A comparison of a quasi-perpendicular method of absolute palaeointensity determination with other thermal and microwave techniques

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Abstract

We describe, test, and explain the relative effectiveness of six thermal and microwave techniques of absolute palaeointensity determination applied to lava samples from Mount Etna (Sicily) that acquired their thermoremanence (TRM) in a known geomagnetic field. The application of a single alternating field demagnetisation treatment prior to each measurement in a standard Thellier experiment is demonstrated to be beneficial: increasing the quality of the results and making the analysis simpler. A new quasi-perpendicular (QP) method dramatically improves the linearity of Arai plots by reducing or altogether eliminating non-ideal effects due to multidomain (MD) grains. Two broad types of sample were encountered by this study and the QP method is the only one capable of producing accurate measurements from them both. Its ease of use, speed, and increased reliability should allow it to replace standard double heating methods in studies such as these where laboratory alteration can be detected by other means than pTRM checks. The results of experiments performed using a microwave palaeointensity system provide further proof that the microwave (de)magnetisation process is equivalent to conventional thermal excitation. They also demonstrate that microwave experiments are subject to the same risks as conventional experiments when performed on samples containing MD grains. The behaviour of the samples in both the thermal and microwave experiments is in good agreement with a recently proposed model of MD TRM which is based on first-order symmetry of the remagnetisation process.

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1. Introduction

Geomagnetic palaeointensity data are currently being produced at a high rate and being applied to diverse and important problems such as constraining geodynamo theory, dating young oceanic crust, and understanding core–mantle interaction (e.g. [1]). Simultaneously, many experimental and analytical methods which claim to improve the reliability or efficiency of the measurements are also been proposed (e.g. [2–8]). This innovation is fuelled by a combination of increased understanding of the potential problems facing palaeoin-
tensity determination together with a growing realisation that traditional methods may often produce untrustworthy results (e.g. [9–12]).

The basic Thellier and Thellier [13] technique remains the most popular method on account of the relative ease with which sample alteration (which can bias the determination) can be detected and avoided in its results. However, there have been a large number of modifications and additions suggested to the basic experimental protocol and these can have profound effects on the results, particularly when the samples contain multidomain (MD) grains [14].

The palaeointensity community has not yet reached a consensus on which precise palaeointensity techniques are optimal. The present study aims to address this to some degree by comparing results produced by them on neighbouring specimens from 13 lava flows (with very similar bulk compositions) which were emplaced by the 1950, 1979, and 1983 eruptions of Mount Etna on the island of Sicily.

The geological background and the rock magnetism of the samples studied here are described in depth by this study’s sister article ([15]; referred to as paper 1 from hereon). Furthermore, the thermal (non-micro-wave) palaeointensity measurements were analysed en masse without differentiating between the methods employed to obtain them. Whilst the choice of palaeointensity technique did exert some influence on the results as we will show, several conclusions could be made independently of this variable. The findings of that study which are pertinent to the present study are listed here.

1. Whilst most samples underwent alteration when heated above 350–450 °C, no alteration whatsoever was evident beneath these temperatures. Nevertheless, only 30% of the 88 measured specimens produced results that were both of reasonable quality and accurate to within 10% of the expected value.

2. Some rock samples which were mainly from close to the margins of the flows contained heavily oxyexsolved titanomagnetite close to pure magnetite. These samples (referred to as hard-type on account of them being resistant to thermal demagnetisation) tended to exhibit concave-up Arai plots typical of those produced by samples containing MD grains. Since the determinations were obtained from the low-temperature portion of this Arai plot, these samples frequently overestimated the actual palaeointensity by 30% or more (Fig. 1).

3. Rock samples (referred to as soft-type) from both the margins and interiors of the flows which were less heavily oxidised and therefore easier to demagnetise tended to underestimate the palaeointensity by an amount generally not exceeding 30% (Fig. 1). This biasing frequently occurred with no associated non-ideal behaviour visible on the Arai plots and was

Fig. 1. Comparison of accuracy of palaeointensity measurements (intensity error fraction – IEF – see text) with the fraction of NRM remaining after thermal demagnetisation to 350 °C (NRM$_{350}$/NRM$_0$). Soft-type samples tended to underestimate the palaeointensity while hard-type samples tended to overestimate it. The stippled area denotes IEF ≤ ±10% which is the only range of values for which both types of samples produced results which passed selection criteria (SELCRIT2 in paper 1).
explained through differences in the natural and laboratory cooling rates. A correction based on this hypothesis was demonstrated to be effective in making the mean determinations more accurate (Fig. 6, paper 1).

4. It was possible to reliably constrain the correct palaeointensity by taking the overlap in the ranges of good-quality measurements produced by soft-type and hard-type samples. This observation was used to produce a new reliability criterion for palaeointensity studies.

This study will breakdown the results used in paper 1 by the method employed and analyse and explain their differences. It will also present results performed on the same samples using a microwave palaeointensity system.

Section 2 of this article will describe the palaeointensity techniques in detail together with the statistical parameters and models of MD TRM that will be referred to later. Sections 3 and 4 will compare the experimental results with one another and with models of multi-domain (MD) thermoremanence (TRM). Section 5 will discuss the observations and their implications for future palaeointensity studies.

2. Palaeointensity techniques

2.1. Techniques used in the thermal experiments

Three different experimental approaches were used to produce the thermal palaeointensity determinations. The first was the Coe-modified Thellier [13,16] method incorporating pTRM checks and pTRM tail checks [17]. This experimental technique is currently that most used by palaeomagnetists. It, and the results it produced, will be referred to as CONV. It was applied to eighteen specimens and the temperature steps used increased progressively from 150 °C to 450 °C in steps of 50 °C.

The heating–cooling procedure at every temperature step was:

1. Heating to Tr and cooling to Tc (room temperature) in a magnetic vacuum (the ‘demagnetisation’ step)
2. Heating to Tp and cooling to Tr in a magnetic field of 50 μT applied along the z-axis of the specimens (the ‘remagnetisation’ step)
3. Heating to Tr and cooling to Tc once more in a magnetic vacuum (the ‘pTRM tail check’ step)
4. Heating to Tp−1 and cooling to Tc in an applied magnetic field of 50 μT along the z-axis of the specimens (the ‘pTRM check’ step)

Samples were positioned in the oven in such a way as to ensure the lab field made an angle greater than 45° (and generally greater than 90°) with the NRM to allow the pTRM tail checks to be used effectively [18].

We wished to investigate the effects of a small alternating field (AF) demagnetisation treatment applied prior to each measurement on the resulting quality of the palaeointensity determination. Consequently, the same Thellier–Coe experiment was undertaken on 18 sister-specimens from the same palaeomagnetic cores as those subject to the CONV experiment. In this case however, the specimens were subject to a pre-measurement demagnetisation (AF demagnetisation with peak field 5 mT) immediately before every measurement. This method and the results it produced will be referred to as AFD. A similar approach has been attempted previously [16,19,20] with mixed results.

Biggin [14] predicted that a Thellier-type protocol that involved only a single remagnetisation treatment at every temperature step would produce significantly improved results when applied to samples containing MD grains. One such method would be the perpendicular protocol [21] employed with no pTRM or pTRM tail checks. This method is impractical in many laboratories because of the difficulties associated with aligning the laboratory field exactly perpendicular to a specimen’s NRM. Therefore we introduce an alternative which we will refer to as the quasi-perpendicular (QP) single-heating approach.

The QP approach has two immediate requirements:

1. The characteristic remanent magnetisation (ChRM) of a sample is isolated by a pre-measurement demagnetisation – the AFD results indicated that 5 mT AF demagnetisation was generally sufficient to achieve this in the samples studied here.
2. The ChRM makes an angle of at least 45° with the lab field. This was ensured by picking those samples with low inclinations of magnetisation in lab coordinates. It is necessary to reduce noise whilst inferring the TRM component during the process that will be outlined below.

The experimental process is fast and simple. The initial measurement is made after the first AF demagnetisation treatment. This provides the vector (X0, Y0, Z0) of the ChRM; the relative size of its components will remain constant through the experiment.

The sample is then subjected to a thermal remagnetisation treatment to some peak temperature. During this treatment, a field is applied along the z-axis (the length of the cylindrical specimen) during both the heating and
cooling phases. After another 5 mT AF demagnetisation treatment, the remanence is measured again. This measurement will be a composite of the ChRM (reduced from its previous value) and the laboratory magnetisation. The latter of these should be entirely aligned with the z-axis so that the measurements will be of $X_{\text{nrm}}$, $Y_{\text{nrm}}$, and $Z_{\text{meas}} = Z_{\text{nrm}} + Z_{\text{lab}}$.

It is then possible to produce two estimates of the size of $Z_{\text{nrm}}$:

$$Z_{\text{nrm1}} = X_{\text{nrm}} \frac{Z_0}{X_0}$$  \hspace{1cm} (1)

$$Z_{\text{nrm2}} = X_{\text{nrm}} \frac{Z_0}{X_0}$$  \hspace{1cm} (2)

To avoid error amplification, in the case where either the $X$ or the $Y$ component constitute less than 15% of the full NRM vector, only the remaining component is used. Otherwise, the average of $Z_{\text{nrm1}}$ and $Z_{\text{nrm2}}$ may be used as $Z_{\text{nrm}}$, the value of the $Z$ component expected for the NRM.

The magnitude of the NRM vector can then be calculated:

$$\text{NRM} = \sqrt{X_{\text{nrm}}^2 + Y_{\text{nrm}}^2 + Z_{\text{nrm}}^2}$$  \hspace{1cm} (3)

with the magnitude of the laboratory TRM vector being given by the residual $Z$ component of the total measurement:

$$\text{TRM} = \sqrt{(Z_{\text{meas}} - Z_{\text{nrm}})^2}$$  \hspace{1cm} (4)

These points are plotted on the Arai plot and the experiment is continued with single remagnetisation treatments made to progressively higher temperatures and interspersed with the fixed AF treatment. Temperature steps were the same as for the Coe-modified Thellier experiment: 50 °C intervals from 100 °C to 450 °C continuing to 500 °C and 530 °C if required. No pTRM checks or tail checks were used in these experiments because the thermal demagnetisations that these require would negate the predicted benefits of the method when applied to MD samples [14].

Fifty-two specimens were treated using the QP approach. These specimens included at least one sister-specimen from those subject to the Coe-modified Thellier experiments (to make comparisons between the methods possible) and totalled specimens from 26 core samples. Of these 26 core samples, ten provided three specimens for the QP approach, six provided two specimens and a further ten provided one specimen only.

### 2.2. Techniques used in the microwave experiments

We undertook microwave palaeointensity experiments successfully on 18 specimens using the 14 GHz system at the University of Liverpool. This system incorporates a resonant microwave cavity and a cryogenic magnetometer, is fully automated, and allows the applied microwave power, magnetic field strength and direction to be defined by the operator. Once the sample is prepared and inserted into the system the orientation remains fixed and so any change in the direction of magnetisation is easily defined. For both systems the sample size is small being a 5-mm-diameter core typically 3 to 6 mm long. Precise orientation of such a small sample is difficult and for the experiments described here no attempt was made to determine absolute directions. A new 14 GHz system currently under development at Liverpool is designed to allow absolute orientation for future experiments.

For microwave demagnetisation the sample is placed in the centre of the resonant cavity. When microwave power is applied the microwave field couples with the magnetic system in the sample producing magnons (spin waves). As the power is increased the number of magnons increases, increasing the energy of the magnetic system and demagnetising the sample. This process can be compared with conventional thermal demagnetisation where heat energy produces lattice vibrations (phonons) in the bulk sample. In order to stay in equilibrium with the bulk sample magnons are generated in the magnetic system and the sample demagnetises. In theory the direct production of magnons by microwaves is identical to magnon production by the heat/phonon mechanism but without heating the bulk sample [8]. Experimental evidence supports this theory [22].

The microwave experiments were performed for two principal reasons. The first was to allow comparisons between the results of conventional and microwave experiments to be made. The second reason was to test three additional palaeointensity methods. The microwave system allowed six different methods to be tested in a relatively short space of time because the experiments are performed on individual specimens one at a time rather than in batches of specimens as in thermal experiments.

The microwave equivalents of the three techniques already described will be referred to as MW(CONV), MW(AFD), and MW(QP). In place of the 5 mT AF demagnetisation treatment used in the conventional AFD and QP experiments, we simply employed microwave demagnetisation treatments. The power of this treatment was adjusted to that necessary to isolate the
ChRM in the particular specimen being measured. As for the conventional experiments, this pre-measurement demagnetisation treatment was made prior to every measurement.

The other three methods were simple Thellier-type protocols with no pre-measurement demagnetisations. The first of these, MW(Aitken), was the technique of Aitken et al. [23] which is essentially a reversed version of Coe’s method [16] (CONV in this study). In Aitken experiments, the remagnetisation treatments are performed prior to the demagnetisation treatments at every temperature step and the pTRM tail check (step 3 in the CONV experiments), is omitted. The second method, MW(Thellier), was the original Thellier and Thellier [13] double-heating technique where two remagnetisation treatments (made using equal and opposite applied fields) are performed at each temperature step. Finally, we used the original perpendicular method as outlined by Kono and Ueno [21]. The MW(Perp) method differs from QP and MW(QP) on three counts. Firstly, it requires that the field be applied precisely perpendicular to the NRM. Secondly, although the ChRM is isolated by demagnetisation treatments at the beginning of the experiment, there are no subsequent pre-measurement demagnetisations. Thirdly, like every other microwave experiment except MW(QP), MW(Perp) includes pTRM check and pTRM tail check measurements.

2.3. Quality parameters

A number of parameters may be produced from the palaeointensity results of individual samples. The ones relevant to the present study are defined here.

The $\beta$ value is simply the standard error of the slope of the best fitting straight line on the Arai plot divided by the value of the slope itself. It is essentially a measure of the linearity of the selected portion on the Arai plot and, when multiplied by the applied field intensity, provides 68% uncertainty bounds for that sample’s palaeointensity determination.

The $q$ (quality) value was developed by Coe et al. [24] and was very commonly cited in palaeointensity studies. It is given by:

$$q = \frac{fg}{\beta}$$

where the $f$ value defines the fraction of the sample’s NRM which is used to produce the determination and the $g$ (gap) value is a measure of the regularity of the spacing of the points along the best-fit line [24].

Max $|\Delta$pTRM$_{\text{check}}|$ refers to the maximum absolute amount of discrepancy between a pTRM check and an original measurement of pTRM that falls within the selected temperature range of the experiment. It will generally be expressed as a percentage of the initial NRM measurement for that sample. When divided by the $j$ value it becomes the maximum DRAT parameter [25]. The cumulative DRAT (CDRAT) parameter is the sum of all DRATs over the range of temperatures used for the palaeointensity measurement. DRAT(Tail) is calculated in the same way as DRAT but using the maximum discrepancy measured by the tail checks [17].

We introduce a new parameter which we call the intercept deviation (ID) and which is only appropriate for use in experiments employing a pre-measurement demagnetisation which is sufficient to isolate the characteristic component of remanence (as it was in most of the AFD and QP experiments performed in this study).

$$\text{ID} = \frac{|\text{NRM}_{\text{int}} - \text{NRM}_0|}{\text{NRM}_0}$$

$\text{NRM}_{\text{int}}$ is the intercept of the best-fit straight line on the $y$-axis of the Arai plot, $\text{NRM}_0$ is the intensity of the sample at the beginning of the palaeointensity experiment (after the pre-measurement demagnetisation). The value of ID is particularly sensitive to progressive changes in the ratio of NRM lost to laboratory TRM gained (Arai plot curvature) which tend to shift the $y$-axis intercept of the best-fit straight line. It therefore has the potential to be a powerful indicator of alteration or MD effects.

When comparing palaeointensity determinations with the expected value from the IGRF (43.3 $\mu$T for the 1950 flows, 44.1 $\mu$T for the 1979 flows, and 44.2 $\mu$T for the 1983 flows), we used the intensity error fraction (IEF) which is simply the difference between the measured and expected values expressed as a percentage of the expected intensity.

In analysing the results of all methods, we did not consider the known field intensity when selecting which points on the Arai plot to use to produce the palaeointensity determination. Instead, we chose them objectively using criteria normal to ancient palaeointensity studies: a primary directional component coupled with the most linear portion on the Arai plot and the best pTRM check results.

It is normal practice in palaeointensity studies to filter the measurements through a set of selection criteria. The set we will use here is the same as SELCRIT2 in paper I. For those experiments in which pTRM checks and pTRM tail checks are employed, the criteria are: $\text{MAD} \leq 15^\circ$, $\alpha \leq 15^\circ$, $N \geq 4$, $f \geq 0.15$, $q \geq 1$, $\beta \leq 0.10$, $\text{DRAT} \leq 10\%$, $\text{DRAT(Tail)} \leq 10\%$ (where $\alpha$ is the angle between the origin-anchored and centre-of-mass-an-
chored vectors on the Zijderveld [26] plot, MAD is the maximum angular deviation, and \( N \) is the number of points used to produce the estimate). Where pTRM and pTRM tail checks were not made, these criteria are modified to \( \text{MAD} \leq 15^\circ, \alpha \leq 15^\circ, N \geq 4, f \geq 0.15, q \geq 1, \beta \leq 0.05, \text{ID} \leq 1\% \).

2.4. Models of multidomain thermoremanence

The primary model used in this study to make predictions of the behaviour of different types of sample when subject to different palaeointensity methods was the phenomenological model developed by Biggin [14]. This was chosen on account of its flexibility with respect to unblocking behaviour and its demonstrated ability to capture first order behaviour very well [14].

We input two different sets of parameters into the model to represent the two broad types of unblocking behaviour we encountered in our samples (see Section 1 and Fig. 1). The \( \lambda \) parameter dictates the size of the pTRM tails in any modelled sample and hence the degree to which its behaviour is ‘MD-like’. This parameter was carefully chosen so that the modelled pTRM tail checks were similar to those observed in the experiments and was identical for both soft-type and hard-type models. The other parameters and the modelled temperature steps were chosen so that the models exhibited unblocking temperature spectra typical of soft-type and hard-type specimens; they are given in Fig. 2. The curvature of the Arai plots produced by the models was not considered when deciding the values for these parameters.

The pre-measurement demagnetisation treatments that were used in the AFD and QP experiments were simulated by maintaining the magnetisation function \( M(T) \) at zero for both \( T < 0.17 \). The majority of thermal experiments that we performed used a laboratory field \( (H_{\text{lab}}) \) applied broadly

Fig. 2. Arai plot produced by two different versions of a phenomenological model of MD TRM [14] subject to three different types of experiment with \( H_{\text{lab}}/H_{\text{nrm}} = 1.5 \), and the two fields applied perpendicular to one another. The soft-type version has \( \alpha_1 = 20, \alpha_2 = 0.25, \alpha_3 = 0.25, \alpha_4 = 0, \lambda = 0.1 \); the hard-type version has \( \alpha_1 = 20, \alpha_2 = 0.8, \alpha_3 = 0.15, \alpha_4 = 0, \lambda = 0.1 \). Note that the virtual temperature steps (given in °C to match the experimental data), which were chosen to fit the straight line to, were selected to match those most commonly chosen in the corresponding experimental results. Open points at higher temperatures will generally have been affected by alteration in the actual experiments. The dashed line shows where the points would fall to give an accurate result. pTRM (pTRM tail) checks are shown as vertical (horizontal) lines.
perpendicular to the NRM so we applied $H_{\text{lab}}$ perpendicular to $H_{\text{nrm}}$ in our simulations of these. In simulating microwave experiments, we matched the angle between the fields to the individual sample.

In paper 1, it was concluded that the difference in natural and laboratory cooling rates biased the mean palaeointensity to low values in our samples. We took this into account in our simulations by using a ratio of $H_{\text{lab}}$ to $H_{\text{nrm}}$ of 1.5. For our experimental value of $H_{\text{lab}}=50 \, \mu T$, this implies $H_{\text{nrm}}=33 \, \mu T$ which, relative to the average expected palaeointensity of $44 \, \mu T$, is at least sufficient to account for these effects. In any case, our decision to include cooling rate effects has little effect on the behaviour of the simulations relative to one another and therefore has no consequence for the conclusions reached by this study.

The MW(Thellier) method entails a sample being subject to three different types of treatment (two anti-parallel remagnetisation and one demagnetisation treatment). This is beyond the simulation capabilities of the phenomenological model in its present form. However, a second, kinematic model was also developed and tested by Biggin [14]. The kinematic model does not feature the same flexibility as the phenomenological model with respect to sample unblocking behaviour but does allow an infinite number of types of treatment to be simulated. Furthermore, it has been shown to give qualitatively identical results to the phenomenological model in the vast majority of cases. It was therefore used in place of the phenomenological model to simulate the MW(Thellier) experiment.

3. Thermal palaeointensity determinations

Fig. 2 provides the Arai plots produced by the phenomenological model for both sample-types subjected to the CONV, AFD, and QP methods. The key points to note are:

1. The QP method is predicted to produce ideal SD-like behaviour.
2. For soft-type samples, the AFD method is predicted to produce a more linear Arai plot than the CONV method.
3. Hard-type samples are predicted to produce larger overestimates of the palaeointensity than soft-type samples.
4. For soft-type samples, the initial pTRM check measurement (for alteration) is less than the original pTRM and the measured pTRM tails (checks for MD behaviour) become smaller through the course of the experiment.
5. For hard-type samples, the measured pTRM tails become progressively larger through the course of the experiment.

The results of the actual palaeointensity experiments for the 88 samples measured are given in Table 1 in a format which allows methodological comparisons to be easily made. More detail about each measurement is provided in Table 2 of paper 1. Fig. 3 provides some example Arai plots (two soft-type and two hard-type) from which it is possible to see the similarities between predicted (Fig. 2) and actual results as well as evidence for all five of the predictions listed above.

Fig. 4 provides one-to-one comparisons between the actual palaeointensity results and various quality parameters for the three methods. The Wilcoxon signed rank test is the non-parametric equivalent of a paired $t$-test [27] used to detect systematic differences in the data produced by the three methods. The $P$ values produced by these tests (i.e. the probabilities of no systematic difference existing) are also given in Fig. 4.

The palaeointensity result produced by sample 311E (140 $\mu T$) is outside the range of linear dependence of TRM on the field and may be disregarded. This result aside, the AFD palaeointensity results are generally similar to or lower than the CONV results (Fig. 4a) although the differences were not statistically significant. However, with high confidence, the AFD Arai plots were more linear (lower $\beta$; Fig. 4b) and of higher overall quality (higher $q$; Fig. 4c). Furthermore, and with extremely high significance, the AFD results produced lower values of max $|\Delta pTRM_{\text{check}}|$ (Fig. 4d) indicating that the pTRM checks agreed far better with the original pTRM measurements than in the CONV experiments.

The QP results tend to be lower than both the AFD and CONV results (Fig. 4e and h). The linearity of their Arai plots was generally better (Fig. 4f and i) as was their overall quality (Fig. 4g and j). Finally, the amount of Arai plot curvature in the QP results as measured by the ID parameter tended to be less that in the AFD results (Fig. 4k).

Table 2 provides the mean values for each of the methods before and after the selection criteria are applied and with and without a cooling rate correction. As we would expect, a significantly larger fraction of the AFD and QP results pass the selection criteria than do the CONV results. However, the high technical quality of the individual measurements produced by the AFD and QP experiments that comprise their selected means does not ensure that their values are accurate (this surprising disassociation between technical quality and
Table 1
Summary of palaeointensity measurements made by method

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<th>Core</th>
<th>Year of flow emplacement</th>
<th>Distance to upper margin (cm)</th>
<th>Type</th>
<th>CONV PI (μT) IEF</th>
<th>IEF</th>
<th>q</th>
<th>AFD PI (μT) IEF</th>
<th>IEF</th>
<th>q</th>
<th>QP1 PI (μT) IEF</th>
<th>IEF</th>
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<td>32.4</td>
<td>-27%</td>
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<td>-29%</td>
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Bold measurements pass the selection criteria outlined in text. The distance to the upper margin is given because of its applicability to original cooling time.
Fig. 3. Examples of Arai and associated Zijderveld plots produced by the three thermal palaeointensity methods tested by this study. Two hard-type and two soft-type samples are included. Asterisks indicate determinations which did not pass the selection criteria described in the text (SELCRIT2 in paper 1). On the Zijderveld plots, filled (hollow) points represent the vertical (horizontal) component (the top of the diagrams is west and up).
accuracy is discussed in detail in paper 1). In fact, both the AFD and QP means require a cooling rate correction (+9% IEF to results from samples taken from more than 50 cm from the upper margin of their lava flow; see paper 1) in order to be made accurate. The CONV selected mean determination does not require this correction to be made accurate. However, given the large amount of scatter in this result (2 of the 3 measurements comprising it are more than 20% inaccurate but fortuitously cancel one another out), no significance can be attached to this observation.

4. Microwave palaeointensity determinations

Table 3 provides details about the microwave palaeointensity determinations and their associated parameters. These displayed similar variations to the results of the thermal experiments: soft-type samples tended to produce underestimates whilst hard-type samples tended to produce overestimates (with some exceptions: see below). The mean determinations before and after selection criteria were applied are reasonably accurate although this is a rather fortuitous result given the large scatter in the data. This higher degree of dispersion is to be expected given the range of protocols used and the fact that a larger proportion of hard-type samples (which tend to be more deviant) were measured than in the thermal experiments.

We will now consider the dispersion in more detail by focusing on the individual protocols and the results they produced. The MW(CONV) results were analogues of their conventional counterparts. The hard-type
sample produced a large (35%) overestimate (Fig. 5a) while the soft-type samples produced results between 0 and 25% below the correct value. The highest quality of these determinations, produced by sample 196A1, was an underestimate associated with a linear Arai plot and good pTRM checks over 88% of its unblocking temperature spectrum (Fig. 5b).

The MW(AFD) experiments were uniquely performed on hard-type samples and three of these gave predictably high results (e.g. Fig. 5c) while the fourth gave a noisy underestimate. The two MW(QP) experiments were performed on specimens from the same cores as two of the MW(AFD) specimens. These produced linear Arai plots (e.g. Fig. 5d) that gave lower palaeointensity determinations than the double-heating method. These microwave results were entirely in keeping with their conventional counterparts and the phenomenological model’s predictions (Section 3).

The phenomenological model predicts the shape of Arai curves produced by Aitken’s method to be extremely sensitive to the direction of the applied field relative to the sample’s NRM. The Arai plot is predicted to be almost perfectly linear when the field is applied parallel to the NRM (Fig. 6a) and to have extreme concave-up curvature when the field is applied anti-parallel or even sub-anti-parallel (Fig. 6c). This prediction was fully supported by the MW(Aitken) results: one sample was treated with the field parallel and produced a linear Arai plot (underestimate) over a significant portion of its unblocking temperature spectrum (Fig. 6b). Two other samples were treated with the field sub-anti-parallel and both produced strongly concave-up Arai plots which resulted in overestimates of the palaeointensity despite their soft-type nature (e.g. Fig. 6d).

The original perpendicular method incorporating pTRM and pTRM tail checks (MW(Perp)) is predicted to produce Arai plots with good linearity but which, unlike QP and MW(QP), slightly (5–10%) underestimate the palaeointensity before cooling rate effects are taken into consideration (Fig. 6e). The two samples measured using this method produce linear Arai plots (e.g. Fig. 6f) which also underestimate the palaeointensity by 20–25%.

The Arai plot predicted by the kinematic model for the MW(Thellier) method (Fig. 6g) is in excellent agreement with that produced experimentally by 196A3 (Fig. 6h). The other samples measured using this method also produced sinusoidal Arai plots in keeping with this prediction although these were not quite so dramatic.

5. Discussion

5.1. The benefits of using a pre-measurement demagnetisation

The AFD and QP methods (and their microwave equivalents) employed a single, consistent demagnetisation treatment prior to every measurement made. Indeed, this was the only difference between the AFD and CONV methods. As Fig. 4 and Table 1 show, this simple
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Mean (all) 46.5±8.7  6±20
CR corrected 49.6±8.3  13±19
Mean (Sel) 41.3±9.5  –6±21
CR Corrected 44.7±9.9  6±21

Bold results pass the same selection criteria as applied to the thermal results. $\theta$ is the angle between $H_{\text{lab}}$ and the ChRM. $\Delta$Power gives the range of applied powers (and the time of application in seconds – 10 s if not stated) used to produce the palaeointensity measurements. The other parameters are explained in the text.
Fig. 5. Examples of Arai and associated Zijderveld plots produced by microwave experiments employing equivalent protocols to those used in the thermal experiments. Where the time of application is not stated, it was 10 s. On the Zijderveld plots, filled (hollow) points represent the vertical (horizontal) component in laboratory coordinates (all samples were unoriented).
Fig. 6. Comparison of modeled and experimental Arai plots for the microwave experiments performed using protocols not employed in the thermal experiments. Note that the differences in the x-axis scales are caused by the laboratory field being varied in the experiments (see Table 3) but remaining fixed (so that $H_{lab}/H_{nrm} = 1.5$) in the simulations.
action was responsible for dramatic improvements in the quality of the measurements leading to a much higher fraction of results passing selection criteria (Table 2).

The AFD and QP results produced by soft-type samples frequently required correcting for a bias imparted by differences in the natural and laboratory cooling rates in order to be made accurate (as did several of the high-quality microwave determinations – e.g. Fig. 5b). Many of the CONV results were accurate before this correction but we point out that this was fortuitous. Of the nine (50%) CONV estimates that were accurate to within 10% of the expected intensity, only one of these was of sufficient quality to pass the set of selection criteria used in this study (Table 1) or any of the three sets of selection criteria used in paper 1. These particular results are most likely simultaneously biased by the effects of both MD grains and cooling rate differences so that they approximately cancel one another out (paper 1). This fortuitous process cannot be relied upon to occur in general. Therefore, for the purposes of future studies, the AFD and QP methods may be considered more trustworthy than the CONV method because they reduce the effects of one source of inaccuracy (MD grains).

In the majority of cases, the pre-measurement demagnetisations were sufficient to isolate the characteristic remanent component (ChRM) of the samples. Any experiments in which this is done can be expected to benefit from the following:

1. Decreased non-linearity due to MD grains providing that points are well spaced on the Arai plot (as for soft-type samples; Figs. 2 and 3). AF demagnetisation in particular has been shown to make samples behave more SD-like with respect to their pTRM behaviour [28].
2. Decreased noise from weak viscous magnetisations acquired in the lab (AF demagnetisation in particular not only removes VRM and soft pTRM but also places the MD grains in a more stable state; e.g. [29]).
3. Decreased noise following a thermal remagnetisation treatment because the softest pTRM is always erased. This makes the precise temperature and time at which the samples are removed from the oven, and the amount of time between this and the measurement, less important. This and point 2 make Arai plots more linear and pTRM checks more reliable.
4. Being able to decompose each measurement into NRM and TRM components which allows the QP method or at least for pTRM tails to be detected without recourse to dedicated pTRM tail check treatments (this second process is fully described and tested in Biggin and Perrin [30]).
5. A best-fit straight line on the Arai plot that is better constrained because the initial point is now usable.
6. The measurement of an ‘ID parameter’ which is very sensitive to any form of Arai plot curvature and which may be used to further evaluate the reliability of the determination.
7. Reduced subjectivity associated with the choice of where, on the Arai plot, to begin the line of best fit. The benefit of always using the initial point will be to make the interpretation of results, from older rocks in particular, far easier and more reliable.
8. Being able to combine the results from multiple specimens on the same Arai plot for analytical (e.g. [7]) or purely visualisation purposes.

5.2. The quasi-perpendicular method

As with all of the palaeointensity techniques employed in this study, the QP method has the full support of Néel’s theory of thermoremanence [31]. However, it differs from the others in that it is also predicted to cause samples containing multidomain (sensu lato) grains to behave in a palaeointensity experiment as if they uniquely contain SD grains (Fig. 2) and therefore to prevent them producing concave-up Arai plots. All of the samples used in this study contain significant quantities of MD grains (paper 1) and, when measured using the QP method, they produce results which are consistently lower in value and higher in quality than when they are measured using the CONV and AFD methods (Fig. 4 e–k). This strongly suggests that MD effects are being minimised in the QP experiments.

Sixteen specimens that were identified as being less than 50 cm from the upper margin of a lava flow were measured with the QP method. The results of these should be largely free of biases due to both cooling rate differences (paper 1) and MD grains. As expected, almost two-thirds of these produced results within 10% of the expected palaeointensity (43.3 to 44.2 μT) and the average of all of these (40.5 ±3.0 μT; 95% confidence limits for the mean) is also within this margin of error.

The hard-type samples universally overestimate the palaeointensity when studied using the CONV and AFD methods because of exaggerated MD effects (Figs. 2 and 3). More than half of the hard-type samples produced accurate determinations when studied using the QP method suggesting that these effects were largely eliminated in these experiments. Fig. 3 shows two examples of hard-type samples which behave much better in the QP than in the CONV and AFD experiments. Nonetheless, four of the hard-type samples measured using the QP method still produced overestimates of the
palaeointensity which suggests that MD effects were still present to some degree. The effectiveness of the QP method in eliminating MD behaviour rests in the symmetry constraints defined by Biggin and Poidras [32] which were clearly not obeyed by these four samples. The most likely explanation for this is that the MD grains in these particular samples formed under stress during their rapid initial cooling and developed more effective wall-pinning sites as a result. This is likely to have caused their domain walls to be pinned during their primary cooling in such a way that their acquired TRM was larger than the equilibrium TRM (for that applied field) at ambient temperatures. As a consequence, they were more susceptible to partial demagnetisation upon subsequent heating than samples containing more equilibrated low-stress grains, and this led them to produce concave-up Arai plots even in QP experiments. This preferential demagnetisation of high-stress grains is most likely exaggerated by experiments that produce closely spaced Arai plots because a substantial amount of thermal energy is supplied to allow the demagnetisation over a relatively small portion of the blocking temperature spectrum. Consequently, we believe that this source of inaccuracy could be reduced in future studies by careful design of the experiments so that points appear well spaced on the Arai plots.

In paper 1, a new robust reliability criterion was developed by stating that, given two distinct types of recorder (e.g. the soft- and hard-type samples in this study), which both produce some good quality palaeointensity measurements for the same time period, the range in which these results overlap will likely include the true palaeointensity. If this were a study of ancient rocks, this criterion could have been applied to the QP data and used to reliably constrain the palaeointensity to within 10% either side of the correct value (Fig. 1). This would not have been possible using the AFD or the CONV data because no reliable results were obtained from hard-type samples and consequently, no range of overlap would have been available to constrain the palaeointensity.

The QP method produces linear Arai plots from MD grains because the magnetic field which is applied during the thermal treatments remains constant (in both intensity and direction) through the entire experiment so that conflicting high-temperature tails are not imparted [14]. Unfortunately, this means that pTRM check measurements for alteration should not be made in the experiments: these would necessitate thermal treatments in a different field and would therefore introduce the same non-linearity into the Arai plots as is found in double heating experiments. Fortunately, while such checks are not only within the palaeointensity community, they are the only reliable guard against obtaining results which are biased by laboratory alteration. Careful rock magnetic measurements can also be used for this purpose (e.g. [33–35]). Indeed, in this study, cyclic $\chi(T)$ measurements were very clear in constraining alteration to 400 °C and above (paper 1) which was entirely consistent with the pTRM check measurements where they were made (Fig. 3).

The only other published palaeointensity technique which is capable of reducing MD effects to the same degree as the QP method is the “multispecimen parallel differential pTRM technique” outlined by Dekkers and Böhlke [6]. However, while this method relies entirely on separate rock magnetic analyses to demonstrate that alteration is not affecting the experimental results, the QP method has an extra safeguard. Like conventional double-heating Thellier-type protocols, the QP method measures the palaeointensity multiple times per specimen, over different portions of the (un)blocking temperature spectrum. In general, most forms of alteration do not occur at the same rate at all temperatures; either they are progressive and get worse at higher temperatures or they occur abruptly over a narrow temperature range. The $\beta$ and ID parameters are sensitive indicators of both systematic curvature and distinct offsets on the Arai plot which these alteration processes would produce. Other selection criteria than those based on pTRM checks can therefore be used to reject data which could be affected by laboratory alteration.

5.3. The equivalence of microwave and thermal (de)magnetisation

Overall, the microwave results are highly consistent with the theoretical predictions for MD grains (Figs. 5 and 6) which are themselves highly consistent with experimental results produced using conventional heating techniques. This strongly supports the notion that the microwave (de)magnetisation process is equivalent to the conventional thermal excitation process. It also shows that microwave experiments are subject to the same dangers and limitations resulting from MD grains and that the same precautions can and must be taken (see paper 1 and [14] for details). The correlation between the phenomenological models’ predictions and the experimental results provide further support for the models’ accuracy and that of the underlying theory of first-order symmetry [32].

We appeal to cooling rate differences between nature and the laboratory to explain the high incidence of
underestimation of the palaeointensity by the soft-samples in both the thermal and microwave experiments. The cooling rate in the microwave experiments is 1–2 orders of magnitude higher than that in the thermal experiments so, using the same approach as paper 1, we would expect that their results underestimated the palaeointensity by a further 4% (for a total of 13% underestimation) [15, 36]. Unfortunately, we are not able to test this prediction for three reasons. Firstly, there are an insufficient number of microwave results produced by soft-type specimens from the same cores as those used in the thermal experiments to allow any meaningful direct comparisons to be made. Secondly, the wide variety of experimental protocols used makes any comparisons of the mean values difficult. Thirdly, the intensive nature of the microwave experiments (producing closely spaced Arai plots even for soft-type samples) might be expected to exaggerate the effects of the MD grains on the results in the same manner as it did for hard-type specimens in the thermal experiments. This would tend to increase the palaeointensity determinations and therefore counteract the effects of the cooling rate differences.

6. Conclusions

1. The adoption of a (ideally AF) pre-measurement demagnetisation treatment in any Thellier-type palaeointensity experiment, especially when it is sufficient to isolate the characteristic component of remanence, increases the quality of the results significantly.

2. The QP method is much faster to use than Thellier and Thellier’s double-heating technique [13] and its derivatives but retains their within-sample consistency check. It is also extremely effective at reducing Arai plot curvature due to MD grains: apparently eliminating it altogether in more than 90% of the samples measured in this study. If this had been a study of ancient rocks then, together with the criterion developed in paper 1, the QP results (and no other) could have been used to produce a robust and accurate palaeointensity determination.

3. We have provided further proof that microwave excitation is precisely equivalent to conventional thermal (de)magnetisation by showing that palaeointensity experiments performed using both methods suffer from the same non-ideal effects due to MD grains. This implies that all of the conclusions of this study and of paper 1 are equally relevant to both thermal and microwave studies. The microwave method does retain the advantages of minimising alteration to the samples and of being fast and flexible.

4. The agreement between the simulated and experimental results observed in this study further demonstrates the usefulness of the models developed by Biggin [14] in informing palaeointensity studies.

Acknowledgements

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