

LABORATORY SIMULATION OF MAGNETIZATION CHANGES
CAUSED BY BURIAL METAMORPHISM

LABORATORY SIMULATION OF MAGNETIZATION CHANGES
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BY

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ABSTRACT

Magnetization associated with the emplacement of a rock body may be thermo-remanent magnetization (TRM) in an igneous body or detrital-remanent magnetization (DRM) in a sedimentary deposit. At the time of formation the acquired remanence will tend to lie in the ambient field direction. This primary remanence may not remain unchanged through geologic time. Viscous demagnetization may progressively destroy the remanence in the primary direction or it may be reset as VRM in a later different ambient field direction. The amount of acquired VRM will depend on the temperature the rock is heated to and the length of time the heating lasts as well as the magnetic properties of the remanence carriers.

The remanence may also be changed by chemical reactions taking place in the magnetic minerals. The remanence acquired during these chemical changes (CRM) is found, by this work, to be a determining factor in the stability of and initial NRM during thermal remagnetization.

Synthetic samples were stored for up to 32 days at 400°C it is possible to access geologic time. To simulate viscous changes over geologic time elevated temperatures applied for laboratory times are related to longer times at lower temperatures using the thermal activation curves. Changes in remanence during the storage were observed at various times throughout the experiment. The remanence in samples with an initial NRM was diminished , remanence in samples with a weak initial NRM increased in

intensity but the remanence direction lay in the field direction after as little as 1/2 a day storage time.

Thermal demagnetization allows separation of the magnetization on the basis of blocking temperature spectra. During the storage the initial magnetite was oxidized to cation deficient magnetite with significantly higher blocking temperatures than those found in the magnetite. Hysteresis measurements, and thermomagnetic measurements indicate that this change results primarily from the shift in T_c due to cation deficiency. Apparently the change in T_c did not significantly affect the mechanisms responsible for blocking remanence but merely shifted the blocking temperatures by a similar amount. In samples with a weak initial NRM the chemical change completely reset the magnetization and in samples stored for times as short as only 8 days the initial remanence direction could not be recovered.

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CHAPTER ONE

A THEORETICAL DESCRIPTION OF MAGNETIZATION CHANGES EXPECTED AS A RESULT OF BURIAL METAMORPHISM

1.0 INTRODUCTION

When a rock containing an initial magnetization is heated a change in that magnetization may occur. This heating could be in response to a variety of geologic processes, burial, contact metamorphism, or regional metamorphism. As heating proceeds the associated thermal energy may partially demagnetize the original remanence or replace it with a viscous remanent magnetization or VRM. The elevated temperatures may also cause chemical changes which will result in the acquisition of chemical remanent magnetization or CRM. In more extreme conditions the blocking temperatures of the pre-existing magnetization or NRM may be exceeded which will have the result of totally resetting the magnetization.

The magnetic viscosity affects the rock by, i) the acquisition of VRM in the direction of the field and, ii) the decay of pre-existing remanent magnetization. (Nagata, 1961) The stability of the NRM depends on the both the relative build up of VRM and the time decay of the initial remanence. Chemical changes of the magnetic minerals carrying the magnetization will result in the acquisition of CRM which may lie in the field direction, (Haigh, 1958) the initial or NRM direction or in a direction somewhere between the initial NRM direction and the

direction of the ambient field (Bailey & Hale, 1981).

1.1 THE THEORY OF VRM

The theory of the viscous magnetization of a single domain magnetic particle was presented in a classic paper by Neel (1949). As a magnetic grain cools through its Curie temperature (T_c), it becomes magnetized in equilibrium with the ambient field H . This magnetization becomes stabilized when the temperature drops below the blocking temperature (T_b) and there is no longer sufficient thermal energy to permit the grain to equilibrate with a changing magnetic field. The magnetization will be given by,

$$m = vJ \tanh(vJ(T_b)H(T_b)/kT_b) \quad [1]$$

where m is the magnetic moment

v is grain volume

J is the spontaneous magnetization

$J(T_b)$ is value of J at T_b

$H(T_b)$ is the coercive force at T_b

T_b is the blocking temperature

A rock can be considered to be an assembly of these grains each contributing to the overall remanence of the rock. The decay of remanence in zero field: the magnetic moment becomes

$$m = m_0 \exp(-t/\tau) \quad [2]$$

where m is the magnetic moment at time t

t is time

m_0 is the initial moment

τ is the relaxation time

The relaxation time is defined as the time required for the

magnetization to decrease to 1/e of its initial value at a given temperature, hence τ is a measure of the stability of the magnetization. This exponential indicates that the magnetization is decreasing with time since the exponent will always be negative.

1.2 TIME TEMPERATURE AND RELAXATION THEORY

Dunlop (1973) has proposed theoretical time-temperature relations using the theory of Neel (1959) to model the decay and acquisition of VRM. The relaxation time of an assembly of single domain grains is given by,

$$1/\tau = C \exp(-vJ_s(T)H_c(T)/2kT) \quad [3]$$

where C - a frequency factor (=10¹⁰ Hz.)

J_s(T) - spontaneous magnetization
as a function of temperature

v - grain volume

H_c(T) - coercive force as a
function of temperature

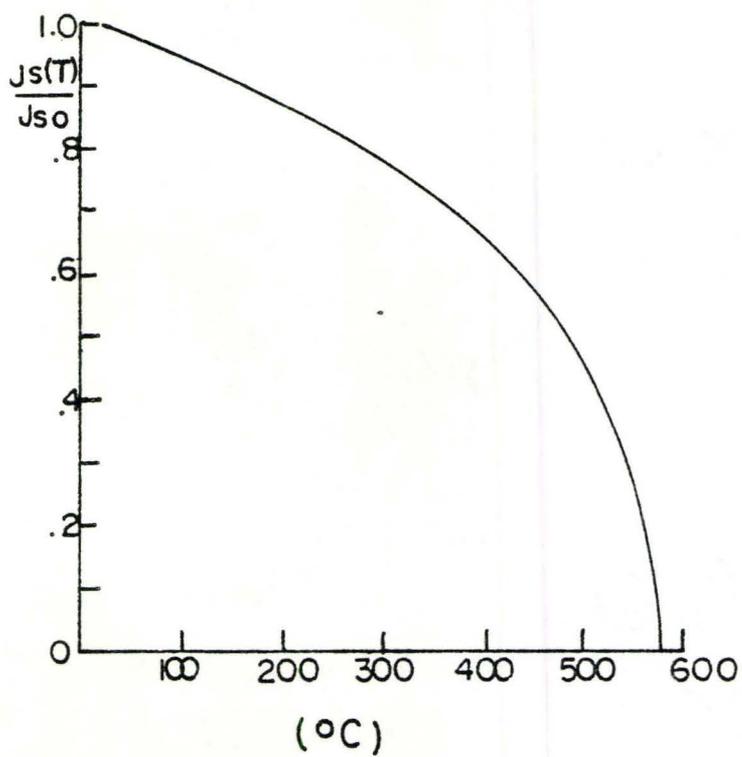
k - Boltzmann's constant

T - absolute temperature

τ - relaxation time

From equ. [3] it can be seen that the stability of the magnetization or τ will increase as the temperature decreases. Hence it will require longer times to reset the magnetization at lower temperatures. Also, the exponential in equ. [3], coupled with the exponential from the magnetization expression, [2], produces a relation which shows that the relaxation time is very

Fig 1-1 Spontaneous magnetization as a function of temperature, $J_s(T)$ for magnetite. J_s vanishes at the Curie temperature.



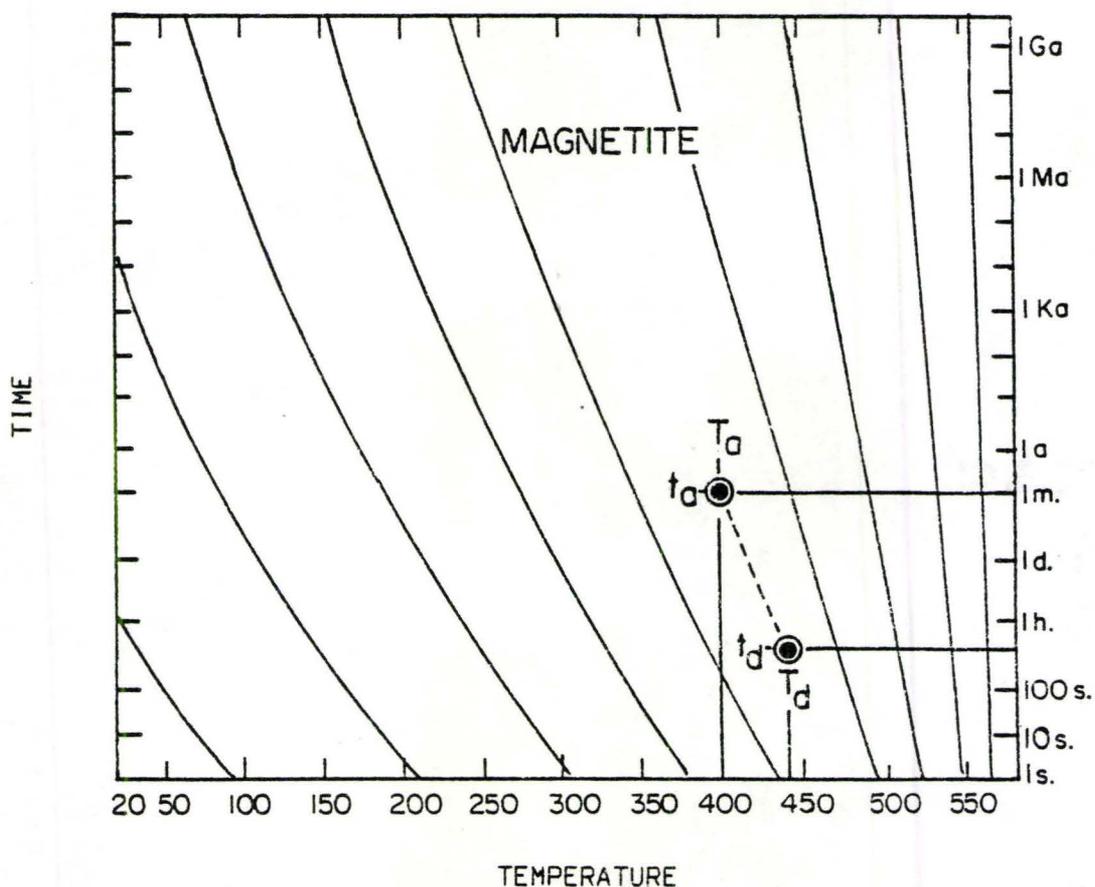


Fig 1-2 Thermal activation curves for magnetite, after Pulliah et. al. (1975). These curves are contours of equivalence between time and temperature in blocking and unblocking of TRM. A remanence acquired at 400°C for one month should demagnetize in 30 minutes at a temperature of 440°C. Geologic time can be simulated since this is equivalent to heating to a temperature of 350°C for 10⁶ years.

sensitive to small changes in the temperature. The blocking temperature is given by,

$$T_b = vJ_s H_k / 2k \log_e(\tau / C) \quad [4]$$

Equ. [3] can be simplified by assuming the grain size to be a single effective grain size for each sample. The dependance of J_s on temperature has been determined experimentally and is shown in figure 1-1. In magnetite, H_k arises from shape anisotropy in even slightly elongate particles (Dunlop, 1981). Shape anisotropy is related to $J_s(T)$ by,

$$H_k(T) \propto J_s(T) \quad (\text{Nagata, 1961})$$

We can now draw the thermal activation curves which are shown in figure 1-2. and result from the more useful relation between time and temperature given by Pulliah et.al. (1975) as,

$$(T_2 \log C t_2) / (J_s(T_2) H_k(T_2)) = (T_1 \log C t_1) / (J_s(T_1) H_k(T_1)) \quad [5]$$

This allows for changes in the spontaneous magnetization and coercivity as the temperature changes. This is critical as the $J_s(T)$ variation will dominate blocking mechanisms as the Curie temperature is approached. The shape of the curves can be used as a guide to give an approximate relation between time and temperature.

1.3 UNBLOCKING OF NRM

At temperatures below the Curie temperature the magnetic behaviour of a rock may be described by the thermal activation theory of Neel (1949). In an attempt to measure the effects of this magnetization, Pulliah et.al.(1975) performed experiments on samples of Matachewan diabase. The magnetic carrier in the samples was magnetite with blocking temperatures of 550°C to

600°C. Pulliah et.al. (1975) found that heating to a temperature of 450°C for 10 days, was sufficient to completely remove the magnetization of these samples. The thermal activation curves predict that an NRM with a laboratory blocking temperature of 550°C would withstand heatings up to 535°C for 10 days. Magnetization carried by hematite, with laboratory blocking temperatures of 600°C to 670°C, was unblocked by heatings to 530°C for 10 days. Again this was lower than the temperature of 550°C for 10 days predicted using thermal activation curves for hematite.

In both cases the unblocking temperature predicted by theory was higher than the experimentally observed temperature. The magnetization was less resistant to thermal demagnetization, suggesting that the modification of remanence in a rock, during metamorphism may be greater than predicted by the thermal activation curves. As an example of this effect suppose that a metamorphic event lasts about 10^6 years. An initial remanence with $T_b = 570^\circ\text{C}$, for laboratory times, should remain in disequilibrium with a weak applied field during heating to temperatures up to 555°C (according to Pulliah et.al's (1975) curves). This means that the NRM should persist through greenschist facies metamorphism, which spans temperatures of 300°C to 500°C (Turner and Verhoogen, 1960). If the experimental results of Pulliah et.al. (1975) can be extrapolated to geologic time, they imply that the NRM will survive temperatures only up to 385°C for 10^6 years. This is equivalent to only middle greenschist metamorphism. If hematite (with blocking temperatures of 670°C, for laboratory times), carries the NRM,

the results of Pulliah et.al.(1975) can be extrapolated to 10^6 years with T_b 's of about 450°C . These results indicate that the initial NRM of a rock will likely be reset by a metamorphic event any more intense than lower greenschist facies metamorphism.

1.4 BLOCKING OF MAGNETIZATION

Pulliah et.al.(1975) also preformed storage tests on other samples of Matachewan diabase and investigated the temperatures required to demagnetize the remanence of a sample acquired during storage for a specified period of time. A sample stored for 10 days at 400°C should aquire a viscous magnetization. From the thermal activation curves one can see that this VRM should be able to withstand heating to about 450°C for twenty minutes. Upon thermal demagnetization, blocking temperatures as high as 550°C were observed by Pulliah et.al. (1975).

Hale (1985) preformed similiar experiments on synthetic samples of magnetite dispersed in a matrix of kaolin. The samples were originally demagnetized then stored in vacuum at a temperature of 400°C in an ambient field strength of 5×10^{-3} T, for one month. According to the thermal activation curves a sample should acquire a magnetization with blocking temperatures no greater than 485°C for even unreasonably short heatings. Blocking temperatures as high as the Curie temperature ($T_c = 585^{\circ}$) were observed in thermal demagnetization experiments.

Pulliah et.al. (1975) found that samples with blocking temperatures of 550°C to 600°C for laboratory times were unblocked by heating to 450°C for 10 days. According to the

thermal activation curves, the diabase should withstand heating to 535°C for 10 days. The blocking temperatures predicted by the thermal activation curves do not agree with the blocking temperatures observed in the blocking experiment. The results of Pulliah et.al. (1975) show that unexpectedly high blocking temperatures can be accessed during storage. The low temperatures required to reset a pre-existing NRM thus might be a result of the same effect which permitted the acquisition of VRM with higher than expected blocking temperatures. Results of both experiments by Pulliah et.al.(1975) and by Hale(1985) do not agree with theory, the results are, however, consistent.

1.5 CHEMICAL MAGNETIZATION

Pulliah et.al(1975) in part attributed the conflict between theory and experiment to chemical changes in the diabase. Stable CRM can result during subdivision of large unstable grains into smaller more stable grains. Also, recrystallization may result in changes of internal stress, changes in shape, changes in J_s , and changes in magnetic interactions (McCelland-Brown, 1982), all of which are important in determining T_b .

The stable CRM can also be described by equ. [3] where J_s and H_k are properties of the new mineral and the critical volume for blocking of the CRM at the temperature of formation will be determined by,

$$vJ_s(T_b)H_k(T_b)=25kT \quad [6]$$

This implies that grains smaller than v will be unblocked, while grains larger than v will be blocked, for times equal to t . If the CRM is due to the growth of fine grained hematite, the

blocking temperatures would be expected to be higher than those of magnetite since hematite has a higher Curie temperature ($T_c = 670^\circ\text{C}$).

Baily and Hale(1981) performed experiments in which CRM was induced in cores of oceanic basalts from the Deep Sea Drilling Project. The principle magnetic carrier of these samples is titanomaghemite which "oxyexsolves", upon heating to 250°C , to intergrowths of magnetite, which becomes the principle magnetic carrier, and ilmenite. This will result in an acquired CRM component which can be studied using thermal demagnetization experiments.

The effects caused by the initial NRM and those caused by the applied field may be separated by heating, in vacuum, one sample which is initially demagnetized and one which has a moderately strong remanence. Results of the thermal demagnetization experiments, of weakly magnetized samples, by Baily and Hale (1981) suggest that the direction of the resulting CRM will depend on both the NRM and the ambient field when stored in weak fields ($<.5 \times 10^{-4}$ T) and will be a stable single component which may not lie in the direction of either the original NRM or the ambient field. Samples with original NRM stored in stronger ambient fields, acquired a CRM in the ambient field direction and did not preserve the original NRM direction.

The experimental results given earlier by Pulliah et.al.(1975) do not agree with Neel's theory, nor do the results of Hale(1985). The explanation of chemical alteration put forth by Pulliah et.al. (1975) as a reason for the variance from

theory, cannot explain Hale's (1985) results since these samples were heated in vacuum and did not alter significantly.

1.6 THERMAL ACTIVATION THEORY WITH AN ASSUMED GRAIN VOLUME DISTRIBUTION

Walton (1980) has proposed a thermal activation theory where the magnetization is integrated over all volumes and all anisotropy constants, which may reconcile the discrepancy reported by Pulliah et.al. (1975) and Hale (1985). If the volume distribution is assumed to be log normal, (which is often but not always the case in nature), the time-temperature relation becomes,

$$(T_1 J_s^2(T_2)) / (T_2 J_s^2(T_1)) = (\ln(Ct_1)) / (\ln Ct_2) \quad [7]$$

Given a particular choice of grain volume distribution this can yield results which are more in agreement with the experimental results of Pulliah et.al.(1975). For instance, over laboratory times blocking temperatures of 550°C are observed in material which according to Pulliah et.al.'s (1975) curves should not withstand temperatures greater than 450°C for even unreasonably short heatings. Using the results of Walton (1980) the maximum expected laboratory blocking temperature increases to 500°C which is closer to the observed results of Pulliah et.al.(1975) than the theory of Dunlop (1973). Walton's (1980) theory assumes a grain size distribution. If however a different distribution is assumed the exact answer can be produced. It then becomes a matter of justifying any assumptions of grain size distribution. This is almost never possible and Pulliah et al.'s (1975) formulation is more general.

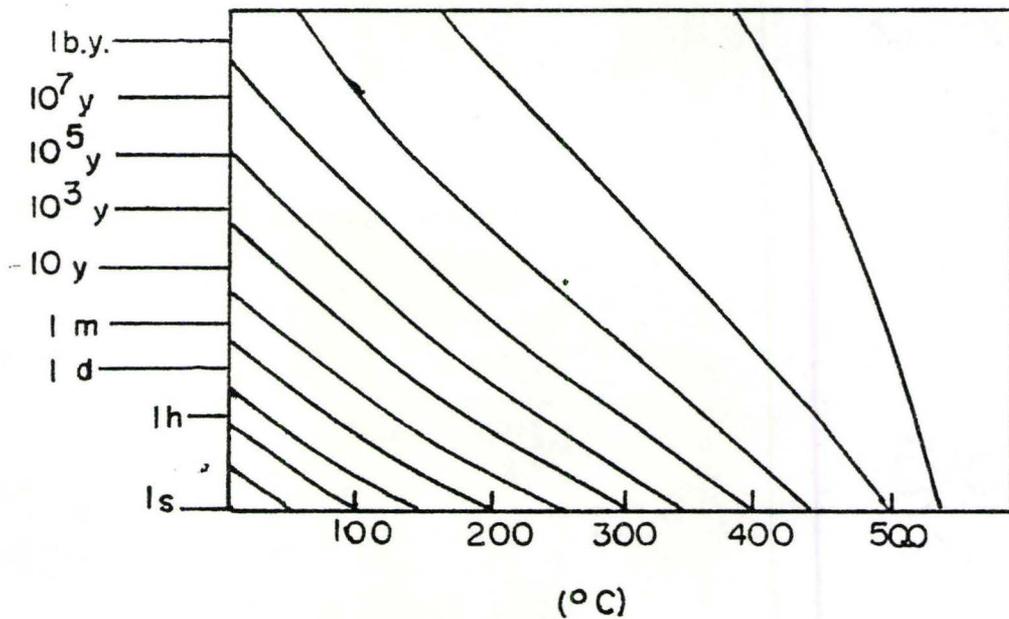


Fig 1-3 Thermal activation curves for magnetite after Schmidt and Middleton, (1982). These curves are like those of Pulliah et. al. (1975) except that a log normal distribution of grain size has been assumed.

Schmidt and Middleton (1982) have also calculated time-temperature curves for a log normal distribution of grain size and these are shown in figure 1-3. They have subsequently been used by Kent (1985) to study the magnetization of Devonian limestones in New York State. The magnetization of these limestones occurs with two distinct components. There is a stable component with blocking temperatures as high as 500°C in 45 minute heatings. There is also a less stable component with blocking temperatures of only 275°C , in 45 minute heatings.

The stable component found in these rocks is characterized by Harris et.al. (1978) to have undergone metamorphism equivalent to heating to 255°C for 10^8 years. The rock then remained at near surface conditions. The low temperature component is also found to be present in conglomerates which were deposited by glaciers 10,000 years ago, Kent (1985). These conglomerates are originally derived from the Devonian limestones and by implication the secondary VRM in the limestone was also acquired over 10,000 years since the glaciation.

The stable component was originally thought to be due to thermal remagnetization of magnetite during burial. Since its blocking temperatures are higher than those predicted by Pulliah et.al. (1975) as 400°C for 45 minutes it seemed that the remanence was not due to the acquisition of VRM. Using the thermal activation curves of Schmidt and Middleton (1982) the blocking temperature becomes 490°C for 45 minute heatings, closer to the observed blocking temperature of 500°C for 45 minute heatings. The VRM acquired in surface conditions for 10,000 years would have blocking temperatures of 240°C for heatings lasting 45 minutes,

closer to the observed 325°C , than the results using the results of Pulliah et.al. (1975) which are 165°C for 45 minutes. The curves of Schmidt et.al. (1982) approximate the experimental data more closely than Pulliah et.al.'s (1975) curves. But Schmidt and Middleton's (1982) curves are only appropriate for material which has a log normal distribution about a single grain size. The curves would have to be modified not only for different material but even for a different grain size distribution, and are thus not generally applicable.

1.7 SUMMARY

Néel (1949) proposed a theoretical approach to the stability of magnetization subjected to randomizing thermal energy. This was applied in experiments by Pulliah et.al.(1975) to diabase dykes carrying an initial NRM. The results found, disagree with the theory. The purpose of the present study is to reconcile theory with observed experimental results.

Since determining the thermal activation curves requires that certain assumptions be made about the magnetic properties of the material, the curves may not describe the temperature behaviour perfectly. Experimentation must deal with the possibility that chemical changes introduce high blocking temperatures due to CRM. Walton (1980) has shown that one of the causes of the disagreement may be due to more than one effective grain size present in the sample. This is a major limitation to the applicability of Walton's (1980), and Pulliah et.al's(1975) theory in a general way since each sample can be expected to have

a unique distribution of grain sizes and determining the actual distribution is difficult.

Careful studies of the different magnetic properties, and how they are affected by chemical changes were undertaken to determine the relative roles of time, temperature and chemical change, in the blocking of remanence in fine grained magnetite. Samples were stored both in air and in vacuum, to allow the separation of blocking of stable CRM components from components due to VRM. Samples with an initial NRM were simulated by inducing a moderate TRM. Samples with a weak NRM (low field TRM) were also studied. This was done to help in determining the role of NRM in the acquisition of VRM. Of the samples containing an initial NRM half were stored with the direction of the NRM perpendicular to the applied field while half were stored with the initial NRM anti-parallel to the applied field. By doing this the effects of ambient field direction may be determined. The effects of time on the acquisition of VRM and CRM were studied by storing the samples for increasingly longer amounts of time.

CHAPTER TWO

THE APPROACH FOLLOWED TO SIMULATE BURIAL METAMORPHISM

2.0 SAMPLE PREPARATION

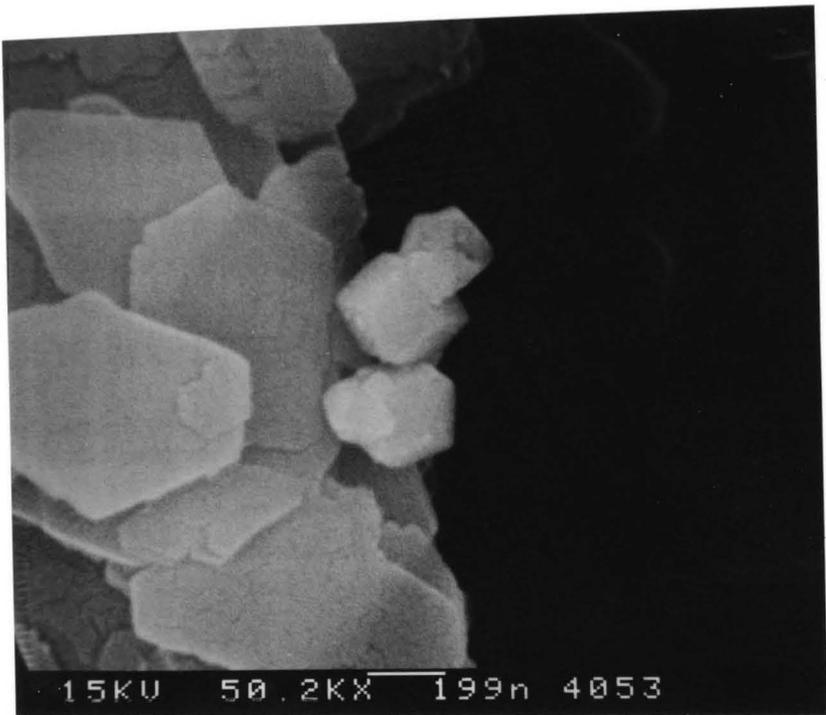
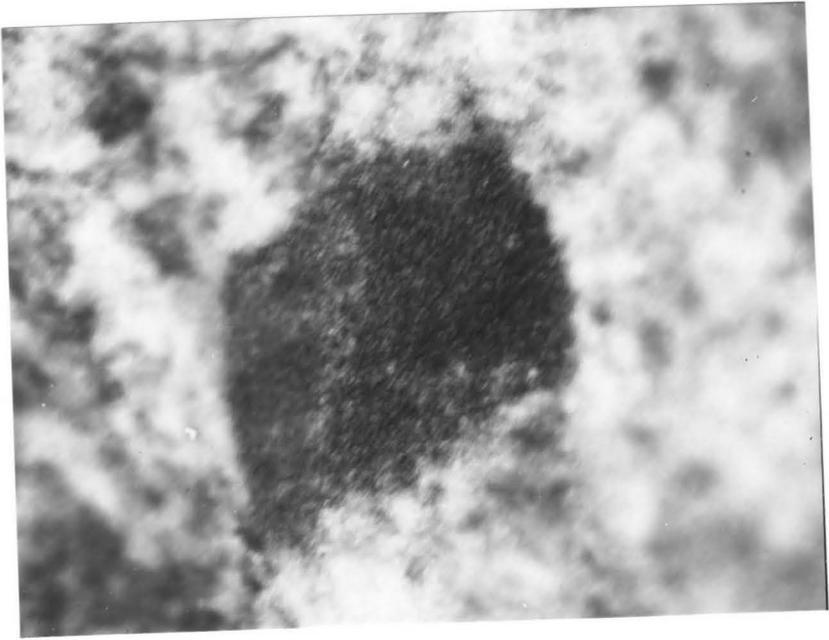
Synthetic samples were made by mixing kaolin with magnetite powder. Three concentrations were made, 0.5%, 1.0%, 2.0%, magnetite by weight. The actual concentrations varied within each group by less than 1%. This mixture was then pressed into samples 1 cm.(o.d.) by 1 cm in length. To ensure that normalization to weight would later be possible, the mass of each sample was measured after drying. Drying was achieved by placing the samples in a dessicator, which was evacuated through a cold trap of liquid nitrogen, to a pressure of 10 mTorr. The dessicator was heated by an infrared lamp to a temperature of less than 50 °C for periods ranging up to 12 hours. An average of 1 cc. of ice was removed from the cold trap after drying each group of approximately 50 samples.

2.1 GRAIN SIZE AND CONCENTRATION HOMOGENEITY DETERMINATION

In an attempt to determine the degree of dispersion between the kaolin and magnetite the samples were examined under a reflecting light microscope. Plate 2-1a, is a photograph of the surface of a 2% sample which was heated at 400°C for twelve hours, in color the grains show deep red internal reflections indicating the presence of hematite. The aggregate (center of photo) is .15 mm in diameter, and appears to be formed from

Fig 2-1 a) Surface of a sample heated for twelve hours at a temperature of 400°C. The surfaces of the dark grains are hematitized and show deep red internal reflections. The magnification is x300. The dark mineral occurs as aggregates and in trails throughout the matrix.

b) Scanning electron microscope image of the initial magnetite. The magnification is x50,000. Note the scale bar indicating 199×10^{-9} m. The magnetite grains show a distinct cubic shape in contrast to the platy hexagonal outline of the kaoline.



grains which are about one thousandth the size of the aggregate giving them a diameter of a few tenths of a micrometer.

The size of an individual grain can be better estimated using a scanning electron microscope (SEM). Plate 2-1b), is a photograph from a 2% concentration sample before any storage. The general shape of the cubic magnetite grain distinguishes it from the kaolin which has a clearly more platy and hexagonal appearance than the equant magnetite grains which are perched on the edge of a kaolin aggregate. The size of these grains is roughly 0.2 micrometers.

The determination of overall grain morphology is important to the results of the experiment. If the particles are not behaving as single-domain (SD) or pseudo-single-domain (PSD) grains the magnetization can not necessarily be expected to behave as predicted by single domain theory. True SD particles will have $J_{rs} = 1/2J_s$, while for multidomain (MD) grains J_{rs} decreases as grain size increases (Dunlop, 1980). The coercivity of SD particles also approaches the maximum microscopic coercive force, but is much less than this for MD grains (Dunlop, 1980).

PSD grains have J_s and H_c properties similar to SD grains even they are not true single domain particles and contain domain walls. Single domain behavior of magnetite (Dickson et.al., 1966 and Dunlop, 1973) occurs in a very narrow range from about 0.025 micrometers to about 0.05 micrometers and for hematite the band is much broader ranging from 0.025 to 10.0 micrometers grain size. In magnetite PSD behaviour is evident in a broad range of grain size from 0.05 to 15.0 micrometers. The grain size of the magnetite used in the present study is within

the range required for PSD behaviour.

The size of the grains can also be estimated by using hysteresis properties. Several samples from each of the various groups were measured on a vibrating sample magnetometer to determine: saturation magnetization (J_s), remanant saturation magnetization (J_{rs}), coercive force (H_c), remanent coercive force (H_{rc}), and initial susceptibility (X_i). In rocks containing PSD grains the J_{rs}/J_s ratio should be between 0.5 and 0.1 and H_{rc}/H_c between 1.0 and 4.0 (Dunlop, 1980). This is approximately the value obtained for the synthetic samples, for both ratios. Figure 2-2 a) shows the relation between J_{rs}/J_s vs. H_{rc}/H_c which plots in a field similar to that found for submarine intrusive rocks and other natural rocks which contain fine grained magnetite (Dunlop and Prevot, 1981), (Day et.al, 1977), figure 2-2 b).

2.2 INDUCING TRM AND THE EFFECTS OF HEATING

TRM was induced in the samples by heating in a vacuum furnace, to 600°C well above the Curie temperature of magnetite ($T_c=570^{\circ}\text{C}$), and then cooling to room temperature in the presence of a magnetic field. To ensure the grains are in equilibrium with the applied field, the field was turned on for the entire heating and cooling process, and the temperature was held above 600°C for at least twenty minutes. The furnace was evacuated to a residual pressure of about 100 mTorr to prevent the oxidation of magnetite to hematite. As a further precaution the samples were placed in a copper box which removed the remaining oxygen, by surface oxidation of the copper.

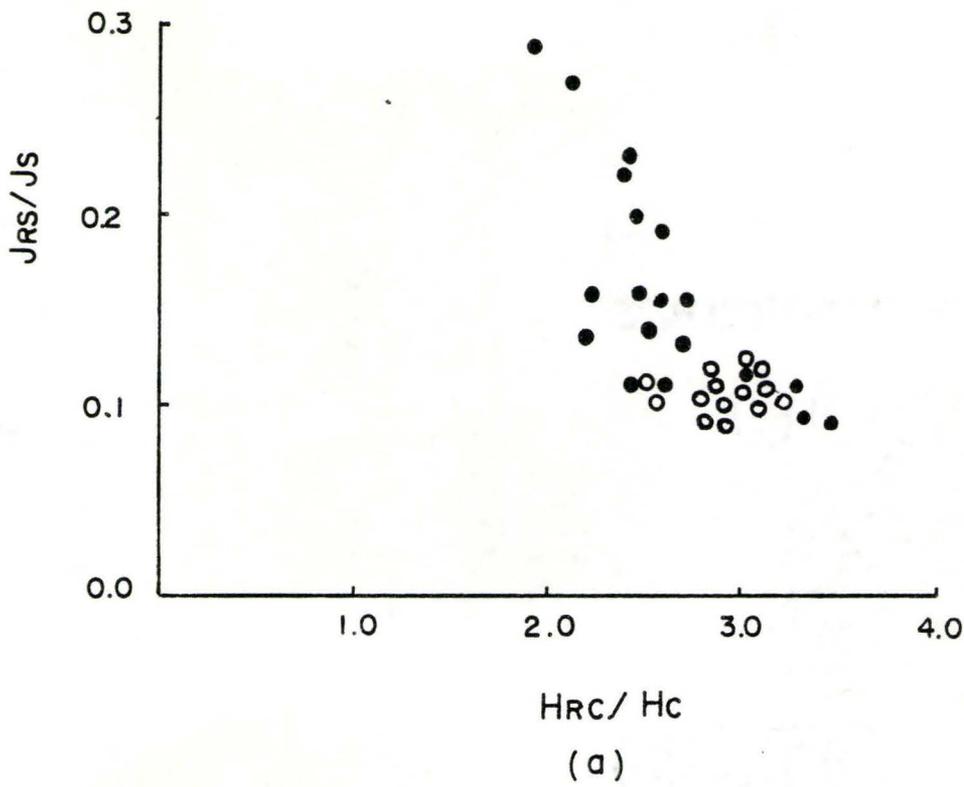
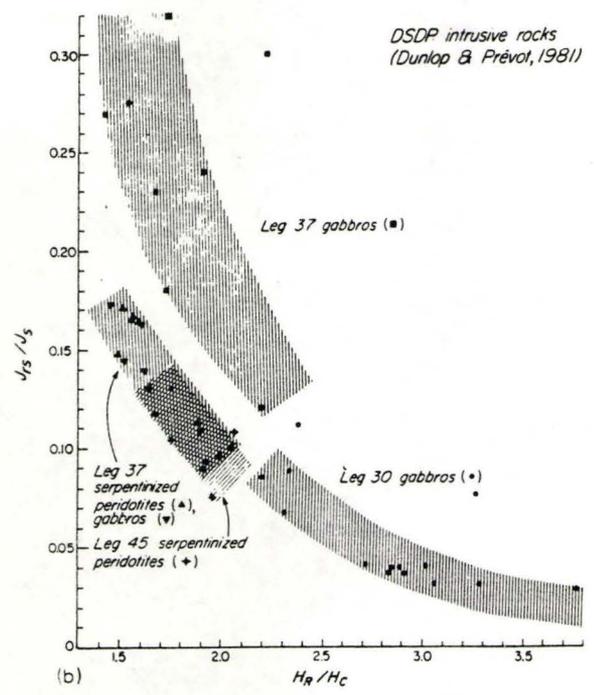


Fig. 2-2 a) H_{RC}/H_C vs. J_{RS}/J_S for unheated samples (closed circles) and samples after TRM acquisition (open circles). These data plot in a localized region after the first heating in vacuum above 600°C .

b) The synthetic sample data plot in a region similar to natural rocks described by Day et al. (1977) and Dunlop and Prevot (1981).

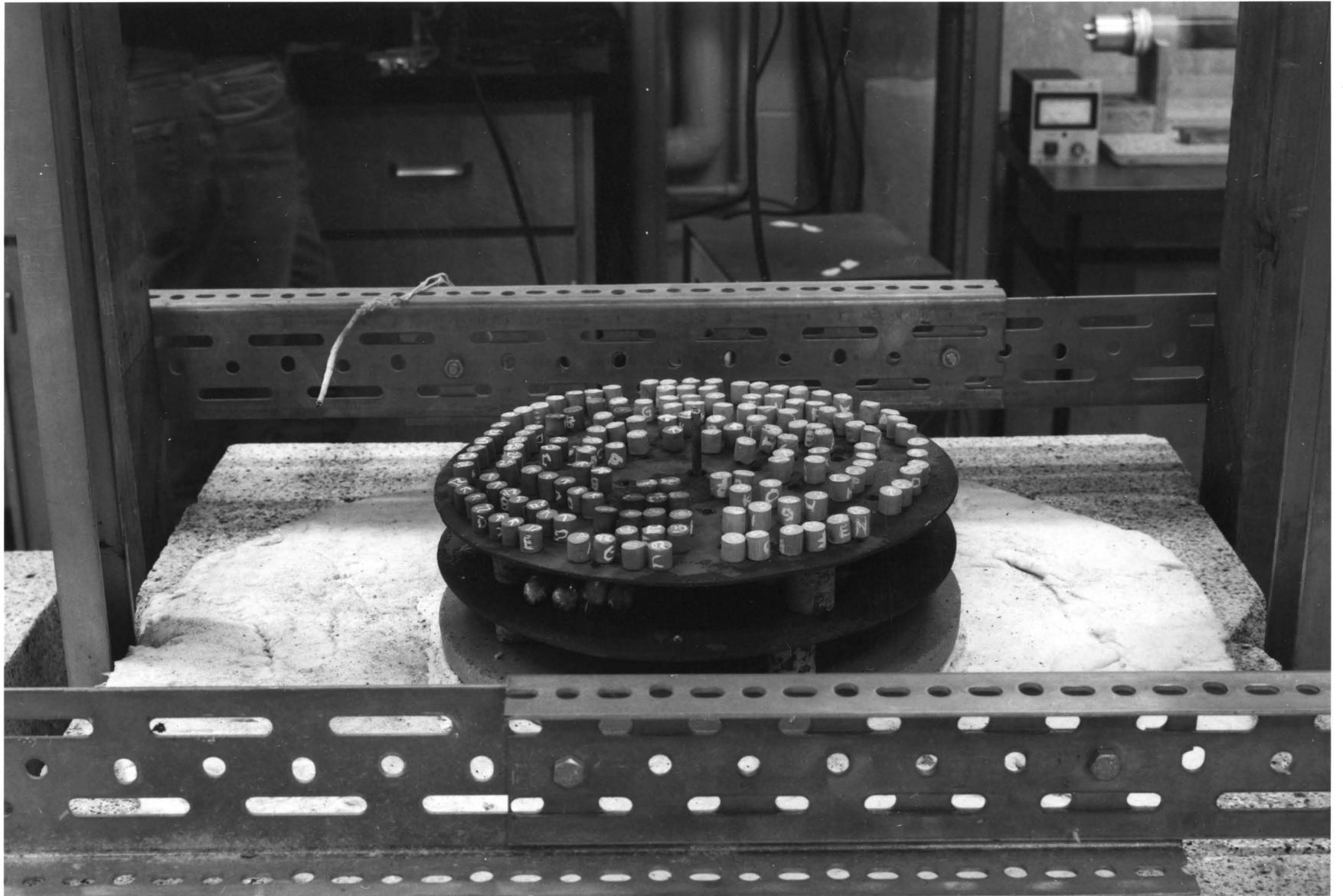


It is a straight forward matter to detect the oxidation of magnetite. First, a visual inspection will be sufficient to distinguish a color change due to the presence of hematite which gives a pink hue to the sample. This is however not a very reliable method as vacuum deposition of copper on the sample surface can also produce a pinkish hue.

A more reliable method of determining the degree of oxidation is to consider the effects on hysteresis parameters. Since hematite has a J_s value of about 2 emu/gram and magnetite has a J_s value equal to 485 emu/gram the relative difference will mean that for practical purposes any decrease in the value of J_s , measured will be a direct measure of the amount of magnetite altered to hematite. The ratio of J_{rs}/J_s when plotted against H_{rc}/H_c plotted as in figure 2-2 a) can also give an indication of any change in the bulk magnetic properties of the material. Figure 2-2a) shows a strong tendency for all the samples to move to a very narrow region at about J_{rs}/J_s of about 0.1 and H_{rc}/H_c of about 3.0. Later it will be necessary to compare samples which have remained in vacuum throughout the experiment to samples which were heated in air. To this end samples were heated in a vacuum of about 100 mTorr to 400°C to drive off any remaining water and then sealed under a high vacuum in a quartz tube.

TRM was induced in the samples in three different ways. TRM along the axis, in 65 samples, was induced by standing the sample in a vertical field. The applied field, (measured on a Hewlett Packard model 428B dc. fluxgate magnetometer) was about $(1.0 \pm .05) \times 10^{-4}$ T. Another 65 samples were oriented with the

Fig 2-3 Placement of samples before storage. The furnace is raised to allow quick removal of the samples. When the furnace is lowered it fits tightly around the sample support. The samples are placed on a copper plate to distribute the heat evenly and holes allow convection to further distribute heat.



long axis perpendicular to the applied field. Finally 90 samples were magnetized in a very weak applied field, typically 200 ± 50 gammas. In each case the field was controlled by a set of three axis Helmholtz coils with the vacuum furnace in the centre of the coils. The field was measured, over the area of the furnace in which the samples are heated, and found to be uniform (± 25 gammas). The NRM produced was measured on a Schonsedt spinner magnetometer model SSM-2 which was interfaced with a Texas Instruments micro-computer.

2.3 STORAGE

To simulate the effects of burial metamorphism, the samples were stored at 400°C for periods ranging from half a day to thirty-two days. The samples were divided into groups based on concentration and direction of remanence with respect to the ambient field. The direction of the remanence was recorded by orienting an arrow on the sample with respect to the ambient magnetic field. The horizontal samples were stored such that the direction of magnetization lay in the horizontal plane but they were not aligned in the same azimuthal direction. The vertical samples were placed so that their remanence was opposed to the applied field which was about 30 degrees from vertical. The samples which were sealed in quartz were also stored with their remanence opposing the applied field. Plate 2-3 shows the arrangement of samples before heating. All the samples are standing up vertically and placed on a copper plate with small holes through it to allow convection which redistributes heat.

The copper plates also distribute heat since they are good conductors.

The samples were heated in a non-inductive furnace (Plate 2-4) connected to a Hewlett Packard model 240 temperature programmer. The temperature inside the furnace was continuously monitored by a platinum-rhodium thermocouple connected to a strip-chart recorder. Between removals of samples, the temperature varied by no more than 1°C .

Samples were removed after 1/2, 1, 2, 4, 8, 16, 32, day(s) with two samples of each concentration and each type of magnetization being removed, a total of eighteen samples with each removal. After 1, 8, 16, 32, day(s) two sealed samples were removed along with the others. These removals required the furnace be raised and the samples exposed to the room temperature. Although the samples were removed as quickly as possible the entire procedure required about two minutes. During this time the temperature of the furnace was maintained by closing the cavity off with a 1/2 cm. sheet of asbestos. After the furnace was relowered the temperature of 400°C (inside the furnace) could be recovered in less than fifteen minutes and the furnace temperature stabilized in about an hour. There were temperature overshoots associated with the first couple of removals but never more than 5°C for more than 1 minute.

It is important that the temperature of the samples does not drop below 400°C since such a cooling will introduce partial TRM which will make interpretation more difficult. This means that it is critical the samples be placed in zero field conditions as quickly as possible after removal from the furnace. To

Fig 2-4 Non-inductive furnace (used to heat samples during storage before being closed over the samples.



facilitate this each sample was removed from the furnace with a pair of aluminium tongs and immediately placed inside a magnetic shield with a residual field of not more than 100 nT and stored there for at least 12 hours before being removed for measurement.

2.4 THERMAL DEMAGNETIZATION

Progressive thermal demagnetization (TDM) was carried out in steps of 20°C to 40°C to examine the blocking temperature spectra of the samples stored for up to 32 days. Each demagnetization step began with first preheating the power supplies to keep drift to a minimum. Also the furnace was heated prior to inserting the samples to reduce the time required to heat the samples to the desired temperature. It generally required 1/2 hour for the samples to reach thermal equilibrium. They remained at this temperature for twenty minutes to ensure thorough heating. After this time the furnace was removed, allowing the samples to cool to room temperature in about 3/4 of an hour.

It is possible to determine the component of magnetization removed during the demagnetization step by measuring the magnetization remaining after each heating and differencing this with the initial remanence. After several successive heating the blocking temperature spectrum for the remanence can be seen since the magnetizations with blocking temperatures lower than the temperature of the demagnetization step are eliminated. The magnetizations with the lowest blocking temperatures will be removed first and demagnetization proceeds at progressively higher temperatures until only the highest blocking temperatures

remain. In fine grained, PSD samples carrying TRM this should be very close to the Curie temperature.

During the demagnetization of two groups of samples, the field was controlled with a three axis helmhotz coil and the samples were heated in the same vacuum furnace used to induce the initial TRM. For a third group of samples the thermal demagnetization was carried out using a Schonstedt thermal demagnetizer. The Schonstedt instrument was used because the residual field in the vacuum furnace may have been as large as several hundred gammas due to remanence of the furnace. This introduces one further complication. Since the Schonstedt demagnetizer does not have the capability to preform the heatings in vacuum, but the other two demagnetizations were done in vacuum, comparisons must be drawn more carefully. The results from the samples demagnetized in air may provide results significantly different from those from samples demagnetized in vacuum and the difference may be advantageous.

The first group of samples was heated to 400°C directly, with no intermediate steps. This was done to save time since the lowest useful blocking temperature must be 400°C from which the samples were cooled in a null field. Since up to 30% of the initial magnetization was removed by 400°C , the second thermal demagnetization was done with two additional steps: one at 260°C and one at 350°C .

CHAPTER THREE
RESULTS OF THE SIMULATION EXPERIMENT

3.0 RESULTS OF HYSTERESIS MEASUREMENTS

Hysteresis measurements can provide an indication of changes in the characteristics of a magnetic material. Changes in hysteresis properties thus were monitored by measurements taken at various times during storage. Figure 3-1a,b, show the results of H_c and H_{rc} measurements made before TRM acquisition, after TRM acquisition, and at each removal time during storage. Averages can be calculated since at least 5 samples were measured at the pre-TRM and post-TRM stage, in each concentration group, and three samples representing each concentration group were measured at each removal time. The values plotted for initial and post-TRM acquisition measurements are the mean and the error bars indicate the width of one standard deviation.

During TRM acquisition H_{rc} and H_c changed significantly, from the initial values. The initial values show significant deviation, (20%) within all three concentration groups, as well as a wide range between groups, (100×10^{-4} to 200×10^{-4} T). After TRM acquisition H_{rc} and H_c have smaller deviations in all groups and there is no difference between concentration groups, to within the error shown. Heating to a temperature greater than 600°C in vacuum, standardized the coercivity of all samples before the storage experiment.

H_{rc} and H_c are also plotted as functions of the length of time during which they were heated at 400°C in air. Although starting H_c and H_{rc} values were similar in all samples, the post storage values plot over a wider range. The initial value of

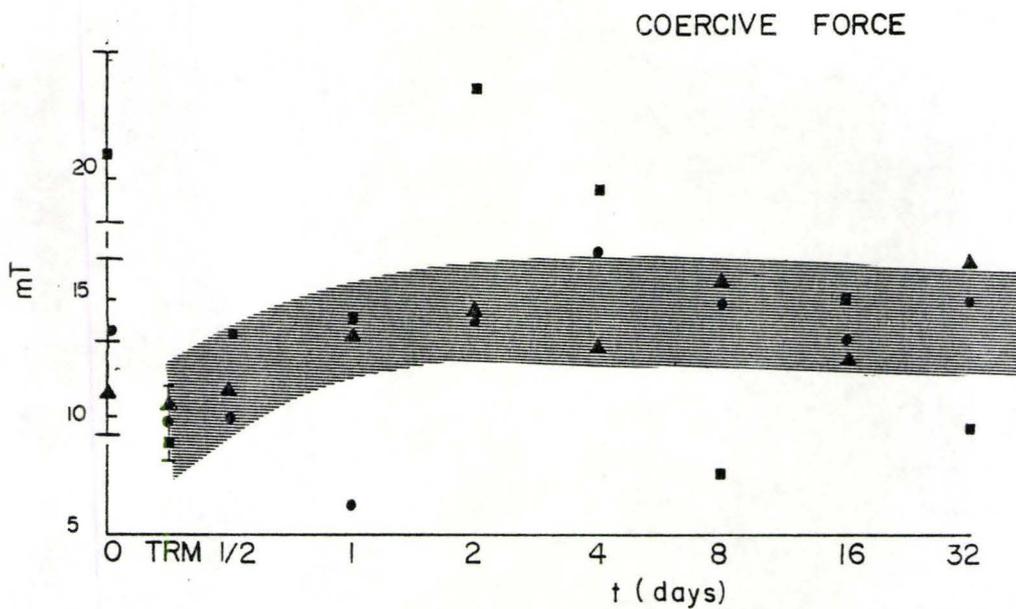
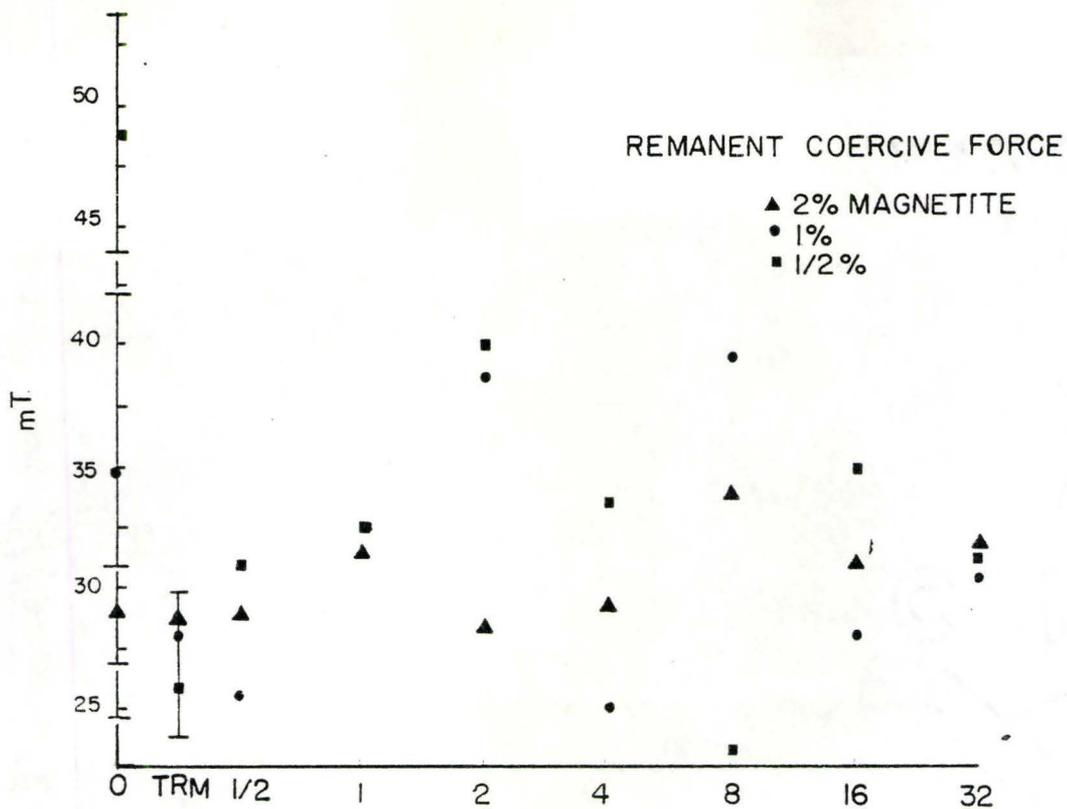


Fig 3-1 a,b Coercivity and remanent coercivity changes during heating for acquisition of TRM. H_c and H_{rc} increase slightly during the first few heatings but then remain roughly constant throughout the storage.

H_c is approximately 100×10^{-4} T, H_c increases after one day storage to an average value of 145×10^{-4} T. for the remaining 32 days. This indicates a degree of "hardening" of the material during the first day of storage. The maximum H_c value is still well below the 2000×10^{-4} T coercive force expected if the magnetite were oxidized to hematite (Dunlop 1971). The change of 50×10^{-4} T seems small in comparison and a more likely explanation is that oxidation of the surface of the grains has tended to reduce the effective grain volume.

J_s and J_{rs} are shown in figure 3-2a,b. Since J_s and J_{rs} depend on the amount of material present, each value has been normalized to account for differences in weight and values are given in Am^{-1} per gram. This normalization assumes a uniform magnetite concentration from sample to sample within each group. Some of the variation observed in values plotted may result from sample inhomogeneity. In spite of this difficulty it appears as though J_s and J_{rs} are reduced, as much as 1/2 in some instances, from the original value. A reduction of J_s results when magnetite ($J_s = 485 \times 10^3 \text{ Am}^{-1}/\text{gram}$) alters to maghemite ($J_s = 435 \times 10^3 \text{ Am}^{-1}/\text{gram}$) and is even greater when fully oxidized to hematite ($J_s = 2 \times 10^3 \text{ Am}^{-1}/\text{gram}$). The decrease in the value of J_s is largest in samples with initial concentration equal to 2% magnetite, J_s values are relatively unchanged in lower concentration groups with only a small decrease in J_s during storage. The erratic behaviour of the more concentrated samples may be due to a greater tendency for inhomogeneities to exist since higher concentrations may tend to form aggregates. Since J_s is much too large for hematite, not all the material has been

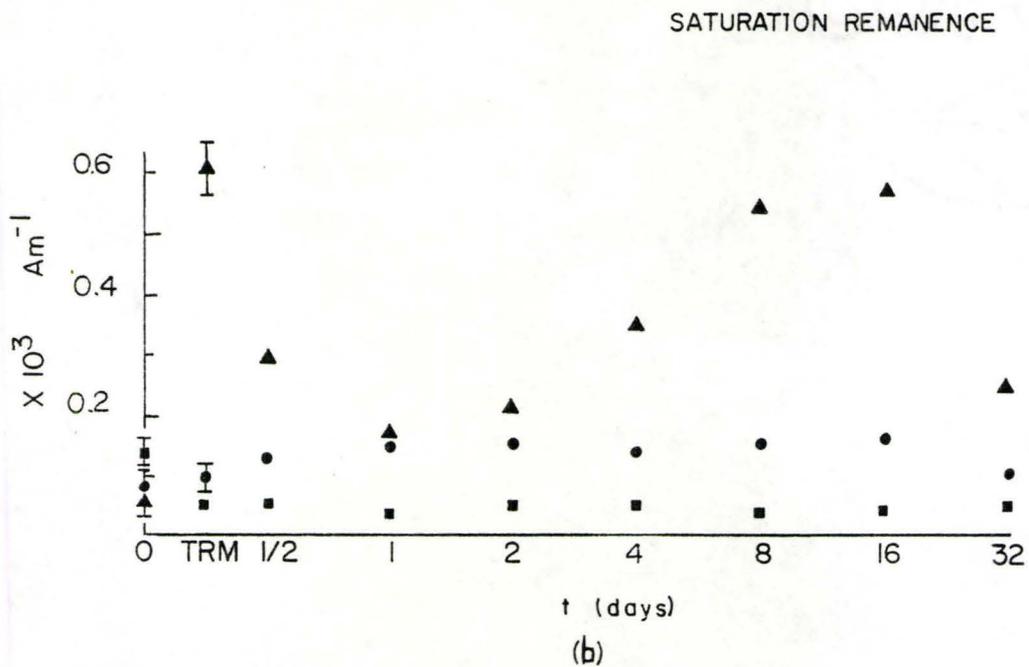
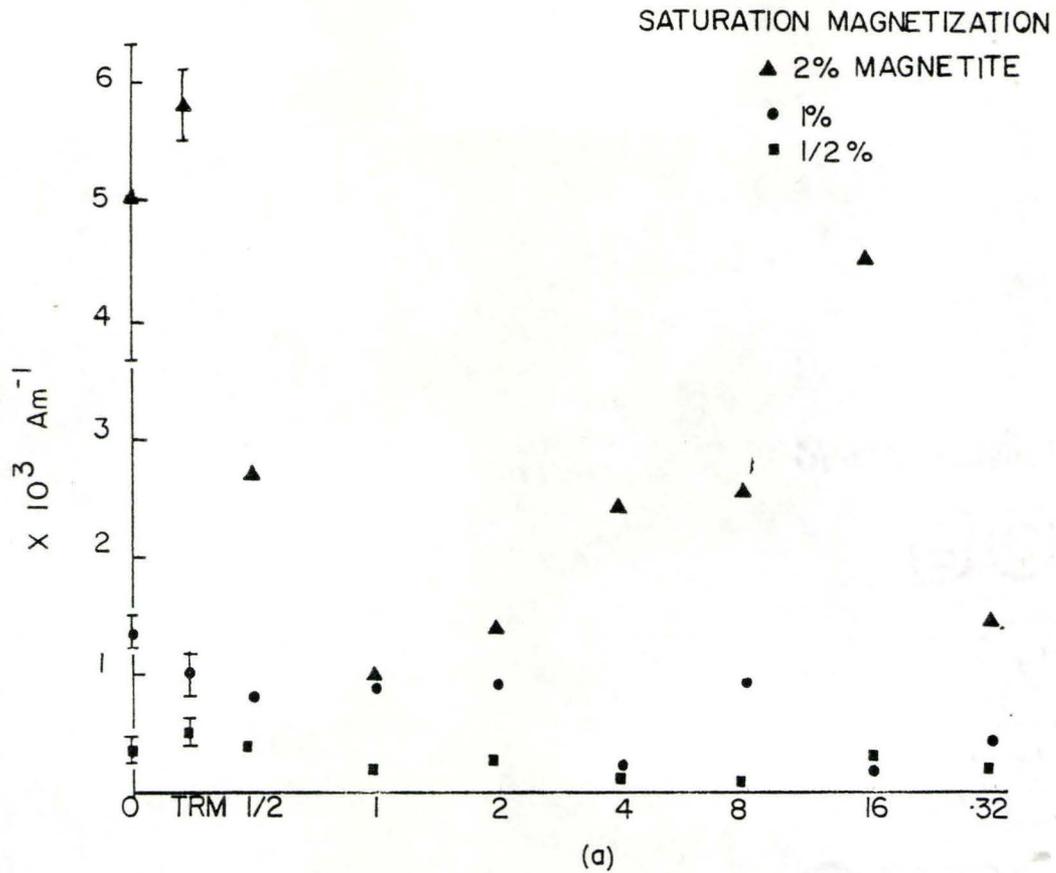


Fig 3-2 a) The saturation magnetization in 1% and 1/2% initial concentration samples decreases only slightly during storage. 2% concentration samples have a decrease in J_s by as much as 1/2. b) Saturation remanence increases slightly during storage.

fully oxidized to hematite by storage and some amount of magnetite or possibly maghemite remains.

The ratios J_{rs}/J_s and H_{rc}/H_c are shown in figure 3-3. Changes in J_{rs}/J_s and H_{rc}/H_c do not appear to depend on the length of time the samples have been stored. The storage results in increasing the range of both ratios from a tightly grouped value after TRM acquisition to a range similar to the deviation measured in the initial material. By plotting J_{rs}/J_s vs. H_{rc}/H_c (figure 3-4) and comparing with natural rocks (figure 2-2b) and initial samples (figure 2-2a), storage appears to have broadened the field in which the ratios plot and returned to a range measured in initial samples and natural rocks.

3.1 SATURATION MAGNETIZATION AS A FUNCTION OF TEMPERATURE AND COERCIVITY RESULTS

The thermomagnetic curve for a sample stored in air at 400°C for 32 days is shown in figure 3-5. The sample was heated in air, so that possibly some continued oxidation occurred during the measurement. The thermo-magnetic curves indicate that there is only one magnetic phase present. This material has a Curie temperature of 630°C after storage for 32 days. The measured T_c is between the Curie temperatures of hematite ($T_c=675^\circ\text{C}$) and magnetite ($T_c=585^\circ\text{C}$). The high Curie temperature may be explained by cation deficiency in the magnetite lattice, brought about by the oxidation of the initial material during storage. Upon cooling, the thermomagnetic curve shows a significantly lower J_s . This indicates an apparent loss of about 1/2 of the material carrying the magnetization. A possible

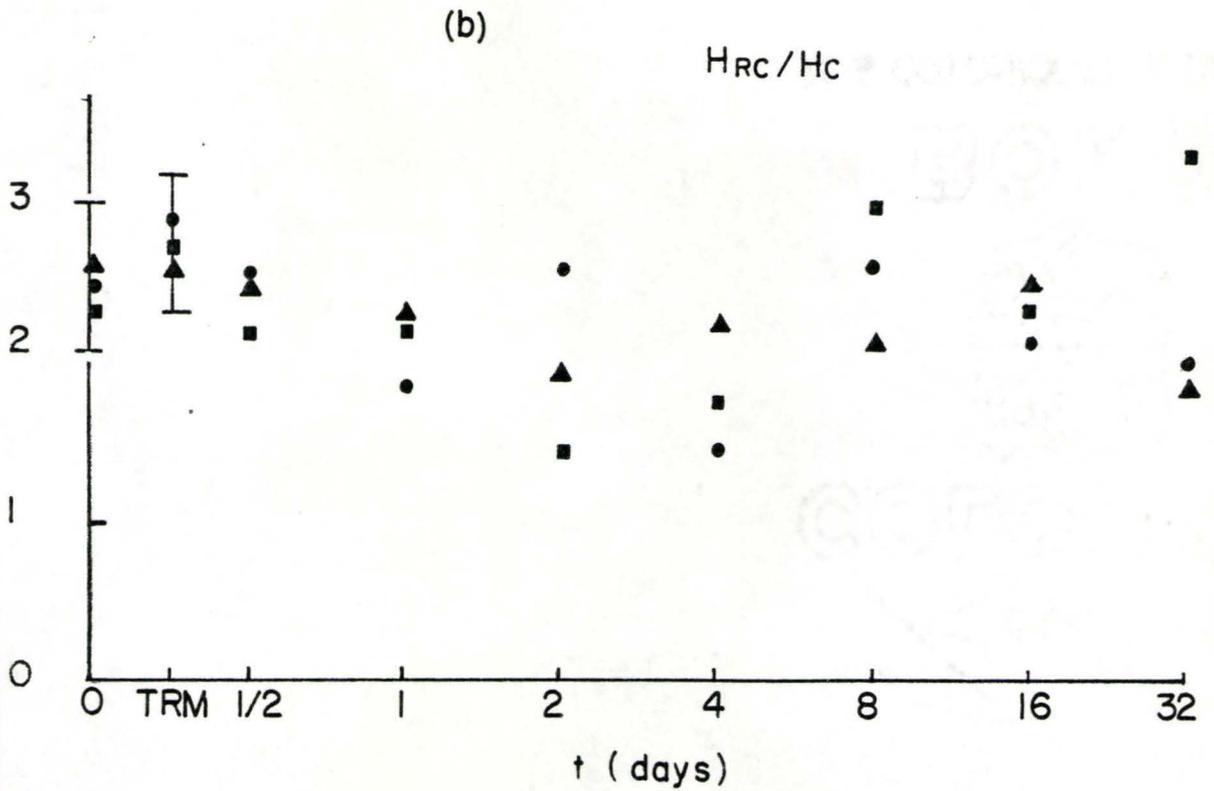
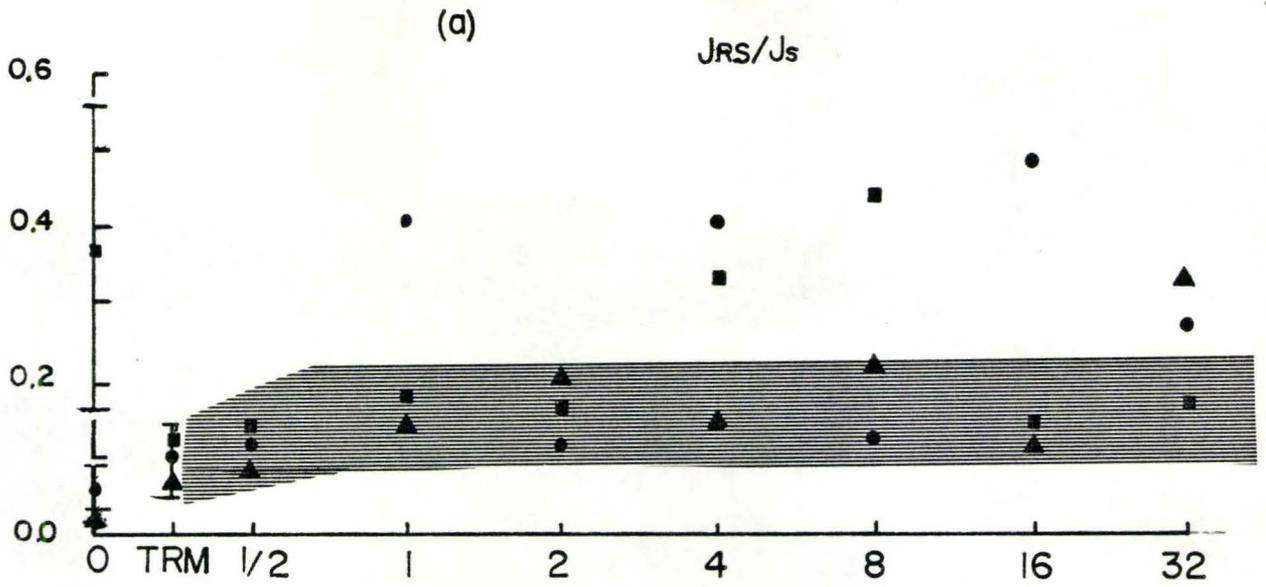
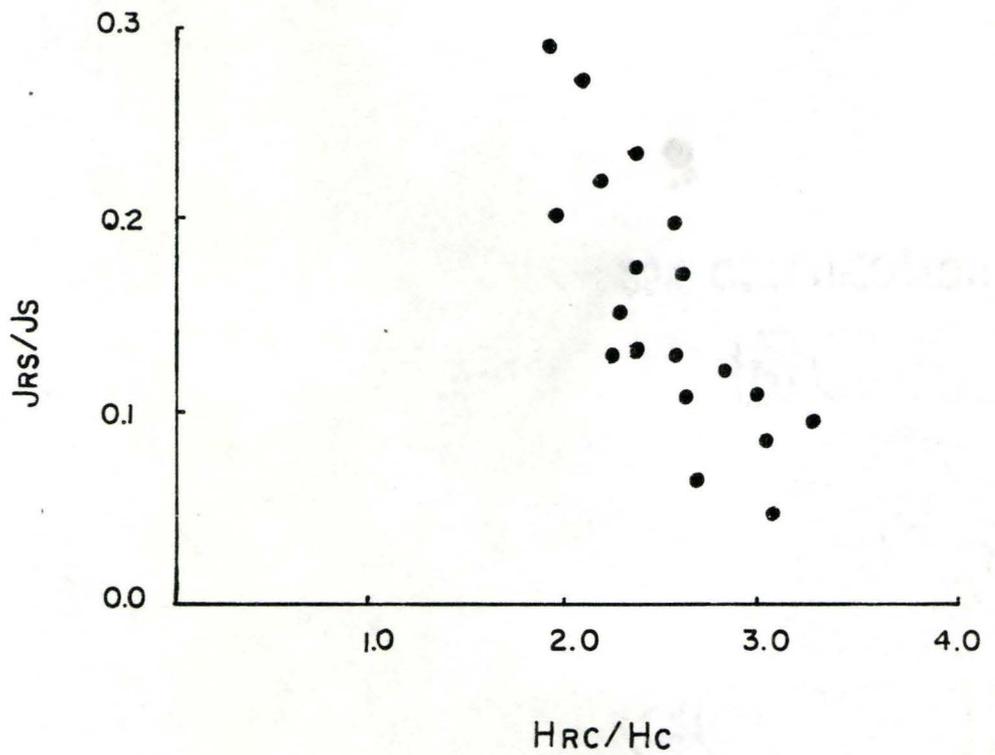


Fig 3-3 a) J_{rs}/J_s and b) H_{rc}/H_c both tighter grouping after TRM acquisition. Storage causes the range of values of J_{rs}/J_s to increase.

Fig 3-4 J_{rs}/J_s vs. H_{rc}/H_c after storage. The range of values widens from values after TRM acquisition. refer to figure 2-2a,b



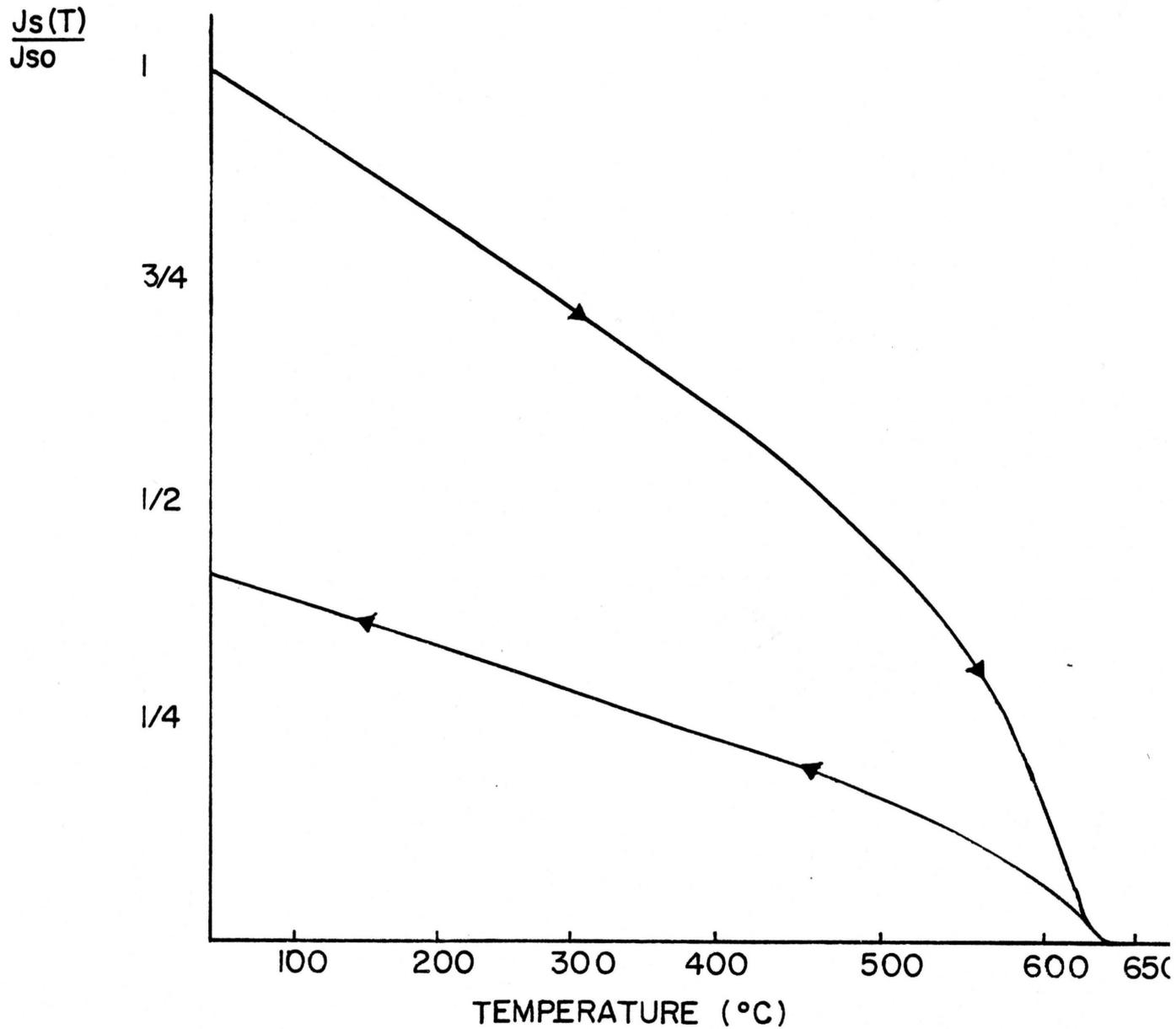


Fig 3-5 Thermomagnetic curve of a magnetite bearing synthetic sample after stored at 400°C for 32 days. The indicated Curie temperature is 640°C . The decrease in J_s during thermomagnetic analysis probably results from hematization of cation deficient magnetite at high temperature.

mechanism to explain the loss in J_s is that heating in air has caused further oxidation and inversion to hematite. This has such a small J_s ($2 \times 10^3 \text{ Am}^{-1}/\text{gram}$) value that when the cation deficient magnetite is oxidized to hematite the magnetization is virtually lost. The lack of any measurable J_s at temperatures above 640°C but still below the Curie temperature of hematite indicates that the amount of magnetization carried by hematite is too small to be measured, compared to the large magnetization of the cation deficient magnetite.

Alternating field demagnetization provides a broad method of distinguishing remanence carried by a material with a large coercivity or a "hard" material such as hematite from a remanence carried by a material with a low coercivity or a "soft" material such as magnetite. Figure 3-6 is the normalized total intensity, which remains after demagnetization, plotted as a function of the demagnetizing field strength, for remanence carried in a sample after TRM acquisition, and in samples stored for 2 days and 8 days. After application of an alternating field with a strength of $400 \times 10^{-4} \text{ T}$., less than 20% of the original remanence remains. Also shown is the result of the AF demagnetization of 0.22 micrometre magnetite (Dunlop 1981).

The initial sample has a coercivity much the same as the 0.22 micrometre magnetite of Dunlop (1981). With increased storage time the coercivity seems to increase indicating the material is becoming "harder". This might be due to continued oxidation of the magnetite during storage which results in a decrease in grain size or an increase in the coercive force. The median demagnetizing field increases from about 0.18 T for

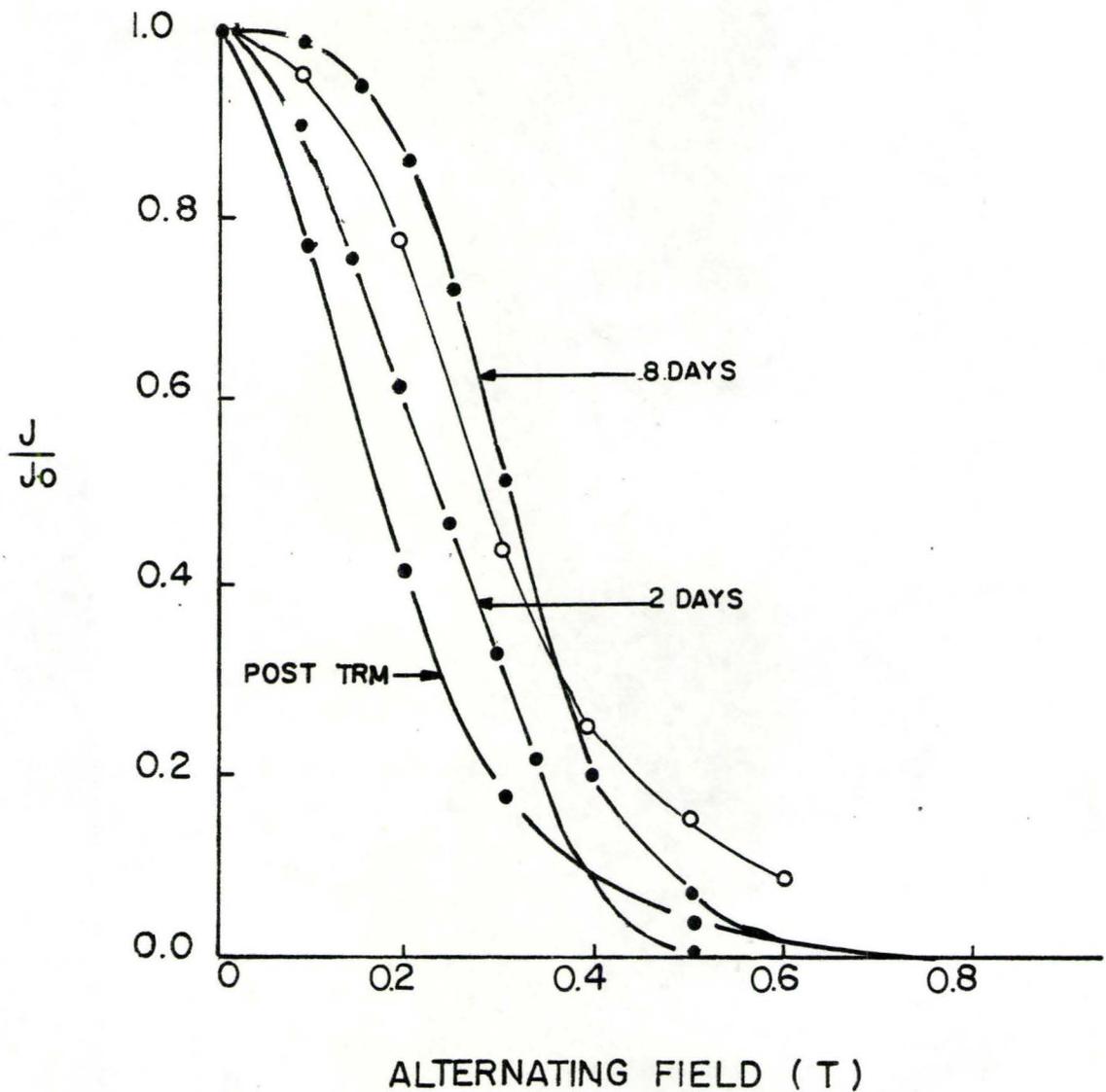


Fig 3-6 AF demagnetization curves obtained after: TRM acquisition, 2 days storage, 8 days storage. Dunlop's (1981) demagnetization curve for 0.22×10^{-6} m magnetite is also shown for comparison.

samples with initial TRM only to 0.24 T after 2 days storage and 0.33 T after 8 days storage. The median demagnetizing field is about 0.3 T for 0.22 micrometer magnetite (Dunlop, 1981) which is similar to the values for samples stored at 400°C.

3.2 VISCOUS MAGNETIZATION OF SAMPLES SEALED IN VACUUM

Magnetic viscosity is a measure of a material's ability to acquire a remanence, at a constant temperature, in a low magnetic field, with time. This phenomenon was first investigated by Thellier (1938) with the result that the time decay of an initial NRM is a linear function of $\log t$. Change in magnetization may be caused by decay of NRM or acquisition of VRM in the ambient field direction. In the storage experiment the initial NRM in some samples opposed the ambient field and in some samples the initial NRM was perpendicular to the ambient field. The NRM was a total TRM acquired by cooling from above the Curie temperature. Magnetization is blocked in individual grains as the temperature drops below T_b for each grain. Differences in T_b will depend mostly on the distribution of grain volumes. The grains with low T_b will be the most susceptible to acquiring low temperature VRM.

Figure 3-7 shows the amount of remanence lost from samples stored in vacuum for 1 to 32 days in a $0.53 \pm .01 \times 10^{-4}$ T field. Since the field was applied antiparallel to the remanence, there is no directional change due to components acquired in the field direction rather, viscous demagnetization of TRM and viscous magnetization both tend to reduce the initial remanence, (figure 3-8). The change in the total intensity after storage becomes larger with increasing time. After 32 days the total change is

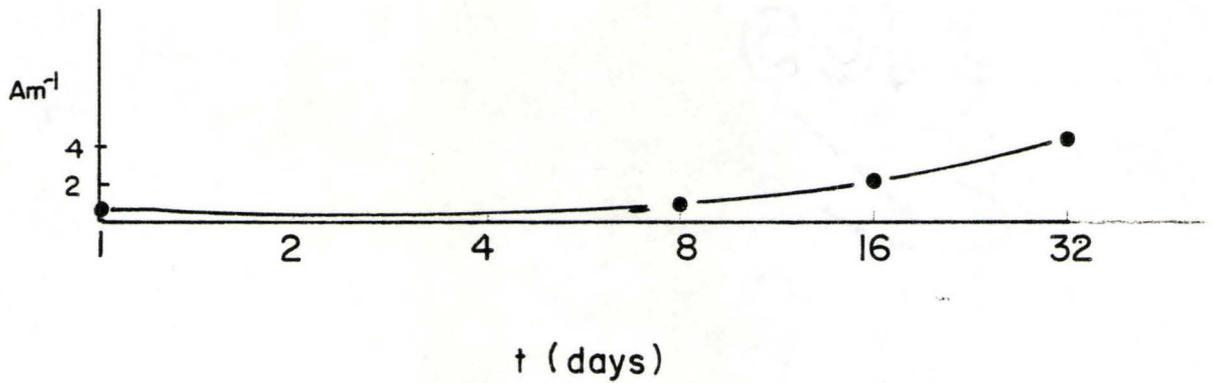


Fig 3-7 Total change in remanence plotted as a function of time. The remanence is modified after only 1/2 day storage followed by a gradual increase in the modification of the remanence.

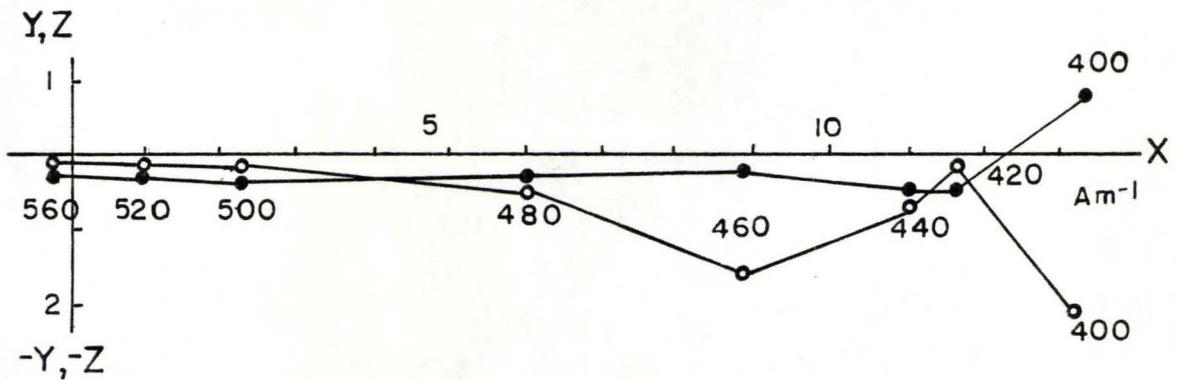


Fig 3-8 Zijderveld plot of a sample with initial remanence perpendicular to the applied field, during thermal demagnetization. (closed circles are projections of the remanence in the horizontal plane (x,y), open circles are in the vertical plane (x,z). The numbers indicate the temperature in degrees C. After an initially vertical segment is removed the remanence direction is constant.

$0.004 \times 10^3 \text{ Am}^{-1}$ and even after only one day the change is $0.001 \times 10^3 \text{ Am}^{-1}$. The decay of NRM appears to be significant even after very short storage times. The observed decrease in remanence normalized to the initial NRM varies depending on the initial magnetization. Normalized remanence in samples with a weak NRM is 0.4% to 0.5% and 0.1% to 0.2% in samples with a strong NRM.

The changes in the remanence of the samples stored in vacuum must be due to VRM since no significant chemical changes have occurred. Using these results as an indication of the behaviour of VRM and comparing with the samples stored in air (chemical reactions may occur), it may be possible to distinguish behaviour which is VRM and behaviour which is CRM.

3.3 CHANGES DURING STORAGE OF SAMPLES WITH INITIAL TRM'S HEATED IN AIR

Samples were oriented during storage so that the initial remanences lay in two different directions relative to the ambient field. Half were stored with remanence antiparallel to the ambient field and half were stored with remanence perpendicular to the ambient field. Figure (3-9) shows the position of the remanence before and after storage in samples with remanence perpendicular and anti-parallel to the ambient field. In both cases the remanence remains stable in the initial direction with only small random changes observed. It appears the direction of the field does not affect the way the direction of the magnetization changes during storage if the initial TRM is large.

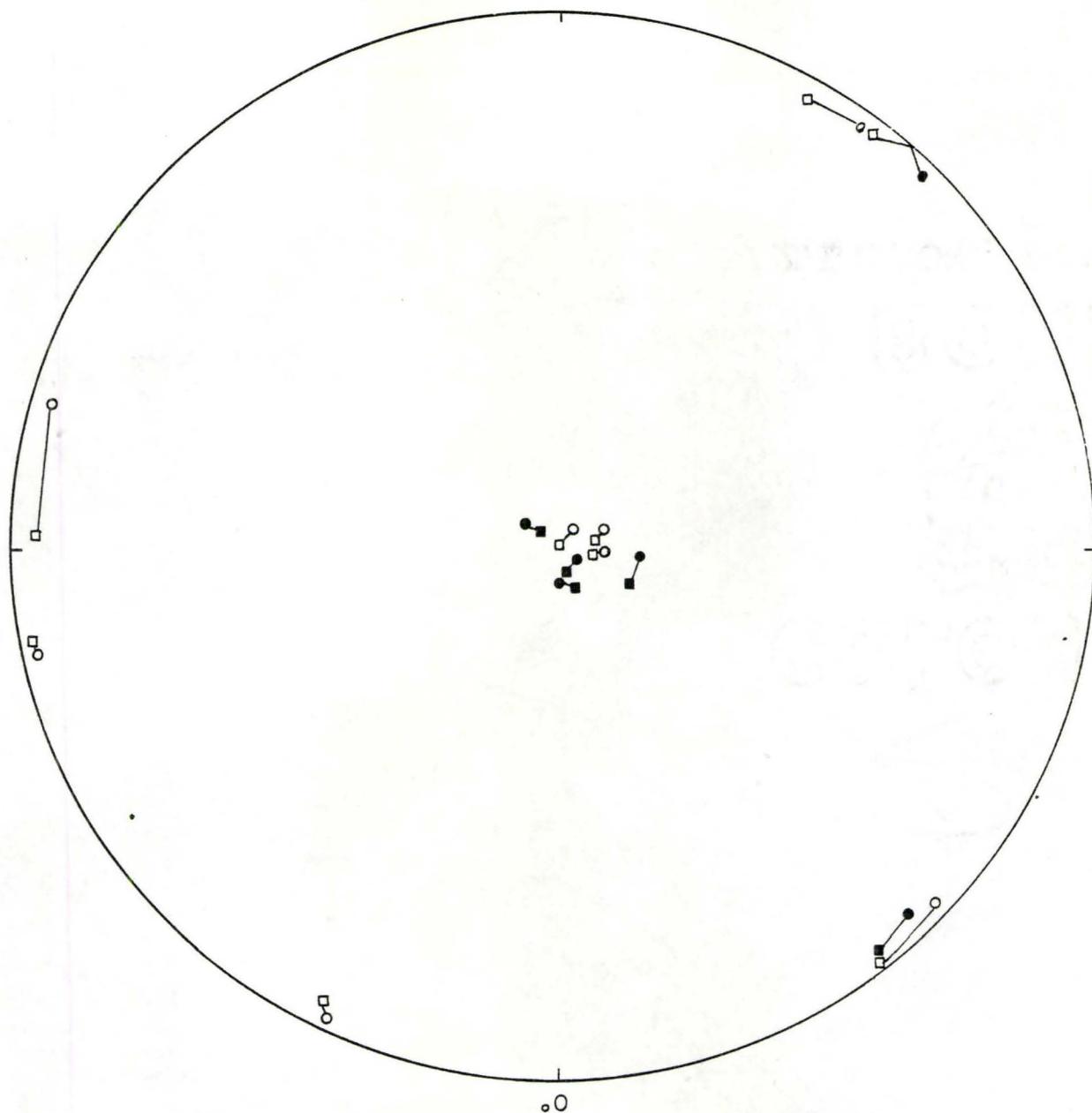


Fig 3-9 Stereonet projection of the remanence direction, before storage and after storage in samples with a strong initial NRM. The position before storage is shown by a circle, the position after storage by a square. Solid symbols are plotted on the lower hemisphere open symbols are on the upper hemisphere. Sample with initial remanence perpendicular to the applied field plot along the primitive and samples with remanence initially parallel to the field plot at the pole. In both cases the post-storage remanence reproduces the NRM direction.

Since little directional change was observed a comparison of total intensities is sufficient to characterize the magnitude of the change in remanence. Figure 3-10 shows the results of the difference between the initial TRM magnitude and the magnitude of the remanence after storage. Each point represents seven samples and the error bar indicates the range of magnitudes after storage. The change in magnitude of the remanence appears to depend on the concentration of magnetite in the initial sample.

The change in remanence during storage normalized to the initial remanence, gives an indication of the relative stability of the remanence. Figure 3-11 is a plot of $(J_{\text{storage}} - J_{\text{initial}})/J_{\text{initial}}$. The amount of change is about 1/4 the NRM magnitude. This change does not depend on the length of time the sample is heated, up to 32 days. This result is similar to the results for the vacuum samples which are, i) the modification of NRM happens in a short time, ii) displacement of the remanence direction is small. The similarity between samples which did not alter chemically and samples which altered chemically indicates the change in remanence was due to VRM.

3.4 CHANGES DURING STORAGE OF SAMPLES WITH WEAK INITIAL NRM HEATED IN AIR

Samples with an initial NRM less than 0.5 Am^{-1} total intensity behaved differently from samples with greater NRM. Figure 3-12 shows the position of initial remanence before and after storage. The samples were oriented so that the remanences were not initially in the same direction. After storage the

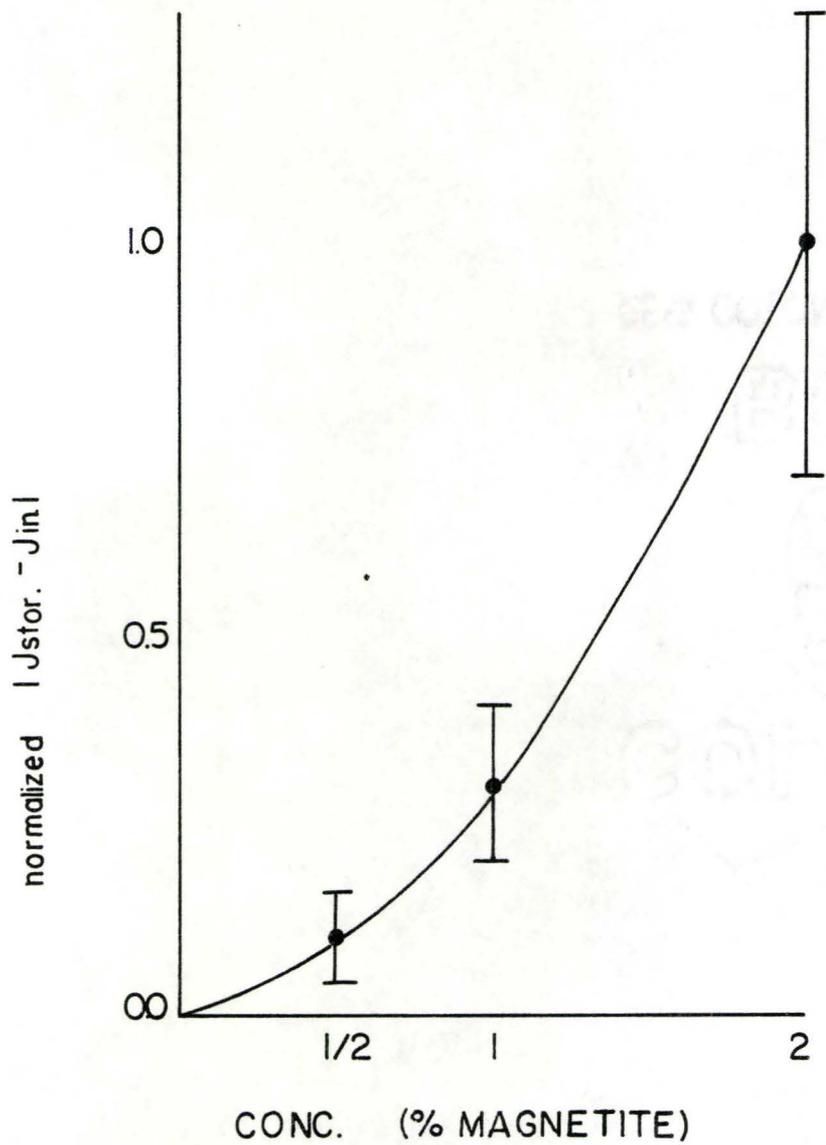


Fig. 3-10 Change in magnetization as a function of initial concentration. Seven samples were measured for each point and the error bars are included.

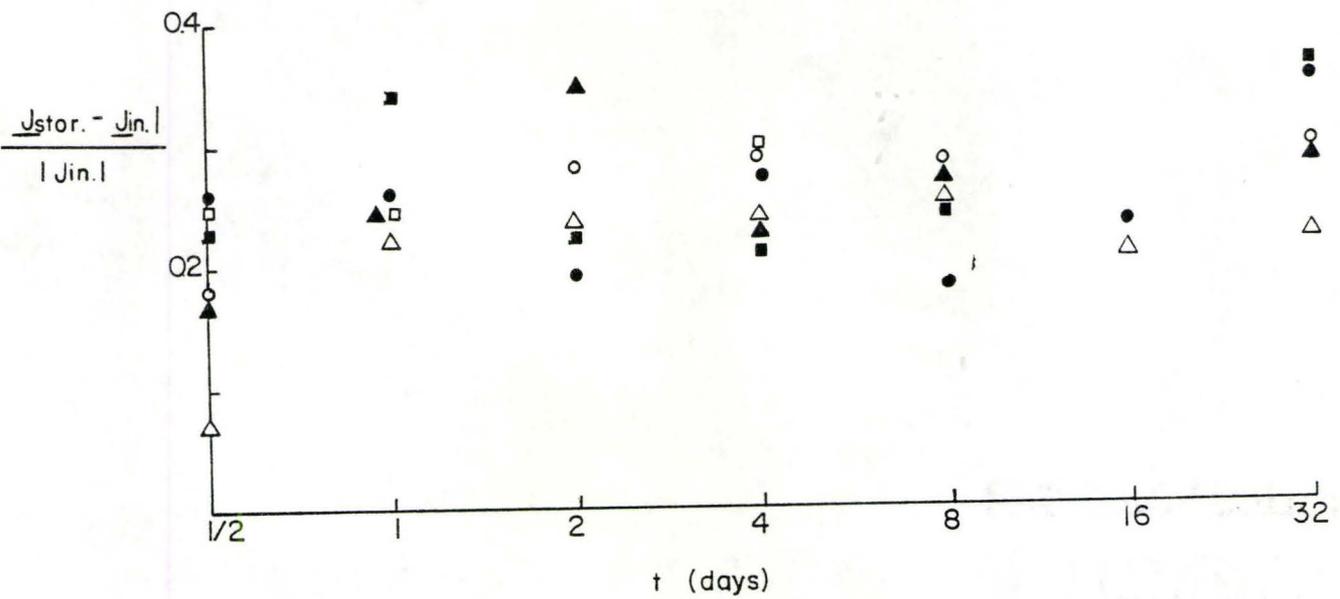


Fig3-11 Change in remanence normalized to the initial remanence. The change in remanence is apparently constant during storage.

triangles - 2% open symbol for vertical NRM
 circles - 1% closed symbol for horizontal NRM
 squares - 1/2%

direction of all the samples moves to lie near the field direction (Dec.=180° Inc.=59°). Even samples stored for 1/2 a day show significant movement of the remanence to align in the field. This behavior of acquiring a remanence in the field direction is typical of VRM (Nagata 1961). Bailey and Hale (1981) have also shown it to be typical of CRM in samples with weak remanence and fields greater than 0.5×10^{-4} T.

The magnitude of the remanence increases in all cases during storage. Figure 3-13 shows the intensity of NRM before storage and after storage at each removal time. The initial intensity varies from sample to sample but is always less than 0.5 Am^{-1} . With the exception of the sample stored for 32 days the magnitude of the remanence after storage always increases to the same intensity. It appears that the magnitude of the post storage remanence lies in the direction of the field (and so cannot be due to PTRM acquired during cooling) and has a constant magnitude which is independent of the initial magnitude or direction. The major distinction between samples with an initial remanence and samples with no initial remanence is that those samples with no initial remanence acquire remanence in the field direction while samples with an initial remanence do not acquire a remanence in the field direction.

3.5 THERMAL DEMAGNETIZATION OF SAMPLES WITH INITIAL NRM

Samples with an initial NRM were demagnetized in vacuum and in air. By demagnetizing samples with an initial NRM the blocking temperature of the initial NRM can be determined. Then the effects of VRM with lower blocking temperatures can be

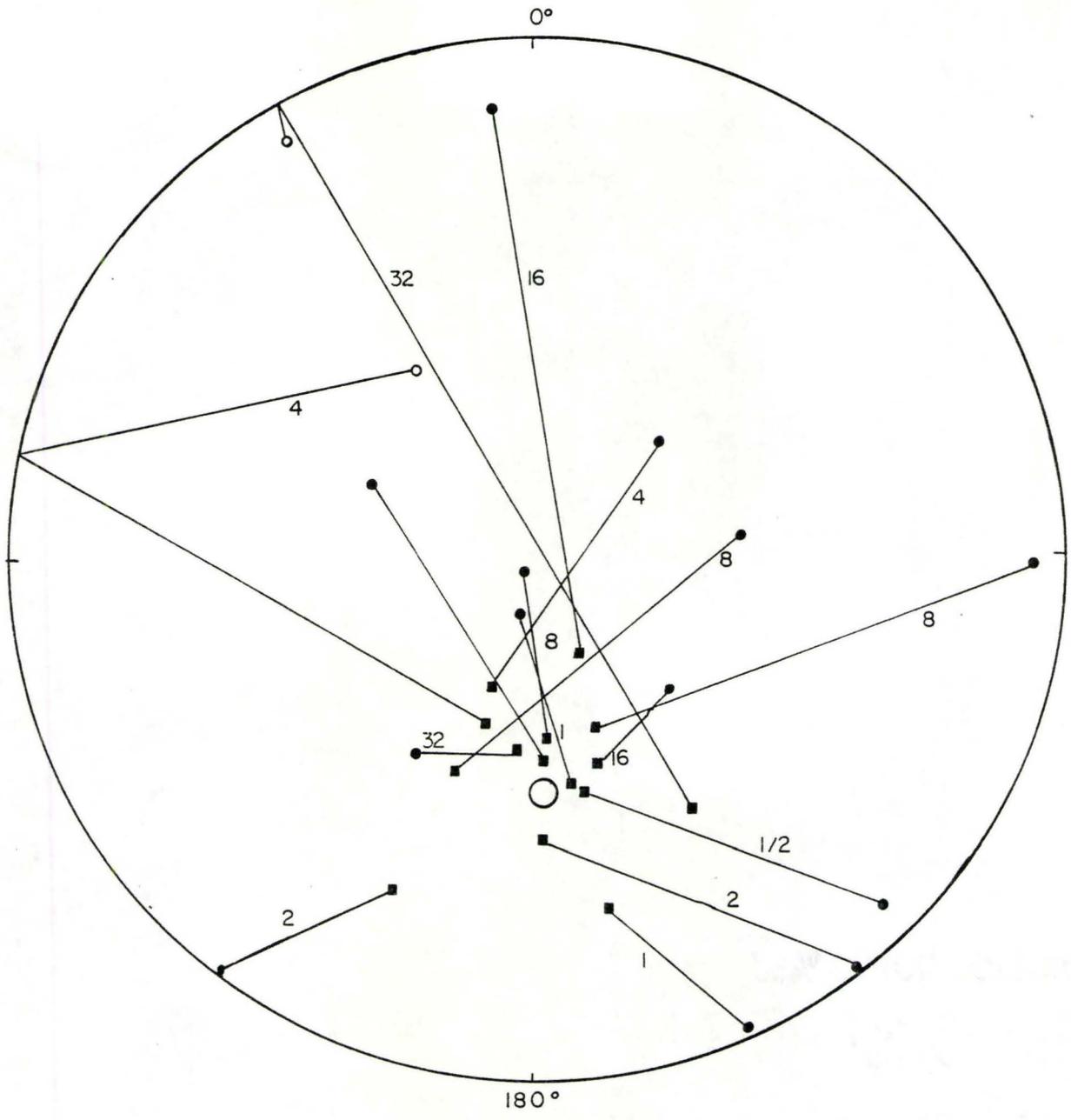


Fig 3-12 Direction of remanence before and after storage of samples with weak initial NRM. The direction before storage is shown as a circle and the direction after storage is shown as a square (solid symbols are in the lower hemisphere, open symbols are in the upper hemisphere). After storage all of the directions cluster near the ambient field direction (0).

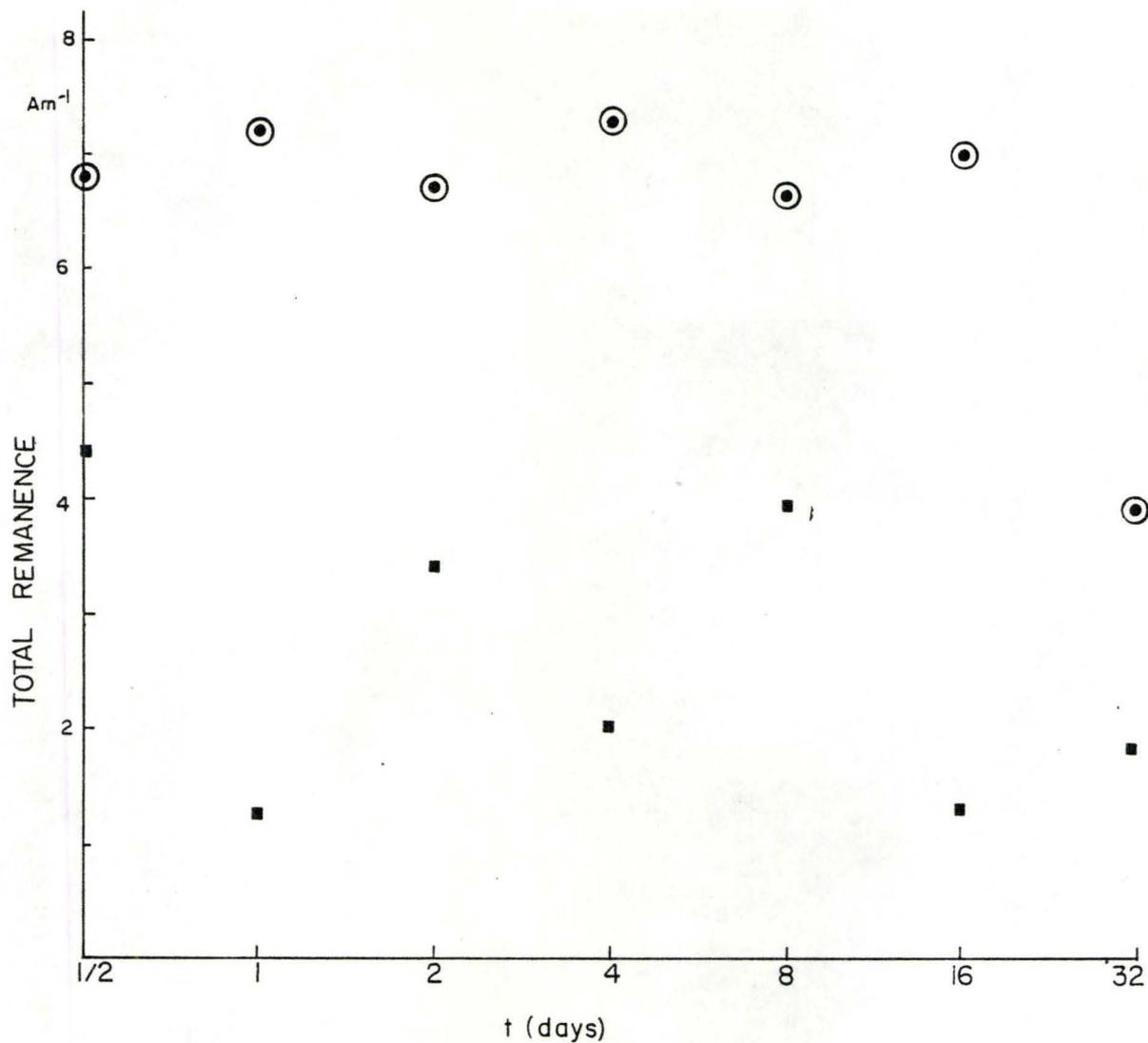


Fig 3-13 The intensity before storage of samples with weak initial NRM (squares), and the intensity after storage (circles), plotted against the length of time stored.

separated from the initial NRM during thermal demagnetization.

The direction of the remanence does not change in these samples during storage. The normalized intensities thus provide an accurate measure of the change in remanence. Figure 3-14 shows that the remanence of samples demagnetized in vacuum is totally removed by heating to 540°C , this is below the Curie temperature of magnetite. Samples demagnetized in air do not have the remanence totally removed until temperatures greater than 620°C , well above the Curie temperature of magnetite but below the CDM Curie temperature given in section 3.1. for the post storage, cation deficient magnetite.

3.6 THERMAL DEMAGNETIZATION OF SAMPLES WITH WEAK INITIAL NRM

Thermal demagnetizations of samples were carried out in air and in vacuum. Since the samples with initial TRM completely demagnetized with heatings close to the Curie temperature of magnetite ($T_c=585^{\circ}\text{C}$), and the color of the samples changed from pink to grey during the vacuum heatings, it appears that the material reverted to magnetite after the first vacuum heating. The samples demagnetized in air did not unblock at 585°C and some remanence remained at temperatures greater than 620°C . The color of these samples remained pink and the material apparently was not reduced to magnetite.

The normalized intensity curves for samples demagnetized in vacuum are shown in figure 3-15 and 3-17. Direction plots are shown in figure 3-18 and 3-19. The intensity of the remanence is stable up to heating to 350°C with about 80% of the remanence remaining. The loss of remanence below 400°C could be due to

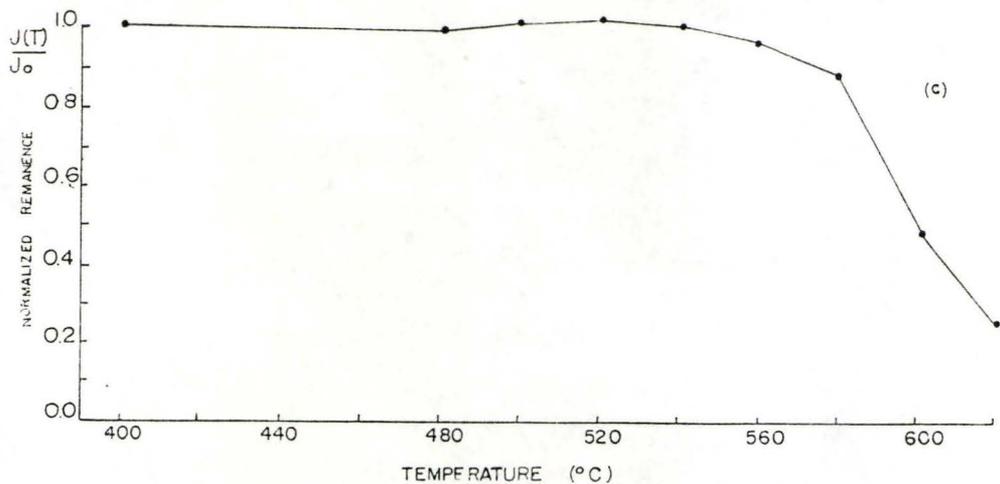
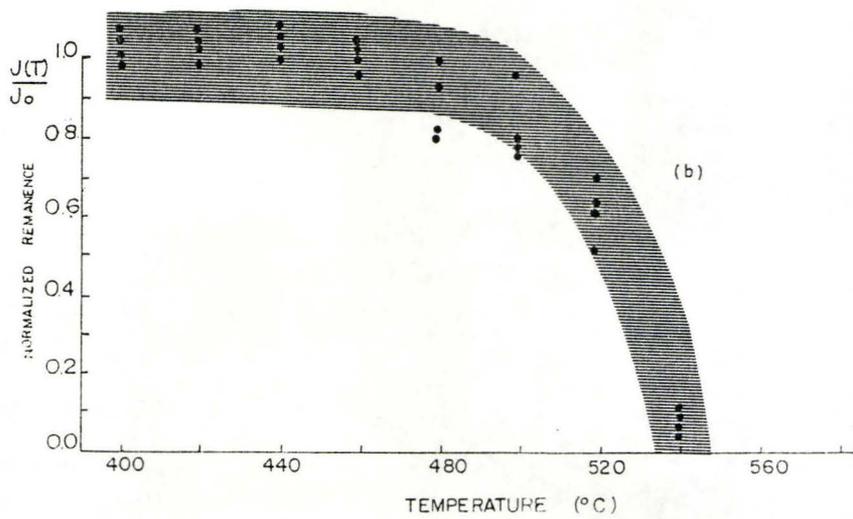
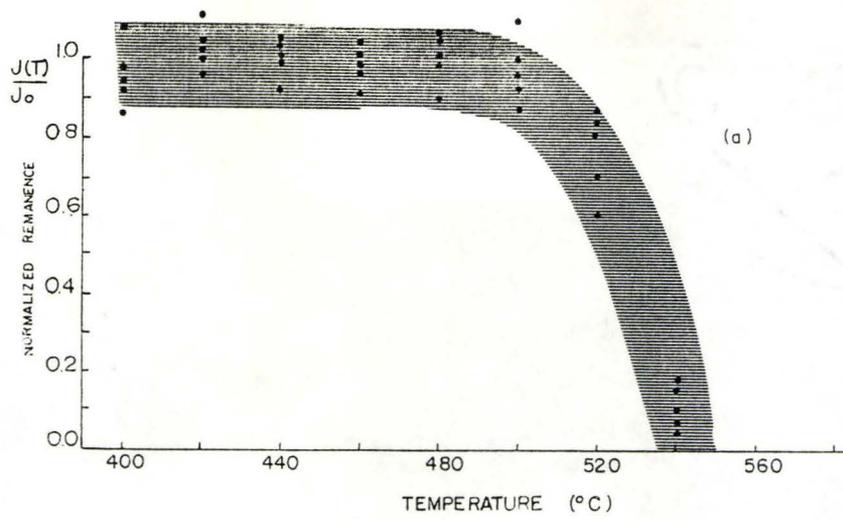


Fig 3-14 a) Vacuum thermal demagnetization of samples with an initially horizontal NRM and b) samples with an initial vertical NRM. The remanence is completely removed by heating above 560°C. c) Air thermal demagnetization of a sample with an initially vertical remanence. The remanence is completely removed only by heating above 620°C.

partial TRM with low blocking temperatures, acquired when the samples were removed from storage.

Demagnetization occurs at temperatures above 400°C . The remanence decreases to about 60 - 40% of the initial remanence after heating to 440°C . The remanence carried in this temperature range lies in the field direction. The demagnetization curves of samples stored for less than 16 days show a component of stable remanence corresponding to the NRM. This stable component appears as a horizontal segment in the intensity curves from about 440°C until the samples become unstable and demagnetize to less than 10% of the initial remanence. This component is not in the field direction but corresponds to a remanence direction moving toward the initial NRM direction.

As the length of storage time increases, the stability of the component in the ambient field direction increases. The remanence in samples stored for only 1/2 a day moves towards the initial NRM direction with the first heating, and the stable portion of the intensity curve spans much of the demagnetization. With longer storage times the remanence remains in the storage field direction at progressively higher temperatures. For storage times lasting four days the remanence begins to move back to the initial NRM direction only after heating to 460°C . The stable portion of the demagnetization curve spans less of the total demagnetization spectrum. After 8 days the remanence no longer returns to the initial NRM direction upon demagnetization. The remanence remains in the field direction or in a direction between the field and initial direction throughout the

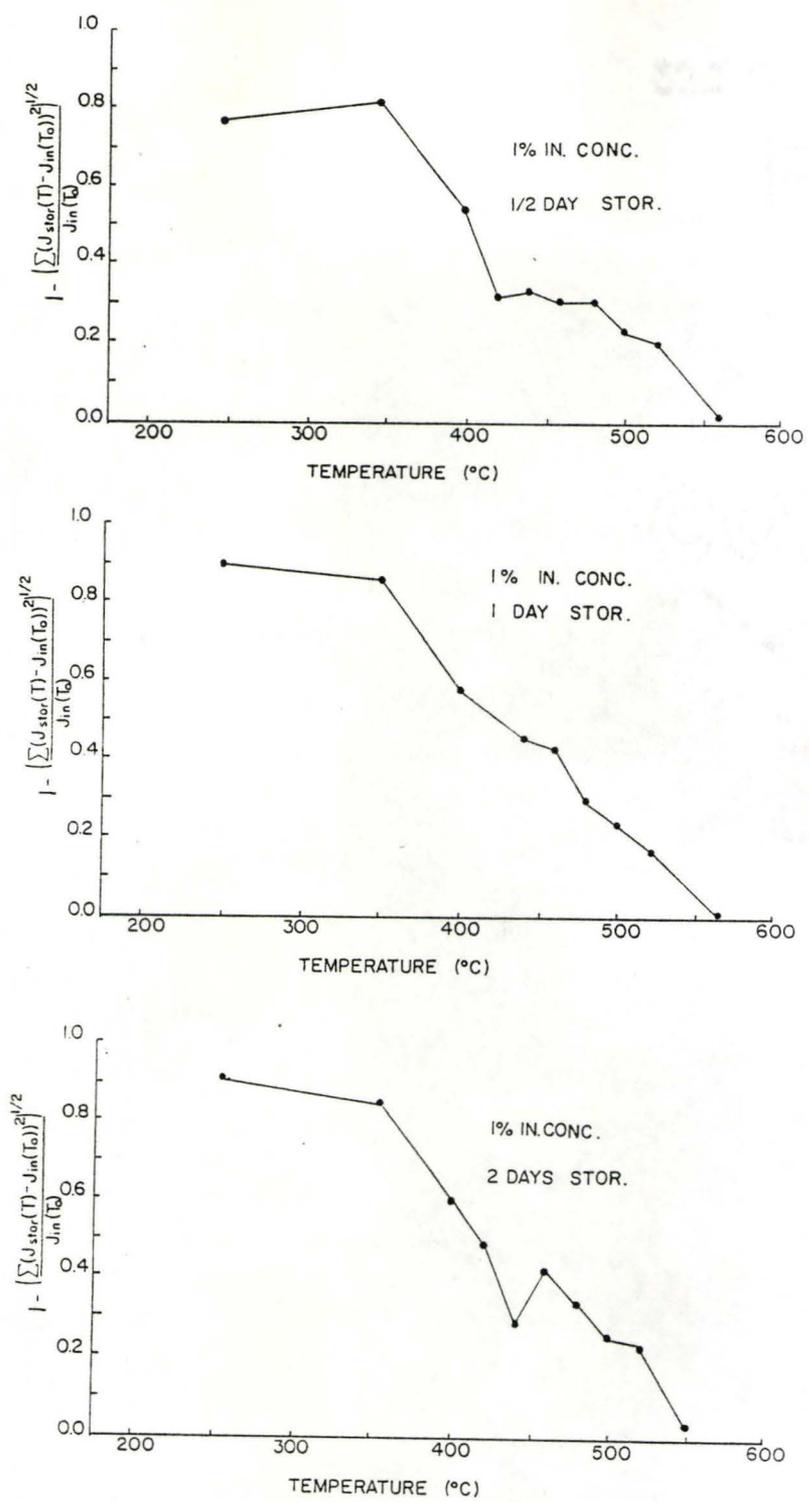
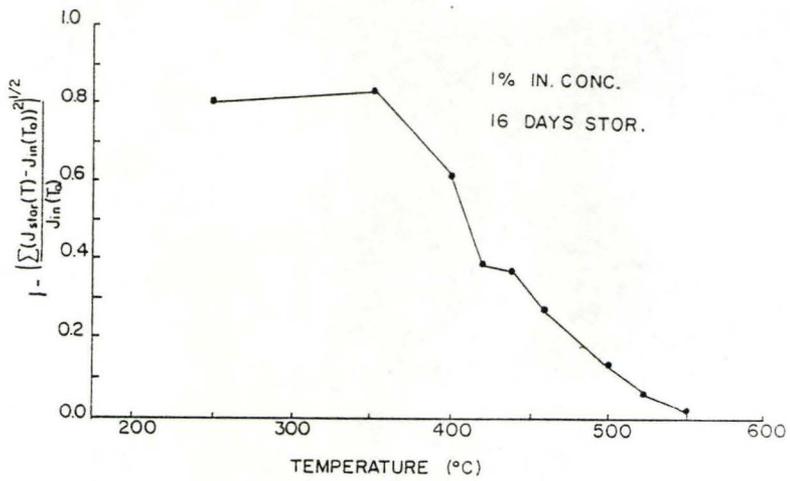
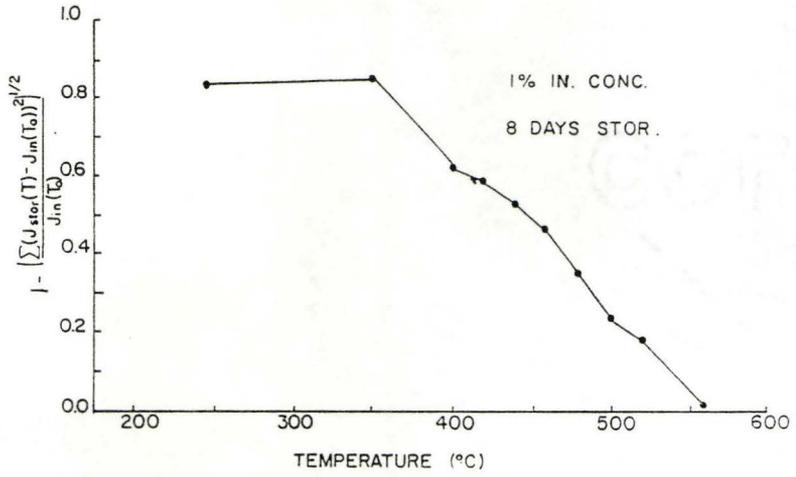
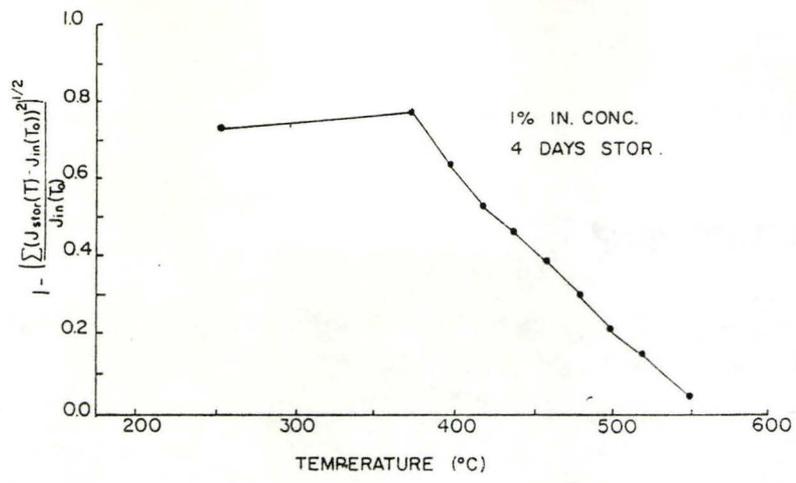


Fig 3-15 Vector difference intensity plots for 1% samples demagnetized in vacuum. The vector difference is used since large changes of direction were observed in the remanence during demagnetization. The remanence has a stable component for temperatures from about 440°C to about 540°C. in samples stored less than 16 days.



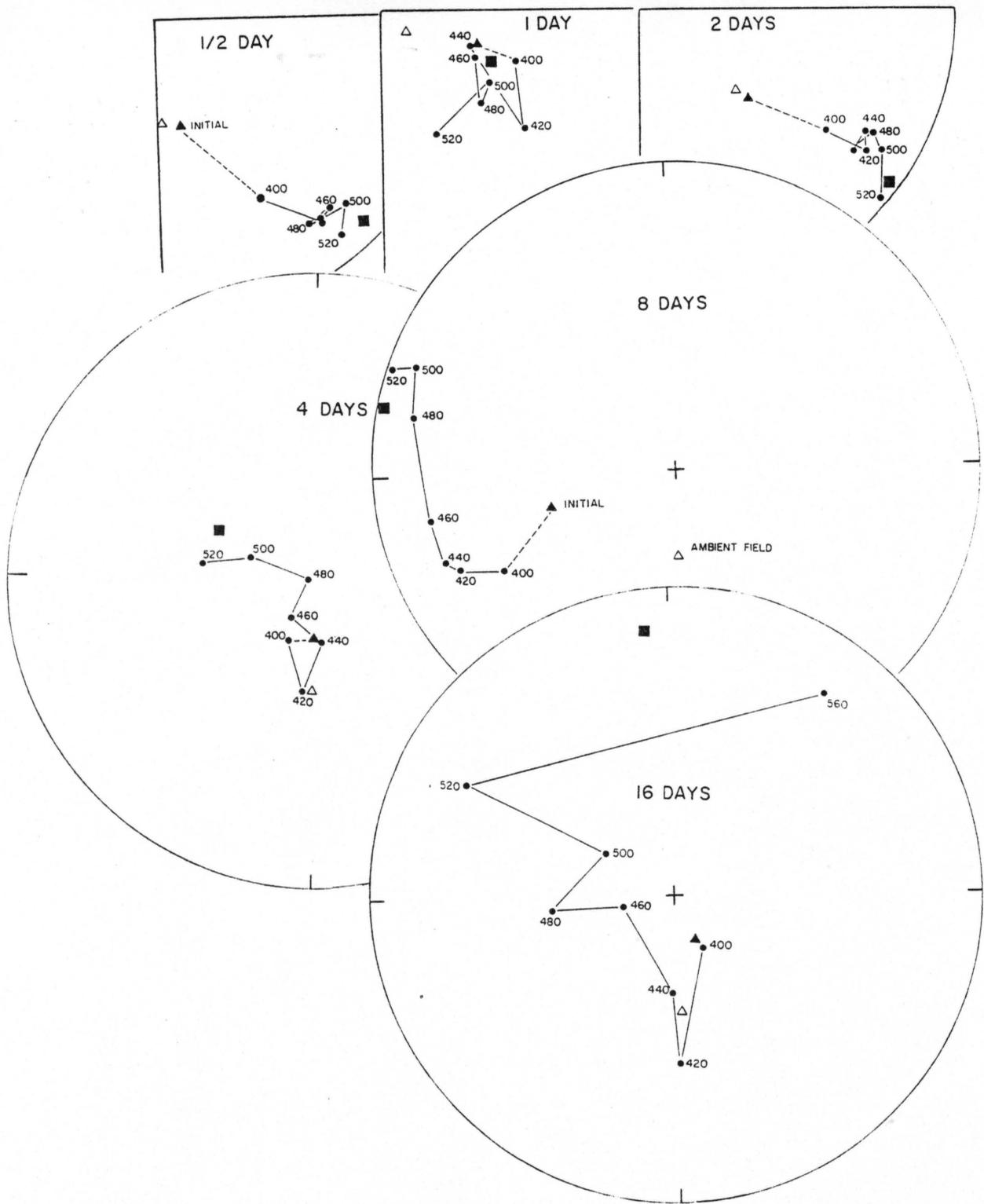


Fig 3-16 stereonet projections of 1% samples demagnetized in vacuum. The remanence direction is initially near the storage field direction. For samples stored less than 16 days the remanence direction moves towards the initial direction with increased heating.

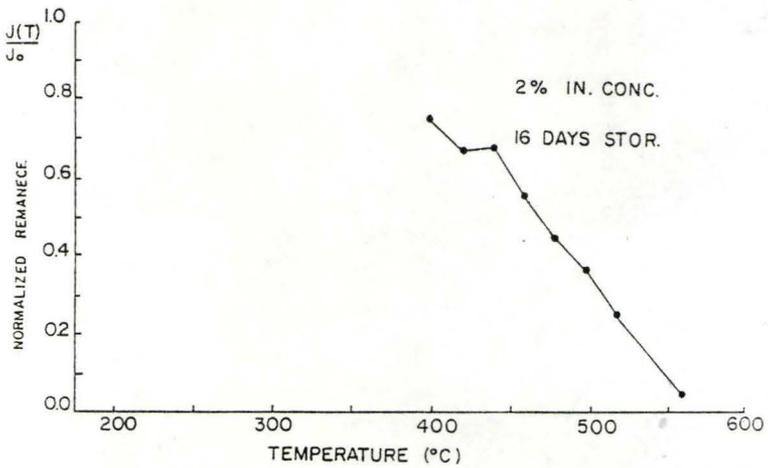
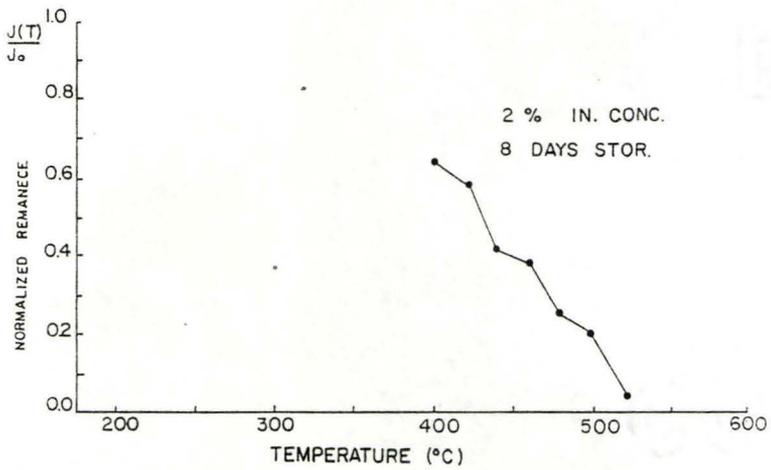
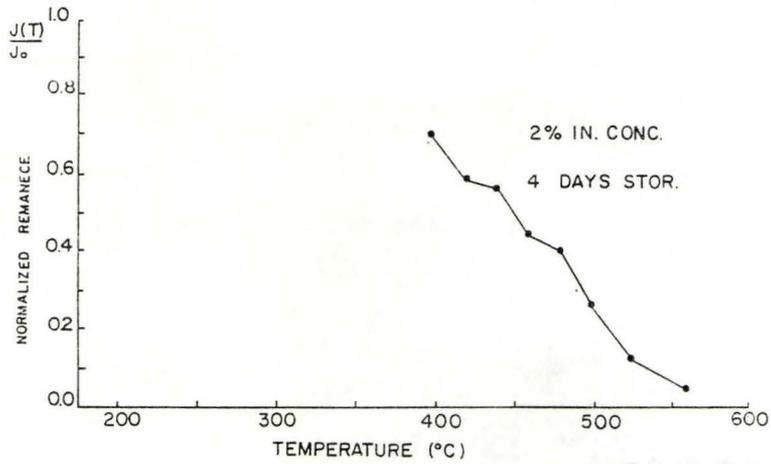
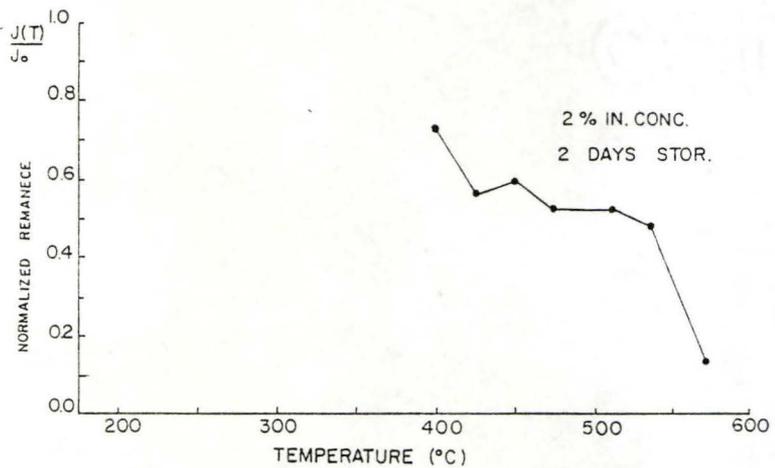
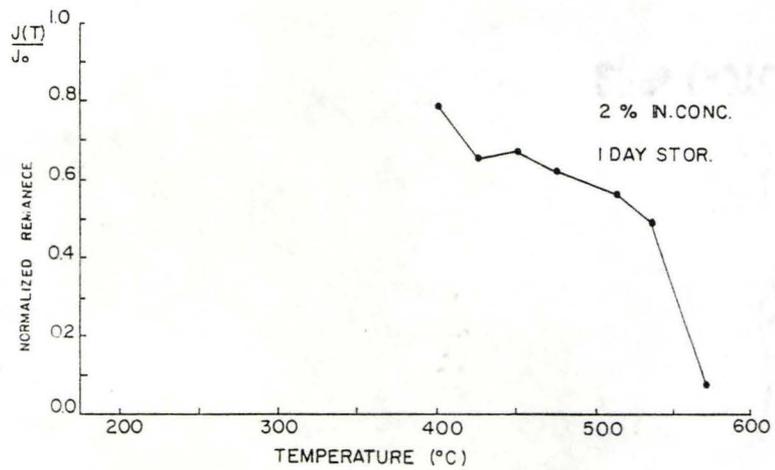
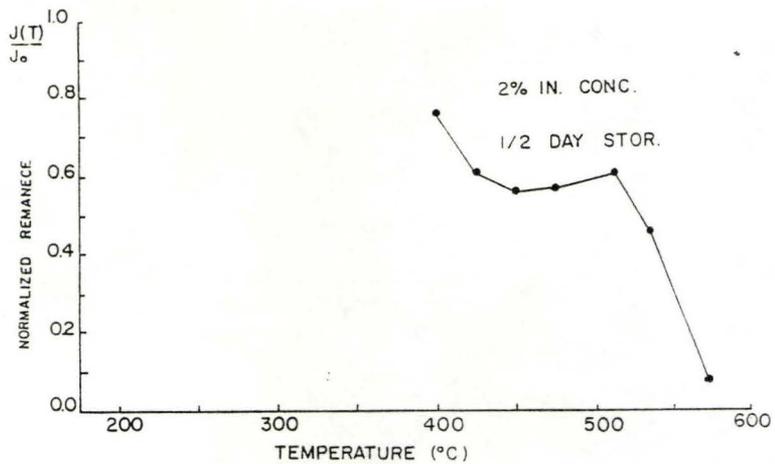


Fig 3-17 Normalized remanence for 2% samples demagnetized in vacuum. The remanence is stable from 440°C to 540°C.



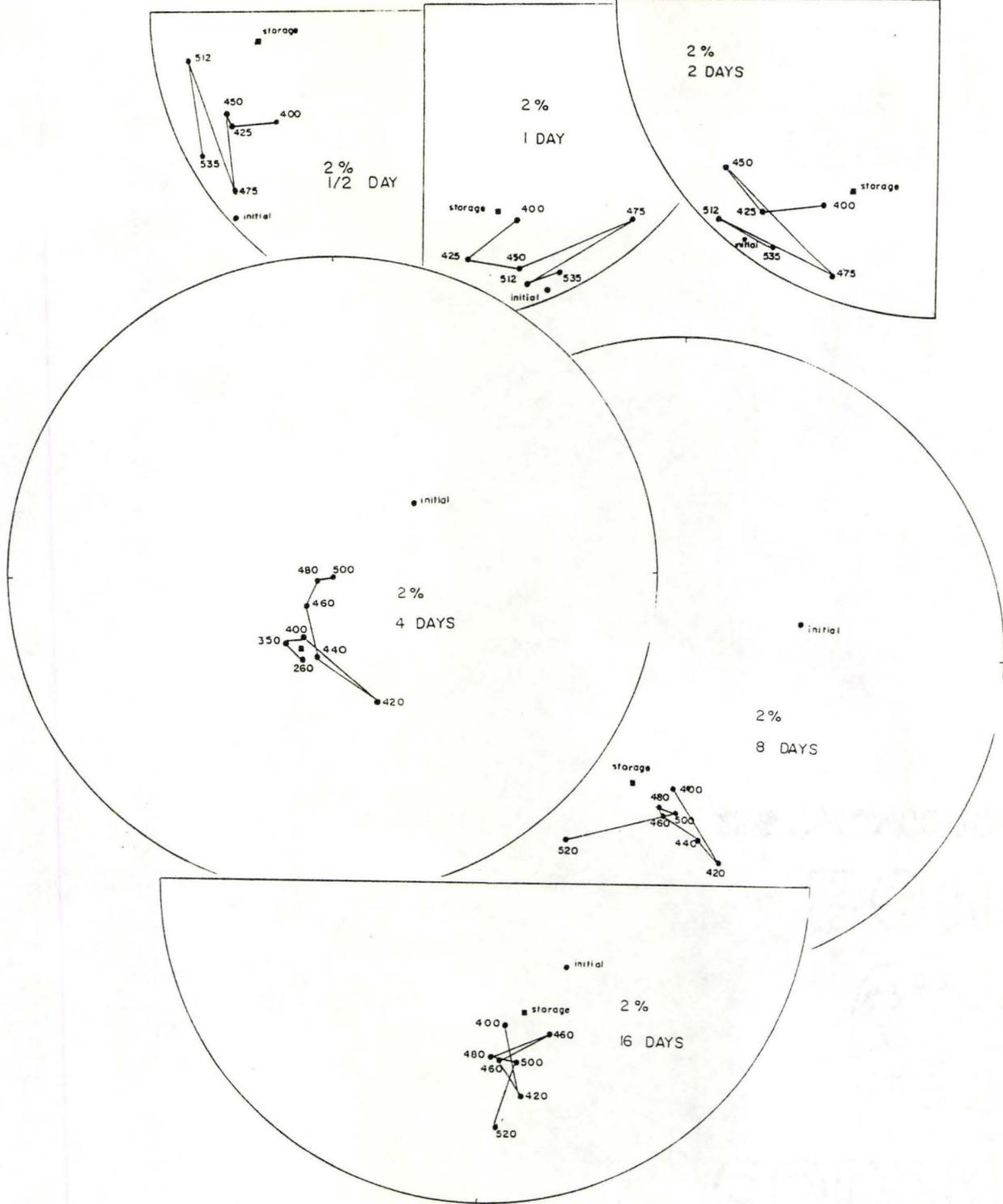


Fig 3-18 Stereonet projections of 2% samples demagnetized in vacuum. The remanence direction of samples stored less than 8 days moves from the storage direction towards the initial NRM direction.

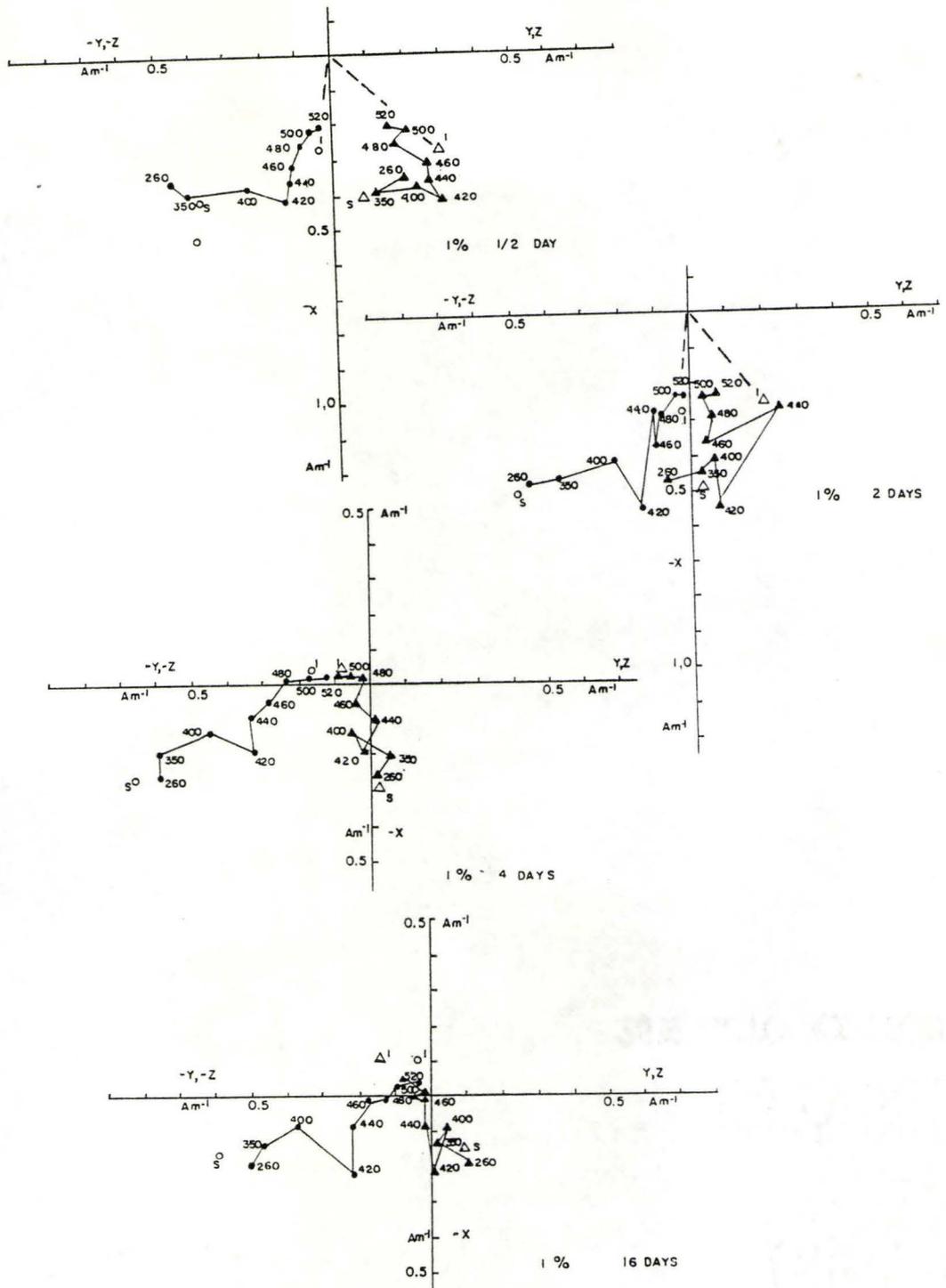


Fig 3-19 Zijdeveld plot of samples demagnetized in vacuum. circles are in the vertical plane, triangles are in the horizontal plane. A line drawn from the origin to the open symbol denoted (I) shows the initial direction. The solid symbols follow linear trends which parallel these directions throughout the T_D range this component is demagnetized.

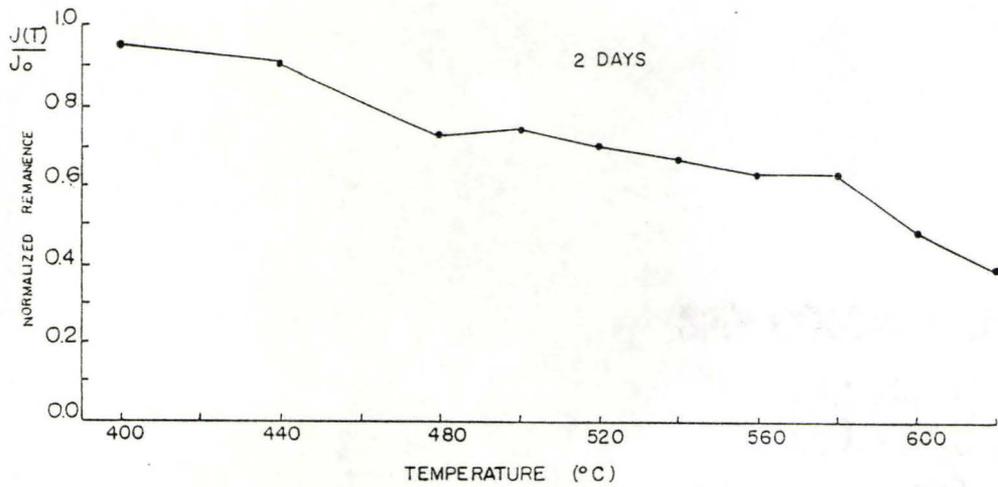
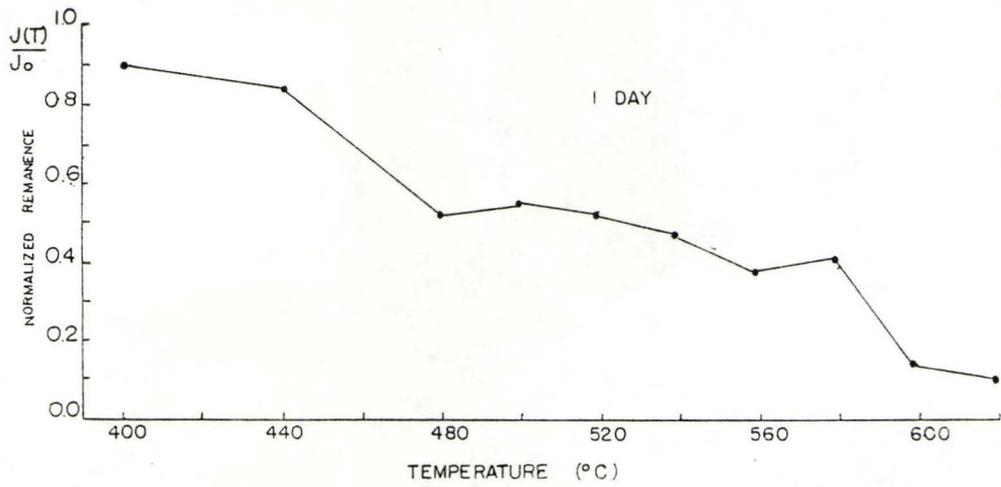
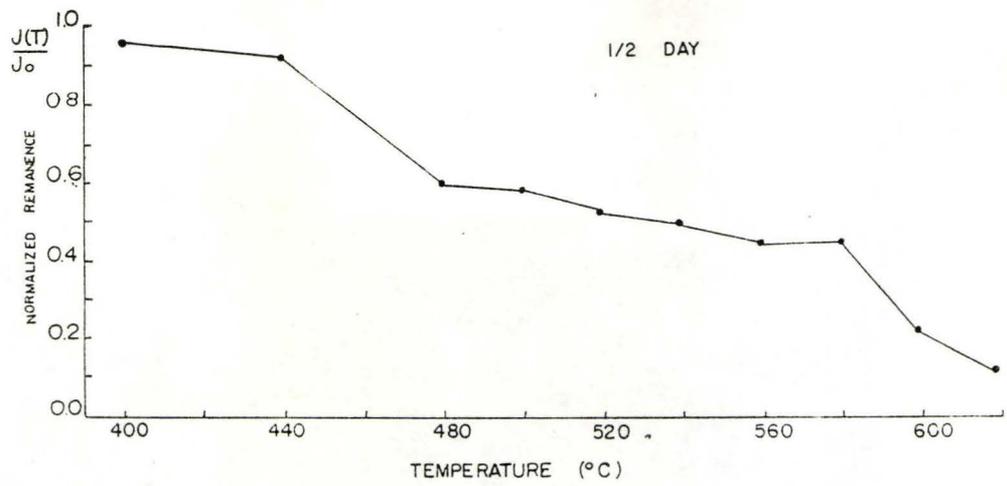
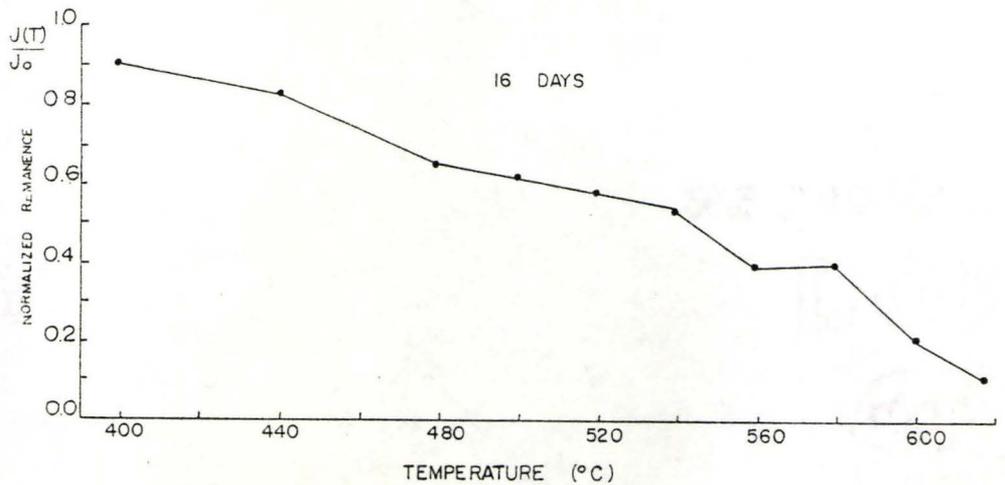
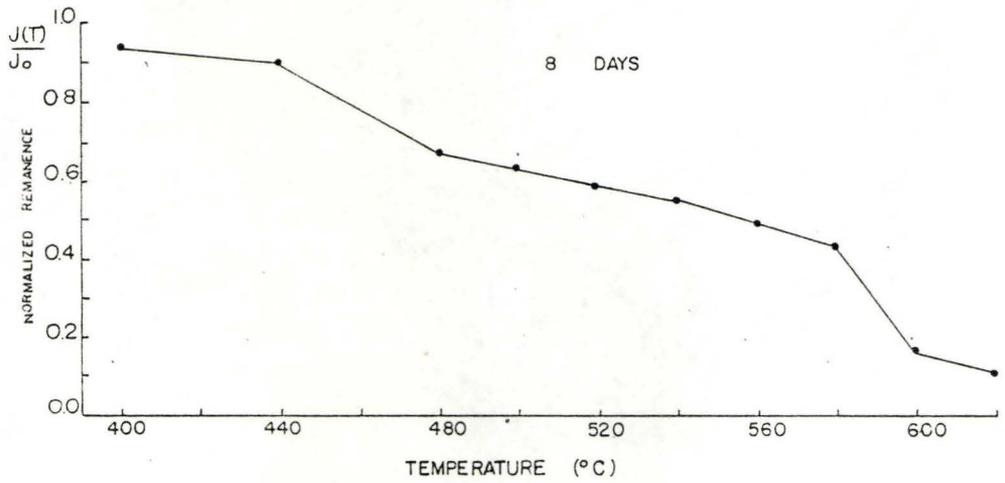
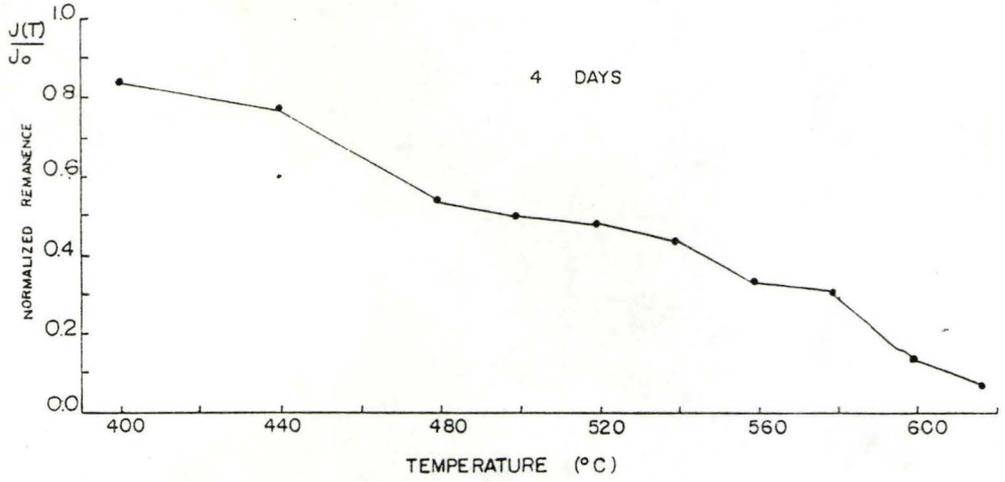
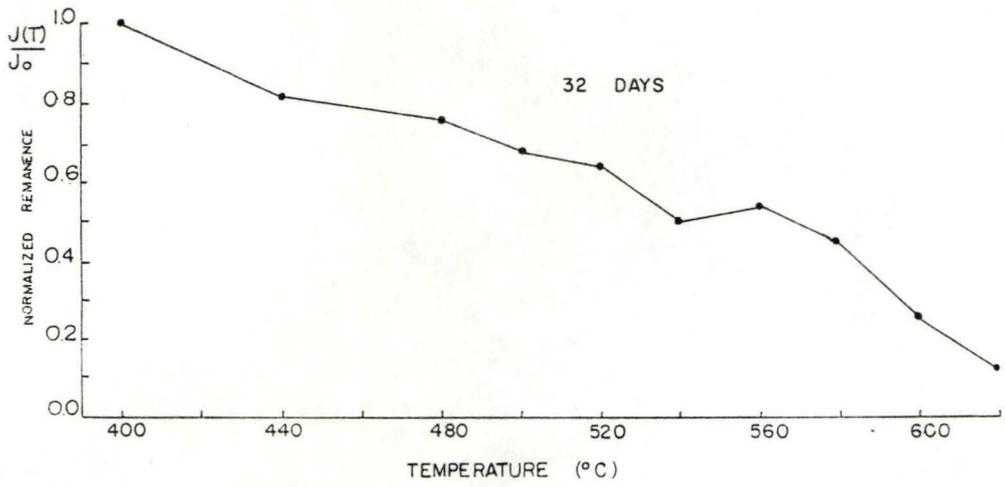


Fig 3-20 Normalized intensity of samples demagnetized in air. The remanence in samples stored less than 8 days is stable between 480°C and 580°C.





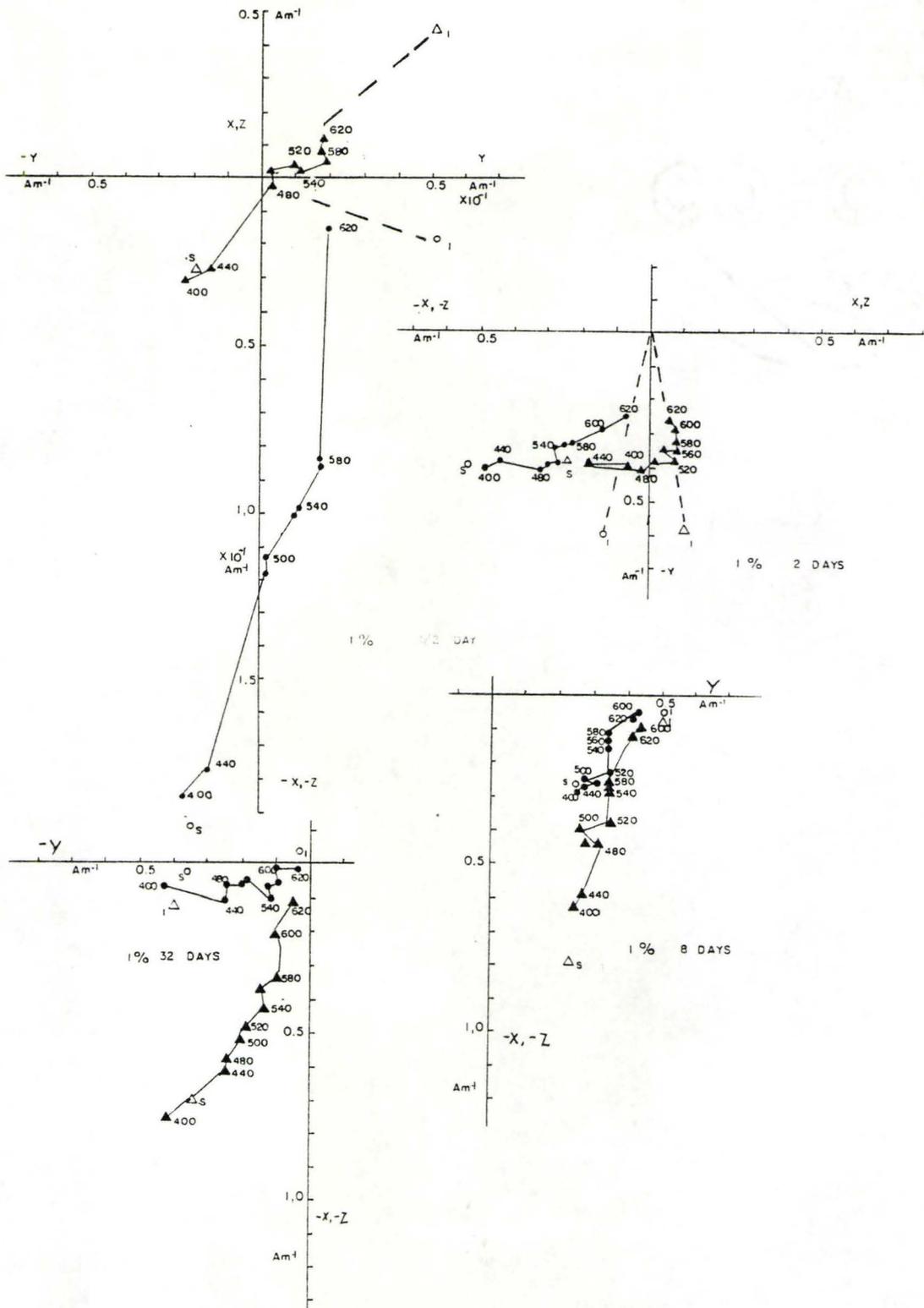


Fig 3-21 Zijderveld plot for samples demagnetized in air.
 circles are in the vertical plane
 triangles are in the horizontal plane
 A line drawn from the origin to the open symbol denoted (I) gives
 the initial direction.

demagnetization. Demagnetization curves no longer have a NRM component and the remanence reduces to less than 10% of its original intensity at a temperature of about 560°C.

Samples demagnetized in air have similar demagnetization curves to those samples demagnetized in vacuum. The main difference is that the temperatures to unblock similar remanence components are higher in samples heated in air. This is due to the fact that the Curie temperature of the oxidized material is greater than the reduced material. Fig 3-20 shows the normalized intensity remaining after each demagnetization step. At temperatures below 400°C the remanence does not demagnetize more than 10%. This loss of remanence is probably due to PTRM acquired during cooling. Subsequent heatings cause a decrease of remanence until about 60% of the remanence remains. The direction of this low temperature component (figure 3-21) is in the storage field direction. This is the same result as obtained from samples demagnetized in vacuum. Heating beyond 480°C is accompanied by a NRM component segment in the intensity curves. In samples heated 8 days the demagnetization curves show a stable component which spans most of the demagnetization spectrum: this again similar to the results previously obtained with samples heated in vacuum. A stable component lies in the NRM direction and it demagnetizes to less than 10% of the initial remanence when heated to 620°C. Samples stored longer than 8 days have a single demagnetization slope which corresponds to a remanence in the storage field direction or on a great circle between the NRM direction and the field direction.

As the length of time the sample is stored increases the

effect of the initial NRM component decreases until for storage times longer than 16 days the component in the NRM direction is completely lost. The samples demagnetize to less than 10% of the initial remanence with heatings to 620°C.

All the samples demagnetized show similar demagnetization characteristics. The remanence is initially in the field direction even after storage times as short as 1/2 a day. Upon thermal demagnetization of samples stored less than 16 days the component in the field direction is removed and the remanence returns to the NRM direction. The remanence in the NRM direction is stable against further heating until the blocking temperature of the initial NRM is reached and the samples demagnetizes. Samples stored longer than 8 days have a remanence with an initial component in the field direction, but in these samples the remanence direction does not return to the NRM direction upon thermal demagnetization. Instead the remanence remains in the field direction, or in a direction between the field and the NRM direction, until demagnetization is complete. Thus, after storage for more than 8 days, the NRM record is entirely obliterated.

CHAPTER FOUR

CONCLUSIONS

4.0 COMPARISON OF RESULTS WITH THEORY

The results of thermomagnetic measurements and hysteresis properties indicate that $J_s(T)$ is similar to magnetite and that H_c remains close to the initial value for magnetite as well. It is interesting to compare the behaviour of the low T , viscous component, with the thermal activation curves of Pulliah et.al. (1975). This component which was acquired in 32 days at 400°C was removed by heating in vacuum to temperatures less than 440°C .

The thermal activation curves of Pulliah et.al.(1975) predicted that a VRM acquired by heating to 400°C for 1 day will require a 20 minute heating at a temperature of 440°C to completely unblock. The experimental results indicate that all of the VRM acquired at 400°C was unblocked with twenty minute heatings at a temperature of 440°C , so the agreement with theory is good.

The results from the thermal demagnetization performed in air do not permit as straightforward a comparison with theory. An allowance must be made for the higher T_c observed in the oxidized material. The shape of the thermal activation curves for magnetite should not need to be changed since the measured J_s and H_c of the material are nearly the same as for the initial magnetite. Although $J_s(T)$ is not a linear function, to a first approximation the thermal activation curves can be applied if the temperature scale is recalibrated to the new T_c of the chemically

altered material. This modification results in the VRM acquired by heating at temperatures of 400°C for one month requiring 20 minute heatings at 480°C to remove the VRM. This temperature agrees with the experimentally observed temperature required to remove the remanence component in the storage field direction. This suggests that the chemical change has most significantly affected the blocking temperature spectrum by a shift in T_c .

Pulliah et.al. (1975) suggested that the enhanced stability of their remanences might result from chemical alteration. This explanation is supported by the present study as the blocking temperature was increased by 40°C.

4.1 IMPLICATIONS OF RESULTS IN TERMS OF PALEOMAGNETISM

The storage experiment provides a close analogue of natural TRM's induced in igneous bodies during cooling and subsequently overprinted during metamorphism. It also closely simulates metamorphic modification of the remanence carried by intrusion-baked sedimentary rocks. The hysteresis data confirmed that the magnetic characteristics of the synthetic samples were similar to those found in a wide range of naturally occurring rocks. For these reasons the results of this study have implications for paleomagnetic work and they are:

1. Remanence acquired during high temperature storage of a sample with a weak initial NRM lies in the direction of the ambient field during storage. It does not depend on the direction of the initial NRM. This result is similar to the results of experiments on VRM by Nagata (1961), and on CRM by Bailey and Hale,(1981). Weak NRM's in paleomagnetic samples thus

cannot be expected to survive metamorphism.

2. The remanence direction in samples with a strong initial NRM moved only slightly towards the direction of the ambient field during high temperature storage largely preserving the initial NRM. The initial NRM direction is recoverable by thermal demagnetization of samples which were stored for short periods of time. In samples stored for longer times the initial NRM direction is completely overprinted. The acquired CRM component can totally reset the remanence so that determining the initial NRM direction is no longer possible. Extrapolating short duration experiments to geologic time is unwise since metamorphism usually results in the acquisition of CRM's. If CRM acquisition has taken place, the NRM of metamorphic rocks cannot be used as an indicator of the pre-metamorphic magnetic field.

3. The VRM acquired during storage can be separated from the CRM, on the basis of blocking temperature. The VRM has distinctly lower blocking temperatures than the CRM which has blocking temperatures which are as high as the blocking temperatures of the TRM.

McClelland-Brown (1982) used the blocking temperature distribution to estimate the maximum temperatures reached in a country rock intruded by a diabase dike. In a similar type of study, Schwarz and Buchan (1981) deduced the amount of uplift in the Subury area by using blocking temperature spectra and an assumed geothermal gradient. Buchan, Berger, McWilliams, York and Dunlop used the blocking temperature spectrum found in highly metamorphosed Grenville terrain to interpret components acquired

during distinct metamorphic events. The results of the present study show that minor chemical alteration can result in blocking temperatures significantly higher than those predicted by the thermal activation curves of Pulliah et.al.(1975). Even though the hysteresis properties measured showed that the material was still magnetically similar to the initial magnetite, after the chemical alteration during storage the Curie temperature was found to be significantly higher than the Curie temperature of magnetite. Only when the temperature axis of the magnetite thermal activation curves is rescaled to the higher Curie temperature of the altered samples are the predicted blocking temperatures close to the experimentally observed values.

The results indicate that the theory of Pulliah et.al.(1975) and Walton (1980) may be applicable to estimation of survival temperatures of paleomagnetic remanences only under the following conditions:

i) Thermomagnetic curves are known for individual samples before and after thermal demagnetization and ideally they show no change.

ii) If there has been chemical alteration, the VRM must be separable from the CRM.

iii) Since chemical alteration may accompany thermal demagnetization the temperature axis of the thermal activation curves must be rescaled to the post-heating Curie temperature.

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