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Magnetic Techniques for Ascertaining the Nature of Iron Oxide Grains in Basalts

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Abstract. Qualitative techniques are described by which the domain nature and magnetite-maghemite oxidation state of the iron oxides in basalts may be rapidly identified through measurements of hysteresis and susceptibility over a wide temperature range. Detailed studies made on different suites of basalts have revealed that their magnetic properties in most cases can be explained only on the basis of single-domain behaviour. Also it has been found that the observed variations in these properties between basalts are usually best explained by differences in the position the iron oxide minerals in the different samples occupy along the magnetite-maghemite oxidation chain. These observations suggest that the role of titanium usually found in association with iron oxides in basalts is to subdivide the grains physically rather than to form solid solutions of titanomagnetites or titanomaghemites. Some implications of these results to basalt formation and the magnetic anomalies such rocks could cause are discussed.

Key words: Magnetic — Techniques — Basalt — Susceptibility — Hysteresis — Oxidation — Magnetite — Titanium — Domains — Superparamagnetic.

1. Introduction

The few percent of iron oxide minerals present in basalts are responsible for the bulk magnetic properties of these rocks. Microscopic observations on thin sections or polished surfaces of the basalts reveal that the usual size of the iron oxide grains is in the range of 1 to 100 microns. Such large magnetic grains are expected to behave like multidomains and in fact Néel (1955) expressed this view although the magnetic properties of basalts could not be adequately explained in terms of multidomain behaviour.

Chemical analysis of the iron oxide minerals separated from basalts usually indicates the presence of some titanium. Chevallier, Bolfa and Mathieu (1955), Uyeda (1958) and others concluded from investigations on synthetic specimens that solid solutions can occur over a wide compositional range between the spinel end members Fe_3O_4 and Fe_2TiO_4, with a

corresponding variation in magnetic properties such as Curie point (T_c). This has led to the widespread adoption by workers in rock magnetism of titanomagnetite and titanomaghemite series, whereby the observed magnetic properties of the rock are interpreted in terms of the place of the iron oxide minerals within either series. However, in actual rocks T_c as estimated from the chemical composition of the grains seldom agrees with that deduced from magnetic measurements. Moreover, the observed Curie points are not well defined, especially for basalts presumed to show values less than 400 °C on the basis of their Ti content. Further difficulties in determining T_c arise from changes the magnetic grains undergo during the laboratory heating itself, resulting in the irreversibility of heating and cooling cycles.

Oxidation of magnetite has engaged the attention of both geologists and chemists for the past fifty years, but still some aspects of this process need further study for a better understanding. Lepp (1957) first pointed out that the product of oxidation depends only on the grain size of the magnetite used and the rate of reaction. Later Feitknecht and Gallagher (1970) showed that grains of magnetite smaller than 3000 A in diameter tend to become oxidized to maghemite, whereas larger ones turn into hematite or they form disproportionately both magnetite and hematite through several intermediate cation deficient phases. The magnetic properties of synthesized cation deficient magnetites with different Ti contents have been studied as a function of degree of oxidation (Readman and O'Reilly, 1972). However, the manner in which the oxidation process itself depends on the grain size of the titanomagnetites is difficult to visualize in the absence of studies similar to those reported on sized grains of pure magnetite.

In a critical study (Radhakrishnamurty, Raja, Likhite and Sahasrabudhe, 1972) of the magnetic behaviour of several hundred basalts, not a single case was found which could be unambiguously attributed to the presence of large titanomagnetite grains with low T_c. All the ten cases encountered in the study which indicated low T_c turned out to be ambiguous due to grain size effects of the magnetic minerals in them. Thus, the occurrence in continental basalts of titanomagnetites of the synthetic type itself is perhaps not as soundly established a fact as has been generally assumed. For these reasons, consideration of the role of titanium in determining magnetic properties along the magnetite-maghemite chain has been omitted as an open question for the sake of the present analysis.

The above features of the iron oxide minerals could certainly cause many complexities in the way of understanding the magnetic properties of basalts. Fortunately, magnetite and maghemite have distinct magnetic properties and hence can be identified by simple magnetic tests. Recently, it was shown (Radhakrishnamurty, Likhite, Raja and Sahasrabudhe, 1971) that such tests can be unambiguously extended to cation deficient (CD) phases of magnetite, as these have specific magnetic properties by which

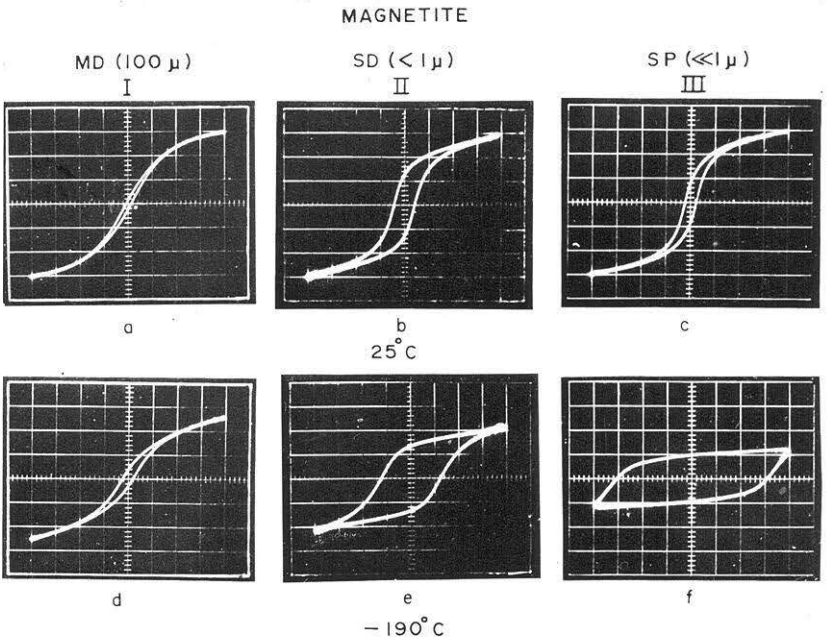

Fig. 1. Typical hysteresis loops for one synthetic and two basalt samples. MD — multidomain, SD — single-domain and SP — superparamagnetic grains. a, b, c, at 25 °C; d, e, f, at −190 °C for the same samples. Scale: X-axis (magnetic field) 1 small division = 125 Oe for a and d; and 62.5 Oe for the rest. Y-axis (magnetic moment) 1 s.d. = 0.9 emu for all

they can be distinguished from pure magnetite and maghemite. However, the first step for an understanding of the magnetic properties of basalts is to ascertain the domain nature of the iron oxide grains in such rocks.

2. Hysteresis of Iron Oxide Grains

The magnetic hysteresis phenomenon depends on the domain structure of the grains in a material. At any particular temperature, there is a narrow range of sizes in which the grains of a magnetic material show maximum coercive force (H_c) and retentivity (J_r) and such grains are termed optimum single-domains (SD); smaller grains exhibit superparamagnetism (SP) due to thermal agitation and much larger grains become multidomains (MD). From simple theory, both SP and MD grains are presumed (Bean, 1955) to show vanishingly small H_c and J_r. However, experimentally, some finite values of H_c and J_r may be observed for clusters of SP particles due to dipole interaction (Evdokimov, 1963; E. Kneller, private communication,

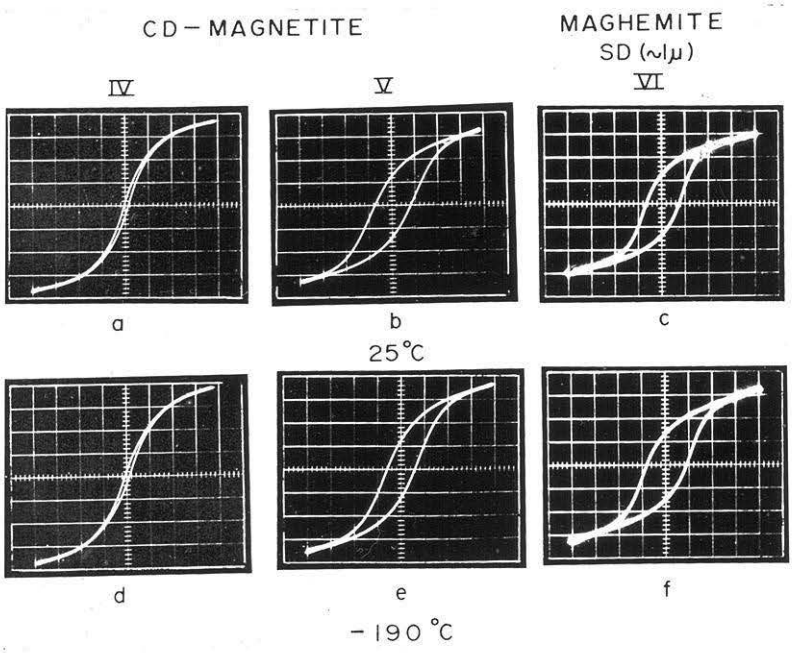

Fig. 2. Hysteresis loops for two basalts containing cation deficient magnetite and a synthetic sample of maghemite. Scale: X-axis 1 s.d. = 75 Oe; Y-axis 1 s.d. = 2.0 emu

1972) amongst them and for MD grains due to crystal defects in them.

Since the terms MD, SD, CD and SP comprise both domain states and composition, it may be explained that in this paper use of "SD" is reserved for "pure" magnetite, whereas "CD" refers to cation deficient grains that may be either SD or MD, though usually the former. "SP" and "MD" terms are applied to grains of any composition.

Magnetite undergoes a phase transition at -150 °C, below which its magnetocrystalline anisotropy constant K increases tenfold compared to that at room temperature (T_r). Morrish and Watt (1958) showed from studies on magnetite micropowders that for SD particles, H_c is several times greater at -196 °C than at T_r and that for MD grains the corresponding increase is by about a factor of two. These observations are consistent with the known positive correlation between H_c and K. Morrish and Watt also found that maghemite shows a small increase in H_c at low temperatures.

In Fig. 1 are shown the typical hysteresis loops for three samples; a, b, c were obtained at 25 °C and d, e, f are corresponding loops for the same samples at -190 °C. The loop in Fig. 1a is for a sample prepared by dispersing a small quantity of 100 micron size grains of magnetite in plaster

of Paris and the peak field used was 2500 Oe. This sample shows the expected increase in H_c at -190 °C (Fig. 1 d) corresponding to MD grains of magnetite. The loops shown in Fig. 1 b, c and 1 e, f are for basalts obtained in a peak field of 1250 Oe. It should be mentioned here that a small field has been chosen for these two basalt samples to show the change in H_c clearly, though this change has the same trend even in much higher fields. The large increase in H_c at -190 °C (Fig. 1 e) compared to that at 25 °C (Fig. 1 b) strongly suggests that the magnetic grains in sample II are SD magnetite. The similar behaviour shown by sample III (Fig. 1 c and 1 f) is again consistent with SD magnetite at low temperature. However, at T_r, this sample is in a metastable magnetic state and shows a Rayleigh loop (hysteresis in 10 Oe) which indicates that a significant fraction of the particles in it are superparamagnetic (Radhakrishnamurty and Sastry, 1970; Néel, 1970); the estimated particle size is about 100 A.

Fig. 2 shows the hysteresis loops for two basalt samples (IV and V) and one synthetic sample (VI) prepared by dispersing commercial maghemite in plaster of Paris. Surprisingly sample IV shows little or no change in its hysteresis with temperature (Fig. 2 a and 2 d), while sample V shows a distinct decrease in H_c at -190 °C (Fig. 2 e) compared to that at 25 °C (Fig. 2 b). This character is just opposite to that of magnetite. Such samples have been shown to contain CD magnetite phases (Radhakrishnamurty, Likhite, Raja and Sahasrabudhe, 1971). The hysteresis behaviour of maghemite sample VI, shown in Fig. 2 c and 2 f conforms to the expectation, namely there is a small increase in H_c at -190 °C compared to that at 25 °C.

Thus, basalts containing the above six kinds of minerals and grain sizes have been identified from their characteristic hysteresis behaviour at different temperatures. Of course, composite mixed behaviour might be, and often is, encountered in basalts and is in principle also detectable by experiment. However, it should be emphasised that the magnetic analysis of such composite rocks is still at best semi-quantitative.

As was pointed out previously (Radhakrishnamurty and Likhite, 1970 b), the presence of SP particles in a basalt can be revealed by the low field (10 Oe) hysteresis loop and that of optimum SD grains by their characteristically large relative remanence (ratio of remanent to maximum intensities) in medium fields (100—500 Oe). The presence of MD and CD magnetite can be established by hysteresis studies in high fields (1000—5000 Oe). However, it should be mentioned in this context that the sensitivities of the three different instruments used (Likhite, Radhakrishnamurty and Sahasrabudhe, 1965; Likhite and Radhakrishnamurty, 1966), for hysteresis studies in low, medium and high fields decrease somewhat with increasing magnetic fields due to increase in noise levels of the instruments. Hence, it may not be possible to observe distortionless loops for all kinds of basalts in all the different ranges of fields. Typically a sample should develop

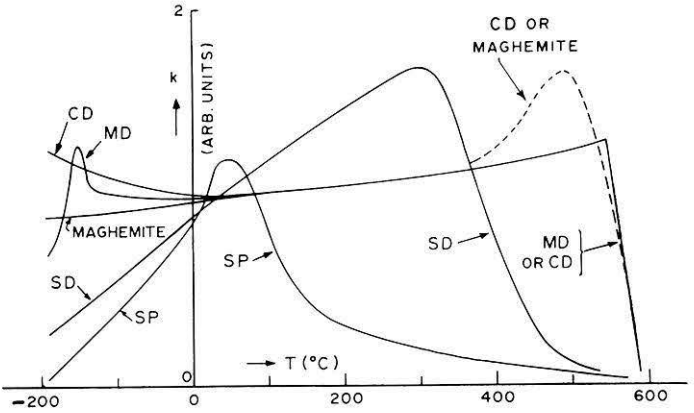

Fig. 3. Initial susceptibility as a function of temperature for basalts containing different types of grains. SP — superparamagnetic, SD — single-domain, MD — multidomain, CD — cation deficient grains of magnetite

maximum intensities of the order of 0.07, 1.0 and 2.0 emu/cc in low, medium and high fields respectively for observing distortion-free hysteresis loops with the three instruments.

3. Studies of Initial Susceptibility with Temperature

Many workers have described apparatus suitable for measuring the susceptibility (k) of rocks in low fields. The various kinds of basalts used in the present study have been measured in a field of 0.5 Oe with the apparatus described previously (Likhite and Radhakrishnamurty, 1965). With low- and high-temperature attachments (Radhakrishnamurty and Likhite, 1970a) it was possible to determine the k of most basalts at any temperature in the range -196 °C to 700 °C.

In Fig. 3 are shown curves depicting the variation of k with T for basalts containing different types of magnetic grains. These curves were based on actual measurements but somewhat idealised to bring out the salient features. Only the heating curves are shown, mainly to avoid congestion and to faciliate comparative analysis. It should be noted that the "SD" and "SP" notations on the curves refer to the respective grain state at T_r only: the actual grain state at high temperature on the "SD" curve tends towards SP, while that at low temperature on the "SP" curve tends towards SD.

All the curves are reversible in the temperature range $-196°$ to 25 °C. However, in the high temperature range, only the MD magnetite curve is

reversible, mainly because the changes taking place on heating in the composition and/or state of large magnetite grains are negligible, and the curve also shows a well defined and sharp T_c. The curves of all other types of grains are more or less irreversible in the high temperature range and some of them may change drastically due to changes in composition and/or grain growth: these features may imply that there would be ambiguity in defining any point of such curves as T_c. Some of the uncertainties in the determination of T_c of basalts were discussed in greater detail elsewhere (Radhakrishnamurty, Raja, Likhite and Sahasrabudhe, 1972).

Also, it was inferred from these magnetic observations that basalts which show SD magnetite behaviour often get oxidized to maghemite when heated beyond 200 °C; sometimes the grains become CD magnetite. Perhaps the most likely explanation for these observation is that when the grains get the necessary oxygen from their surroundings, maghemite might form and if oxygen was not available the grains might turn into CD magnetite. Cation migration in SD magnetite might start around 200 °C and the associated changes could even cause ambiguities in the determination of blocking temperature (T_b) or T_c of the relevant grains.

The following important features emerge from a comparative study of the different curves shown in Fig. 3.

1. While it may be difficult to distinguish between MD and CD magnetite from the high temperature part of the k-T curve, they may be resolved clearly from the low temperature side. MD magnetite shows the characteristic sharp k-peak at -150 °C due to phase transition and CD shows an increase in the value of k at -196 °C compared to its value at T_r.

2. SP and SD magnetite grains show a large decrease in k at -196 °C compared to their respective values at T_r. It is very difficult to identify the magnetite transition peak at -150 °C in the k-T curves of SP and SD samples, probably due to the large increase in H_c with decreasing temperature around the transition point.

3. Maghemite shows a small decrease in k at -196 °C compared with its T_r value.

The above features of the different kinds of grains can be explained by variations in the effective contributions by shape and magnetocrystalline anisotropies. Particularly, the inconspicuousness or absence of the k-peak at the transition temperature in the k-T curves of SD magnetite may be due to the undiminishing or somewhat increasing effect of shape anisotropy of the grains. The magnetic properties of CD magnetite grains are quite peculiar and at present stand out simple as experimental observations. The decrease of H_c and increase of k at low temperatures observed for CD grains are mutually supporting and consistent and it may be recorded in this context that an explanation for these, based on more fundamental properties of the material, is not yet possible.

Table 1. Magnetic granulometric data for stable basalts from Deccan and Rajmahal traps

	Flow No.	Rock type	Q_n[a]	Mineral nature	
				Virgin	After heating (600 °C)
1	2	3	4	5	6
	2	FGB	3.1	Maghemite	Maghemite
	4	PB	4.1	CD + SD	Maghemite
	5	AB	6.1	CD	Maghemite
	7	AB	5.3	CD	CD
	8	CGB	4.1	CD	CD
	9	AB	3.4	CD + SD	CD
Deccan	13	PB	6.8	CD	CD
traps	14	AB	8.0	CD	CD
	17	AB	1.3	CD	Maghemite
	21	PB	1.3	CD	CD
	22	PB	3.7	CD	CD
	24	PB	4.2	CD	Maghemite
	I	FGB	40.6	SD	Maghemite
	II	PB	71.4	SD	Maghemite
	V	FGB	36.0	SD	CD
	VI	PB	10.0	CD	CD
	VII	CGB	45.8	SD + CD	Maghemite
	X	PS	116.0	SD	SD
Rajmahal	XI	PB	7.8	CD + SD	CD
traps	XII	CGB	55.0	SD	Maghemite
	XIII	PB	24.8	SD + CD	CD
	XIV	CGB	9.9	CD	CD
	XV	PB	78.8	SD	Maghemite
	XVI	CGB	23.0	SD + CD	CD
	XVII	CGB	5.2	CD + SD	CD

[a] Ratio of remanent intensity to that induced in 0.5 Oe. FGB — Fine grained basalt, CGB — Course grained basalt, PS — Pitchstone, AB — Amygdaloidal basalt, PB — Porphyritic basalt, CD — Cation deficient, SD — Single-domain and referring to magnetite.

4. Granulometry of Basalts

Several techniques have been developed (Dunlop, 1965; Stacey, 1967; Lowrie and Fuller, 1971) for studying the domain nature of the magnetic grains in basalts. However, these are mainly meant for identifying the domain nature of part of the grains responsible for the remanent magnetization of the rocks. The techniques described in this paper are useful

for studies on the total magnetic mineral content of the basalts and include both domain nature and chemical compositional aspects of the minerals concerned.

The granulometry of several suites of basalts has been studied. In Table 1 are given magnetic data for palaeomagnetically stable rocks from the Deccan and Rajmahal traps. The important points to be noted are as follows:

1. Values of Koenigsberger ratio (Q_n) are in the range 1 to 10 for basalts containing CD and 10 to 100 for SD magnetite.

2. The mineral in some of the samples gets converted to maghemite when heated to 600 °C and cooled; this may imply that the grains in the virgin samples are less than 3000 A in size.

3. Multidomain magnetic behaviour is very rare in these basalts and this indicates that the magnetic grain size may be greatly different from the physical grain size derived from optical observations.

4. The flows not listed in Column 2 among the two suites of basalts in Table 1 were found to be unstable or partially stable, mainly due to the presence of SP particles in samples derived from the respective flows.

In Table 2 granulometric data are given for some younger and older basalts. In cases where uniformity of grain nature in rock bodies is known the totals shown in Column 3 represent flows or sites regarded as units; in all other cases, numbers refer to the total of samples or specimens. Cases like SP, SD etc. indicate that these grains predominate. Composite notations like SP+SD, MD+SD etc. mean that the magnetic characteristics of both types of grains could be clearly identified for the sample studied. More than two kinds of grains may also be present in basalts, but this would be difficult to determine and hence was not attempted.

The data presented in Table 2 indicate that in basalts younger than Cretaceous, MD behaviour is rare while it is fairly common in older basalts. CD behaviour, when its occurrences both in pure and composite form are counted, is far more prevalent than others.

5. Implications of Magnetic Granulometry to Petrography

The most important result obtained from these studies on different kinds of basalts is that SP behaviour has been found even for some coarse grained samples. This result clearly indicates that each iron oxide grain in a basalt might be behaving like a cluster of much smaller grains due to some yet unknown mechanism of subdivision.

In some Deccan trap flows and dykes, entire rock masses were found to contain SP particles. It was impossible to separate the magnetic grains from crushed samples. These cases may indicate very rapid chilling of the magmas concerned, as a result of which SP-size (100 A) magnetite particles were formed more or less uniformly in the rock bodies. The presence of even

Table 2. Magnetic granulometric data for some young and old basalt formations

Formation	Age	No. of samples (or sites)	SD	SP	CD	MD	SP+SD	SP+CD	SD+CD	MD+SD	MD+CD	SP+MD
1	2	3	4	5	6	7	8	9	10	11	12	13
Deccan Traps	Tertiary	36[a]	—	—	21	—	—	12	3	—	—	—
DT Dykes	Tertiary	15[a]	6	2	3	—	2	1	1	—	—	—
Rajmahal Traps	Cretaceous	17[a]	6	—	2	—	4	—	5	—	—	—
Columbia River Basalts	Miocene	8[b]	—	—	—	—	5	3	—	—	—	—
Greenland Basalts	Tertiary	43[b]	—	—	25	—	—	—	18	—	—	—
Ophiolites (Newfoundland)	Ordovician	56[b]	—	—	1	2	—	16	1	28	1	7
Cuddapah Traps	Precambrian	15[a]	—	—	—	—	—	—	—	15	—	—
Dykes	Precambrian	6[a]	—	—	—	—	—	—	—	—	6	—

[a] Represents sites or flows. [b] denotes samples or specimens. SD — single-domain, SP — superparamagnetic, CD — cation MD — deficient,multidomain grains.

much smaller particles which are superparamagnetic at −196 °C has been inferred in some samples of ophiolite complexes in Newfoundland (Deutsch, Radhakrishnamurty, Strong, Rao, Pätzold and Likhite, 1973). However, such small particles in these ophiolites seem to have been formed due to alteration.

It is possible to study the granulometry of a large number of samples in situ using portable instruments. The essential magnetic grain nature can be ascertained at the rate of a sample per few minutes. A few expeditions made with portable instruments to the rock outcrops have already revealed that in rock bodies such as flows or dykes the magnetic grain nature is sometimes uniform and is variable at other times. The latter case might mean that normal weathering and alteration have caused grain subdivision. Such studies in situ can aid the choice of samples best suited for palaeomagnetic work.

The magnetic grains in basalts constitute a very interesting area for future work and may contribute to a better understanding of basalt formation and subsequent alteration.

6. Implications to Geomagnetic Anomalies

As described in previous sections, the magnetic properties of basalts can be quite complex and these in turn determine the nature of the geomagnetic anomalies, especially the marine magnetic anomalies. Studies on dredged samples showed that Q_n values varied from 190 to 1 for samples taken from the axis to the edges of the Mid-Atlantic ridge (Irving, 1970); the natural remanent intensity also varied in a similar proportion. It was reported that the T_b of the grains in the samples obtained from the axis was about 200 °C and that of edge samples was around 500 °C. These features have been explained on the basis of conversion of the original titanomagnetites to titanomaghemites (Marshall and Cox, 1972) and SD titanomagnetite to SP (Butler, 1973), both operating through an oxidation process. Another somewhat different but likely explanation for such observations may be the conversion of SD grains to CD type. The change of SD to CD nature involves a sharp fall in remanent intensity and also an increase in T_b or T_c. The magnetic contrast produced by such changes could cause some anomalies in a profile and these may not be distinguishable from other anomalies caused by geomagnetic reversals.

Since the anomalies are believed to be caused by remanent intensity of the rock body, T_b of the grains is a very important parameter in estimating the thickness of the basaltic layer. Due to the increase of temperature with depth, thinner layers of low-T_b rock mass and thicker layers of high-T_b rocks would both contribute to the magnetic anomalies and this may result in underestimating the thickness of the low-T_b rock layers.

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