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Inside...

Seven Secrets to Sample Prep Set Stage for Success!

(or: What to Do Before You Come to the IRM)

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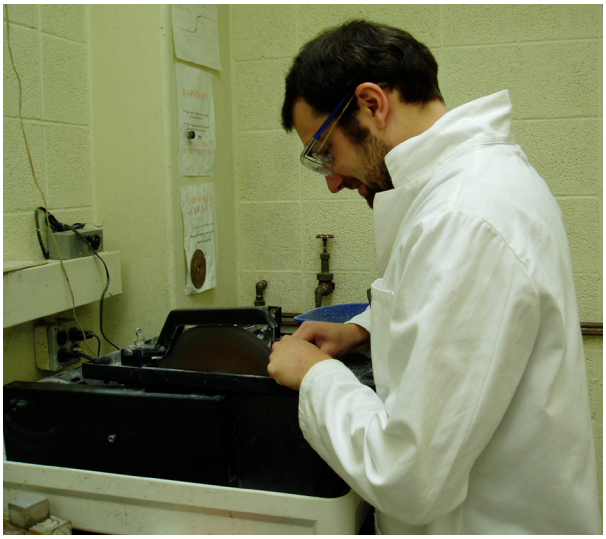
IRM



IRM Postdoc Maxwell Brown uses the high-speed rock saw to prepare basalt cores for paleomagnetic work.

Visitors to the IRM generally find that at least some sample preparation is required prior to many types of measurements. Sometimes this is quite simple (scoop up a bit of sediment and mash it into a gelcap), and sometimes it is more complex (magnetic separation or reduction of an oxidized sample). To make the most of your visit to the IRM, sample preparation should be completed in advance to the extent possible; excessive on-site prep work can eat into valuable instrument time. However, in cases where it is not possible or feasible to complete all preparation in advance, visitors should be aware of what can (and cannot) be done at the IRM. Additionally, while it is possible to do certain types of prep work at the IRM, the amount of time involved may be prohibitive. It is important to let us know in advance if you will have extensive or unusual sample preparation needs.

Previous *Quarterly* articles have described some sample preparation specifications (see v. 6 no. 4 and v. 17. no. 1), mostly regarding sample size and shape. Here, we focus a bit more on the ways and means available at the IRM to get your sample into the proper size and shape, as well as other types of equipment and procedures.

Making small samples out of big samples

Rock saws (high-speed and slow-speed)
Rock crusher
Ball miller

The most frequent sample preparation needs involve manipulating the sample into a size or shape compatible with a particular instrument. The vibrating sample magnetometers (VSMs) when used at room temperature can accommodate a wide variety of sample sizes and shapes, including standard paleomag mini-cores. For high- or low-temperature VSM work, however, sample size is limited to ~4 mm x 4 mm x 2 mm, and for high-temperature work the sample MUST be attached to the ceramic sample mount with a high-temperature cement (provided by the IRM). For work on the Magnetic Properties Measurement System (MPMS), the maximum sample diameter is ~8 mm, but to avoid problems, we recommend staying under 6 mm. A typical MPMS sample configuration involves placing the sample in a pharmaceutical gelcap (~5 mm diameter x 14 mm length) which is in turn inserted into a drinking straw, but other configurations are possible. Sample shape issues are typically only a concern with the "Roly-Poly" anisotropy bridge and when measuring high-field anisotropy on the VSM. Perfect cubes are ideal, but a cylinder with a height/diameter ratio of 0.9 is also acceptable.

For sediments, subsampling is typically fairly straightforward, and we have a wide variety of scoops, spatulas, knives and other instruments for slicing, dicing, and scooping. For hard rocks, we have both a high-speed rock saw and a slow-speed wafering saw. To make small chips, we have a rock crusher as well as an assortment of mortars and pestles. To make a fine powder (e.g. for Mössbauer work), we have a ball miller that can be used in conjunction with crushing/chipping equipment in the main Geology department. However, it is usually simpler to use a mortar and pestle to pulverize the small amount of material typically required.

VSM or MPMS samples need to be firmly immobilized. Common problems when measuring powders or fine-grained sediments include erratic, irreproducible,

cont'd. on pg. 5...

Undergrad Research at the IRM

Most people are aware that many graduate students and post-Ph.D. researchers pass through the IRM each year. Fewer people may recognize that the IRM also hosts a large number of undergraduate students, providing them with access to a large, well-equipped lab and exposure to cadre of friendly researchers with diverse interests. These students come to us in a variety of ways, their visits range from a few days to many months, and they have explored a wide range paleomagnetic and rock magnetic topics.

This summer, two students joined us for 10 weeks as part of intensive summer internship programs. Becky Smith (U.C. Berkeley) participated in the Department of Geology and Geophysics NSF-sponsored summer internship program and did an experimental mineralogy project focused on iron segregation in silicate minerals. Alissa Morson (Carleton College) was a Summer Research Fellow from the Hubert Humphrey Medical Institute and explored the feasibility of correlating volcanic ash units based on magnetic properties. Both Becky and Alissa worked under the supervision of Joshua Feinberg.

Over the past year six University of Minnesota students have worked with IRM faculty, staff, postdocs, and graduate students on a variety of projects. Evan Finnes is currently undertaking a senior thesis project focused on layering in the Duluth Gabbro for which he was awarded funding from the Precambrian Reseach Center at University of Minnesota-Duluth. Charissa Johnson received funding from Sigma Xi to pursue investigations into the magnetic properties of obsidian. Leta Schoeller has been working on paleointensity of samples from the Juan de Fuca Ridge, and Susanna Webb is studying the magnetization of cave sediments. Charissa, Leta and Susanna were all awarded funding through the university's Undergraduate Research Opportunities Program. Joe Cropsey has been assisting with research into different paleointensity techniques, and Ryan Swanson examined spin glass behavior in oxidized magnetite. Evan and Joe are working with Maxwell Brown and Josh Feinberg; Charissa and Susanna are working with Josh Feinberg; Leta is working with Julie Bowles; and Ryan worked with Bruce Moskowitz.

Finally, students from neighboring colleges often come to the IRM to do work for their senior theses or as part of a work-study project. Recently, Maria Princen and Madelyn Mette have visited from Macalester College, and Chelsea Scott from Carleton College.

The work these students do often culminates in a presentation at major professional meeting. Look for them this December at AGU, and welcome them into the greater rock magnetic community!

Current Articles

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 abstracts are taken from INSPEC (© Institution of Electrical Engineers), Geophysical Abstracts in Press (© American Geophysical Union), and The Earth and Planetary Express (© Elsevier Science Publishers, B.V.), 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 Abstracts section of the IRM Quarterly are always welcome.

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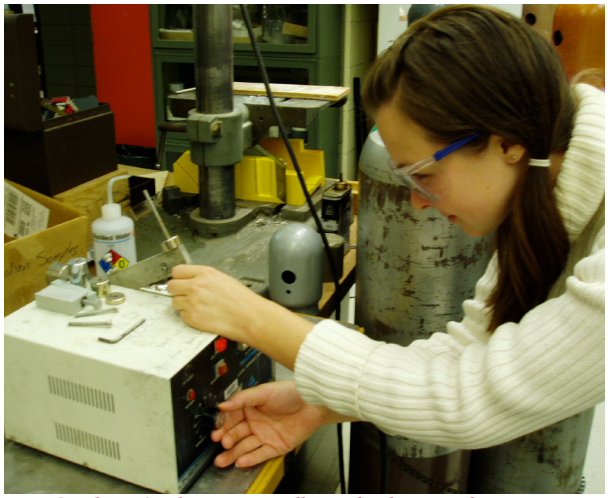

IRM Graduate Student Jessica Till uses the slow-speed saw to prepare a sample for MFM work.

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IRM Visiting Student Haitao Wei demonstrates use of the magnetic separator.

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Other

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IRM Sample Prep, continued from pg. 1

or discontinuous jumps in low-temperature remanence sweeps. These jumps often occur disturbingly close to the Verwey transition of magnetite (120K), and it can be unclear whether they result from particle reorientation, a change in magnetic state, or some combination of the two. To mitigate these problems, gelcaps should be tightly packed full, but we have our little tricks for partially full gelcaps. Immobilizing small rock chips or crystals often requires the addition of foreign material (Kimwipe, quartz wool, calcium fluoride, etc.). This in turn adds new problems, such as magnetic contaminants or difficulty in separating the sample out for later work. People have also experimented with epoxy, wax, crystal bond, glue, gelatine and powdered sugar with varying success; chemical reactions between the sample and some of these immobilizing substrates can be an issue.

Extractions and Separations

Magnetic extractor
Heavy liquid separation capabilities

Visitors working with sediments occasionally find that the ferromagnetic signal is weak either in an absolute sense and/or relative to a dia- or paramagnetic signal. In this case, it may be desirable to concentrate the (ferro)magnetic particles. In other cases, a visitor may be interested in a specific mineral fraction that can be separated based on its density. To these ends, the IRM has a recirculating magnetic extraction system to separate magnetic particles. Potential users should note that the system has a bias in favor of the most magnetic particles (i.e. magnetite will be more efficiently extracted than hematite or goethite, and large magnetite grains will be favored over very fine grains). Depending on the amount of magnetic material present in the original sample, the extraction typically takes 1-2 days per sample. In contrast, heavy liquid separation provides a density-based separation and involves using a low-toxicity heavy liquid (lithium heteropolytungstate; LST) in conjunction with a centrifuge. Again, the process takes 1-2 days, although multiple samples can be processed simultaneously.

Changing the state of a sample: drying, oxidizing, reducing

Drying furnace (100°C)
Gas-mixing furnace (1400°C)
Muffle furnace (1100°C)

Visitors often arrive with wet samples, which in certain cases absolutely must be measured while wet (e.g. magnetosome cultures or other particle suspensions). For such samples, the IRM has non-soluble polycarbonate capsules and special handling procedures.

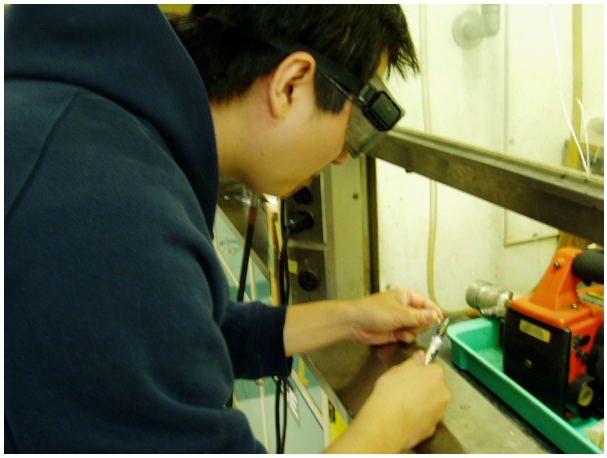
For visitors with wet sediments, there are advantages and disadvantages to measuring wet, and the choice of whether or not to dry may depend on exactly how wet the

samples are. If the sediments contain a significant fraction of water, particles can more easily mobilize and rotate during measurement in both the VSM and MPMS. These rotations can lead to noisy data or seemingly mysterious variations in magnetization. A common observation in low-temperature remanence measurements is a magnetic "transition" at 273K; as water passes through the freezing point, particles rotate, causing a decrease in total moment. This is sufficiently close to the Morin transition in hematite (260K) to sometimes generate ambiguity in interpretation. Finally, to properly measure variations in bulk magnetic properties downcore (for example), samples should be normalized by their dry mass.

It might seem that drying samples in advance of measurement is preferred, but unfortunately dry sediments can also cause problems. If the starting sample is wet but cohesive, particle rotation during measurement can increase after drying the sample, and as mentioned above (*Making Small Samples out of Big Samples*), erratic or discontinuous jumps in data often occur in dry sediments or powders. A rule of thumb might be that if your sample is oozing water and would experience what we might call the "milkshake" effect on the VSM, you may want to dry your samples. Otherwise, it may be wise to leave them wet. Samples can then be dried after measurement for dry-mass re-normalization. If you have questions or concerns about whether or not to dry your samples, please don't hesitate to consult with IRM staff prior to arrival.

Although the IRM has a drying oven that operates at temperatures ≤100°C, if drying prior to measurement we usually recommend drying most sediments at room temperature, as oxidation and alteration can occur even at these low temperatures. This can take hours to days depending on the size of the sample and is preferentially done prior to arrival at the IRM.

Occasionally, a visitor will have a synthetic sample that needs to be oxidized or reduced immediately prior to measurement. For example, a fine-grained magnetite powder can rapidly oxidize during storage in ambient atmospheric conditions, and the IRM has a gas-mixing furnace (T_{max} = 1400°C) that can be used to reduce the sample. Reduction to stoichiometric magnetite usually takes 4-12 hours at 400-600°C in an appropriate CO-CO_2 mix. We also have a muffle furnace that can be used to heat samples up to 1100°C in air.



IRM Graduate Student Yifan Hu prepares a sample mount for high-temperature VSM use.

Making samples pretty

Polishing (and sample mounting) equipment

Microscopes

Domain imaging at the IRM with the magnetic force microscope (MFM) or with Bitter techniques requires a polished sample. While Bitter can be done on a standard petrographic thin section, the MFM requires a sample no bigger than 1 cm in diameter. The polish must be finished with amorphous silica which both produces a smooth surface and removes stress induced by earlier polishing steps. Because an insufficient polish can result in rough topography or stress-dominated domain structures, it is often necessary to re-polish after your first attempt at imaging if non-ideal features are observed. This type of "touch-up" polishing can be done at the IRM. Although it is possible to mount and polish your samples from scratch at the IRM, we typically do not recommend this because the process takes a considerable amount of time away from imaging. Visitors generally find that a lot of time and patience is required to obtain even a single good image, and it is not unusual to spend 8-10 days working on the MFM and come away with only a couple usable images. For this reason we suggest that visitors not waste valuable time on polishing at the IRM if at all possible.

If you do find you need to make use of our polishing equipment, we describe the typical process here. A sample is embedded in a 1" diameter resin cylinder using a hot press. These cylinders (up to 6 at a time) fit in an attachment for a Buehler Ecomet 5 Two-Speed Polisher, which has an automated, rotating head. This apparatus is typically used to polish samples in diamond slurries down to the 1 μm level. To fit in the MFM, the samples must then be cut down on the slow-speed saw to the requisite 1 cm x 1 cm (x 0.5 cm) dimension. The final polish is ac-

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complished on a Buehler Minimet "finger" polisher, which can accommodate one sample at a time and generally requires a bit of babysitting. The entire process will take at least one day, and if you do not have much polishing experience (or are working with new samples), it can be difficult to gauge how much time is required at each step of the polishing process. Samples typically have to be removed frequently from the polisher for examination under a microscope to evaluate progress. The system is not presently set up to easily handle standard thin sections, but it can be done. A slide holder allows the user to manually hold a thin section on the rotating platen, which requires much more user interaction and skill than the automated head.

Other Special Needs

Visitors with needs not described here should not hesitate to ask us. We can usually find a way to make things work for you, but we may need advance notice. In the past, for example, we have been able to provide visitors with access to a glove box in another lab, or to advanced imaging and analytical facilities in the university's Characterization Facility (www.charfac.umn.edu).

Data Preparation

In addition to sample preparation, the first day at the IRM can go much more smoothly if sample information is well organized in advance. Because the IRM uses a centralized, internal database for all data acquisition, a minimal amount of sample information must be uploaded to the database prior to measurement. At a minimum, each specimen (sub-sample) measured must have a unique name and be linked to a sample name. At a maximum, optional metadata includes sample description, mass, volume, composition, oxidation state, site location information, stratigraphic depth, and more. A spreadsheet and instructions can be downloaded from the IRM website so that you may organize your sample information in advance. Upon arrival, it is then a matter of minutes to upload the spreadsheet to the database, and data acquisition can begin. Visitors often start by weighing samples, and the computer-controlled balance will link the mass to your specimen in the database; all data can then easily be mass normalized and exported in consistent units across all instruments. Database use is optional, but most visitors find that it allows for an efficient way of tracking, organizing, and processing data. It also provides tools to simplify and automate preparation of datafiles for uploading to the MagIC database (http://earthref.org/MAGIC/). Remote database access is now available, though in "beta" format. Past visitors who are interested should e-mail us for required software, drivers and password.

Bernard Brunhes
b. 3 July, 1867, Toulouse, France
d. 10 May, 1910, Clermont-Ferrand, France

Eldest son of a physicist father, Bernard Brunhes followed in his father's footsteps to become a professor at the University of Lille and then the University of Dijon in 1897. During his early career, he focused on optics, as well as electricity, acoustics, and thermodynamics. It was not until he was appointed director of the Puy-de-Dôme observatory in Auvergne in 1900 that he became interested in magnetism. The observatory had largely focused only on meteorology until Brunhes decided to broaden the research to include seismology and magnetism. It was in 1905 that Brunhes discovered a magnetization nearly anti-parallel to today's field in the Miocene basaltic lava flows at Pontfarein. Although he suggested in 1906 that they reflected a reversal of the magnetic field, it was more than 50 years before this was widely accepted by the scientific community. In honor of his ground-breaking discovery, the present magnetic polarity epoch is named for Brunhes.

Congratulations to IRM Graduate Student Jessica Till

who was awarded one of two 2010 Mineralogical Society of America Grants for Student Research in Mineralogy and Petrology for her work on

"Rutherford backscattering spectrometry studies of Fe chemical diffusion in plagioclase"

Operator Caffeination

Coffee grinder
Automated drip coffee maker (ADCM)
Espresso machine
Tea pot

Because the attentiveness of the operator is a key variable in the acquisition and interpretation of data, we provide a wide array of equipment to support artificial alertness heightening.

The IRM Quarterly is always available as a full-color pdf online at www.irm.umn.edu

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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.

The *IRM* staff consists of **Subir Banerjee**, Professor/Founding Director; **Bruce Moskowitz**, Professor/Director; **Joshua Feinberg**, Assistant Professor/Associate Director; **Jim Marvin**, Emeritus Scientist; **Mike Jackson**, **Peat Sølheid**, and **Julie Bowles**, Staff Scientists.

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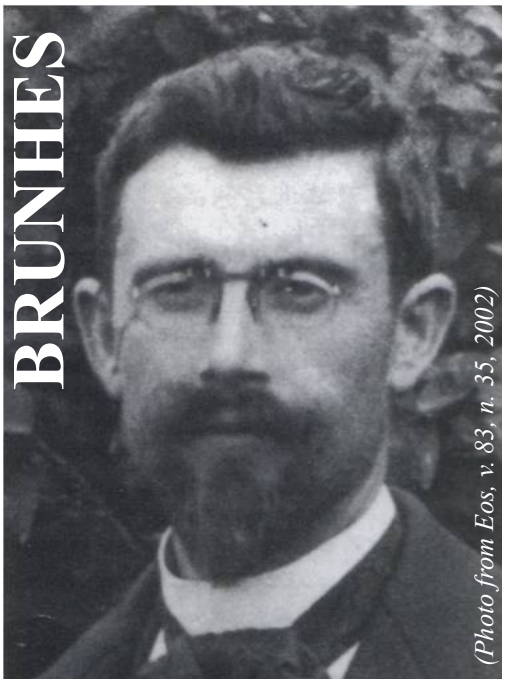

(Photo from Eos, v. 83, n. 35, 2002)

IRM

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