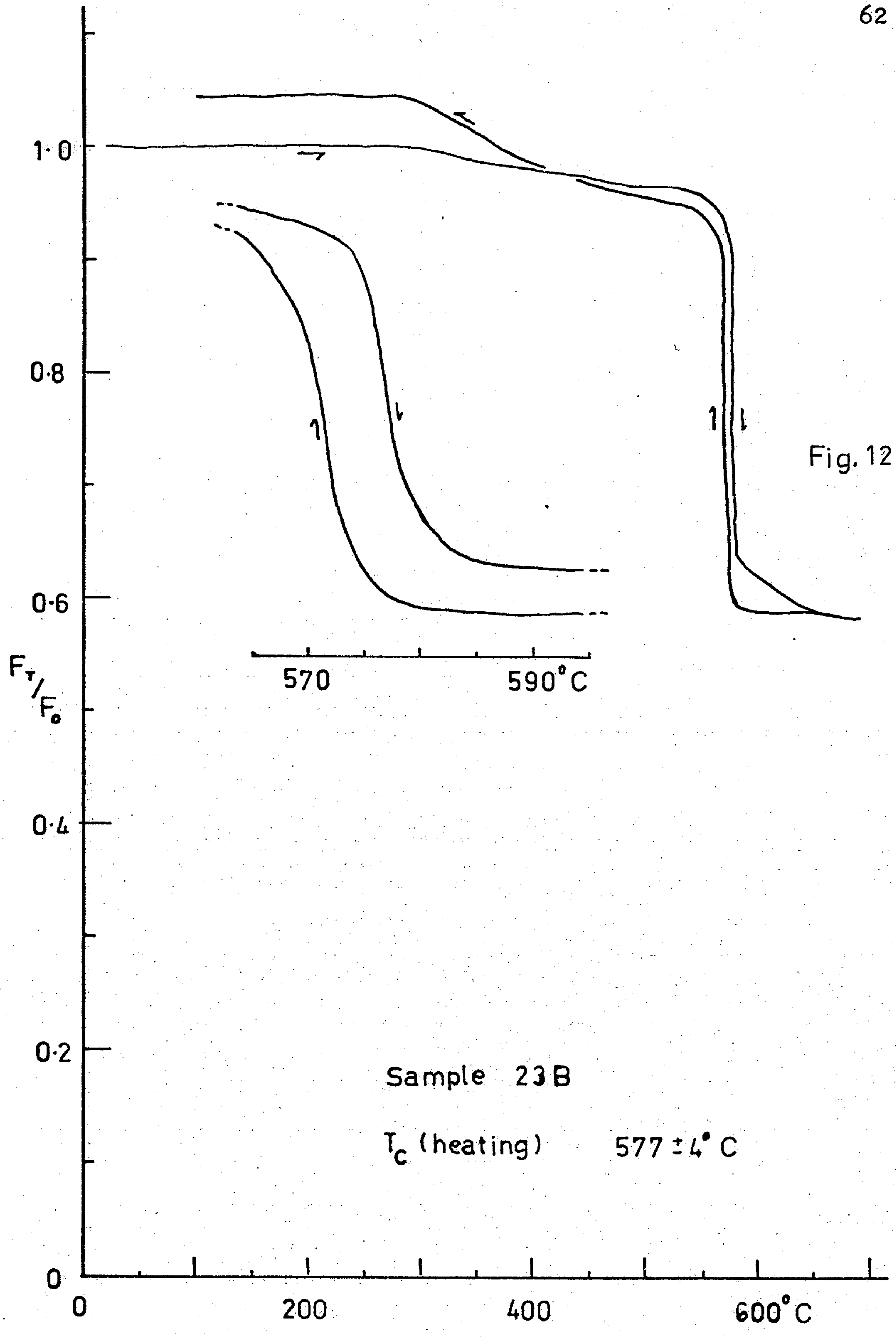


Fig. 12

Sample 23B

T_c (heating) 577 ± 4° C

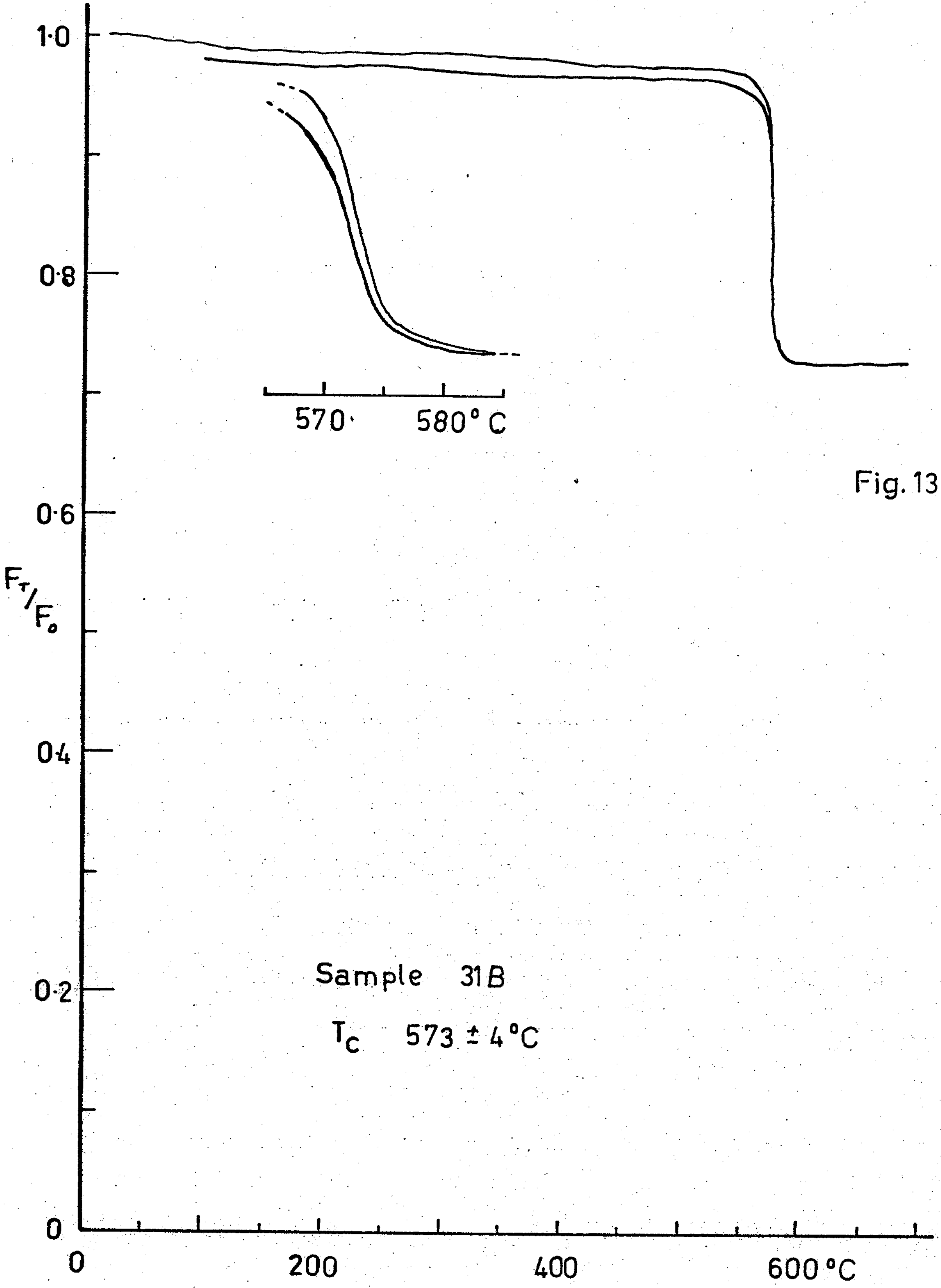


Fig. 13

Sample 31B

T_C 573 ± 4°C

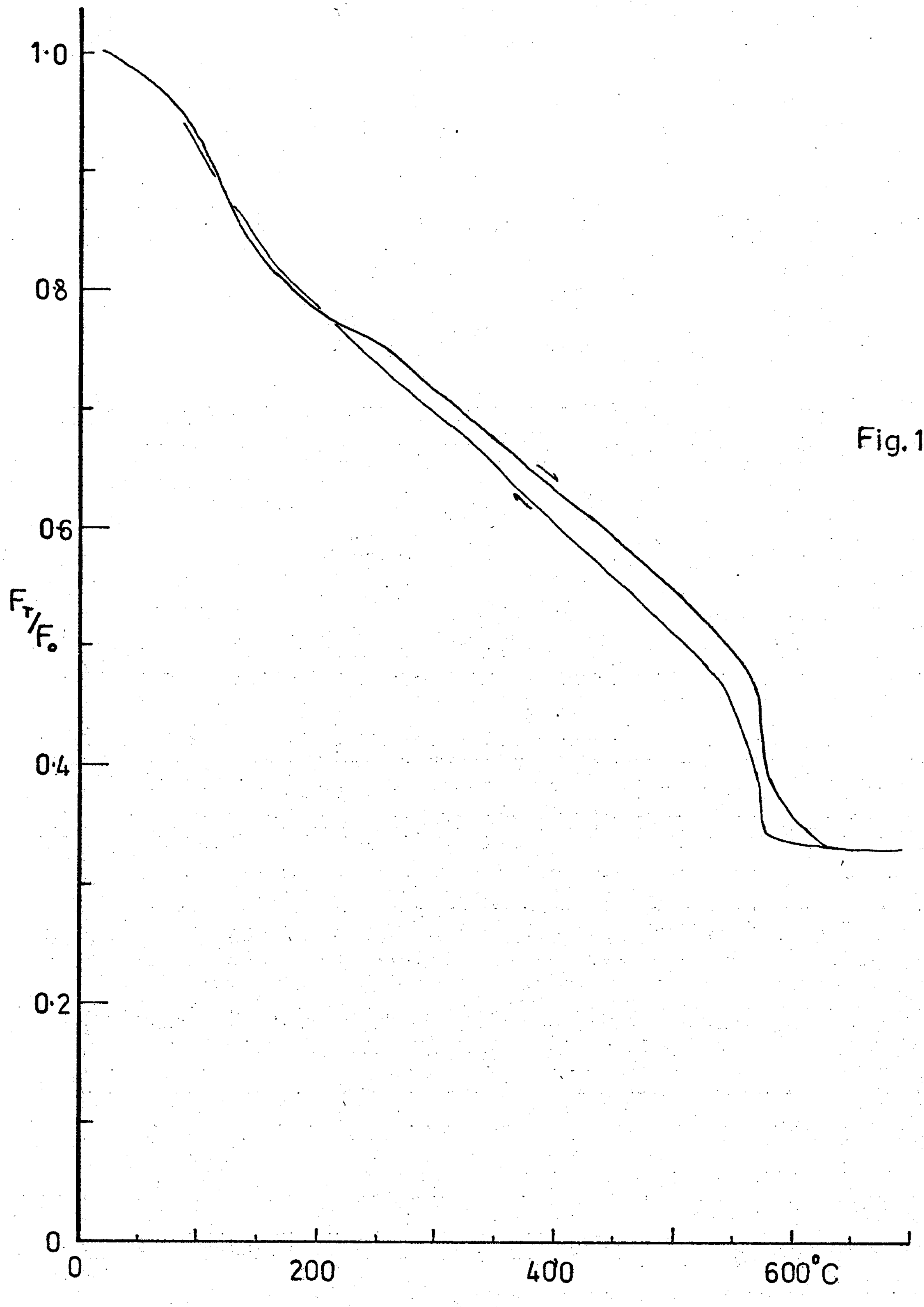


Fig. 14

Sample 32B T_c 575°C + Range

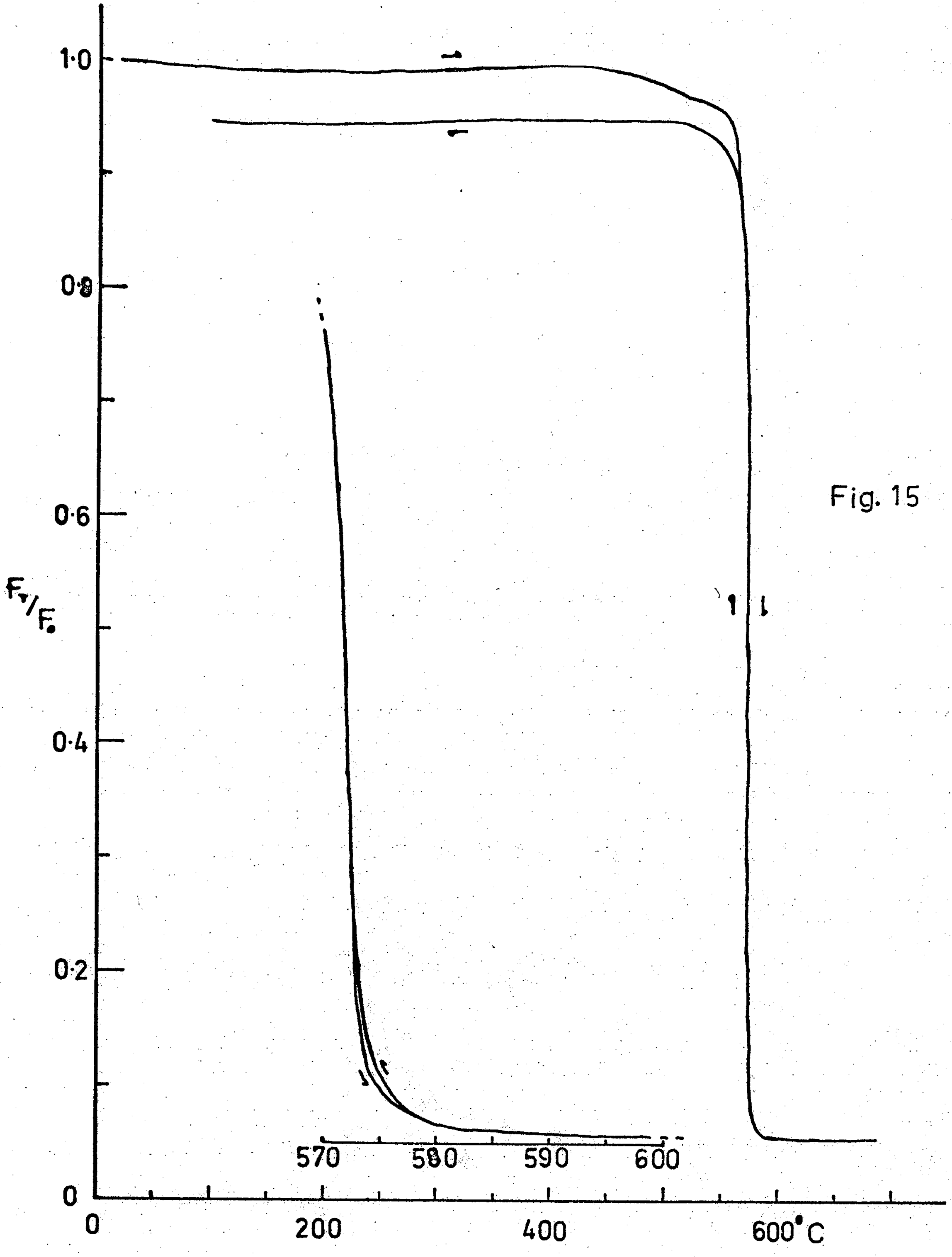


Fig. 15

Sample 33 B T_c 573 ± 4° C

CHAPTER VIII

COMMENTS AND CONCLUSIONS

Modifications to equipment

The equipment has been adequate for the present purpose. However, a number of modifications and additions would render it capable of other measurements, and would increase its versatility.

A vacuum, or controlled atmosphere system would reduce any oxidation effects which might occur in some samples (although reduction processes might still be significant). A vacuum arrangement would be the better of the two, as it would also eliminate aerodynamic effects, which do present a minor problem in the equipment.

An alternative, considerably less expensive method would be to enclose the sample in an evacuated small quartz ampoule. However, an objection is that gases may be evolved during heating, with possible explosion of the ampoule. The possible use of this method in the present equipment should be investigated.

For the present work an air-cored coil system was used. However, if at a later stage, magnetic saturation of samples is required, for saturation magnetization or hysteresis measurements, an iron-cored electromagnet would be required.

A furnace controller will be available in the near future, which will free the operator from frequent adjustments to the furnace supply. The furnace has been found to be slightly slower in cooling than anticipated. Experimentation with smaller amounts of calcined magnesia insulation may be worthwhile, if a shorter cooling time can be obtained without an excessive rise in coolant temperature.

For very sensitive work, attention will have to be given to the support framework, and in fact to the location of the equipment, since building vibrations cause a noisy trace on occasions.

Future investigations

The Curie temperatures determined for the samples, as discussed in Chapter VII, would indicate that the ferrimagnetic phase might be almost 100% magnetite, with little titanium in solid solution.

Microscopic investigations of opaque minerals may well indicate the presence of exsolved titanium minerals, ilmenite and/or ulvospinel. Chemical analyses and X-ray diffraction tests may be used to verify the presence or absence of maghemites and to obtain a measure of the titanium content (and other elements, if present).

It may be possible to correlate the above analyses with geophysical measurements. Heating experiments have been performed by Vincent et al.(1957) which suggest the possibility of a method for estimating titanium content. Furthermore, heating experiments may provide additional information on the possible nature of ferrimagnetic minerals at depths below the surface of the earth.

Measurements of natural remanence and the subsequent determination of paleomagnetic pole positions will serve to indicate the long-term magnetic stability of the surface rocks. Demagnetization procedures will be necessary during this work, in order to remove any secondary magnetizations.

Measurements of thermal decay of NRM and TRM may help to determine the temperature or temperatures to which the rocks have been subjected in the past.

It has yet to be established whether the regional anomalies are due to remanent or induced magnetization. If the latter is so, then Curie point determinations are most relevant to the problem. If, however, remanence is found to be the main contributing factor, then a study of blocking temperatures may be more significant, since these govern the thermal dependence of remanent magnetization. (Nagata, 1961, Irving 1964, 1970).

More extensive sampling of surface rocks should be undertaken over larger areas over the regional magnetic anomalies in the Canadian Shield.

At this point, it should be added that integrated studies of heat flow and of the radioactivity of the rocks in this region of the Canadian Shield would be most relevant to the problems of the Curie isotherm, of the magnetic anomalies, and of crustal structure in general.

Conclusions

1. It has been shown that a commercial electrobalance, in conjunction with an air-cored coil system, can be used to determine sub-saturation thermomagnetic curves, and Curie temperatures, with a minimum of ancillary equipment and in a mechanically noisy environment.

2. Curie temperatures have been determined for a series of 70 samples, and these indicate a mean Curie temperature of 573°C for the rock unit sampled.

3. The ferrimagnetic phase present in these rocks is most probably a magnetite, with little titanium in solid solution, but may be a titanomaghemite.

4. If this rock unit continues to depths below 20 km. without changes in the nature of the ferrimagnetic phase, it is improbable that the Curie level would occur at 20 km.

5. Rapid thermomagnetic analyses in air to 620°C for high Curie point minerals have caused no significant changes in Curie points.

6. Brief heating to 700°C may produce alterations in composition, which are not oxidation processes, of the ferrimagnetic phases.

APPENDIX 1

COIL DESIGN

The magnetizing field coils were constructed prior to this project. They are a modified Helmholtz system and provide a reasonably uniform field of about 400 oersteds over a volume of about 10 cc.

The gradient field coils were designed specifically for this equipment. For a solenoid, the maximum gradient occurs at each end of the winding and if two identical solenoids are placed end to end, with magnetic fields in opposition, the point on the axis at their adjacent ends will have zero magnetic intensity and maximum gradient.

The equation for the field intensity at a point on the axis of a solenoid follows from a standard derivation (e.g. Cheston 1964).

For a ring of current, the field dH_z at a point P on the axis is given by

$$dH_z = \int_c \frac{I\,dl\cos\theta}{\rho^2}$$

(Symbols as in fig.16)

By integrating over the whole volume of the coil, one obtains the field H_z

$$H_z = \frac{2\pi I mn}{10}\left[(z+c)\log_e \frac{b+\sqrt{b^2+(z+c)^2}}{a+\sqrt{a^2+(z+c)^2}} - z\log_e \frac{b+\sqrt{b^2+z^2}}{a+\sqrt{a^2+z^2}}\right]$$

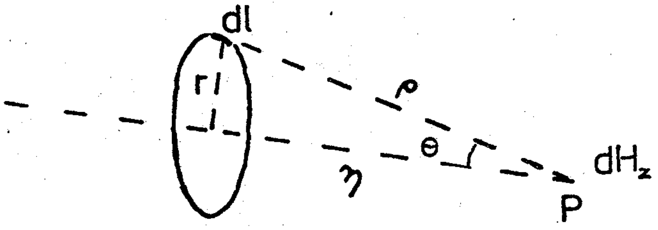


Fig. 16. Magnetic Field due to Current Loop.

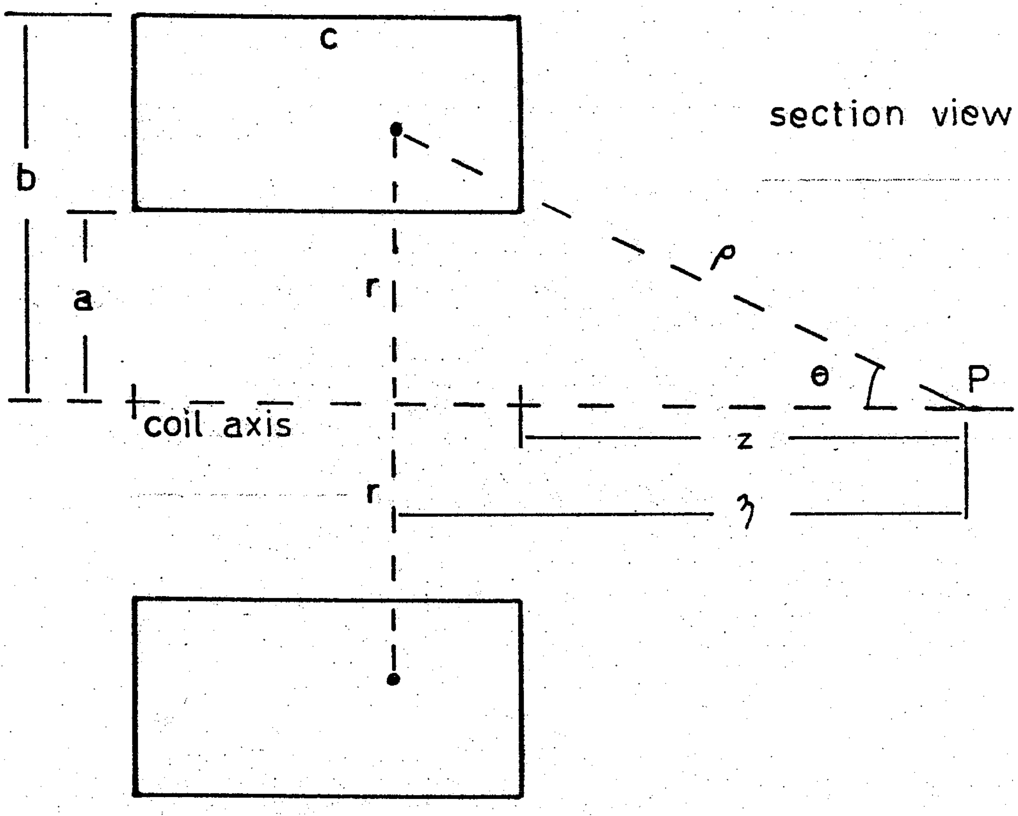


Fig. 17. Magnetic Field due to Coil - Dimensions for Equation

where I is the current in amperes; m,n, are turns per unit length and layers per unit length. Other symbols are shown in fig. 17.

The magnetic field profile along the axis was computed for a given set of dimensions and plotted. The region near the point Z = 0 was seen to have a reasonably uniform gradient.

The dimensions of the coils were limited by those of the older coils and of available materials for the new coil former. In order that the former would safely withstand any heat from the furnace, heat resistant Transite was chosen. The minimum value of 'a' was 4.5cm. as a result of this choice. The older coils restricted 'b' to not greater than 10cm. and the maximum allowable value 'c' was considered to be about 10cm. By computing the field and the ratio of gradient at Z = 0 to volume of the coils for various lengths of 'c', it was found that for C = 10cm. about 90% of the maximum gradient for the above 'a' and 'b' could be achieved. To use the value of 'c' for optimum use of copper (about 5.5cm.) would not have provided a sufficient range of gradient. It was decided to use C = 10cm. in order that the coils need not normally be operated near maximum current ratings.

During construction an improved method of interlayer insulation was used, which reduced the stresses on the ends of the former. This method reduces the number of turns which may be wound into a given space. In these coils, the decrease of about 12% was not considered too great, as sufficient margin was available, with c = 10cm., for present requirements. Maximum working gradients were expected to be of the order of 100 oersted/cm. and with a current of 4 amperes, a gradient of about 115 oersteds/cm. can be achieved.

The wire used was 14 gauge enamelled copper wire, for which the maximum rated current is 8 amperes and the resistivity at 20°C is 2.525 ohm per 1000 feet. The number of turns wound on each coil is 1681, and the measured resistance of the coils is 12.8 ohm. The power dissipation at 4 amperes is 200 watt and the coils are found to be warm after several hours at this rate.

Using a standard formula (Harnwell 1949, p.472), the inductance L was estimated.

$$L = \frac{0{\cdot}32\,a^2 n^2}{6a + 9b + 10c}$$

a, b, c in cm.

where n is the total number of turns,
a is the average radius of windings,
b is length of coil,
c is depth of windings.

A value of about 0.25 henry per coil was obtained.

The coils are connected in series, giving a total inductance of about 0.5 henry. The magnetizing field coils are connected in parallel, with a combined inductance of about 0.5 henry and combined resistance of 12.8 ohms.

APPENDIX 2

DESIGN OF THE FURNACE

The basic requirement was to obtain a temperature of about 800°C maximum in a time of the order of 30 minutes, and to be able to cool in about the same time. Water cooling was considered advisable in view of the small amount of insulation to be used.

Estimates were made of the heat budget in the furnace in order to permit computation of the electrical requirements. The design was limited by available materials. As an outer sheath, a 2 inch I.D. copper pipe was suitable, and as a refractory a 1 inch I.D. Alundum core, of 3/16 inch wall thickness, 6 inches long was obtained. A commonly used high temperature insulation is calcined magnesia.

An estimate of the radial heat loss was made using the standard radial heat flow equation for a multi-layer cylinder, as found in textbooks on heat (e.g. Noakes 1957).

$$\frac{dQ}{dt} = \frac{(\theta_1 - \theta_2)\, 2\pi l}{\sum_n \frac{1}{k_n} \log_e (r_{n+1}/r_n)}$$

where $\frac{dQ}{dt}$ is rate of flow of heat

θ_1, θ_2 are inner and outer temperatures respectively

l is length of cylinder

r_{n+1}, r_n are inner and outer radii of the nth layer

k_n is thermal conductivity of the nth layer.

Using the above equation, and the approximate dimensions and characteristics of the materials, an estimate of 50 calories per second radial heat loss was obtained.

Assuming the use of a linear rate of increase of temperature for the furnace, the average radial heat loss was about 25 calories per second.

An estimate of the heat required by the Alundum refactory, which will be at a temperature close to that at the centre of the furnace, gave a value of 30,000 calories to reach maximum temperature.

The mass of magnesia was estimated at 5 grams, the packing density not being known. Taking the average temperature of the magnesia to be of the order of 350°C, an estimate of 400 calories was obtained.

Heat is also supplied to the central portion of the quartz hangdown tube. Accurate estimates would not be warranted, but a rough estimate of 20,000 calories total requirement was made.

Other losses such as radiation, convection in the quartz tube and losses from the end plates were not computed in detail, but were considered to be relatively small. Taking this into consideration, a total heat requirement for the furnace was estimated at 100,000 calories.

Using this figure for a 30 minute heating period gave an average electric power requirement of approximately 250 watts, with a maximum of about 500 watts.

The heat to be removed in cooling amounted, on the above estimates, to about 50,000 calories. Assuming an average loss of 25 calories per second, the cooling time would be about 35 minutes.

The electrical power requirements are a maximum of 500 watts, operating at 110-120 volts A.C. Alumel was chosen as a suitable resistance wire for the element and the manufacturers' tables indicated that for 22 gauge wire at 115 volts, 500 watts, a length of 24.3 feet was required.

The inside diameter of the refactory is 1 inch, and the length of one turn would be about 3 inches, giving the number of turns as 97. The diameter of the wire is 0.0253 inch, and with spacing one diameter, the length of winding would be about 5 inches. The winding is non-inductive.

For 500 watts, a current of 4.35 amps. is required. The maximum safe current for 22 gauge Alumel wire is indicated in the wire tables to be 5 amps. During the construction of the furnace it was found to be safer to use a slightly larger spacing and a compromise of 21 feet of wire was used. This practical matter is not serious since the furnace is controlled by a variable transformer, or a furnace controller. The required power is still available.

The maximum radial heat flow is 50 calories per second; that is, the heat is transferred to the cooling water at 50 calories per second. For a maximum rise in temperature of water of 25 degrees the mass flow of water is given by:

$$\text{Mass/sec.} \times 1 \times \text{Temperature rise} = \text{Radial heat flow}$$

$$\text{i.e. Mass/sec} = 50/25 \text{ gm/sec.}$$

Thus, the rate of flow is 2 cc/sec. which is quite feasible. A much smaller temperature rise can be maintained in practice, using a greater flow.

APPENDIX 3

USE OF ALTERNATING CURRENT FOR THE PRODUCTION OF THE MAGNETIC FIELDS

The idea presented itself that perhaps alternating current might be used in the presant arrangement to produce a force on the sample.

The field H will be of the form $H = H_0 \cos \omega t$ and the gradient $\frac{\partial H}{\partial z} = \frac{\partial H_0}{\partial z} \cos \omega t$.

In the simple case, where magnetization J is proportioned to field strength, H,

$J = kH$, k being constant,

then the force F exerted on the sample, volume v will be

$$F = vkH_0 \frac{\partial H_0}{\partial z} \cos^2 \omega t$$

If the balance and recorder effectively integrate this force over a number of cycles, the force recorded will be an average over a cycle, and its relation with F may be calculated.

In the more general situation, $J = J(H)$, the dependence not being known, prior to measurement.

Then, $F = v \nabla (H_0 . J) \cos \omega t$.

J will still be periodic, and if the balance system can integrate effectively over all relevant periods, the force recorded may be related to the true force F.

This method has some advantage in that D.C. rectification and smoothing circuits would not be necessary. Consequently trials were made with A.C. in the Helmholtz coils, placing a sample in a region of appreciable gradient. Although the system worked effectively in producing a force, integrated by the balance and the recorder, more problems arose which precluded the use of A.C. in the system in its present form.

Excessive vibrations occurred in the coils and were transmitted to the balance and sample suspension, producing a noisy trace. The periods of the vibrations covered a broad spectrum and could not be adequately filtered out by the balance controller. It was considered unlikely that rigid clamping could effectively eliminate this.

A second problem arose with the production of eddy currents in metal parts. These currents caused considerable heating in certain places and it was felt that damage to electronic equipment might occur if A.C. were to be used. It was noticed that one moving coil meter gave false readings when the fields were on.

It was concluded that, although not usable in the present system, alternating current supplies to air core solenoids could provide an inexpensive and flexible means of producing a force on a magnetizable sample.

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