Comparison and calibration of nonheating paleointensity methods: A case study using dusty olivine

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[1] We present a comparative study of nonheating paleointensity methods, with the aim of determining the optimum method for obtaining paleointensities from “dusty olivine” in chondritic meteorites. The REM method, whereby thermoremanent magnetization (TRM) is normalized by saturation isothermal remanent magnetization (SIRM), is shown to “over normalize” TRM in dusty olivine due to the transformation of stable single-vortex (SV) states to metastable single-domain (SD) states in a saturating field. The problem of over normalization is reduced in the REMc and REM' methods, which more effectively isolate the high-coercivity stable SD component of remanence. A calibration factor of $f = 1600 \ (1000 < f < 2900)$ is derived for the REM' method. Anhysteric remanent magnetization (ARM) is shown to be a near perfect analogue of TRM in the stable SD component of dusty olivine. ARM normalization of the high-coercivity (100–150 mT) remanence with a calibration factor $f_{\text{ARM}} = 0.91 \ (0.7 < f_{\text{ARM}} < 1.2)$ yields paleofield estimates within $\pm 30\%$ of the actual field values for SD dominated samples. A Preisach method for simulating TRM acquisition using information extracted from first-order reversal curve (FORC) diagrams is shown to work well for SD dominated samples, but fails when there is a large proportion of SV remanence carriers. The failure occurs because (1) SV states are not properly incorporated into the Preisach distribution of remanence carriers, and (2) the acquisition of TRM by SV states is not properly modeled by the underlying SD thermal relaxation theory.

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1. Introduction

Magnetic fields are thought to have played an important role in the evolution of the solar nebula [Salmeron, 2012]. Although information about the strength of these magnetic fields may, in principle, be recoverable from primitive chondritic meteorites, the question of whether such meteorites recorded a preaccretionary remanence, and whether they retain a faithful record of this remanence today, remains open. The task of identifying, quantifying, and interpreting paleomagnetic signals from extraterrestrial materials poses a major technical challenge that probes the limits of current rock magnetic techniques.

The ideal carriers of primary paleomagnetic remanence are noninteracting single-domain (SD) grains with high coercivity. The primary remanence carriers in meteorites should, in addition, be very stable with respect to chemical and thermal alteration on the parent body, shock remagnetization and terrestrial weathering. It has been proposed that dusty olivines in chondrules fulfill these requirements [Uehara and Nakamura, 2006; Lappe et al., 2011]. Dusty olivines are high-Mg olivine grains containing submicrometer metallic Fe-Ni particles formed by subsolidus reduction of the fayalite component of the olivine during chondrule formation [Boland and Duba, 1975]. They are thought to be relict grains which survived the chondrule heating process unaltered and can be found in unequilibrated ordinary chondrites [Nagahara, 1981]. The first to consider dusty olivine as a potential carrier of preaccretionary remanence were Uehara and Nakamura [2006]. In a previous study, we investigated the mineral magnetic properties of synthetic dusty olivine and performed electron holographic imaging to determine the magnetic domain states found in Fe-Ni particles with a range of sizes and aspect ratios [Lappe et al., 2011]. It was concluded that a significant proportion of the exsolved Fe-Ni grains in dusty olivine are in the SD state and that some of these particles would acquire a remanence immediately on cooling through their Curie temperature ($T_c$). Furthermore, it was predicted that this remanence could remain stable for $>4.6$ Ga, even if the meteorite were heated to temperatures approaching $T_c$. In this study, we focus on the rock magnetic properties of synthetic dusty olivine produced in known magnetic fields, with the aim of comparing the accuracy of various nonheating methods for paleointensity determination and providing new paleointensity calibration curves specific to dusty olivine. It is anticipated that scanning magnetic microscopy techniques [Weiss et al., 2007] will soon enable remanence measurements to be made from individual regions of dusty olivine at sub chondrule length scales. The results presented here will permit the intensity of the magnetizing field to be calculated from such measurements with increased accuracy and confidence. Additionally, the manner in which the most successful nonheating paleointensity technique was identified in this study may serve as a case study for future researchers whose samples are prone to mineral alteration during heating.

2. Experimental Procedures

2.1. Sample Preparation

The magnetic properties of dusty olivine are sensitive to the Ni content of the metallic grains [Lappe et al., 2011]. For this study we created three suites of samples using olivine precursors with Ni contents spanning the range observed in natural dusty olivine [Ruzicka et al., 2008]: (1) synthetic Ni-free olivine, (2) synthetic olivine with 0.1 wt% Ni and (3) natural olivine crystals from an Icelandic basalt with 0.34 wt% Ni [Maclennan, 2008]. In all three samples, the olivine contained 90% forsterite ($\text{Mg}_2\text{SiO}_4$), and $\sim10\%$ fayalite ($\text{Fe}_2\text{SiO}_4$). For the preparation of the synthetic olivine precursors, stoichiometric mixtures of $\text{SiO}_2$, $\text{MgO}$, $\text{Fe}_2\text{O}_3$, and $\text{NiO}$ powders were ground together under acetone and pressed into pellets. These pellets were then heated up to 1300°C under a gas flow of 50% CO and 50% CO$_2$ for 20 h and quenched in water. This procedure was repeated up to 4 times to ensure complete reaction. The pellets were reground and repressed between each heating cycle. The fayalite contents of the olivine starting materials were estimated from their lattice parameters determined by X-ray powder diffraction using the calibration of Schwab and Kustner [1977].

The olivine precursors were reduced at high temperature in a flowing CO gas to form dusty olivine and then quenched into a known magnetic field to impart each with a laboratory thermoremanent magnetization (TRM). The rapid cooling rates used in these experiments are designed to mimic the acquisition of TRM during chondrule formation, where cooling rates are estimated to be of the order 10–1000 K/h [Ciesla, 2005; Jones et al., 2000]. Calibration factors derived here may not, therefore, be directly applicable to more
slowly cooled materials without performing an appropriate cooling-rate correction [Halgedahl et al., 1980]. The olivine precursors were ground into powders under acetone using an agate mortar and packed tightly into either a 2×2×2 or a 3×3×3 mm³ mould cut into a graphite crucible (Figure S1; supporting information). A short length of platinum wire was attached to the bottom of the mould so that an arrow-shaped indentation was preserved in the sample to mark its orientation. The graphite crucible was rigidly fixed to the end of a 1 m long rod of 1 cm diameter alumina using Pt wire. The rod was partially inserted through a hole in the top of a vertical-tube gas-mixing furnace, sealed with a gas-tight O-ring. The furnace was then heated from room temperature to 1350°C at a rate of 25°C/min under a pure CO gas flow. After 10 min at 1350°C, the samples were quenched by pushing down on the alumina rod until the sample was fully immersed in a beaker of water attached to the bottom of the furnace with a gas-tight O-ring seal. In this way, the sample was not exposed to air during quenching, which inhibits the formation of magnetite [Lappe et al., 2011]. Care was taken to keep the rod in a fixed orientation as it was pushed down, ensuring that the sample remained in a fixed orientation with respect to the applied magnetic field. Helmholtz coils were arranged on either side of the water beaker to provide a magnetic field varying between 200 µT and 1.5 mT during the quench. The field at the sample position was measured in all three directions using a LakeShore Model 455 DSP Gauss meter prior to heating up the furnace. The maximum standard deviation in field measurements taken over an interval of 30 min prior to each run was 0.005 mT. One to three samples were produced per run. Detailed information about the applied field in all three directions, as well as sample IDs and starting materials used can be found in Table S1 (supporting information).

2.2. Rock Magnetic Measurements

Rock magnetic measurements were performed at the Institute for Rock Magnetism, University of Minnesota. For all samples we measured (in order) alternating-field (AF) demagnetization of the laboratory TRM, acquisition of anhysteretic remanent magnetization (ARM), AF demagnetization of ARM and AF demagnetization of saturation isothermal remanent magnetization (SIRM). The bias fields for ARM acquisition were between 0.02 mT and 0.4 mT, applied along z. The maximum AF amplitude for the ARM acquisition was 150 mT. AF demagnetization of ARM acquired in a 0.4 mT bias field was performed over 10 steps with AF amplitudes between 4 mT and 150 mT. SIRM was induced using a field of 1 T applied along z. AF demagnetization of TRM and SIRM was performed over 21 steps with AF amplitudes between 2 and 150 mT. All remanence measurements were conducted at room temperature using a 2G Enterprises 755-4K superconducting rock magnetometer with a nominal sensitivity of 10⁻¹² Am². AF demagnetization was conducted using a 2G Enterprises in-line degausser, while ARMs were imparted using a Schonstedt alternating field demagnetizer outfitted with a pARM device. First-order reversal curve (FORC) measurements [Pike et al., 1999; Roberts et al., 2000] were performed on all samples using a Princeton Measurements vibrating sample magnetometer (VSM) at room temperature. The applied saturation field was between 0.8 and 1 T. A total of 106–132 curves per sample were measured, giving a field step width for adjacent FORCs between 6 and 8 mT. FORCs were processed using FORCinel [Harrison and Feinberg, 2008] applying a smoothing factor of 3 or 4. Room temperature susceptibility measurements at different frequencies were obtained using a MAGNON Variable Frequency Susceptibility Meter (VFSM). The temperature dependence of magnetic susceptibility was measured using an MFK1-FA MultiFunction Kappabridge (AGICO). An AC field amplitude of 711 A/m, a frequency of 976 Hz and a temperature sweep rate of 11°C/min was used for all measurements.

3. Results

A summary plot of remanence measurements for sample cube13 is shown in Figure 1. Additional summary plots for all other samples are provided in Figure S6 (supporting information). All samples acquired a single-component TRM during the quench and display a univectorial demagnetization trend heading toward the origin (Figure 1a). Remanence directions remain very stable during demagnetization and are coincident with the applied field direction, after accounting for minor rotation of the alumina rod during quenching (Figure 1b). The TRM could not be fully demagnetized in the maximum AF field amplitude of 150 mT, so a significant residual TRM remained in each sample prior to the ARM acquisition experiment.
For this reason, AF demagnetization of ARM does not trend toward the origin (Figure 1d). The residual TRM was subtracted from the ARM measurements prior to subsequent data analysis. The ARM acquisition is directly proportional to the bias field for all samples (inset to Figure 1c). AF demagnetization curves of TRM and ARM are very similar in shape (Figure 1c). AF demagnetization of SIRM, on the other hand, decays more rapidly at lower AF amplitudes.

No frequency dependence of room temperature magnetic susceptibility was detected in any sample, indicating an absence of superparamagnetic behavior. Magnetic susceptibility measurements as a function of temperature were conducted on all samples (see Figure S2; supporting information). With the exception of cube4, none of the “as-quenched” samples showed evidence for magnetite, as shown by the lack of an obvious Verwey transition in the low-temperature susceptibility curves. During the high-temperature measurements, all samples showed a very clear drop in susceptibility at around 770°C, consistent with the $T_c$ for pure metallic Fe. Some of the Ni-bearing samples showed hints of the martensitic bcc to fcc transition between 650°C and 750°C, characteristic of Ni-bearing Fe [Lappe et al., 2011]. There was little evidence of the quenched metastable fcc phase observed by Lappe et al. [2011], which gave rise to a sudden increase in magnetic susceptibility at $\sim$300°C caused by the transformation from fcc to bcc on heating. However, most samples showed signs of magnetite formation during the heating run above 300°C, which may obscure a possible signal due to the fcc to bcc transition. Two samples showed hints of a magnetic transition at about 200°C, indicating the presence of small amounts of Cohenite (Fe₃C) [Lappe et al., 2011].

[8] FORC diagrams calculated by averaging up to 10 individual FORC measurements for each suite of samples are shown in Figure 2. Note that the FORC diagrams have been processed in order to remove a common instrumental artifact, as described in Figure S5 (supporting information).
All FORC diagrams display a prominent central ridge (1) with broad coercivity distribution extending up to 600 mT, which can be attributed to noninteracting single-domain (SD) particles [Egli et al., 2010]. In addition, FORC diagrams display a broad, vertically spread positive peak (2) accompanied by a broad horizontally spread negative peak (3) just underneath the central ridge. This combination of positive and negative peaks can be attributed to the presence of single-vortex (SV) states [Pike and Fernandez, 1999]. Average SV nucleation and annihilation fields of 58 ± 55 mT and 170 ± 55 mT, respectively were extracted from the FORC diagrams, indicating that AF demagnetization to \( \gg 170 \) mT would be required to completely isolate the stable SD signal [Lappe et al., 2011]. Samples derived from the synthetic olivine precursors (Figures 2a and 2b) have more prominent SV signals than samples derived from the natural olivine precursor (Figure 2c). The synthetic samples also display more variability in the intensity of the SV signal from sample to sample.

4. Nonheating Paleointensity Methods

[10] Most methods of absolute paleointensity determination require the sample to be heated. However, in the case of extraterrestrial materials (many of which contain highly reduced or unstable magnetic minerals), heating can lead to severe thermal alteration of the specimen. Alteration would not only invalidate the paleointensity measurement, but also prevent the sample from being used for subsequent analysis. For these reasons, a number of nonheating methods are commonly employed to obtain relative paleointensity estimates for extraterrestrial materials [Kletetschka et al., 2003; Gattaacceca and Rochette, 2004; Carporzen et al., 2011]. Combined with an empirical calibration curve, these methods can also be used to determine absolute values of paleointensity, albeit with significantly larger uncertainties than the heating protocols more frequently used on terrestrial rocks [e.g., Coe, 1967; Shaw, 1974; Tauxe and Staudigel, 2004].

4.1. REM, REMc, and REM’

[11] The simplest nonheating method of paleointensity determination is the ratio of equivalent magnetizations (REM) method [Kletetschka et al., 2003]. Here the natural remanent magnetization (NRM) is normalized by the saturation isothermal remanent magnetization (SIRM) to give the REM ratio (REM = NRM/SIRM). An empirical calibration factor \( f = 3000 \) was determined by Kletetschka et al. [2003], such that the magnetizing field \( B \) (in \( \mu T \)) is equal to \( f \times \text{REM} \). A plot of REM versus \( B \) for each sample is shown in Figure 3a. Despite the scatter in the data, the REM acquisition curve is clearly nonlinear, as is expected for the range of \( B \) used in this study [Day, 1977; Parry, 1979; Shcherbakov et al., 1993]. Fields smaller than 200 \( \mu T \) could not be used in the TRM acquisition experiments due to the presence of a large stray magnetic field in our high-temperature laboratory. Note, however, that linearity of ARM acquisition in fields less than 200 \( \mu T \) was observed (Figure 1c). To describe the nonlinear acquisition of TRM, we fit the data using the expression (adapted from Tauxe [2010]):

\[
\text{REM} = \frac{\text{TRM}}{\text{SIRM}} = 2\text{REM}, \quad \int_{0}^{90} \tan \left( \frac{MB \cos \theta}{kT} \right) \cos \theta \sin \theta \, d\theta,
\]
where $\theta$ is the angle between the easy axis and the applied field, $M$ is an adjustable parameter representing the average magnetic moment of the particles and $REM_s$ is the saturation value of the REM ratio (i.e., the REM value that would be obtained after cooling in infinite field). When $REM_s = 1$, equation (1) describes the equilibrium magnetization of an assemblage of randomly oriented SD grains with uniaxial shape anisotropy [Tauxe, 2010]. Given the scatter in the data, the result of any individual fit depends on the starting values provided for $M$ and $REM_s$. The red solid curve in Figure 3a represents the average fit obtained after performing 120 individual fits to the data with randomly chosen starting guesses for $M$ and $REM_s$. The blue dotted curves are confidence bands calculated from the average fit curve $\pm 1\sigma$.

Figure 3. Plot of (a) REM and (b) REMc versus $B$ for each sample. Open circles, filled circles and diamonds represent samples made from synthetic Ni-free olivine, synthetic Ni-bearing olivine and natural olivine precursors, respectively. The red solid curve shows the average fit to all data using equation (1). Blue dotted curves represent the average fit $\pm 1\sigma$. Calibration factors ($f$) derived from the average and $\pm 1\sigma$ curves are shown in the inset.

describing the range of individual measurements may be slightly wider. Fitting produces a value of $REM_s = 0.36(7)$, where the number in parentheses represents the standard deviation in the last decimal place. The calibration factor $f = B/REM$ is shown in the inset to Figure 3a. The nonlinearity of the acquisition curves means that $f$ is not a constant, but increases with increasing $B$. The calibration factor tends to a constant value of $f = 2850$ in the low-field region of the data, where the remanence acquisition is linear with $B$. This calibration factor is consistent with the value of $f = 3000$ that is commonly used in the literature [Kletetschka et al., 2003; Gattacceca and Rochette, 2004]. Minimum and maximum $f$ factors were derived from the dashed curves, giving an $f$ factor range of $\sim 1850$–5040. This range is typical of the large uncertainties associated with REM paleointensity estimates. For example, for $REM_s = 0.2$, paleofield estimates would lie between $\sim 400$ and $\sim 1300 \mu T$.

[12] A key limitation of the REM method is that it is based on the total NRM of the sample and therefore cannot be used for samples that contain multiple remanence components. Several methods have been suggested to overcome this problem. The simplest modification is the REMc method [Acton et al., 2007; Kohout et al., 2008], where instead of using total NRM and SIRM, the REMc ratio is calculated using the NRM and SIRM that remain after a chosen AF demagnetization step. This procedure is intended to remove unwanted low-coercivity signals or overprints. Figure 3b shows the REMc ratio calculated from the residual TRM and SIRM remaining after AF demagnetization to 150 mT. A value of $REM_s = 0.6(1)$ is now obtained from the fit to equation (1), which is considerably higher than that obtained using raw REM values (Figure 3a). Despite the increase in $REM_s$, a similar average calibration factor for the linear region of $f = 2700$ is obtained.

[13] A second modification is the REM’ method proposed by Gattacceca and Rochette [2004]. Here the slope of the NRM demagnetization curve (dNRM/dAF) is normalized by the slope of the SIRM demagnetization curve (dSIRM/dAF) giving $REM’ = \Delta NRM / \Delta SIRM$. REM’ values have been evaluated at each AF demagnetization step from the ratio of slopes of the TRM and SIRM demagnetization curves (Figure 4a). All samples show a marked increase in REM’ with increasing AF demagnetization step up to AF amplitudes of 100 mT, followed by roughly constant REM’ values between 100 and 150 mT (boxed region in Figure 4a). Average REM’ values for the 100–150
mT field range are shown in Figure 4b. A value of REM = 0.6(1) is obtained from the fit to equation (1). An average calibration factor for the linear region of $f = 1600$ is obtained. This is roughly a factor of two smaller than the value of $f = 3000$ normally assumed for the REM' method [Gattacceca and Rochette, 2004]. Figure 4c shows a TRM fit applied to the REM’ of the samples produced from natural olivine only. These samples show less scatter than those made from synthetic olivine. The fit to equation (1) gives a value for REM of 0.60(7) and an average $f$ factor of 2120 (Table S2; supporting information).

### 4.2. ARM Normalization Methods

[14] An alternative approach to nonheating paleointensity determination uses ARM rather than SIRM as a normalizing factor [Stephenson and Collins, 1974; Yu, 2010; Carporzen et al., 2011]. This approach is based on the theory that ARM provides a better approximation to TRM than SIRM. The relative efficiency of TRM versus ARM acquisition can be described by an empirical calibration factor $f_{\text{ARM}}$:

$$f_{\text{ARM}} = \frac{\chi_{\text{TRM}}}{\chi_{\text{ARM}}}, \quad (2)$$

where $\chi_{\text{TRM}}$ and $\chi_{\text{ARM}}$ are the susceptibilities of TRM and ARM acquisition, respectively. If TRM was acquired in a field $B_{\text{ancient}}$, while ARM was acquired in the laboratory using a bias field $B_{\text{bias}}$, then the calibration factor can be calculated as:

$$f_{\text{ARM}} = \frac{\text{TRM}}{\text{ARM}} \frac{B_{\text{bias}}}{B_{\text{ancient}}}, \quad (3)$$

where TRM and ARM are total remanences. To calculate the intensity of the ancient field equation (3) can be rearranged:

$$B_{\text{ancient}} = \frac{\text{TRM}}{\text{ARM}} \frac{B_{\text{bias}}}{f_{\text{ARM}}}. \quad (4)$$

[15] As in the REM’ method, the total remanences can be replaced by the demagnetization slopes $d\text{TRM}/d\text{AF}$ and $d\text{ARM}/d\text{AF}$. Hence the ratio of total remanences (TRM/ARM) becomes $(d\text{TRM}/d\text{AF})/(d\text{ARM}/d\text{AF}) = (d\text{TRM}/d\text{ARM})$:

$$B_{\text{ancient}} = \frac{d\text{TRM}}{d\text{ARM}} \frac{B_{\text{bias}}}{f_{\text{ARM}}}. \quad (5)$$

Figure 4. (a) REM’ values plotted as a function of AF demagnetization step for all samples. Dotted lines, dash-dotted lines and solid lines represent samples made from synthetic Ni-free olivine, synthetic Ni-olivine and natural olivine precursors, respectively. Line colors refer to different applied fields (blue: 211 μT, green: 352 μT, brown: 619 μT, pink: 948 μT, red: 1233 μT, black: 1547 μT). The boxed region shows the 100–150 mT range which was used to calculate average REM’ values. (b, c) Average REM’ values as a function of $B$. Samples made from synthetic Ni-free olivine, synthetic Ni-olivine and natural olivine are represented by open circles, filled circles and diamonds, respectively. The red solid curves show the average fit to (Figure 4b) all samples and (Figure 4c) natural samples only using equation (1). Blue dotted curves represent the average fit ± 1σ. Insets shows $f$ factor values derived from the average and ± 1σ curves.
The slope dTRM/dARM can be determined from a plot of TRM lost versus ARM lost during AF demagnetization. Multiple remanence components can be accounted for by evaluating dTRM/dARM over a restricted range of AF amplitudes (Figure S3; supporting information). As with the REM’ method (Figure 4a), we restricted the calculation of dTRM/dARM to AF amplitudes between 100 and 150 mT by fitting a straight line to the last three data points of each curve. A plot of dTRM/dARM versus the applied laboratory field is shown in Figure 5. The solid curve in Figure 5a shows the average fit to all samples except cube4 using equation (1). Cube4 was excluded from the fit as this sample was shown to contain an anomalous amount of magnetite. There is a systematic difference in the behavior of samples synthesized with natural olivine (diamonds) compared with those made with synthetic olivine (circles). The average fit to the natural samples only is shown in Figure 5b. Note the significantly lower degree of scatter in the natural samples compared to the synthetic samples.

The absolute values of dTRM/dARM in Figures 5a and 5b are dependent on the value of bias field used for the ARM acquisition (0.4 mT in this case). Rearranging equation (5), however, we can use the data to calculate the calibration factor, $f_{\text{ARM}}$, which can be used to determine paleointensity using any bias field. Values of $f_{\text{ARM}}$ calculated both from the data and from the average fitted curves in Figures 5a and 5b are shown in Figure 5c. Values of $f_{\text{ARM}}$ calculated from the fit to all samples vary from $\sim 2$ at low fields to $\sim 1$ at high fields, which compares well with the value of $f_{\text{ARM}} = 2.60 \pm 1.32$ determined by Yu [2010]. The natural samples show considerably less scatter and a much more restricted range of $f_{\text{ARM}}$ (0.7 < $f_{\text{ARM}}$ < 1.2) in comparison with the synthetic samples (1 < $f_{\text{ARM}}$ < 1.85).

4.3. FORC Method

A new method of nonheating paleointensity determination has recently been proposed by Muxworthy and Heslop [2011] and Muxworthy et al. [2011]. Their method uses Preisach distributions derived from FORC diagrams as empirical input to a numerical simulation of TRM acquisition. The acquisition of TRM is physically modeled using thermal relaxation theory, assuming uniaxial
SD particles dominated by shape anisotropy. The simulations described here were performed using FORCintense [Harrison et al., 2012], a modified implementation of the Muxworthy-Heslop method within FORCinel [Harrison and Feinberg, 2008]. For a detailed description of the theoretical basis of the method see Muxworthy and Heslop [2011]. Details specific to this study, as well as a description of modifications made in FORCintense, are described below.

[19] FORC diagrams with a smoothing factor (SF) of 3 were used as input to the simulations (Figure 2). Smoothing has the effect of broadening the central ridge, and this broadening needs to be corrected before the smoothed average FORC diagrams can be used to derive the Preisach distribution. We determined the width of the central ridge, \( W \), by fitting a Gaussian function to a vertical profile averaged between 0.2 < \( B_c < 0.3 \) T. Widths were calculated for 1 ≤ SF ≤ 5 and an estimate of \( W_{\text{SF}=0} \) was determined by linear extrapolation. A corrected FORC diagram with SF = 3 was obtained by multiplying \( B_u \) values by a factor \( W_{\text{SF}=0}/W_{\text{SF}=3} \). The corrected FORC diagram was then converted to a Preisach distribution by symmetrical averaging about the \( B_u \) axis [Muxworthy and Heslop, 2011].

[20] To simulate TRM acquisition, the Preisach distribution was sampled at 200,000 points, each point representing a randomly oriented hysteron. Each hysteron represents an ensemble of magnetic particles that share similar characteristics (i.e., they have the same \( B_c, B_u \) and easy axis orientation). The probability of picking a hysteron at a given position in \( B_c-B_u \) space is proportional to the Preisach distribution at that point. The program written and used in Muxworthy et al. [2011], erroneously used the Preisach distribution not only to control the probability density of hysterons but also to weight the magnetic contribution of each hysteron, i.e., this procedure weighted the hysteron contribution by a factor proportional to the square of the Preisach distribution itself. This leads to calculated AF demagnetization curves that decay far too rapidly with AF amplitude. FORCintense corrects for this computational mistake by using the Preisach distribution to control the probability density of hysterons, but does not apply a Preisach weight to the magnetic contribution of each hysteron. This modification was found to yield much improved agreement between calculated and observed AF demagnetization curves for a range of test samples [Harrison et al., 2012].

[21] The total TRM is calculated during stepwise cooling from \( T_c \) to room temperature, with increasing numbers of hysterons passing from an unblocked to a blocked state as temperature decreases. Since the Preisach distribution yields information about the coercivity but not the volume of particles, blocking temperatures must be estimated using an empirical relationship that links the thermal fluctuation field acting on a particle to its coercivity [Barbier, 1954; Wohlfarth, 1984; Muxworthy et al., 2009]. FORCintense uses the empirical calibration of this relationship by Muxworthy et al. [2009]. Values for \( T_c \) and the temperature dependence of saturation magnetization appropriate to pure metallic Fe were used [Garrick-Bethell and Weiss, 2010]. A total cooling time of 0.02 h was assumed (an estimate of the total quench time of the synthetic dusty olivine samples).

[22] After calculating the total TRM at room temperature, the Preisach distribution was used to simulate AF demagnetization curves according to the procedure described by Muxworthy and Heslop [2011]. A comparison of calculated versus observed AF demagnetization curves is shown in Figure 6. Data points show the observed TRM normalized by the room temperature SIRM (i.e., REM). Lower, middle and upper curves are simulated REM values for fields that are a factor of 0.5, 1, and 2 times the actual laboratory field used, respectively. Typically there is poor agreement between the calculated and observed demagnetization curves for AF amplitudes < 100 mT. Above 100 mT, however, the agreement between calculated and observed remanence values improves significantly. The shape of the AF curves is generally better described for the natural samples (Figure 6c). The failure to get good agreement over the whole range of AF amplitudes meant that it was not possible to obtain a consistent estimate of the paleointensity at each AF amplitude step, as required by the protocols of Muxworthy and Heslop [2011] and Harrison et al. [2012]. Instead, we have used the FORC method to simulate both TRM and SIRM AF demagnetization curves and thereby predict values for REM, REMc, and REM′ which can be compared directly to the experimental observations. REMc values were calculated from the residual simulated TRM and SIRM after 150 mT AF demagnetization. REM′ values were calculated from the ratio of the slopes of simulated TRM and SIRM demagnetization curves, averaged over the 100–150 mT range (Figure S4; supporting information).
Simulated REM, REMc, and REM’ acquisition curves are shown in Figure 7 and compared with the experimental data from Figures 3 and 4. Calculations were performed for applied fields up to 10 mT in order to explore the nonlinearity of remanence acquisition. The overall agreement between the calculated curves and the data is good. The agreement is worst for the REM method (Figure 7a) and best for the REM’ method (Figure 7c). There is particularly good agreement if one considers only samples made with natural olivine precursors (diamonds in Figure 7c). The factors calculated from the FORC method are 3400, 3620, and 2760 for the REM, REMc, and REM’ curves, respectively.

5. Comparison of Methods

5.1. REM, REMc, and REM’

The average fits in Figures 3a, 3b, and 4b provide calibration curves for the REM, REMc, and REM’ methods, respectively (Table S2). Using these calibration curves we have obtained an estimate of the laboratory field intensity for each sample. Histograms of the normalized field intensity (i.e., the calculated field divided by the actual field) are shown in Figures 8a–8c. All three methods show similar distributions: normalized field estimates are centered around 1, the lowest normalized field estimates are around 0.5 and the maximum normalized field estimates are around 2.

5.2. FORC Method

Normalized field estimates based on simulated REM, REMc and REM’ values obtained using the FORC method are shown in Figures 8d–8f. Each field estimate was obtained by simulating the remanence acquisition curve using the FORC diagram for that specific sample. The distributions of normalized field estimates are similar to those of the conventional REM, REMc, and REM’ methods, although there is a greater tendency to overestimate the paleofield and there are more samples with normalized paleofield estimates greater than 2. Both the conventional REM and FORC methods perform slightly better when only the samples made from natural olivine precursors.

Figure 6. Observed and calculated TRM/SIRM for three different samples. Open circles represent the measured AF demagnetization curve, the blue dash-dotted line, the green solid line and the red dashed line correspond to an AF demagnetization curve simulated at 0.5, 1 and 2 times the actual applied laboratory field value. (a) Cube12 (synthetic Ni-bearing olivine). (b) Cube28 (synthetic Ni-free olivine). (c) Cube11 (natural olivine).
are considered (Figure 9). For these natural samples, normalized field estimates are $\geq 0.5$ and $< 2$. Particularly noteworthy are the clusters of points close to 1 for the REMc and REM’ FORC methods (Figures 9e and 9f).

5.3. ARM Methods

[26] The fit curve in Figure 5a provides a calibration curve for the ARM method with a bias field of 0.4 mT. A histogram of normalized field estimates based on this calibration curve is shown in Figure 10a. Like the REM and FORC methods, the spread of normalized field estimates is consistent with an uncertainty of around a factor of 2. The method performs considerably better when only samples made with natural olivine precursors are considered. The histogram in Figure 10b was obtained using the calibration curve from Figure 5b. This calibration yields normalized field estimates between 0.8 and 1.3 which are centered around 1, suggesting an uncertainty in paleofield estimates of around $\pm 30\%$.

6. Discussion

6.1. Remanence Acquisition

[27] All samples show highly stable remanence directions during AF demagnetization to 150 mT (Figures 1a and 1b). This demonstrates that (a) our experimental design (Figure S1) was successful in keeping the samples in a fixed orientation with respect to the applied field during rapid quenching, and (b) that dusty olivine is a reliable carrier of high-coercivity remanence. The REM, REMc, and REM’ data show that TRM acquisition is non-linear at fields over 400 mT and suggests that TRM acquisition saturates at REM$_s$ values less than 1 (Figures 3 and 4; Table S2). This effect can be explained by the presence of SV states. Indirect evidence from FORC diagrams (Figure 2) and direct evidence from electron holography [Lappe et al., 2011] suggests that SV states are common in dusty olivine. SV states are found in particles above a critical size threshold [Butler and Banejree, 1975]. Lappe et al. [2011] identified particles close to this threshold that can adopt either a stable SV state or a metastable SD state depending on their temperature and field history. Such particles are more likely to adopt their stable state during acquisition of weak-field TRM, and so we expect a significant proportion of TRM to be carried by SV states. Acquiring TRM in larger fields will cause more vortex cores to become aligned with the field, but the total remanence stays low as long as the applied field is lower than the vortex annihilation field (>170 mT at room temperature). Exposing the sample to a saturating field at room temperature, however, will cause some particles to switch to their metastable SD state. Hence, saturation TRM
(stable vortex core moments aligned with the field) is potentially much lower than SIRM (metastable SD moments aligned with the field), causing REM, to be less than 1. In other words, it is important to emphasize that samples characterized by SV states will have their TRMs “over normalized” by SIRM due to the fact that these remanences are carried by different micromagnetic states. The problem of over normalization is worst for the REM method ($\text{REM}_s = 0.36$), which includes the maximum contribution from SV states. The problem is less of an issue for the REMc and REM’ methods ($\text{REM}_s = 0.6$), in which a higher proportion of remanence is carried by SD states. We will discuss the relative merits of SIRM and ARM normalization below in section 7.3.

6.2. Data Scatter

[28] Considering all samples together, there is a very large degree of scatter in the normalized remanence values. The degree of scatter is similar for both SIRM and ARM normalization methods. The degree of scatter is considerably less, however, when the samples made with natural olivine precursors are considered on their own, particularly when using the REM’ and ARM methods (Figures 9 and 10). A comparison of FORC diagrams for the three suites of samples (Figure 2) indicates that the natural samples contain, on average, a much smaller proportion of particles in the SV state. The samples made with synthetic olivine precursors not only contain a much larger proportion of SV particles but also display much larger variability in the SD/SV ratio from sample to sample. We suggest, therefore, that the scatter observed in the normalized remanence is linked to the variable amounts of SV versus SD particles in the samples. The reason for the larger proportion of SV particles in the synthetic samples is related to the finer grain size of the olivine precursors. Lappe et al. [2011] found that larger Fe-Ni particles typically nucleate at grain boundaries. The fine grain size of the synthetic olivine precursors creates more opportunities to nucleate and grow large (i.e., SV) Fe-Ni particles. This conclusion is supported by scanning electron microscopy observations (Figure S7; supporting information). There is little change in the degree of scatter when comparing REM, REMc, REM’, and

Figure 8. Histograms showing normalized paleofield estimates (calculated field/applied field) for tested paleointensity methods for all samples. Normalized paleofield intensities calculated from the average fit curve fitted to the (a) REM, (b) REMc, and (c) REM’ data. Normalized paleofield intensities calculated from the individual simulated (d) REM, (e) REMc, and (f) REM’ curves for each cube.
ARM methods for the synthetic samples alone. This suggests that the origin of the scatter is related to a fundamental difference in the TRM acquisition process for SD versus SV particles rather than the ‘over normalization’ problem discussed in section 7.1. Samples made with natural olivine precursors have a magnetic mineralogy that corresponds closely to the paleomagnetic ideal of noninteracting SD particles dominated by shape anisotropy, making this suite of samples arguably the best of the three for calibration purposes.

6.3. SIRM Versus ARM Normalization

[29] Calibration factors for REM, REMc, and REM’ methods are in good agreement with literature values [Kletetschka et al., 2003; Gattacceca and Rochette, 2004]. Out of the three REM-derived methods, REM’ yields marginally less scatter in recovered field values and an REMs value closest to 1. This suggests that the REM’ method comes closest of the three to isolating the SD signal. We recommend REM’ as the preferred method of SIRM normalization.

[30] By far the best results, however, are obtained for the natural samples using the ARM normalization technique (Figure 5). The close similarity between TRM and ARM demagnetization curves indicates that ARM provides a much better analogue of TRM in these samples than SIRM. Taking all samples together, our calibration factor, $f_{\text{ARM}}$, is in good agreement with the previous results of Yu [2010], although there is still considerable scatter in the data caused by variations in the SD/SV ratio of the synthetic samples. Taking only the natural samples, which have a higher proportion of SD particles, we find an average value of $f_{\text{ARM}} = 0.91 \pm 0.20$. A value of $f_{\text{ARM}} \sim 1$ implies that ARM is an almost perfect analogue of TRM acquisition in the SD component of dusty olivine (equation (2)). This greatly simplifies the process of extracting paleointensities from dusty olivine. It should be noted, however, that this calibration factor strictly applies only to the specific range of AF demagnetization fields used in this study (100–150 mT). It remains to be seen whether the factor decreases further when higher demagnetization fields (i.e., $> 170$ mT) are employed in an attempt to better isolate the SD component.
6.4. FORC Method

This study represents the first test of the FORC/Preisach method proposed by Muxworthy and Heslop [2011] and Muxworthy et al. [2011] using well-characterized synthetic samples. The method fails to describe the shape of the AF demagnetization curves correctly, especially at low-AF amplitudes (Figure 6). This prevents the method from being used in its original form to recover estimates for the paleofield. The reason for the failure is, again, due to the preponderance of SV states, which pose several problems for the FORC method. Firstly, FORC signals associated with SV states tend to occur well away from the central ridge (signal 2 in Figure 2). In the FORC method, such hysterons become field-blocked in positive and negative saturation states in equal proportions, so their contribution to the overall remanence cancels out. In addition, both signals 2 and 3 in Figure 2 are associated with SV nucleation and annihilation processes, rather than SD switching processes. Assigning hysterons to these signals results in the creation of SD particles with incorrect values of coercivity and interaction field. This means that the coercivity distribution of remanence-carrying particles (and, therefore, the AF-demagnetization curve) is not accurately reproduced. Lastly, as argued in section 7.2, our results point (perhaps not unsurprisingly) toward a fundamental difference in the efficiency of TRM acquisition for SD versus SV particles. The FORC method is based on SD thermal relaxation theory, and therefore is, by definition, unlikely to perform well for samples dominated by SV particles.

Despite these limitations, our results demonstrate that the modified FORC method performs well in terms of predicting REM’ values (Figure 7c). The results are particularly impressive for the natural samples where the influence of SV states is smaller (Figure 9f). These results confirm, therefore, that when the magnetic mineralogy of a sample corresponds closely to the assumptions and theoretical basis of the method (i.e., SD particles dominated by shape anisotropy), the FORC simulations provide a good model of TRM acquisition. Potential improvements to the method could be made by measuring higher resolution FORC diagrams, which allow more efficient separation of the SD and SV contributions [Egli et al. 2010]. While the FORC method does not, in its current form, provide a significant improvement in accuracy or precision compared to the empirical calibration of our preferred ARM normalization technique, it does at least provide a theoretical foundation for the empirical calibration. If the presence of SV states can be properly accounted for, the FORC method may offer a way to significantly reduce the scatter associated with natural variability in the SD/SV ratio in the future.

7. Conclusions

(1) The acquisition of TRM in dusty olivine is nonlinear for fields greater than \( \sim 400 \mu\text{T} \).

(2) SIRM is larger than saturation TRM due to the transformation of SV states to metastable SD states in a saturating field. This transformation leads to ‘over normalization’ of TRM by SIRM in the REM method.

(3) Over normalization is reduced in the REMc and REM’ methods, as more of the SD component of remanence is isolated. The preferred
method of SIRM normalization is REM’ with a calibration factor of $f = 1600$ (1000 < $f$ < 2900).

[40] (4) The high degree of scatter in the normalized remanence values correlates with high variability in the SD/SV ratio of the samples detected using FORC diagrams.

[41] (5) ARM provides a closer analogue of TRM than SIRM. For samples dominated by SD states we observe $\chi_{\text{ARM}} \sim \chi_{\text{TRM}}$, indicating that ARM is a near perfect analogue of SD TRM in this material.

[42] (6) The recommended method of non-heating paleointensity determination for dusty olivine is ARM normalization of the high-coercivity (100–150 mT) remanence using a calibration factor $f_{\text{ARM}} = 0.91$ (0.7 < $f_{\text{ARM}}$ < 1.2). Using this method, recovered field estimates are expected to be within ±30% of the actual field values for SD dominated samples.

[43] (7) The modified FORC method works well for SD dominated samples, but fails when there is a large proportion of SV remanence carriers. The failure occurs for two reasons: (i) SV states are not properly incorporated into the Preisach distribution of remanence carriers, and (ii) the acquisition of TRM by SV states is not properly modeled by the underlying SD thermal relaxation theory. If both these problems can be solved, the FORC method has the potential to further reduce the uncertainties inherent in non-heating paleointensity methods.

[44] (8) On the basis of results from the different techniques compared here, the SD/SV balance exerts a strong control on final paleointensity estimates. The use of FORC diagrams is recommended, therefore, as an essential component of sample selection.

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