GEOLOGICAL SURVEY OF CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

PAPER 68-37

A RECORDING THERMOMAGNETIC BALANCE

(Report and 6 figures)

E. J. Schwarz

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Abstract

An instrument is described that records continuously the force acting on a small specimen kept stationary in an inhomogeneous magnetic field produced by an electromagnet with specially cut pole caps. The force is displayed along one of the axes of an xy recorder while either the temperature of the specimen (variable between -196 and 800°C) or the magnetic field strength (variable between 0 and 7900 oe) is read along the other axis. The smallest magnetic moment that can be displayed per inch of the recorder chart is 1.27×10^{-5} cgs emu + 3%. The sensitivity may be decreased in calibrated steps to a maximum of 2.5 cgs emu/inch deflection during operation. The system may be evacuated to below 10^{-3} torr. Some typical results are added and briefly discussed.

A RECORDING THERMOMAGNETIC BALANCE

Introduction

Magnetic balances form part of the standard equipment of many laboratories, and are used in determining certain magnetic properties of rocks and individual minerals. These properties usually include the saturation magnetization, the thermal change of the saturation magnetization, and Curie points. The determination of these properties yields information on the magneto-mineralogy of the specimens under study as in most cases these properties uniquely define the minerals present regardless of grain size. Moreover, the abundances of such minerals can be estimated. This information is useful to paleomagnetists particularly if thermal magnetic washing of a series of specimens is being considered. Finally, the determination of the change of the saturation magnetization with temperature indicates whether or not physico-chemical changes affecting the magnetic minerals occur at certain temperatures during heating. By this means, it may be possible to obtain information on the thermal history of the rock, information that is useful in petrogenic and paleomagnetic investigations.

Magnetic balances of the types already in existence may be sufficiently sensitive for these purposes, but they have the drawback that their sensitivity cannot be changed during operation. However it is desirable that sensitivity be variable especially in the investigation of rocks containing both magnetite and hematite, which are not only the most common magnetic minerals but which may be chemically transformed from one into the other during an experiment. It should be possible to increase the sensitivity of an instrument when the temperature of the specimen equals or exceeds the Curie point (580°C or less) of the strongly magnetic magnetite so that the magnetization due to the weakly magnetic hematite fraction (Curie Point 670°C) can be examined in detail. The problem is encountered for instance in the study of red beds. Much paleomagnetic work has been carried out on red beds because their high magnetic stability is attractive. However, in many cases it is uncertain when these sediments acquired their stable magnetization so that paleomagnetic pole positions derived from these rocks may not correspond to the geomagnetic pole at the time of formation of the rocks.

A magnetic balance with variable sensitivity also allows the detailed investigation of the magnetic susceptibility of minerals to be extended above their Curie points. This type of investigation may reveal the occurrence of physico-chemical processes affecting the mineral under study. An example is the complicated thermomagnetic properties of pyrrhotite which may have a potential as a geothermometer.

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In the following, a brief description is given covering the important points of a magnetic balance of variable sensitivity designed and built at the Geological Survey of Canada. Results obtained for magnetite, hematite, and pyrrhotite are added to illustrate their quality and diagnostic value.

Principle of design

A magnetic balance measures the translational force ($\bar{F}$) exerted by an inhomogeneous magnetic field ($\bar{H} = \bar{H}(\bar{r}) = \bar{H}(x, y, z)$) on a specimen suspended from one of the beams of the balance. Usually, an expression for $\bar{F}$ is derived on the basis of the potential energy of a specimen in $\bar{H}$ (e.g. Bates, 1951). Another approach is given below.

A small specimen may be regarded as a dipole of variable moment $\bar{p} = m\bar{l}$, in which $\bar{l}$ is the distance between the poles -m and +m. The force $\bar{F}$ is the resultant of the forces acting on both poles:

$$\bar{F} = m\bar{H}\,(\bar{r} + \bar{l}) - m\bar{H}\,(\bar{r}) = \nabla\,(\bar{p}.\bar{H}), \qquad (1)$$

in which $\bar{p} = m\bar{l}$.

In the present case, the specimen is vertically suspended in $\bar{H}$ while $\bar{F}$ is measured with a balance. Thus, the horizontal components F_x and F_y are to be minimized by suspending the specimen along the designed axis of symmetry of $\bar{H}(\bar{r})$ while the vertical component F_z is to be maximized for optimum sensitivity. The magnitude of this component in terms of the Cartesian coordinate system is:

$$F_z = \frac{\delta(p_x H_x)}{\delta z} + \frac{\delta(p_y H_y)}{\delta z} + \frac{\delta(p_z H_z)}{\delta z} = v\,\frac{\delta(\bar{J}.\bar{H})}{\delta z}, \qquad (2)$$

v and J (x, y, z) denoting respectively the volume and the magnetization of the specimen.

The magnetization is a linear function of the magnetic field acting on the specimen if the latter is paramagnetic or diamagnetic. For isotropic specimens and J constant one obtains

$$F_z = v\,k\,H\,\delta H/\delta z\,, \qquad (3)$$

in which k indicates the volume susceptibility. Hence, it is required to select the region of maximal and reasonably constant $H\,\delta H/\delta z$ for the specimen.

The relation between J and H is complicated for specimens showing magnetic hysteresis. However, J tends to become virtually constant in saturating fields so that the following relations are obtained:

$$F_z = v\,\bar{J}.\frac{\delta\bar{H}}{\delta z}\,,$$

and for isotropic specimens

$$F_z = v\, J \frac{\delta H}{\delta z} \qquad (4)$$

Consequently, a saturated specimen should occupy a position along the z axis such that $\delta H/\delta z$ is maximal and is reasonably constant over its volume.

Equations (3) and (4) may be used to determine J or k from the measured value for F_z. The effect of the medium of susceptibility k_m, displaced by the introduction of the specimen in H, may be taken into account by replacing k or J by, respectively, $(k-k_m)$ and $(J-k_mH)$.

Description of the instrument

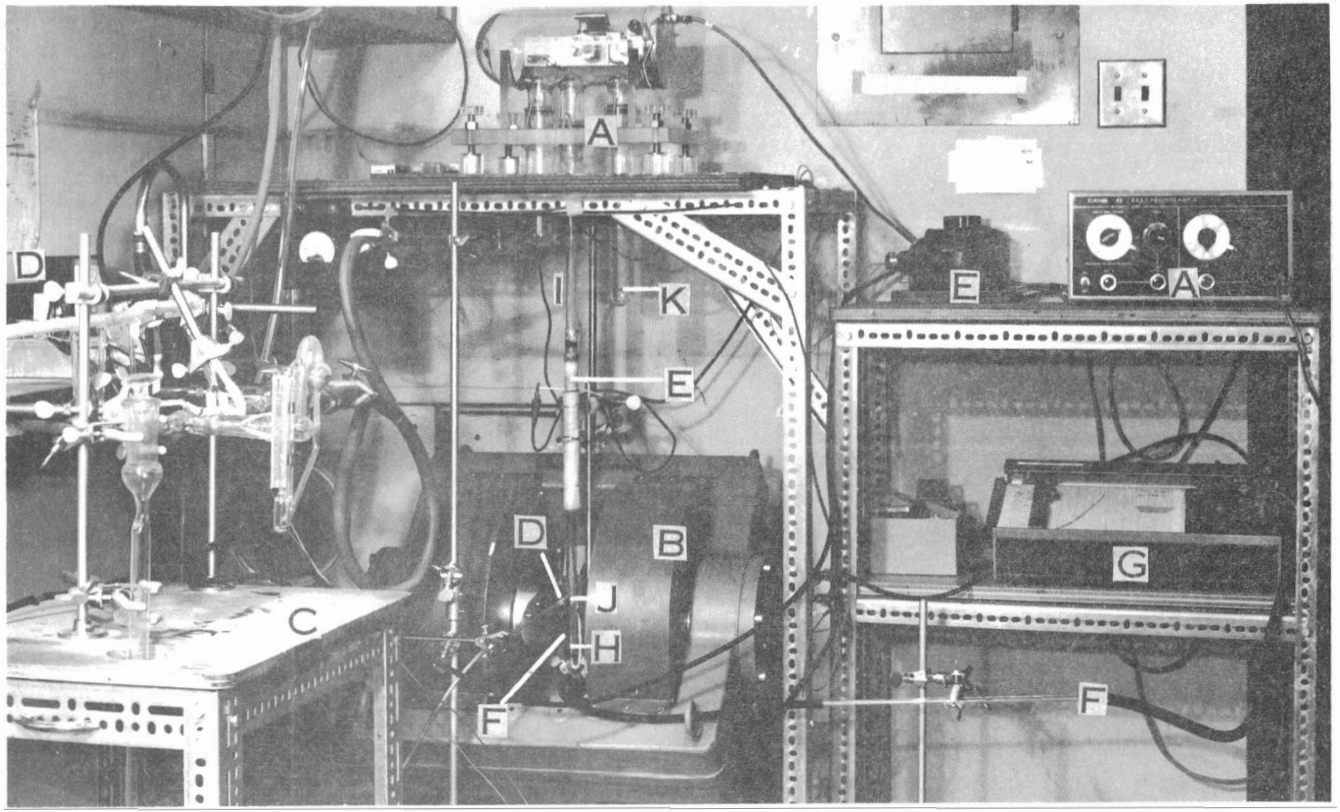
The main parts of the instrument are the electrobalance, the electromagnet, the oven and Dewar flask assembly, and the vacuum system as shown on Figure 1.

The balance is a commercially available unit (Cahn Co., model RG). Its beam is kept at a fixed position without noticeable oscillation by a rapidly responding photoelectric feed back system. In addition, the feed back current is amplified and fed into an xy recorder so that the force acting on the specimen is continuously recorded.

The sample is placed in a quartz-glass bucket (J on Fig. 1) which is suspended by quartz-glass fibre and nichrome fibre from one of the beams of the balance inside a closed quartz glass tube (I on Fig. 1). This tube is attached by a ground joint to the glass vessel in which the balance is placed. A counterweight is placed on a pan (K on Fig. 1) so that virtually the full capacity (up to 1000 mg) of the automatic feed back system of the balance may be used in recording F_z.

A pole cap design reported earlier by Garber et al. (1960) and Keyding et al. (1961) was adopted for a 6-inch electromagnet to meet the requirements of maximal and constant H $\delta H/\delta z$ and $\delta H/\delta z$ over the volume occupied by the specimen. Figure 2 shows some of the characteristics of the field produced at 70 per cent of the capacity of the electromagnet. The field gradient $\delta H/\delta z$ at the location of maximal H $\delta H/\delta z$ is comparable to the maximum gradient reached in the z direction but the sections of reasonably constant $\delta H/\delta z$ and H $\delta H/\delta z$ do not overlap.

H is measured with a small Hall effect sensor located in a position at which the field characteristics are comparable to those at the position of the specimen. A scanner is used to vary H continuously. A continuous record of F_z (H) is obtained by recording simultaneously the output of the sensor and that of the balance. The field produced by the magnet does not interfere noticeably with the electromagnetic system that provides the force for keeping the beam of the balance in its original position. Changes in specimen weight during heating can be detected by checking the recorder output after nulling H, and can be taken into account in the determination of F_z.



(GSC 200443-B)

Figure 1. General view of the instrument. The labelled components are:

A. the electrobalance and its control unit.
B. the electromagnet.
C. the pump unit.
D. the Hall effect probe and its power supply.
E. the furnace and its power supply.
F. the Dewar flask and the liquid N_2 line.
G. the xy recorder.
H. the insulated thermocouple.
I. the detachable quartz-glass tube.
J. the specimen container.
K. the counterweight pan.

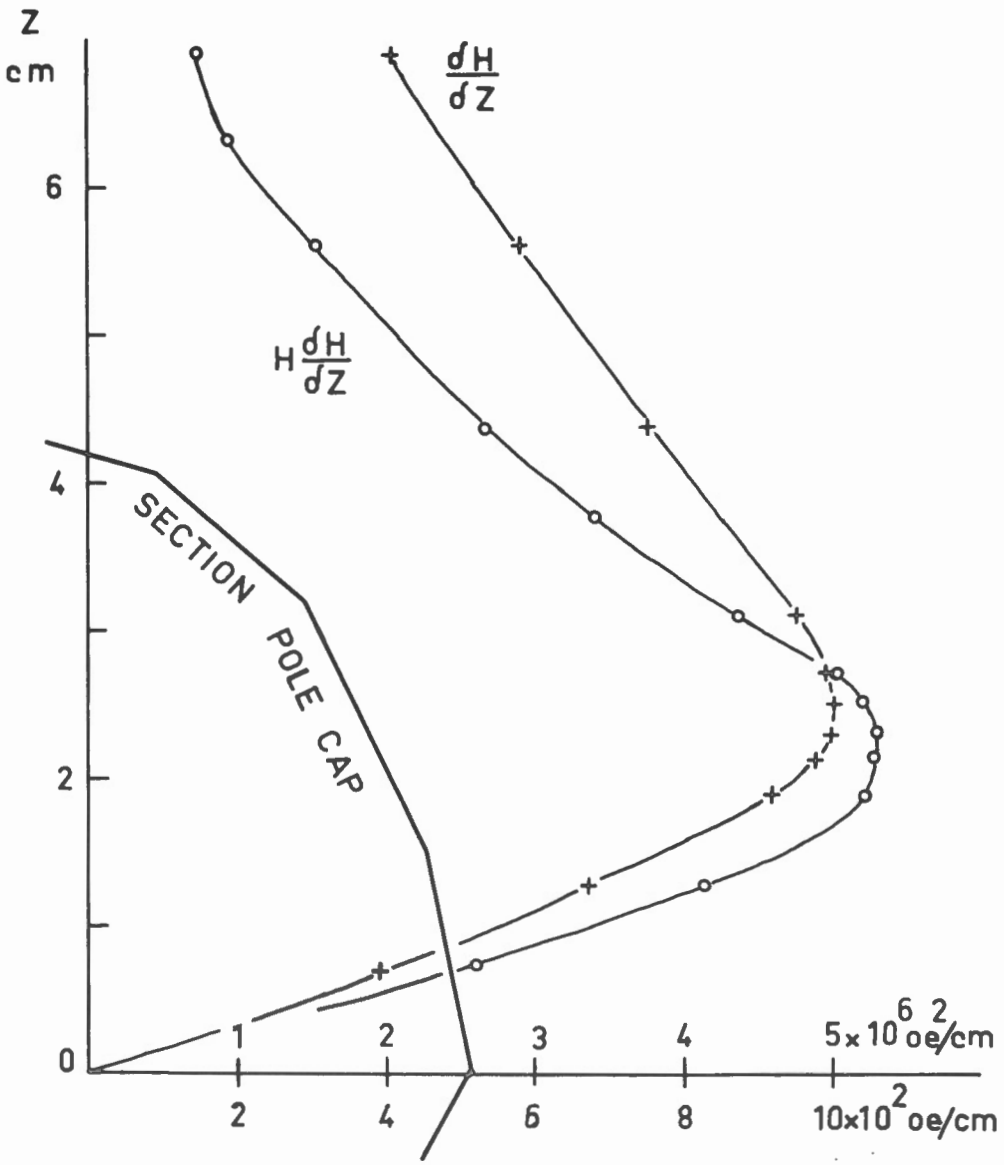


Figure 2. Some characteristics of the magnetic field measured along the vertical axis of symmetry (Z) of the pole cap configuration. The section of a part of one of the pole caps is added (scale in centimetres). The energizing current (1 amp) was 70 per cent of the maximum available.

The temperature of the specimen may be varied between -196 and about 800°C and is measured with a thermocouple. The Dewar flask is easily removed after cooling the specimen to -196°C (liquid N_2 temperature) and subsequent warming to 20°C. Then the furnace is moved down along tube I (see Fig. 1) to take the position of the Dewar flask and heating of the specimen above 20°C may be initiated. The variable transformer in the oven circuit is replaced by a programmable temperature controller (F & M model 240 M) for automatic operation. A continuous record of the thermal change of F_z is obtained by recording simultaneously the output of the balance and that of the thermocouple.

Calibration and sensitivity

The standard sample suspension system was used in the calibration tests and its contribution was subtracted from the total F_z recorded for the system and specimen to obtain the contribution to F_z of the specimen alone (F_z^s). The result of one test with 19.55 mg $Fe(NH_4)_2 \cdot 6H_2O$ is shown on Figure 3. The susceptibility k may be calculated using equation (3) from the F_z^s value recorded for H = 5350 oe and $\delta H/\delta z$ = 1000 oe/cm (Fig. 2). The k value obtained in this manner is accurate to within 2 per cent and is within 1 per cent of that (31.4×10^{-7} cgs emu at the temperature of measuring, 22.5°C) obtained from the data listed by Hodgman (1952).

The sensitivity of the instrument may be given in the following terms:

1. The smallest magnetic moment that can be displayed per inch of the recorder chart is 1.27×10^{-5} cgs emu. This figure can be increased to a maximum of about 2.5 cgs emu. For each of the sensitivity scales, the resolution is 1 per cent of the moment corresponding to the full scale deflection of the recorder, and the absolute accuracy is better than 3 per cent.

2. Specific magnetic susceptibility of the order of 1.10^{-8} emu may be determined with an accuracy better than 3 per cent for samples weighing about 300 mg. The requirement of reasonably constant $H\,\delta H/\delta z$ over the volume occupied by such relatively large specimens still is fulfilled.

Typical results

The described instrument is used in the investigation of magnetic properties of minerals and rocks. Typical results obtained with the instrument for commonly occurring minerals are shown on Figures 4, 5, and 6.

Figure 4 shows the results for a specimen of coarsely crystalline hematite. Abrupt changes in the magnetization are observed at 258°K ± 2 (Morin transition) and at 951°K ± 3 (Curie point). The occurrence of these changes in specimens of rocks indicates the presence of αFe_2O_3 and yields information on its composition and grain size.

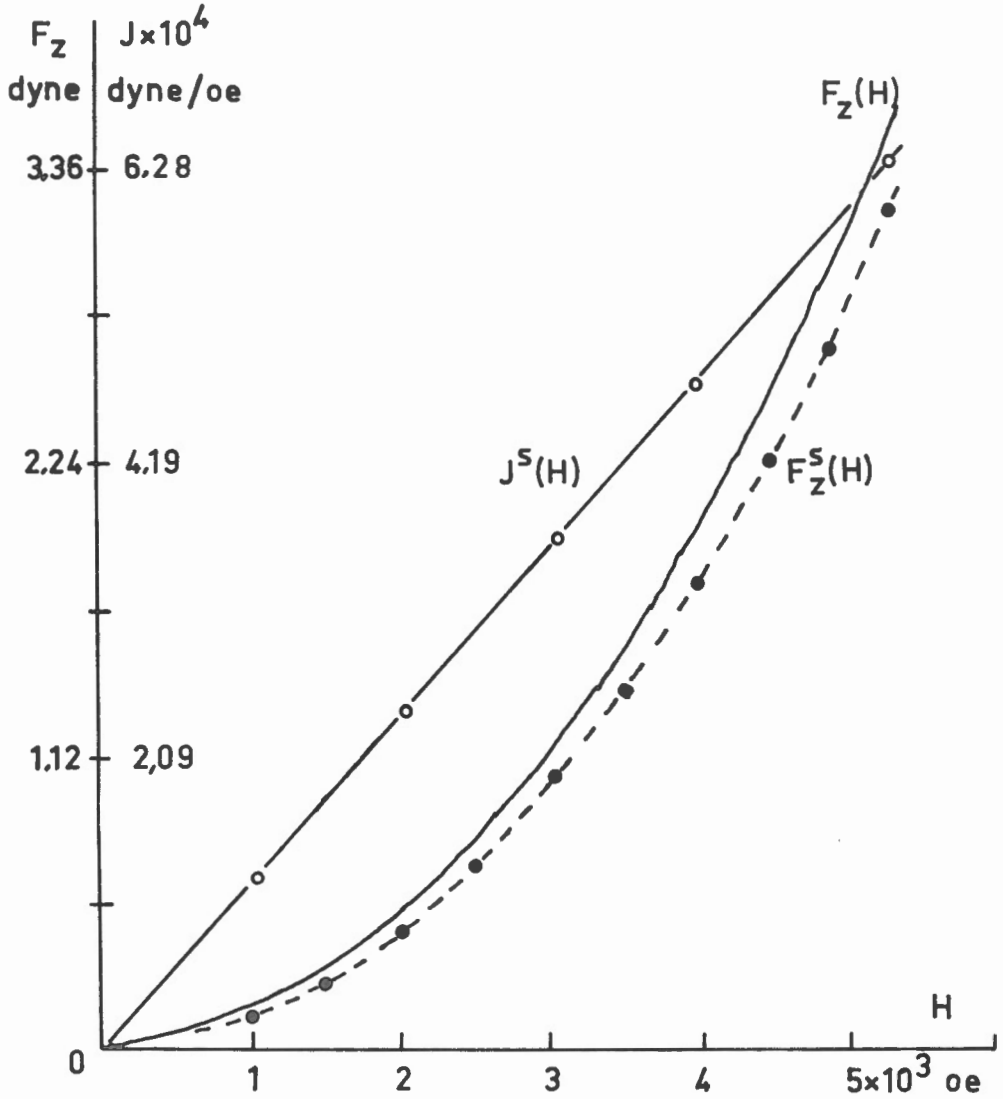


Figure 3. The results for a sample of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ at 22.5°C. The curve $F_z(H)$ was traced from the original record, $F_z{}^s(H)$ shows the vertical component of the force on the sample only, and $J^s(H)$ shows the dependence of the magnetization of the sample on the applied field.

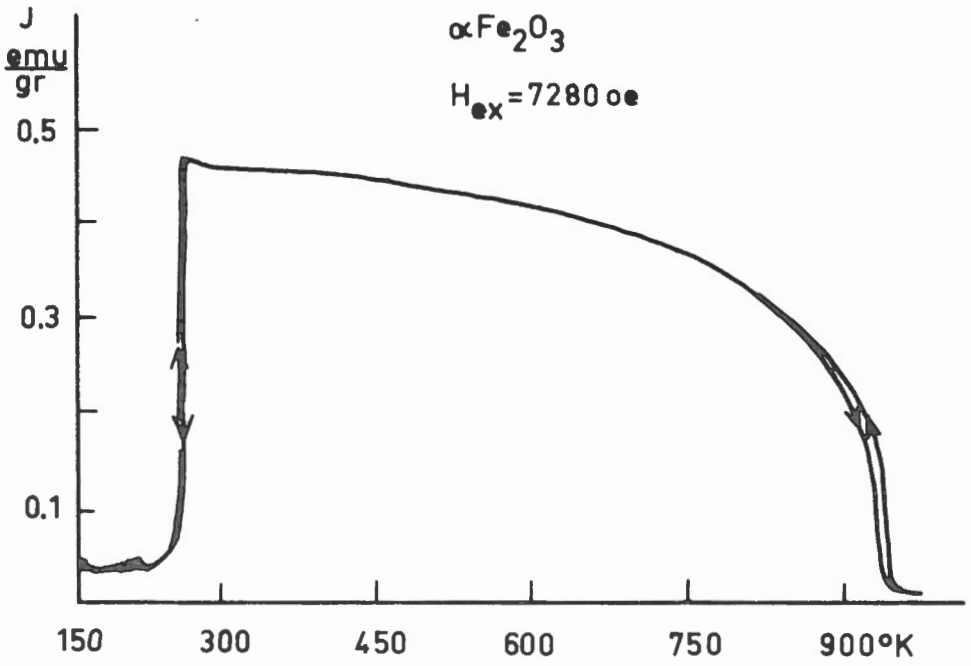


Figure 4. Recorded variation in magnetization of a sample of hematite during heating and subsequent cooling.

Figure 5 shows the results for a single crystal of pyrrhotite of bulk chemical composition $Fe_{0.91}S$. A strong increase in magnetization is observed between 483 and 493°K during heating. This increase may be due to generation of long range order of vacant cation sites (9 per cent of total cation sites). Moreover, it is thought that the heating curve indicates the presence of two simultaneously occurring phases: (1) is ferrimagnetic up to Curie point at 573°K + 3 and its composition is about $Fe_{0.88}S$ whereas (2) becomes ferrimagnetic during heating at 483°K and shows a Curie point at 533°K ± 4. The J (T) curve can be displayed in considerably more detail than shown on Figure 5. It is hoped that the analysis of such curves will lead to a better understanding of the interesting thermal variation of the magnetization of pyrrhotite.

The results for 1.84 mg of magnetite are shown on Figure 6. The Curie temperature is 853°K + 2. A decrease in magnetization is also observed at about -123°K during cooling of the specimen. This isothermal decrease in magnetization is generally thought to be caused by a strong increase in magnetocrystalline anisotropy during the first order transition of cubic to orthorhombic crystal symmetry of magnetite. The transition is reversible and the change in susceptibility associated with its occurrence is suppressed if saturating magnetic fields are used.

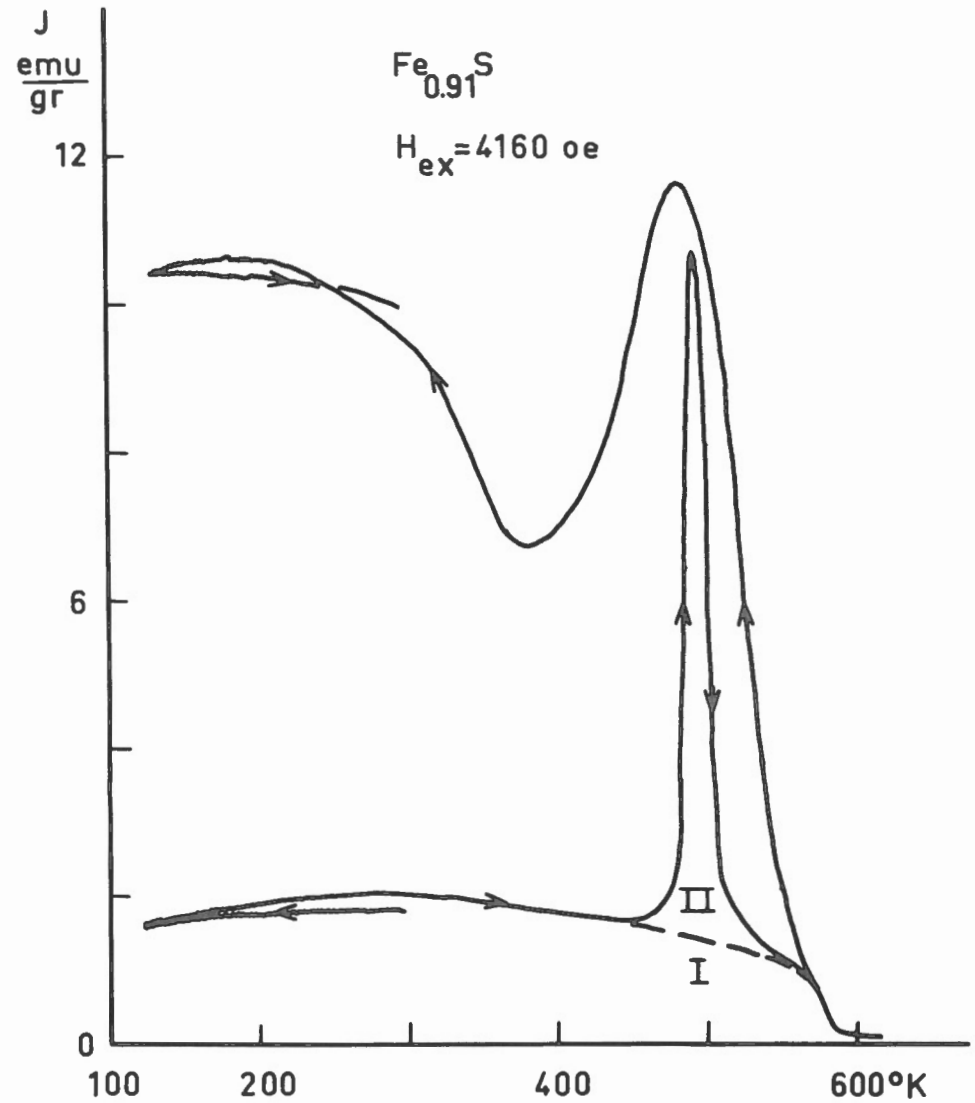


Figure 5. Thermal change of the magnetization for 34 mg of $Fe_{0.91}S$ as drawn from the record.

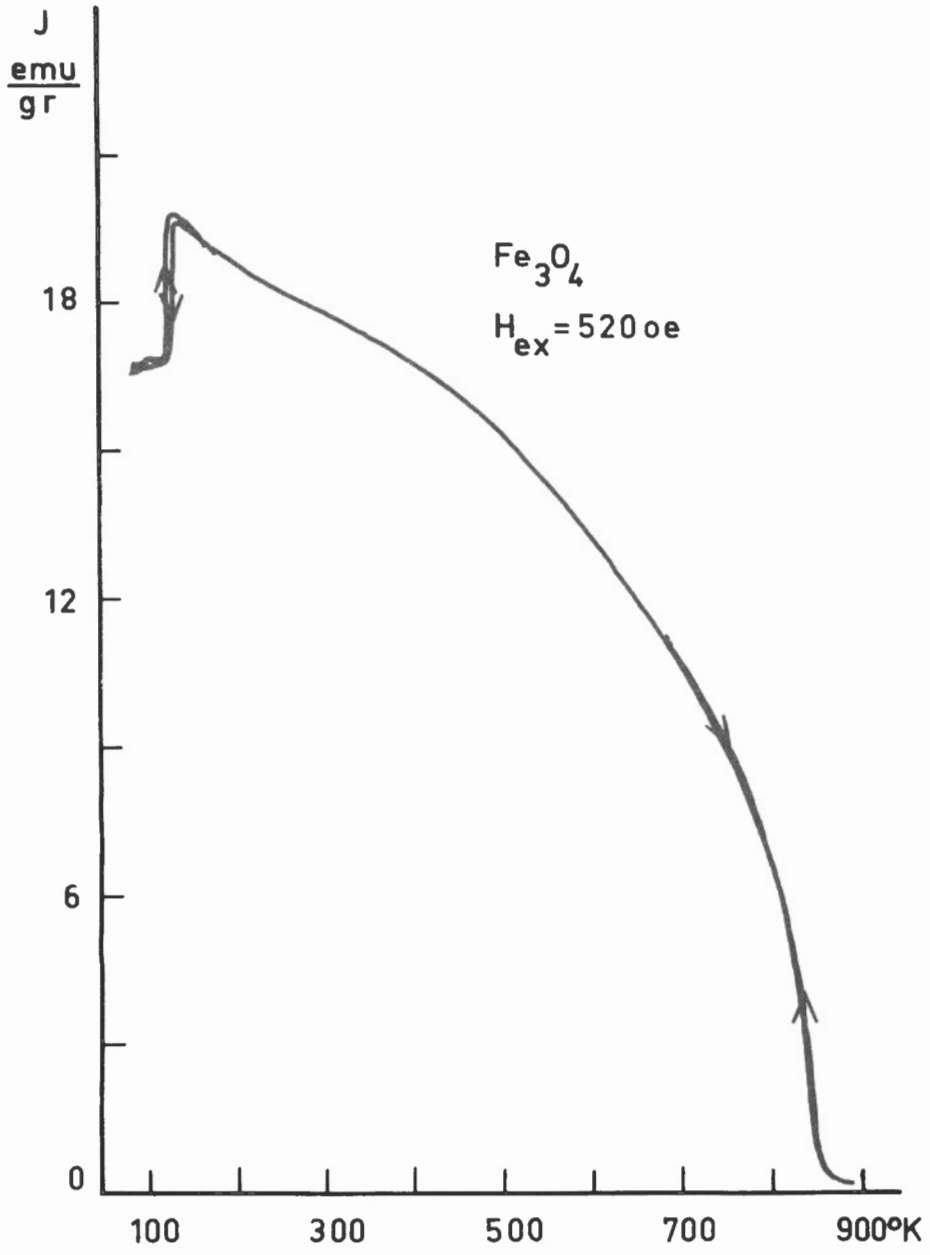


Figure 6. Variation of the magnetization acquired by a sample of magnetite during thermal cycling.

Summary

The general specifications of the instrument are:

1. The smallest magnetic moment that can be displayed per inch of the recorder chart is 1.27×10^{-5} cgs emu $\pm$ 3 per cent. This figure can be increased in calibrated steps to a maximum of about 2.5 cgs emu.

2. The magnetic field acting on the specimen may be varied between 0 and 7900 oe.

3. The temperature of the specimen may be varied between -196 and about 800°C.

4. Either the field dependence or the thermal dependence of the specimen magnetization is recorded by the instrument. The variation of the magnetic field or the temperature may be programmed to a large extent.

The instrument is used in the determination of magnetic phases in rocks. Recorded abrupt changes in magnetic moment between -196 and about 700°C are valuable for this purpose. These changes may be due to a variety of causes but have a high diagnostic value. In simple cases, the abundance of magnetic minerals may be estimated from the record of the field dependence of the specimen magnetization. Because the sensitivity may be changed at any time during the experiment, the instrument is very well suited for the investigation of rocks such as red beds which may contain minerals of both high and low magnetic susceptibility and of different Curie points. Moreover, the high sensitivity allows investigation of magnetic properties of minerals above their Curie points. Such experiments may be desirable in the investigation of magnetic structures and the possible effect of lattice component order-disorder transitions and crystallographic transitions on magnetic parameters.

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