

# Final Report “Electrical and mechanical characterization of rocks at the sub-millimeter scale” DE-SC0000757

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## **Abstract**

The broad purpose of CSM’s 6-year (3 years plus renewal) DOE project was to develop and apply new experimental physics technology to the material characterization of rocks at the grain scale or smaller. This is motivated by a knowledge that the bulk chemistry and physics of rocks are strongly influenced by processes occurring at the grain scale: the flow of fluids, cation exchange, the state of cementation of grains, and many more. It may also be possible in some cases to “upscale” or homogenize the mesoscopic properties of rocks in order to directly infer the large-scale properties of formations, but that is not our central goal. Understanding the physics and chemistry at the small scale is. During the first 3 years, most effort was devoted to developing and validating the near-field scanning technology. During the 3 year renewal phase, most effort was focused on applying the technology in the labs Professors Batzle (now deceased) in Geophysics and Prasad in Petroleum engineering.

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# 1 CSM DOE personnel

Rezwanur Rahman got his PhD in Applied Physics under the aegis of this project by developing a method for using cavity perturbation to determine the electrical properties of thin films, rock sections, to observe in real time the effects of minute fluid saturation, and to correlate thermal maturation with directly observable dielectric properties. Nathan Greeney got his PhD in Applied Physics developing the near-field scanning techniques we used to perform laser ultrasound for meso-scale elastic properties as well as the near-field THz techniques that can be used to image the surface of rocks and other materials at much less than a wavelength. In addition Hans Ecke (MS, Geophysics) developed all of the data logging and archiving tools as well as an open source database containing all of our measurements.

## 1.1 DOE sponsored papers from CSM as of 1/1/2016

1. Weiss, M., S. Schafer, B.J. Zadler and J.A. Scales, *Near field millimeter wave microscopy with conical Teflon probes*, Journal of Applied Physics, **106**, 044912:1-8, 2009.
2. Zadler, B.J. and J.A. Scales, *Resolving small changes in elastic moduli due to oriented fracturing*, Journal of Applied Physics, **104**, 023563, 2008.
3. Greeney, N. and J.A. Scales, *Dielectric microscopy with submillimeter resolution*, Applied Physics Letters, **91**, 222909, 2007.
4. Greeney, N.S., K.M. Strovink, J.A. Scales, A.M. Jessop, J. Stuart Bolton, C.C. Watson and D.E. Adams, *Non-contacting transfer of elastic energy into explosive simulants for dynamic property estimation*, Journal of Applied Physics, May 2014.
5. Greeney, N. and J.A. Scales, *Dielectric microscopy with submillimeter resolution*, Applied Physics Letters, **91**, 222909, 2007.
6. Rezwanur Rahman, Tim R. Ohno, P.C. Taylor, John A. Scales *Optically activated sub-millimeter dielectric relaxation in amorphous thin film silicon at room temperature*, Applied Physics Letters May 2014
7. Rezwanur Rahman · P C Taylor · John A Scales *A system for measuring complex dielectric properties of thin films at submillimeter wavelengths using an open hemispherical cavity and a vector network analyzer*, Review of Scientific instruments, , Aug 2013
8. Greeney and J.A. Scales *Non-contacting characterizing the electrical and mechanical properties of rocks at the submillimeter scale*, Applied Physics Letters, **100**, 124105, 2012.

## 2 New Technology

The two basic techniques we use for achieving sub-millimeter resolution are both based on scanning a source and receiver with sub-wavelength step-size control and using energy that directly traverses a thin sample. With a sample that's "thin" relative to a wavelength (sub-millimeter), it is possible to use only energy that is directly propagating across the sample. In practice this means that for our CW THz source we can use the amplitude and phase of the directly propagating energy. On the other hand, to achieve this resolution ultrasonically, we use lasers (thus avoiding

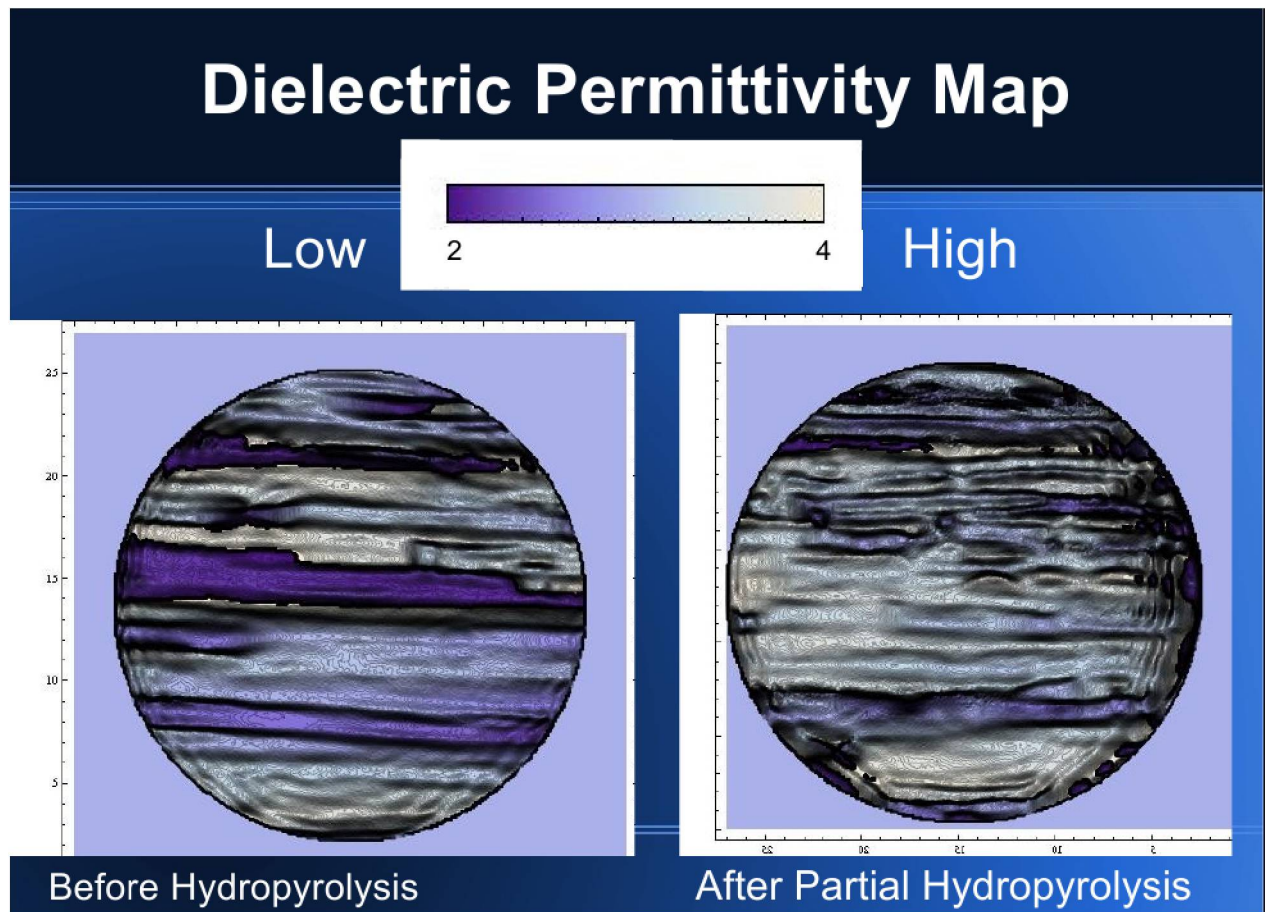


Figure 1: A near field THz scan of an oil shale, before and after pyrolysis. Kerogen/oil has a low dielectric permittivity compared to the host rock, making it relatively easy to image with our THz technique. The samples are 25mm in diameter.

mechanical, contacting transducers). But in this case, since the source is impulsive, it is necessary to carefully window (or time-gate) the data so that only the directly propagating energy is used (i.e., reverberations and mode-converted energy is not used).

## 2.1 Near Field THz Scanning

As described in the papers Weiss et al. (2009) and Greeney and Scales (2007, 2012) our EM scanning technique relies on coupling the propagating THz waves (generated by harmonic multiplication of highly stabilized microwaves) into conical dielectric probes with a sub-wavelength aperture comprising the tip. The rest of the cone is coated with metal to prevent leakage of the EM field. With identical cones on either side of a thin sample, it is easy to scan the rock sample between the two probes and directly measure the complex transfer function at each frequency in some range. Thus we measure the amplitude and phase of both the transmitted field and the reflected field. Examples of such scans are shown in Figure 1. The phase of the transmitted field can be readily converted into an index of refraction; in this case since hydrocarbons have a much lower index than the host rock, it is fairly easy to visualize the spatial distribution of hydrocarbons.

### 3 Near Field Ultrasonic Scanning

One of our key goals was to be able to compare mechanical and electrical properties of samples at the same wavelength. But non-contacting CW sweeps with ultrasound are more difficult to achieve. Non-contacting sources have limited power and while we can still use the laser vibrometer as a receiver, we chose to use an impulsive laser source and time-gate the data for first arrivals. The time gating ensures that only the directly propagating displacement field is used to make inferences about the elastic moduli. The technical details are described in Greeny and Scales (2012).

### 4 Outreach and Technology Transfer

The first part of the project was devoted to the development of the scanning microscopy technology. But after both the THz and the ultrasound system were working and had been thoroughly tested, we sought important applications related to rock physics, in particular.

While pursuing our near-field THz scanning we realized that it would be useful to be able to measure the complex dielectric properties of mounted thin sections and other samples that were too small to be used in our near-field scanning. To that end we were able to build a high-finesse THz cavity. Here the idea is cavity perturbation: namely measure the unloaded cavity resonances, insert a sample and repeat the measurement. We used our Sub-millimeter Wave Network Analyzer, connected via waveguide to a home made 15 cm diamond polished Copper mirror. Rectangular waveguides were electron-discharge milled through the center of the copper and a custom waveguide connector was used.

In cavity perturbation one, in general, needs to know and account for the geometry of the sample. But as described in paper attached below, by measuring a bare substrate as well as a substrate plus film, we are able to cancel all geometrical factors.

The sensitivity of our cavity is such that not only can we measure the complex permittivity of rock thin sections (which exist in abundance waiting to be analyzed), we are even able to see resonance shifts due to a single atomic layer of graphene. The technique drew a great deal of interest from our thin film group so the first application described below is to semi-conductor solar cells.

The details of our cavity perturbation method and a description of the THz experimental system are described in Rahman, Taylor and Scales (2013).

### 5 Conclusions

The success of any project such as this cannot be measured only in the number of papers published or the number of students graduated or even in the technology transfer from the lab to the field. All of these are important. But also important are the new questions asked (as well as the original questions answered). In our case, we have demonstrated

- rapid non-contacting measurement of the spatially varying dielectric constant and elastic modulus of rock samples at length scales much less than a wavelength (either EM or Acoustic). This allows us to make actual maps of these properties. In the EM case, no approximations are made and the method is fully phase sensitive. So the real and imaginary parts of the permittivity or conductivity are directly accessible.

- A sensitive cavity perturbation technique using a high-finesse THz cavity for measuring the complex dielectric properties of rock samples that are too thin or just too small for our scanning system. This has huge application for measuring the dielectric properties of rock thin sections (and subtracting the effects of the substrate).
- the cavity method can be used to study the percolation of minute boluses of fluids as they spread through the sample. We simply keep the sample in the cavity and monitor a particular eigenfunction/frequency over time.
- Scanning of samples in pressure vessels to study processes such as hydro-pyrolysis. We have demonstrated that it is possible to follow the movement of hydrocarbons in real time.