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Application Note
Measuring Calibrated Magneto-electric Samples
Rev A

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Introduction

The Vision Library for Radiant's non-linear materials testers offers the Magneto-electric Response Task to measure the magneto-electric coupling coefficient in multiferroic materials and composite magneto-piezoelectric devices. The test stimulates a sample with a small AC magnetic field while measuring its charge generation. Provisions are available to apply a background magnetic bias field to the sample in addition to the AC stimulus. Figure 1 diagrams the test configuration for measuring magneto-electric properties with a Precision tester. The sample is electrically biased at zero volts in Figure 1.

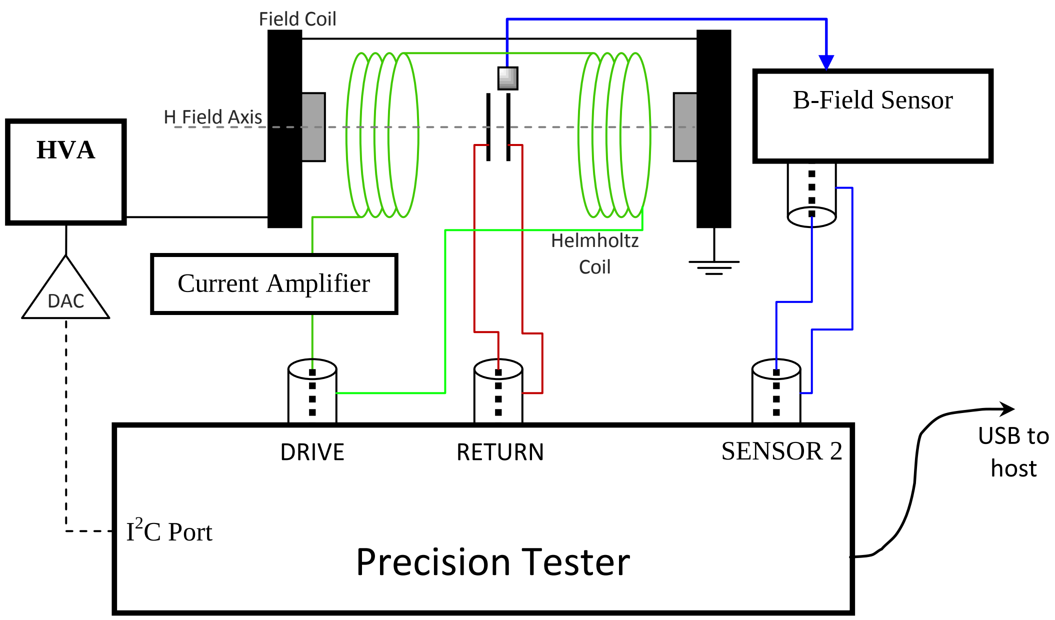

Fig. 1: Measuring magneto-electric response with a bias magnetic field.

Dr. Chee-sung Park, then at the Center for Energy Harvesting Materials and Systems (CEHMS) at Virginia Tech, manually executed this Charge vs. B-field measurement in 2010 on a known sample using test elements in the Vision Library running on a Precision Premier II test system. Exporting the results and performing the proper mathematics on the data, he was able to show that the results from the Charge vs. B-field test were identical to the results of the traditional Voltage vs. B-field test. His experiment was published in the *Journal of Smart Materials and Structures* [Smart Mater. Struct. **20** (2011) 082001 (6pp)].

Radiant created the MR Task to execute automatically that same Charge vs. B-field measurement of magneto-electric devices under Vision's control. The purpose of this application note is to replicate Dr. Park's experiment in the opposite direction: use the MR Task to measure the Charge vs. B-field for calibrated ME samples provided by CEHMS, mathematically convert the results to the Voltage vs. B-field format and compare the predicted ME coefficient to that measured by CEHMS on the calibrated samples.

Theory:
The magneto-electric coupling coefficient causes a capacitor to generate a voltage or a charge or a combination of the two when the sample is exposed to a magnetic field. The process can be thought of as the sample storing some of the energy of the magnetic field in its lattice, causing a change in its lattice parameters and consequently changing the associated electrical field balance associated with that lattice. All of the useful non-linear materials environmental responses can ultimately be traced to changes in the lattice:

1) Dielectric response - Otherwise known as electrical capacitance, this is the change in the lattice charge balance as the result of an external electric field applying a force to the lattice.

2) Piezoelectric response - The change in the lattice arrangement due to the application of an external force.

3) Pyroelectric response - The change in the lattice arrangement due to a change in the temperature of the lattice.

4) Magneto-electric response - The change in the lattice arrangement due to the application of a force to the lattice from an external magnetic field. This property requires that the lattice have some magnetic properties in order for the external magnetic field to apply force to atoms in the lattice.

A lattice in steady state with no external forces applied to it will nominally emanate no electric field. Electrodes on opposing surfaces of the lattice will be neutral electrically. A change of the energy state of an asymmetrical lattice will cause a net electric field to emanate into the electrodes. If the electrodes are open-circuit, the resulting voltage will be measureable by external instruments. This approach is the traditional test for the magneto-electric coefficient. It is defined as the change in the voltage across the sample with the change of the applied magnetic field.

$$\alpha_{ME} = \delta E / \delta H \qquad \text{Eq (1)}$$

Eq (1) above is Eq (2) in Dr. Park's referenced paper. If the electrodes are shorted together, no voltage can occur between the electrodes. Instead, current will flow between the two electrodes, producing excess charge on each electrode of the proper polarity and amplitude to cancel the electric field emanating from the lattice. Dr. Park provides this relationship as Eq (1) in his paper

$$\alpha = P/H \qquad \text{Eq (2)}$$

The relationship between α and α_{ME} is given by Dr. Park as

$$\alpha_{ME} = \alpha / \varepsilon_0\varepsilon_r \qquad \text{Eq (3)}$$

In summary, a capacitor has a net polarization state in equilibrium with any external magnetic field (α) and that net polarization is converted to a voltage across the capacitor (α_{ME}) by the dielectric constant of the capacitor material. *The magneto-electric coefficient for the sample may be determined by measuring either the open circuit voltage across the sample or the net charge transfer between the capacitor plates of the sample when an external magnetic field is applied.*

The measured response of the magneto-electric sample to an applied magnetic field will be determined by 1) the value of the magneto-electric coupling coefficient, 2) the electrical capacitance of the sample, and 3) electrical load across the sample. If the sample electrodes are open circuit, the dielectric constant of the sample will create a voltage across the sample. If the sample electrodes are shorted together, current will flow between the electrodes as the magnetic field changes to maintain a zero voltage difference between the electrodes. If an intermediate impedance is placed between the two electrodes, the end result will be the same as if the electrodes are shorted but a temporary voltage will develop across the impedance while the current is flowing between the plates. If the magneto-electric coefficient and the dielectric constant are both constant, the relationship will be linear as shown in Figure 1.

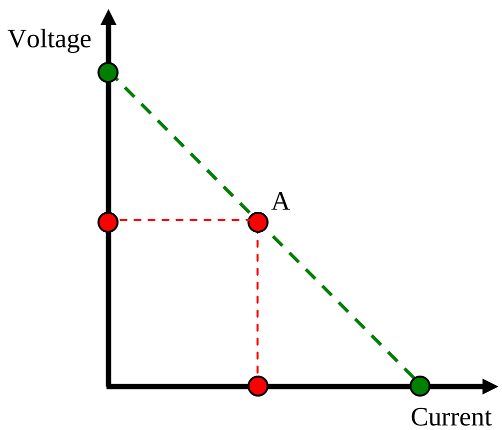

Figure 2: Tradeoff of voltage and current between sample electrodes as a magnetic field changes for a fixed time period after the magnetic field changes.

There are several complications to the simple relationship diagrammed in Figure 2. First, the magneto-electric coefficient and/or the dielectric constant of the material may not be constant with magnetic field. The dielectric constant did change with the magnetic field in the samples that Dr. Park measured and he had to characterize this change in order to properly compare the voltage-based and charge-based measurement techniques. (See Figure 4B of the Park. Et all paper.) For such a sample, the linear Voltage/Current trade-off shown in Figure 1 would acquire a non-linear shape.

A second source of complexity in the magneto-electric measurement arises from dynamics. Point A in Figure 2 represents the response of the sample with an intermediate impedance between its plates. The value of the impedance determines the amount of voltage produced across the sample as the applied magnetic field changes but the value of the impedance is itself determined by the test frequency. If the test frequency is slow enough, the impedance will reduce to zero. This effect puts a floor on the slowest frequency that can be used to execute a voltage-based magneto-electric test. *Since all magneto-electric samples have leakage between electrodes through their dielectric material, all voltage-based magneto-*

electric tests have a lower frequency test floor. Another consideration is that the capacitance of the cables connecting the sample to the test instrumentation will be in parallel with the capacitance of the magneto-electric sample and must be charged by the magneto-electric response of the sample. This effect will reduce the measured voltage across the sample resulting in an artificially low value for the magneto-electric coefficient. The magnitude of the error will be related to the ratio of the sample capacitance to the capacitance of the test fixture cables.

Charge based measurements can be made with a Sawyer-Tower circuit but suffer the same parasitic losses mentioned above. Sensitive charge-measurement systems like the Radiant testers use a virtual ground input. Both the parasitic capacitance and the parasitic leakage mentioned above require a voltage to activate them so by maintaining zero volts across the sample during a test, the parasitic effects are eliminated. The Radiant test systems also have a slow frequency test floor determined by the ability of their charge integrator circuits to accurately hold onto small amounts of charge. The slow frequency test limit of the Radiant Premier II or Multiferroic testers will is set at 1 Hz to guarantee a 2% maximum charge loss for a 10 pC test over one second. (See the application note "Application_Note_-_Measuring_Calibrated_ME_Samples_with_the_MR_Task.pdf".) This test frequency limit is more conservative than the 30 second specification given for Premier II or Multiferroic testers when executing hysteresis loops. The reason is that the typical magneto-electric measurement may generate 10 pC or less – a value much smaller than the typical hysteresis loop.

Effect of Coil Inductance on Test Frequency:

The Premier II and Multiferroic testers can make measurements as fast as 250 kHz. However, it is highly unlikely that the current amplifier shown in Figure 1will be able to go much faster than 10 Hz. The reason relates to the voltage limit of the current amplifier. During a test, the current amplifier delivers into the coil the current specified by the DRIVE output of the tester. To maintain that current, the amplifier must overcome the back voltage generated across the magnetic coil it is driving according to the equation:

$$V_{Coil} = L\ \delta I/\delta t \qquad \text{Eq(4)}$$

where L is the inductance of the coil. The coil requires a specific current to generate a specific magnetic field but the voltage across the coil is a function of *the rate of change of current*. That rate of change increases linearly with test frequency. When the back voltage generated by the coil equals the maximum voltage the amplifier can generate, the test cannot be run any faster while still generating the specified magnetic field. It is best to stay away from maximum limits during tests. A typical electrical engineering rule of thumb is a factor of two. Therefore, typical Helmholtz coil installations hooked to typical current amplifiers will not be able to go much above 20 Hz without severe distortion of driving magnetic field.

Noise:

An important distinction occurs between the measurement of electrical hysteresis and magneto-electric response. For electrical hysteresis, it is possible to use coaxial cable from the tester BNC all the way to the sample. Given the virtual ground on the RETURN BNC, the shielded coaxial cable has no impact on the test results. That is not true for any non-shielded wire that extends beyond the shield protection of the coaxial cable. It will act as an antenna, causing electrons to move along the wire with any change of local EMF, no matter how small. Since the tester measures the electrical hysteresis *by counting electrons that move into and out of the virtual ground at the RETURN BNC*, this EMF noise will show up in the measurements. EMF noise comes from power supplies and fluorescent lights in the room. Most samples, such as a thin PZT capacitor only 100 microns on a side, generate so much charge that it swamps external EMF noise sources. Small area capacitors that generate polarization on the same order as does EMF noise must be placed in a shielded box grounded to the shield of the RETURN BNC connector. As noted earlier, most magneto-electric measurements will generate charges on the order of 1 pC to 100 pC. Placing the sample in a non-magnetic aluminum or brass box and grounding that box to the tester will greatly reduce the impact of EMF noise on the measurement results.

A second method by which noise will couple into the measurement is through the magneto-electric coupling coefficient itself. The EMF, usually varying at 50 Hz or 60 Hz, will couple into the Helmholtz coil directly, causing a systematic variation of the current in the coil. This translates into variations of the magnetic field the coil generates and consequently a direct modulation of the magnetic stimulation of the sample under test. The efficiency of this coupling has yet to be explored under controlled test conditions for its potential effect on the MR Task.

A third source of EMF noise arises from the static electric field that surrounds everything everywhere. Since it is static, it should have no impact on a test. However, a person walking near to the Helmholtz coil while a test is being executed will causes changes in the local electric field around the coil that will couple into the test results by both paths described above. Normally, for large samples generating a large charge signal, this EMF effect should be small or even unnoticeable. Tests capturing small, picocoulomb-scale charge levels may be affected.

The electrical noise across the sample in the shield box in the test fixture is in Figure 5 below. The peak electrical noise is 250 fC peak-to-peak.

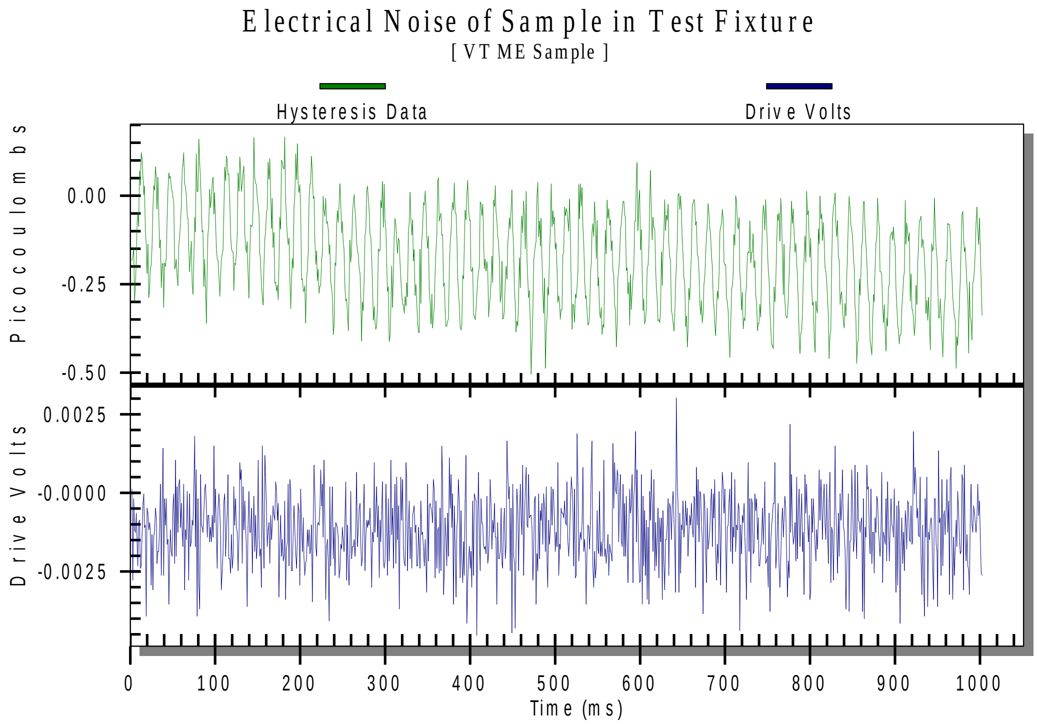

Figure 3: Electrical noise of shielded sample in test fixture of Figure 5.

Calibrating the Magnetic Field:
The purpose of this experiment is to determine the accuracy of the magneto-electric measurement using all of the possible test configurations. The final accuracy number will be a combination of the accuracy of the charge measurement, specified as 2% for the MR Task run on the Premier II or Multiferroic testers, and the accuracy with which the magnetic field across the sample is known. This second subject is covered in detail in the application note from Radiant Technologies named "Application Note - Calibrating the Magnetic Field for ME Testing.pdf". Please review it carefully before conducting any tests with the MR Task.

Test Configuration:
The test configuration will be as follows:

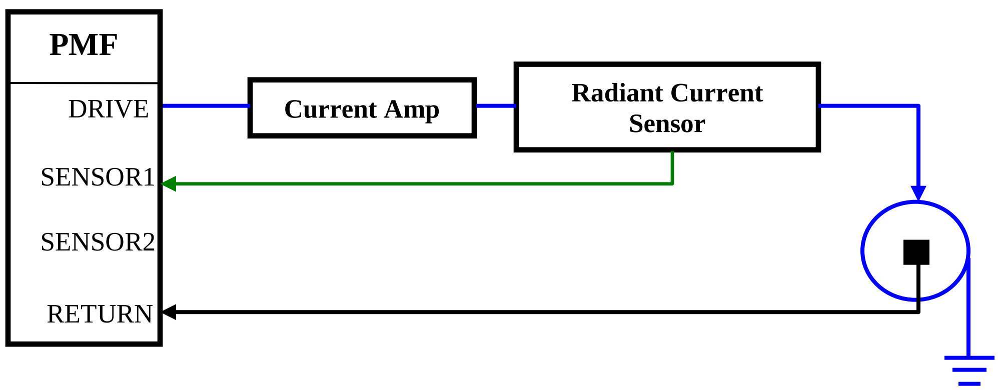

Figure 4: Test configuration for this experiment.

The Lakeshore gaussmeter was the best choice for measuring the magnetic field during the execution of MR Tasks but it was not possible to position its Hall Effect sensor next to the sample in the test fixture. The Radiant current Sensor RCSh proved accurate enough to use it for the magnetic field mapping during execution of the MR Task in place of the gaussmeter.

The result expected from this experiment is a comparison of the α_{ME} measured by Virginia Tech of its samples and that determined by the MR Task.

Measuring the Small Signal Capacitance vs. Magnetic Field:
The small signal capacitance of the sample at different magnetic fields can be measured by the Radiant testers. It requires a change in the test configuration to do so. The DRIVE output must be connected to the sample and a Radiant I^2C DAC must be connected to the current amplifier. The procedure for making this test is described in the application note "Application Note - Measuring Small Signal Capacitance vs H-field.pdf".

Results:
A photograph of the test configuration, including a shield box for the sample is shown in Figure 5.

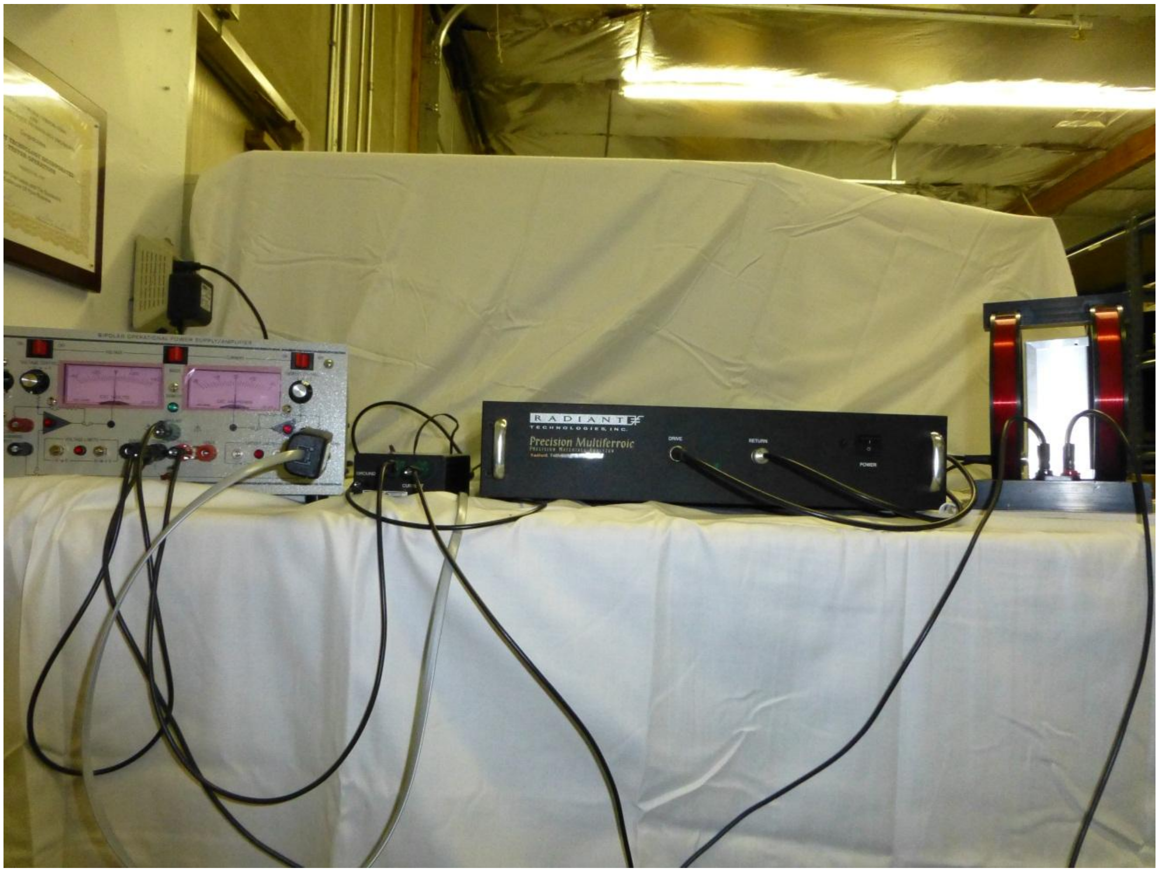

Figure 5: The test configuration.

The Helmholtz coil is a Lakeshore MH-6 with a 6 inch diameter. The current amplifier is a Kepco 36-6 unit capable of 36 amps up to 20 volts. The Gaussmeter is a Lakeshore Model 425 with a Hall Effect sensor. The current sensor in-line with the Helmholtz coil is a Radiant RCSh which uses a Hall-effect sensor to measure the current. Because of its internal Hall-effect sensor, the RCSh must be placed away from the Helmholtz coil during measurements. As an alternative, the Radiant RCSi current sensor uses an instrumentation amplifier to measure the current. It has more resolution than the RCSh but is only isolated to 200 V while the RCSh is isolated to 1500 V. The RCSh was used for this experiment.

The shield box is aluminum and non-magnetic. The coax cables go into the box to connect to a small PC board holding the sample. The BNC connectors have their shields connected to the box metal so connecting coax cables to the connectors will automatically connect the outside box to the ground shields of the tester connectors. A non-magnetic battery holder conveniently holds the sample. The shield box is attached to a wooden base to place the sample at the correct height to be at the central axis of the Helmholtz coil.

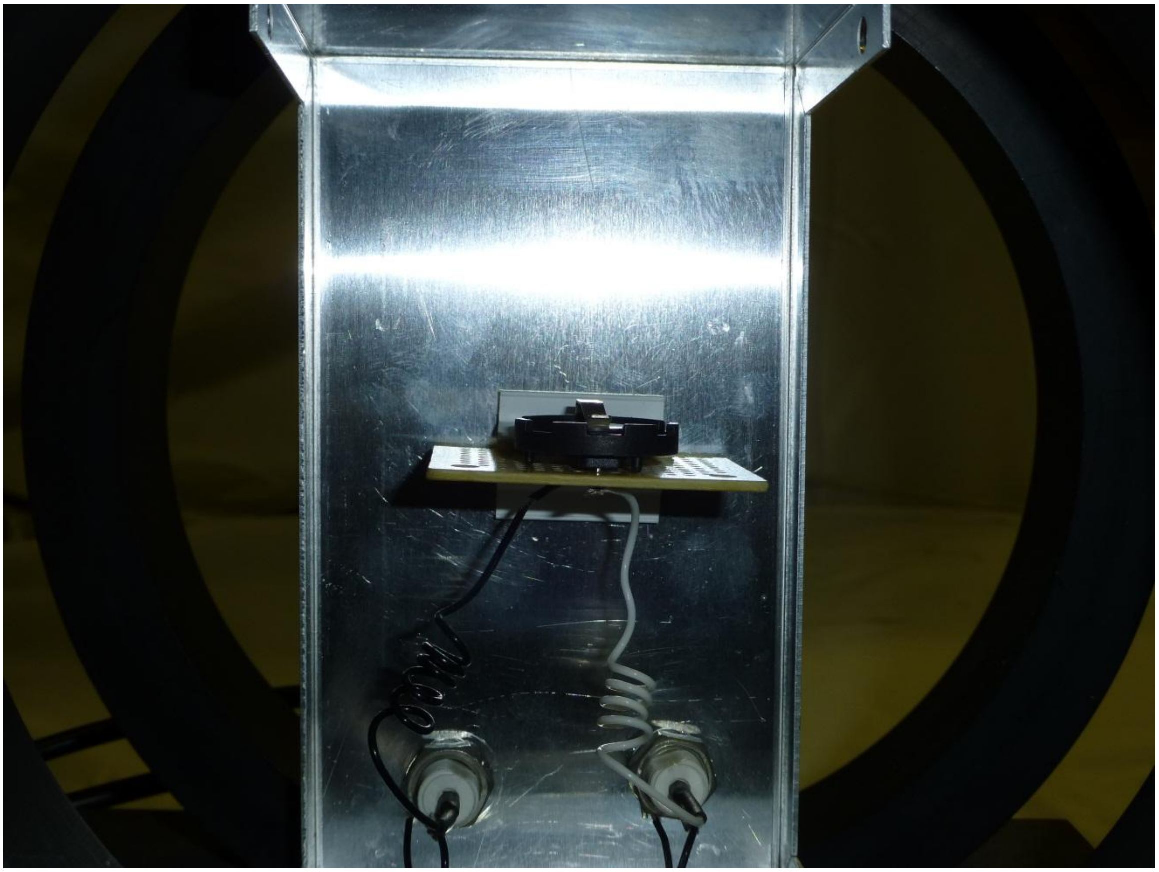

Figure 6: The sample shield box.

Technically, since one side of the sample is grounded during the MR Task execution, two BNCs are not needed on the shield box. The second BNC allows us to execute Polarization Hysteresis vs. Magnetic Field and Small Signal Capacitance vs. Voltage tests on the sample since both tests require the DRIVE and RETURN to be connected to the sample.

The sample was fabricated by Mr. Su Chul Yang and Mr. Shashaank Gupta of Virginia Tech's Center for Energy Harvesting Materials and Systems (CEHMS). It consists of KNNLS-NZF cut into disks with a diameter of 0.785 cm^2 and a thickness of 0.05 cm. The disks are bonded to a nickel foil.

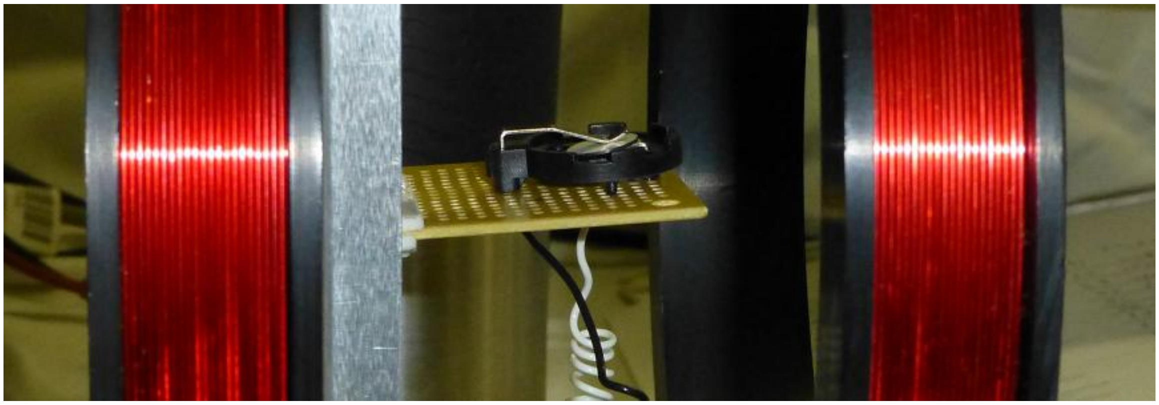

Figure 7: The sample in the shield box sample holder.

This sample is a “composite” magneto-electric sample physically attaching a ferromagnetic material to a piezoelectric ceramic. A magnetic field applies a force to the ferromagnetic material which in turn creates stress changes in the piezoelectric material to which it is bonded. Although it is physically different than a true multiferroic material, the outcome is the same: an applied magnetic field creates a voltage across the sample or charge flow from the sample.

The experiment will consist of four measurements.

1) The first will be a dry run of the MR Task with no sample inserted into the sample holder. The sample holder will be shorted. This test will evaluate the amount of current generated in the RETURN cable that enters the active area of the Helmholtz coil. If the current is significant, this data will be used to correct the actual measurement for this parasitic contribution. See the application note “Summary of the Magneto-electric Response Task.pdf” from Radiant for more details.

2) The second will be the MR Task measurement of the sample itself. There will be three measurements.

    - ±45.0 (Oe) at 0.0 (Oe) bias.
    - ±45.0 (Oe) at +56.0 (Oe) fixed bias.
    - ±45.0 (Oe) at -56.0 (Oe) fixed bias.

3) The third step will be a zero-field measurement of the MR Task to determine the noise coupling into the sample from external sources.

4) The final measurement will be the Capacitance vs. H-field Test Definition described earlier.

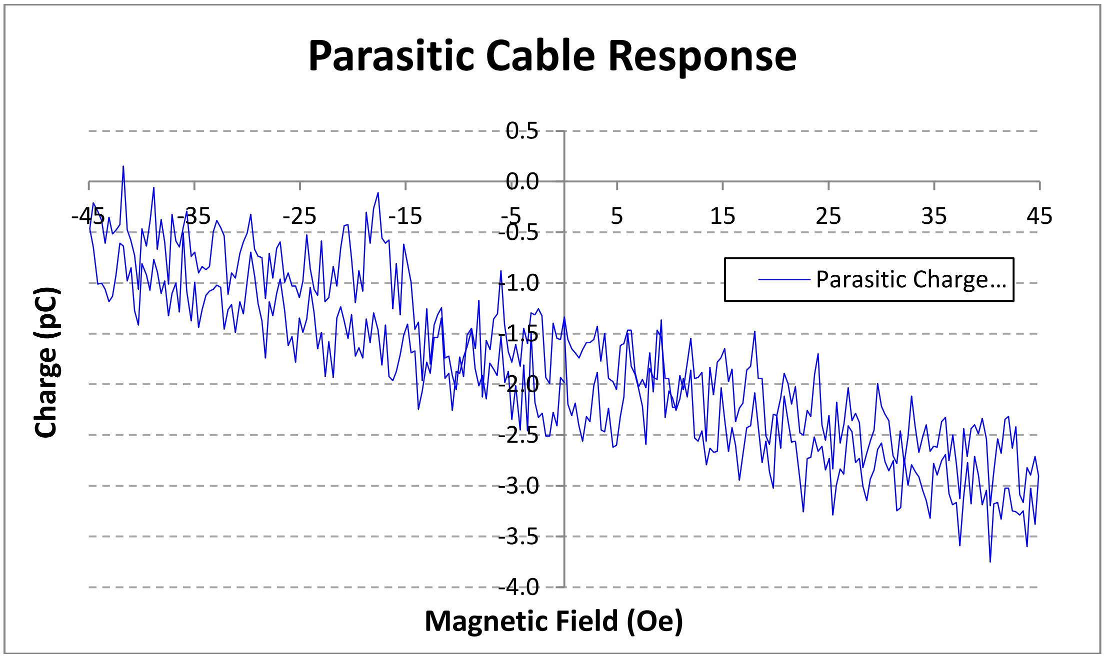

Figure 8: Magnetically induced current in the sample cables with no sample.

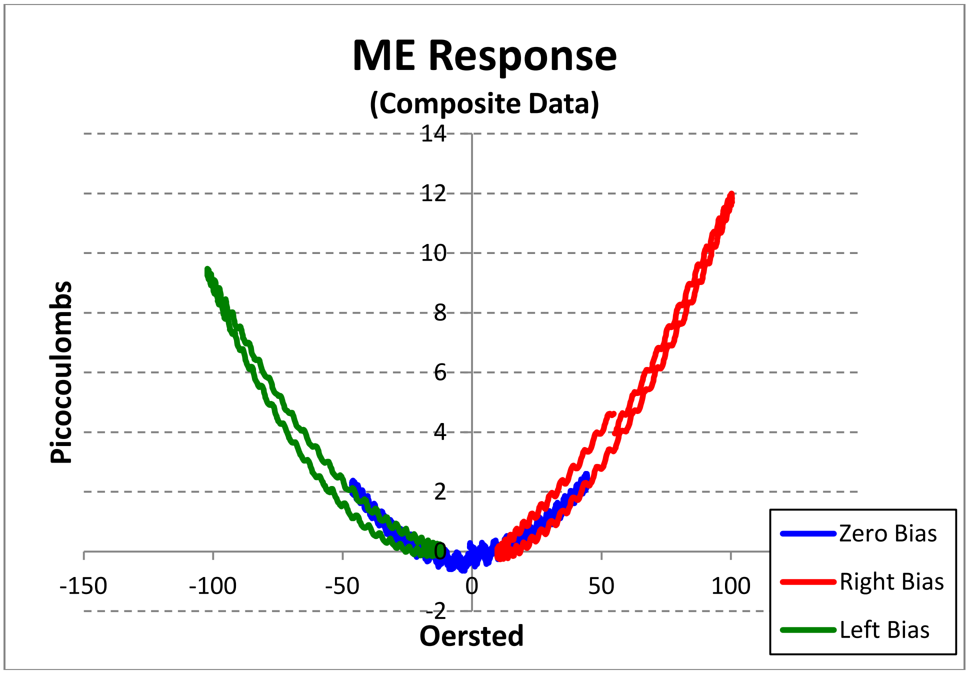

Figure 9: Magneto-electric response of the sample.

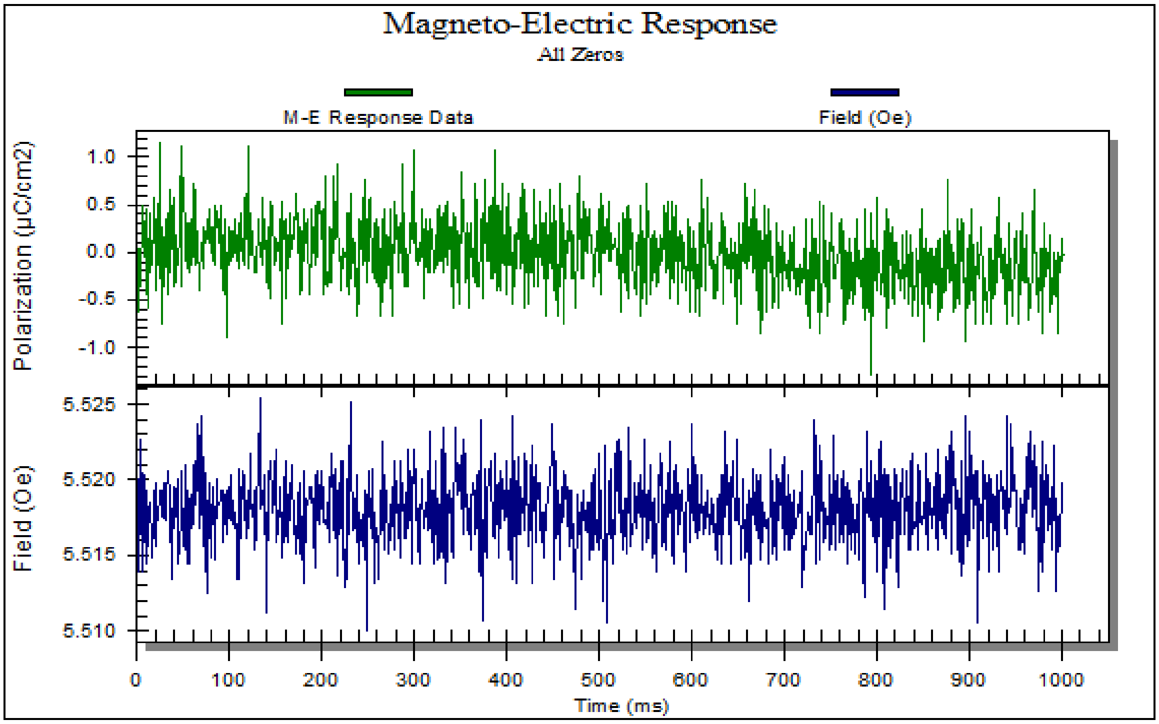

Figure 10: Ambient noise level at zero magnetic field.

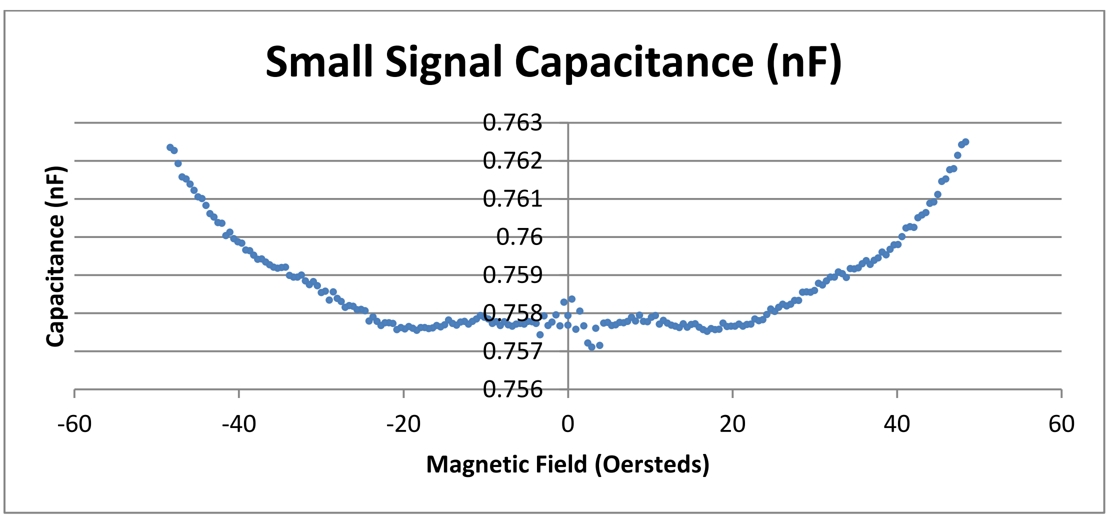

Figure 11: Sample Capacitance vs. Applied Magnetic Field.

Analysis:
The α = P x H coefficient at a particular field value is the slope of the MR Task measurement of the sample at the field point in Figure 8. A 2nd degree polynomial fit of the measurement is given by:

$$Q = 0.0012\ H^2 + 0.006\ H - 0.2126 \qquad \text{eq (5)}$$
$$R^2 = 0.9664$$

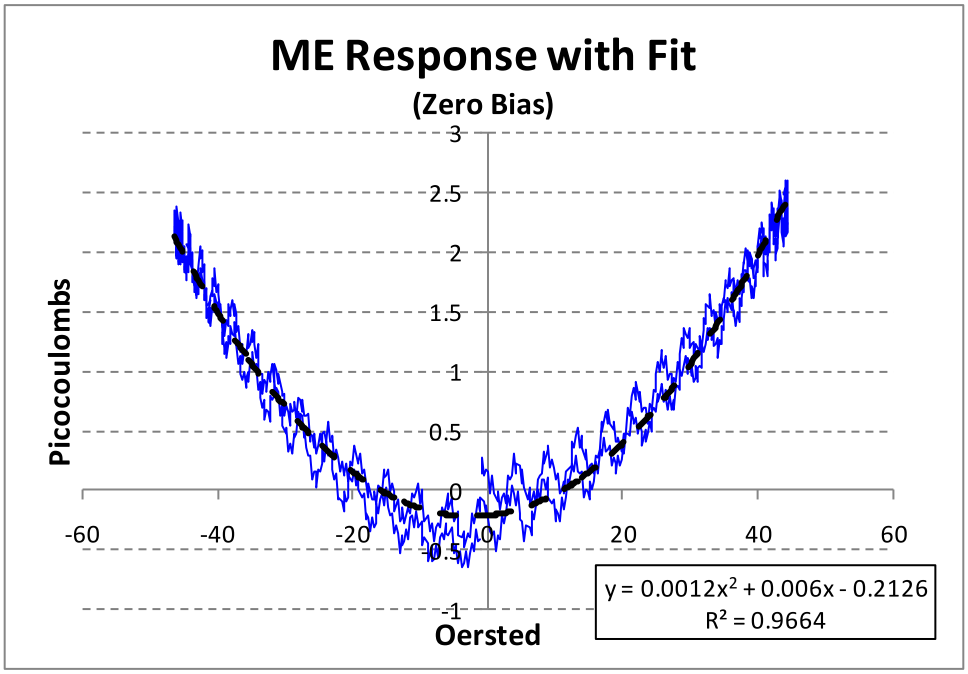

Figure 12: Zero Bias Magneto-electric response with 2nd order polynomial fit.

The slope of eq (5) as a function of field is:

$$\delta Q/\delta H = 0.0024\ H + 0.006\ (pC/Oe) \qquad \text{eq (6)}$$

$$\alpha = \delta P/\delta H = \delta(Q/A(cm^2))/\delta H = 0.00306\ H + 0.0076\ (\delta pC/cm^2/\delta Oe) \qquad \text{eq (7)}$$

Selecting a sample point of +43.0 Oe, (7) gives:

$$\alpha = 0.00306\ H + 0.0076 = 0.1391\ (pC/cm^2/Oe) = 139\ (fC/cm^2/Oe) \qquad \text{eq (8)}$$

To calculate the α_{ME}, start with (6) and note that the capacitance of the sample in Figure 11 at 43.0 Oe is 761 pF:

$$\delta V/\delta H = (6)\ /\ \text{Capacitance Density}$$

$$\delta V/\delta H = 3.15 \times 10^{-6}\, H + 7.88 \times 10^{-6}\ mV/cm^2/\Delta Oe \qquad (9)$$

Now, dividing (9) through by the sample thickness of 0.05 cm to convert δV to δE produces:

$$\alpha_{ME} = \delta E/\ \delta H = 6.3 \times 10^{-5}\, H + 1.576 \times 10^{-4}\ mV/cm^2/\Delta Oe \qquad (10)$$

$$\alpha_{ME} = \delta E/\ \delta H = 2.87\ mV/cm/Oe \text{ at } 43\ Oe.$$

Conclusion:
The MR Task in Vision reports the value of α as polarization per unit of magnetic strength. When converted to α_{ME} using the capacitance of the device under test, the Task is shown to be a valid substitute for traditional measurements. The Polarization method has the advantages of:

- Being accessible to the Precision family of testers and Vision program.
- Being fully automated – Can measure a broad sweep of field levels in a single measurement and center the measurement sweep about a variable set of stepped DC Bias field values.
- Rapid experiment completion requiring no manual adjustment during the experiment.
- Not being subject to parasitic cable capacitances that can affect lock-in amplifier measurements.