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Comparison of Thermal and Microwave Paleointensity Estimates in Specimens Displaying Non-Ideal Behavior in Thellier-Style Paleointensity Experiments

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Abstract Determining the strength of the ancient geomagnetic field is vital to our understanding of the core and geodynamo, but obtaining reliable measurements of the paleointensity is fraught with difficulties. Over a quarter of magnetic field strength estimates within the global paleointensity database from 0 to 5 Ma come from Hawaii. Two previous studies on the SOH1 drill core gave inconsistent, apparently method-dependent paleointensity estimates, with an average difference of 30%. The paleointensity methods employed in the two studies differed both in demagnetization mechanism (thermal or microwave radiation) and Thellier-style protocol (perpendicular and original Thellier protocols)—both variables that could cause the strong differences in the estimates obtained. Paleointensity experiments have therefore been conducted on 79 specimens using the previously untested combinations of thermal-perpendicular and microwave-original Thellier methods to analyze the effects of demagnetization mechanism and protocol in isolation. We find that, individually, neither demagnetization mechanism nor protocol entirely explains the differences in paleointensity estimates. Specifically, we found that non-ideal multidomain-like effects are enhanced using the original Thellier protocol (independent of demagnetization mechanism), often resulting in paleointensity overestimation. However, we also find evidence, supporting recent findings from the 1960 Kilauea lava flow, that microwave-perpendicular experiments performed without partial thermal remanent magnetization checks can produce underestimates of the paleointensity due to unaccounted-for sample alteration at higher microwave powers. Together, these findings support that the true paleointensities fall between the estimates previously published and emphasize the need for future studies (thermal or microwave) to use protocols with both partial thermal remanent magnetization checks and a means of detecting non-ideal grain effects.

1. Motivation

The Pacific Ocean covers 30% of the Earth’s surface but has few islands, with the Hawaiian Islands being some of the most easily accessed. Volcanic islands, like Hawaii, contain some of the best records of the temporal variation in Earth’s magnetic field over the last few million years. Paleosecular variation timescales of this length are necessary to better understand long-term variations in geomagnetic behavior, as well as crust, mantle, and core interactions (e.g., McElhinny & Merrill, 1975). Accurate paleointensity (PI) data in the 0–5 Ma period are crucial because only in this interval is there enough spatial and temporal global coverage of data to characterize long-term (million years) variations. In order to obtain accurate PI data, an appropriate PI method for the mineral magnetic characteristics of any particular site must be used.

Many paleomagnetic studies over the last 60 years (e.g., Coe et al., 1978; Cromwell et al., 2018; de Groot et al., 2013; Doell & Cox, 1963, 1965; Teanby et al., 2002) have found the Hawaiian Islands ideal for studying magnetic field variations in the central Pacific Ocean over the past hundred to few million years. Hawaiian absolute PIs have substantial temporal coverage and comprise 28% of the global PI (PINT) database in this interval (Biggin et al., 2015) and are therefore important to study to understand long-term field behavior over this time interval. Numerous studies have taken advantage of the drill core from the Scientific Observation Hole (SOH) and Hawaii Scientific Drilling Project (HSDP) to extract the required PI data over the last 45 kyr
The paleomagnetism of the SOH1 core was studied twice previously—once by Teanby et al. (2002) using thermal PI experiments and again by Gratton et al. (2005) using microwave (MW) PI experiments and a different PI protocol. Teanby et al. (2002) and Gratton et al. (2005) each sampled the core independently and extracted PIs from 83 common flows. Teanby et al. (2002) additionally reported a new inclination record and dated the flows from 0 to 45 ka. The mean PIs reported by the two studies, 33.5 (Teanby et al., 2002) and 25.1 μT (Gratton et al., 2005), differ by approximately 33%. Both studies reported mean uncertainty estimates of approximately 10%, which implies the possibility for a resolvable difference between them.

At this stage, it is useful to introduce our nomenclature that a given PI "method" is composed of a combination of a specific "demagnetization mechanism" and a specific "protocol." Teanby et al. (2002) used the conventional thermal demagnetization mechanism paired with the original Thellier (OT) protocol (Thellier & Thellier, 1959), while Gratton et al. (2005) used the MW demagnetization mechanism paired (predominantly) with the perpendicular (Perp) protocol (Kono & Ueno, 1977). A third study, by Laj et al. (2011), used the raw data from Teanby et al. (2002) (reanalyzed with their updated selection criteria to give an SOH1 average of 29.7 μT) combined with additional (non-SOH1) data (acquired using the same method) from the SOH4 and HSDP1 drill cores, which cover additional flows, to create a more complete and statistically rigorous Hawaiian PI record. Even after the Laj et al. (2011) reassessment, there remains nearly a 20% discrepancy between the thermal mechanism OT protocol and the MW mechanism Perp protocol results.

To investigate discrepancies between PI results obtained using different demagnetization mechanisms (i.e., thermal and MW), a meta-analysis of 13 paired studies (including that of the SOH1 drill core) was undertaken by Biggin (2010). It is important to note that the studies assessed in the Biggin (2010) analysis differed not only in demagnetization mechanism but also in protocol. Biggin (2010) concluded, first, that systematic differences existed between PI estimates derived from thermal and MW experiments performed on the same rocks, with the former tending to be significantly higher than the latter (at the 95% confidence level). We carried out a further analysis (available in Supporting Information A), which showed this as well at the flow level. Second, Biggin (2010) suggested that the most plausible explanation for the bulk of these discrepancies resided in unrecognized biasing from multidomain-like effects being more prevalent in the thermal results. Importantly, it was suggested that the discrepancies were more or less entirely due to the difference in the protocols used rather than in the demagnetization mechanism. With respect to the SOH1 case, Biggin (2010) hypothesized that the thermal results being higher than the MW results was likely unrelated to the choice of thermal or MW energy for demagnetization. Rather, the discrepancy was due to Teanby et al. (2002) employing the OT protocol while Gratton et al. (2005) employed the Perp protocol.

Since our initial flow-level analysis of the SOH1 data confirmed the results of Biggin (2010), the aim of the present study is to test the hypothesis that the differences found in PI results from the SOH1 core are entirely due to protocol and not due to demagnetization mechanism. We hypothesize that if the demagnetization mechanism-protocol pairs are reversed, such that MW demagnetization is paired with the OT protocol and thermal demagnetization is paired with the Perp protocol, then the sense of the discrepancy between the MW and thermal results should reverse such that the former should give higher estimates than the latter. This study will explicitly test this hypothesis using new experiments performed on 24 of the same SOH1 flows as studied originally by both Teanby et al. (2002) and Gratton et al. (2005).

The results of this study are important on several levels. First, they provide improved insight into the strength of the magnetic field at the time of emplacement of the 241 flows sampled by the SOH1 drill core data set. Second, they have implications for how the swathes of PI estimates obtained by similar methods from rocks elsewhere in the world should be interpreted. As such, they expand the results of a recent restudy of the 1960 Kilauea lava flow by Grappone et al. (2019) to more Hawaiian lava flows. Lastly, they can provide guidance on how future PI experiments should be performed and analyzed in order to maximize their reliability.

2. Drill Core Geology and Sampling

The samples used in this study are from the SOH1 drill core, which was drilled from the Kilauea volcano on Hawaii Island between 1989 and 1991 at 19°29′N, 154°54′W, to a total depth of 1,685 m. The Hawaii Institute
of Geophysics and Planetology and the Hawaii Natural Energy Institute drilled the borehole to assess the viability of using geothermal energy in the area (Quane et al., 2000). The core consists primarily of ‘a’a (~66%) and pahoehoe (~22%) lavas from 241 aerial, subaerial, and submarine flows with thicknesses varying from 0.3 to 17.4 m (Gratton et al., 2005; Teanby et al., 2002). The remaining ~11% of the core consists of dyke intrusions. Samples were taken from 196 lava flows from the upper 779 m of the core to avoid the increasing number of dyke intrusions and apparent alteration at greater depths (Gratton et al., 2005). This portion of the core has been modeled with an age range of 0–45 ka (Teanby et al., 2002).

The portions of each drilled 2.5-cm diameter core that were saved by Gratton et al. (2005) from their SOH1 study were retrieved from the University of Liverpool archive and subsequently used in this study. We cut 120 new specimens for the restudy from the material remaining from 24 flows that span the range of flows sampled in Gratton et al. (2005). Although the specimens used in this study are sister specimens from Gratton et al. (2005), we cannot rule out the possibility that the flows may be inhomogeneous even on a centimeter scale.

Flows were selected based on the degree of disagreement in PI estimates between the Gratton et al. (2005) and Teanby et al. (2002) studies, number of samples previously studied, and the availability of specimens. For the flows selected for this study, the Teanby et al. (2002) PI estimates, at the flow level, ranged from 21% lower to 53% higher (with a mean of 27% higher) than the Gratton et al. (2005) estimates.

3. Rock Magnetism

Gratton et al. (2005) undertook an extensive survey of the hysteresis loop parameters of the SOH1 borehole. The raw data from their rock magnetic survey were reanalyzed here using HystLab’s automatic hysteresis loop processing program (Paterson et al., 2018) and are replotted in Figure 1, with the flows investigated herein highlighted. The bulk domain stability (BDS) trendline from Paterson et al. (2017) lies below the main sequence of hysteresis loop parameters for SOH1 flows, which indicates that the data have a mixture of magnetic domain types, potentially also including superparamagnetic grains. The specimens used in this study sample the main sequence of SOH1 data observed in Figure 1. The ratio of magnetic remanence to saturation magnetization ($M_r/M_s$) values of the main sequence of SOH1 data lie consistently above the values that would be expected for multidomain grains of magnetite ($M_r/M_s > 0.1$), which implies the presence of single domain and non-single domain grains. Gratton et al. (2005) also determined thermomorphic behavior for all the flows in their study. They found highly reversible thermomorphic curves with 98% of Curie temperatures falling in the range from 520°C to 600°C, with a mean of 561°C and median of 570°C. Twenty-two percent of flows also contained a secondary ferrimagnetic phase with Curie temperatures below 340°C.

The hysteresis and thermomorphic parameters are typical of low-Ti magnetite-rich basaltic lavas found on Hawaii, and these have been the focus of other Hawaiian PI surveys (e.g., Cai et al., 2017; Cromwell et al., 2018; Hill & Shaw, 2000). Additional, new rock magnetic information (first-order reversal curve [FORC] and scanning electron microscopy [SEM] analysis) from the main sequence can be found in Supporting Information B.

4. Methods

All new thermal tests were carried out in air using a Magnetic Measurements MMTD-80 thermal demagnetizer, and specimens were cooled quickly using a built-in cooling fan. The specimens were then measured on the University of Liverpool Geomagnetism Laboratory’s 2G SQUID Magnetometer and RAPID system. All MW tests were run on the 14-GHz Tristan Microwave System (Hill et al., 2008), also at the University of

Figure 1. Hysteresis parameters plot for all the flows in the SOH1 borehole (as studied by Gratton et al., 2005), highlighting which flows were studied herein for new PI estimates. $M_r/M_s$ refers to the ratio of remanent saturation magnetization to the saturation magnetization and $H_{cr}/H_C$ refers to the ratio of coercivity of remanence to coercivity. The bulk domain stability trendline derives from the results of Paterson et al. (2017). The three named flows have new PI estimates, as well as first-order reversal curves (FORCs) and scanning electron microscopy (SEM) analysis in Supporting Information B.
Liverpool. The goal of this study was to replicate the experimental conditions of the previous studies as closely as possible to properly isolate each variable of interest.

In the OT protocol (Thellier & Thellier, 1959), each specimen is heated to a given temperature, \( T_N \), in a known, non-zero intensity magnetic field and then cooled to room temperature and measured. The polarity of the magnetic field is then reversed, and the sample is taken to \( T_N \) again. The protocol can also be referred to as in-field-infield, or “II.” The process is then repeated at \( T_{N+1} \). Partial thermal remanent magnetization (pTRM) checks were included after every second step (i.e., from \( T_{N+1} \) to \( T_{N-1} \)). For consistency with the PINT database (Biggin et al., 2009), when used with thermal energy, this protocol (with pTRM checks) will be referred to as Th-OT+, to acknowledge the pTRM check addition. The MW variant of the OT protocol simply replaces the heat with MW power integrals and will be referred to as MW-OT+. We used powers ranging from 5 to 40 W in 3 to 5 W steps, applied for 5 to 15 s and assumed that any power not reflected was absorbed by the specimen-cavity coupled system. For MW-OT+, we used a steady magnetic field applied parallel/antiparallel to the natural remanent magnetization (NRM), which Biggin (2010) predicted to be the least affected by non-ideal Arai plot behavior. For Th-OT+, we used an applied field with an inclination of ±90° in specimen coordinates.

The Perp protocol is a modification of the OT protocol that only requires a single thermal or MW treatment (Kono & Ueno, 1977). Samples first get stepwise demagnetized in a zero field to remove any soft magnetic overprints. Once the primary component of magnetization is identified as beginning at some \( T_p \), the sample is then heated to \( T_{P+1} \) in a magnetic field applied in the direction perpendicular to the characteristic component (the remaining NRM). The process is then repeated for \( T_{P+2} \) and higher. Gratton et al. (2005) determined that many samples had a second ferrimagnetic phase with an unblocking temperature around 300°C, which they interpreted to be the result of oxyexsolution into Ti-rich and Ti-poor lamellae during extrusion. Therefore, samples undergoing Th-Perp were first stepwise demagnetized in 40–50°C steps from 100°C to 300–340°C to ensure that the perpendicular field was applied only to the high-temperature ferromagnetic phase. After successfully finding the characteristic direction, the field in the oven was switched on. The steps continued to 590°C in 10–30°C increments. The process is the same using the MW system (MW-Perp), but with power integral steps instead of temperature steps until a consistent magnetic direction is obtained (Hill & Shaw, 2007). We did not include any pTRM checks in our new Perp experiments in order to replicate the methods used by Gratton et al. (2005). All data were analyzed using the methods described in Hill and Shaw (2007).

Laboratory field strengths were selected that were as close to the original analyses as possible. All experiments in Teanby et al. (2002) were carried out using an applied field of 40 µT, as were the new MW-OT+ experiments completed herein. MW studies are carried out one sample at a time, so the field strength often varied specimen to specimen within a flow in Gratton et al. (2005). For the specimens we selected, the mean applied field used in Gratton et al. (2005) for MW treatments was 31 ± 1.3 µT, so we used a field of 31 µT in our Th-Perp experiments.

A summary of the experiments carried out in this study is given in Table 1. We ran 79 specimens using either MW-OT+ or Th-Perp. Additionally, 19 specimens were tested using Th-OT+ and 22 were tested using MW-Perp, replicating the original studies in order to confirm the previous results. The median number of specimens tested from each of the 24 flows we examined was 3, with a range of 1–11.

Our selection criteria are based on the MC-CRIT.C1 selection criteria, without tail checks, from Paterson et al. (2015). These were also used successfully by Grappone et al. (2019) to study the 1960 Kilauea lava flow. We relaxed the FRAC/c criterion (Shaar & Tauxe, 2013) (a measure of the proportion of the NRM used to determine the result) from 0.45 (used in Grappone et al., 2019) to 0.35 because of extensive alteration observed at higher temperatures/power integrals and because of difficulties in demagnetizing the specimens using MWs. Relaxing this criterion yielded seven additional specimen-level estimates most notably from MW-Perp experiments, without changing any flow-level estimates in a statistically significant manner. The selection criteria are detailed in Table 2.

For our analysis, we use two statistical tests: the \( t \)-test and the Wilcoxon signed rank test (see, e.g., Klugh, 1986). For the study-level data, which are normally distributed (see the failure to reject the null hypothesis in Kolmogorov-Smirnov test in Supporting Information A), we use a two-sample, unpaired \( t \)-
test to test if the method-level means are equal. A paired difference test is more appropriate for the flow-level data, which have flow-level data pairings. The paired data do not visually appear to be normally distributed (a weak assumption required for a paired t-test), so we should choose a non-parametric test. Biggin (2010) used the Wilcoxon signed rank test in their analysis of the SOH1 data set, so for consistency, we also use that for flow-level data.

### 5. Paleomagnetic Results and Analysis

#### 5.1. New Data

A summary of all experiments run herein is given in Supporting Information C, and these new experiments are described in this subsection. PI estimates that passed the selection criteria were obtained from experiments performed on 73 specimens from 20 flows. A pass rate of >50% at the specimen level was achieved for every experimental method tested. The most common reason for failure of the new MW-Perp and MW-OT+ experiments was low FRAC/f, as the MW often could not fully demagnetize each specimen. The most common reason for failure for the new Th-OT+ experiments was high DRAT (i.e., pTRM check failures), and for Th-Perp, it was high scatter around the best fit line (β). The only clear difference in Arai plot shape observed between the different methods is that the new Th-OT+ data often show two slopes where for some specimens, both slopes passed the selection criteria. In these cases, the low-temperature slope was selected, as they had the higher FRAC. These new Th-OT+ data additionally show the highest mean curvature values (as defined by |K′|) at 0.30, compared with those of the Th-Perp (0.22), MW-OT+ (0.21), or MW-Perp (0.093). All new measurement data can be found on the MagIC database. Examples of passed MW-OT+ and Th-Perp data can be found in Figures 2a.ii, 2a.iii, 2b.ii, 2b.iii, 2c.ii, and 2c.iii. Examples of passed Th-OT+ and MW-Perp data can be found in Supporting Information C.

Flow mean results are detailed by PI method in Supporting Information C. The new Th-OT+ and MW-OT+ flow-level mean PI estimates tend to yield higher values with 44.0 ± 16 μT across five flows and 29.5 ± 9.2 μT, across 19 flows, respectively. New Th-Perp and MW-Perp experiments tend to yield lower mean PI estimates with estimates of 27.8 ± 8.1 μT, across 11 flows, and 18.5 ± 10 μT, across five flows, respectively. If we assume that our new PI estimates are normally distributed, which is noted (see Supporting Information A), we can use a two-sample t-test to determine whether observed differences between experiments are statistically significant. From the new data, the Th-OT+ mean estimate (44.0 μT) is higher than the MW-OT+ mean (29.5 μT), the Th-Perp mean (27.8 μT), and the MW-Perp mean (18.5 μT), at the 95% confidence level, with p values of 0.0147, 0.0176, and 0.0001, respectively. The new data’s Th-Perp mean estimate is not statistically distinct from the new data’s MW-OT+ mean estimate at the 95% confidence level (p = 0.62). The new data’s MW-OT+ flow-level mean PI estimate of 18.5 μT is lower than the new data’s MW-OT+ mean estimate at the 95% confidence level, with a p value of 0.0015. The new data’s Th-Perp and MW-Perp estimates are not statistically different at the 95% confidence interval, with a p value of 0.0676.

### Table 2

**Selection Criteria**

<table>
<thead>
<tr>
<th>N</th>
<th>FRAC/f</th>
<th>β</th>
<th>q</th>
<th>[K′]</th>
<th>MADANC</th>
<th>∆β</th>
<th>DRAT</th>
<th>∆θ</th>
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<tr>
<td>≥4</td>
<td>≥0.35</td>
<td>≤0.1</td>
<td>≥1</td>
<td>≤0.480</td>
<td>≤10</td>
<td>≤10%</td>
<td>≤1°</td>
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</tr>
</tbody>
</table>

Note. N is the number of data points, FRAC/f are measures of the NRM used, β is a measure of scatter around the best fit line, q is a measure of the data quality, [K′] is a measure of Arai plot curvature, and MAD and α determine the scatter of the specimen’s paleodirection. ∆θ is the change in the θ1 + θ2 value, an indication of the perpendicularity between the NRM and TRM directions (Hill & Shaw, 2007) for the perpendicular experiment. For further details, the reader is referred to Paterson et al. (2015).

*aFRAC is used for OT+ experiments and f for Perp experiments. bOT+ techniques use these criteria, but Perp does not, for technical reasons. cUsed only for Perp experiments.*
Figure 2. Normalized Arai plot examples comparing the different methods used. (a) Flow 2 (503-cm thickness), (b) Flow 23 (137-cm thickness), and (c) Flow 206 (290-cm thickness). The data are normalized by NRM0. The filled circles are accepted data points, with the solid black line being the best fit line. Open circles are rejected data points. The black right angle lines are $pT(M)RM$ checks, which are only present in OT+ data. Orthogonal vector plots are provided in core coordinates for the OT+ data. All specimens presented pass their original study’s selection criteria. The microwave data are visually more linear than the thermal data, and the OT+ data are often two-sloped. The powers given for the Gratton et al. (2005) MW data are power applied and the power integrals given in the new data are (inferred) energy absorbed.
5.2. Incorporation of Existing Data Sets

The new Th-OT+ results reported here are broadly consistent with their original counterparts reported by Teanby et al. (2002) and Laj et al. (2011). Both have the same two-slope (concave up) behavior (see Figure 2 for Teanby et al., 2002, results and Supporting Information C for a direct comparison). When considering only the four flows tested in both the present study and by Teanby et al. (2002), the mean PI values (43.5 ± 19 and 30.8 ± 14 μT) have overlapping uncertainty bounds and are not statistically distinct from each other at the 95% confidence level ($p = 0.3232$).

The new MW-Perp data reported here also broadly replicate the lower estimates reported by Gratton et al. (2005). The Arai plots appear single sloped (see Figure 2 for Gratton et al., 2005, results and Supporting Information C for a direct comparison) and the mean PI values for the nine flows tested here and in Gratton et al. (2005) are 18.2 ± 10 and 18.6 ± 9.8 μT, respectively. These mean values are not statistically distinct at the 95% confidence level ($p = 0.9462$). This result supports Grappone et al.’s (2019) finding that the different generations of the MW systems give equivalent results.

The consistency of our newly obtained results with those from the previous studies enables us to conclude that we may reasonably combine our new Th-OT+ data with the Teanby et al. (2002) data and our new MW-Perp data with the Gratton et al. (2005) data. All the data are therefore combined to create one data set which forms the basis for discussion in the following section. The combined SOH1 data set, consisting of data from this study, Teanby et al. (2002), and Gratton et al. (2005), is summarized in Table 3.

### Table 3

<table>
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<tr>
<th>Flow</th>
<th>Th-OT+ (μT)</th>
<th>$N_{pass}/N_{tested}$</th>
<th>MW-OT+ (μT)</th>
<th>$N_{pass}/N_{tested}$</th>
<th>Th-Perp (μT)</th>
<th>$N_{pass}/N_{tested}$</th>
<th>MW-Perp (μT)</th>
<th>$N_{pass}/N_{tested}$</th>
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<td>2</td>
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<td>4/4</td>
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<td>6</td>
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<td>45.1 ± 2.8</td>
<td>2/2</td>
<td>36.5 ± 0.6</td>
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<td>21.3 ± 9.5</td>
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<td>18.5</td>
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<td>0/1</td>
<td>12.6</td>
<td></td>
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<tr>
<td>196</td>
<td>24.7 ± 1.3</td>
<td>2/2</td>
<td>15</td>
<td>1/1</td>
<td>18.7 ± 3.6</td>
<td>0/3</td>
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</tr>
<tr>
<td>206</td>
<td>40.1 ± 29</td>
<td>2/5</td>
<td>18.7</td>
<td>1/3</td>
<td>26.2 ± 1.5</td>
<td>0/3</td>
<td></td>
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<tr>
<td>220</td>
<td>46.4 ± 7.9</td>
<td>4/6</td>
<td>37.2 ± 6.1</td>
<td>2/3</td>
<td>33.8</td>
<td>1/1</td>
<td>26.0 ± 4.7</td>
<td>7/12</td>
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<tr>
<td>221</td>
<td>40.9 ± 2.1</td>
<td>3/3</td>
<td>36.7</td>
<td>1/4</td>
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<td>2/4</td>
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<td>4/5</td>
</tr>
<tr>
<td>222</td>
<td>39.8 ± 3.3</td>
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<td>33.7</td>
<td>1/2</td>
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<td>37.8 ± 1.6</td>
<td>3/3</td>
<td></td>
</tr>
<tr>
<td>237</td>
<td>25.5 ± 8.2</td>
<td>3/9</td>
<td>14.3 ± 8.5</td>
<td>4/5</td>
<td>10.4</td>
<td>1/2</td>
<td>19.1 ± 0.2</td>
<td>2/4</td>
</tr>
<tr>
<td>Mean</td>
<td>39.0 ± 12</td>
<td>18/22</td>
<td>29.5 ± 9.2</td>
<td>19/21</td>
<td>27.8 ± 8.1</td>
<td>11/18</td>
<td>24.8 ± 8.5</td>
<td>22/23</td>
</tr>
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</table>

Note. $N_{pass}$ is the combined number of specimens that passed the PI selection criteria. $N_{tested}$ is the combined number of specimens that were tested from a given flow with a given method. Empty cells indicate that no experiments were attempted because of a lack of material. For the mean row, $N_{pass}$ and $N_{tested}$ reference the number of flows.

5.2. Incorporation of Existing Data Sets

The new Th-OT+ results reported here are broadly consistent with their original counterparts reported by Teanby et al. (2002) and Laj et al. (2011). Both have the same two-slope (concave up) behavior (see Figure 2 for Teanby et al., 2002, results and Supporting Information C for a direct comparison). When considering only the four flows tested in both the present study and by Teanby et al. (2002), the mean PI values (43.5 ± 19 and 30.8 ± 14 μT) have overlapping uncertainty bounds and are not statistically distinct from each other at the 95% confidence level ($p = 0.3232$).

The new MW-Perp data reported here also broadly replicate the lower estimates reported by Gratton et al. (2005). The Arai plots appear single sloped (see Figure 2 for Gratton et al., 2005, results and Supporting Information C for a direct comparison) and the mean PI values for the nine flows tested here and in Gratton et al. (2005) are 18.2 ± 10 and 18.6 ± 9.8 μT, respectively. These mean values are not statistically distinct at the 95% confidence level ($p = 0.9462$). This result supports Grappone et al.’s (2019) finding that the different generations of the MW systems give equivalent results.

The consistency of our newly obtained results with those from the previous studies enables us to conclude that we may reasonably combine our new Th-OT+ data with the Teanby et al. (2002) data and our new MW-Perp data with the Gratton et al. (2005) data. All the data are therefore combined to create one data set which forms the basis for discussion in the following section. The combined SOH1 data set, consisting of data from this study, Teanby et al. (2002), and Gratton et al. (2005), is summarized in Table 3.

5.3. Analysis of Combined Data Set

Figures 3–5 display a series of one-to-one comparisons of the PI data produced by different methods at the flow level, utilizing data from the combined data set, which allows the influence of demagnetization technique and PI protocol to be scrutinized. From visual inspection, the data do not appear to be symmetrically
random about the 1:1 line nor cluster close to it. Thus, the PI estimate data pairs do not visually appear to be consistently normally distributed about the 1:1 line, which indicates that a two-sample t-test may be insufficient. We instead use the Wilcoxon signed rank test (see Biggin, 2010) to examine if the respective deviation of the data sets from the 1:1 line is significant at the 95% ($\alpha = 0.05$) confidence interval. The null hypothesis is that the data scatter about the 1:1 line is random.

In keeping with Biggin (2010), we first confirm that the Th-OT+ data are consistently higher than the MW-Perp data (Figure 3a). The Wilcoxon signed rank test gives $W = 1$, which corresponds to a $p$ value of 0.0008 for 15 data points, so we can reject the null hypothesis that the deviation from the 1:1 line and hence the data scatter is random.

Next, we test the hypothesis of Biggin (2010) that the primary cause for the Th-OT+ data being consistently higher than the MW-Perp data is due to the differing PI protocol (OT+ vs. Perp) and not demagnetization mechanism (MW vs. thermal). We do this by comparing the MW-OT+ and Th-Perp data (i.e., the inverse combination) to see if the OT+ protocol continues to yield systematically higher values than the Perp protocol. It can be seen in Figure 3b that in fact, MW-OT+ data are not consistently higher than Th-Perp data; the paired results are significantly closer to and fall on either side of the 1:1 line. The Wilcoxon signed rank test gives $W = 18$, which corresponds to a $p$ value of 0.33 for 10 data points, so we cannot reject the null hypothesis that the deviation from the 1:1 line is random. Having failed to support the simple hypothesis that the protocols are entirely responsible for the differences in PI results, we now examine demagnetization mechanisms and protocols in isolation to probe deeper into these specimens’ behavior.

If the cause of the discrepancy between the Th-OT+ and MW-Perp data was purely the demagnetization mechanism, then we would expect that estimates from MW-OT+ and MW-Perp would be similar and would cluster around the 1:1 line. Similarly, estimates from Th-OT+ and Th-Perp would also be similar, clustering around their 1:1 line. These cases are plotted in Figure 4. For the flows they have in common (Figure 4a), the mean PI estimate for the MW-OT+ data is $30 \pm 8.8 \mu T$ and $26.1 \pm 7.7 \mu T$ for the MW-Perp data. The Wilcoxon signed rank test gives $W = 23$, which corresponds to a $p$ value of 0.0065 for 18 data points, so we can reject the null hypothesis that the deviation from the 1:1 line is random. Next, we investigate the Th-OT+ and Th-Perp data (Figure 4b). For the flows they have in common, the mean PI estimate for the Th-OT+ data is $38.3 \pm 14 \mu T$ and $27.5 \pm 8.3 \mu T$ for the Th-Perp data. The Wilcoxon signed rank test for Th-OT+ versus Th-Perp gives $W = 3$, which gives a $p$ value of 0.05 for seven data points, therefore also rejecting the null hypothesis that the deviation from the 1:1 line is random. Therefore, changing the protocol to OT+ from Perp does indeed cause higher PIs to be measured, but this is not the entire explanation for the discrepancy illustrated in Figure 3a.

Figure 3. Testing Biggin’s (2010) hypothesis that only paleointensity protocol affects PI estimate. Flow-level paleointensity estimates are plotted against each other for completely distinct PI methods (no shared protocol or demagnetization mechanism). (a) Confirming that the Th-OT+ data are higher than the MW-Perp data. (b) Checking if MW-OT+ data are higher than Th-Perp. The mean PIs listed are for the flows the methods have in common. $N$ is the number of data points, and $W$ and $p$ are the statistics from the Wilcoxon signed rank test.
For the flows that have both Th-OT+ and MW-OT+ data (Figure 5a), we observe mean PI estimates of 40.7 ± 13 and 28.8 ± 10 μT, respectively. The Th-Perp and MW-Perp data give more similar estimates (Figure 5b). For the flows they have in common, the mean PI estimates are 28 ± 8.6 and 26.6 ± 5.9 μT, respectively. For Th-OT+ versus MW-OT+, the Wilcoxon signed rank test gives $W = 1$, which corresponds to a $p$ value of 0.0012 for 14 data points. We can therefore reject the null hypothesis that the deviation from the 1:1 line is random, which indicates that Th-OT+ data are higher than the MW-OT+ data. For the Th-Perp and MW-Perp data, the Wilcoxon signed rank test gives $W = 25$, which corresponds to a $p$ value of 0.24 for 11 data points. Thus, we cannot reject the null hypothesis that the Th-Perp and MW-Perp data’s deviation is random, which suggests that the Th-Perp data are not higher than the MW-Perp data. This result agrees with the $t$-test in section 5.1.

We therefore observe that neither the demagnetization mechanism nor the protocol used can, in isolation, fully explain the differences in PI estimates observed. We additionally find that the Th-OT+ method yields results that are consistently (and statistically) higher than the other three methods used.
6. Discussion

6.1. PI Methodology Differences

Double-heating Thellier protocols have long been known to have problems with multidomain components causing non-linear Arai plots (Levi, 1977). Hodgson et al. (2018) showed that this can apply to non-single domain oxygen-exsolved titanomagnetite grains as well, which are common in basaltic lavas such as the Hawaiian lavas of the SOH1 drill core. These non-single domain components can lead to concave up (two-slope) Arai plots. If the low blocking temperature (power integral) portion is used, the data give PI over-estimates, and conversely, underestimate are obtained if the high blocking temperature (power integral) portion is used (Levi, 1977; Smirnov et al., 2017; Thomas, 1993; Xu & Dunlop, 2004). Grappone et al. (2019) studied samples from the 1960 Kilauea lava flow, another Hawaiian lava flow. They showed that the high estimates often found using thermal double-treatment methods (of which Th-OT is one) (e.g., Yamamoto et al., 2003) are not replicated when using the MW demagnetization mechanism. Our data here confirm this conclusion, as the Th-OT method yields the highest estimates, but the MW-OT data align much more closely with the Perp data sets obtained using either demagnetization mechanism.

A recent study by Cromwell et al. (2018) (using the thermal-IZZI method and strict selection criteria) reports PI estimates from 22 surface lava flows across the island of Hawaii. They found estimates consistent with geomagnetic field models in the 270–10,000 years range. However, the study was unable to reproduce the high estimates found in Teanby et al. (2002) and Lav et al. (2011). Cai et al. (2017) additionally found systematically lower estimates than Lav et al. (2011) using subaerial glassy basaltic margins on older flows from the HSDP2 core, using the same techniques as Cromwell et al. (2018). Based on the data herein and the other studies, the thermal OT method appears to give higher PI estimates than other PI methods and is likely an overestimate of the true palaeointensity due to exaggerated multidomain-like effects and the lack (in these experiments) of any mechanism to detect these.

The original MW study for the 1960 Kilauea lava flow, by Hill and Shaw (2000), gave site-level estimates up to 20% lower than the expected value of 36.5 μT. In both the Hill and Shaw (2000) and the Gratton et al. (2005) studies, an older MW system was used with the Perp protocol (Kono & Ueno, 1977), which uses only a single treatment per step and no pTRM checks. Grappone et al. (2019) showed that changing the MW experimental protocol to a more common double-treatment technique including alteration (pTRM) checks (in their case the IZZI protocol; Yu et al., 2004) gave the correct answer of 36.5 μT for the 1960 Kilauea lava flow. Although the OT protocol’s additional treatments cause more thermochemical alteration than the Perp protocol, it is likely that the SOH1 samples behave in a similar way to the 1960 Kilauea flow and that the Perp data sets give low estimates due to undetected alteration.

The MW-Perp and the Th-Perp data sets are not statistically distinct, implying that it is not something inherent to the MW causing the differences between the original Teanby et al. (2002) and Gratton et al. (2005) data sets. It has been shown that for well-behaved (single-domain [SD]) grain-containing ceramics, there is a detectable difference in PI estimates due to differences in cooling rate (Poletti et al., 2013). Since the MW-Perp and the Th-Perp data sets are not statistically distinguishable, any cooling rate effect would be relatively minimal. Further, the MW-OT data have a faster cooling rate but give lower estimates than the Th-OT data, which is the opposite of the expected cooling rate effect (Poletti et al., 2013).

Additionally, the Perp protocol having less than half the number of treatments as in the OT protocol introduces another complication if these specimens undergo stress relaxation during the experiment. If even mild stress relaxation affects the specimens, the Perp data will appear too low because pTRMs would be gained more efficiently than NRM would be lost (Kosterov & Prevot, 1998).

That the MW-OT results are lower than their Th-OT counterparts suggests that something inherent to the MW demagnetization mechanism dampens the effects on the data from non-ideal vortex-state grains. If this finding was the result of the MW not causing the same magnitude of stress relaxation as in thermal experiments, the MW-Perp data would be expected to be even lower than the Th-Perp data (Kosterov & Prevot, 1998). This does appear to be the case here (Figure 5b), as the differences are not statistically significant.

Based on the above discussion, explanations therefore exist both for why the thermal OT experiments overestimate the true value (multidomain-like effects) and for why Perp experiments (both thermal and MW)
underestimate it (unrecognized alteration), which implies that the best estimate should fall between these two extremes.

The inherent uncertainty in the data and techniques used to obtain PI estimates can and should be expressed in the stated uncertainties associated with a flow-level mean (e.g., averaging the Th-OT+ and MW-Perp and providing the resulting large standard deviation). Until new, high-quality data exist for all the flows studied here from the SOH1 drill core, the authors propose that for each flow, the available flow-level Th-OT+ and MW-Perp data sets, which are the largest two, be averaged. Supporting Information D provides an example of this averaging for the flows containing at least two specimens in each Th-OT+ and MW-Perp data set (i.e., the flows studied in the meta-analysis described in Supporting Information A). This method gives a mean PI estimate of 29.2 ± 12 μT. This mean value is virtually identical to the mean MW-OT+ estimate of our newly collected data, 29.5 ± 9.2 μT, but higher than the equivalents measured by Gratton et al. (2005) and lower than those in Teanby et al. (2002). Flows characterized by data produced from only a single method (more than half; 97/181) across these studies (and the Laj et al., 2011, reanalysis) should not be considered accurate to within stated uncertainties.

6.2. Implications for Future Experiments

The Perp protocol has largely fallen out of use due to the lack of pTRM checks, but the OT protocol lives on as do the IZZI (Yu et al., 2004), Coe (ZI) (Coe, 1967), and Aitken (IZ) (Aitken et al., 1988) protocols. Modeling done in Biggin (2010) carries the implication that the OT protocol has the potential to exaggerate multidomain behavior compared with other Thellier-style double-treatment experiments, which is largely consistent with the study carried out here. We therefore suggest that any future experiments performed with the OT protocol (or indeed the Coe or Aitken protocols) should incorporate pTRM tail checks alongside pTRM checks such that multidomain-like effects may be detected. The IZZI protocol has the built-in advantage of allowing detection of non-ideal behavior via zig-zags in the Arai plot.

This study is consistent with previous studies (e.g., Grappone et al., 2019) that have shown that while different methods can give seemingly reliable data, non-ideal effects (multidomain behavior, stress relaxation, undetected alteration) can be a biasing influence. We therefore concur with the finding in Grappone et al. (2019) that at least pilot specimens should be run using at least two Thellier-style protocols (or the IZZI protocol) as a first order check for non-ideal PI behavior. Better still, different methods (e.g., thermal Thellier, MW Thellier, or multispecimen) should be employed to produce reliable multimethod PI estimates (de Groot et al., 2013), which, ideally, should be internally consistent. Biggin and Paterson (2014) provided a set of quantitative criteria for evaluating the reliability of PI estimates. Such PI estimates are required to satisfy the TECH QPI criterion of Biggin and Paterson (2014), which requires PI estimates to come from multiple techniques.

7. Conclusions

In this paper, we have sought to identify the cause for systematic discrepancies between previously published PI studies on the SOH1 drill core. New PI data confirm the systematic offset observed from previous studies when using the same methods, namely, Th-OT estimates were ~30% higher than MW-Perp estimates. For the first time, Th-Perp experiments and MW-OT experiments were undertaken on these rocks. Our results confirm that Th-OT data can be too high in the presence of magnetic carriers that do not behave as non-interacting SD grains. We further confirm that Perp data, which lack pTRM checks for alteration, can be too low due to undetected thermochemical alteration.

Until new measurements are made using reliable methods, results previously obtained from the SOH1 drill core using different methods should be combined at the flow level. The resulting enhanced standard deviation will accurately reflect the intrinsic uncertainty associated with the mean. The potential for biasing in those flows only represented by estimates produced by only one of the previously applied methods should be recognized.

Future studies undertaken using the thermal and/or MW demagnetization mechanisms should avoid any protocols which do not contain within them checks for both lab-induced alteration and non-ideal multidomain-like behavior. The IZZI protocol with pTRM checks satisfies both of these criteria.
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References


References From the Supporting Information


