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Investigations of Some Magnetic and Mineralogical Properties of the Laschamp and Olby Flows, France

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Rock-magnetic, paleomagnetic and petrologic properties of samples from the Laschamp and Olby basalt formations in France were studied to aid in determining the validity of the Laschamp geomagnetic field reversal reported by Bonhommet and Babkine. The Laschamp flow contains ilmenomagnetite, with partial alteration of the magnetite to hematite. Ilmenomagnetite in the Olby flow has largely recrystallized at high temperatures to a composite mozaic intergrowth of pseudobrookite, titanohematite and magnesioferrite, with rare residual magnetite and lamellae of ilmenite. The remanent magnetization is stable and resides primarily in single-domain magnetite particles. Our results indicate that the magnetizations of the Laschamp and Olby flows faithfully record the direction of the ambient magnetic field in which they cooled.

INTRODUCTION

Bonhommet and Babkine (1967) reported that the Laschamp and Olby formations of the Chaîne des Puys volcanic chain of Auvergne, France, were reversely magnetized. Later Bonhommet and Zähringer (1969) and Bonhommet (1970) reported that the ages of the flows are bracketed by 50,000 and 8,000 years before present. Thus they concluded that in the Brunhes Epoch of normal magnetic polarity there is a period, within the last 50,000 years, in which the earth's magnetic field was reversed—the Laschamp event. Such a young magnetic reversal would be an extremely useful stratigraphic index.

Although actively sought by some investigators, we know of no independent confirmation of the Laschamp event, either in hard rock or in sediments. Denham and Cox (1970) sampled sediments in Mono Lake, California, deposited from 30,000 to 12,000 years before present. Although they observed a maximum deviation from an axial dipole of approximately 60°, they concluded that they did not observe the Laschamp event. We measured the magnetization of a sedimentary core from the Bering Sea. The dangerous assumption of constant deposition, together with a ^{14}C date of 16,600 years, led to a sedimentation rate in excess of 25 cm per 1,000 years. The oldest portion of the core had a ^{14}C date in excess of 27,000 years. We sampled the core at 15-cm intervals and found no segment with reversed polarity.

The lack of independent confirmation of the Laschamp event has led to the suggestion that the magnetization of the Laschamp and Olby flows is caused by a self-reversing magnetic mineral. Another possibility is that

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they represent a local field reversal, where, during an epoch of a relatively weak dipole, a nondipole component of reversed polarity dominated the total magnetic vector. From the stability of his samples against alternating field (AF) demagnetization and from the character of their thermal demagnetization, Bonhommet (1970) concluded that the self-reversal hypothesis was highly unlikely. Through the courtesy of N. Bonhommet, we received oriented samples of both the Laschamp and Olby flows. Here we report on our studies further exploring the self-reversal hypothesis and our experiments to ascertain the nature of the minerals carrying the remanence in the Olby and Laschamp formations.

EXPERIMENTS AND RESULTS

We had a dozen samples from four discrete locations in each flow. The virtual geomagnetic pole positions obtained from samples of both flows agreed within a few degrees with those reported by Bonhommet and Zähringer (1969). Also, the stability of the natural remanent magnetization (NRM) against AF demagnetization has a character similar to the example given by Bonhommet (1970). The resistance of the remanence to AF demagnetization revealed high coercivities with $J/J_N = \frac{1}{2}$ occurring near 350 Oe.

Following the AF demagnetization, samples were subjected to total thermoremanent magnetization (TRM) in the laboratory field of .75 Oe. In both flows the magnetization was acquired parallel to the ambient magnetic field—thus these samples are not now self-reversing. Alternating field demagnetization spectra of the laboratory TRM for both flows are very similar to those of the original NRM. This suggests that the magnetic character of these rocks was not drastically altered upon heating and is consistent with the NRM's being largely TRM.

In an attempt to estimate the ambient field in which the Laschamp and Olby formations acquired their magnetizations, we performed a Thellier test (Thellier and Thellier, 1959) with a sample from each flow. Heating in air, we used temperature increments of 100°C until the samples reached 500°C, and increments of 25°C until they reached 600°C. The NRM *vs.* TRM plot was highly nonlinear, and apparently our samples were unsuitable for intensity determination. The thermal demagnetization, however, supports the result of Bonhommet (1970) that the magnetization decreases monotonically with temperature and has a Curie value of 570° ± 10°C.

Whole rock chips were subjected to high field magnetization *vs.* temperature (J–T) cycles. The runs were performed in air at atmospheric pressure. All samples showed a single Curie temperature of 562° ± 10°C as illustrated in Fig. 1.

Subsequently, fresh rock samples were crushed and ground in water to a fine powder from which the magnetic minerals were separated with a hand magnet. X-ray diffraction patterns were obtained using the separated powders of both flows. Both patterns had all the characteristic spinel lines, as well as the major lines of a member of the ilmenite–hematite solid solution series. Both patterns also had peaks corresponding to plagioclase, olivine and augite. The unit cell dimension for the spinel species was calculated to be 8.39 ± 0.01 Å for both Laschamp and Olby flows, a value identical to that of stoichiometric magnetite. The rhombohedral titanohematite phase of the separated powders of both flows had the following cell parameters: a = 5.04 ± .01 Å and c = 13.76 ± .02 Å with c/a = 2.73 ± .01. The unit cell volumes are consistent with the values for hematite which has an ilmenite content of between 10–20%. A titanohematite of such a composition has a Curie temperature between 500°C and 600°C. J–T cycles were performed with the separated powders in helium gas and at atmospheric pressure—usually a slightly oxidizing environment due to incomplete oxygen flushing.

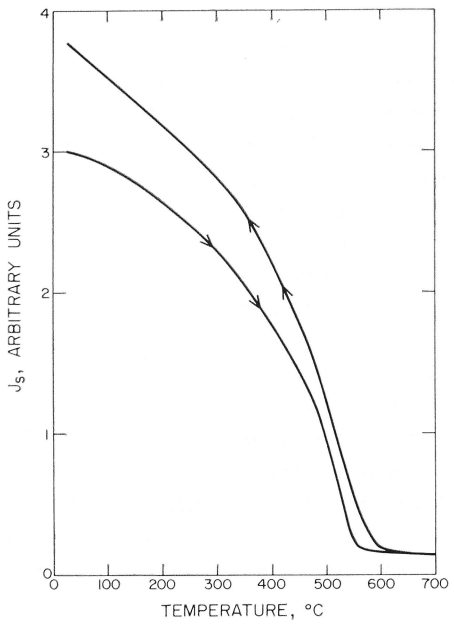

FIG. 1. Saturation magnetization as a function of temperature for sample 285 of the Laschamp flow. The applied field was 7200 Oe and the sample was run in air at atmospheric pressure. The difference in the heating and cooling Curie temperatures is due to the thermal hysteresis of the oven. Samples from the Olby flow exhibited the same behavior.

Single Curie temperatures of 556° ± 10°C were obtained for both flows. The saturation magnetizations at room temperature were 22.2 emu/g and 14.5 emu/g for the Laschamp and Olby separated powders, respectively. At room temperature the saturation magnetization of magnetite is approximately 92 emu/g, while $x\mathrm{FeTiO_3}$ $(1\text{-}x)$ $\mathrm{Fe_2O_3}$ with $x<.20$ is in the antiferromagnetic region of the ilmenite–hematite solid solution series with a feeble, parasitic ferromagnetism superimposed. The separated powders contained significant amounts of plagioclase, olivine and pyroxene, which would reduce the saturation magnetization of a pure magnetic phase by an unknown amount. It is thus evident that the saturation magnetization of the separates is dominated by the magnetite fraction.

To further isolate the predominant magnetic mineral(s), low temperature experiments were performed. Near 130°K, magnetite undergoes a magnetocrystalline anisotropy transition such that saturation isothermal remanent magnetization (IRM) acquired at a temperature below the transition temperature is greatly reduced in multidomain magnetite upon warming to room temperature in a nonmagnetic environment. The same behavior is observed for a room temperature IRM cycled below the transition temperature. It is known that hematite undergoes a magnetic transition near −15°C from a spin-canted moment in the basal plane to an antiferromagnetic moment parallel to the ternary axis (Shull *et al.*, 1951). Remanence-bearing hematite loses its spin-canted magnetization when cooled across this transition temperature—the Morin transition. As the sample is reheated to room temperature, partial recovery occurs. The Morin transition is dependent upon the grain size as well as the impurity concentrations. Haigh (1957) observed that the transition tempera-

ture drops very rapidly with the introduction of small quantities of titanium in artificially prepared samples. With 4-mole percent ilmenite content, the Morin transition, T_m, is at $-60°C$, and at less than 20% ilmenite content the transition appears to hold at $-60°C$. On the other hand, Kaye (1962) has observed the effect of titanium on the transition in natural crystals of hematite and concluded that 0.4% ilmenite was sufficient to depress T_m to $-85°C$, whereas no transition was detected for ilmenite concentrations greater than 2%. Apparently the Ti ion concentration affects the Morin transition in an ill-defined manner. For hematite grains smaller than 0.1 μ, the Morin transition is not observed.

Using these identifying characteristics, we applied the following procedure, as suggested by Nagata (1965). Magnetic separates of both Olby and Laschamp were given a room temperature IRM in an 8 kOe field. Then, the samples were cooled in the residual field (*ca.* 30 Oe) of the vibrating sample magnetometer to liquid nitrogen temperature at which point the samples were again given an IRM in an 8 kOe field in the same direction. Reheating the samples to room temperature produced the behavior shown in Fig. 2. On both the heating and cooling legs a marked decrease in intensity is seen at around 130°K. This is easily identified as the magnetite transition. Since hematite becomes antiferromagnetic below its Morin transition, it should show no response to the low temperature IRM, while still exhibiting a memory for the room temperature IRM. No indication whatsoever was seen for a Morin transition on either the Laschamp or Olby flows.

While being a positive test for magnetite, this strong field IRM, low temperature cycle is not a completely negative test for titanohematite of $x = .10$–$.20$, consistent with Curie temperature, microprobe and x-ray results. Such a Ti ion concentration may be entirely outside the region where the Morin transition occurs. Alternatively, extremely small titanohematite grains could repress the transition. A third possibility could be a dominance of a defect magnetic moment which would be insensitive to low temperature treatments.

To investigate the seat of the actual remanence, samples possessing NRM and samples that were given anhysteretic remanent

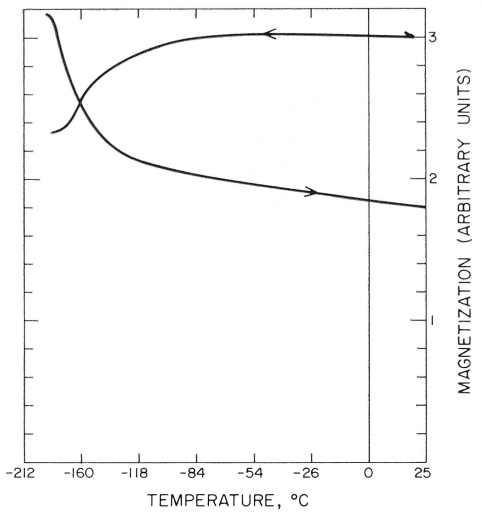

FIG. 2. Low temperature magnetization curves for Laschamp sample 285. The magnetic transition that takes place at $-150°C$ is indicative of magnetite. No Morin transition is evident.

magnetization (ARM) of 12 Oe dc field with peak ac fields of 2500 Oe were subjected to various low temperatures in a field-free space. The magnetizations were measured at room temperature after each cycle. The results are listed in Table 1. The Morin transition is observed neither after the −39°C cycle nor after the −76°C cycle. The much more pronounced decrease in the remanence at liquid nitrogen temperatures is consistent with the presence of magnetite cooled across its magnetocrystalline anisotropy transition temperature near 130°K. The magnetization of Olby sample 269D was observed to increase slightly after each low temperature cycle. In the case of all three temperature cycles the magnetic directions remained unchanged.

These low temperature experiments strongly suggest that the remanence of the Laschamp and Olby flows is held largely in the magnetite. No strong field or remanence experiment provides any positive evidence for a magnetically active titanohematite phase. With no such evidence, we conclude that it is very unlikely that the titanohematite plays any significant role in controlling the remanence in the Laschamp and Olby basalts.

Polished sections of two samples from the lower, reversed, Olby flow (261C, 269) and two samples from the Laschamp reversed flow (279, 285) were examined at high magnification under a reflected-light microscope, and mineral identification was confirmed with an electron microprobe.

In all samples an original homogeneous titanomagnetite crystallized alongside olivine, augite and plagioclase. Subsequent to crystalization, oxidation occurred producing ilmenite lamellae in the magnetite host; these lamellae are preserved in the Laschamp samples, but in the Olby flow, as a result of further oxidation, they have been extensively pseudomorphed by pseudobrookite. Despite much alteration of magnetite to hematite, the Laschamp flow still contains appreciable quantities of magnetite (Fig. 3a). The state of oxidation corresponds to Classes 3 and 4 of Ade-Hall *et al.* (1968). At ×1500 in oil, the magnetite shows an unresolvable mozaic texture, suggesting that it is no longer one-phase. A component of this mozaic is probably the relatively pure fine-grained magnetite responsible for the NRM.

In addition to pseudobrookite, the Olby samples contain well-crystallized titanohematite—to the exclusion of pseudobrookite in many grains—less common magnesioferrite

TABLE 1

THE CHANGES IN REMANENCE AS A FUNCTION OF LOW TEMPERATURE TREATMENT
SAMPLES WERE COOLED TO THE TEMPERATURE INDICATED AND THEN HEATED
TO ROOM TEMPERATURE IN A NONMAGNETIC SPACE

Sample	Type of remanence	$J(T)/J_R$ 20°C	$J(T)/J_R$ −39°C	$J(T)/J_R$ −76°C	First cycle $J(T)/J_R$ −196°C	Second cycle $J(T)/J_R$ −196°C
285C Laschamp	NRM	1	.995	.991	.910	.885
269D Olby	NRM	1	1.010	1.020	1.041	1.045
285B Laschamp	ARM	1	1.010	.986	.854	.889
269E Olby	ARM	1	.995	.981	.861	.868

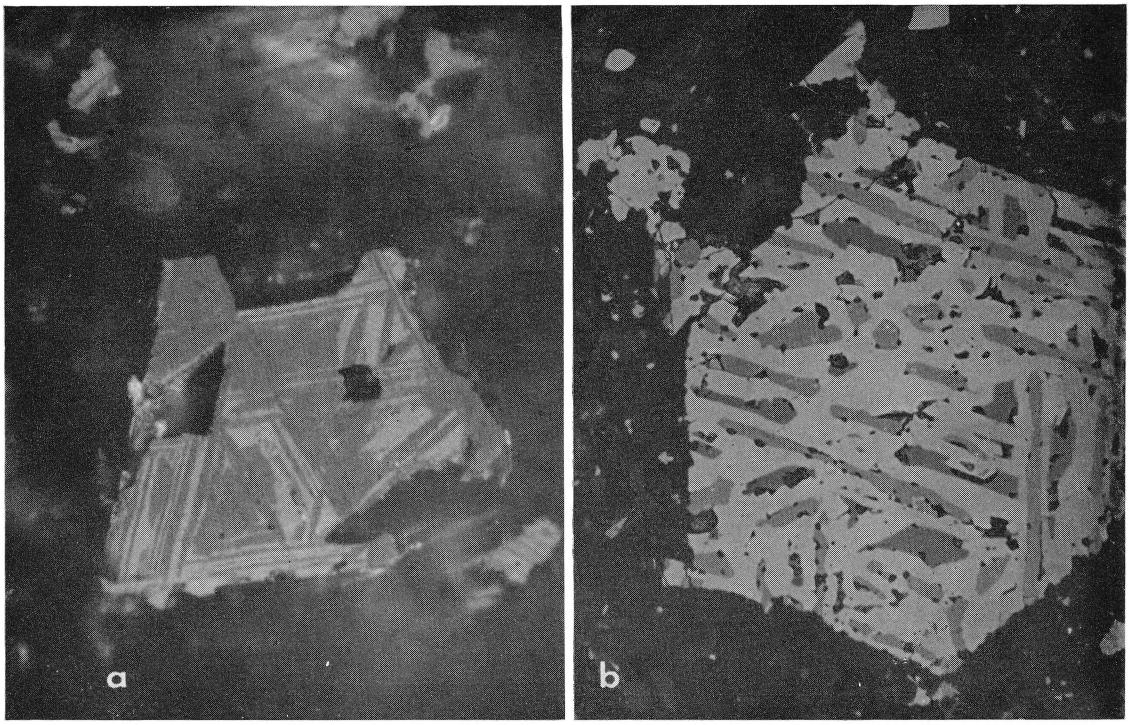

FIG. 3a. Ilmenomagnetite grain from Laschamp basalt (279). ×900, oil immersion. Magnetite gray, ilmenite lamellae lenticular, shades of gray due to reflection pleochroism, hematite pale gray.

FIG. 3b. Titanohematite–pseudobrookite–magnesioferrite pseudomorph of ilmenomagnetite grain from Olby basalt (261). ×370, air. Titanohematite shades of pale gray due to reflection pleochroism, pseudobrookite medium gray, elongate areas, magnesioferrite dark gray, angular areas.

and rare residual magnetite. In some composite grains the original trellis form of the ilmenite lamellae has been almost obliterated; however, it is generally clear that pseudobrookite has replaced the ilmenite, titanohematite has formed marginally to it, and magnesioferrite represents the remains of the former host magnetite (Fig. 3b). Oxidation has proceeded to Classes 5 and 6 on the scale of Ade-Hall *et al.* (1968), although rutile was not a product phase. Rare residual brown magnetite contains poorly reflecting exsolution lamellae of spinel. The pseudobrookite, titanohematite and magnesioferrite appear to have formed in mutual equilibrium. Temperatures were probably in excess of 600°C (Haggerty and Lindsley, 1970).

Compositions of the minerals in two composite grains in sample 261C, determined by microprobe analysis, are given in Table 2. Composite grain A can be regarded as typical of the iron-titanium oxide assemblage in 261C (Fig. 3b). Relatively high MgO is not unusual for pseudobrookites (Otteman and Frenzel, 1965; Carmichael and Nicholls, 1967). In molecular percent, the titanohematite in A contains 77% Fe_2O_3, 3.3% R_2O_3, 8% $FeTiO_3$, and 11.6% $R^{2+}TiO_3$, and the magnesioferrite contains 18.6% Fe_3O_4, 49% $R^{2+}Fe_2^{3+}O_4$, 30% $R^{2+}R_2^{3+}O_4$, and 2.4% Fe_2TiO_4. However, compositions vary considerably from grain to grain, particularly Ti, Cr, Mg and Mn in hematite. Strong enrichment in Mg, Al and Mn in the cubic phase during progressive oxidation of the titanomagnetite was described by Wright and Lovering (1965). The TiO_2-poor magnetite core in composite grain B possibly

TABLE 2

MICROPROBE ANALYSES OF COEXISTING IRON–TITANIUM OXIDES IN OLIVINE BASALT 261C FROM OLBY

	Composite grain A			Composite grain B		
	Magnesio-ferrite	Titano-hematite	Pseudo-brookite	Magnetite core [b]	Titano-hematite	Pseudo-brookite
TiO_2	1.03	10.25	47.3	.47	10.09	46.7
Al_2O_3	16.0	1.84	2.24	6.29	1.69	1.11
Cr_2O_3	.07	.07	.01	.40	.34	.04
V_2O_3	.18	.46	.24	.10	.25	.17
Fe_2O_3 [a]	57.2	80.5	42.5	62.2	78.3	43.4
FeO [a]	8.9	3.7	0.0	24.9	7.3	2.4
MnO	2.75	.41	.31	2.23	.27	.33
MgO	15.3	2.8	6.5	3.5	.75	4.5
CaO	.0	.05	.1	.1	.1	.1
Total	101.43	100.08	99.20	100.19	99.09	98.95

[a] Iron has been distributed on the basis of ideal stoichiometry.

[b] Magnetite analysis includes spinel exsolution lamellae not resolvable by the microprobe.

represents the magnetite formerly in equilibrium with the ilmenite lamellae; by chance it survived the high temperature oxidation that produced the pseudobrookite–titanohematite–magnesioferrite assemblage.

Clearly, the reversed NRM is possessed by samples containing a range of secondary iron–titanium oxide assemblages.

The magnesioferrite phase in the Olby basalt sample 216C, analyzed by the microprobe and listed in Table 2, is a potential carrier of NRM. The lattice parameter of this spinel phase should be approximately 8.31 Å, which is considerably lower than the 8.39 Å determined for magnetic separates. In addition, the Curie point of the microprobe-analyzed magnesioferrite would be considerably lower than that determined by the J–T measurements and by thermal demagnetization. Also, no magnesioferrite was detected in the Laschamp flow. It thus appears highly unlikely that the magnesioferrite plays a role in the magnetizations of the Laschamp and Olby basalts.

The following experiments were performed in an attempt to determine whether the remanence-bearing magnetite is single- or multidomain. Kobayashi *et al.* (1965) found that magnetite particles in the single-domain range can be distinguished from clearly multidomain magnetite ($d > 37\ \mu$) again by low temperature work. As they are heated to room temperature across the magnetocrystalline anisotropy transition temperature near 130°K, multidomain particles lose most of their remanence. The relative survival of remanence across the transition point increases markedly with decreasing grain size, such that Kobayashi *et al.* concluded that magnetite particles whose remanence is unaffected by the magnetocrystalline anisotropy transition are single-domain in character. We applied this test to Laschamp and Olby samples. Untreated samples were placed in a field-free environment and then cooled to the liquid nitrogen temperature before being returned to room temperature. Decreases of the total NRM vectors of 15.7% and 6.8% were observed for the Laschamp and Olby samples, respectively. An identical sequence was repeated for previously demagnetized samples exposed to a 1 kOe IRM. The remanence of the Laschamp sample dropped by 13.2% and that of the Olby sample by 28.4%. Magnetic separates were given an 8 kOe IRM and cooled down to the liquid nitrogen temperature, while we continuously monitored their

TABLE 3

LISTING OF THE SAMPLES USED AND THE EXPERIMENTS PERFORMED ON THEM

	Measure NRM	AC demag-netize	Thermal demag-netize	TRM acquisi-tion	Curie tempera-ture measure	Low temp test NRM	Low temp test IRM	Low temp test ARM	Magnetic separate and x-ray	Lowrie Fuller–test	Micro-probe	Flow
261C											X	Olby
262C	X	X		X			X					Olby
273D	X		X	X								Olby
269 A–E (5 cores + chips)	X	X		X	X	X		X	X	X	X	Olby
279C											X	Laschamp
282C	X	X		X			X					Laschamp
289D	X		X	X								Laschamp
285A–C (3 cores + chips)	X	X		X	X	X		X	X	X	X	Laschamp

magnetization and temperature. Drops in magnetization of 7% and 22% were observed for the Olby and Laschamp powders, respectively, as the temperature decreased through the magnetocrystalline anisotropy transition temperature. These experiments suggest that the remanence is held largely by submicron-sized particles mostly single-domain. This is consistent with the observation of phase inhomogeneity of the magnetite, observed at high magnification under the microscope.

Noting that the stability against AF demagnetization of TRM in single-domain magnetite grains decreases with increasing magnetic fields, while the stability of TRM in multidomain magnetite grains increases with increasing magnetic fields, Lowrie and Fuller (1971) devised a test to determine whether the remanence is held in single- or multidomain particles. The test compares the NRM's (assumed TRM) AF demagnetization spectrum to that of saturation IRM of the same sample. The relative stability against AF demagnetization of saturation IRM responds in the same manner as saturation TRM. The stability against AF demagnetization of a TRM acquired in a weak field (*ca.* .5 Oe) and carried by single-domain particles should be relatively greater than the stability of the saturation IRM. In contrast, a weak field TRM carried by multidomain particles will be less stable than saturation IRM. Application of this test to samples of the Laschamp and Olby formations seems to indicate that the remanence is carried by single-domain particles, as seen in Fig. 4.

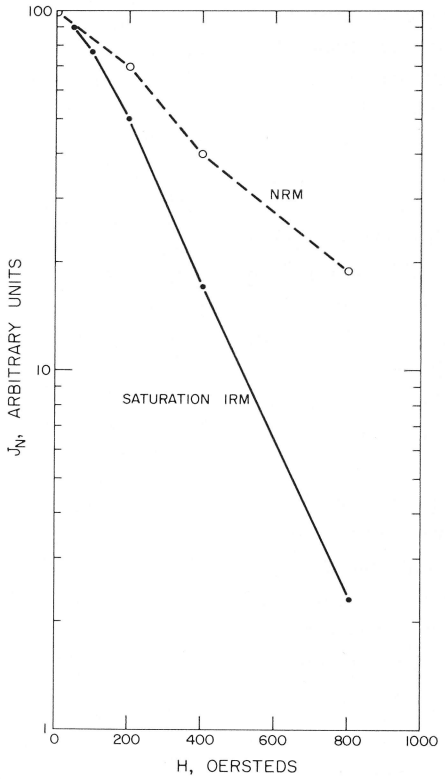

FIG. 4. Comparison of NRM and saturation IRM stability against AF demagnetization for sample 285. The higher relative stability of the NRM satisfies the Lowrie–Fuller criterion for single-domain remanence, providing there is only one magnetic mineral present in the sample.

CONCLUSIONS

The oxide minerals now present in the Olby and Laschamp flows are magnetite, hematite (including titanohematite), ilmenite, pseudobrookite, magnesioferrite and spinel. The last three occur in the Olby flow. The degree of oxidation ranges from moderate (Classes 3 or 4) to extreme (Class 6). Thus it appears that the reversed NRM is not dependent on the state of oxidation and recrystallization of the Fe–Ti minerals. X-ray diffraction patterns of the magnetic separates confirmed the presence of two magnetic minerals—magnetite and titanohematite. While the Curie temperature eliminated neither species, the absence of the Morin transition, observation of the magnetite magnetocrystalline anisotropy transition, and the value of the saturation magnetization suggest that the predominant magnetic mineral wherein most of the remanence resides is probably nearly stoichiometric magnetite. The resistance of the remanence to destruction by temperature cycles below the magnetocrystalline anisotropy transition temperature and the fact that the AF demagnetization spectrum of the NRM is more stable than that of saturation IRM suggest that the remanence is probably carried by single-domain particles. Because we know of no mechanism by which single-domain magnetite could have self-reversed, these conclusions argue against the possibility that the magnetization of the Laschamp and Olby flows have self-reversed.

Since the virtual geomagnetic pole positions obtained from the two flows are approximately 35° from the rotational axis, the possibility of a localized field reversal is not eliminated. Until independent discoveries of the Laschamp event are made at different geographical locations, this will remain a topic for speculation.

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