

Magnetic Microscopy Studies of Magnetotactic Fossils on Martian Meteorite ALH84001 and Related Earthbound Analog Systems

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Abstract—Major objectives of this work are to further develop high frequency techniques for use in non-distractive characterization of magnetite crystals found in Martian meteorites and related earthbound simulant analog systems. These techniques are utilized mainly for the complex permittivity, permeability, and conductivity characterization of both solid-state and biological samples. Researchers at the University of Houston have made further progress in the development of three characterization tools: (a) we extended the frequency range up to mm-wavelengths using novel approaches, including an open confocal resonator technique, (b) developed a high-spatial resolution near-field microwave microscopy probe to provide a unique method of characterizing tiny magnetite samples with lateral dimensions of an order of 1 micron, and (c) demonstrated that the ferromagnetic resonance technique (FMR) can provide a rapid, easy-to-use method for detecting signatures of magnetic components of even very small samples. Tiny super paramagnetic particles used in medicine as magnetic resonance contrast agents were employed for these preliminary studies.

MICROWAVE TECHNIQUES OF material characterization have been used in a large number of nondestructive testing (NDT) applications, some of which could potentially be used for astrobiology studies. In particular, microwave microscopy and related techniques could potentially be used to characterize the magnetite crystals found on Martian meteorite ALH84001,^{1,2} proposed to be of biological origin because of their unusual structure. Additional, terrestrial applications range from large-scale remote sensing to detection of tumors or plaque in the human body. In general, these techniques can be classified as far-field, for well-defined resonance electromagnetic wave modes, or near-field, where an evanescent electromagnetic field is used. During the last ten years, considerable interest in such techniques has been expressed by the biophysics community.

Our group, while working on characterization of magnetic, dielectric, and superconducting materials, has designed and fabricated a variety radio frequency (rf), microwave and mm wave resonators in the frame of several different projects.³⁻⁶ These



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include lambda/4 miniature resonators for intravascular plaque characterization; cylindrical TE₀₁₁ copper cavities for microwave spectroscopy and Josephson junctions characterization; normal metal or superconducting enclosure dielectric cavities for magnetic, dielectric, and superconducting materials characterization; coaxial cavities; split resonators for dielectrics; and uniquely sensitive Fabre-Perot (confocal) resonators for microwave and mm-wave characterization. In all these techniques the sample under investigation was placed at the point of maximum amplitude of the high frequency electric or magnetic fields. In addition, high-quality Q-factor, superconducting resonators enable one to measure magnetic susceptibility (for highly insulating samples), dielectric permeability, and complex conductivity, depending on characteristics of the sample, by placing the sample inside the resonator and using the cavity perturbation technique.

Confocal or dielectric resonator techniques are considered far-field techniques, which, while allowing quantitative large area sheet resistance imaging, are limited by wavelength to relatively low resolution (not smaller than 1 mm). Near-field microwave scanning microscopy was originally proposed by Ash and his colleagues in 1972, when the ability to resolve metallic gratings, metallic images, cracks in planar metallic surfaces, and dielectric discontinuities was demonstrated.⁷ Spatial resolution as small as $\lambda/2000$ was shown. In 1987, Gutmann and his colleagues demonstrated the capability of this technique to

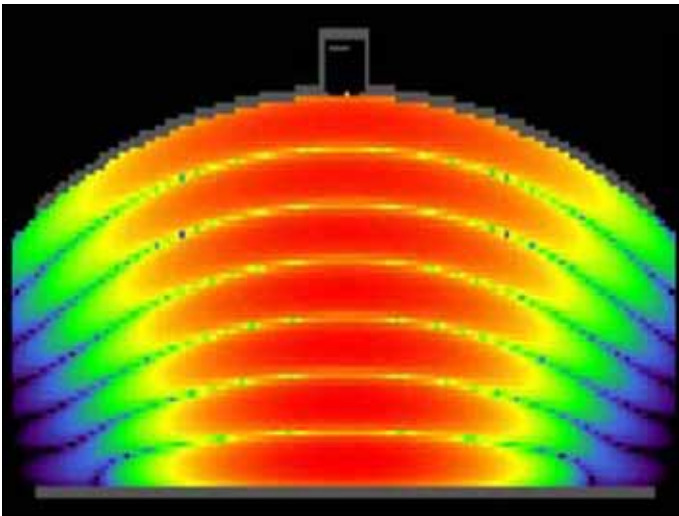


Figure 1. Normalized E-plane 78.163 GHz TEM007 theoretical Ex. It is clear that seven standing half-waves are excited between two confocal mirrors. Red indicates the largest electric field values, and blue indicates the smallest electric field values.

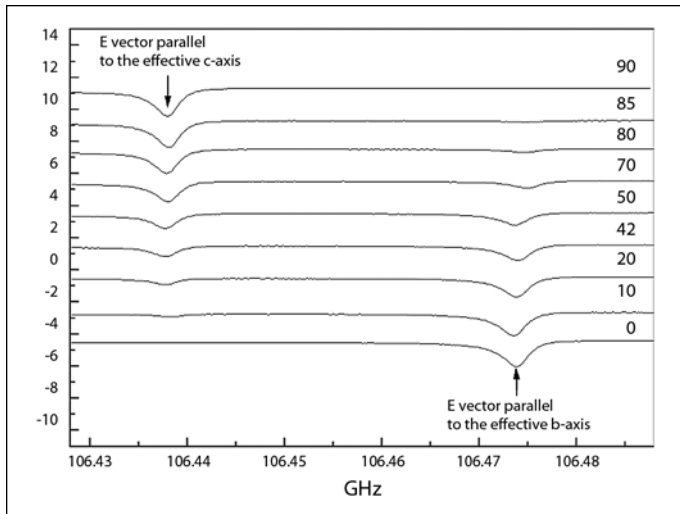


Figure 2. An example is shown of measurements of anisotropic properties of the sample using a brass mirror W-band resonator. A single crystal sample, 0.51 mm thick GaAlO₃, was selected for measurements of reflection response from the resonator versus electric field and material-axes relative-orientation angle (changing from 0 degrees to 90 degrees when the E-vector is parallel and perpendicular to the c-axis, respectively).

measure also depth profiles dopants with a resolution of a few microns.⁸ Currently, because of recent development in near-field microwave microscopy, the resolution better than 1 μm had already been demonstrated,⁹ making microwave NTD very attractive for testing not only the surface of metals and superconductors but also other nonmetallic materials as well as biological samples. In recent years, researchers have published results of biological sample characterization using the

microwave and mm-wave technique. It includes, for example, conductivity of DNA double helix measurements.¹⁰

The objective of this work was to modify our confocal, coaxial line and split resonator techniques to render them specialized for characterization of magnetic and biological samples. (See Fig. 1.) The ultimate goal of this work is to have both hardware and software used in these techniques modified, optimized, and fully tested for measurements related to astrobiology.

Methodology

Fabry-Perot Resonator Technique

The Fabry-Perot or open resonator technique possesses a unique combination of advantages over the more prevalent closed-cavity and parallel-plate resonator methods for materials testing.¹¹ The focused energy allows raster scanning of the sample surface for which effective resolution can be improved using Fast Fourier Transform techniques. By mechanical translation of the substrate, a scanning open resonator can map surface morphology. The open resonator has a relatively sparse mode spectrum. No mode degeneracy, however, at least five to ten distinct W-band (75-110 GHz) fundamental modes, can be probed at each mirror separation distance. Open resonators have a particularly useful physical size at W-band frequencies where pillbox cavities become inconveniently small and wall conductivity losses begin to dominate Q .

A final powerful advantage of an open resonator is that the two mirrors can be thermally independent, enabling each to be constructed of different metals at different temperatures. For example, the planar mirror can be a thin-film HTS at 4.2 or 77 K in a windowed cryostat, while the curved mirror can be constructed of a normal metal at room temperature. More importantly, a curved mirror constructed of a low-temperature superconductor, for instance niobium, at 4.2 K produces a significant Q increase and greatly enhanced resonator sensitivity at W-band frequencies.

Because of the predominantly linear field polarization of TEM_{00q} modes, the open resonator is well-suited to diagnose anisotropic dielectrics and conductors.¹²

In general, for any open resonator TEM_{00q}-mode electric-field vector aligned with a principal axis in the plane of an anisotropic sample (ϵ_{\parallel} aligned, for instance), one resonant mode is observed. As the sample is rotated about the resonator longitudinal axis, a second uncoupled mode with the same mode number q appears at a different frequency and grows in intensity as the relative angle increases, while the intensity of the first mode decreases. At an angle of 90° from the initial orientation (ϵ_{\perp} aligned in this instance), the first mode will have disappeared completely, and the amplitude of the second mode will be at a maximum.

To verify this finding, we measured the unloaded Q and frequency of the brass resonator with a 0.51 mm thick NdGaO₃ substrate inserted. In Fig. 2, confirmation of such behavior is shown.

In addition to the useful anisotropic permittivity measurement technique just discussed, it is expected that an open resonator can be used to measure anisotropic properties of biological samples biased by either electric or magnetic external fields. The sensitivity of this method can be increased even further by replacing a copper enclosure with a superconducting enclosure, while keeping the sample at room temperature.

Microwave Microscopy

A probe for a scanning tip microwave microscope (STMM) has been developed which achieves micron-level resolution combined with high sensitivity. This device will allow the non-destructive testing and analysis of any surface that needs high spatial resolution impedance characterization. It is already known that STMM has potential applications in non-contact, non-destructive imaging of surface resistivity/dielectric constant profiles; screening of material for desired characteristics including conductivity, superconductivity, dielectric properties, and ferroelectricity; ion-implanted wafers and other critical surfaces; and the measurement of protein in microbiology research.

The STMM built in our lab probe consists of a high Q-factor transmission line (with single crystal sapphire dielectric) $\lambda/2$ resonator in which a sharp point tip extends over the inner conductor end. Resistivity or permittivity mapping can be made by measuring the reflection or transmission microwave signal from the probe (Fig. 3b). Areas under development that will use this design include feedback control of tip and sample distance and the integration of STMM with an anti-vibration table. The STMM may also be coupled with external electric and magnetic fields.

For this project, a computer controlled X-Y scanning station was built with 1 micron step motors. The test of both mechanical parts and software was accomplished by imaging a small metal particle x-y scanned inside of a split-dielectric resonator. As a result of measuring shifts of the resonator frequency versus x-y station position, an image of the microwave field distribution was extracted (Fig. 3).

Ferromagnetic Resonance Technique

Ferromagnetic resonance, on the other hand, measures the absorption by spin waves of microwaves incident on a magnetic material. Ferromagnetic resonance (FMR) is a microwave spectroscopy technique for probing the magnetization of ferromagnetic materials. Basics of the FMR methods are very similar to nuclear magnetic resonance (NMR) except that FMR probes the magnetic moment of electrons whereas in NMR the magnetic moment of protons is probed.

The basic setup for an FMR experiment is a microwave resonant cavity placed inside an electromagnet. The resonant cavity is designed to work at a fixed frequency. A detector is placed at the end of the cavity to detect microwaves. The magnetic sample is placed between the poles of the electromagnet, and the dc magnetic field is swept while the absorption of the microwaves

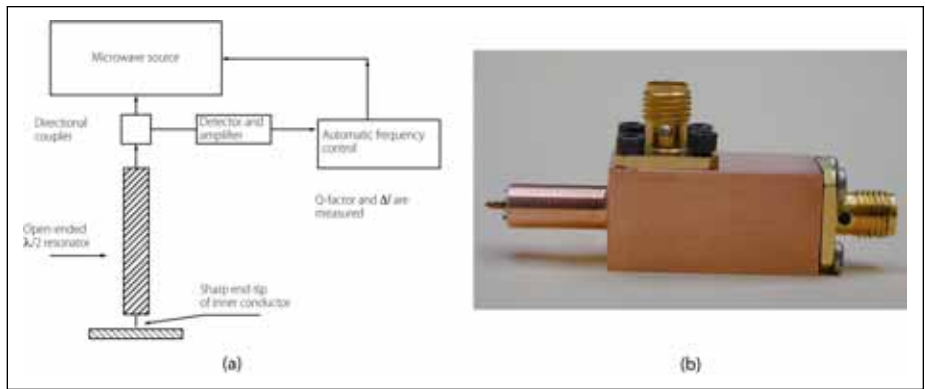


Figure 3. (a) Experimental set-up for near-field microwave microscopy. (b) The photo shows a prototype of a coaxial transmission line probe that UH researchers designed.

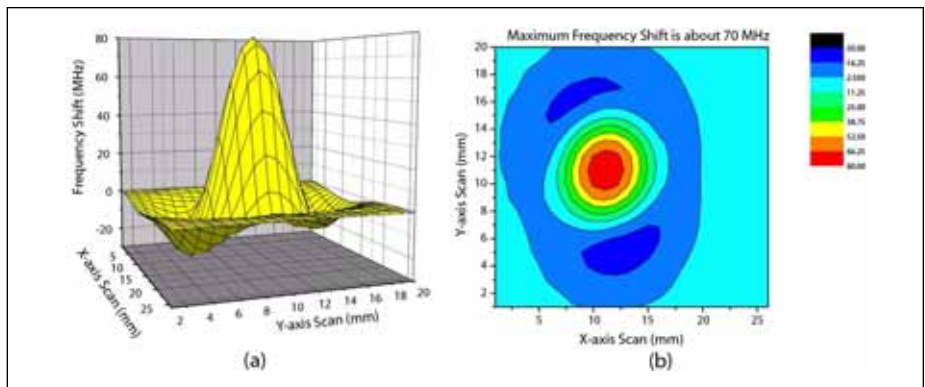


Figure 4. An example of the electromagnetic response of a single dielectric-split resonator to x-y scanning over the whole resonator area with a 0.3 cubic mm of metallic particle. Shown are (a) the shift of the resonant frequency vs. x-y position of the scanning station and (b) a contour map of the frequency shift.

is measured. Typically, it is done as a function of angle, temperature, and applied dc magnetic field. When the precession frequency and the resonant cavity frequency are the same, absorption increases, as indicated by a decrease in intensity in the detector. We have tested this method for sensitivity and its ability to probe magneto-crystalline anisotropy. FMR can identify an anisotropy of the crystallites, which makes it a very powerful method. In experiments which will follow up our current system modifications and testing, we plan to study not only Martian soil simulants provided by our JSC collaborators, including David S. McKay, but we will also check the usability of our techniques for measurements of dielectric and magnetic properties of biological samples.

References

- ¹P. McKay, E. I. Friedman, R. B. Frankel, and D. A. Bazylinski, "Magnetotactic Bacteria on Earth and on Mars," *Astrobiology* 3.2 (2003): 263-70.
- ²B. P. Weiss, S. S. Kim, J. L. Kirschvink, R. E. Kopp, M. Sankaran, A. Kobayashi, and A. Komeili, "Ferromagnetic Resonance and Low-Temperature Magnetic Tests for Biogenic Magnetite," *Earth Planet. Sci. Lett.* 224 (2004): 73-89.

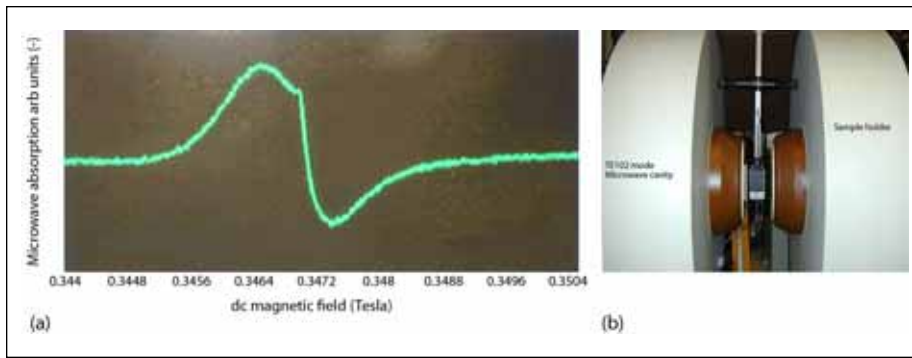


Figure 5. (a) Plots of FMR spectra of superparamagnetic iron oxide nano particles. Anisotropy as well as the evidence of existence of two different phases are seen. (b) Picture of TE₁₀₂, 10 GHz microwave cavity, which was used for the FMR measurements in order to test the sensitivity of the method. The cavity shown here between the poles of the 1 Tesla electromagnet is a part of a Bruker 300 ES spectrometer. The sample is placed inside the cavity in the maximum of the rf field (center of the cavity). This photo shows the end of the quartz rod holding superparamagnetic nanoparticles used for MRI contrast in medical applications.



MEDICAL ADVANCES—Chinmay Darne (*l.*), a doctoral student in electrical and computer engineering, conducts research under the supervision of Dr. Jarek Wosik, research associate professor in the Texas Center for Superconductivity (TCSUH). Mr. Darne completed his baccalaureate studies at Bombay University, India.

³Z. Zhai, C. Kusko, N. Hakim, and S. Sridhar, “Precision Microwave Dielectric and Magnetic Susceptibility Measurements of Correlated Electronic Materials Using Superconducting Cavities,” *Review of Scientific Instruments* 71.8 (2000): 3151-60.

⁴J. Wosik, L.-M. Xie, M. Strikovski, and P. Przyslupski, “Characterization of Ferromagnetic Perovskites for Magnetically Tunable Microwave HTS Resonators,” *J. Appl. Phys.* 91 (2002) 5384-90.

⁵J. Wosik and J. Krupka, “Superconducting Niobium Split-Post Sapphire Resonator for Characterization of Dielectric and Single- and Double-Sided HTS Thin Films,” *Applied Superconductivity Conf.*, Virginia Beach, VA, Sept. 22–29, 2000.

⁶A. Raj, W. S. Holmes, and R. S. Judah, “Wide Bandwidth Measurements of Complex Permittivity of Liquids Using Coplanar Lines,” *IEEE Trans. on Instrumentation and Measurement* 50.4 (2001): 905-09.

⁷J. Ash and G. Nicholls, “Super-Resolution Aperture Scanning Microscope,” *Nature* 237 (1972): 510-12.

⁸R. J. Gutman, S. M. Borrego, P. Chkrabarti, and M.-S. Wang, *IEEE MTT-S Digest* (1987): 281.

⁹M. Golosovski and A. Davidow, “Novel Millimeter-Wave Near-Field Resistivity Microscope,” *Appl. Phys. Lett.* 68 (1996): 1579.

¹⁰P. Tran, B. Alavi, and G. Gruner, “Charge Transport along the Lambda DNA Double-Helix,” *Phys. Rev. Lett.* 85 (1999): 1564-67.

¹¹T. Harrington, J. Wosik, and S. A. Long, “The Field Pattern of the Confocal Resonator for Characterization of Anisotropic Dielectrics and HTS Thin Films,” *IEEE Trans. on Applied Superconductivity* 7.2 (1997).

¹²T. Harrington, “Open Resonators for Superconducting and Dielectric Anisotropy Testing,” University of Houston, 1997.

Presentations

Wosik, J. “Superconducting Receiver Coil Array for Structural MRI of Trabecular Bone for Therapy Response Monitoring,” U. of Pennsylvania Medical School, Nov. 2005.

Wosik, J., M. Kamel, L. Xue, L.-M. Xie, K. Nesteruk, and J. Bankson. “Superconducting Coil Array for Parallel Imaging,” *Proc.*, 13th Annual Meeting of the Intl. Society for Magnetic Resonance in Medicine, Miami, FL, May 7–13, 2005.

Xue, L., M. Kamel, L.-M Xie, J. Wosik, and P. Narayana “SNR Limit for Cryogenic Arrays,” *Proc.*, 13th Annual Meeting of the Intl. Society for Magnetic Resonance in Medicine, Miami, FL, May 7–13, 2005.

Funding and Proposals

Wosik, J. “Structural MRI of Trabecular Bone for Therapy Response Monitoring,” NIH (Solicitation PAR-04-023, Bioengineering Research Partnership, F. Wherli University of Pennsylvania), Sept. 23, 2005–June 30, 2010, \$456,000 for UH; total proposal: \$3,097,000. (*Submitted.*)