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# Instability of thermoremanence and the problem of estimating the ancient geomagnetic field strength from non-single-domain recorders

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**Data on the past intensity of Earth's magnetic field (paleointensity) are essential for understanding Earth's deep interior, climatic modeling, and geochronology applications, among other items. Here we demonstrate the possibility that much of available paleointensity data could be biased by instability of thermoremanent magnetization (TRM) associated with non-single-domain (SD) particles. Paleointensity data are derived from experiments in which an ancient TRM, acquired in an unknown field, is replaced by a laboratory-controlled TRM. This procedure is built on the assumption that the process of ancient TRM acquisition is entirely reproducible in the laboratory. Here we show experimental results violating this assumption in a manner not expected from standard theory. We show that the demagnetization−remagnetization relationship of non-SD specimens that were kept in a controlled field for only 2 y show a small but systematic bias relative to sister specimens that were given a fresh TRM. This effect, likely caused by irreversible changes in micromagnetic structures, leads to a bias in paleointensity estimates.**

paleomagnetism | paleointensity | thermoremanent magnetization | multidomain

The aim of paleointensity research is to reconstruct variations in the absolute intensity of the ancient geomagnetic field throughout Earth history. Paleointensity data are essential for constraining the conditions in the core (1–4), for studying the role that the geomagnetic field plays in controlling Earth's atmosphere (5–7), and as a geochronological tool (8, 9). Despite the necessity for a large amount of reliable paleointensity data, there are still significant ambiguities in the available paleointensity information (10–12); these call for a reevaluation of the existing database (13).

One fundamental problem in paleointensity research arises from the difficulty in locating dateable ancient materials that fulfill the most basic requirement of the absolute paleointensity method. This requirement states that the natural remanent magnetization (NRM) should be a pure thermoremanent magnetization (TRM) carried exclusively by noninteracting single-domain (SD) particles (14). As purely SD materials are rare in nature, much of the published data are based on materials that do not entirely fulfill the strict assumption of pure SD, but still demonstrate a reasonably satisfying relationship between the blocking and unblocking temperatures. That is to say, pseudo SD (PSD) or even small multidomains (MD) are frequently assumed to carry stable and reproducible magnetizations.

We first outline the principles of the absolute paleointensity method (14, 15), as most of the data in the paleointensity database rely on some variant of this method (10, 16). The basic underlying assumptions of any absolute paleointensity method are that TRM is quasi-linearly proportional to the intensity of the field ($B$) in which it was acquired ($\mathrm{TRM} = \alpha \cdot \mathsf{B}$), and that in the absence of chemical and physical alteration, the proportion between the TRM and $B$ is an intrinsic property of the sample that does not change with time. The laboratory procedure in the Coe variant of the Thellier approach (17), widely considered one of the most robust of the many methods, involves a series of double heating steps at progressively elevated temperatures through which the ancient TRM ($\mathrm{TRM_{anc}}$) is gradually replaced by a laboratory TRM ($\mathrm{TRM_{laboratory}}$) acquired in a controlled field. Each double heating step includes one demagnetization procedure in zero field and one remagnetization procedure in the presence of a fixed laboratory field ($\mathrm{B_{laboratory}}$). After each procedure, the magnetization vector is measured and the portion of NRM lost and the partial TRM (pTRM) gained are calculated and plotted on an Arai diagram (18) (Fig. 1). Ideal Arai plots yield data points scattered along a perfectly straight line with a slope of $\mathrm{TRM_{anc}}/\mathrm{TRM_{lab}}$, from which the paleointensity can be calculated by $B_{\mathrm{anc}} = B_{\mathrm{laboratory}} \cdot \mathrm{slope}$.

In this work, we inspect the most fundamental assumption of all paleointensity methods, taken as true a priori: that TRM (not overprinted by viscous remanent magnetization, VRM) is stable through time unless chemical or physical alteration occurs. If this assumption is true, then the process of TRM acquisition can be reproduced in the laboratory, and $\mathrm{TRM_{laboratory}}$ can be compared with $\mathrm{TRM_{anc}}$. A violation of this assumption calls the reliability of all absolute paleointensity procedures into question. Putting this assumption more simply, if paleointensity experiments were carried out on two identical specimens that acquired their TRMs in the presence of the same field but at different times, one holding a fresh TRM and another holding an aged TRM, the results of the paleointensity experiments should be exactly the same. To test this assumption, we examine two groups of sister specimens, one with a "fresh TRM" and another with 2-y-old "aged TRM." Surprisingly, we find a small but systematic change in the shape of the remanence decay curves, which affect the Arai plots and lead to a systematic bias in the interpretation.

## Significance

**The evolution of Earth's magnetic field is one of the greatest riddles of Earth's past. Despite decades of paleomagnetic research, some fundamental properties of the geomagnetic field, such as the nature of its intensity fluctuation (paleointensity), are still elusive. Paleointensity is recovered from ancient materials that were thermally magnetized in the presence of the ancient field. The paleointensity procedure is based on the assumption that the ancient magnetization is stable with time. Here we show that this assumption is violated for many of the widely used materials, such as, for example, crystalline volcanic rocks. Our results put in question the reliability of much of the available paleointensity information, posing new challenges to our understanding of the ancient Earth.**

Author contributions: R.S. and L.T. designed research, performed research, analyzed data, and wrote the paper.

The authors declare no conflict of interest.

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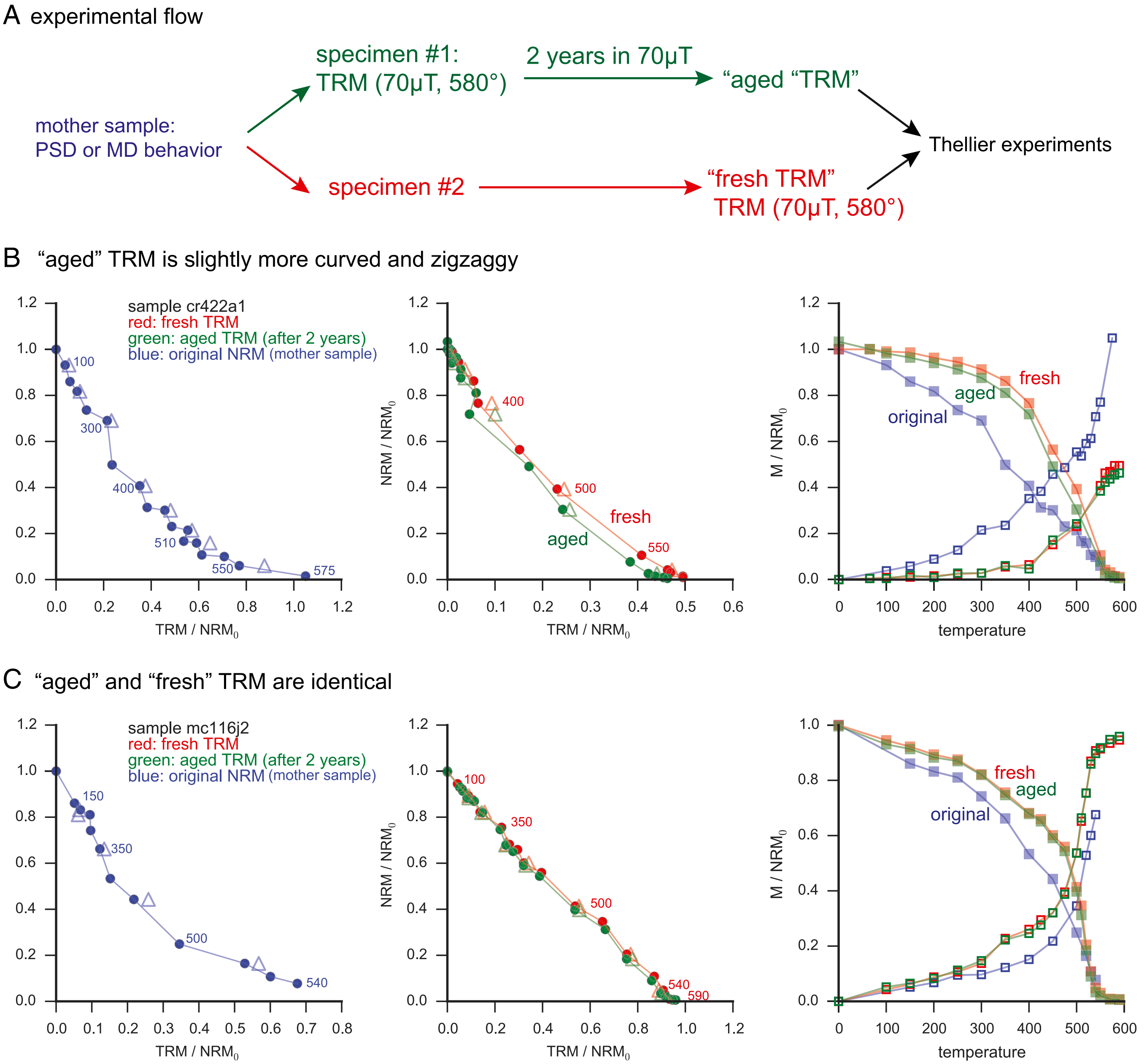

**Fig. 1.** Paleointensity experiments on fresh and aged TRM. (*A*) Experimental workflow. (*B*) A representative case showing the typical difference between the aged (green) and the fresh (red) TRM, where the aged TRM specimen has a slightly more curved and zigzagged Arai plot. (*C*) An example of two sister specimens yielding identical behaviors, demonstrating that *B* is not a result of experimental noise. All measurements are normalized to NRM$_0$, the initial measurement after TRM acquisition, before TRM aging. (*B* and *C*) (*Left*) Arai plots of the mother sample with the original ancient NRM. (*Middle* and *Right*) Arai plots (*Middle*) and demagnetization/remagnetization plots (*Right*) of the aged and fresh TRM.

## Methods

In our experiments (shown schematically in Fig. 1*A*), we used samples that had already undergone Thellier-type experiments in the paleomagnetic laboratory at the Scripps Institution of Oceanography (SIO), showed no alteration in the original experiments, and demonstrated behaviors attributed to small MD or PSD (i.e., curved and/or zigzagged Arai plots, Fig. 1 *B* and *C*, *Left*). From a collection of "mother samples," we assembled two groups of nearly identical "sister specimens" by breaking each sample into smaller pieces. The samples were mostly volcanic rocks from different locations: McMurdo (19), the Island of Socorro (20), the Snake River Plain (21), Jan Mayen (22), Costa Rica (23), Spitsbergen (22), and Hawaii (24); some specimens are archaeological and synthetic slag samples (25, 26).

The experiments were done in the SIO paleomagnetic laboratory. We first thermally demagnetized all of the specimens using an air-cooled laboratory-built paleointensity oven at 580 °C (Fig. 1*A*). After the magnetizations were measured, one group, consisting of 181 specimens, was given TRM in a 70-μT field by cooling from 580 °C. Specimens from this group were kept in a fixed field of 70 μT parallel to the direction of the NRM, and were allowed to age for 2 y at room temperature. According to Néel theory (27), uniaxial SD particles should not experience any change in their net magnetization, as there is insufficient energy to switch between the two energetically stable magnetization states. However, it is not clear if non-SD particles that have multiple stable magnetization states can experience change in their NRM in these conditions. For this reason, the NRMs of the specimens from this group were routinely measured. As shown below, we observed that some specimens did gain some VRM.

Two years after putting the first group in a fixed field, we gave the second set a fresh TRM in the same field, i.e., 70 μT, by cooling from 580 °C. This resulted in two groups of specimens (aged and fresh) that differ in only one significant respect: the time elapsed from the TRM acquisition. We carried out the "IZZI" variant of the Thellier experiment (28) on a subset of 118 pairs of sister specimens (aged and fresh) from both groups using identical procedures and temperature steps using oven fields of 30 μT or 70 μT. The data were analyzed using the PmagPy Thellier GUI program (29). Under the assumption of a stable TRM, the specimens in each pair should behave exactly the same. However, as mentioned above, the aged and the fresh TRM specimens yielded surprisingly different behaviors.

## Results

**Viscous Remanent Magnetization.** Fig. 2 shows the normalized magnetization of the specimens from the first group (total of 181 specimens) during the 2 y of aging. The magnetization of most specimens did not exhibit significant change. However, several specimens demonstrated a rapid increase in magnetization of up to 12% over a period of 2 wk, followed by a slower increase in the subsequent measurements. When we switched off the external field after about 2 y, this VRM decayed and approached the initial magnetization. There are some differences between the locations, whereby the volcanic rocks of Costa Rica showed the largest change in magnetization.

**Different Behaviors in the Paleointensity Experiments.** The most striking result from our TRM aging experiments is that the fresh TRM and the 2-y aged TRM demonstrated small but systematic differences in behavior during the paleointensity experiments. Fig. 1*B* shows a representative result where the shape of the Arai plots and the demagnetization curves show dependency on the age of the TRM. The aged TRM specimen yielded a slightly more scattered and curved Arai plot, and the demagnetization temperature spectrum shifted toward lower temperatures. This result was not observed in all of the specimens. Fig. 1*C* shows an example of identical behavior for the fresh and the aged TRM (Arai plots similar to Fig. 1 and measurement data file are given in Datasets S1 and S2). This confirms that the difference shown in Fig. 1*B* is not caused by an experimental error but, instead, by a viscous mechanism.

We note that there is also a difference between the behavior of the original mother sample (Fig. 1 *B* and *C*, *Left*), and our current experimental results. The original experiments yielded curved and zig-zagged Arai plots attributed to non-SD. However, our experiments on the laboratory TRMs showed suppressed MD behavior, i.e., straighter Arai plots. This effect may, in part, be due to the difference in the experimental protocols. In the original experiments, the angle and ratio between $B_{\mathrm{laboratory}}$ and $B_{\mathrm{anc}}$ were arbitrary whereas, in the new experiments, $B_{\mathrm{laboratory}}$ is parallel to $B_{\mathrm{anc}}$ and the ratio is 1 (when the oven field was 70 μT) or smaller (when the oven field was 30 μT). The suppression of MD behavior is in agreement with Shaar et al. (26) and Yu et al. (30), who demonstrated that $B_{\mathrm{laboratory}}$ parallel and smaller than $B_{\mathrm{anc}}$ minimizes non-SD effects in IZZI Thellier experiments.

To quantitatively assess the differences between the Arai plots of the fresh and the aged TRM specimens, we calculate two key paleointensity statistics for all of the specimens. The $k$ (curvature) statistic (31) provides an indication for the fit of the Arai plot to a circle. The $\beta$ (scatter) statistic (16, 32) quantifies the scatter of the data points about the least-squares best-fit line. The calculation was made using the entire temperature spectrum (i.e., all of the points in the Arai plot). Fig. 3 *A*, *B*, *D*, and *E* shows, for each pair of sister specimens, the difference between the aged TRM statistic and the fresh TRM statistic. Under the assumption that both TRMs are identical, the data points should be equally scattered around a zero value (red line in Fig. 3). However, a large majority of the pairs show systematically higher values of $k$ and $\beta$ for the aged TRM (91 and 103, respectively, out of 118). This means that the aged TRM specimens have more curved and more scattered Arai plots than the fresh TRMs. Fig. 4 shows that this difference in the shape of the Arai plot affects the paleointensity estimation. In almost all of the pairs of aged and fresh TRM specimens, the paleointensity estimation is higher for the aged TRM (102 out of 118). In our experiments in which the aging spanned only 2 y, this difference is in the range of a few microteslas (Fig. 4 *A* and *B*).

Because Figs. 3 and 4 show paleointensity statistics calculated using the entire temperature interval, we repeated the paleointensity calculations for different starting temperatures, ranging from 0 °C to 350 °C (*SI Appendix*, Fig. S1). We found a substantial bias in $k$ statistic, $\beta$-statistic, and paleointensity estimate for the other temperature bounds. This indicates that the entire blocking/unblocking temperature spectrum is affected by the aging of the TRM and not just the low temperature spectrum and implies that any absolute paleointensity method, or method that relies on calibration by absolute methods, will be affected.

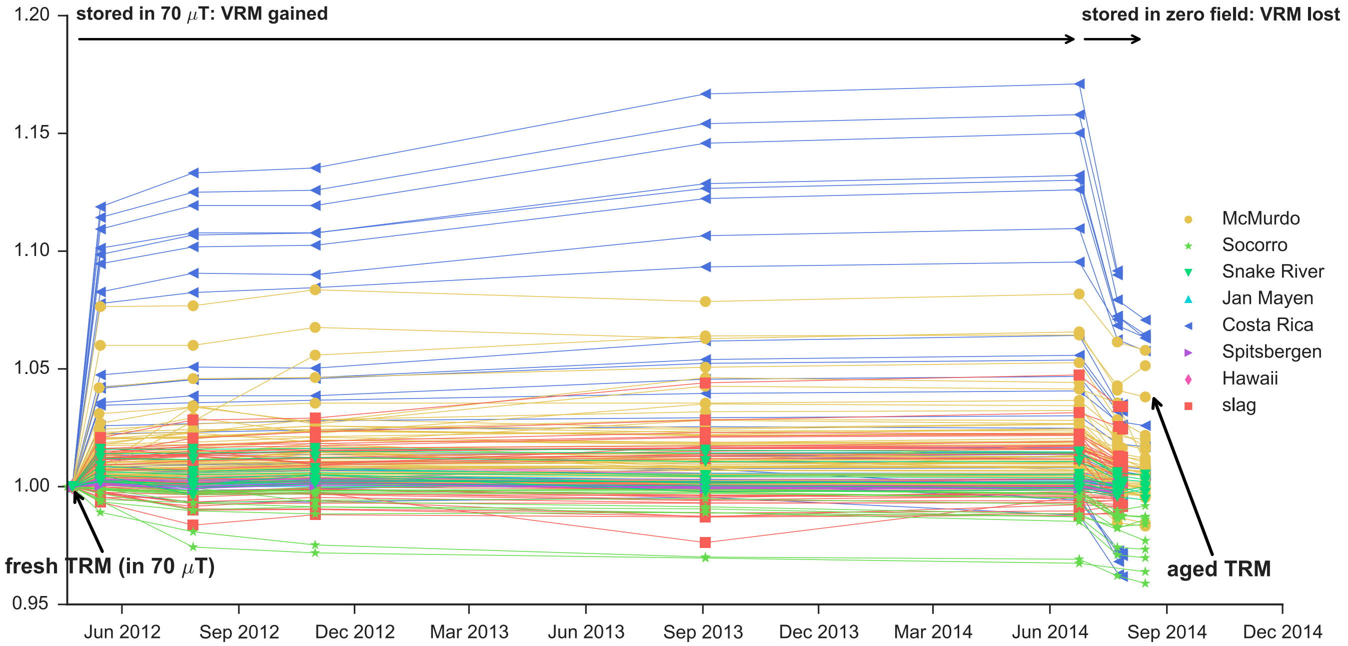

**Fig. 2.** Magnetization measurements during TRM aging. A total of 181 specimens were given a fresh TRM in $B_{\mathrm{laboratory}} = 70$ μT and then stored in a field parallel and equal to $B_{\mathrm{laboratory}}$. After about 2 y, the field was turned off. Some specimens show rapid increase in magnetization during the first 2 wk of the experiment and a decrease at a much slower rate after switching of the field.

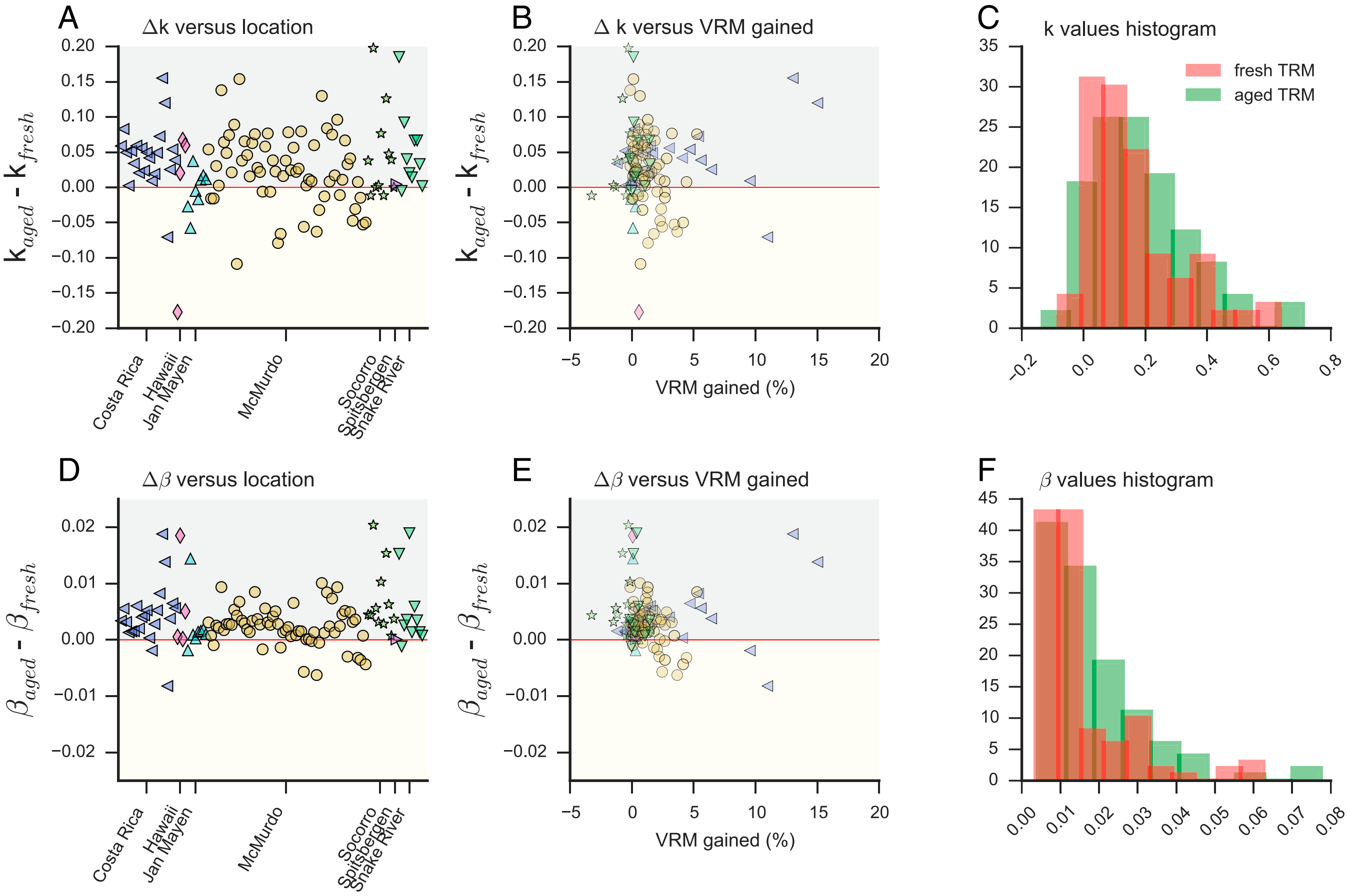

**Fig. 3.** Increase of the curvature and the scatter of the Arai plots caused by TRM aging. A total of 118 pairs of sister specimens with aged and fresh TRMs are shown. (*A*, *B*, *D*, and *E*) Difference between the value of the aged TRM specimen and the fresh TRM specimen. (*A*–*C*) Curvature statistics *k* (31): 91 pairs show increased curvature in the aged TRM specimen. (*D*–*F*) Scatter statistic *β* (32): 103 pairs of specimens show increased scatter in the aged TRM specimen. (*A* and *D*) Results plotted versus location. (*B* and *E*) Same as *A* and *D*, but plotted versus VRM gained (percent), calculated from Fig. 2. (*C* and *F*) Histogram of the calculated values.

**Paleointensity Dependency on Curvature, Scatter, and VRM Gained.** To see if there is a dependency of the intensity estimates on the values of the *k* statistic and *β*-statistic, we plot in Fig. 5 *A* and *B* the paleointensity values obtained from all specimens (aged and fresh) versus the paleointensity statistics. Fig. 5 shows that for high values of scatter and curvature, it is likely that an inaccurate paleointensity value will be obtained.

Fig. 5*C* displays the paleointensity values of the aged specimens versus VRM gained (the percentage of NRM increase after stored in constant field for 2 y). It can be seen that there is a dependency

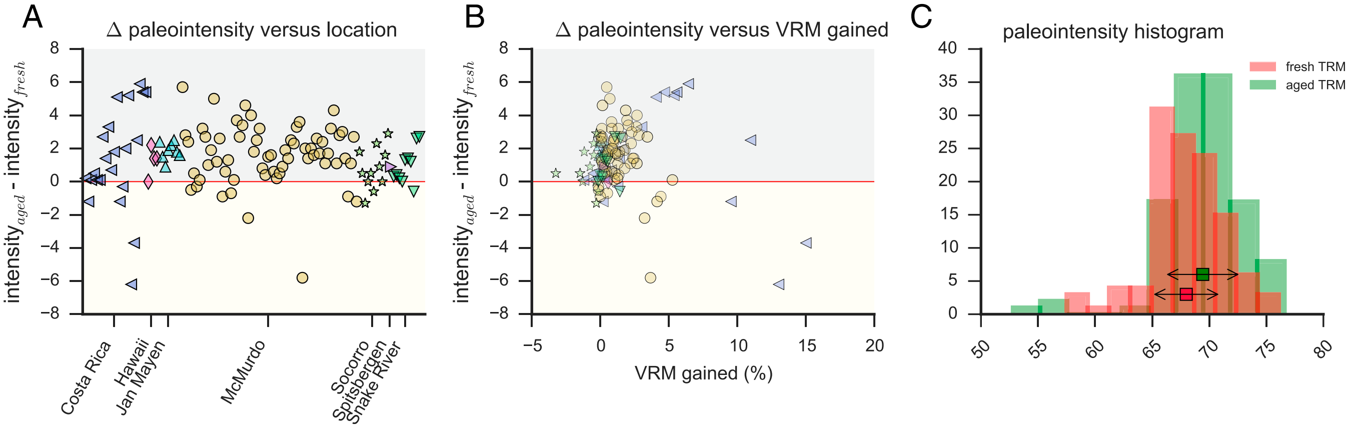

**Fig. 4.** Increase of the paleointensity estimation (calculated using the entire temperature spectrum) caused by TRM aging. A total of 118 pairs of sister specimens with aged and fresh TRMs are shown; 102 pairs show higher paleointensity for the aged TRM. Colors and symbols are as in Fig. 3. (*A* and *B*) Difference between the paleointensity values of the aged TRM specimen and the fresh TRM specimen. (*C*) Histograms of the paleointensity values. Symbols and arrows in the histogram show the mean and the SD.

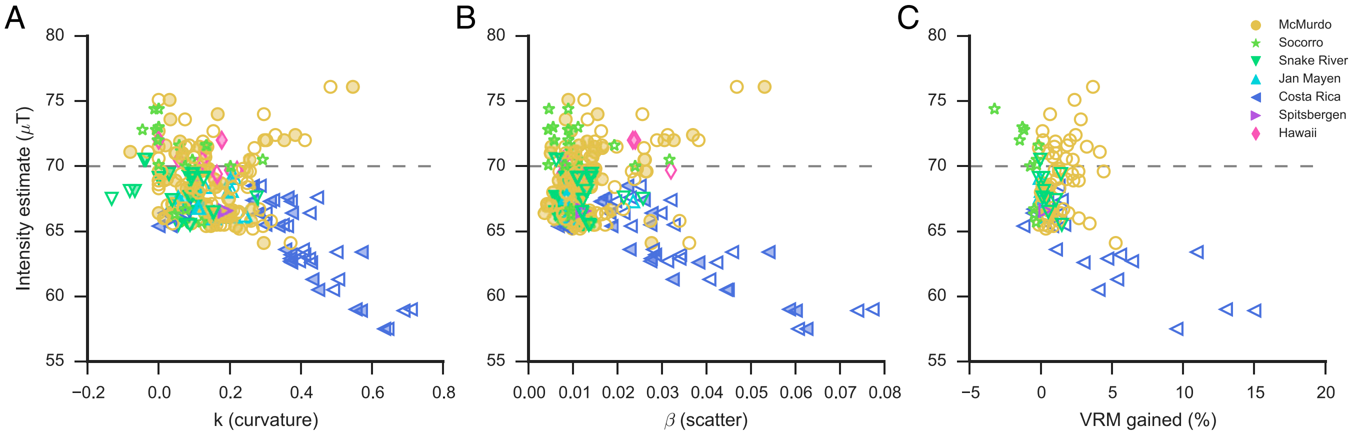

**Fig. 5.** Dependency of the paleointensity estimates (calculated using the entire temperature spectrum) on the curvature statistic (*A*), the scatter statistic (*B*), and the VRM gained (*C*). Open (closed) symbols denote aged (fresh) TRM. Despite none of the specimens being ideal SD, as demanded by the paleointensity theory, the accuracy of most specimens is in the range of ±10%. However, curved and scattered Arai plots tend to yield underestimated paleointensities. Specimens that gained more than 4% tend to yield inaccurate paleointensity.

of the paleointensity accuracy on the VRM gained. The viscous magnetization effect yields underestimated paleointensities.

## Discussion

**Summary of the Key Observations.** In our experiments, we conducted paleointensity experiments on 118 pairs of nearly identical sister specimens. The only difference between the sister specimens is the time elapsed between the TRM acquisition and the Thellier experiments. We observed a small but systematic difference between the aged TRM and the fresh TRM specimens. The difference is observed in the curvature and the scatter of the Arai plot, and the demagnetization temperature spectrum. These differences lead to a systematic bias in paleointensity calculation (Fig. 4). Figs. 4*B* and 5*C* show that the bias in the paleointensity may be linked to the VRM effect shown in Fig. 2, as specimens that exhibited a change of more than 4% in their NRM during the aging process tended to yield inaccurate paleointensity estimation.

Our sample collection comprises samples collected from different locations and geological settings. Figs. 2–4 show that the results depend on the geological setting (influenced by the dominant mineralogy and grain size of the ferromagnetic particles). For example, the Costa Rican samples were most sensitive to VRM acquisition (Fig. 1) and showed the most significant bias in paleointensity (Fig. 4*A*). On the other hand, the Jan Mayen and the Hawaiian samples were less sensitive.

**Possible Mechanisms.** We suggest that the primary cause for the behavior observed in our experiments is instability of TRM in non-SD particles. This instability is driven by irreversible changes in complicated micromagnetic structures. Complicated micromagnetic structures such as, for example, "vortex" (33), unlike the SD state, have multiple metastable energy states. Theoretically, a particle can experience a shift between nearby local energy minima (LEM), depending on the energy barriers and the amount of external energy applied. In our experiments, the aged TRM specimens were kept at room temperature in a fixed field parallel and equal to the TRM field. This was done in an effort to minimize the changes in external energy. We conclude that irreversible changes in the micromagnetic structure at room temperature do occur frequently in many samples generally used for paleointensity experiments. This can be explained qualitatively by considering TRM as a nonequilibrium state of the magnetization with the external field. With sufficient time and energy, the micromagnetic structure will approach equilibrium with the external field. We see that approaching this equilibrium state (with stronger magnetization; Fig. 2) can occur in a very short time span at room temperature. Moreover, we suggest that natural materials, such as volcanic rocks, which are exposed to constant changes in the ambient magnetic fields and temperatures, can be affected more profoundly by thermoremanent instability.

Instability of metastable partial TRM was recently reported by de Groot et al. (34). They used magnetic force microscopy to observe changes in domain configurations after imparting partial TRMs (pTRM). They showed that pTRM of large MD particles are metastable and can experience significant change in domain configuration in zero-field conditions. Their concluding remarks are that whether such type of viscous magnetization driven by LEM reconfiguration can affect the NRM of a natural sample is enigmatic. Here we amplify on their observations and demonstrate that TRM instability may be a primary cause for irreversible change in the NRM.

**Implication for Paleointensity Studies.** Our results are applicable to the general field of paleointensity estimation, as they are based on typical rocks chosen for paleointensity experiments (mostly basaltic lava flows) with PSD grain sizes. One important consequence of TRM instability is that ancient materials affected by this mechanism can yield biased paleointensity estimates regardless of the paleointensity method used, including ones involving the use of total TRMs (ref. 35 and its derivatives), microwave-induced TRMs (ref. 36 and its derivatives), or partial TRM replacement (37).

The theory underlying the Thellier method demands that TRM can be treated as the sum of independent and reciprocal pTRMs, and that pTRMs are gained and removed at the same temperature (14). These properties are attributed only to SD. Non-SD particles can have complicated blocking/unblocking temperature functions spanning the entire temperature range below the Curie temperature (38, 39). Furthermore, non-SD can show dependency on the experimental history of the sample (40, 41) that leads to complicated behavior during the paleointensity experiment (42). Hence, irreversible changes in the micromagnetic structure of non-SD alters the blocking–unblocking relationships as well as the demagnetization–remagnetization relationships over the entire temperature spectrum below the Curie temperature, and not only the low-temperature portion. Consequently, the shape of the Arai plot is irreversibly distorted. Therefore, excluding the low-temperature segments in the Arai plots is not necessarily helpful. Also, thermoremanence instability is not associated with a magnetization overprint in a direction different than the NRM, and is therefore not detectable in Zijderveld plots.

Interestingly, despite the fact that none of the specimens is SD, the accuracy of most of the paleointensity estimates is in the range of ±10% (Fig. 5). Also, the 1σ intervals of the averaged paleointensities of both aged and fresh TRMs include the expected value of 70 μT (Fig. 4*C*). This is an encouraging result. The aged

TRM yields a higher paleointensity, very close to the expected field, but the scatter of the results is higher.

The observation of higher paleointensity values for aged TRM is consistent with the results of Cromwell et al. (24), who analyzed 13 published paleointensity studies of 1960 Kilauea flow and compared them to the expected International Geomagnetic Reference Field. This flow was kept in a relatively stable field for few decades in a "natural lab." Using bootstrap resampling, Cromwell et al. demonstrated that there is an overall high bias of the results regardless the method and the criteria used (the exception being the truly SD behavior observed in the glassy flow tops, which showed no such bias). This biased high result may be, in part, due to the effect discussed here. In another effort, Paterson et al. (13) analyzed published datasets from samples that acquired magnetizations in known fields. They showed that the distribution of the paleointensity data, regardless of the acceptance criteria typically used, is systematically skewed. Altogether, these observations lead us to posit that, perhaps, the overall paleointensity database may be largely biased, as many results are derived from nonideal SD-like materials. We note that the use of very strict acceptance criteria in Cromwell et al. (24) resulted in accurate and precise estimates of historical field values from Hawaiian glassy lava flow tops, but these criteria are rarely met in paleointensity experiments using typical (non-SD) materials.

We do not know if the paleointensity database tends to be biased high or low, as the bias may be dependent on the ambient condition. Still, it is reasonable to infer that ancient rocks that acquired their TRM thousands or millions of years ago might have a profound paleointensity bias, much larger than in our experiment spanning only 2 y. This bias is difficult to assess without access to the original data to calculate curvature and scatter.

Given the results reported here, one of the greatest challenges in the next generation of the paleointensity databases is to estimate the degree of this so-called "non-SD thermoremanent instability." To put forward a methodology for evaluating possible bias in the overall published data, it is essential to have the measurements data from which the published interpretations are derived. This can be done through the MagIC database (43) (earthref.org/MAGIC/), and we stress the necessity of publishing the entire measurement data of any paleomagnetic experiment.

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