Commonly used experimental parameters for acquisition of anhysteretic remanent magnetization (ARM) and its anisotropy (AARM): Results and recommendations from a rock magnetic community survey

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1. Motivation

Anhysteretic remanent magnetization (ARM) and its anisotropy (AARM) are properties widely used in environmental magnetism, paleomagnetism, and magnetic fabric studies, to gain information about magnetic remanence carriers and their preferred alignment. These data, in turn, can help define depositional environments for sediments, emplacement mechanisms for intrusive rocks, or deformation histories for metamorphic rocks. In environmental magnetism, ratios of ARM to susceptibility or isothermal remanent magnetization (IRM) (amongst other parameters) help determine the magnetic mineralogy, grain size and domain state, and hence the environmental processes under which rocks formed or altered [Liu et al., 2012]. Being the best room temperature equivalent for a natural thermal remanence [Potter, 2004], ARM and AARM experiments are also important in paleomagnetic studies, where they are widely used to better understand remanence acquisition and its anisotropy. In fabric studies, AARM as well as anisotropy of partial anhysteretic remanence (ApARM) are characterized to determine the preferred alignment of the remanence-carrying grains, or a sub-population of remanence-carrying grains [Jackson and Tauxe, 1991].

For the community to continue to use these ARM and AARM applications and innovate methods, we should encourage comparable ARM and AARM protocols across different laboratories. Reaching consensus on the standard procedure(s) and parameters for ARM and AARM experiments, will also help define suitable parameters for automated systems. Note that different types of studies employing ARMs, e.g. environmental vs. fabric studies, may call for different sets of experimental parameters. Here, we will focus particularly on how ARMs are imposed on samples. Imparting an ARM is a complex physical process, and the intensity of an ARM will depend not only on a specimen’s inherent magnetic properties, but also on a number of experimental variables, such as the strength of the DC bias field, the peak alternating field (AF) used during the application of the ARM, and the decay rate of the AF [Egli, 2006; Egli and Lowrie, 2002; Sagnotti et al., 2003; Yu and Dunlop, 2003]. These same experimental parameters can also change the perceived AARM for a specimen [Bilardello and Jackson, 2014], which may be further affected by the number of directional ARMs measured, and whether or not samples are demagnetized between directional measurements. Decay rates for AF are rarely reported in published studies, making it difficult to compare results obtained from different laboratories.

Given these concerns, and because 15 years have passed since the inter-laboratory calibration study by Sagnotti et al. [2003], we conducted a survey to determine the range of experimental parameters for ARM and AARM measurements that are currently employed within our community.
Preparation of samples and magnetic analysis on filters with atmospheric particles.

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Project background
Atmospheric aerosols, once completed their cycle in the atmosphere, return to the surface of the planet where they interact in different ways depending on the physical-chemical typology of the environment (hydrosphere, lithosphere, biosphere or anthroposphere) where they end up depositing. Depending on the medium to which they are transferred and based on their chemical or mineralogical composition, the associated impacts may be relevant.

The DONAIRE project has carried out an integrated study of atmospheric deposition in a wide geographic area, using a common methodology and covering different microenvironments. The project proposed the geochemical, magnetic and mineralogical characterization of the atmospheric deposition in the NE of Spain in biweekly monitoring for a full year in 12 different locations (from pristine areas to heavily anthropogenic environments).

The amount of atmospheric deposition by mass, its chemical composition and a complete characterization of its magnetic properties are being quantified, together with the relative enrichment of anthropogenic zones with respect to their closest remote counterparts. The final aim of the project is to establish relationships between geochemical species and / or sources with respect to magnetic properties in order to define magnetic-geochemical proxies. Special attention is paid to events of interest such as episodes of Saharan dust, pollution events or particular emission sources.

Methods at IRM
The low total particle mass collected in the filters (0.005-0.05 g) led to having to optimize sample preparation. The main goal was to conserve the total mass of the filter to be able to estimate the concentration of the magnetic minerals and to evaluate the magnetic properties per unit of mass.

Because the samples were distributed over the 47 mm (diameter) surface of the air filters, specific precautions were taken to guarantee centering of the specimens within the instruments’ measurement regions (Fig. 1). The samples were folded over in four, rolled and inserted into 8 mm diameter straws before being compressed to a ~ 1 cm high cylinder.

Hysteresis loop determinations, Isothermal Remanent Magnetization acquisition and backfield demagnetization curves at ambient temperature where acquired in a Vibrating Sample Magnetometer (VSM) in maximum fields of 1-1.5 T, depending on the specimen. Because the specimens possessed weak magnetizations, averaging times of 20-30 seconds were used, resulting in typical experiment durations of 0.5-1 hours.

Remanent magnetic measurements and AC susceptibility as a function of temperature and frequency were performed on Magnetic Properties Measuring Systems (MPMS). Proceeding sequences consisted of: i) cooling to 20 K in the presence of a saturating 2.5 T field (FC) and subsequent measuring of the remanence on warming; ii) After cooling, in absence of magnetic field (ZFC), a low temperature saturation isothermal remanent magnetization (SIRM) of 2.5 T was imparted and the remanence was measured again on warming to room temperature; iii) Subsequently, a room temperature SIRM (RTSIRM) was measured upon on cooling; iv) Lastly, frequency dependent (1, 10, 100 Hz) in-phase and quadrature magnetic susceptibilities were measured upon warming between 20 and 300 K.

Results
The M (T) (magnetization versus temperature) curves have been used to identify magnetic mineral diagnostic transitions for matter collected in the atmospheric filters (Fig.2). In the RTSIRM curves on cooling (black curves in Fig. 2), a transition near 120 K was determined, identified as the Verwey transition and compatible with the
Were magnetotactic bacteria around in the Precambrian?

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The conventional fossil record spans the recent most ~0.6 Ga, documenting about 15% of evolutionary history on Earth (Knoll et al., 2016). The overwhelming majority of these are products of controlled biomineralization (CBM) by complex, multicellular life forms. Bacteria and Archaea can also biomineralize, but this does not typically occur in a controlled fashion. The only group of bacteria that are capable of CBM are magnetotactic bacteria, who produce iron-bearing nanocrystals in membrane-bound cellular compartments (Uebe and Schuller, 2016). Inside these compartments, magnetotactic bacteria synthesize crystals of magnetite and/or greigite a few tens of nanometers in size, which can be preserved in the sedimentary record as “magnetofossils” (Kopp and Kirschvink, 2008). Metagenomic analyses and molecular dating of deep-branching magnetotactic bacteria phyla have shown that the genes responsible for controlled magnetite biomineralization originated during the Archean, more than 3 Ga ago (Lin et al., 2017).

Samples from Archean and Precambrian rocks found in the Natural History Museum collections were brought to the Institute for Rock Magnetism for preliminary screening for magnetofossil signatures. The focus of this project were samples from ~1.9 Ga rocks in the Lake Superior region, specifically the Gunflint and Biwabik Formations. The main magnetofossil trait that can be determined magnetically is the alignment of magnetofossils in chains. First-order reversal curve (FORC) diagrams are sensitive to non-interacting single domain (SD) particles and undisrupted chains, and were the main tool in identifying samples potentially carrying magnetofossils. In addition, low temperature magnetism was used for diagnosing magnetic mineralogy, using ordering temperatures or phase transitions.

A number of samples were found to contain central ridges in FORC diagrams, indicating the presence of non-interacting SD particles/chains. The most tantalizing results come from a chert specimen from the Gunflint Formation containing magnetite (Fe$_3$O$_4$), iron-manganese carbonates ([Fe,Mn]CO$_3$), and goethite (FeO(OH)). The magnetic signatures of Fe-Mn carbonate and magnetite can be seen in the low temperature remanence curves, where two transitions occur, respectively at ~25-35 K and 120 K (Fig. 1a). Goethite can be gleaned from the separation of the field cooled (FC) and zero-field cooled (ZFC) curves at T>120 K. The FORC diagram of this sample (Fig. 1b) is characterized by a narrow central ridge with a peak coercivity around 60 mT, superimposed on a weak positive background from magnetostatic interactions. Other samples also exhibit central ridges in FORC diagrams, but do not have ‘textbook’ magnetosome signatures. A hematite-bearing jasper specimen from the Biwabik Formation exhibits a sharp central

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ridge with a coercivity tail up to 140 mT and asymmetric lobes characteristic of single vortex (SV) particles (Fig. 2a). A carbonate from the Gunflint Formation shows a central ridge with a bimodal superparamagnetic (SP) – SD distribution and no background features (Fig. 2b).

These samples are currently analyzed using electron microscopy techniques to determine whether the signatures are indeed coming from magnetofossils. If successful, this would extend the fossil record of controlled biomineralization by 1 Ga, and would have a number of implications for magnetotaxis, the origin of biomineralization, the timing of oxygenation of the global ocean, the Precambrian behavior of the magnetic field, etc. The magnetofossil hunt continues!

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Figure 1. Gunflint chert: a) FC-ZFC remanence curves with $\delta$FC/$\delta$ZFC > 2. $T_N$ – Néel temperature; $T_V$ – Verwey transition. b) FORC diagram showing textbook magnetofossil features.

Figure 2. FORC diagrams for the Biwabik jasper (a) and the Gunflint limestone (b).
A list of current research articles dealing with various topics in the physics and chemistry of magnetism is a regular feature of the IRM Quarterly. Articles published in familiar geology and geophysics journals are included; special emphasis is given to current articles from physics, chemistry, and materials-science journals. Most are taken from ISI Web of Knowledge, after which they are subjected to Procrustean culling for this newsletter. An extensive reference list of articles (primarily about rock magnetism, the physics and chemistry of magnetism, and some paleomagnetism) is continually updated at the IRM. This list, with more than 10,000 references, is available free of charge. Your contributions both to the list and to the Current Articles section of the IRM Quarterly are always welcome.

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SANTA IS COMING BACK TO TOWN:
The 2019 Santa Fe Conference on Rock Magnetism is on its way!
The 11th Santa Fe Conference on Rock magnetism will be held at St. John's College in Santa Fe New Mexico from June 6 - 9th 2019.

Following the previous Santa Fe formats, the conference will feature invited talks on selected topics and provide ample space for discussion. This year’s proposed topical sessions will be:

1) Highs and lows of short-term geomagnetic field behavior
2) Pitfalls of Protocols and Processing Procedures
3) Fundamental rock-magnetism
4) Magnetic imaging

On Sunday June 9th there will be an all-day MERRILL workshop on micromagnetic modeling led by Wyn Williams (University of Edinburgh) for those who wish to attend.
An optional field trip will be offered on Thursday June 6th led by John Geissman (UT Dallas) and Mike Petronis (Highlands University, Las Vegas NM) to admire the local geology (and possibly archaeology)

Registration and travel information will be available as we finalize details, (we anticipate early Spring), on the IRM website and the usual email lists. Stay tuned!
2. Survey
The survey contained questions about the instruments available in each laboratory and their typical operating parameters. The survey was circulated to the gpmag, emrp, and latinmag e-mail lists in January and February 2018, and asked respondents to provide information for the following questions:

1. Instruments used for AF demagnetization and imposing of ARMs?
2. What AF ranges (in mT) are possible with these instruments?
3. Which AF range is typically used?
4. What is the frequency of the AF (in Hz)?
5. What DC fields (in mT) are possible?
6. What DC fields are typically used?
7. If AF decay is defined by a decay rate, then what decay rates (in mT/per half cycle) are possible?
8. What decay rates are typically used?
9. If AF decay is defined by a translation speed, then what values are possible?

Figure 1: Possible, typically used, and published values for DC fields, maximum AF, decay rate and AF frequency. NS means the parameter was not specified, or is not known. M indicates that several sets of parameters were used (published studies), or that any decay rate is possible/used in a certain range. Decay rates controlled by translation speed were classified as M.
what translation speeds (in cm/s) are possible?

10. What translation speeds are typically used?

3. Survey results

22 laboratories participated in the survey. ARM and AARM experiments are conducted in 21 of these, and some have multiple instruments available. The most commonly used instruments for ARM and AARM experiments are various models of the 2G Enterprises systems (configured for either in-line, or offline treatment), the ASC Scientific/Precision Instruments D-Tech (for AF/ARM treatment), and the Molspin and Magnon systems. Figure 1 offers a snapshot of the ranges of values that are currently possible within our community, and typically used for DC fields, AF windows, decay rates, and AF frequencies. These values are also compared to experimental parameters reported in AARM literature from 72 studies published between 1985 and 2017.

Maximum possible DC fields vary between 0.1 mT and 1.5 mT, although the most commonly used DC fields are 0.05 mT (i.e. close to the Earth’s field) and 0.1 mT. Note that even in these low bias fields, ARM is not always a linear function of field (Figure 2). The maximum possible AF fields range from 90 to 470 mT. It is interesting to note that – although AF fields up to 470 mT are possible for some instruments in some laboratories, the maximum field used in published AARM studies is 240 mT. Most laboratories report using a maximum AF field of 100 mT. This common range of 0-100 mT is not dictated by the instrumental capability or scientific rationale. Many of the available instruments are capable of applying larger AF fields of 200 mT or 300 mT. However, for many years the Schoenstedt AF demagnetizers, which had a peak field of 100 mT, were very common, and their limitation led to a de facto experimental standard. It is worth noting that it is often desirable to apply ARMs using a smaller peak AF field than the maximum AF field, to ensure complete demagnetization of the laboratory remanence, if needed.

Decay rates can be defined in several different ways depending on the instrument used. Please note that the survey question about decay rates was only answered by 14 out of 21 laboratories employing ARM/AARM systems. There are three general classes of instruments that are used to impart ARMs. The first class of instrument is common on 2G systems with in-line AF/ARM systems and produces a field that simply varies between positive and negative directions for a peak value. The electronic components of this kind of instrument do not control the rate of field decay. Instead, decay rates are determined by the translation speed over which the samples are moved through the alternating field. Translation speeds can be as high as ~20 cm/s, although more commonly used speeds are around 8 - 15 cm/s, chosen depending on the strength of the samples’ magnetization. (Strongly magnetized samples are more likely to cause flux jumps in SQUIDs at higher translation speeds.) A second class of instruments reports the decay rates in mT/half-cycle. Laboratories using the D-Tech instrument have access to decay rates of 0.0001-0.1 mT/half-cycle, and either use a fixed rate of 0.001 or 0.005 mT/half-cycle, or vary the decay rate depending on the AF window. The Molspin offers a smaller range of decay rates, 0.002-0.016 mT/half-cycle, and typically used are 0.004 mT/half-cycle, similar in magnitude to fields used on the D-Tech when they are not varied with AF. The Sapphire Instruments SI-4 has a faster decay, with rates of 5-40 mT/cycle, i.e. 2.5-20 mT/half-cycle, and the rates are adjusted based on the AF window. The third group of instruments reports the decay rate in units of mT/s with values of 5, 10 and 20 mT/s (Magnon), or 1, 1.5, 3 and 9 mT/s (Agico LDA5 & PAM1), with decay rates of 3, 5 and 10 mT/s being used most often. Because the AF frequency is known, these mT/s decay rates can be converted into mT/half-cycle decay rates by DecayedRate [mT/half-cycle] = DecayedRate [mT/s] / (2 * AF Frequency [Hz]). With this conversion, the 5, 10 and 20 mT/s of the Magnon, operating at 105 Hz, translate to 0.024, 0.048 and 0.095 mT/half-cycle, and the 1, 1.5, 3 and 9 mT of the LDA5 & PAM1 operat-
ing at 43 Hz translate to 0.012, 0.017, 0.035, and 0.10 mT/half-cycle. The AF frequencies at which the instruments operate vary by over almost an order of magnitude, from 43 and 300 Hz, and are not always known.

4. Discussion

The survey shows that our community uses a number of different instruments for ARM and AARM experiments. The most frequently used DC field, 0.05 mT, is smaller than the maximum DC field that can be applied in most laboratories, 0.1 mT. Similarly, 100 mT is often used as the upper limit of the AF window, although available instrumentation would allow higher fields. Again, this suggests that the instrument capabilities are not the limiting factors in these experiments.

Decay rates are defined in two profoundly different ways, (1) via the speed with which a sample is moved through a constant-amplitude AF field so that the field strength experienced by the sample depends on its position, or (2) by applying an AF that slowly decreases in intensity over many cycles. In particular, the latter decay rates vary over several orders of magnitude. Some laboratories report using a decay rate of (maximum AF)/(1000 half-cycles). Note that decay rates given in mT/s are not strictly comparable, because the AF frequency has an additional influence.

It is interesting to compare these self-reported values with experimental parameters described in the AARM literature [Biedermann et al., in review]. For example, most published studies report having used DC fields of 0.1 mT, which corresponds to the maximum DC field available in many laboratories. Conversely, the most commonly used DC field by far, according to our survey, is 0.05 mT. We are not sure what the reasons for this discrepancy are, however, some possibilities include the following: (1) The survey respondents may tend to work in different laboratories than the authors of previously published AARM studies. This may reflect different practices, for example, in labs that do a lot of environmental magnetic work versus those that focus more on anisotropy. (2) The standards may have evolved broadly across the whole community, i.e. DC fields on 0.1 mT were used earlier, but nowadays fields of 0.05 mT are more frequently used.

Survey responses agreed well with published studies for the most typical AF window during ARM experiments: 0-100 mT. However, published studies report a larger number of AF windows than our survey. This is most likely related to the different sizes of the datasets. Decay rates and AF frequencies are generally not reported in published studies, and can therefore not be compared to our survey results.

In addition to the parameters discussed above, other details of the experimental setup may cause changes in the measured data, and further work will be needed until we thoroughly understand every factor that influences an ARM measurement. Examples of such possible factors are (1) how the AF is increased to its maximum value prior to the AF decay (rate of increase, shape of increase, whether or not the DC field is on during the increase), (2) how long the sample is exposed to the maximum AF (and whether or not the DC field is on during that time), (3) whether the DC field is turned on at the maximum AF or a slightly lower field, and (4) the shape of AF decay, in addition to the decay rate. The complexity of the physics of ARM acquisition is such that neither analytical models (limited to SD populations, e.g. Egli [2006]), nor micromagnetic models [e.g. Conbhuí et al., 2018] predict ARM response to these factors, and experimental work is required to document the effects and their dependence on other key factors such as particle size and interactions. Additionally, viscosity may affect the measured ARMs.

5. Summary and recommendation

A wide range of parameters are currently being used in (A)ARM experiments, and a universal understanding of how each of these parameters affects the resulting magnetization and its anisotropy has yet to be established. When comparing results from different studies, it is important to be aware that experimental variables can and will affect the reported results. For example, Sagnotti et al. [2003] reported a range of ~75% in ARM intensities acquired on identical PSD samples in different labs, using nominally identical AC and DC fields (AF of 100mT and DC bias field of 0.05 mT. The most important recommendation for our community is to report all experimental parameters in our future studies. While many researchers report the DC bias field and peak AF magnitude, we also encourage researchers to report the frequency of the AF and its decay rate (in mT/half-cycle or the translation speed).

The most suitable set of experimental parameters will depend on the specific goals of any given study. Nevertheless, we present some considerations below that may help in determining the optimal parameters. Larger DC fields lead to stronger ARMs, and therefore also a better signal-to-noise ratio. On the other hand, ARM intensity is not always linear with DC field, and to avoid any problems due to non-linearity, smaller DC fields may be favourable (Fig 2). How the ARM varies with DC field, and whether or not this variation is linear, can be determined experimentally. It has been shown that AARM can vary with DC field [Bilardello and Jackson, 2014]. Therefore, when AARMs are used to anisotropy-correct paleomagnetic data, it is advisable to use a DC field close to the geomagnetic field.

The influence of the AF window on AARMs and ApARMS is related to different sub-populations of grains carrying distinct anisotropies. This can be used in fabric studies, e.g. to characterize different deformation stages. Anisotropy corrections on samples with multiple sub-fabrics are challenging, and several ApARM tensors will be needed, with the AF windows defined e.g. by determining the magnetization directions during an ARM demagnetization.

Decay rates should ideally be chosen based on the maximum AF field and the instrumental limitations of the equipment in a given laboratory. Weaker AF peak fields call for slower decay rates, whereas higher fields
require faster decay rates. (Too slow decay rates in high fields would lead to overheating coils, whereas too fast decay rates in weak fields would be unable to demagnetize the sample completely.) Some instruments automatically change the decay rates as a function of the peak AF designated by a user, e.g. when the decay rate is defined via a fixed translation speed. Other instruments require that decay rates be adjusted manually. Ultimately, we hope that this study will help our community achieve more comparable ARM and AARM results through a higher awareness of the variety of experimental parameters associated with ARMs.

Acknowledgments

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The IRM Quarterly

The Institute for Rock Magnetism is dedicated to providing state-of-the-art facilities and technical expertise free of charge to any interested researcher who applies and is accepted as a Visiting Fellow. Short proposals are accepted semi-annually in spring and fall for work to be done in a 10-day period during the following half year. Shorter, less formal visits are arranged on an individual basis through the Facilities Manager.

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