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Electron Holography and Rock Magnetism

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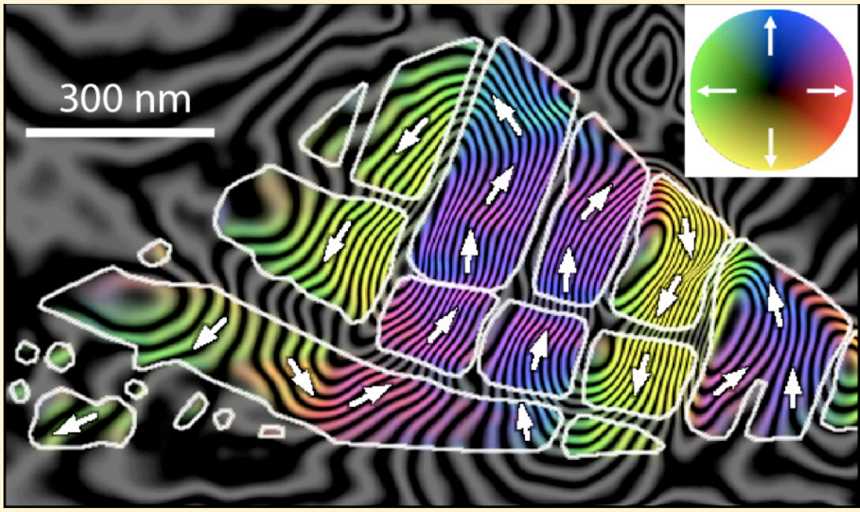

Figure 1--An example of a fully processed electron hologram showing a cross section through a magnetite/ulvöspinel inclusion exsolved in clinopyroxene. This particular image was collected at 89K (-184˚C). White lines indicate the outline of individual magnetite grains. The magnetization in the plane of the image is indicated using contours, colors, and arrows. The hologram shows the magnetic induction within and between magnetite grains, allowing for the study of non-uniform magnetization within individual grains as well as magnetostatic interactions among populations of grains. Data collected April 2005.

Over the last five years a magnetic imaging technique called electron holography has started to be used to image the magnetic flux in geologic materials at nanometer scales (Harrison et al, 2002, 2005; Simpson et al., 2005; Kasama et al., 2006; Feinberg et al., 2006; Harrison et al., 2007). The signature images from these studies are colored, contoured figures showing a sample's magnetic induction and direction (Figure 1). These images are processed versions of data originally collected in a transmission electron microscope (TEM) and contain quantitative information about the magnetization of a sample. The aim of this short article is twofold: to briefly describe the history and procedures intrinsic to electron holography and to encourage others in the rock magnetic community to consider using this technique in their own research by conveying the broad potential the technique has for rock magnetism studies.

History of Electron Holography

Dennis Gabor first described electron holography in 1948 as part of a technique proposed to improve the resolution of TEM images (Gabor, 1948). He envisaged an "electron interference microscope" that would capture interference patterns by overlapping two coherent waves of electrons: a "primary" wave that traveled through a region of vacuum, and a "secondary" electron wave that passed through the sample. His interference patterns were characterized by alternating light and dark fringes produced by constructive and destructive interference of the electron waves. When Gabor's interference patterns were re-illuminated using light similar to the "primary" wave, the interference fringes acted as a diffraction grating and produced a virtual image of the original sample. This image reproduction is possible because the interference fringes record information about both the amplitude and the phase of the electron wave leaving the sample.

Even though this discovery would eventually earn Gabor a Nobel Prize in Physics, the development of electron holography lagged until field emission gun (FEG) electron sources became widely available. FEGs provided a reliable source of bright, coherent electrons and they remain a prerequisite for the successful application of the technique. Research groups specializing in electron holography multiplied throughout the 1980s and 1990s and by 1992 over 20 distinct approaches to electron holography had been devised (Cowley, 1992). The brand of holography most useful to rock magnetic studies is "off-axis electron holography" because it

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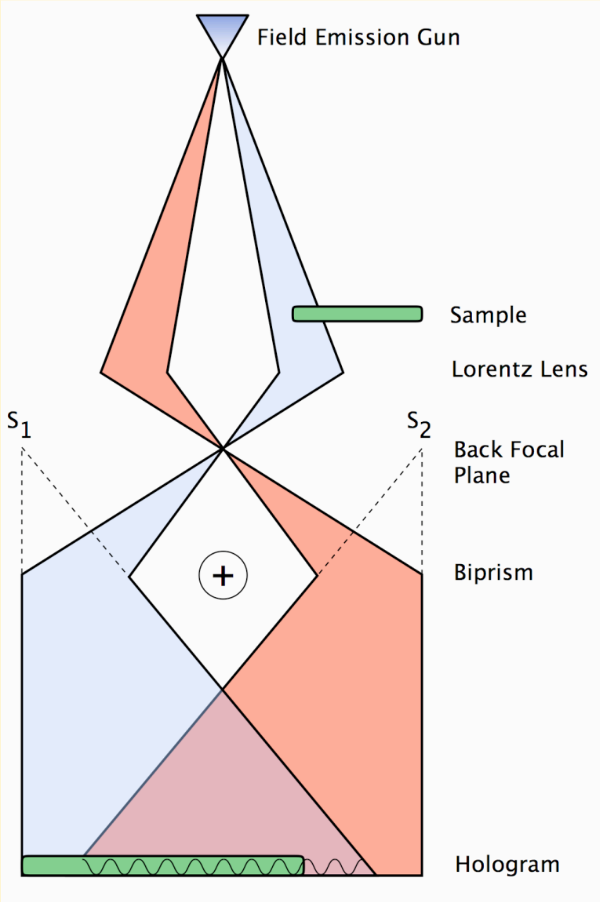

Figure 2--Schematic illustration of the TEM setup used in off-axis electron holography. The sample occupies approximately half the field of view. Critical components are the field emission gun, which provides a source of coherent electron illumination, and the positively charged electron biprism wire, which acts to overlap the sample and (vacuum) reference waves. The Lorentz lens allows imaging of magnetic minerals in a close-to-field-free environment (<0.5 mT). (Adapted from Harrison et al., 2007)

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focuses on isolating the phase shift induced by a magnetic material rather than on producing virtual images. A detailed explanation of the theory of off-axis electron holography requires more space than is available here and readers are urged to explore the excellent reviews of the technique by Dunin-Borkowski & McCartney (2002) and Dunin-Borkowski et al., (2004).

Experimental Design

A schematic figure of the experimental setup for an off-axis electron holography study of magnetic materials is shown in Figure 2. In addition to the FEG, two important hardware modifications are required: the installation of a Lorentz lens and an electrostatic biprism. In conventional TEM practice, the current in an electromagnetic objective lens is used to produce high magnification images of a sample. However, the objective lens is unsuitable for imaging during off-axis electron holography of magnetic materials because it produces a large magnetic field that can alter the magnetic structure in a mineral sample. Instead, a Lorentz lens is installed below the conventional objective lens to allow for work at high magnification while keeping the sample in a relatively field-free environment (<0.5 mT). The objective lens is turned off, or is turned on temporarily to impart fields of up to 2 T to the sample. An electrostatic biprism, typically a gold-coated quartz wire (or platinum wire), is installed in one of the selected-area apertures of the TEM. When a voltage is applied to the biprism (between 50-250 V), portions of the electron beam are deflected so that they overlap (Figure 2). This overlap produces an interference pattern (a hologram), which appears as a typical bright-field TEM image overlain by interference fringes. The broader interference fringes are called Fresnel fringes and are due to diffraction around the edges of the biprism wire, while the fine-scale interference fringes contain information about both the amplitude and the phase shift experienced by the electrons as they pass though the mineral sample. The magnitude of the phase shift is due to factors such as the sample's thickness, its mean inner potential (a measure of a material's ability to slow passing electrons), and its magnetization. Because rock magnetists are primarily interested in the magnetically induced phase shift, the contributions from the sample's thickness and mean inner potential must be removed. Thus, off-line processing is required to extract the magnetic data from a raw holographic image. This processing is made significantly easier if the original holograms are collected on a charged-coupled device (CCD) camera, rather than on photographic film.

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Processing the Holograms

At the University of Cambridge, holograms are digitally processed using a series of routines that run in a software environment named Semper. An example of an unprocessed hologram is shown in Figure 3a. A magnetically induced phase shift can be seen in the fine-scale interference fringes as they are displaced while passing over a maghemite inclusion exsolved in hematite (inset in Figure 3a). An unprocessed hologram from a region of vacuum is shown for comparison in Figure 3b. A Fourier transform of the hologram in 3a is shown in Figure 3c, comprising a central peak, two side bands, and a diagonal streak due to the Fresnel fringes. In order to isolate the phase shift information and remove the portions of the image due to direct transmittance and Fresnel fringes, a "complex image wave" is produced through an inverse Fourier transform of an isolated side

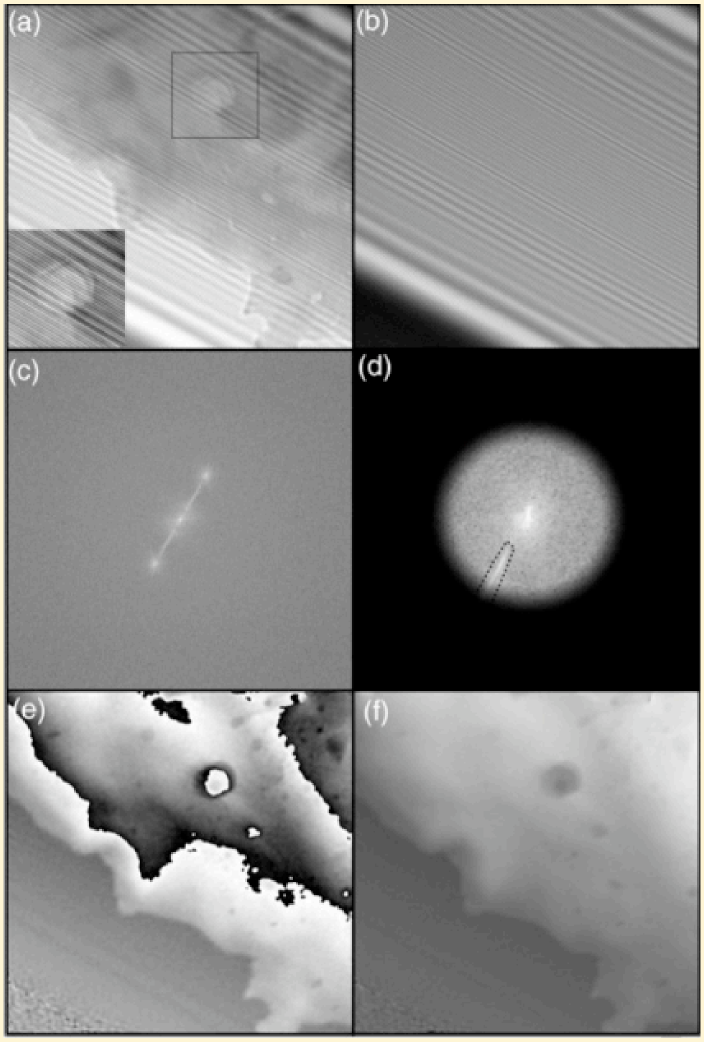

Figure 3--The sequence of image processing steps required to convert an electron hologram into a phase shift image. (a) Original electron hologram of the region of interest (in this instance a maghemite-bearing hematite). Broad Fresnel fringes caused by diffraction around the edge of the biprism wire are visible in the upper right and lower left. The inset is a magnified image of the outlined region, showing the change in position of the fine-scale holographic fringes as they pass through a maghemite inclusion. (b) A reference hologram recorded over a region of vacuum. (c) Fourier transform of the electron hologram shown in (a), comprising a central peak, two side bands, and a diagonal streak due to the Fresnel fringes. (d) A mask is applied to the Fourier transform in (c) in order to isolate one side band. The Fresnel streak is removed by assigning a value of zero to pixels falling inside the region shown by the dashed line. (e) Inverse Fourier transform of (d) yields the complex image wave, which in turn yields a modulo 2π image of the holographic phase shift. (f) Automated phase unwrapping algorithms are used to remove the 2π phase discontinuities from (e) to yield the final phase shift image. (Adapted from Harrison et al., 2007)

band (Figure 3e). The sample's complex image wave is divided by that of the vacuum hologram in order to remove phase shifts caused by heterogeneities in the charge and thickness of the biprism wire. Because the total range of phase shifts across the final complex image wave is greater than 2π, phase shift discontinuities form across the image. These phase discontinuities can be removed (or "unwrapped") using automated image processing routines to yield the final phase shift image (Figure 3f).

The next step is to remove the mean inner potential contribution to the phase shift. If a sample's magnetization can be perfectly reversed (e.g., by saturation in two opposite directions), then the magnetic contribution in the resulting phase shift images will be opposite in sign. Such images can be straightforwardly gathered by using the magnetic field generated by the objective lens to saturate the sample in opposite directions. By adding two reversely magnetized images, the magnetic contribution to the phase shift cancels out, leaving behind twice the mean inner potential contribution. In practice, some samples do not reverse precisely the same way, and the reversal measurements must be repeated multiple times so that nonsystematic differences between reversed images average out. Once the mean inner potential contribution has been determined in this way, it can be removed from the final phase shift images, leaving behind an image of only the magnetically induced phase shift.

Visualizing the resulting phase shift image can be accomplished using contours, colors, and arrows. Contours can be produced by plotting the cosine of the phase shift. The spacing of the contours can be adjusted by multiplying the phase shift by a constant (an "amplification factor") before taking its cosine. For example, the simulated contour plots shown in Figure 4g-i were produced using an amplification factor of four. By calculating the vertical and horizontal derivatives of the phase shift, the direction and magnitude of the projected in-plane magnetic induction can be represented by the hue and intensity of a color. Alternatively, this vector field can be portrayed using arrows.

> *"[E]lectron holography offer[s] us the opportunity to visualize otherwise invisible magnetic structures at unprecedented spatial resolution (~5 nm)."*

One of the strengths of the off-axis electron holography technique is that lines of magnetic flux are imaged quantitatively both within and between magnetic grains. This style of visualization allows researchers to study non-uniform magnetizations within grains as well as magnetostatic interactions among populations of grains. However, like any technique, off-axis electron holography has its limitations. The magnetization of a sample must be relatively strong (>50 kA m-1) in order to discern the magnetic component of the phase shift at a scale of ~10 nm. If such high resolution is not required then it is possible to study more weakly magnetized materials such as hematite or goethite. Additionally, holograms show only the in-plane magnetization of a sample. One highly anticipated development in off-axis electron holography is the development of vector field tomography, which may allow a three-dimensional image of the magnetic induction throughout a sample to be produced.

Additional Advantages

THERE ARE NUMEROUS additional advantages to using electron holography in rock magnetic studies. Recent TEM models allow users to adjust the environment inside the TEM column, so that the conditions under which a mineral acquires its natural remanent magnetization can be reproduced. A sample's temperature can be varied; mixtures of oxidizing or reducing gases can be introduced; and the ambient magnetic field can be carefully controlled. The composition of magnetic minerals can be precisely determined using energy dispersive spectroscopy (EDS) or electron energy loss spectroscopy (EELS). A mineral's orientation and crystal structure can be determined using standard selected-area electron diffraction (SAED) or the more advanced technique of convergent beam electron diffraction (CBED). The distribution of grain shapes and sizes in a TEM specimen can be measured in three-dimensions using electron tomography (Figure 5). There is also the potential to use alternative magnetic imaging modes such as the Frsenel mode of Lorentz microscopy.

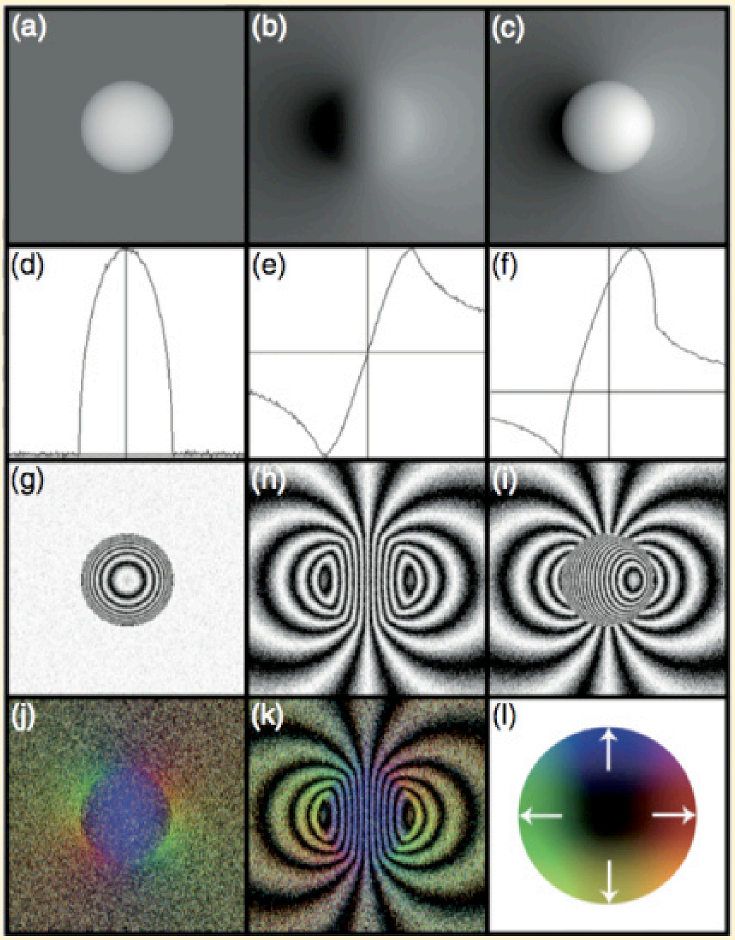

Figure 4--Simulation of the holographic phase shift associated with a 200 nm diameter spherical particle of magnetite. The particle is uniformly magnetized in the vertical direction. The mean inner potential contribution to the phase shift is shown in (a), the magnetic contribution in (b), and the sum of the two is shown in (c). (d-f) Profiles of (a-c), taken horizontally through the center of the particle (normal to the magnetization direction). (g-i) Cosine of 4 times the phase shift shown in (a-c). (j) Color map derived from the slope (gradient) of the magnetic contribution to the phase shift (b). The hue and intensity of the color indicates the direction and magnitude of the integrated in-plane component of magnetic induction, according to the color wheel show in (l). The color can be combined with the contour map (k). (Adapted from Harrison et al., 2007.)

The number of laboratories around the world capable of conducting electron holography studies is increasing. Established laboratories with interests in rock magnetism already exist at the University of Cambridge and Arizona State University. New facilities are being built at the University of Alberta and the Technical University of Denmark and should be operational by late summer of 2007.

Magnetic imaging is advancing quickly. Techniques such as electron holography offer us the opportunity to visualize otherwise invisible magnetic structures at unprecedented spatial resolution (~5 nm). Images at this scale are introducing us to never-before-seen magnetic structures that challenge our existing models of fundamental magnetic phenomena such as exchange coupling and magnetostatic interactions. But as Freddie Mercury once said, "If you see it, darling, then it's there." The magnetic structures seen using electron holography and other imaging techniques such as magnetic force microscopy are ushering in an exciting era, where new micromagnetic models are needed to help understand these latest high-resolution observations.

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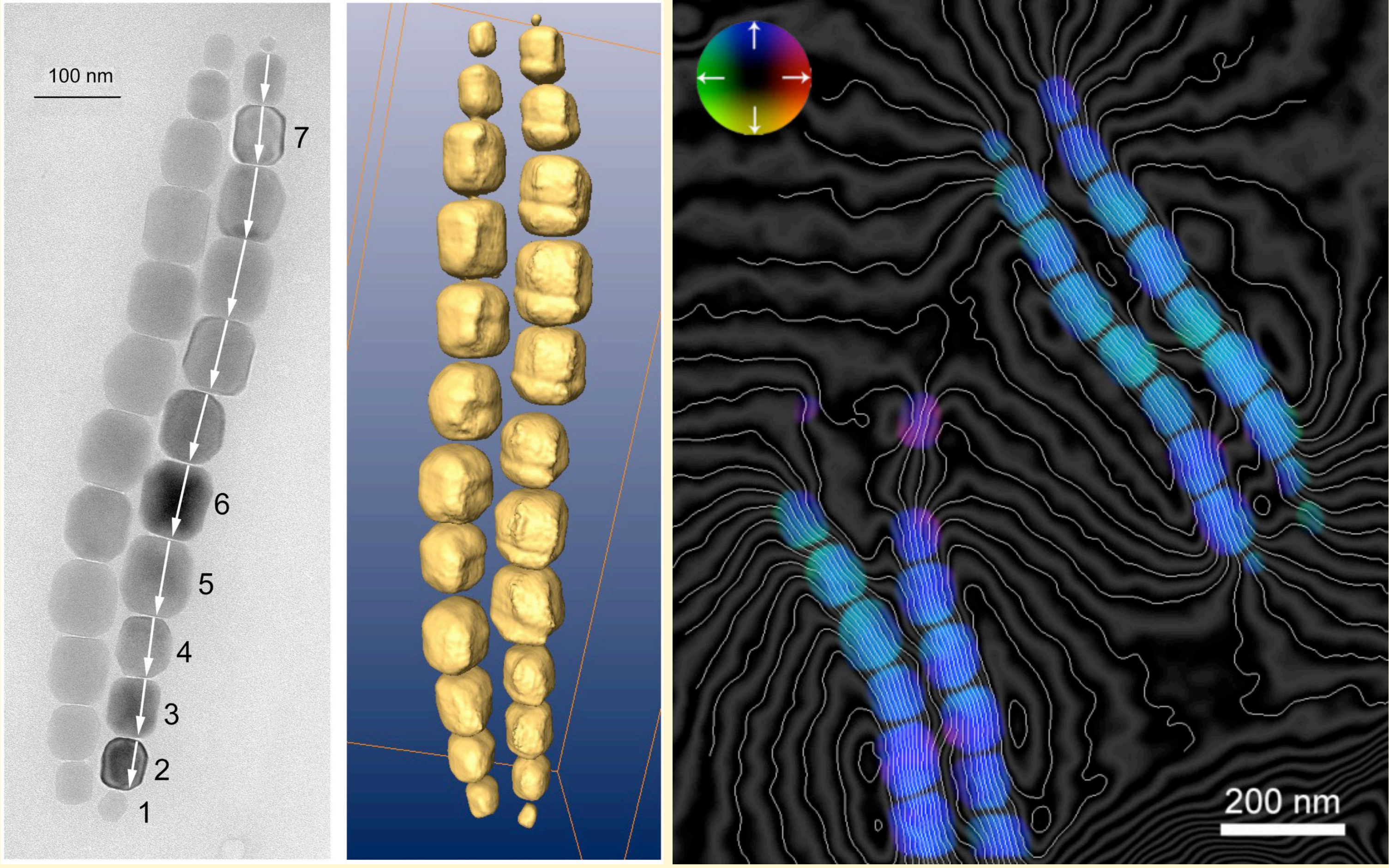

Figure 5--TEM study of magnetotactic bacteria. The image on the left shows a bright-field TEM image of magnetotactic bacteria, where white arrows indicate the approximate orientation of the magnetite [111] axis in each magnetosome. The middle image shows a three-dimensional reconstruction of the same magnetosomes using electron tomography. The image on the right is an electron holography image of bacteria in the same sample. The direction of in-plane magnetization is given by the contours and colors. (See the color wheel inset.) Magnetic interactions between magnetosomes force the final magnetization direction to be parallel to the elongation of the chain. (Adapted from Harrison et al., 2007.)

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New Faculty Member at IRM

Josh Feinberg will be starting as an assistant professor of Geophysics this fall at the University of Minnesota. We, here at IRM, are excited to have Josh join us. Josh's arrival is exciting on two fronts: We are thrilled to begin exploring unfamiliar avenues of research under Josh's lead as a new colleague; and we are equally thrilled to welcome Josh into the IRM fold as an old friend. We are sure that *Quarterly* readers will join us in welcoming and congratulating Josh.