

Inferring domain state from magnetic hysteresis in high coercivity dolerites bearing magnetite with ilmenite lamellae

J.P. Hodych *

Department of Earth Sciences, Memorial University of Newfoundland, St. John's, Nfld., A1B 3X5, Canada

Received 6 March 1996; accepted 20 May 1996

Abstract

Hysteresis loops were measured for 16 dolerite samples from separate Precambrian dykes whose remanence is probably primary. Remanence is carried by grains of magnetite subdivided into fine particles, usually by ilmenite lamellae, as seems common in mafic igneous rocks of high coercivity. Coercive force H_C ranges from 9 to 40×10^3 A/m (110–500 Oe). The ratio J_{RS}/J_S of saturation remanence to saturation magnetization ranges from 0.10 to 0.45. J_{RS}/J_S varies in approximate proportion to H_C with a constant of proportionality appropriate for pseudo-single-domain magnetite with one or two domain walls per particle. A cooling cycle to 77K causes large (40% on average) demagnetization of J_{RS} in most samples, supporting the dominance of pseudo-single-domain magnetite. Most samples also show a large (29% on average) decrease in H_C on cooling to 140K, suggesting magnetostrictive control of H_C , perhaps through internal stresses opposing domain wall motion. Only the three dolerites with H_C greater than 30×10^3 A/m (380 Oe) show little change in H_C on cooling, suggesting dominance by single-domain particles with shape anisotropy.

In magnetite grains subdivided into single-domain particles by ilmenite lamellae, magnetic interaction may lower J_{RS}/J_S from the expected 0.5. Some theories [1,2] suggests that this is due to a self-demagnetizing field from magnetic poles on the surface of each grain of magnetite subdivided by ilmenite and should be corrected for. Another theory [3] suggests that this correction is not necessary because the self-demagnetizing field is already approximately cancelled, due to magnetic poles inside each subdivided grain, at the ends of magnetite particles. The latter suggestion is favoured by our three highest H_C dolerites, whose J_{RS}/J_S averages 0.42 ± 0.04 before and 0.66 ± 0.06 after correction for the self-demagnetizing field from poles on the surface of magnetite–ilmenite grains. Applying this correction to our other dolerites usually raises J_{RS}/J_S significantly above 0.5, which is unreasonable for pseudo-single-domain magnetite. Hence, correcting for self-demagnetizing fields from poles on the surface of magnetite–ilmenite grains in rocks is not recommended, whatever the domain state.

Keywords: magnetic hysteresis; magnetic domains; diabase; magnetite

1. Introduction

Grains consisting of magnetite finely subdivided by ilmenite lamellae are common in mafic igneous rocks and are important recorders of the Earth's

magnetic field [4]. They may, for example, be the main recorders of primary remanence in Precambrian dolerite dyke swarms that have passed baked contact tests. Such dyke swarms are very important to Precambrian palaeomagnetism [5] because they often yield very precise baddeleyite or zircon U–Pb dates [6].

* Fax: 709 737 2589. E-mail: hodych@kean.ucs.mun.ca

In this paper, the magnetic hysteresis of Precambrian dolerite dyke samples is measured to help determine the source of the high stability (coercivity) of their remanence. Graham [7], studying some Precambrian dolerite dykes, first suggested that high coercivity remanence might be due to fine subdivision of magnetite by ilmenite lamellae. Strangway et al. [8] and Larson et al. [4] demonstrated that such subdivision was common in basalts and that it might be fine enough to produce single-domain magnetite grains whose shape anisotropy could be responsible for the high coercivity fraction of remanence. However, pseudo-single-domain magnetite grains can also produce high coercivity remanence. For example, Hodych [9,10] showed that a Matachewan dolerite dyke that carries a 2.5 billion year old remanence has its coercivity magnetostrictively controlled, presumably through internal stresses opposing domain wall motion.

Magnetic hysteresis loops (Fig. 2) yield saturation remanence (J_{RS}) and saturation magnetization (J_S) whose ratio allows single-domain grains to be distin-

guished from pseudo-single-domain grains. For the latter, J_{RS}/J_S should be lower than the 0.5 expected of randomly oriented elongated single-domain grains. However, in mafic igneous rocks, paramagnetic ilmenite lamellae commonly subdivide the magnetite grains into separate fine particles. Magnetic interactions between these magnetite particles may lower J_{RS}/J_S , making it harder to distinguish single-domain from pseudo-single-domain particles. Davis and Evans [1] suggested a method for correcting for these interactions in the case of single-domain particles. The consequences of applying this correction will be explored for the dolerite samples.

2. Palaeomagnetism and magnetic mineralogy of the dolerites

The samples (Table 1) are all dolerites from separate Precambrian dykes. Most samples are from a swarm of east–west trending dykes sampled along ~ 130 km of coast north and south of Nain, Labrador.

Table 1
Magnetic properties of the dolerites

Sample	$H_C \times 10^3$ (A/m)	$H_1 \times 10^3$ $\frac{1}{2}$ (A/m)	$H_{CR} \times 10^3$ (A/m)	$\% \Delta H_C$	$\% \Delta J_{RS}$	J_{RS}/J_S	J_{RS}^*/J_S	Fe_3O_4 (%)	X	f
9116	8.8	13.0	16.6	-37	-50	0.10	0.38	1.9	0.73	0.49
BX86	10.3	10.3	18.8	-35	-49	0.13	0.62	1.0	0.34	0.76
SD78	12.1	12.9	20.4	-35	-52	0.18	0.56	1.0	0.60	0.58
RE88	12.7	15.4	24.5	-24	-39	0.16	0.59	2.2	0.70	0.51
3301	14.5	13.8	22.6	-19	-34	0.17	0.53	2.3	0.58	0.60
9138	17.5	19.9	31.4	-18	-32	0.20	0.57	1.1	0.51	0.65
3203	17.7	16.7	27.4	-31	-50	0.22	0.53	0.9	0.63	0.56
2701	21.8	25.8	31.3	-30	-42	0.29	0.60	0.7	0.68	0.53
3101	22.5	23.7	34.1	-27	-42	0.28	0.62	1.3	0.65	0.55
4602	22.9	30.6	35.3	-32	-38	0.28	0.53	1.1	0.77	0.47
5601	25.4	35.5	42.2	-25	-40	0.29	0.60	2.2	0.76	0.48
9102	29.3	36.8	44.2	-22	-20	0.35	0.63	1.4	0.66	0.54
9128	30.4	35.8	43.7	-40	-36	0.32	0.58	1.5	0.70	0.51
5901	31.5	32.6	43.8	-6	-16	0.37	0.60	1.3	0.86	0.41
4305	32.5	36.8	43.7	+1	-11	0.42	0.68	0.4	-	0.41
9144	40.1	48.9	62.4	-5	4	0.45	0.71	0.3	-	0.53

H_C is coercive force ($\pm 0.3 \times 10^3$ A/m); H_1 is the alternating field that halves the natural remanence intensity; H_{CR} is remanence coercivity; $\% \Delta H_C$ is the change ($\pm 3\%$) in H_C on cooling to 140K; $\% \Delta J_{RS}$ is the change ($\pm 3\%$) in saturation remanence due to a cooling cycle to 77K; J_{RS}/J_S and J_{RS}^*/J_S are the ratio of saturation remanence to saturation magnetization before and after 'correction' for self-demagnetizing field of poles on the surface of intergrown magnetic grains; Fe_3O_4 is the volume percent magnetite in the sample; X expresses the Ti content of the intergrown magnetic grains; f is the volume fraction of magnetite in the magnetic grains.

The exceptions are samples RE88, SD78 and BX86 which are from the Mackenzie, Matachewan and Biscotasing dyke swarms, respectively. All the samples probably carry a primary remanence (with only a small secondary overprint). This has been proven with positive baked contact tests for the Mackenzie, Matachewan and Biscotasing dyke swarms [5]. The Nain east–west dyke swarm has an age (1.28 Ba, pers. commun., J.C. Roddick, 1994) and remanence direction similar to the Mackenzie dyke swarm. For the 81 Nain dyke samples that carry this remanence direction, the alternating field strength (H_1) required

to halve the natural remanence intensity ranges from 6×10^3 to 54×10^3 A/m (80 and 680 Oe) and averages 25×10^3 A/m (320 Oe). The 13 Nain dyke samples of the present study were chosen to represent this range in H_1 (Table 1), and because each sample has an alternating field demagnetization curve that suggests a relatively narrow range of coercivities.

The magnetic grains in the dolerites are expected to have crystallized as a magnetite–ülvöspinel solid solution with the formula $(1 - X) \text{Fe}_3\text{O}_4 \cdot X \text{Fe}_2\text{TiO}_4$ and to have oxidized on cooling to produce an intergrowth of nearly pure magnetite with ilmenite lamellae along [111] planes [11]. The weight percent of TiO_2 , and of total iron as FeO, were measured for six intergrown grains in a polished section of each sample. Then X was calculated (Table 1) using the relation $X = 215.4(\text{TiO}_2/\text{FeO})(79.9 + 71.8 \text{TiO}_2/\text{FeO})^{-1}$. The volume fraction f of magnetite in the intergrown grains was estimated (Table 1) using $f = [1 + 31.7X/44.2(1 - \frac{2}{3}X)]^{-1}$. An electron microprobe was used for most of these determinations of X , and a scanning electron microscope (SEM) for the remainder (samples 9144, 4305, 3301, 4602 and 9102). Although the SEM analyses are only semi-quantitative, they were found to yield values of X within 0.10 of the microprobe determinations. The SEM analyses suggest that the magnetite is intergrown with silicate minerals as well as ilmenite in sample 4305 and mainly with silicate minerals in sample 9144. The silicate minerals in the grains (as anorthite and enstatite) as well as the ilmenite were taken into account in estimating f for these two samples.

Polished sections of each dolerite were examined with the SEM in back-scattered mode (Fig. 1). Magnetite–ilmenite intergrowth is visible in all specimens with $H_C < 23 \times 10^3$ A/m (290 Oe). For higher H_C , it becomes increasingly difficult to resolve the intergrowth texture. In the highest coercive force sample (9144), most of the intergrowth texture is probably finer than the $\sim 0.2 \mu\text{m}$ resolution limit of the SEM in back-scattered mode.

Curie points lie between 560°C and 580°C for all samples, suggesting that the magnetite is now almost titanium-free. Low temperature demagnetization curves for saturation remanence (as in [12]) give evidence of sharp Verwey transitions in most samples, suggesting that the magnetite is not significantly oxidized. The exceptions are the three highest H_C samples (9144, 4305 and 5901) in which the Verwey transition seems largely suppressed, perhaps by oxidation [13].

3. Hysteresis loop and low temperature measurements

Hysteresis loops (Fig. 2) for dolerite samples of about 1 cm^3 volume were measured to a maximum field of about 294×10^3 A/m (3700 Oe). A vibrating sample magnetometer [14] and an electromagnet with a laminated core were used.

The loops were corrected for paramagnetism in the manner of Gee and Kent [15]. The analyses of Wiebe [16] for 11 Nain east–west trending dyke samples were averaged. All the Fe_2O_3 was assumed to be in magnetite, along with a corresponding amount of FeO, leaving 9.79 ± 2.27 (S.D.) wt% FeO in paramagnetic minerals. Since 1 wt% FeO contributes $2.07 \times 10^{-8} \text{ m}^3/\text{kg}$ to the paramagnetic susceptibility [17] and since the density of the Nain dolerite samples averages $2.82 \text{ g}/\text{cm}^3$, the paramagnetic volume susceptibility, k , of our Nain dolerite samples should average $570 \pm 130 \times 10^{-6}$ ($46 \pm 11 \times 10^{-6}$ in cgs). This susceptibility was used to correct each of our Nain dolerite samples and its error was taken into account in subsequent error estimates. This susceptibility was also used for the other three dyke swarms since their samples had high enough magnetite contents for paramagnetic corrections to be very small.

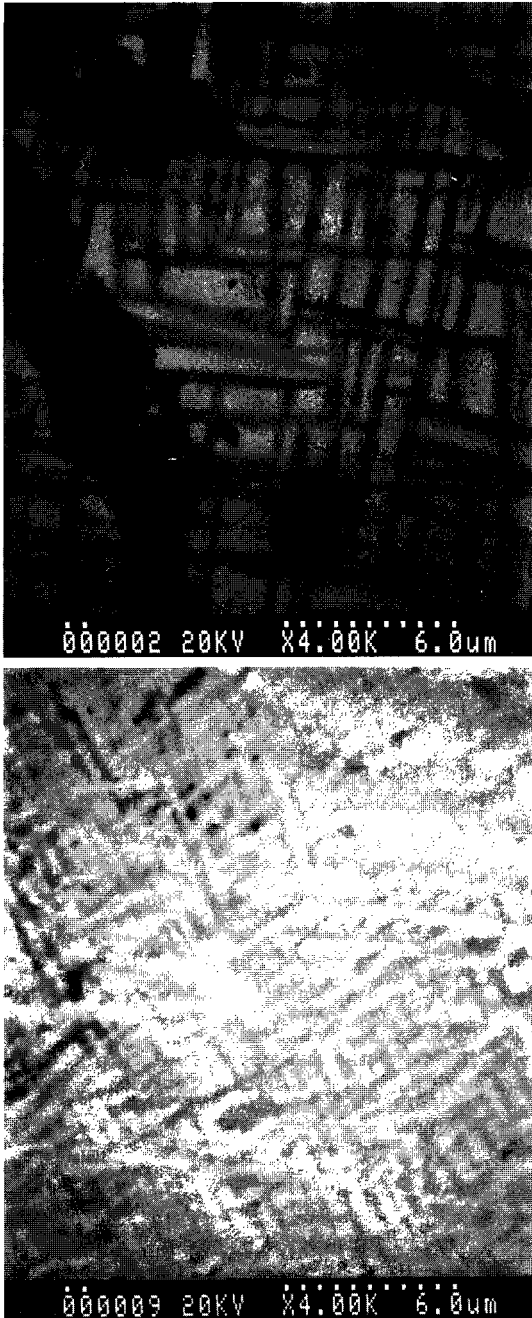


Fig. 1. Scanning electron microscope backscatter images of magnetite (light grey) subdivided by ilmenite lamellae (dark grey) in two Nain dolerite samples. (Silicate minerals are black). The coarser magnetite–ilmenite intergrowth (upper photograph) is from sample 9116 with $H_C = 8.8 \times 10^3$ A/m (110 Oe). The finer intergrowth (lower photograph) is from sample 4602 with $H_C = 22.9 \times 10^3$ A/m (288 Oe). The dotted scale bars represent 6 μm .

After correcting for paramagnetism, saturation magnetization, J_S , was estimated by extrapolating a plot of J versus $1/H$ to $1/H = 0$ [18]. Then, J_{RS}/J_S and H_C were measured (Table 1). The values of J_{RS} and H_C should be within 3% of saturation, judging by extrapolations analogous to those for J_S made for some of the higher H_C samples (3101, 4602, 9102, 5901 and 4305). Neglecting the paramagnetic correction would lower H_C by 3%, or less, and J_{RS}/J_S by 5%, or less, except for 9144, 4305 and 2701 whose J_{RS}/J_S would be lowered by 17%, 13% and 9%,

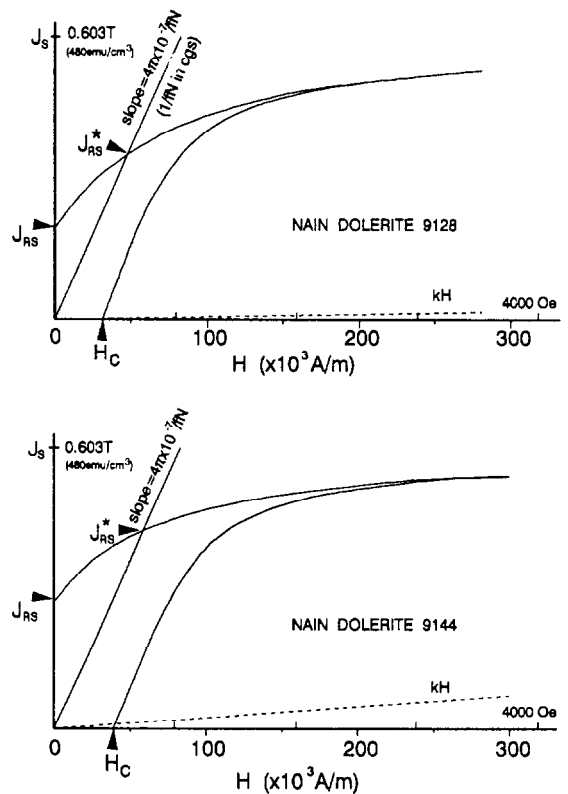


Fig. 2. Hysteresis loops for Nain dolerite samples 9128 (likely pseudo-single-domain magnetite dominant) and 9144 (likely single-domain magnetite dominant). The loops have been corrected for the paramagnetic magnetization, kH , shown by the dotted line. The magnetization (J) axis is calibrated assuming the values shown of saturation magnetization, J_S , per unit volume of magnetite in the sample. Coercive force, H_C , and saturation remanence, J_{RS} , are shown. The method (after Davis and Evans [1]) of obtaining a saturation remanence, J_{RS}^* , 'corrected' for the self-demagnetizing field of magnetic poles on the surface of intergrown magnetite–ilmenite grains is shown (where f is the volume fraction of magnetite in these grains and $N = 1/3$ ($4\pi/3$ in cgs)).

respectively. Magnetite contents of the samples were estimated assuming $J_s = 0.603T$ (480 emu/cm^3) for magnetite.

Remanence coercivity H_{CR} was also measured for each sample (Table 1). This is the reverse field that, when applied and removed, reduces J_{RS} to zero.

The apparatus used for room temperature hysteresis loop measurement was also used to measure H_C as a function of low temperature [14]. The percent change in H_C ($\% \Delta H_C$) observed on cooling from room temperature to 140K is listed in Table 1.

Each sample was given a saturation remanence J_{RS} at room temperature and was placed in zero field where it was cooled to 77K with liquid nitrogen and then warmed back to room temperature. The percent change in J_{RS} ($\% \Delta J_{RS}$) due to this cooling cycle is listed in Table 1.

4. Inferring domain state from magnetic hysteresis

4.1. Non-interacting grains

For randomly oriented elongated single-domain grains, theory [19] predicts $J_{RS}/J_s = 0.5$. As pointed out by Néel [20], for multidomain grains (grains with many domain walls), shearing of the hysteresis loop by self-demagnetizing fields should result in:

$$\frac{J_{RS}}{J_s} \approx 4\pi \times 10^{-7} \frac{H_C}{NJ_s} \left(\frac{J_{RS}}{J_s} \approx \frac{H_C}{NJ_s} \text{ in cgs} \right) \quad (1)$$

The self-demagnetizing factor, N , of the grains is $1/3$ for spherical grains ($4\pi/3$ in cgs). Grains with few domain walls (one type of pseudo-single-domain grain) should behave similarly to multidomain grains, but N should be reduced. For example, $N \approx 0.2$ (≈ 2 in cgs) is expected for cubes with 1 or 2 domain walls [21]. The grains are assumed to be far enough apart to ignore their magnetic interaction in all the above theories.

Fig. 3 is a plot of J_{RS}/J_s versus H_C in which open squares represent the data of Worm and Markert [22] for various sizes of non-interacting magnetite particles (~ 1.5 – 1 elongation) that they precipitated in silicate glass. Only samples with magnetite grain sizes between $2 \mu\text{m}$ and $0.06 \mu\text{m}$ are plotted so that

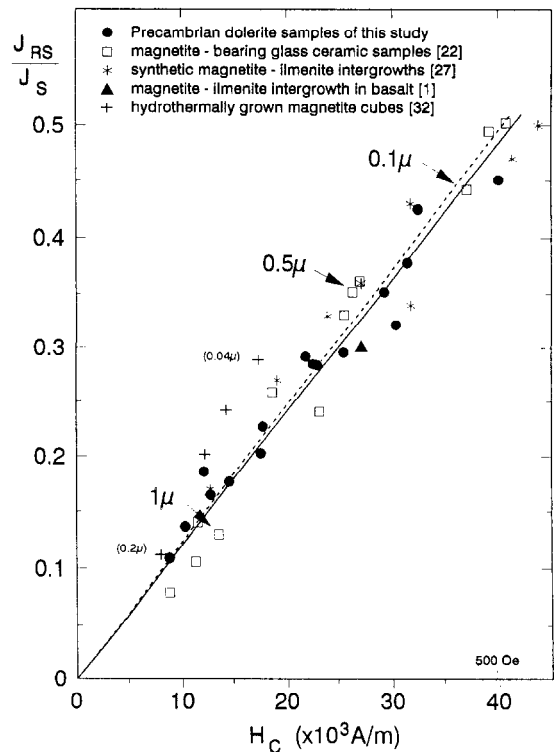


Fig. 3. The ratio of saturation remanence, J_{RS} , to saturation magnetization, J_s , versus coercive force, H_C . The solid line is the least-squares fit forced through the origin for the dolerites, and the dashed line is the same for the glass ceramic samples. Arrows indicate magnetite grain size estimates [22] for some of the glass ceramic samples. The relatively stress-free hydrothermally grown magnetites are shown by crosses, with some grain size estimates [32] in brackets.

pseudo-single-domain magnetite should dominate, except for the samples with grains $< 0.1 \mu\text{m}$, which should be single-domain. (Worm and Markert place the multidomain to pseudo-single-domain transition at $3 \mu\text{m}$, at which they observe a change in the grain-size dependence of H_C and J_{RS}/J_s .) The data show an approximate proportionality between J_{RS}/J_s and H_C as expected from Eq. (1). The proportionality constant is $\sim 1.2 \times 10^{-5}$ (1.0×10^{-3} in cgs), implying that N is ~ 0.17 on average (2.1 in cgs). This low value of N may be due to most of the magnetite grains having only one or two domain walls each. One or two domain walls each have indeed been imaged in 0.5 – $2 \mu\text{m}$ synthetic magnetite grains in a glass ceramic sample [23], as well

as in 0.5–1 μm natural magnetite grains [24].

Perhaps low N can result from other types of pseudo-single-domain grains, including the vortex domain structures that micromagnetic theory predicts should exist in ~ 0.7 to ~ 0.1 μm magnetite grains [25]. However, for ~ 0.7 μm to ~ 0.2 μm magnetite grains with vortex structures, theory predicts $H_C < 7 \times 10^3 \text{ A/m}$ (90 Oe) and $J_{RS}/J_S < 0.05$ [25], which are much lower than observed for magnetites in this size range in the glass ceramic samples.

The proportionality between J_{RS}/J_S and H_C in Fig. 3 extends to the 0.06 μm grains whose $J_{RS}/J_S = 0.5$, as expected of single-domain grains. The proportionality should not extend farther since J_{RS}/J_S should not exceed 0.5. By 0.03 μm grain size, H_C drops to $4.8 \times 10^3 \text{ A/m}$ (60 Oe), presumably because of abundant superparamagnetic grains [22].

5. The dolerite samples

As shown by the dots in Fig. 3, J_{RS}/J_S also increases in approximate proportion to H_C in our dolerites. The constant of proportionality and the range of values is not significantly different from that for the glass ceramic samples with non-interacting pseudo-single-domain magnetite particles. This suggests that most of the dolerite samples are dominated by pseudo-single-domain rather than single-domain magnetite. It also implies an average effective $N \approx 0.17$ agreeing with $N \approx 0.14$ (1.8 in cgs) determined for the Matachewan sample (SD78) using low temperature variation of susceptibility [26].

Lewis [27] synthesized titanomagnetite samples with X from 0.2 to 1, and oxidized them in air at 600°C. Judging by the Curie points, which rose to near that of pure magnetite, magnetite–ilmenite intergrowths were probably produced, although other phases are possible [28]. His hysteresis data for these samples (shown by stars in Fig. 3) resemble data for our dolerites. This suggests that the relation between J_{RS}/J_S and H_C displayed in Fig. 3 should be common in igneous rocks containing grains of intergrown magnetite–ilmenite. This is supported by the J_{RS}/J_S versus H_C plot of Dunlop [29] for mafic igneous rocks (Icelandic basalts, Steen's Mountain basalts and Coronation sills) with Curie points near

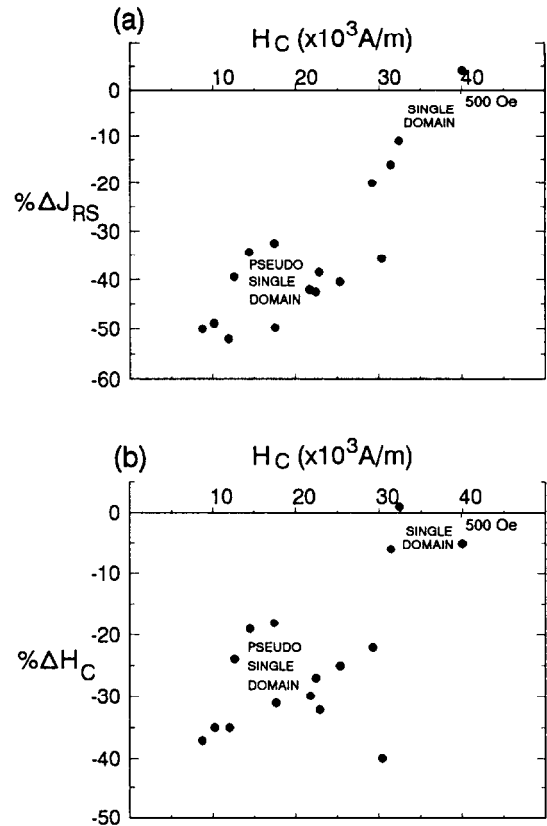


Fig. 4. (a) The percent change in saturation remanence ($\% \Delta J_{RS}$) of the dolerites on demagnetizing with a cooling cycle to 77K is shown versus coercive force (H_C). (b) The percent change in coercive force ($\% \Delta H_C$) on cooling the dolerite samples to 140K is shown versus coercive force.

that of pure magnetite and H_C as high as $19.9 \times 10^3 \text{ A/m}$ (250 Oe).

For our dolerites, the percent change observed in J_{RS} , when demagnetized by a cooling cycle to 77K, is plotted versus H_C in Fig. 4a. The three highest H_C samples (9144, 4305 and 5901) show little demagnetization (8% on average), as expected if they are dominated by single-domain magnetite with high shape anisotropy. The rest of the samples (except possibly 9102) show significantly greater demagnetization, averaging 40%. However, this is smaller than the 70% or more of low temperature demagnetization reported for multidomain crushed magnetite grains [12,30]. This supports the dominance of pseudo-single-domain magnetite in most of the dolerite samples.

A more definitive discrimination between single-

domain and pseudo-single-domain magnetite should be possible by studying the variation of H_C on cooling. For the Matachewan dolerite sample (SD78), I have previously shown that cooling to $\sim 125\text{K}$ causes H_C to decrease in approximate proportion to $\bar{\lambda}_S/J_S$, as expected if H_C is controlled by internal stresses opposing domain wall motion [9,10]. (The variation of polycrystalline saturation magnetostriction $\bar{\lambda}_S$ on cooling used in my past and present studies is from the measurements of Bickford et al. [31]). This supports the dominance of pseudo-single-domain rather than single-domain magnetite, since H_C for the latter would likely be controlled by J_S , through shape anisotropy, and should show little change on cooling. The percent change in H_C on cooling to 140K (well above the Verwey transition) is plotted in Fig. 4b. Only the three dolerites of highest H_C show little decrease (averaging 3%), suggesting the dominance of single-domain magnetite with shape anisotropy. For the rest of the dolerites, the decrease in H_C on cooling to 140K is much larger, averaging 29%. This agrees with the 30% decrease in $\bar{\lambda}_S/J_S$ on cooling to 140K, suggesting that magnetostrictive control of H_C , perhaps through internal stresses opposing domain wall motion, may dominate in the dolerites until H_C exceeds $30 \times 10^3 \text{ A/m}$ (380 Oe). This suggestion will be tested in a forthcoming paper using measurements of H_C and J_{RS}/J_S as a function of low temperature, as in [10].

The great similarity between the glass ceramic and the dolerite plots in Fig. 3 suggests that the magnetite particle size in the glass ceramic samples may roughly predict the particle size to which the magnetite is subdivided by ilmenite in a dolerite of similar J_{RS}/J_S and H_C . This requires testing, since the internal stresses may differ (although they are probably high in both). Also, the magnetite particles in the dolerites may be considerably more elongated.

Davis and Evans [1] observed $J_{RS}/J_S = 0.30$ and $H_C = 22.3 \times 10^3 \text{ A/m}$ (280 Oe) in basalt with magnetite-ilmenite intergrowth. The corresponding glass ceramic sample has $\sim 0.5 \mu\text{m}$ magnetite particles. This does agree with the average dimension of the $\sim 1.2 \mu\text{m} \times \sim 0.2 \mu\text{m}$ acicular magnetite particles observed in the intergrown grains (width from [1], elongation from [2]).

Polished sections of all the dolerites were exam-

ined with the SEM in backscattered mode. Particle size estimation is very difficult because of the variability of the intergrowth texture. Also, for dolerites with $H_C > 23 \times 10^3 \text{ A/m}$ (290 Oe), the intergrowth becomes finer than in sample 4602 (Fig. 1) and difficult to resolve. However, magnetite particle size in the intergrown grains does generally seem to decrease as H_C increases. Also, for most dolerites with $H_C < 23 \times 10^3 \text{ A/m}$, the average magnetite particle size does seem to be within a factor of 2 of that of the ceramic glass samples of similar H_C and J_{RS}/J_S . In contrast, the size of hydrothermally grown magnetite cubes [32] of similar H_C and J_{RS}/J_S is an order of magnitude smaller (Fig. 3), perhaps because of much lower internal stresses.

6. Effect of interaction between single-domain particles

In the above discussion of the dolerites, magnetic interaction between the magnetite particles in each intergrown grain has been neglected. This requires justification because, although the magnetite particles are well separated by ilmenite, they are close together. Consider the case of single-domain magnetite particles in intergrown grains.

For a given intergrown grain (Fig. 5), interaction of the magnetite particles can be considered to have two effects. One effect is that of magnetic poles on the *surface* of the intergrown grain which produce a self-demagnetizing field in the grain of magnitude $fNJ/4\pi \times 10^{-7}$ (fNJ in cgs [1]). Here f is the volume fraction of magnetite in the grain, J is the net magnetization of this magnetite fraction and N is $1/3$ ($4\pi/3$ in cgs) for a spherical grain. The other effect is that of magnetic poles *inside* the intergrown grain. From the theory of Bertram and Bhatia [3], these poles should produce a magnetizing field that approximately cancels the self-demagnetizing field, assuming an equidimensional grain containing randomly oriented, elongated, single-domain particles (a poor assumption for a single grain, but perhaps adequate for the assemblage of grains in a rock). If so, a correction for the self-demagnetizing field of the surface poles is not necessary. However, Davis and Evans [1] did apply a correction, in effect argu-

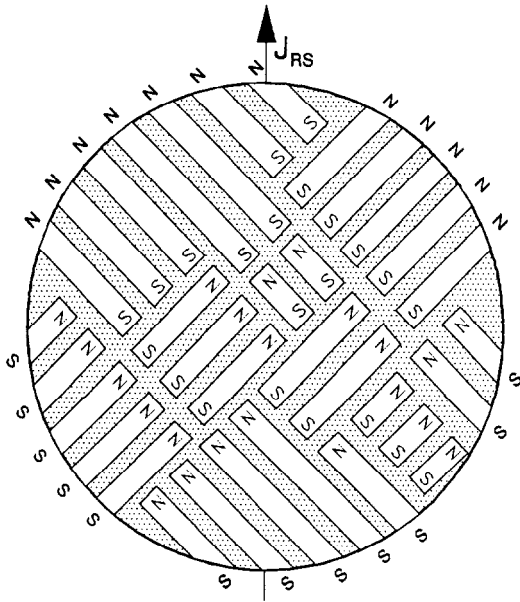


Fig. 5. A schematic diagram of an equidimensional magnetite grain subdivided into single-domain particles by ilmenite lamellae (dotted) and given a saturation remanence J_{RS} . Magnetic poles (N and S) on the surface of the intergrown grain (which produce a self-demagnetizing field in the grain's interior) are shown in bolder letters than magnetic poles within the grain (whose net effect is contentious).

ing that the poles inside the intergrown grains produce no net field.

Davis and Evans [1] corrected for the self-demagnetizing field due to poles on the surface of intergrown grains by drawing a new J axis with a slope of $4\pi \times 10^{-7}/fN$ ($1/fN$ in cgs) on the hysteresis loop (Fig. 2). The height at which this new J axis cuts the hysteresis loop gives the corrected saturation remanence J_{RS}^* . Correcting their basalt containing magnetite-ilmenite intergrowth caused a J_{RS}/J_S of 0.30 to become a J_{RS}^*/J_S of 0.51. They interpreted this as evidence that the magnetite was single-domain.

The correction method of Davis and Evans [1] was applied (using f from Table 1) to our three dolerites of highest H_C , which are likely dominated by single-domain magnetite. Before correction, the average $J_{RS}/J_S = 0.42 \pm 0.04$. After correction, the average $J_{RS}^*/J_S = 0.66 \pm 0.06$, which is much higher than the $J_{RS}^*/J_S = 0.5$ predicted by Davis and Evans

[1]. In contrast, the theory of Bertram and Bhatia [3] would predict $J_{RS}/J_S = 0.5$ before correction, and would consider the correction of Davis and Evans [1] unnecessary, predicting $J_{RS}^*/J_S = 0.68$ if it were applied (assuming that the grains contain randomly oriented, elongated (4 to 1), single-domain particles with $f = 0.5$). Our observations are in better agreement with the theory of Bertram and Bhatia [3]. This justifies our neglecting magnetic interaction between single-domain magnetite particles in intergrown grains in dolerites (as a first approximation).

Note, however, that Davis [2] opposed the theory of Bertram and Bhatia [3]. For a set of artificial samples with various fractions of elongated ($0.5 \times 0.08 \mu\text{m}$) single-domain magnetite particles, he measured J_{RS}/J_S corrected for the self-demagnetizing field of poles on the sample surface. As the volume fraction of magnetite was increased from 0.05 to 0.5, he found that corrected J_{RS}/J_S remained at 0.5, whereas the theory of Bertram and Bhatia [3] predicts that corrected J_{RS}/J_S should increase from 0.5 to 0.65. However, for his most dilute samples (0.005 volume fraction of magnetite) corrected $J_{RS}/J_S = 0.3$, which he disregarded as too low because of probable clumping of the magnetite particles. It seems likely that all his artificial samples suffered from clumping of magnetite particles and that they do not satisfactorily model the well separated magnetite particles in intergrown grains in mafic rocks.

7. Effect of interaction between pseudo-single-domain particles

There is experimental support for neglecting particle interaction in equidimensional intergrown grains containing pseudo-single-domain or multidomain magnetite particles.

Dankers and Sugiura [33] prepared equidimensional samples with different concentrations of crushed magnetite with various particle sizes from multidomain ($\sim 125 \mu\text{m}$, $\sim 65 \mu\text{m}$, and $\sim 27 \mu\text{m}$) to possible pseudo-single-domain ($\sim 17 \mu\text{m}$ and $\sim 7 \mu\text{m}$). For all these grain sizes, J_{RS}/J_S was little affected by varying the volume concentration of magnetite from 0.002% to 100%. Schmidbauer and Veitch [34] prepared spherical samples with various

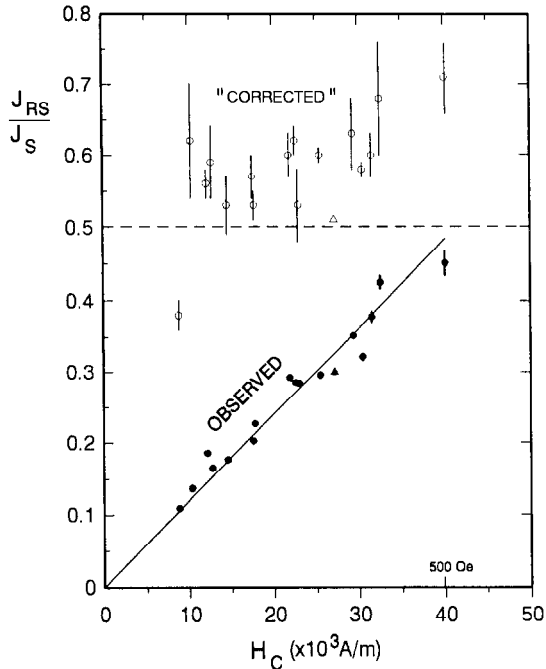


Fig. 6. Comparing J_{RS}/J_S before (dots and filled triangles) and after (circles and open triangles) 'correction' for the self-demagnetizing field of magnetic poles on the surface of grains of magnetite intergrown with ilmenite (or silicate). The correction method is shown in Fig. 2. The dolerites are represented by circles with error bars and the basalt of Davis and Evans [1] by triangles.

concentrations of *well dispersed* $\sim 0.3 \mu\text{m}$ magnetite spheroids that should be pseudo-single-domain. As the volume concentration of magnetite particles was increased from 2.5% to 20%, J_{RS}/J_S remained equal to about 0.25. Presumably, in both sets of experiments, the self-demagnetizing field due to poles on the surface of the sample (which was not corrected for) was approximately cancelled by a magnetizing field due to poles inside the sample.

Evidence for a magnetizing field due to poles inside the intergrown grains in our dolerites comes from correcting for the self-demagnetizing field of surface poles using the method of Davis and Evans [1] (Fig. 2) and obtaining J_{RS}^*/J_S values that are unreasonably high. The J_{RS}^*/J_S values are plotted as circles versus H_C in Fig. 6. Note that J_{RS}^*/J_S significantly exceeds 0.5 for most of the samples. This is unreasonably high, particularly for those dolerites which cannot be dominated by single-do-

main magnetite with shape anisotropy because they show a large decrease in H_C on cooling.

8. Conclusion

In our dolerites with coercive force (H_C) between 9×10^3 and 30×10^3 A/m (110 and 380 Oe), the ratio of saturation remanence to saturation magnetization (J_{RS}/J_S) varies in approximate proportion to H_C . The size of the proportionality constant is consistent, with the magnetite being pseudo-single-domain with one or two domain walls per particle. The dominance of pseudo-single-domain magnetite in most of the dolerites is supported by the size of the low temperature demagnetization (40% on average) shown by J_{RS} when cycled to 77K. The 29% average decrease in H_C on cooling to 140K in these samples is consistent with magnetostrictive control of H_C , perhaps through internal stresses opposing domain wall motion. Only for the three dolerites with H_C above 30×10^3 A/m (380 Oe) do J_{RS} and H_C show little change on cooling, suggesting that their magnetite particles are single-domain with shape anisotropy dominating. This suggests that pseudo-single-domain magnetite, perhaps with domain wall motion impeded by internal stresses, is palaeomagnetically more important than single-domain magnetite with shape anisotropy, in these Precambrian dolerites (and possibly in most others).

The magnetite grains in our dolerites are subdivided into fine particles, usually by ilmenite lamellae, as seems common in mafic igneous rocks of high coercivity. Correcting for the self-demagnetizing field of magnetic poles on the surface of such intergrown magnetite-ilmenite grains (as in [1]) is not recommended. In most of our dolerites J_{RS}/J_S becomes unreasonably high after this correction. It is better to neglect magnetic interaction in intergrown grains (as a first approximation), and use the observed values of J_{RS}/J_S , uncorrected. However, interaction in intergrown grains with single-domain particles may reduce J_{RS}/J_S by $\sim 15\%$ from 0.5, making it difficult to use J_{RS}/J_S to distinguish single-domain from high coercivity pseudo-single-domain magnetite particles. Fortunately, low temperature demagnetization of J_{RS} can help distinguish the two domain states, since there should be relatively

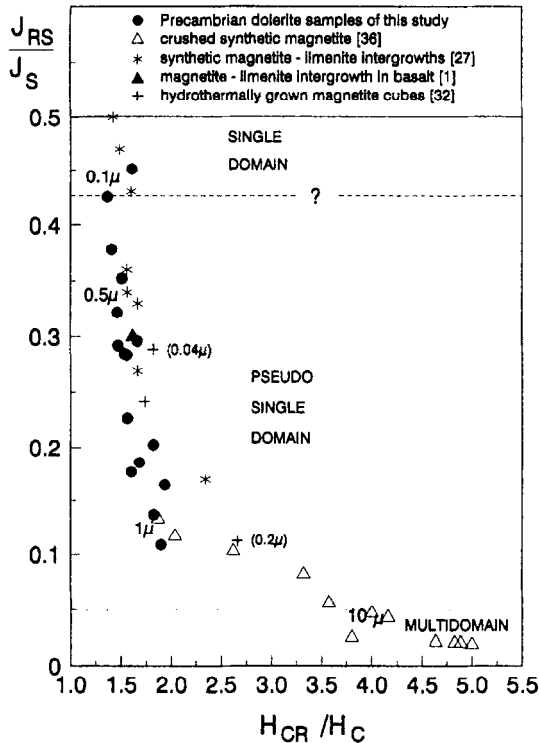


Fig. 7. A plot of J_{RS}/J_S versus H_{CR}/H_C . The size estimates in microns are for the high internal stress magnetite grains in the synthetic samples of Worm and Markert [22] and Day et al. [36] and may give a rough estimate of the size of magnetite particles in intergrown magnetite–ilmenite grains in the dolerites. (The relatively stress-free hydrothermally grown magnetites of Dunlop [32] are shown by crosses with size estimates in brackets.)

little demagnetization for single-domain magnetite with high shape anisotropy.

Plots of J_{RS}/J_S versus H_C (Fig. 3) or J_{RS}/J_S versus H_{CR}/H_C (Fig. 7) are sometimes used for magnetic granulometry [35,36]. If intergrown magnetite–ilmenite grains dominate, note that the size estimate will be that of the magnetite particles within the intergrown grains. Furthermore, the magnetite particles will probably have high internal stresses, making the glass ceramic samples of Worm and Markert [22] a better analogue than hydrothermally grown magnetite.

Acknowledgements

This research was supported by a grant from the Natural Sciences and Engineering Research Council of Canada. B. Ryan of the Department of Natural

Resources of the Government of Newfoundland and Labrador and K.L. Buchan of the Geological Survey of Canada are thanked for help in obtaining the dolerite samples. I thank G.M. English, R.I. Mackay, A.J. Gollop and S.C. Hodych for help with the low temperature measurements and P.J.A. McCausland, P.V. King and C.J. Emerson for help with the electron microprobe and SEM analyses. R.R. Pätzold is thanked for measuring H_{CR} and magnetite content and for preparing the diagrams. [RV]

References

- [1] P.M. Davis and M.E. Evans, Interacting single-domain properties of magnetite intergrowths, *J. Geophys. Res.* 81, 989–994, 1976.
- [2] P.M. Davis, Effects of interaction fields on the hysteretic properties of assemblies of randomly oriented magnetic or electric moments, *J. Appl. Phys.* 51, 594–600, 1980.
- [3] H.N. Bertram and A.K. Bhatia, The effect of interactions on the saturation remanence of particulate assemblies, in: *IEEE Trans. Magnetics*, MAG-9, 127–133, 1973.
- [4] E. Larson, M. Ozima, M. Ozima, T. Nagata and D. Strangway, Stability of remanent magnetization of igneous rock, *Geophys. J.R. Astron. Soc.* 17, 263–292, 1969.
- [5] K.L. Buchan and H.C. Halls, Palaeomagnetism of Proterozoic mafic dyke swarms of the Canadian Shield, in: *Mafic Dykes and Emplacement Mechanisms*, A.J. Parker, P.C. Rickwood and D.H. Tucker, eds., pp. 209–230, Balkema, Rotterdam, 1990.
- [6] T.E. Krogh, F. Corfu, D.W. Davis, G.R. Dunning, L.M. Heaman, S.L. Kamo, N. Machado, J.D. Greenough and E. Nakamura, Precise U–Pb isotopic ages of diabase dykes and mafic to ultramafic rocks using trace amounts of baddeleyite and zircon, in: *Mafic Dyke Swarms*, H.C. Halls and W.F. Fahrig, eds., *Geol. Assoc. Can. Spec. Pap.* 34, 147–152, 1987.
- [7] J.W. Graham, Changes of ferromagnetic minerals and their bearing on magnetic properties of rocks, *J. Geophys. Res.* 58, 243–260, 1953.
- [8] D.W. Strangway, E.E. Larson and M. Goldstein, A possible cause of high magnetic stability in volcanic rocks, *J. Geophys. Res.* 73, 3787–3795, 1968.
- [9] J.P. Hodych, Evidence for magnetostrictive control of intrinsic susceptibility and coercive force of multidomain magnetite in rocks, *Phys. Earth Planet. Inter.* 42, 184–194, 1986.
- [10] J.P. Hodych, Magnetic hysteresis as a function of low temperature in rocks: evidence for internal stress control of remanence in multi-domain and pseudo-single-domain magnetite, *Phys. Earth Planet. Inter.* 64, 21–36, 1990.
- [11] A.F. Buddington and D.H. Lindsley, Iron–titanium oxide minerals and synthetic equivalents, *J. Petrol.* 5, 310–357, 1964.
- [12] J.P. Hodych, Low-temperature demagnetization of saturation

- remance in rocks bearing multidomain magnetite, *Phys. Earth Planet. Inter.* 66, 144–152, 1991.
- [13] Ö. Özdemir and D.J. Dunlop, The effect of oxidation on the Verwey transition in magnetite, *Geophys. Res. Lett.* 20, 1671–1674, 1993.
- [14] G.M. English, A novel vibrating-sample magnetometer used to measure magnetic hysteresis of rock at low temperature, M.Sc. Thesis, Memorial Univ. Newfoundland, 1995.
- [15] J. Gee and D.V. Kent, Magnetic hysteresis in young mid-ocean ridge basalts: dominant cubic anisotropy?, *Geophys. Res. Lett.* 22, 551–554, 1995.
- [16] R.A. Wiebe, Proterozoic basalt dikes on the Nain anorthosite complex, Labrador, *Can. J. Earth Sci.* 22, 1149–1157, 1985.
- [17] D.W. Collinson, *Methods in Rock Magnetism and Paleomagnetism*, 503 pp., Chapman and Hall, London, 1983.
- [18] B.D. Cullity, *Introduction to Magnetic Materials*, 666 pp., Addison-Wesley, Reading, MA, 1972.
- [19] E.C. Stoner and E.P. Wohlfarth, A mechanism of magnetic hysteresis in heterogeneous alloys, *Philos. Trans. R. Soc. London A* 240, 599–642, 1948.
- [20] L. Néel, Some theoretical aspects of rock magnetism, *Adv. Phys.* 4, 191–242, 1955.
- [21] D.J. Dunlop, On the demagnetizing energy and demagnetizing factor of a multidomain ferromagnetic cube, *Geophys. Res. Lett.* 10, 79–82, 1983.
- [22] H.U. Worm and H. Markert, Magnetic hysteresis properties of fine particle titanomagnetites precipitated in a silicate matrix, *Phys. Earth Planet. Inter.* 46, 84–92, 1987.
- [23] C.E. Geiss, F. Heider and H. Soffel, Magnetic domain observations on magnetite and titanomaghemite grains (0.5–10 μm), *Geophys. J. Int.* 124, 75–88, 1996.
- [24] P.P.K. Smith, The application of Lorentz electron microscopy to the study of rock magnetism, *Conf. Ser. Inst. Phys.* 52, 125–128, 1980.
- [25] W. Williams and D.J. Dunlop, Simulation of magnetic hysteresis in pseudo-single-domain grains of magnetite, *J. Geophys. Res.* 100, B3, 3859–3871, 1995.
- [26] J.P. Hodych, Determination of self-demagnetizing factor N for multidomain magnetite grains in rock, *Phys. Earth Planet. Inter.* 41, 283–291, 1986.
- [27] M. Lewis, Some experiments on synthetic titanomagnetites, *Geophys. J.R. Astron. Soc.* 16, 295–310, 1968.
- [28] P. Tucker and W.O. O'Reilly, The laboratory simulation of deuteritic oxidation of titanomagnetites: effect on magnetic properties and stability of thermoremanence, *Phys. Earth Planet. Inter.* 23, 112–133, 1980.
- [29] D.J. Dunlop, Determination of domain structure in igneous rocks by alternating field and other methods, *Earth Planet. Sci. Lett.* 63, 353–367, 1983.
- [30] R.L. Hartstra, Grain size dependence of initial susceptibility and saturation magnetization-related parameters of four natural magnetites in the PSD–MD range, *Geophys. J.R. Astron. Soc.* 71, 477–495, 1982.
- [31] L.R. Bickford Jr., J. Pappis and J.L. Stull, Magnetostriction and permeability of magnetite and cobalt-substituted magnetite, *Phys. Rev.* 99, 1210–1214, 1955.
- [32] D.J. Dunlop, Hysteresis properties of magnetite and their dependence on particle size: a test of pseudo-single-domain remanence models, *J. Geophys. Res.* 91, B9, 9569–9584, 1986.
- [33] P. Dankers and N. Sugiura, The effects of annealing and concentration on the hysteresis properties of magnetite around the PSD–MD transition, *Earth Planet. Sci. Lett.* 56, 422–428, 1981.
- [34] E. Schmidbauer and R.J. Veitch, Anhysteretic remanent magnetization of small multidomain Fe_3O_4 particles dispersed in various concentrations in a non-magnetic matrix, *J. Geophys.* 48, 148–152, 1980.
- [35] K.L. Verosub and A.P. Roberts, Environmental magnetism: past, present, and future, *J. Geophys. Res.* 100, B2, 2175–2192, 1995.
- [36] R. Day, M. Fuller and V.A. Schmidt, Hysteresis properties of titanomagnetites: grain-size and compositional dependence, *Phys. Earth Planet. Inter.* 13, 260–267, 1977.