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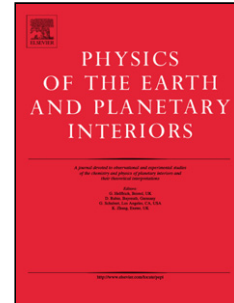
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An assessment of the reliability of palaeointensity results obtained from
the Cretaceous aged Suhongtu Section, Inner Mongolia, China

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Abstract

Here we present microwave palaeointensity results from 89 sister samples from the study of Zhu et al. (Phys. Earth Planet. Int. this issue) who carried out Thellier palaeointensity analysis as part of their integrated palaeomagnetic and $^{40}\text{Ar}/^{39}\text{Ar}$ dating study of Cretaceous lava from the Suhongtu section, Inner Mongolia, China. Additionally, a comprehensive rock magnetic investigation has been carried out in order to determine the mineralogy and hence the validity of assuming that the remanence is a thermal remanent magnetisation (TRM). The microwave results are of apparent high quality and give flow mean palaeointensity estimates ranging from 13 to 49 μT corresponding to virtual dipole moment (VDM) estimates ranging from 2.5 to $8.9 \times 10^{22} \text{ Am}^2$, and an overall mean VDM of $5.5 \pm 1.9 \times 10^{22} \text{ Am}^2$ for the 24 flows (aged $110.6 \pm 0.1 \text{ Ma}$). When the microwave results (using the perpendicular applied field method with partial microwave thermal remanence ($p\text{T}_{\text{M}}\text{RM}$) and $p\text{T}_{\text{M}}\text{RM}$ tail checks) are compared to those obtained with the Thellier method (Coe version with $p\text{TRM}$ but not tail checks, and heating in argon atmosphere) differences are seen at the sample, flow and palaeomagnetic unit level however the overall means and spread in palaeointensity estimates are consistent. Some discrepancy is due to the differing sized sample sets and sample inhomogeneity but discrepancy is also interpreted to be due to the differing protocols, methodology, plus the subjectivity in interpretation. Considering only those results that are consistent to within 20% the spread in palaeointensity estimates remains.

There is substantial rock magnetic evidence from progressive heating in air and argon experiments (both showing irreversible thermomagnetic behaviour) as well as looking at samples under the scanning electron microscope to suggest that maghaemite is present (albeit to varying degrees) in many of the samples. Alteration therefore occurred in nature and it is likely that the remanence will have been affected to differing degrees potentially causing underestimates in palaeointensity. Maghaemite is interpreted to be a remanence carrier where a component of remanence remains after heating to 580°C and loss of pTRM acquisition capacity is found on heating. No correlation was found between the estimated palaeointensity (microwave or Thellier datasets) and amount of high temperature remanence or any rock magnetic parameter. This could suggest that the palaeointensity estimates are reliable or as seems likely more than one factor (such as methodology, protocol, interpretation, CRM contamination and the geomagnetic field) are influencing the palaeointensity. In an attempt to remove the influence of palaeointensity protocol and methodology the palaeointensity datasets were reduced to consist only of those results mutually consistent to 20% (N=22). No obvious correlation was again seen between the inferred maghaemite contribution and palaeointensity. There is therefore no clear evidence for the biasing of results due to CRM contamination (assuming a simple relationship between biasing and maghaemite contribution) and it can thus be inferred that the geomagnetic field was a major influencing factor. Further studies are however needed to fully elucidate the extent and result of each possible influencing factor and ultimately the reliability of palaeointensity estimates from rocks of this type.

Keywords: Palaeointensity, microwave, CRM, Cretaceous

1. Introduction

In order to obtain an absolute palaeointensity estimate, a laboratory induced thermal remanent magnetisation (TRM) acquired in a magnetic field of known strength is compared to the natural remanent magnetisation (NRM). Palaeointensity methodology is advancing and checks are routinely in place to check the so-called Thellier laws of reciprocity, independence and additivity (Thellier & Thellier 1959). A key problem with absolute palaeointensity determinations has always been thermochemical alteration of samples during the laboratory experiments. Some

palaeointensity methods, such as the microwave method, aim to minimise the problem of alteration (Hill et al. 2002; Walton 1993) whilst other methods rely on alteration detection and discarding affected samples (see for example Valet 2003 for discussion of palaeointensity methods). Our understanding of how non-ideal recorders such as grains larger than the ideal single domain grain size behave is advancing (e.g. Xu & Dunlop 2004; Biggin 2006) and there is healthy debate over which methodological protocol and material is the most reliable (e.g. Yu & Tauxe 2004; Tarduno et al. 2006).

The premise behind absolute palaeointensity determination is that the NRM is a TRM acquired on cooling. If however the NRM is not a pure TRM, then the palaeointensity result may not be reliable. This assumption needs to be examined carefully in order to obtain reliable estimates of past geomagnetic field strength. Rock magnetic experiments are usually routinely carried out to determine grain size and mineralogy. Whilst it is sometimes very easy to identify weathering products such as titanomaghaemite which will contain a chemical remanence (CRM) formed by low temperature oxidation (e.g. Dunlop & Ozdemir 1997) it is often not straightforward to be certain that a remanence is, or is not, a TRM. New magnetite grains can be precipitated as a secondary process, for example by the action of hydrothermal fluids (e.g. Dunlop & Ozdemir 1997). Once the precipitated grains grow large enough they are able to acquire a CRM. Most experimental and theoretical evidence suggests that grain growth CRM is weaker than TRM (see Draeger et al. 2006 and references therein). This means that if CRM is not recognised, errors are introduced and the palaeointensity results are likely to be under estimates. By careful heating and cooling regimes Draeger et al. (2006) produced basalt samples containing CRM formed in a known laboratory field. The ratio of CRM/TRM was found to be always less than one but increased as the temperature of the acquisition of CRM increased, in qualitative agreement with theoretical considerations for non interacting single domain grains. Tarduno & Smirnov (2004) believe that the large majority of palaeointensity data, especially from older times, is likely to have been affected by some form of CRM contamination resulting in a low field bias in the palaeointensity database.

Contamination of the original remanence with CRM is not the only process of concern. Deuteric oxidation can occur at temperatures below the Curie temperature and hence a thermal chemical remanence (TCRM) can be formed (Dunlop & Ozdemir 1997; Grommé et al. 1969). Draeger et al. (2006) also produced basalt samples with

TCRM and found that the ratio of TCRM/TRM was slightly greater than one, but when cooling rate was considered suggest that TCRM approximates TRM. Yamamoto (2006) however suggests that TCRM may account for over estimations found in Thellier experiments from historical lava flows.

Zhu et al. (this issue) present an integrated palaeomagnetic and $^{40}\text{Ar}/^{39}\text{Ar}$ dating study of a Cretaceous andesite-basaltic lava sequence, the Suhongtu section, from Inner Mongolia, China. The lava flows were extruded in two episodes at 114.1 ± 0.3 Ma and 110.6 ± 0.1 Ma. The palaeomagnetic study of 51 lava flows demonstrates that as expected for lava extruded during the Cretaceous Normal Superchron (CNS) all flows were normally magnetised. Using an F test the flows were grouped in to 31 separate units to give a mean direction that is consistent with the Eurasia pole derived from the apparent polar wander path (APWP) for the early Cretaceous. The accepted palaeointensity results, obtained using the Thellier method (Thellier & Thellier 1959; Coe 1967), from 15 palaeomagnetic units (comprising 136 samples from 20 flows) indicate a fluctuating field with a virtual dipole moment (VDM) ranging from 2.53×10^{22} Am² to 9.92×10^{22} Am². Eighty-nine samples from 27 flows (all with prefix WK) were provided for a comparative microwave palaeointensity study. Twenty six flows were from the younger 110.6 ± 0.1 Ma episode and one flow from the 114 ± 0.3 Ma episode. By utilising different palaeointensity methods and / or experimental protocols each with their own advantages and disadvantages, consistency and hence reliability can be investigated (e.g. Thomas et al., 2004; Pan et al., 2004; Biggin et al. 2007). In order to try to fully understand the mineralogy of the WK samples and hence the validity in assuming that the remanence is a TRM detailed rock and mineralogical experiments have also been carried out.

2. Rock Magnetic Investigation

Samples range in colour from grey to reddish brown. Sixty-four out of the 89 samples were studied using the Liverpool Variable Field Translation Balance (MMVFTB). Isothermal remanent magnetisation (IRM) acquisition and backfield, hysteresis loops and high field thermomagnetic (magnetisation M_s with temperature T) curves were measured. Samples exhibit a range of behaviour with Figure 1a giving two examples. Whilst the magnetisation of some samples clearly saturates by 300 mT, for more than half of the samples the hysteresis loops are not fully closed nor the IRM

acquisition saturated by 300 mT as shown for sample WK80-8 in Figure 1a (and consistent with the findings of Zhu et al. though more prevalent in this sample set). This is evidence for a component being present that has a higher coercivity than magnetite. Hysteresis parameters for all samples are given on a Day et al. (1977) plot (Figure 1b). The results plot near to the Dunlop (2002) mixing curves for mixtures of MD and SD magnetite grains however these samples probably have a more complicated mineralogy than pure magnetite so the relevance of this is unclear.

Curie temperatures calculated from the $M_s(T)$ curves (using the second derivative method (Tauxe 1998) with the rockmag analyser program (Leonhardt 2006)) range from 531 to 634°C. Curie temperatures greater than 580°C suggest something other than magnetite as the mineral responsible. A reduction in magnetisation on heating is seen for all samples with the cooling curve lying below the heating curve (Figure 1a). The difference in magnetisation between the heating and cooling curves at 100°C ranges from 14 % (sample WK75-1) to 87 % (WK70-3) (average of 43%). To a greater or lesser degree there is an apparent second Curie point around 400°C (see Figure 1a and Figure 2). This was also seen in the temperature dependence of susceptibility $\chi(T)$ curves measured in argon atmosphere by Zhu et al. Progressive heating experiments (Figure 2) of both $M_s(T)$ in air (Liverpool VFTB) and $\chi(T)$ in argon (Beijing Kappa bridge KLY-3 with CS-2 attachment) demonstrate that this drop in magnetisation at around 400°C is irreversible and is not a Curie point but due to magnetomineralogical alteration. As the same behaviour is seen in both argon and air atmosphere this strongly suggests that it is inversion probably of maghaemite to haematite. Hysteresis measurements were also made as part of the VFTB progressive heating experiments (Figure 3) and the Henry et al. (2005) difference of hysteresis loops method employed. For samples WK70-3 and WK76-5 heating between 150 and 250°C creates a new component where after the signal is dominated by the loss of a strongly magnetic component (250-700°C). The biggest loss is found looking at the difference after heating to 400°C and 350°C as expected from the thermomagnetic curves. The low temperature component corresponds to a small increase in M_s and would also no doubt correspond to the increase in susceptibility seen on the $\chi(T)$ plots for similar samples. The higher temperature component corresponds to a loss in M_s (Figure 3). After heating to 600°C and higher, an increase in coercivity and coercivity of remanence is seen. This

behaviour is similar to that seen by Henry et al. (2005) for dyke samples which they interpreted as the result of temperature induced inversion of low titanium titanomagnetite / maghaemite. The creation of a low temperature phase is attributed to stress release and the loss of a strongly magnetic component to the inversion. Whilst the creation of a high coercivity phase is not seen in the difference plots it can be inferred from the hysteresis parameters with temperature plots (Figure 3).

The WK samples show variable stability to heating with the alteration interpreted as being maghaemite inversion. For samples which have the most reversible $M_s(T)$ curves such as sample WK75-1 (Figure 2a) there is no obvious evidence for the inversion of maghaemite in the progressive heating experiments.

The thermomagnetic curves found in this study are remarkably similar to those seen in dykes by Krasa & Herrero-Bervera (2005) which were interpreted as showing variable amounts of alteration and the presence of maghaemite. They found that the degree of alteration was variable within a dyke consistent with the variability seen within a flow in this study. The WK samples that are unstable during heating are also similar to the hydrothermally altered basalt samples studied by Ade-Hall et al. (1971). The one difference being that after heating to low temperature a drop in coercivity is not seen nor is the Ade-Hall et al. 'kink' readily visible on the $M_s(T)$ curves.

Three samples from flows that were part of the microwave study (samples WK86-6, WK89-4 and WK93-8) were analysed in a Camscan X500 crystal probe Scanning Electron Microscope (SEM) which also incorporates an Energy Dispersion Spectrum (EDX) analyser at the Department of Earth & Ocean Sciences, University of Liverpool. The samples were initially hand polished before being mechanically polished for 5 hours using a suspension of 0.05 μm colloidal silicon (SYTON™). The samples were finally given a very thin carbon coat in order to minimise charging effects (Prior et al. 1996). In the SEM the polished sample surface was at 20° to the incident beam. An accelerating voltage of 20 kV and a beam current of 40 nA was used. The step size ranged from 0.1 – 1 μm . Electron Backscatter Diffraction, EBSD data have been processed using the Tango function within Channel 5 (HKL Technologies Ltd., Denmark). Although the resolution of the EDX function is ~ 5 μm , a smaller step size effectively creates an image comprised of a running average for each point.

Examples from the SEM analysis are shown in Figure 4. Sample WK86-6 contains a small volume of visible iron oxide crystals, predominantly rich in titanium

and 4 - 10 μm in size. Cracks indicative of maghaematisation are seen in some grains suggesting varying degrees of low temperature oxidation. Figure 4a taken from sample WK89-4 is a secondary electron image, SEI indicating the proportion and size of titanomagnetite crystals with respect to the groundmass of the sample. The crystals are skeletal and not well evolved, suggesting rapid cooling. Shrinkage cracks were not as apparent as in sample WK86-6. Backscattered Electron Imaging, BEI combined with EDX and EBSD analyses (Figure 4b and c) suggests significant oxidation within sample WK93-8. The crystal shown in Figure 4b has a speckled appearance and is very similar to that shown in Haggerty (1991 page 166) identified as titanomaghaemite (lighter sections) replacing titanomagnetite (darker sections) along fractures within the crystal at low temperatures. EDX analyses indicates a significant proportion of titanium (Ti) within the crystal. Figure 4c is a combined EBSD and EDX map indicating the presence of iron (Fe), superimposed on a Band Contrast, BC map where the brighter the red, the higher the Fe content. The image shows varying amounts of Fe within the iron-oxide crystals and also on the left hand side of the image Fe can be seen along a crack cutting through the iron-oxide and plagioclase crystals. Occurring at temperatures within the brittle failure regime, this cracking highlights the presence of a later, second period of oxidation. Most probably originating from the iron-oxide crystals, EDX imaging suggests that Fe has migrated out of the crystals and collected along these cracks. Hydrothermal activity during successive lava flow emplacement is a possible cause of this.

The samples contain a higher content of Ti than is suggested by the thermomagnetic behaviour. Krasa and Herrero-Bervera (2005) in their Hawaiian dykes similarly found a discrepancy between the Curie temperature and evaluated Ti content and suggest that the magnetic signature is being dominated by a grain size fraction that has different composition and is too small to analyse with the SEM or electron microprobe. Late stage interstitial crystallisation could account for this.

3. Microwave Palaeointensity Study

All samples supplied, irrespective of their rock magnetic behaviour, were investigated. Microwave palaeointensity experiments were carried out at the Geomagnetism Laboratory, University of Liverpool. Two microwave systems both operating at 14 GHz were used. The majority of experiments (63 out of 89 samples)

were carried out using the older system that uses a FIT liquid nitrogen cooled, high temperature SQUID magnetometer (see Böhnelt et al. 2003 for description). The newest system is similar to the older one however it incorporates a more sensitive 3-axis liquid helium cooled SQUID magnetometer made by Tristan Technologies (Shaw & Share 2007). The perpendicular applied field palaeointensity protocol with partial microwave thermal remanence (pT_{MRM}) and pT_{MRM} tail checks (Hill & Shaw 2007) was used as the samples showed good directional stability to thermal and microwave demagnetisation. All results can be found in the supplementary data and examples of individual results are shown in Figure 5. High powers and / or microwave exposure times were needed to de(re)magnetise the samples. This is consistent with the samples having high Curie temperatures. Alteration during the experiment is evident for some samples (indicated by failure of checks and break in slope on the NRM T_{MRM} plot) indicating that samples were experiencing some heating though this will be less than the bulk sample experiences when heated in an oven (e.g. Hill & Shaw 2000).

Using acceptance criteria similar to those described in Hill et al. (2005) results were accepted from 70 samples (19 samples were deemed unacceptable). This equates to a 79% success rate at the sample level. Importantly pT_{MRM} tail checks were analysed in several ways; the scalar difference in intensity (accepted if $\leq 10\%$ normalised to the NRM), the angle between the demagnetisation tail check measurement and the NRM direction (accepted if $\leq 15^\circ$) and, the pT_{MRM} tail in the direction of the applied laboratory field (accepted if $\leq 10\%$ when normalised to the total T_{MRM}).

Reasons for sample failure were non-linearity of the NRM T_{MRM} plot, failure of checks and in three cases the direction wasn't stable so that the perpendicular applied field method failed. Accepted palaeointensity estimates range from 13 to 57 μT . The mean NRM fraction f of the accepted results is 0.5 and the mean Coe et al. (1978) quality parameter q is 12. In order to limit possible biases in palaeointensity estimates (in particular multi domain behaviour) it has been suggested that as large a fraction of NRM as possible should be used with $f \geq 0.5$ a suggested criteria (eg see Zhu et al. this issue and refs. therein). It can be seen in Figure 6a that for these results there is no dependency on the palaeointensity estimate and NRM fraction used. If acceptance criteria of $f \geq 0.5$ were imposed the number of accepted results would decrease from 70 to 51 (57% success rate).

The weighted mean palaeointensity has been calculated for each flow (weighted using the quality factor q as in Coe et al. 1978) and are given in Table 1 and plotted in Figure 6b. No results were obtained from flows WK70, WK96 and WK97, which all exhibited curved NRM T_M RM plots and / or failure of tail checks. WK70 was the only flow from the 114 Ma episode so all accepted results are from the 110 Ma episode. Flow means range from 13 to 49 μ T with the overall mean for the 24 flows being $31 \pm 10 \mu$ T. Twelve flows have only one or two estimates. The within flow consistency for the 12 flows with more than 2 samples per flow is between 3 and 17%. Using the inclination data from Zhu et al. (this issue) the corresponding VDMs range from 2.5 to $8.9 \times 10^{22} \text{ Am}^2$ with a mean VDM of $5.5 \pm 1.9 \times 10^{22} \text{ Am}^2$ ($N=24$). Also plotted in Figure 6b are the weighted flow mean palaeointensities for the more restrictive $f \geq 0.5$ dataset. It can be seen that it makes minimal difference to the flow means (as expected when using a weighted mean as the lower quality data has less influence) however only 4 flows have more than 2 estimates per flow and there is no data for a further 3 flows (WK72, WK73 and WK95).

4. Comparison to Thellier data

Once all data had been analysed, all palaeointensity results obtained from WK samples of the Suhongtu section were compared. All microwave experiments were carried out and the data analysed by Hill independently, and separately, from the Zhu et al. Thellier study. Eighty-nine samples were investigated in the microwave study compared to 220 in the more extensive Thellier study. Five samples in the microwave study were not part of the Thellier study (flow WK86 and sample WK83-8) but all others were. The top and bottom flows in the WK section (WK69 and 99) were not included in the microwave study nor was flow WK87. None of these 3 flows yielded any acceptable results with the Thellier method.

Zhu et al. used the Coe version of the Thellier method (Coe 1967; Thellier & Thellier 1959) with pTRM (but not pTRM tail) checks and heated in an argon atmosphere (see Zhu et al. this issue for further details). Thus the two studies used different experimental protocols (perpendicular applied field method with tail checks as opposed to Coe version of the double heating Thellier method without tail checks) as well as different de(re)magnetising regimes (high frequency microwaves as opposed to heating in argon atmosphere). Both studies only accepted data with good

directional stability of the NRM during the experiment, the ratio of the standard error of the best fit line to the slope of the same line $\beta \leq 0.1$, and pTRM ($PT_{M}RM$) checks that are $\leq 10\%$ when normalised to the total TRM ($T_{M}RM$). In order to limit possible biases of multi domain grains the Thellier study used strict criteria accepting only those results with $f \geq 0.5$ (pTRM tail checks were not carried out). As shown in Figure 6 restricting the microwave dataset to only those results with $f \geq 0.5$ serves mainly to reduce the dataset and does not make any significant changes to the flow means. Independence to f was also discussed by Thomas et al. (2004) in their investigation utilising the microwave perpendicular applied field method with Tertiary intrusives. Both microwave datasets will however be compared (at the sample level) to the Thellier dataset to enable as direct a comparison as possible.

Palaeointensity estimates should not be biased by the quality parameters used however there does appear to be a relationship between the Thellier palaeointensity (using both Zhu et al's total WK dataset and the smaller microwave sister sample set) and the quality factor q . As can be seen in Figure 7a the highest palaeointensity estimates are only found for the lower q values. The majority of the data has q less than 15 however the higher the q , the lower the highest palaeointensity found. No such relationship is seen with the microwave results (Figure 7b) or for any other quality parameter (e.g. f , g , β etc).

Of the 84 sister samples investigated 42 samples gave acceptable results using both methods (using the full microwave data set). Seven samples yielded unacceptable results with both methods, 7 samples that failed the microwave experiments gave acceptable estimates with the Thellier method and 28 samples that failed the Thellier study provided acceptable estimates with the microwave method. The samples that failed with both methods showed non linear NRM TRM ($T_{M}RM$) behaviour and / or failed tail checks. Much of the failed Thellier study data had a tendency for curved NRM TRM plots. In two cases the estimated palaeointensity is roughly consistent with the accepted microwave estimate. Where the microwave yielded unacceptable results due to directional instability it is expected that if the experiment were repeated using a different methodology it may be possible to obtain an acceptable estimate. The reason that some samples were acceptable with one method and not with the other will no doubt in part be due to sample inhomogeneity.

There were cases where sister samples investigated with the Thellier method yielded one acceptable and one unacceptable result.

When the $f \geq 0.5$ microwave dataset is compared to the Thellier study the number of samples that both give acceptable results reduces to 28 and the number of samples that both give unacceptable results increases to 24. Samples that failed the microwave experiments but gave acceptable estimates with the Thellier method increases to 20 and samples that failed in the Thellier study but gave acceptable estimates with the microwave method reduces to 12.

Before comparing the duplicate microwave and Thellier datasets it is instructive to see the consistency between sister samples which underwent the same treatment. In the microwave study only one estimate per sample was obtained however, duplicate sister samples were occasionally used in the Thellier study and the palaeointensity results from these are plotted against each other in Figure 8a. It can be seen that whilst there is some variability results are generally consistent to within 10%. Plotted in Figure 8b are two Thellier microwave comparisons; one using the complete microwave dataset and the other the more restrictive $f \geq 0.5$ microwave dataset. Consistency does improve when the $f \geq 0.5$ microwave dataset is used indicating that the highest quality microwave data are more consistent with the Thellier data however this is a smaller dataset and equal numbers (7) of consistent to within 20% and inconsistent data points are removed. There are still outliers that show greater than 20% discrepancy. Sample inhomogeneity could explain some of the inconsistent results however, as shown in Figure 8a sister samples investigated with the Thellier method are more consistent than the duplicate Thellier and microwave results. Looking at only those results that are consistent to within 20% of each other the spread in palaeointensity estimates stays similar therefore the range in palaeointensity estimates does not appear to be biased to the particular methodology / protocol.

Figure 9 shows NRM T_M RM and NRM TRM plots for 3 samples marked in Figure 8b (WK85-5, WK81-8 and WK80b-2) that gave the most inconsistent results. The microwave results all exhibit linear NRM T_M RM plots however the Thellier NRM TRM plots are not as linear with an initial steeper slope below 350°C. Note also that after the last temperature step, 610°C, there is still NRM remaining (15%, 16% and 33%). The first pTRM check shows an increase in TRM acquisition capacity on heating for samples WK85-5 and WK80b-2 whilst the checks for all samples at higher

temperatures tends to show a decrease in acquisition capacity. The pT_{MRM} checks are generally better however there are not as many. In fact the microwave result for WK85-5 which has only 4 data points is an example of the lowest quality accepted microwave result. There is however a second microwave result from flow WK85 (of higher quality) which is consistent with WK85-5. Similarly, there are other Thellier results from flow WK85 and they are all consistent with each other and all lower than the microwave results. Hence the reason for the discrepancy does not appear to be due to the lower quality microwave result but due to some other difference between the two studies. For sample WK81-8 the microwave result is this time lower than the Thellier result. The microwave result from WK80b-2 is higher than the Thellier result however when looking at the variation seen within the flow, both methods give a range of values though the highest Thellier result 50 μT is still less than 57 μT found in two samples in the microwave study. This flow exhibits the most variable behaviour for both studies.

A greater number of samples per flow were studied with the Thellier method. In order to have comparable numbers of palaeointensity estimates per flow as well as the fact that the microwave study flow means do not differ significantly with the larger dataset will be used for the following flow mean and palaeomagnetic unit comparison. Flow mean results (weighted by quality factor q) are compared in Figure 8c and plotted in stratigraphic order in Figure 10a. In the Thellier study the duplicate sister samples used (where present) were treated as separate estimates of the field when calculating the flow mean. As the within flow dispersion is low this will not affect the flow mean palaeointensity significantly. For flows with more than 2 estimates the error about the mean ranges from 2 to 13% for the Thellier data compared to 3 to 17% for the microwave. The 3 flows (WK70, WK96 WK97) which did not give any acceptable results in the microwave study also gave unacceptable results in the Thellier study. All accepted palaeointensities are from the 110 Ma episode.

Zhu et al. used an F test to combine flows with similar vector results. Results averaged in this way are listed in Table 2 and shown in Figures 8d and 10b. The microwave within palaeomagnetic unit consistency is generally worse than that obtained within a flow (and worse than the Thellier within unit consistency). This is especially so for the unit comprising flows WK91 to 94. Whilst the microwave results

for flows WK91, 92 and 93 have similar palaeointensities, the palaeointensity for flow WK94 is less than half the mean of the other flows (no acceptable results were obtained for flow WK94 in the Thellier study). The overall mean for the 17 units from the microwave study is $29 \pm 9 \mu\text{T}$ with a corresponding overall mean VDM of $5.1 \pm 1.6 \times 10^{22} \text{ Am}^2$.

It is clear that the data from the microwave and Thellier studies differ on all levels however the overall mean and standard deviation of the palaeointensity at the sample, flow and palaeomagnetic unit level are all remarkably similar. It should be remembered though that differing numbers of samples were used in the two studies.

5. Discussion

The rock magnetic and SEM investigation indicates that the WK samples have undergone varying amounts of alteration with clear evidence for the presence of maghaemite in many samples. The consequence for palaeointensity interpretation and the differences seen between the Thellier and microwave studies are discussed before discussing the evidence and implications for the presence of maghaemite remanence carriers and the reliability of the palaeointensity estimates.

Whilst both the microwave and Thellier generated datasets can be classed as 'high quality' the discrepancies seen between them are likely to be due to the different protocols used (perpendicular with tail check as opposed to Coe version Thellier without tail checks), the different de(re)magnetising regimes (microwaves as opposed to heating in argon atmosphere) and the different acceptance criteria. The comparison thus highlights the difficulty in determining unbiased results and the inherent subjective nature of palaeointensity interpretation. This has also been found in many other comparative studies. For example differences at the sample level were found for some samples in the study of Thomas et al. (2004) where the perpendicular applied field microwave method and the Thellier Coe method were used on high titanium titanomagnetite samples of Tertiary age. The palaeomagnetic unit means derived for each method were however indistinguishable.

Whilst in the Thellier study the criteria of accepting only those data with $f \geq 0.5$ has been used to limit the possible biases of multi domain behaviour, pTRM tail checks also check for alteration that creates higher blocking temperature components. This is of importance for the WK samples as the inversion of maghaemite to

haematite occurs at a lower temperature than the blocking temperature of haematite. Heating of the bulk sample is expected to be less in the microwave method than the Thellier therefore any heating induced alteration / inversion should be less significant in the microwave results. In the sample comparison (Figure 9) it can be seen that whilst the individual Thellier pTRM checks pass, they mainly indicate a loss of pTRM acquisition capacity. This can also be seen in some cases in the microwave study though not until the later stages of the experiment, for example the final unaccepted data point for sample WK80b-2 in Figure 9. It is however also possible that the more prevalent alteration during the Thellier experiment acts as an automatic rejection of samples that are inherently unsuitable. For example palaeointensity results were accepted from the microwave study for flow WK94 (all Thellier results rejected) and these are significantly lower than those from the other flows in its defined palaeomagnetic unit. Another explanation here however is that flow WK94 should not be included in that palaeomagnetic unit but without palaeointensity results it was not possible for Zhu et al. to ascertain this.

When different acceptance criteria are used (such as $f \geq 0.5$) the consistency found at the sample level between the two studies changes. If only those data that are consistent to within 20% are considered then the palaeointensity estimates range from 17 – 49 μT . This variation is greater than the maximum variation seen between duplicate samples suggesting that something other than the different palaeointensity methods / protocols is influencing the results.

The failure of samples in both studies is predominantly due to non linear NRM TRM ($T_{\text{M}}\text{RM}$) plots and in the case of the microwave study the failure of tail checks. This could be due to multi domain behaviour or alteration such as the inversion of maghaemite. Samples from flow WK70 which exhibited the biggest difference between heating and cooling $M_s(T)$ curves (Figure 2) all failed. Zhu et al. (this issue) noted that during thermal demagnetisation the magnetisation dropped significantly between 300-450°C for samples from flows WK70, WK71, WK86-88, WK95 and WK96 (similar to that seen in the $M_s(T)$ curves) which they interpreted as maghaemite inversion. All Thellier experiments on samples from these flows failed. However, acceptable results were obtained for all samples investigated from flows WK71 (1 sample), WK86 (4 samples) and WK95 (1 sample) in the microwave study and the palaeointensity results are in no way anomalous (Table 1). Acceptable

Thellier palaeointensity estimates were obtained from sample WK93-8 which showed considerable alteration in the SEM investigation (Figure 4b and c) and both Thellier and microwave studies yielded acceptable results from sample WK76-5 which exhibited inversion of maghaemite in the progressive heating VFTB experiments. It is recognised however that the bulk rock characteristics may not be representative of the remanence carriers. In order to test this Curie temperature has been compared to the highest temperature needed to remove all remanence during thermal demagnetisation experiments (carried out by Zhu et al.) and correlation is indeed found.

The presence of maghaemite in many samples suggests it likely that the remanence will have been affected, and the NRM contaminated to some extent by CRM thus potentially causing underestimates in palaeointensity. Maghaemite is interpreted to be a remanence carrier where a component of remanence remains after heating to 580°C and loss of pTRM acquisition capacity is found on heating. To investigate the influence of the inferred maghaemite component on palaeointensity Zhu et al. (this issue) compared the mean palaeomagnetic unit palaeointensity to the percentage of TRM gained in different temperature intervals. They did not find any correlation. Here we compare the microwave and Thellier palaeointensity data sets to the amount of remanence left after heating to 600°C (from both the thermal demagnetisation experiments carried out in air and the Thellier experiments carried out in argon atmosphere by Zhu et al.) and again no correlation is found (Figure 11). Comparison to rock magnetic properties such as hysteresis parameters, Curie temperature, difference in heating and cooling $M_s(T)$ curves at 100°C also fails to show any correlation.

There are a number of possible reasons why there is not a detectable correlation between the inferred maghaemite contribution and palaeointensity. Firstly, the palaeointensity results may be unaffected by the maghaemite formed due to alteration and are in fact reliable estimates of the field. Zhu et al. cite their excellent within flow consistency as strong defence against possible CRM contamination however the microwave results show similar consistency at the flow (but not the palaeomagnetic unit) level and they are not always consistent with the Thellier results. An alternate explanation (and we believe probably more credible) is that there are a number of factors influencing the palaeointensity results such as experimental protocol and methodology, as well as CRM contamination and fluctuations in the strength of the geomagnetic field. By restricting the datasets and only comparing

those palaeointensity results that are mutually consistent to 20% (N=22) the influence of methodology / protocol can be potentially removed. No obvious correlation is again seen between the inferred maghaemite contribution and palaeointensity (Figure 11). Evaluating the influence of all possible influencing factors and elucidating geomagnetic field behaviour is not straightforward. For example if CRM were gained at different temperatures then this could influence the results by different amounts (Draeger et al 2006). However, assuming a simple relationship between dominance of maghaemite and palaeointensity bias this study lends support to the conclusion of Zhu et al. (this issue) that the palaeointensity results are reflecting real fluctuations in the geomagnetic field. Further studies are however needed to fully understand the influence of biasing factors and ultimately the reliability of palaeointensity estimates from rocks of this type.

6. Conclusions

From the rock and mineralogical study it can be concluded that maghaemite is present (albeit to varying degrees) in many of the samples. Alteration therefore occurred in nature and it is likely that the remanence will have been affected in some way. All samples (irrespective of rock magnetic behaviour) underwent microwave palaeointensity experiments using the perpendicular applied field protocol with pT_MRM and pT_MRM tail checks. Accepted flow mean palaeointensity estimates range from 13 to 49 μ T corresponding to VDM estimates ranging from 2.5 to 8.9 $\times 10^{22}$ Am². The overall mean VDM from 24 flows (⁴⁰Ar/³⁹Ar age 110.6 \pm 0.1 Ma) is 5.5 \pm 1.9 $\times 10^{22}$ Am². Comparison of these results to the more extensive Thellier study (Coe version with pTRM checks but not tail checks and heating in argon atmosphere) by Zhu et al. (this issue) shows differences at the sample, flow and palaeomagnetic unit level however, the overall mean results (at all levels) are remarkably similar. Additionally, those results from the two studies consistent to within 20% of each other cover the range in palaeointensity estimates. By considering the different de(re)magnetising regimes and the different protocols explanations for the differences have been attempted. In order to further investigate this however, additional experiments are required to limit the variables (e.g. microwave experiments using the Coe protocol and without tail checks and Thellier experiments using the perpendicular applied field method with tail checks).

Taking the inferred maghaemite contribution as an indicator for the likely contamination of the NRM by CRM, correlation to the palaeointensity results has been attempted. No correlation was found when using complete datasets (Thellier or microwave) or only investigating those results that are consistent between methods. There is therefore no clear evidence for the biasing of results due to CRM contamination (assuming a simple relationship between biasing and maghaemite contribution). It can thus be inferred that the geomagnetic field was fluctuating during the Cretaceous Normal Superchron at 110 Ma with a mean VDM approximately 70% of the present day field (as concluded by Zhu et al. this issue). Further studies are however needed to fully elucidate the extent and result of each possible influencing factor and ultimately the reliability of palaeointensity estimates from rocks of this type.

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Tables

Table 1. Flow mean microwave palaeointensity results.

Flow	n/N	F μ T	σ	VDM $\times 10^{22}$ Am ²
98	1/1	32.66		5.67
97	0/2			
96	0/3			
95	1/1	13.13		2.50
94	3/3	13.60	2.36	2.35
93	2/3	35.81		6.19
92	5/6	41.60	2.03	7.17
91	3/4	32.07	2.66	5.56
90	1/3	22.05		3.88
89	7/7	22.27	0.79	4.04
86	4/4	32.09	0.91	5.33
85	2/2	40.45		7.48
84	4/5	21.93	0.56	4.06
83	3/3	24.79	2.88	4.66
82	5/7	23.44	3.20	4.12
81	4/4	18.45	2.36	3.41
80b	5/7	49.01	4.08	9.23
80	2/2	51.26		9.39
79	2/2	47.32		8.27
78	2/2	31.40		5.58
77				
76	2/2	30.89		5.92
75	5/6	33.43	3.15	5.84
74	3/3	29.52	1.77	5.22
73	2/2	25.77		4.17
72	1/2	35.90		6.30
71	1/1	40.66		7.15
70	0/2			

Flow number is prefixed by WK. n is the number of acceptable determinations and N the total number of samples available per flow. F is the weighted flow mean and σ the associated standard deviation. VDM is the virtual dipole moment.

Table 2. Palaeomagnetic unit microwave palaeointensity results

Unit	Flows	n/N	F μ T	σ	VDM $\times 10^{22}$ Am ²
2	WK97-98	1/3	32.66		5.76
3	WK96	0/3			
4	WK95	1/1	13.13		2.50
5	WK91-92-93-94	13/16	20.02	4.94	3.46
6	WK89-90	8/10	22.25	4.29	3.98
7	WK86-87-88	4/4	32.09	0.91	5.63
8	WK83-84-85	9/10	23.14	3.02	4.31
9	WK82	5/7	23.44	3.20	4.12
10	WK81	4/4	18.45	2.36	3.41
11	WK79-80-80b	9/11	49.17	2.30	8.94
12	WK77-78	2/2	31.40		5.48
13	WK76	2/2	30.89		5.92
14	WK74-75	8/9	30.74	1.71	5.40
15	WK73	2/2	25.77		4.17
16	WK72	1/2	35.90		6.30
17	WK71	1/1	40.66		7.15
18	WK70	0/2			

Giving unit number and the flows that comprise the unit. n is the number of acceptable determinations and N the total number of samples available per flow. F is the unit mean and σ the associated standard deviation. VDM is the virtual dipole moment.

Figure Captions

Figure 1. Rock magnetic results. a) From left to right hysteresis loop, IRM acquisition and $M_s(T)$ curve. Magnetisation in arbitrary units (AU). The solid (dashed) line on the $M_s(T)$ plot is the heating (cooling) curve. b) Hysteresis properties plotted on a Day et al plot (Day et al 1977) along with the Dunlop mixing curves for magnetite (Dunlop 2002)

Figure 2. Progressive heating thermomagnetic curves. a) $M_s(T)$ and b) $\chi(T)$ with magnetisation and susceptibility in arbitrary units (AU). Sister sample plots for single heating up to 700°C are also shown. The percentage difference between the heating and cooling curves at 100°C are also plotted with in a) the result for a sample (WK76-5) showing mid range alteration also given.

Figure 3. Coercivity results from progressive heating experiment for sample WK70-3. Remanence and induced difference curves for the temperature intervals 150-250°C and 250-700°C and coercivity with temperature plots.

Figure 4. Examples from the SEM analysis a) SEI image of sample WK89-4 groundmass with arrows pointing to the iron oxide grains b) Backscattered Electron Image, BEI for a speckled grain from sample WK93-8 c) Combined EBSD and EDX map indicating the presence of Fe, superimposed on a Band Contrast, BC map from sample WK93-8. The brighter the red, the higher the Fe content. Note Fe filled crack.

Figure 5. Examples of microwave palaeointensity results, a) and b) are successful results whereas the result from c) was rejected. On the NRM T_M RM plots solid (open) diamonds represent accepted (rejected) data points, narrow lines with arrows are pT_M RM checks, and dashed lines with open squares at the end represent the pT_M RM tail checks. The applied power and time is shown beside selected data points. The angle between the tail check measurement and the NRM direction, and the percentage of pT_M RM tail in the direction of the applied field is given near the tail check. The applied laboratory field F_{lab} and the palaeointensity estimate F are indicated. a) and b)

are results from the new Tristan microwave system and c) from the older FIT microwave system.

Figure 6. The influence of the fraction of NRM f on the microwave palaeointensity results. a) Palaeointensity versus f b) Flow mean results plotted in stratigraphic order for the complete dataset and the dataset consisting only of those data with $f \geq 0.5$. Open symbols indicate results obtained from only 1 or 2 samples.

Figure 7. Correlation of quality parameter q with palaeointensity (PI) from a) Zhu et al's Thellier dataset and b) the microwave study.

Figure 8. Comparison of palaeointensity (PI) results where the long dashed line is the 1-1 correspondence line and the short dashed lines are the lines that deviate by 10 and 20% as labelled. a) Comparing results from sister samples in the Zhu et al Thellier study. b) c) and d) Comparison of microwave and Thellier results at b) the sample, c) the flow and d) the palaeomagnetic unit level. In b) the two plots are using firstly the full microwave dataset and secondly the more restrictive dataset with $f \geq 0.5$. The full dataset has been used in c) and d). The mean palaeointensities (PI) are given. Results for samples WK85-5, 81-8 and 80b-2 are indicated on b). Samples with zero PI are those that failed to give an acceptable result. Open symbols on c) and d) indicate those results where one of the means is from only 1 or 2 samples.

Figure 9. Comparison of microwave and Thellier results for the 3 samples highlighted in Figure 3b that gave inconsistent palaeointensity estimates. Microwave NRM $T_{M}RM$ plots are to the left and Thellier NRM TRM plots to the right. Symbols as in Figure 5. All microwave measurements were made with the old FIT microwave system. Samples WK81-8 and WK85-5 were measured when the maximum power available was 40W.

Figure 10. Microwave and Thellier palaeointensity results at the flow and palaeomagnetic unit level plotted in stratigraphic order. Open symbols indicate results obtained from only 2 or 1 samples.

Figure 11. Comparison of Thellier and microwave palaeointensity (PI) to the percentage of NRM left after heating to 600 °C during the Thellier experiment. Open (closed) symbols are the complete (consistent between studies to 20%) datasets.

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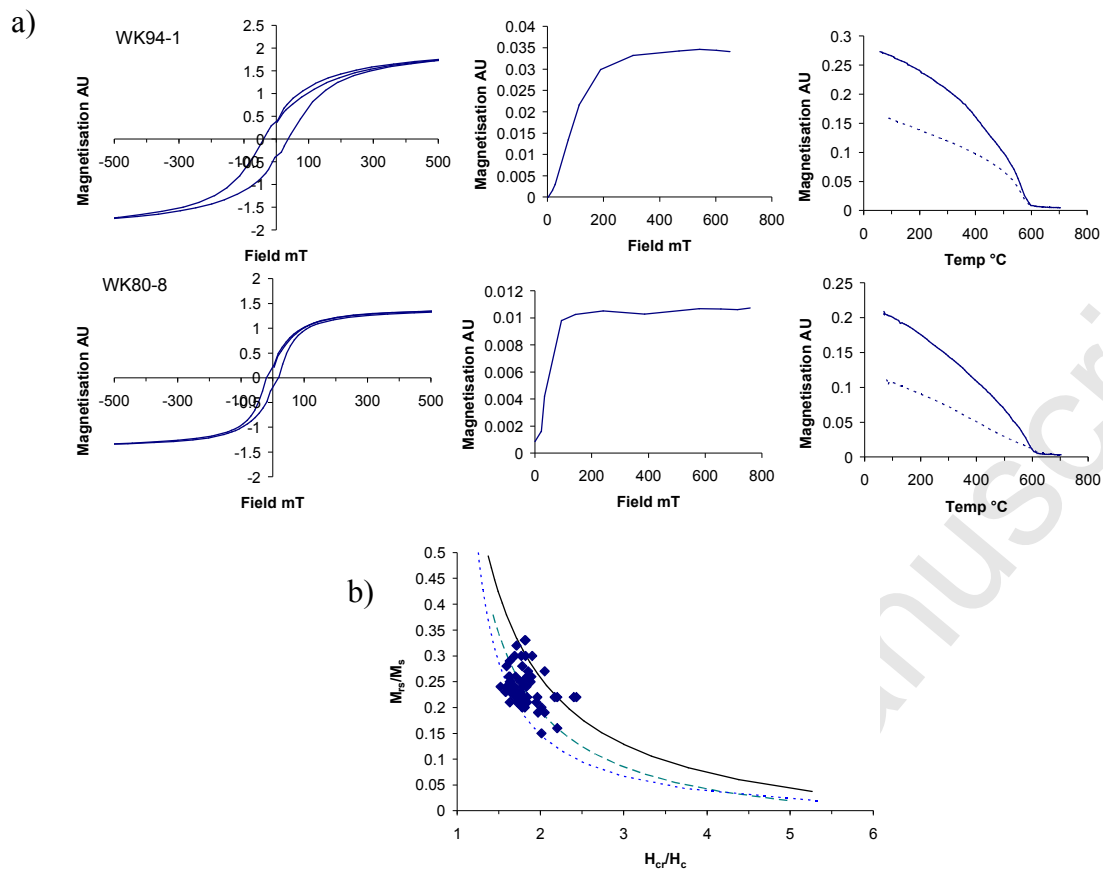


Figure 1.

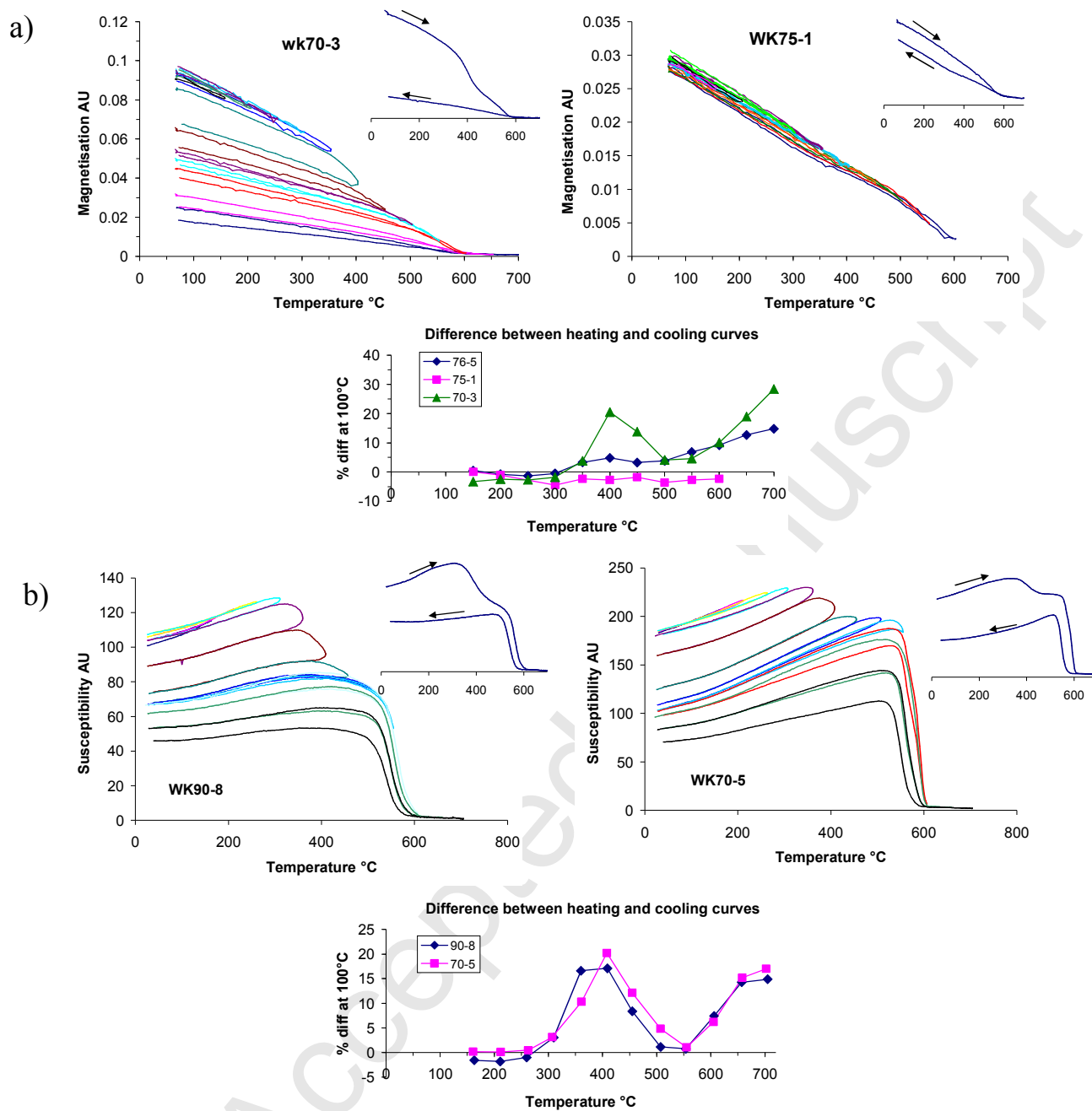


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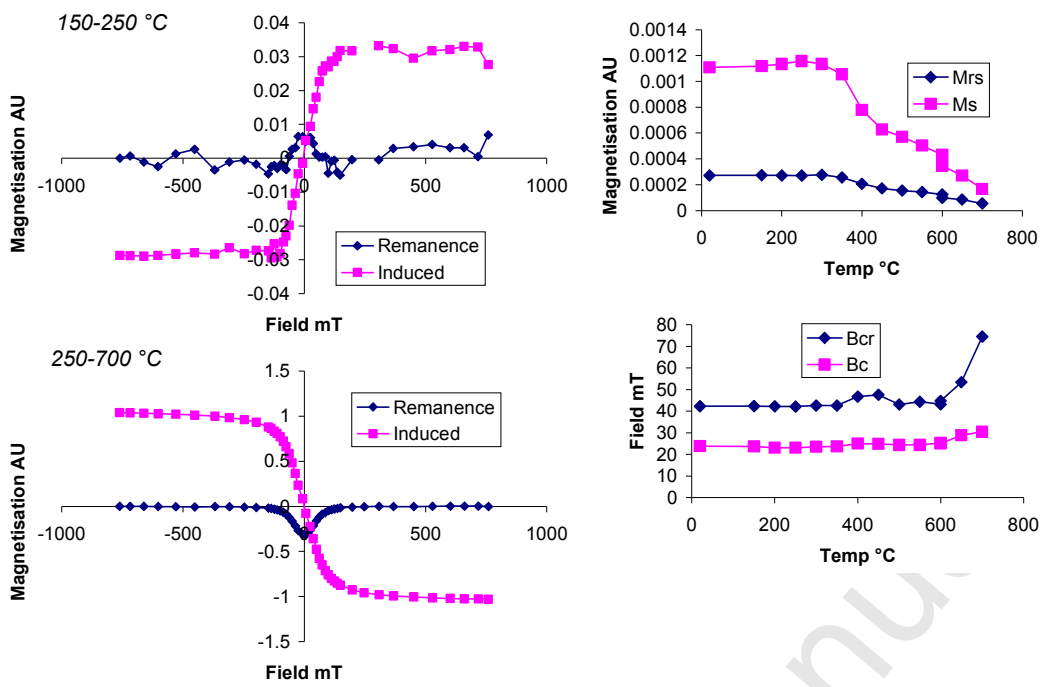


Figure 3

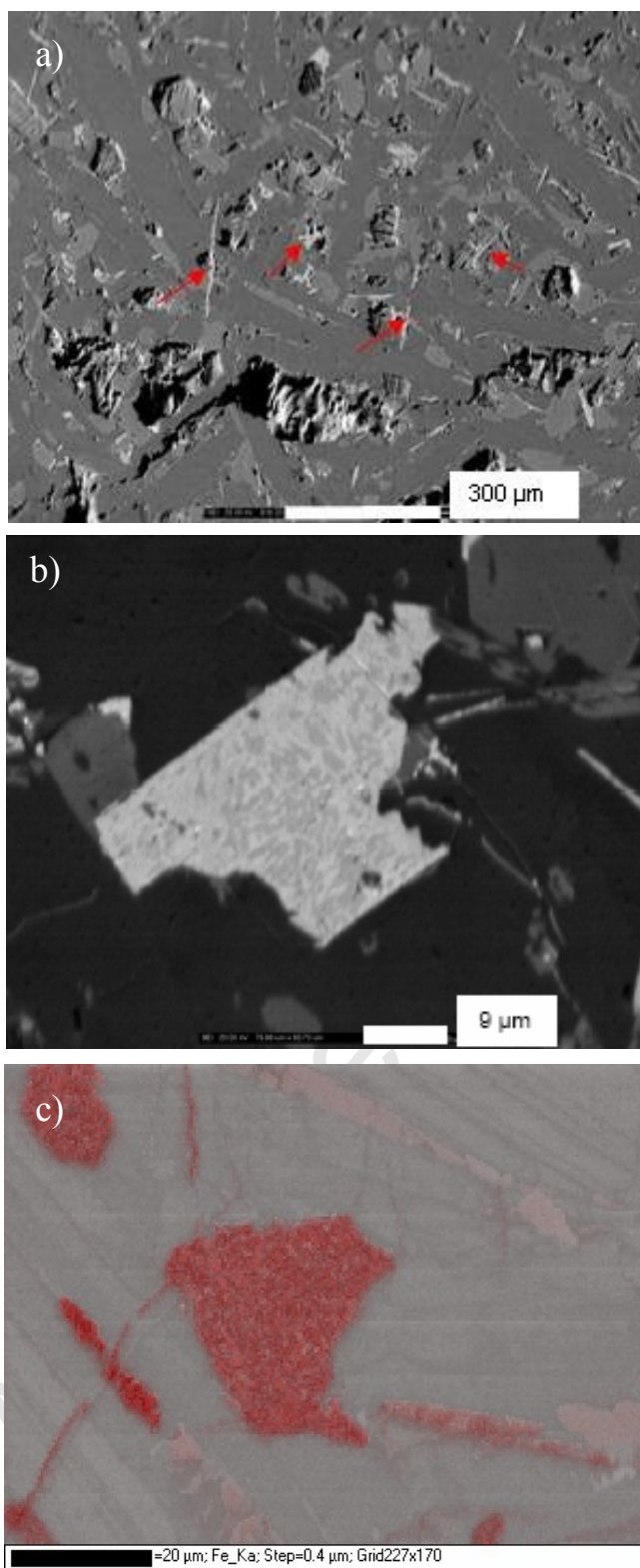


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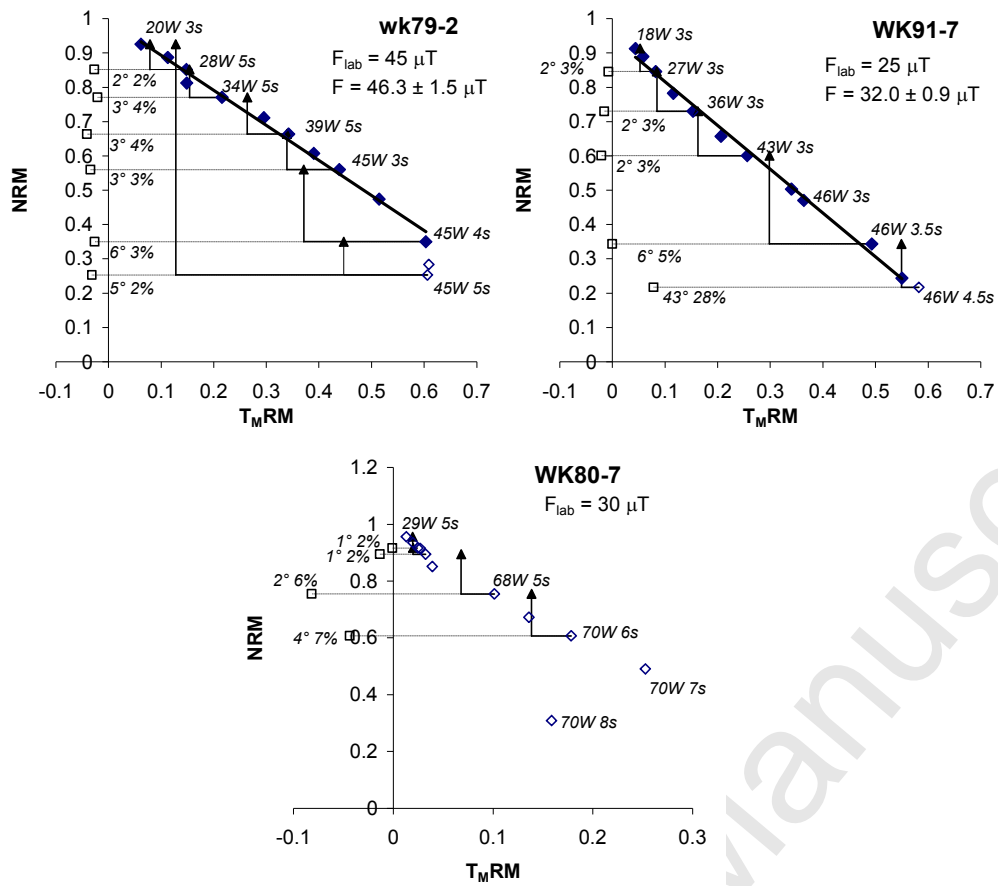


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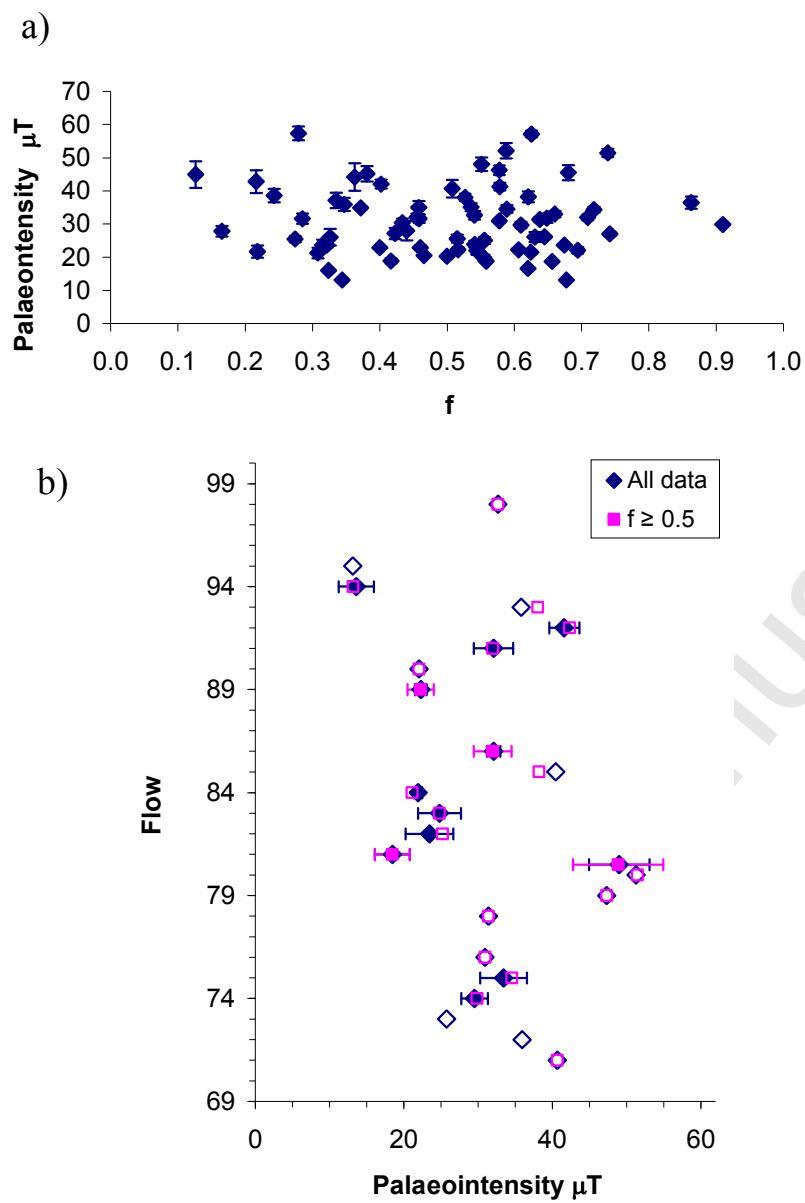


Figure 6.

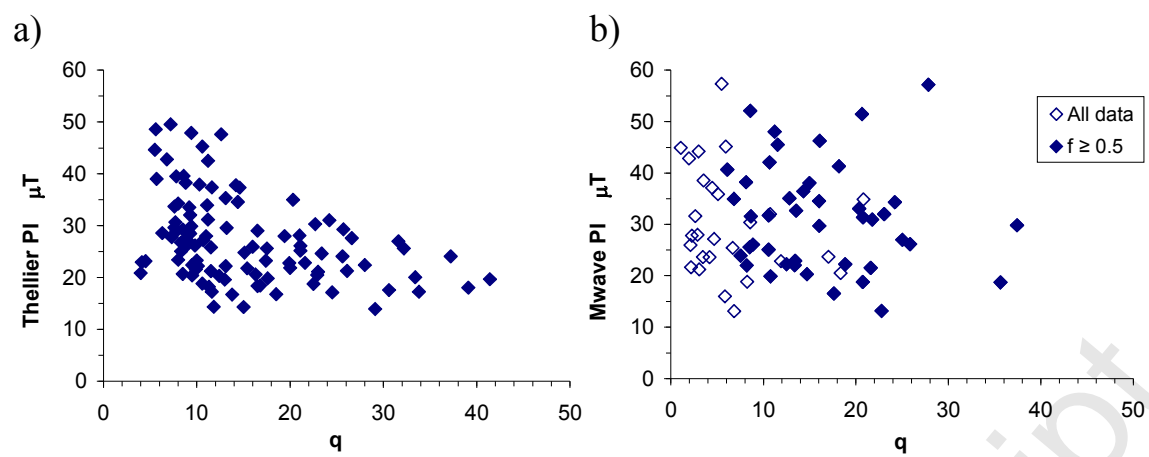


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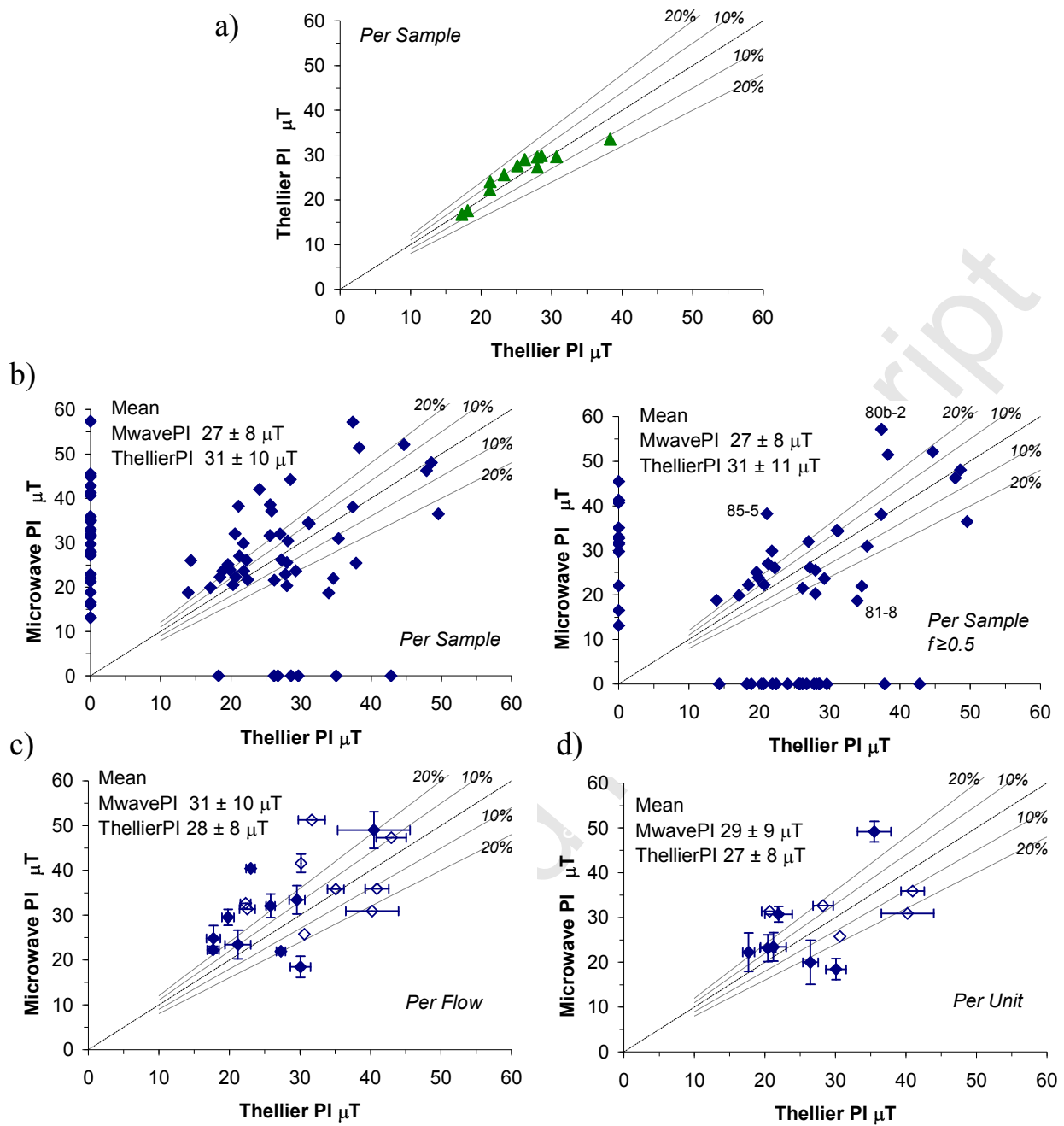


Figure 8

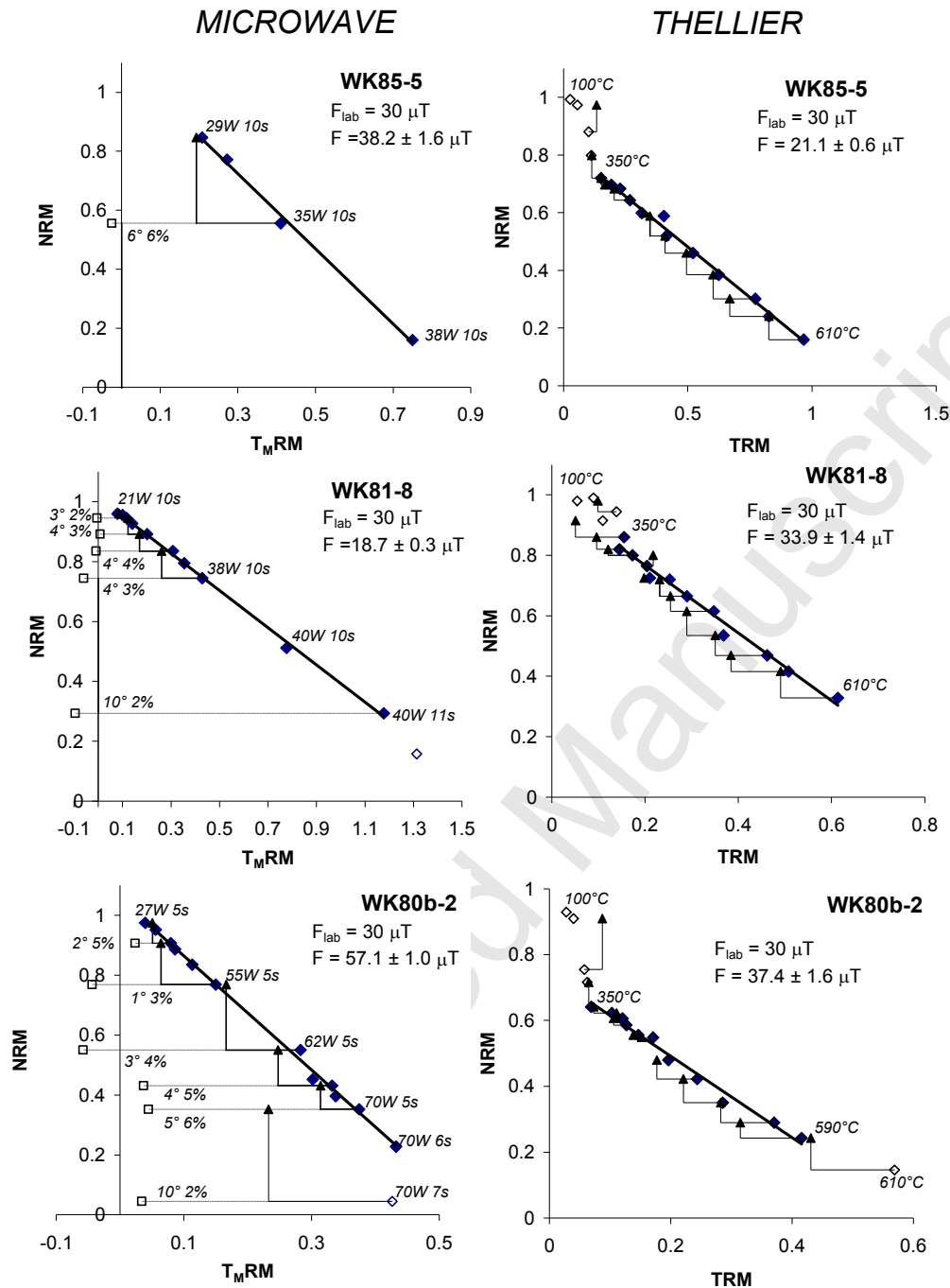


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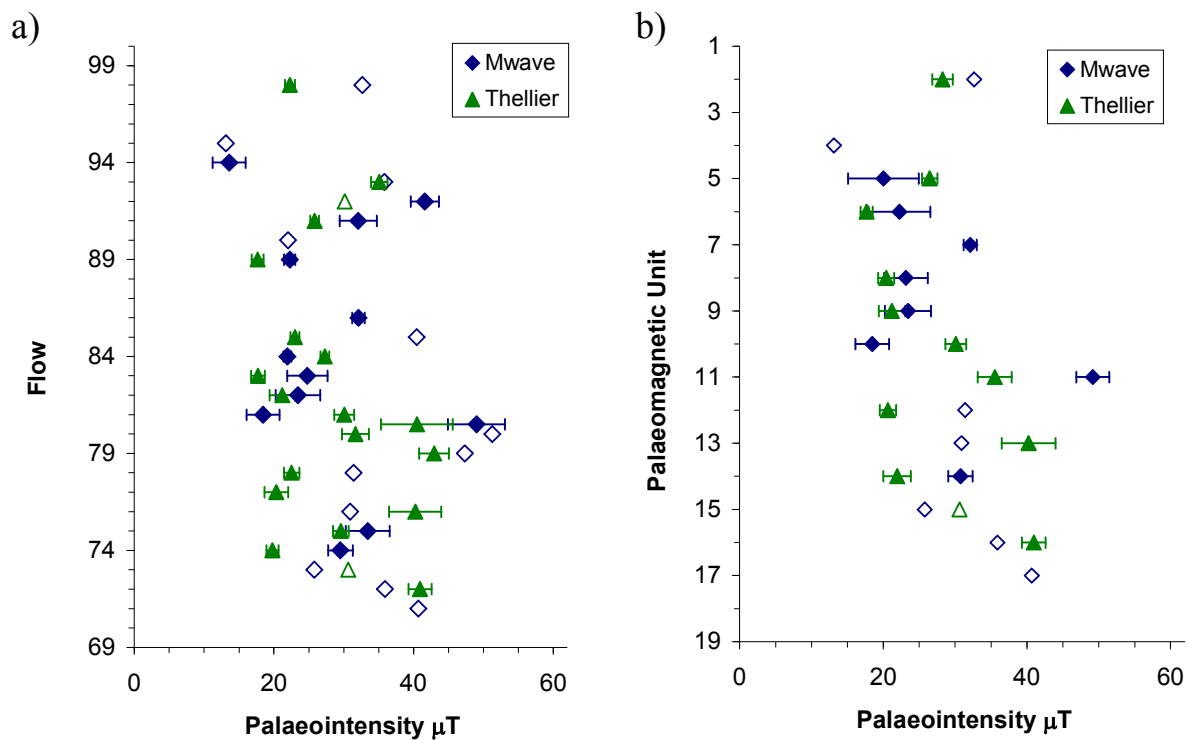


Figure 10.

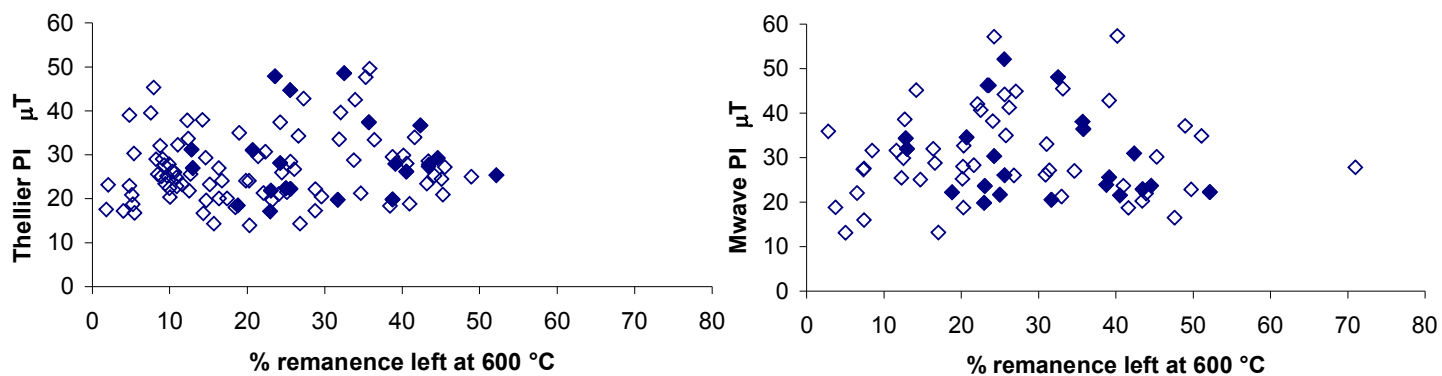


Figure 11