27. INFERENCES ON THE MAGNETIC DOMAIN STATE OF LEG 37 BASALTS

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INTRODUCTION

We report results of the magnetic experiments conducted on samples from Leg 37 of the Deep Sea Drilling Project. The objective of the experiments is to infer the domain state of the magnetic minerals of the oceanic Layer 2 basalts. The following magnetic properties were studied: Rayleigh loops in low fields; high-field hysteresis parameters at room temperature and at liquid nitrogen temperature; temperature dependence of low-field susceptibility k (k-T curves) and of high-field magnetic moment (M-T curves); and the comparison of AF demagnetization characteristics of natural remanence with those of isothermal saturation remanence in the way suggested by Lowrie and Fuller (1971). Samples were available from Hole 332B and Sites 334 and 335. Results of each experimental study are reported individually and a brief summary with conclusions is given.

EXPERIMENTAL RESULTS

Rayleigh Loops

Hysteresis loops in a field of 10 oe, usually called Rayleigh loops, were obtained using apparatus described by Radhakrishnamurty et al. (1969). Radhakrishnamurty and Sastry (1970) observed open or constricted Rayleigh loops in many, especially Cenozoic, basalts and attributed them to the presence of interacting "very fine single-domain grains" with relaxation times of the order of experimental time, i.e., grains that lie in the superparamagnetic (SP) size range. Although Rayleigh loops may arise in other ways, e.g., from large multidomain (MD) grains, the loops in that case should have BH_c/A values in the range 0.03 to 0.1 (Néel, 1955), where A and B are the Rayleigh constants and H_c is the bulk coercivity; such loops are too narrow to be observed with standard equipment. Radhakrishnamurty and Sastry point out that, since the BH_c/A values ($\sim$20) for their samples showing wide loops exceed the theoretical MD values by two or three orders of magnitude, the actual loops are unlikely to be due to MD grains, a conclusion supported by Néel (1970).

Based on low-field hysteresis, the samples available from Leg 37 can be grouped as follows (mainly as in Deutsch and Pätzold, 1975):

Type I: Open Rayleigh loops: only one sample (332B-11-1) showed this.

Type II. For all other samples, the Rayleigh-loop tracer produced a straight line; i.e., Rayleigh loops were absent. For most of these samples the slope of this line was relatively small, corresponding to low initial susceptibility, k (Type IIa). However, for five samples (332B-35-1, 4-7 cm; 332B-35-4, 23-30 cm; 332B-37-1, 126-128 cm; 332B-37-2, 102-105 cm; and 334-26-2, 4-7 cm) the slope and hence k are relatively large (Type IIb). As will be seen, subtypes IIa and IIb are distinct also in their high-field hysteresis behavior.

High-field Hysteresis

Hysteresis loops were also obtained in fields of the order of 1200 oe using apparatus described by Likhite et al. (1965). Dunlop (1969), Radhakrishnamurty et al. (1971), and Lowrie and Fuller (1971), among others, have all recognized the importance of hysteresis studies in knowing the domain structure of the magnetic grains in a rock. Radhakrishnamurty et al. (1972) have successfully extended hysteresis measurements of rocks to liquid nitrogen temperature, and, based on earlier theoretical and experimental work by Bickford (1950), Bean (1955), and Morrish and Watt (1958), have developed "magnetic granulometric" experimental procedures which are quite useful in delineating the single- or multidomain character of samples and in some cases to identify the magnetic minerals in them. Using very similar procedures, we have obtained hysteresis loops for some samples of Leg 37 basalts both at room and liquid nitrogen temperatures. The results for coercivity (H_c) and relative remanence ($J_s r/J_r$) at both 20°C and -196°C are listed in Table 1.

k-T and M-T Curves

Variation of susceptibility with temperature in the range of -196°C to 650°C was measured with an a.c. bridge (Pätzold, 1972) in a low field of 0.31 oe. The M-T curves were obtained in air in fields of 1300 oe, using apparatus described by Deutsch et al. (1971). Results are listed in Table 1.

AF Demagnetization Characteristics and the Lowrie-Fuller Test

In Lowrie and Fuller's (1971) test for distinguishing multidomain from single-domain carriers of remanence, use is made of the distinctive AF demagnetization characteristics of the two domain states. We have applied this test to eight samples of Leg 37 basalts. First, we demagnetized the NRM of the samples up to 900 oe peak field (Table 2). The NRM is quite stable, the directional change being small in all cases, with median destructive fields of the order of 300 oe. The samples were then given IRM in fields of 8600 oe, after which this IRM was demagnetized in alternating fields, again to 900 oe. The results are presented in Table 2 and, for two representative samples, are shown in Figure 1. For all the eight samples, this test showed that the remanence is carried by pseudo single-domain (PSD) (Johnson et al., 1975) or single-domain grains.

TABLE 1
Summary of Experimental Results

Sample (Interval in cm)	k - T Curves Location of Peaks T_p(°C) Heating	Cooling	M - T Curves T_C(°C), Heating	Rayleigh Loops (10 oe)	Hysteresis Room Temperature J_r/J_s	H_c(oe)	-196°C J_r/J_s	H_c(oe)
Hole 332B								
9-2, 92-94				No	0.4	187	0.5	624
11-1, 110-112	90	90	150	Yes	0.2	72	0.6	624
17-1, 22-24			310, 550	No				
22-4, 48-50	190, 405	310	250, 510	No	0.5	204	0.5	480
27-2, 112-114				No				
35-1, 4-7				No	0.3	96	0.3	130
35-1, 46-49			255, 375, 520	No				
35-1, 104-107				No				
35-1, 136-139				No	0.5	132	0.4	384
35-2, 33-36	275, 455	510		No				
35-2, 91-94				No				
35-3, 28-31	300, 510			No				
35-3, 81-84				No	0.5	240	0.25	336
35-4, 23-30			380	No				
36-1, 104-106				No				
36-2, 3-12				No				
36-2, 59-61			425	No				
36-2, 113-115				No	0.7	312	0.5	384
36-3, 46-48				No				
36-3, 143-145				No				
36-5, 120-122				No	0.7	264	0.4	360
36-6, 66-68	410	400	No					
37-1, 73-75				No	0.25	168	0.25	180
37-1, 126-128				No	0.25	132	0.2	120
37-2, 43-45				No	0.5	168	0.5	312
37-2, 102-105				No	0.3	156	0.3	144
42-1, 13-15	450	450	350, 565	No				
Site 334								
20-1, 114-116	230, 470	500		No				
26-2, 4-7	-155, 550	-155, 500	630	No	0.25	120	0.25	144
Site 335								
6-1, 53-55	325, 455	500		No				
6-1, 62-64			360	No				
6-1, 75-77				No				
6-1, 83-85				No				
6-1, 89-91				No	0.45	240	0.5	312
6-1, 94-96				No	0.4	216	0.35	270
6-1, 99-101	250, 510	520		No				
6-1, 105-107	250, 450, 525	525	310	No	0.5	288	0.6	480
6-1, 112-114			285	No				
6-1, 122-124				No				
6-1, 129-131	255, 425	150 - 350		No				
6-2, 5-7				No	0.5	360	0.3	276
10-1, 33-35				No				
10-5, 43-45				No	0.8	372	0.8	528
13-1, 78-88				No				
13-3, 100-111	275, 455, 510	495	550	No				

Note: T_c = True or apparent Curie temperature obtained from temperature dependence of magnetic moment (M-T curves) in 1300 oe, using powdered samples; J_r/J_s = Ratio of remanence to saturation magnetization in 1200 peak oe; H_c = Coercivity.

DISCUSSION

First we discuss two samples (332B-11-1 and 334-26-2) that showed quite exceptional behavior compared with all others. For example, they had the smallest Koenigsberger ratios (Q_n = 2.1, 1.5) and largest k-values (1.67×10^{-3}, 4.22×10^{-3} Gauss/oe); in the other samples, Q_n tended to be one or two orders of magnitude larger, and k one order of magnitude less.

Sample 332B-11-1 (Figure 2)

This was the only sample showing an open Rayleigh loop (Type 1). The k-T curves also are unique, with a spectacular peak on heating at 90°C, followed by very low k values and a lowered peak on cooling. As the loop parameters are similar to values reported by Radhakrishnamurty and Sastry (1970) for open Rayleigh loops, we attribute our loops to the presence

TABLE 2
Lowrie-Fuller Test Results

Field	332B-9-2, 92-94 cm						332B-35-1, 136-139 cm						332B-36-2, 113-115					
	NRM			IRM			NRM			IRM			NRM			IRM		
(peak oe)	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o
NRM	177	-2	1.00	0	0	1.00	343	-23	1.00	0	-2	1.00	230	8	1.00	6	3	1.00
25	175	-5	0.99	0	0	0.95	338	-32	0.95	0	-2	0.86	230	8	1.00	5	3	0.98
50	174	-9	0.94	0	0	0.79	338	-39	0.97	0	-3	0.54	230	8	1.00	5	3	0.93
100	173	-10	0.80	0	1	0.50	339	-41	0.70	0	-3	0.21	230	8	0.98	5	3	0.79
150	173	-12	0.66	0	1	0.29	343	-43	0.52	0	-4	0.14	230	7	0.92	5	3	0.65
200	175	-12	0.47	0	1	0.18	338	-23	0.39	359	-4	0.10	230	7	0.77	5	3	0.52
250	178	-14	0.31	0	1	0.12	339	-43	0.35	359	-4	0.08	230	6	0.63	5	3	0.41
300	175	-8	0.19	0	1	0.09	336	-44	0.27	359	-4	0.07	231	6	0.53	5	3	0.33
400	198	-3	0.17	0	1	0.05	339	-38	0.19	0	-4	0.05	230	3	0.36	5	3	0.22
600	246	-42	0.07	0	1	0.03	338	-44	0.11	359	-4	0.03	231	8	0.17	6	2	0.11
750	–	–	–	0	0	0.02	337	-37	0.09	359	-4	0.02	213	6	0.06	4	3	0.07
900	273	7	0.22	359	0	0.01	312	-47	0.09	359	-4	0.02	224	3	0.05	3	3	0.05

Field	332B-36-5, 120-122 cm						332B-37-1, 126-128 cm						332B-37-2, 43-45					
	NRM			IRM			NRM			IRM			NRM			IRM		
(peak oe)	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o
NRM	213	-4	1.00	356	3	1.00	292	-8	1.00	180	-2	1.00	308	-38	1.00	1	-5	1.00
25	213	-4	1.00	355	3	0.97	292	-9	0.99	180	-1	0.88	307	-39	1.00	1	-5	0.93
50	213	-4	0.99	356	3	0.91	292	-10	0.94	180	-1	0.72	307	-39	1.00	1	-5	0.77
100	213	-4	0.94	356	3	0.75	292	-13	0.71	180	-1	0.40	306	-39	0.82	1	-5	0.46
150	212	-5	0.81	356	3	0.58	292	-17	0.48	180	-1	0.21	305	-39	0.57	1	-5	0.27
200	211	-6	0.61	355	3	0.43	291	-20	0.31	180	-2	0.12	306	-39	0.39	1	-5	0.17
250	210	-8	0.45	366	4	0.31	292	-22	0.21	180	-2	0.07	306	-39	0.27	1	-4	0.10
300	211	-8	0.32	355	4	0.23	290	-22	0.14	180	-1	0.05	306	-39	0.18	1	-4	0.07
400	201	-13	0.17	355	4	0.13	292	-19	0.09	180	-1	0.04	304	-41	0.09	1	-4	0.04
600	184	-13	0.06	356	4	0.05	282	-17	0.05	–	–	–	298	-26	0.03	0	-5	0.01
750	201	-16	0.04	354	5	0.03	263	-23	0.05	–	–	–	320	-43	0.02	2	-3	0.01
900	238	-28	0.02	359	1	0.02	336	-14	0.01	–	–	–	275	18	0.02	0	6	0.01

Field	335-6-1, 89-91 cm						336-5-2, 5-7 cm					
	NRM			IRM			NRM			IRM		
(peak oe)	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o	*D*	*I*	J/J_o
NRM	275	-60	1.00	184	-1	1.00	341	-66	1.00	180	0	1.00
25	274	-60	1.00	184	-1	0.98	341	-66	1.00	180	0	0.99
50	273	-60	0.98	184	-1	0.91	340	-66	0.99	180	0	0.96
100	271	-60	0.91	184	-1	0.75	340	-66	0.98	180	0	0.88
150	269	-60	0.85	184	-1	0.61	340	-66	0.95	180	0	0.78
200	268	-60	0.76	184	-1	0.50	340	-65	0.89	180	0	0.68
250	268	-50	0.55	184	-1	0.40	340	-65	0.80	180	0	0.58
300	266	-60	0.56	184	-2	0.33	340	-65	0.71	180	0	0.50
400	266	-59	0.43	184	-2	0.24	340	-65	0.55	180	0	0.37
600	271	-60	0.23	184	-1	0.13	344	-67	0.29	180	-1	0.20
750	269	-56	0.18	184	-1	0.10	321	-65	0.18	180	0	0.15
900	254	-71	0.10	184	-2	0.07	346	-69	0.13	180	-1	0.10

of an SP fraction, as discussed earlier. The high-field hysteresis loops show a J_r/J_{max} value of 0.2 at 20°C, increasing to 0.6 at -196°C along with an order-of-magnitude increase in H_c. These increases seem to reflect passage of the material through the Verwey transition, indicating SD magnetite or titanomagnetite as the main carrier; this conclusion is compatible with the same material being largely superparamagnetic at 20°C. Possibly the increased size of the hysteresis loop at 20°C after heating and the irreversible features of the *k-T* and *M-T* curves all can be explained by conversion of SP into SD particles due to grain growth or oxidation during heating.

"Type I" behavior very similar to that described above was observed in Leg 34 basalts (Deutsch and Pätzold, 1975), where the majority of the samples produced Rayleigh loops which were explained on the SP model; they showed an excellent one-to-one correlation with *k-T* curves and high-field hysteresis loops of the kind shown in Figure 2.

A polished section made of this sample was observed under an optical microscope (up to ×1000) showing that coarse magnetite or titanomagnetite grains with diameters of several hundred microns, typically ~300 μm, are ubiquitous, though (titano)magnetite grains down to a few microns in size were also found. This finding is similar to that of Ade-Hall and Johnson (1975), who observed characteristically coarse-grained (titano)magnetite in Leg 34 basalts yielding Type I properties. At first sight these results seem to call for

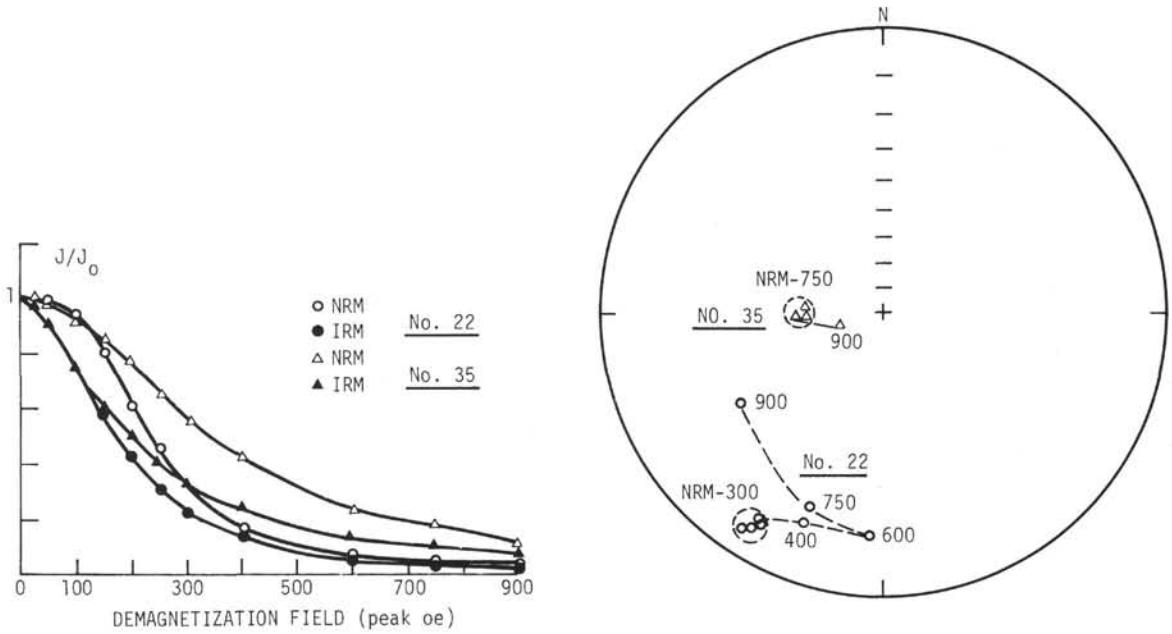

Figure 1. *Left. Normalized AF demagnetization curves of natural remanence and of saturation isothermal remanence produced in a field of 8600 oe for Samples 22 (332B-36-5, 120-122 cm) and 35 (335-6-1, 89-91 cm). Right. Directional changes of NRM of the two samples during AF demagnetization plotted on a Wulff's net.*

linking of observed VRM and other Type I properties in basalts with multidomain (MD) structure, as indeed several authors have done. Against this we argue that the entire SP-SD range is suboptical, so that interpretation of domain structure based on optical (probably even electron) microscopy can be misleading: microscope studies cannot rule out the possible presence of very fine subdivisions in the observed coarse magnetite grains, or of single-domain grains or both. We conclude that the SP model favored by us in explaining the "Type I" properties of Sample 332B-11-1 is not contradicted by the optical findings.

The k peak in Figure 2a constitutes a susceptibility enhancement analogous to Dunlop's (1974) mechanism where high-temperature Hopkinson peaks point to deep-seated induced anomaly sources. In Leg 37 basalts, with only one sample of this type found, this effect is probably unimportant.

Sample 334-26-2 (Figure 3)

This sample gave strikingly different results from the sample previously discussed. The k-T curve clearly indicates multidomain magnetite (Radhakrishnamurty and Deutsch, 1974), showing near-reversibility, an abrupt drop of k at the Curie temperature, and a peak near the Verwey transition (-155°) that would be suppressed in single-domain magnetite due to shape anisotropy. The M-T curve, being obtained on a powder instead of the disc used in k-T experiments, indicates oxidation of original magnetite (titanomagnetite) to hematite, with higher T_c (630°C) and a depressed cooling curve. This is also consistent with an original large-grain or multidomain state (Feitknecht and Gallager, 1970). There is no Rayleigh loop and the high-field loops at 20°C and -196°C are very similar. However, both H_c and J_r/J_{max} of purely multidomain magnetite (titanomagnetite) should be very small at either temperature, though larger at -196°C, whereas stoichiometric single-domain magnetite would have produced at room temperature a loop with J_r/J_{max} of 0.5 and at -196°C a loop similar to that in Figure 2c (middle diagram). Thus the k-T curves and the hysteresis loops indicate that this sample probably contains MD magnetite as well as strongly cation-deficient SD or PSD magnetite. After heating (Figure 3, bottom), H_c increased moderately, probably due to some oxidation.

"Type II" Behavior

The k-T curves on 11 additional samples (Table 1) are different from the "SP" and "MD" curves (Figures 2a, 3a) by peaking initially at intermediate temperatures (~200°-500°C) and by their pronounced irreversibility. Despite large sample-to-sample variations in the location and height of the k peaks, two main kinds of heating curves appear: In Samples 332B-37-2, 102-105 cm (Figure 4a) and 332B-42-1, 13-15 cm, k rises continuously to a single broad peak near 450°C, while Figure 4a (Sample 335-6-1, 105-107 cm) typifies heating curves having two main peaks, shown also by Samples 332B-22-4, 48-50 cm; 332B-35-2, 91-94 cm; 332B-35-3, 28-31 cm; 334-20-1, 114-116 cm; 335-6-1, 112-114 cm; and 335-6-1, 129-131 cm (Table 1), though not all these have a split peak at high temperature.

Type IIa: A grouping into Subtypes IIa and IIb, based on relatively smaller and larger k values measured by Rayleigh-loop tracer, was noted earlier. For Type IIa samples, the high-field hysteresis loops at room temperature typically (Figure 4c, bottom, upper loop) showed large H_c and J_r/J_{max} ratios close to 0.5, indicating SD magnetite or titanomagnetite, with both H_o and J_r/J_{max} increasing substantially below the Verwey transition (lower loop). The pronounced irreversibility shown by the *k-T* and *M-T* heating-cooling curves, and the upswing of the *k-T* cooling curve with decrease in temperature below 0°C, all suggest that relatively unoxidized SD material (as indicated by the hysteresis loops) has become (more) cation-deficient due to heating (Radhakrishnamurty and Deutsch, 1974, figure 3).

Type IIb: Behavior similar to that shown in Figure 4 was observed in most other samples studied by us, except for the following cases: Samples 332B-35-1, 4-7 cm; 332B-35-4, 23-30 cm; 332B-37-1, 126-128 cm; 332B-37-2, 102-105 cm; and 334-26-2, 4-7 cm showed very little change in their hysteresis behavior when cooled to liquid nitrogen temperature. Similar cases of apparent

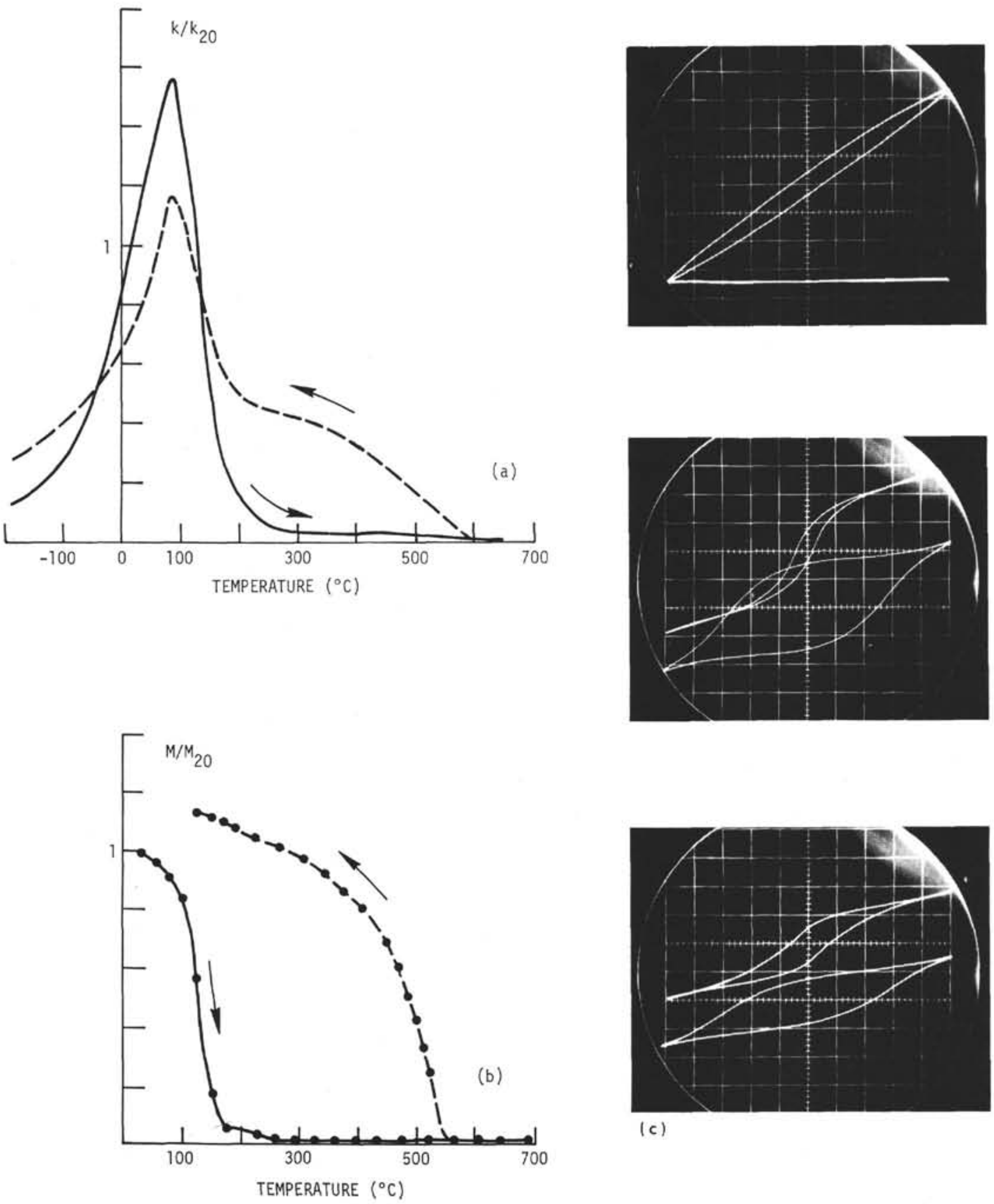

Figure 2. *Sample 332B-11-1, 110-112.* (a) *Normalized susceptibility versus temperature* (k-T *curve*) *in 0.31 oe, heating and cooling.* (b) *Normalized magnetic moment versus temperature* (M-T *curve*) *in 1300 oe, heating and cooling.* M-T *rather than* J_S-T *is quoted as this field is not always sufficient for saturation.* (c)*Top: Photograph of Rayleigh loop in 10 peak oe, with horizontal reference trace. Center: Photograph of hysteresis loops in 1200 peak oe of a fresh sample portion at 20° C (upper trace) and –196° C (lower trace). Bottom: Same as center diagram, measure on sample following* k-T *experiment.*

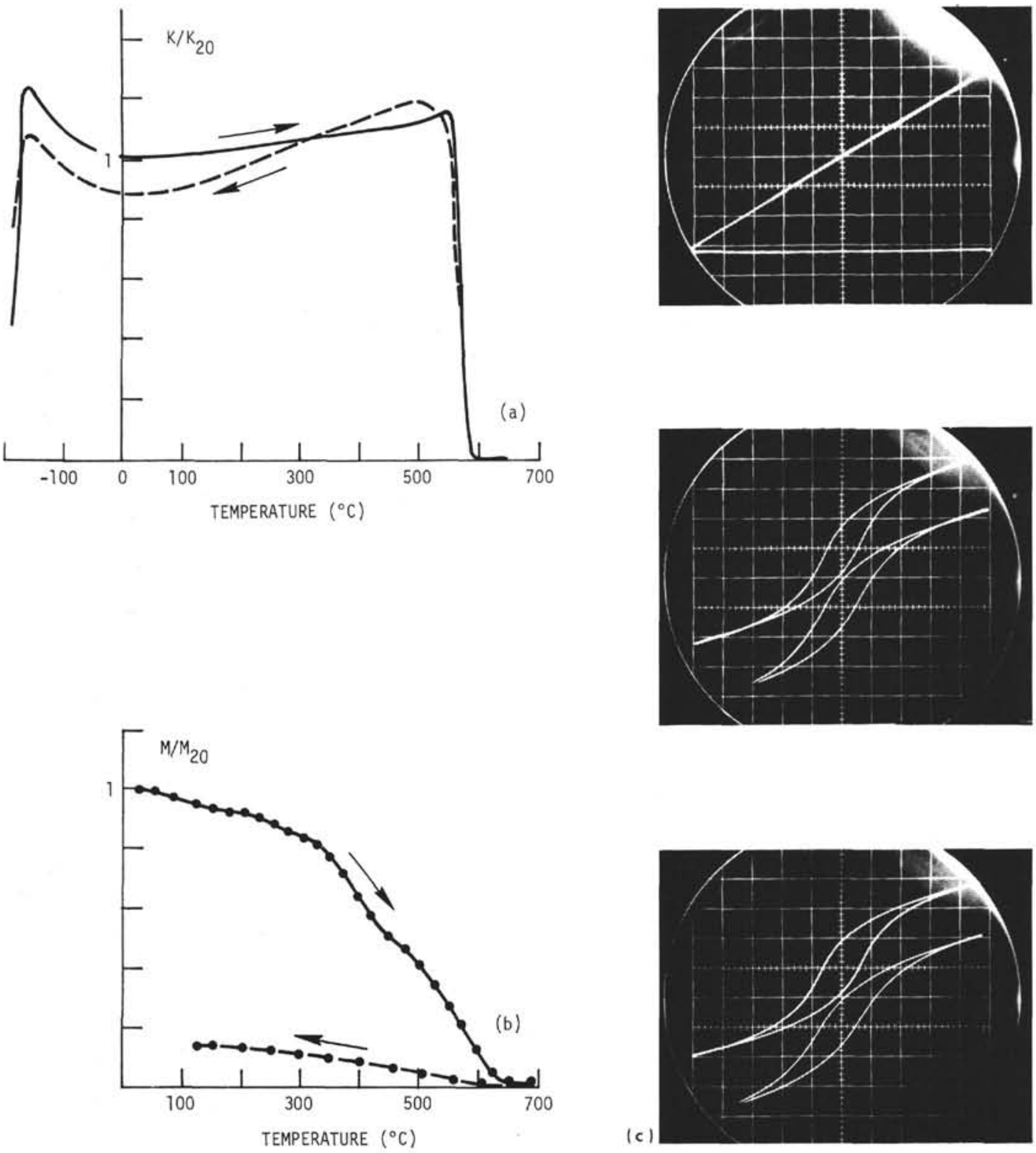

Figure 3. *Sample 334-26-2, 4-7.* (a), (b), (c) *as in Figure 2.*

absence of the Verwey transition in magnetite have been noted in high-field hysteresis experiments with basalts by Radhakrishnamurty et al. (1971) and an explanation in terms of cation-deficiency has been proposed (Radhakrishnamurty and Deutsch, 1974, fig. 2b). Figure 1 shows the results of some of the experiments performed on Sample 332B-37-2, 102-105 cm. The presence of a cation-deficient phase is suggested also by the shape of the *k-T* heating curve, in particular by the decrease in susceptibility as the sample is heated from -196°C to room temperature. The large slope of the low-field hysteresis trace is evident.

SUMMARY AND CONCLUSIONS

We have conducted two types of tests on Leg 37 basalts to infer the domain state of the magnetic minerals contained. The first one involved a measure of the bulk magnetic properties of the rock and included both the susceptibility and hysteresis measurements. The second approach involved a study of the remanence carriers through the Lowrie-Fuller test. Not surprisingly, the Lowrie-Fuller study and most of the hysteresis measurements indicated that the remanence carriers of the samples are of a single-domain nature. On the other hand, low-field susceptibility measurements tend to show up preferentially any magnetic instability, and for this reason the *k-T* curves sometimes indicated different bulk magnetic properties from those inferred on the basis of hysteresis measurements. These apparently conflicting results can be reconciled only by invoking the presence in these basalts of mixtures of grains.

With a single exception, where an MD magnetite fraction was clearly indicated, the magnetic carrier in these samples was identified as pure or cation-deficient single-domain magnetite or titanomagnetite. One sam-

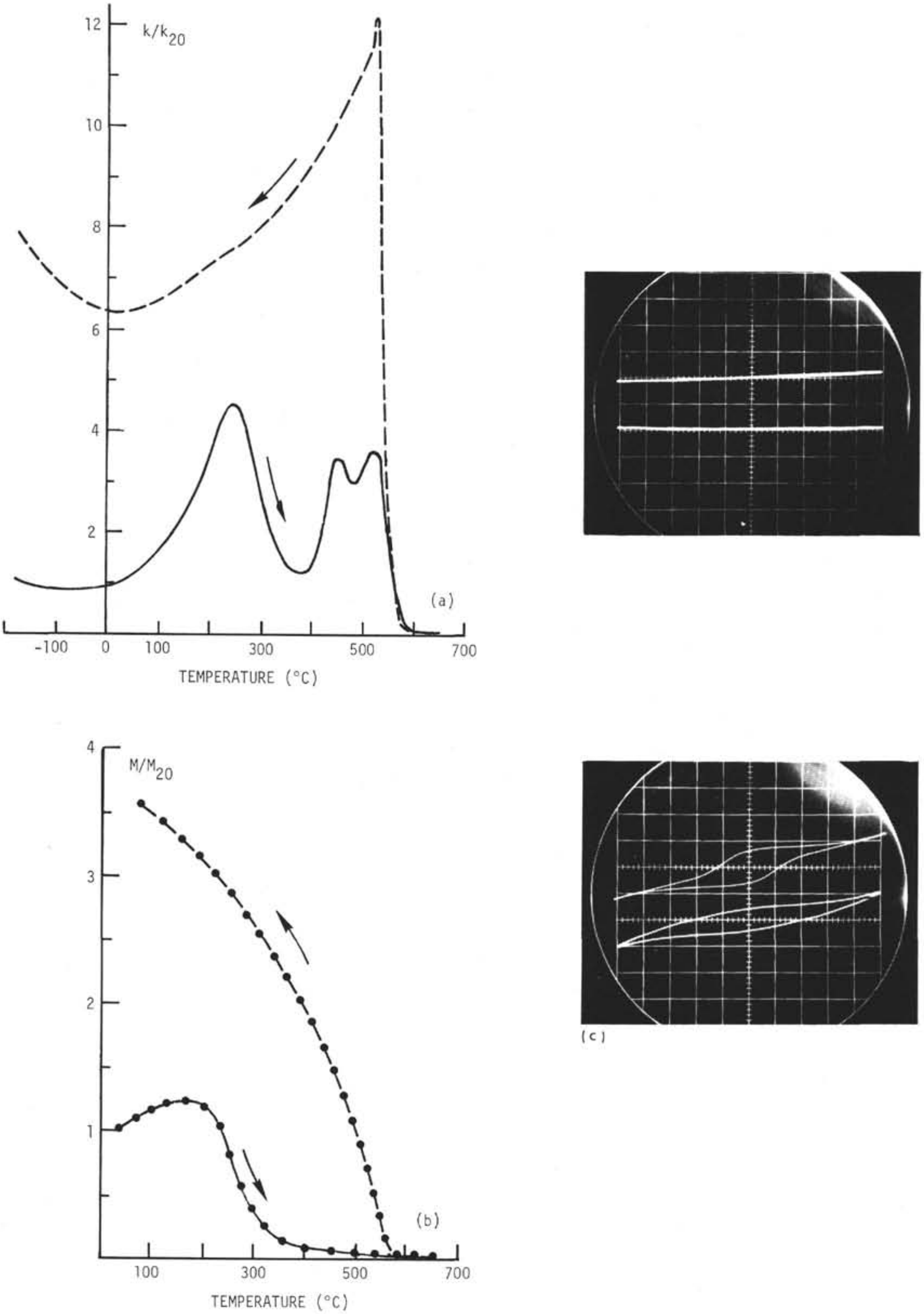

Figure 4. *Sample 335-6-1, 105-107 cm.* (a), (b) *as in Figure* 3a, b. (c) *Top: Photograph of Rayleigh loop in 10 peak oe, with horizontal reference trace. Bottom: Photograph of hysteresis loops in 1200 peak oe of fresh sample at 20°C (upper trace) and –196°C (lower trace).*

ple only showed behavior attributable to grains in the superparamagnetic size range.

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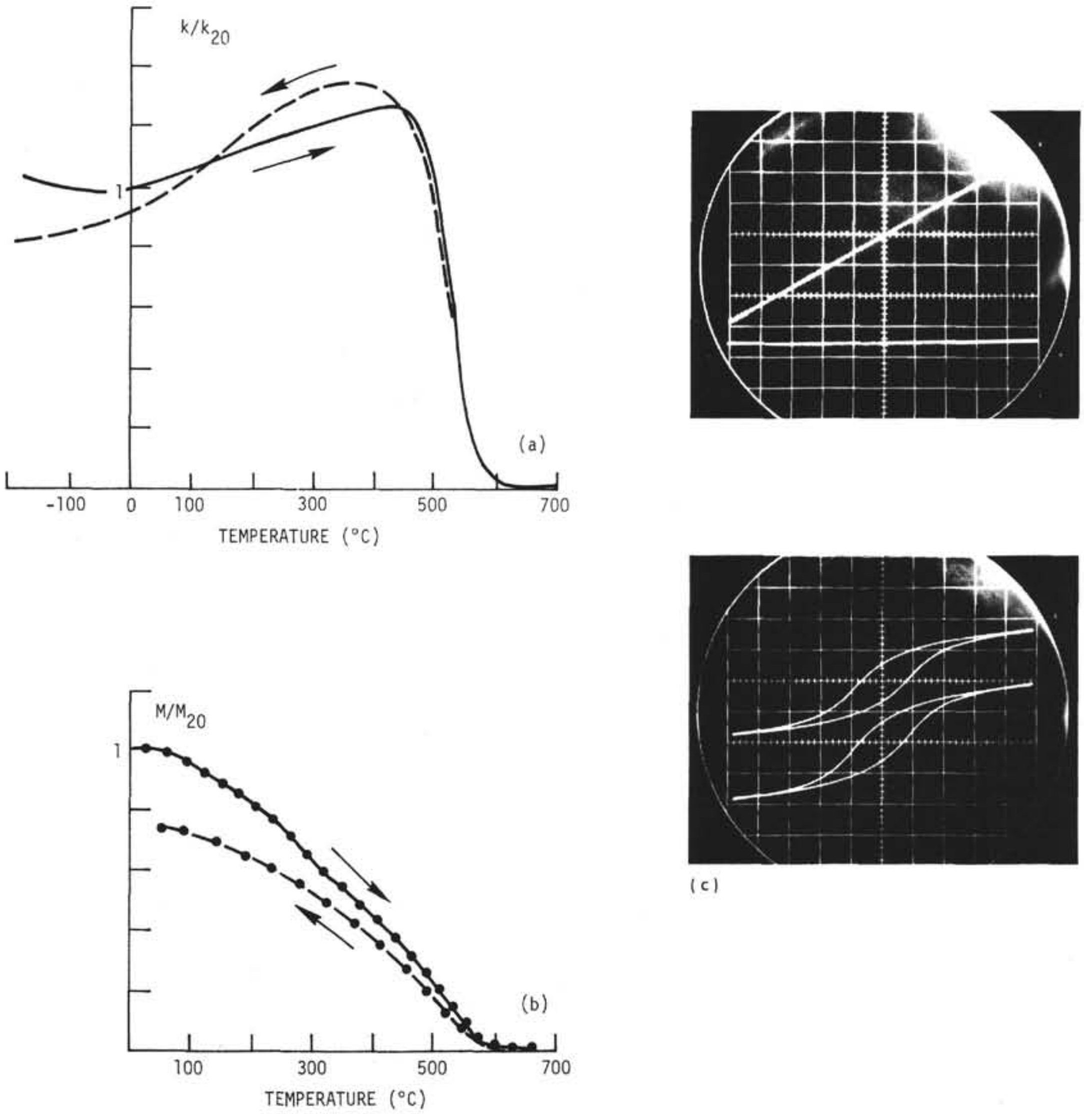

Figure 5. *Sample 332B-37-2, 102-105 cm.* (a), (b), (c) *as in Figure 4.*

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