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Alteration and dissolution of fine-grained magnetite and its effects on magnetization of the ocean floor

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Abstract

Scanning and transmission electron microscopy (STEM) of ocean-floor pillow basalts with ages between 0 and 70 Ma reveals progressive alteration of submicrometer titanomagnetite to phases such as goethite and clays. In contrast, larger titanomagnetite grains (> 1 μm) oxidize to titanomaghemite without apparent change in crystal morphology. Rock magnetic experiments are consistent with a selective removal of the submicrometer grains as the basalts age, and show good correlations between the ranges of grain sizes observed by STEM and those indicated by hysteresis properties. Remanence contributions from the larger (pseudo-single domain and multi-domain) titanium–iron oxides are inferred to decrease only slightly as the ocean floor becomes older, whereas the overall remanence decays with age as the substantial contribution from stable, single-domain titanomagnetite grains diminishes greatly due to their alteration to other phases. Although more work on many more samples is required to verify our conclusions, the current data imply that this alteration is one of the reasons that amplitudes of marine magnetic anomalies diminish with age over time scales of tens of millions of years. © 1997 Elsevier Science B.V.

Keywords: ocean floors; magnetization; titanomagnetite; maghemite; electron microscopy; alteration

1. Introduction

The pronounced decrease in amplitude of marine magnetic anomalies with increasing age of the ocean floor, recognized more than 30 years ago [1], has been attributed to decreasing intensities of natural remanent magnetization (NRM) [2–4]. It has generally been assumed that this decrease reflects oxidation of titanomagnetite to titanomaghemite [5–8]. However, it is difficult to verify this assumption by direct measurements. In nature the oxidation of titanomagnetite takes place over geological time scales at ambient upper-crustal temperatures. Although oxidation to titanomaghemite can also be achieved in the laboratory, the experiments must typically be performed at elevated temperatures to attain reasonable reaction rates [9–11]. In such higher-temperature reactions, experimentally induced changes in NRM cannot be quantified. This is because the

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temperatures during the experiments are typically close enough to the Curie points of the titanium-rich magnetite to partially demagnetize the original NRM, although synthetic, Al-doped titanomagnetite is a useful substitute [12] and the use of an oxygen plasma has produced titanomaghemite at temperatures as low as 50°C [13]. It is not surprising, nevertheless, that there is little consensus regarding the relationships between NRM intensities and maghemitization [7,14,15]. A second obstacle to better understanding concerns the lack of observations of submicrometer-sized magnetic minerals in natural samples. Although bulk rock magnetic properties of mid-ocean ridge basalts (MORB) typically indicate pseudo-single domain (PSD) behavior, this may be an average of SD and multidomain (MD) characteristics, or even of SD and superparamagnetic (SP) contributions.

The samples used in this study were dredged at four sites from the Atlantic Ocean and have ages of < 1 Ma (site 1), 9, 26 and approximately 70 Ma (site 4), as determined from marine magnetic anomalies [16]. From site 1, two separate pillows were studied (site 1A and 1B), whereas one pillow was examined from each of the other sites. Several 1 inch diameter cores were drilled from these pillows and these will be called samples in this paper. Specimens, chips of about 10 mg, or thin sections were obtained from the samples for rock magnetic experiments and electron microscopy. Descriptions of the samples, rock magnetic measurements, and degree of oxidation (maghemitization) of the pillow lavas can be found elsewhere [16–18]. Observations of grains larger than 1 μm in our samples have also been described earlier [17] and include scanning and transmission electron microscopy (SEM and TEM), selected-area electron diffraction (SAED), analytical electron microscopy (AEM), X-ray diffraction and Mössbauer data. The samples display a strong decrease in NRM intensity accompanied by an increase in Curie temperature with increasing age (Fig. 1), in agreement with previous observations [2,6]. The saturation magnetiza-

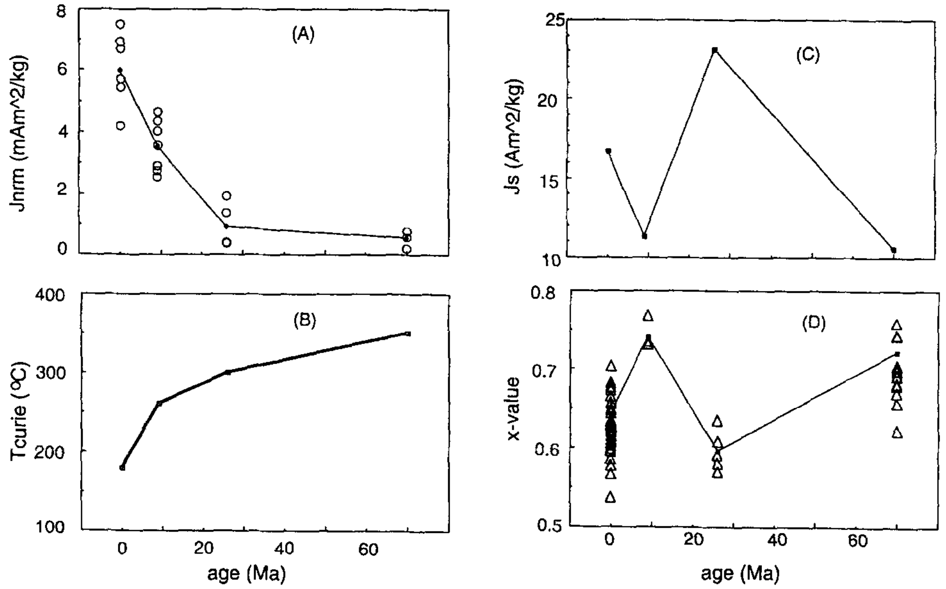


Fig. 1. Magnetic parameters as a function of age for samples from the four sites used in this study. (A) Natural remanent magnetization intensity J_{NRM}. (B) Curie temperatures averaged by site, consistent with an increasing degree of maghemitization as the pillows aged. (C) the saturation moment, J_s, of titanomagnetite–titanomaghemite, averaged by site and normalized by the concentrations estimated from refined X-ray data [17,18]. (D) Titanium content, measured by AEM or microprobe analysis, plotted as x, which is the fraction of ulvöspinel (Fe_2TiO_4) in a titanomagnetite (or equivalent titanomaghemite) of composition $Fe_{(3-x)}Ti_xO_4$. Note the inverse correlation, as expected [19], between J_s and x.

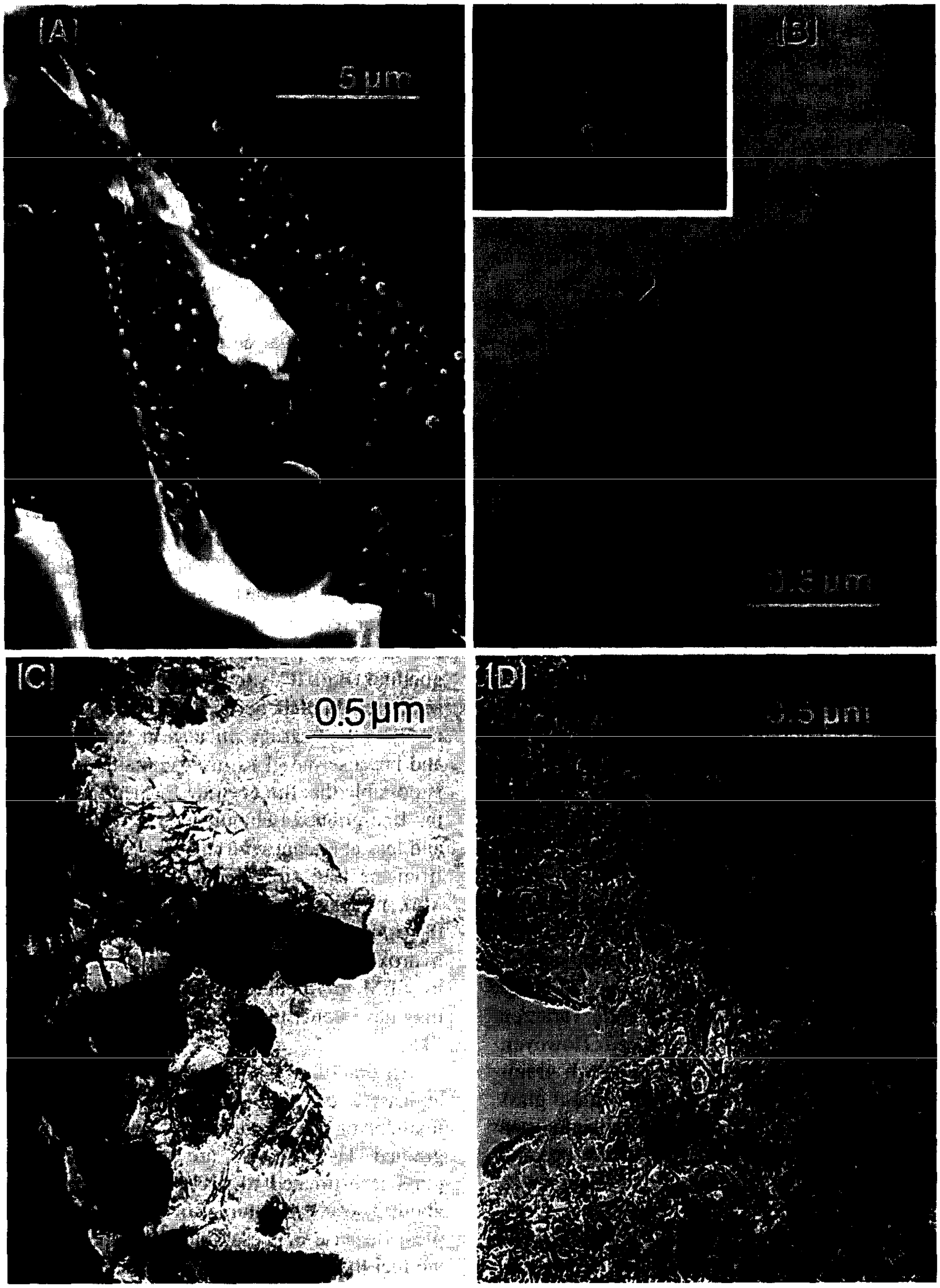
tion of the samples, J_s (Fig. 1C), does not correlate with age, suggesting at first glance that potentially magnetic material is not lost in the oxidation process. J_s does correlate (inversely) with titanium content in the titanomagnetite (Fig. 1C,D) but this is to be expected [19]. Others have suggested that the original NRM intensity of ocean floor pillow basalts is degraded or replaced by secondary magnetizations during maghemitization with only slight volumetric changes in the Fe–Ti-oxides [6–8,20–22] and we cannot exclude this possibility. However, one could also hypothesize that a volumetrically minor fraction of magnetic material is lost due to alteration to non-magnetic minerals, but that this fraction carries a proportionally large portion of the stable NRM before alteration. Following a related study on very young MORB [23], we here report SEM and TEM observations that reveal and confirm the presence of abundant titanium–iron oxides in the SD or even SP range as products of rapid quenching within interstitial glass in young MORB. We also observe in this study that these submicrometer grains are apparently altered progressively with age, implicating dissolution of submicrometer-sized iron oxides in glass as an important contributor to the decrease of NRM with increasing age. Given the provenance of the dredged samples, this alteration must have been related to the interaction between the pillow lavas and seawater at ocean-bottom-water temperatures.

2. Electron microscopy observations of Ti–Fe oxides

Titanomagnetite as well as titanomaghemite grains commonly show dendritic and cruciform textures, indicating that they originated by quenching [24]. More than 90% (by volume) of the Fe–Ti-oxide grains have sizes of 1–10 μm with little variation from sample to sample and site to site. However, clusters of very small titanium- and iron-rich grains were also observed by SEM in fresh interstitial glass of our youngest samples (Fig. 2), as previously seen in other studies [23,25]. Energy-dispersive spectral (EDS) analysis shows that these grains have much larger Fe and Ti contents than surrounding glass. They were shown to be well crystallized titanomagnetite grains by SAED patterns using a TEM (Fig. 2B). Titanium contents show more variation and are often lower (see also [23]) than those of the larger grains, for which x is shown in Fig. 1D. As the interstitial glass is progressively more altered in older samples, the submicrometer titanomagnetite grains within the glass are also altered.

Two kinds of glass have been observed in our pillow basalt samples: quenched pillow rim glass and interstitial glass in pillow interiors. Because only one of our samples came from a pillow rim, we have not included glasses of pillow rims in our study. The glassy groundmass occurs between dendritic clinopyroxene crystallites, in triangular areas between plagioclase laths (Fig. 2A), and in vesicles, indicating that the interstitial Si-rich glass was formed in the latest stage of pillow basalt crystallization. Interstitial glass was observed in all samples from sites 1, 2 and 3, but the freshness and amount are strikingly different, whereas in the samples from site 4 the interstitial glass appears to have been completely altered. In samples from site 1 (0–1 Ma), interstitial glass is black, consistent with fresh glass, and is easily observed with SEM and TEM (Fig. 2A,B). In samples from sites 2 (9 Ma) and 3 (26 Ma), the glass is brown or red-brown under the binocular microscope and has been partially altered to clay minerals and goethite (Fig. 2C, site 2). Goethite has been reported in altered MORB before [26], and because there are no other Fe-bearing silicates in the interstitial glass, and because the glass itself contains very little Fe, it is possible that the goethite formed from alteration of the fine-grained titanomagnetite, which becomes less and less abundant with increasing age of the samples from the four sites. In the samples from site 4 (70 Ma), no glass has been positively identified under the optical microscope, but phyllosilicates, iron oxyhydroxide minerals, and even calcite, were observed by SEM to fill the groundmass area that originally may have consisted of interstitial glass (Fig. 2D, site 4).

All our studied samples are from pillow interiors, at least 2 cm from the rinds, and grain sizes and textures of the plagioclases, pyroxenes, and large-grained Ti–Fe-oxides are similar. The interstitial glass is estimated to occupy, or to have occupied, about 3–6%, by volume, in each of the basalt samples. Because of these similarities between the sites, we feel that it is reasonable to assume that grain size


[A]
5 µm
[B]
0.5 µm
[C]
0.5µm
[D]
0.5 µm

distributions of the Fe–Ti-oxides were originally similar as well.

3. Rock magnetic experiments

Three sets of experiments, designed to test whether the observed presence (or absence) of submicrometer Fe–Ti oxides can be correlated with rock magnetic parameters, show that the fine-grained material may be a significant carrier of the magnetic remanence, albeit dominant only in the younger samples (sites 1 and 2). One of these experiments (Fig. 3) involves imparting a three-component isothermal remanent magnetization (IRM) to a sample which is then thermally demagnetized to determine how unblocking temperatures relate to coercivities [27]. The second set of experiments yielded hysteresis curves (Fig. 4) and hysteresis parameters, which were plotted on a Day diagram [28] in order to determine the average domain states of the magnetic carriers in the rock chips (Fig. 5A). Because many of our hysteresis curves are wasp-waisted (e.g., [29]), they were analyzed in order to see whether there is a trend in decreasing constriction with increasing age. Third, low-temperature IRM was imparted to thin sections used earlier for SEM observations, and this IRM$_{77K}$ was measured while the temperature of the thin sections increased to room temperature (Fig. 5B). These measurements revealed a significant SP contribution to the rock magnetic characteristics of site 1, but not for sites 2–4.

The thermal demagnetization of three-component IRM reveals unblocking temperatures of the lowest coercivity fraction (triangles in Fig. 3) that agree well with the decay of strong-field magnetizations induced during Curie runs in samples from site 1. This shows that the larger remanence-carrying grains and the grains that are magnetically activated in a strong field have the same temperature-dependent behavior, and presumably the same oxidation state and/or titanium content. The higher-coercivity fractions (circles and crosses in Fig. 3) in site 1 have unblocking temperatures that are slightly higher than the bulk of the Curie temperatures. This is reminis-

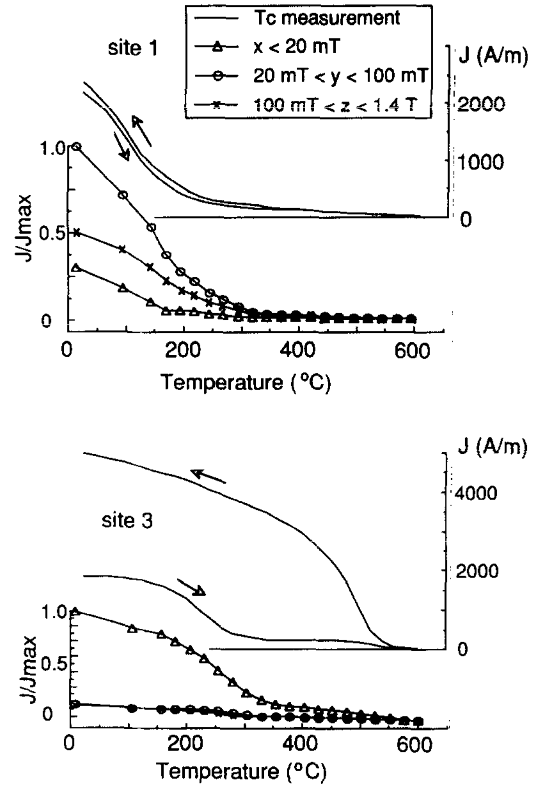


Fig. 3. Three-component IRM [27] that has been thermally demagnetized and Curie runs for parts of the same samples from sites 1 and 3. Note that in site 1 (Pillow 1A), the medium-coercivity component (between 20 and 100 mT) is much larger than the < 20 mT component, whereas in site 3 (and also sites 2 and 4, not shown) the < 20 mT fraction predominates. This means that in the older samples the grain sizes are likely to be more PSD and MD than in site 1, where the behavior is more SD-like.

Fig. 2. Electron microscope images of submicrometer grains in interstitial glass or their alteration products. (A) fresh interstitial glass in a triangular area between plagioclase, with submicrometer titanomagnetite observed by SEM in a sample from site 1 (Pillow 1B). (B) TEM bright field image of tiny grains of titanomagnetite, in an edge thinned by ion milling in the same sample from site 1, with (inset) selected area electron diffraction (SAED) pattern showing that the tiny grains in glass are well crystallized titanomagnetite. (C) TEM bright field image showing that interstitial glasses are partly replaced by needle-shaped iron oxides (goethite) and phyllosilicates (halloysite) in a sample from site 2 (age = 9 Ma); a few submicrometer Fe–Ti oxides are still preserved. (D) TEM bright field image of goethite and halloysite (as in C) that have completely replaced interstitial glass in a sample from site 4 (age = ~ 70 Ma).

cent of the effect noted in very young MORB samples by Kent and Gee [20,21], who observed that stable NRM is carried, in part, by grains with higher unblocking temperatures than the bulk Curie point of the samples. A lower Ti content in several of the smaller grains has been measured for our samples by analytical electron microscopy [18], and has also been reported [23] for the same samples as studied by Kent and Gee. The lower Ti content of some of the very small grains appears to be responsible for the small difference in maximum unblocking temperatures between low- and high-field measurements. That this difference is not greater is due to the fact that many of the submicrometer grains have x values varying little from 0.6, whereas others have much lower x values (see also [23]). More important for this study, however, is the observation that, in samples from site 1, the coercivities of the highest-intensity fraction are greater than those of the older MORBs (sites 2–4; results from site 3 are shown as a representative example in Fig. 3). This is consistent with grain sizes of potential remanence carriers being smaller in samples from site 1 than those of the older sites. Although it has been observed [12] that progressive maghemitization (up to a value of the oxidation parameter, z, of 0.91) of synthetic monodomain titanomagnetites causes a four-fold decrease in coercive force H_c (see also [10]), their H_{cr}/H_c ratio appears to decrease with oxidation, instead of the increases that we observe in sites 2–4.

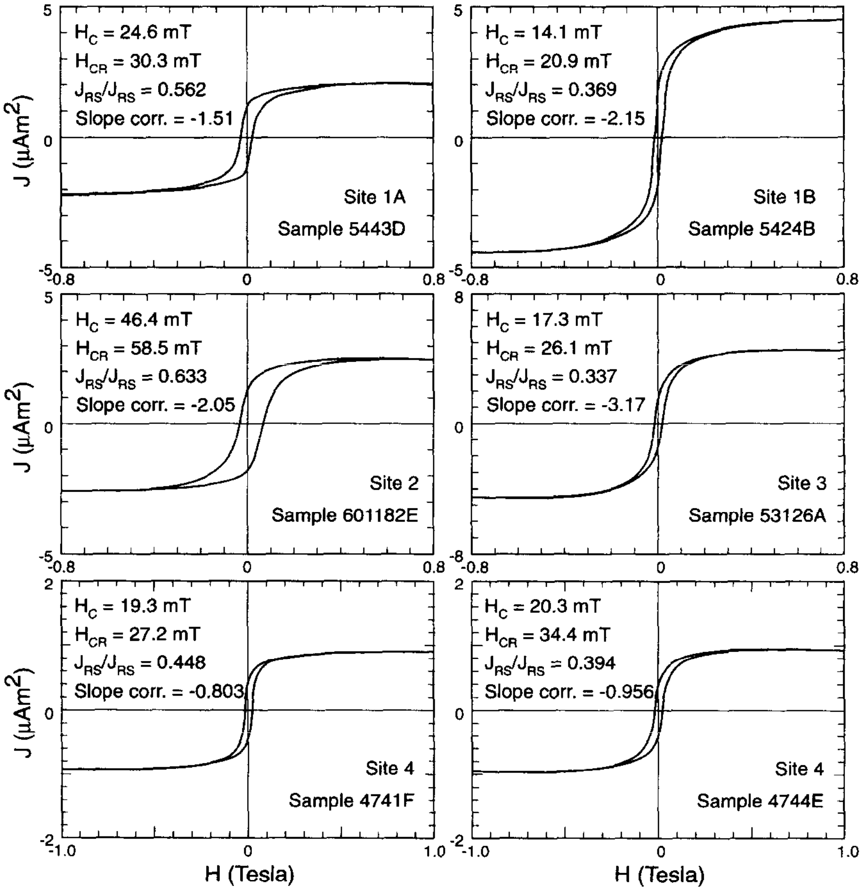


Fig. 4. Hysteresis loops, corrected for paramagnetic slopes (correction factor in $\mu Am^2/T$), for representative chips from samples from pillows 1A and 1B (site 1), from one chip each from sites 2 and 3, and from two chips from different samples from site 4. The chips weighed between 6 and 12 mg.

Moreover, the J_{rs}/J_s ratios in the synthetic samples remain higher than 0.47, whereas in our samples these ratios clearly decrease to levels as low as 0.3, indicating greater contributions from PSD and MD grains. Hysteresis parameters (Figs. 4 and 5A) for sites 2–4 thus show a mixing trend towards more PSD and MD behavior with increasing age. The sample shown in Fig. 3 (bottom) came from the group of site 3 samples (triangles in Fig. 5) with clear PSD-type behavior — its J_{rs}/J_s ratio is about 0.33 — which explains the predominance of lower coercivities (H_c is about 17 mT for this sample; see also the hysteresis loop for site 3 in Fig. 4). If we can assume, as argued earlier, that our MORB samples started with a similar grain size distribution of silicates as well as Fe–Ti-oxides when they formed at the Mid-Atlantic Ridge, then this implies that the SD fraction of MORB pillow basalts was gradually lost as they aged from 9 to 70 Ma.

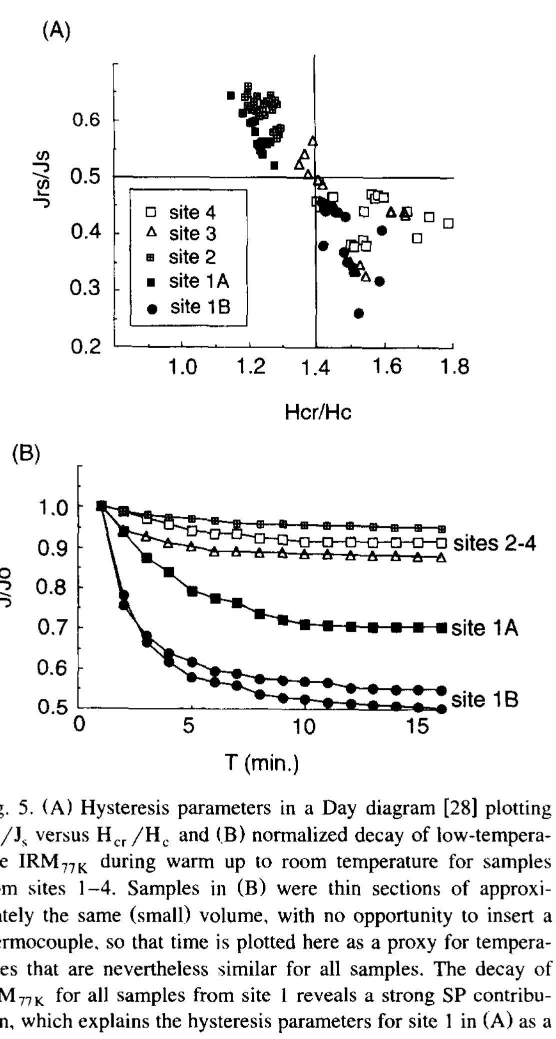


Fig. 5. (A) Hysteresis parameters in a Day diagram [28] plotting J_{rs}/J_s versus H_{cr}/H_c and (B) normalized decay of low-temperature IRM_{77K} during warm up to room temperature for samples from sites 1–4. Samples in (B) were thin sections of approximately the same (small) volume, with no opportunity to insert a thermocouple, so that time is plotted here as a proxy for temperatures that are nevertheless similar for all samples. The decay of IRM_{77K} for all samples from site 1 reveals a strong SP contribution, which explains the hysteresis parameters for site 1 in (A) as a mixture of SP and SD grains. In contrast, samples from sites 3 and 4 have predominantly PSD grains or mixtures of SD and MD fractions.

At first glance, it appears that the hysteresis parameters of site 1 do not follow this trend. However, it is known that bulk-rock PSD behavior during hysteresis measurements can be caused by a mixture of SD and SP grains, as well as by a mixture of SD and MD grains or by relatively homogeneous distributions of PSD grains. To resolve this ambiguity, the IRM_{77K} values and their decay as the samples warmed up to room temperature were used to distinguish between three samples with significant SP contributions from site 1, and samples with hardly any SP behavior (one sample each from sites 2–4). There is also good correlation for the samples *within* site 1: two samples from one pillow (labeled site 1B) have PSD-type behavior (Fig. 5A) and reveal very pronounced decay during warm up to room temperature, whereas one sample from a different pillow (site 1A) with more SD-type behavior is intermediate in its decay behavior (Fig. 5B).

It must be noted, however, that two alternative explanations for the marked decay of low-temperature IRM exists. The first is that SP-like behavior of large (2 mm) titanomagnetite crystals has been attributed to a rapid decay of the magnetocrystalline anisotropy constant with increasing temperature [30]. However, the maximum grain size in the sample of site 1B was only about 15 μm. The second alternative explanation is that surface-oxidized pure magnetite reveals a suppressed Verwey transition [31], which resembles the behavior of site 1B samples in that up to 70% of IRM_{77K} is lost during warm up. However, the Verwey transition in titanomagnetite with x of about 0.6 occurs some 100° higher than in pure magnetite. Moreover, we find that, regardless of increased oxidation, as seen in increased Curie temperatures (Fig. 1B) and increasing z values [17], the Verwey transition is nearly completely absent in all samples, and that just the opposite trend is seen in terms of correlation between oxidation and strong IRM decay during warm up. Thus, the observed relationships between magnetic behavior and grain

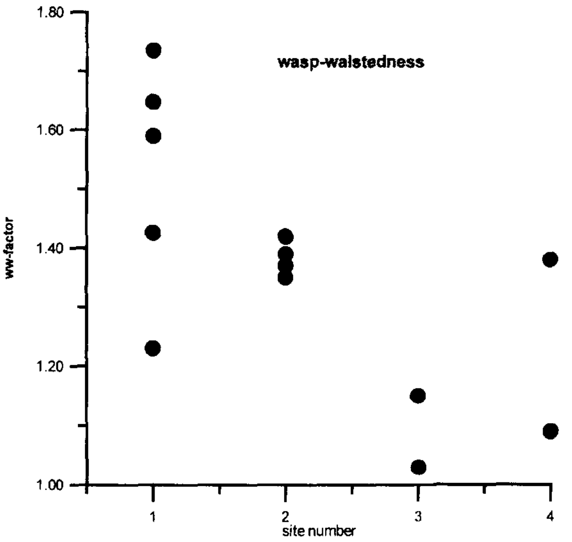


Fig. 6. The wasp-waisted (ww-)factor, which measures the degree of constriction in the hysteresis loops, as explained in the text, plotted versus site number. The higher the ww-factor, the greater the degree of constriction; only a few chips had a ww-factor of one, and no samples showed potbellied loops [29]. ww-Factors have been measured for about 75 chips and have been averaged by sample.

size remain entirely consistent with an interpretation of the decay of IRM_{77K} during warming as being due to the presence of SP grains.

A separate line of evidence is, moreover, supportive of the presence of SP grains; namely the wasp-waisted nature [29] of the hysteresis curves (Fig. 6). With the exception of samples from site 3, where the ascending and descending lines of the hysteresis curve are mostly parallel until they become converging, the curves from site 1 and 2, and to a lesser extent from site 4, are constricted. In order to quantify the constriction, a wasp-waisted (ww-) parameter has been devised, by taking the ratio of two values, ΔH_{max} and ΔH_0. ΔH_0 is measured parallel to the x axis at $y = 0$, whereas (ΔH_{max} is also measured parallel to the x axis but at that value of the magnetization moments where the ascending and descending curves are farthest apart. Wasp-waisted hysteresis curves will thus have a ww-factor that is > 1.0, and the greater the ww-factor the greater the relative constriction. For potbellied curves (for definition, see [29]), where $\Delta H_0 = \Delta H_{max}$, this ww-parameter has no applicability, but we have not observed any potbellied curves. The ww-factors, averaged by core sample, are plotted versus site number in Fig. 6, and it can be seen that the degree of constriction decreases, on average, with increasing age. This is again consistent with a decreasing contribution in older samples from superparamagnetic grains. However, other possible explanations must once more be discounted before we can accept this as firm evidence. It has long been recognized that constriction can also be caused by a bimodal coercivity distribution, such as might arise from a mixture of titanomagnetite and titanomaghemite or titanohematite. In order to see whether bimodal coercivity distributions are present, we have analyzed ΔJ curves (the difference between the y values of the ascending and descending curves as a function of H), as suggested by Tauxe et al. [29]. No significant difference was found between the ΔJ–H plots for selected samples from sites 1, 2 and 4, and a smooth decay of ΔJ versus H was observed; we conclude that the absence of ''roller coaster'' behavior (as defined in [29]) suggests that the ww-factors may indicate variable, but (on average) decreasing contributions from SP grains.

4. Discussion

It appears that, as our MORB rocks aged, their Fe–Ti-oxides became more PSD- to MD-like. We infer from this that, as the pillow lavas aged and the larger titanomagnetites became progressively more oxidized [17], they also lost SP and SD titanomagnetite grains, due to alteration to goethite or nonmagnetic phases. From these observations and from our study of maghemitization [17], we conclude that alteration processes appear to continue on a timescale of tens of millions of years.

It is not possible to quantify either the earlier volumetric importance or the magnetic contributions of the SD grains that are no longer present in samples from sites 3 and 4, but a semi-quantitative model of the contributions to J_{NRM} as a function of age is presented in Fig. 7. As the large and optically visible MD grains are oxidized to titanomaghemite without much apparent volume loss [17,18], the smallest SP grains are lost first and the submicrometer SD grains later, by alteration to non-magnetic

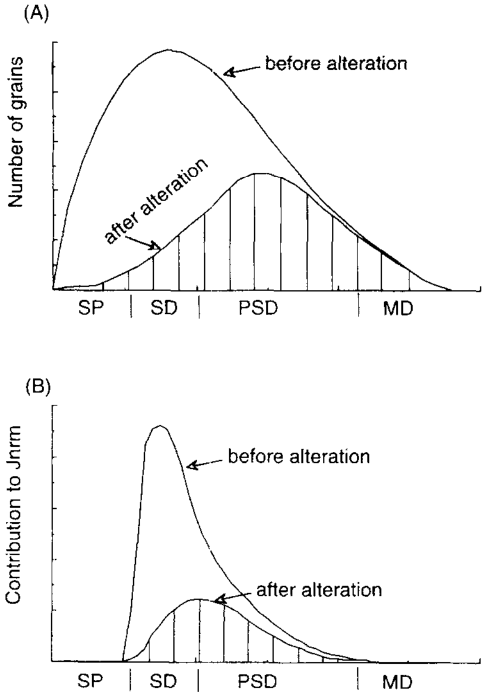


Fig. 7. Model of the impact of alteration, as the MORB samples aged up to 70 Ma, on size distribution of magnetic grains (A) and their inferred contribution to the intensity of NRM (B). Diagram axes are not to scale and are qualitative only.

phases. This not only explains the lack of significant change in J_s (Fig. 1C), which is dominated by the volumetrically important MD grains, but also the change in hysteresis parameters which are sensitive to the SD fraction. Whereas it is possible, even likely, that maghemitization causes some reduction in J_{NRM}, we conclude that an important, and perhaps the principal, contribution to J_{NRM} of single-domain sized titanomagnetite in fresh (young) MORB samples is degraded by alteration processes that produce magnetically unimportant phases. If our conclusions can be substantiated by future work on many more samples, and provided that the alteration patterns in dredged basalts are representative of the magnetic source layer (which certainly also needs further study), then it can be argued that the pronounced decay of marine magnetic anomalies over times of tens of millions of years could be caused, in large part, by this alteration.

Our study is also of importance for paleointensity studies of glasses in older MORB [32], because knowledge about the alteration, or lack thereof, is critical for the assumption that a primary thermoremanent magnetization is being analyzed in paleointensity experiments. While rim-glass and, occasionally, interstitial glass can apparently survive without alteration, it is clear from this study, as well as our work in progress, that much of the glass in samples of Paleogene or Cretaceous MORB is completely altered to clays, and that the fine-grained titanomagnetite in the glass is also subject to alteration and dissolution, rendering the material less appropriate for paleointensity studies.

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