

Application Note
Ferroelectric Properties vs Magnetic Bias Field
Rev -

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Introduction

The Vision Library offers the Magnetolectric Response Task to measure the magnetolectric coupling coefficient in multiferroic materials and composite magnetopiezoelectric 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.

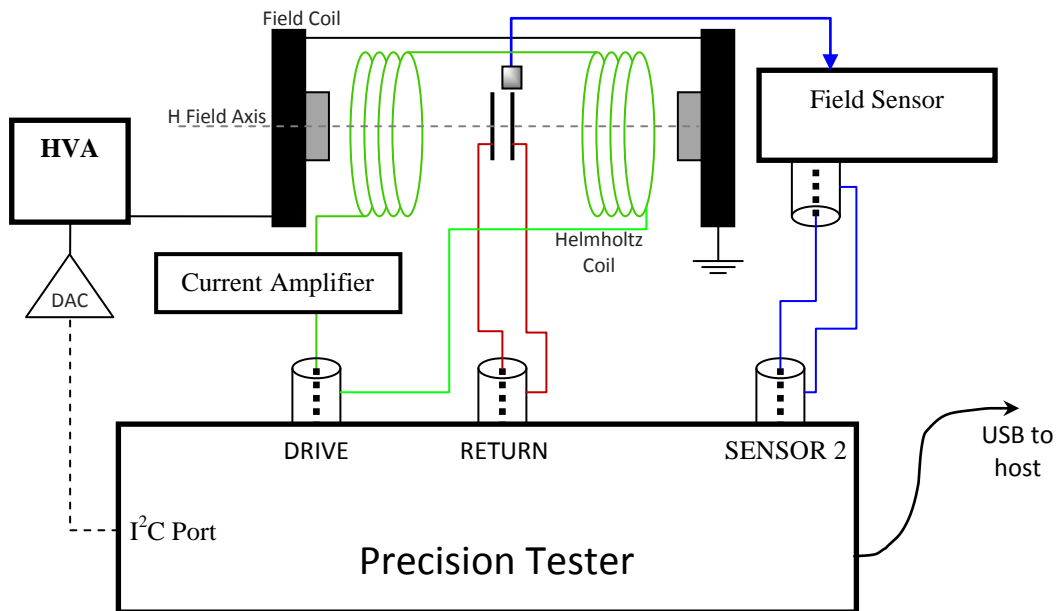


Fig. 1: Measuring magnetolectric response with a bias magnetic field.

The test configuration in Figure 1 will measure the coupling coefficient α between the magnetic field and the piezoelectric response of the sample:

$$\mathbf{P} = \alpha \mathbf{H} \quad \text{Eq(1)}$$

This measurement technique along with an explanation of error sources and example test results are described in the document “Application Note - Magnetolectric Response Task.pdf” available on Radiant’s web site.

There are a wide variety of measurements besides the magnetoelectric response necessary to fully characterize these unique samples.

- 1) Polarization Hysteresis vs Magnetic Field
- 2) Remanent Polarization vs Magnetic Field
- 3) PUND vs Magnetic Field
- 4) Conduction vs Magnetic Field
- 5) IV vs Magnetic Field
- 6) Small Signal Capacitance vs Magnetic Field

All of these measurements require only a fixed magnetic bias field from the field coil in Figure 1, eliminating the need for the Helmholtz coil.

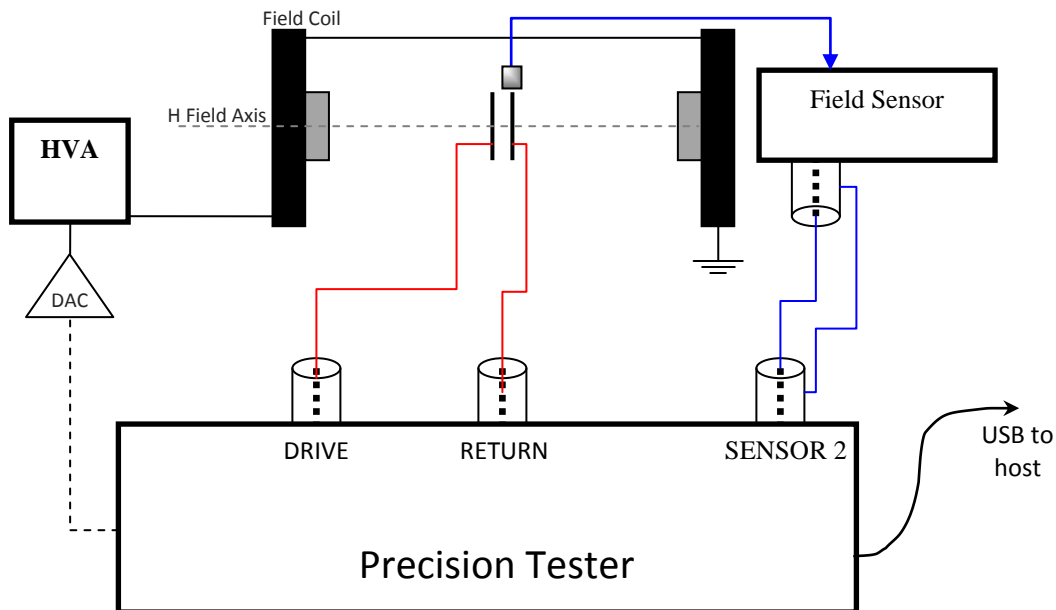


Fig. 2: Measuring ferroelectric responses versus a bias magnetic field.

The sample is connected to the tester DRIVE and RETURN ports in the standard manner necessary to measure ferroelectric properties. An I²C DAC module controls the high voltage amplifier of the magnetic field coil. The field coil establishes a DC magnetic field across the sample. Without the Helmholtz coil, the test frequencies for the sample can be expanded to the frequency limits of the tester and sample.

This application note describes below how to create User Defined Tests in the Vision Editor to make the measurements listed above. It is assumed that the researcher is familiar with creating, executing, and analyzing the results of Vision Test Definitions. If not, please refer to the Tutorials chapter of the Vision HELP pages for instructions about custom test programs in Vision. Select “Help-> Help Topics” in the

Vision menu. This selection takes the user to the table of contents for all HELP pages. As an alternative, each task menu has a HELP button which will take the user directly to the HELP page associated with that task.

NOTE: It is impossible in this application note to describe every aspect of every test described below. Please utilize the extensive HELP pages provided for each task in Vision to find more detailed information about that task and its setup menus. Also, Radiant provides example programs as [User Defined Tests](#) in the [Customized Tests](#) folder of the Vision Library. These examples may be moved to the Editor and modified as necessary to match the sample and test conditions.

Using the Vision Editor and the test fixture configuration in Figure 2, the researcher may measure a wide variety of changes to sample response as a function of magnetic fields including the characterization of long term reliability effects.

User Defined Tests

The Vision Editor allows the user to create Test Definitions for execution by a dataset. A test definition consists of a list of tasks from the Vision Library. Upon execution, Vision executes the tasks in the sequential order they occur in the Test Definition and stores the results in the Archive database of a Dataset. Vision datasets end in the extension “.dst” and have two parts: the Current Test Definition (CTD) and the Archive. The Archive holds a record of each execution of the CTD in the time order of execution. The CTD may be changed at any time so the records in the Archive may represent a variety of Executed Test Definitions with a variety of data formats. The user creates a Test Definition by selecting the desired tasks from the Vision Library and moving them to the Vision Editor. After all of the tasks in the Editor have been configured properly, the user moves the TD to the selected dataset to become its CTD. When executed, the tasks of the CTD are run in the order they appear in the CTD and the results are stored as a record in the Archive. Each record holds a copy of the Executed Test Definition (ETD) as well as all of the data collected during the program execution. Records in the Archive *may not be changed once recorded*. Even Radiant does not have the capacity to change archived records.


CTDs with repetitive or shmoo measurements become very compact using Branch Loops. CTDs with Branch Loops have two important characteristics: 1) there may be no nested Branch Loops and 2) data and filters are stored in the Archive record in the order they are executed. Therefore, a simple four line CTD with a Branch Loop Task set to loop 100 times will store a linear list of 400 entries in the record in the time order of execution.


One of the strengths of Vision is its ability to embed data analysis tasks in the program execution. These analysis tasks are called filters. Filter tasks collect data from measurement tasks or other data filters executed earlier in the test definition, perform the specified function on the data, and plot the results. The plots may be recalled from the Archive and transferred directly into word processing programs, spread sheets, or presentation programs.


Vision has a feature that allows the user to create flow diagrams of test definitions. For each test definition described in this application note, there will be a flow diagram imported into this application note directly from Vision.

Example Test Definition:

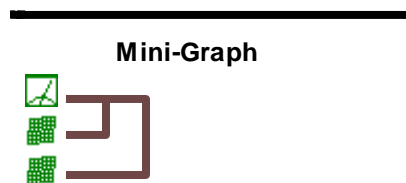
The following program executes a single hysteresis loop on the sample and creates two plots: 1) the hysteresis loop and 2) the normalized CV of the hysteresis loop. (The nCV is the mathematical derivative of the polarization vs voltage hysteresis loop).

Task V:4.6.4	9/21/11	Measurement Task	
Task Type: Hysteresis			
Task Name: Hyst 5V 1ms			

Task V:4.6.0	5/31/11	Filter Task	
Task Type: Hysteresis Filter			
Task Name: Plot Loop			


Task V:4.6.0	5/31/11	Filter Task	
Task Type: Hysteresis Filter			
Task Name: Plot nCV			

The execution flow looks like this:






The brown lines represent the data flow from the hysteresis test to the two data filters.

The Test Definition above simply makes one measurement and plots it in two different ways. By adding a Branch Loop, the Test Definition can be made to collect and plot all of the hysteresis loops of the sample from 1V to 5V in half-volt steps.


Task V:4.6.4 9/21/11 Measurement Task 

Task Type: Hysteresis
Task Name: Hyst 1V->5V 1ms
Voltage: 1.00
Period: 1.00 (ms)
Profile: Standard Bipolar
Adjust Volts in a Branch Loop
Voltage Increment: 0.50 (Volts)


Branch Target 
Filter Target 

Task V:4.6.0 5/31/11 Filter Task 

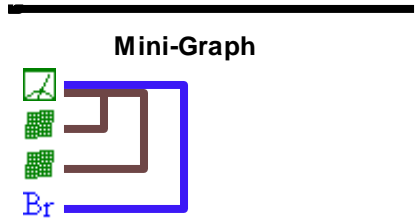
Task Type: Hysteresis Filter
Task Name: Plot Loop
Append Data
Filter Type: Centering
Number of Associated Tasks: 1
Input Task Names...
Task 1: Hyst 1V->5V 1ms

Task V:4.6.0 5/31/11 Filter Task 

Task Type: Hysteresis Filter
Task Name: Plot nCV
Append Data
Filter Type: Normalized Capacitance Vs Voltage
Number of Associated Tasks: 1
Input Task Names...
Task 1: Hyst 1V->5V 1ms

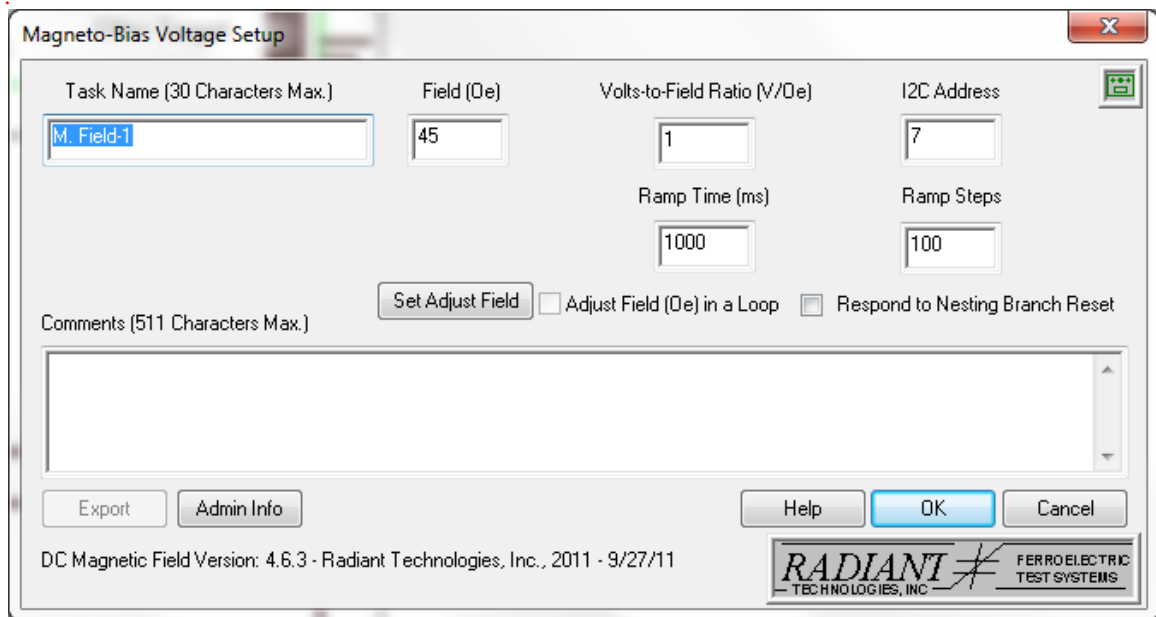
Task V:4.6.0 5/31/11 Branch Task 

Task Type: Branch Task
Task Name: Loop to 5V
Logic: if Hysteresis: Current Volts < 5, then Branch
Target Task: Hyst 1V->5V 1ms



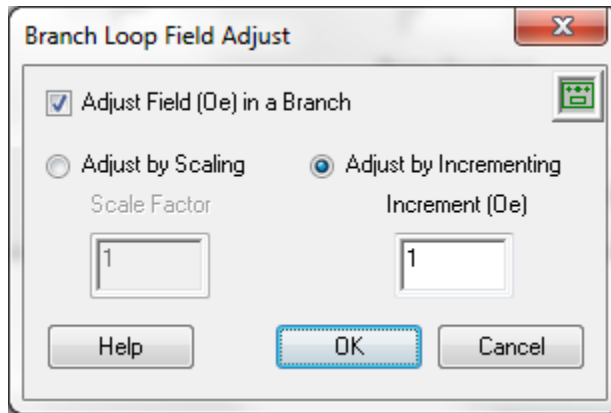
The blue line in the program flow chart is the Branch Loop retrace. Note that the Hysteresis Task has been modified from the first example to start at 1V and increment its Vmax by 0.5V each time a new loop is executed. Also note that the two Hysteresis Filter tasks have been adjusted to append (red circle) each hysteresis loop to its record so they all appear in one plot. Without the append setting, this program would create 22 plots, one for each filter task in each loop, a total of eleven each for the two filters.

The key to the usefulness of this Test Definition is the ability of the Hysteresis Task to adjust its test parameters on each loop. There are other, more complex methods for adjusting test parameters during a test but the Branch Loop is enough by itself to create very useful measurements of ferroelectric properties as a function of magnetic field. The menu for the task controlling the DC magnetic bias field is below.



This task sets a voltage on the I²C DAC module of Figure 1 to generate the specified magnetic field. The Volts-to-Field Ratio parameter converts the assigned magnetic field amplitude to the appropriate input voltage for the high voltage amplifier. This ratio must be calculated and calibrated by the user. The task does not step directly to the set field. Instead, it ramps to the specified field according to the Ramp Time and Ramp Steps parameters set by the user. Pressing the HELP button on this menu will open the HELP page that describes the meaning of the parameters and the operation of this task.

By clicking the “Set Adjust Field” button, this task will adjust the assigned magnetic field in a Branch Loop.



Other tasks that will be useful for characterizing magneto-electric properties include the Waveform Task and the Delay Task, both of which have adjustable properties inside a Branch Loop.


Below are a series of recommended Test Definitions for magneto-electric measurements that may be constructed using the Vision Library. The examples below do not represent the full array of custom tests that may be constructed in Vision. For any information or assistance in constructing a test, please contact us at Radiant Technologies (1-505-842-8007, radiant@ferrodevices.com).


Test #1: Hysteresis vs Magnetic Field


A simple test is to examine the change in the polarization hysteresis loop as a function of a background magnetic field strength. This is done in a Branch Loop using a Hysteresis Task to measure the loop, a Hysteresis Filter to capture and plot the polarization loops, and a Single-Point Filter to retrieve and plot the loop parameters like coercive voltage and $\pm Pr$.

There are two generic plot construction tasks in the Vision Library. Most measurement tasks store not only the captured data but also points of interest from that data. For instance, the Hysteresis Task captures the hysteresis loop of the sample but also derives and stores the Pmax value, Vc, and Pr values. The first generic plot constructor in vision is the Collect/Plot Filter. It plots the captured data such as the hysteresis loop or PUND results. It can collect the data from multiple instances of a specified task and plot all that data together using the standard plot format for that measurement task. The standard plot format for a measurement task is the default plot format encountered when executing that task from QuikLook. The Collect/Plot Filter setup menu provides a list of all tasks in the Library from which the user chooses which task type to plot.


The Single-Point Filter collects and plots the defined points of interest identified by measurement tasks. It must be assigned another parameter against which to plot the special points and it *must* be placed inside a Branch Loop where the X-value varies in each loop. For instance, a Hysteresis Task may be placed inside a Branch Loop with a Single-Point Filter. By varying the Hysteresis test period in each loop, the Single-Point Filter can be configured to plot the coercive voltages of the loop as a function of test frequency. The Single-Point Filter is of special interest in this application note because it can be used inside Branch Loops to plot changes to sample characteristics as a function of the DC magnetic field set by the I²C DAC module. That function is used in the test flow diagram displayed below.


Task V:4.6.3 9/27/11 Hardware Task 


Task Type: DC Magnetic Field
Task Name: Set Magnetic Field
Field: 1.00 (Oe)
Adjust Field in a Branch Loop
Increment Volts By: 1.00 (Oe) **Branch Target** 
Volts-to-Field Ratio (V/Oe): 1.00
I2C Address: 7
Ramp Time (ms): 1000.00
Ramp Steps: 100

Task V:4.6.0 5/31/11 Program Control Task 

Task Type: Delay
Task Name: Delay 30 seconds
Delay for 30 Seconds

Task V:4.6.4 9/21/11 Measurement Task 

Task Type: Hysteresis
Task Name: Hyst 5V 10ms
Voltage: 5.00
Period: 10.00 (ms)
Profile: Standard Bipolar **Filter Target** 

Task V:4.6.1 6/24/11 Filter Task 

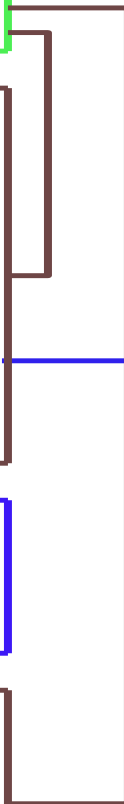
Task Type: Single-Point Filter
Task Name: Single Point Filter
Input Task Type: Hysteresis Task
X-Axis: User Variable DC Magnetic Field: Current Fiel
Captured Parameters: 5
Measured Parameter 1: PMax ($\mu\text{C}/\text{cm}^2$)
Measured Parameter 2: Pr ($\mu\text{C}/\text{cm}^2$)
Measured Parameter 3: -Pr ($\mu\text{C}/\text{cm}^2$)
Measured Parameter 4: Vc
Measured Parameter 5: -Vc
Number of Associated Tasks: 1
Input Task Names...
Task 1: Hyst 5V 10ms

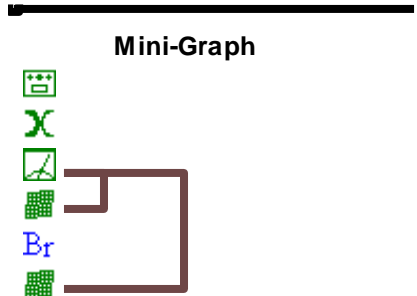
Task V:4.6.0 5/31/11 Branch Task 

Task Type: Branch Task
Task Name: Loop to 64 Oe
Logic: if DC Magnetic Field: Current Field < 64, then Branch
Target Task: Set Magnetic Field

Task V:4.6.0 5/31/11 Filter Task 

Task Type: Hysteresis Filter
Task Name: Plot Loops vs Mag Field
Accumulate Data
Filter Type: Centering
Number of Associated Tasks: 1
Input Task Names...
Task 1: Hyst 5V 10ms





In this test, the hysteresis loop is set so as *not* to change in each loop. Instead, it executes the same electrical measurement each time. The DC Magnetic Field Task, on the other hand, is set to start at 1 Oersted and increment this value by 1 Oersted each time it repeats. The Branch Loop is set to continue looping until the magnetic field value reaches 64 Oersted. A 30 second delay between the DC Magnetic Field Task and the Hysteresis Task ensures that the magnetic field has stabilized prior to execution of the hysteresis loop. The Delay Task is capable of much longer delays if necessary or it may be left out of the program. The Single-Point Filter Task is positioned to collect coercive voltages, P_r values, and the P_{max} value from each hysteresis loop and plot them against the DC magnetic field value. The Single-Point Filter Task *must be inside the Branch Loop*. Notice that on this test, the Hysteresis Filter is positioned *outside the Branch Loop*. From outside the branch loop, the Filter Task must be set to the *accumulate mode* (see **red circle**) instead of the *append mode* as in the earlier example. In the accumulate mode, the Hysteresis Filter Task will look inside the loop and plot every execution of the Hysteresis Task on a single graph. For information on selecting between accumulate and append mode for the filter tasks, press the HELP button on the task menu. The accumulate mode reduces the size of the dataset but does not plot its results until all measurements have been completed. To see plots real time as they are taken, use the append mode inside the loop. In the given program, the results of the Single-Point Filter will be plotted as they are taken since this filter resides inside the loop.

This program will take about one hour to run but plots the results after completion. After execution of this test definition, the Archive Record will contain 64 hysteresis loops and 64 Single-Point Filter tasks along with a single plot at the end with all 64 hysteresis loops. Executing this same number of loops manually, exporting the data to a graphics program, and graphing the results by one person would probably take a week.

Other Parameters vs Magnetic Field

Other ferroelectric measurements may be characterized as a function of the magnetic field. By substituting the Advanced CV Task, the I/V Task, or the Remanent Hysteresis task for the Hysteresis Task in the example above, it is possible to characterize differences in the small signal CV, the sample leakage, or the remanent polarization switching as a function of the strength of a background magnetic field.

Test #2: Switching Polarization with Magnetic Field

A fascinating possibility with magnetoelectric materials is that remanent electrical polarization may be switched by an external magnetic field. This characteristic may be tested with the following series of tests that replicate in magnetic field the well established electrical PUND test for ferroelectric materials.

Any measurement of remanent polarization is complicated by the fact that even a saturating electric pulse applied to a ferroelectric capacitor in the same direction the capacitor is already saturated will show apparent remanence. That means that the polarization does not return to zero as the voltage returns to zero. The fact that this excess polarization remaining outside the capacitor decays into the capacitor in a few seconds indicates that it is not generated by switching domains but instead by some internal parasitic response to the applied voltage. Assuming that the same parasitic occurs whether domains switch or not, the accepted method for determining domain-generated remanent polarization requires the differencing of the results of a switching test and an identical non-switching test. The test electrical profile is shown below.

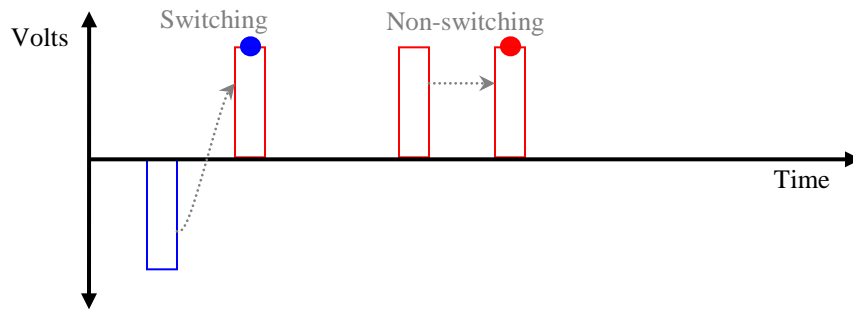


Fig. 3: Classic remanent polarization measurement using pulses.

The red and blue dots represent the points at which the polarization state is measured by the test instrument. The blue dot contains the sum of the switched remanent polarization plus the parasitic response. The red dot theoretically contains only the parasitic response. Subtracting the value of polarization contained in the red dot from that contained in the blue date should yield the switched remanent polarization, i.e. the memory. The test profile in Figure 3 uses the Single Pulse Task to emulate the PUND test common in the ferroelectrics community. The pulses may be replaced by half-hysteresis loops using a Hysteresis Task. Instead of the single polarization values measured at the red and blue dots above, the switching and non-switching half-loops are subtracted from each other point-for-point to show the remanent hysteresis loop. The subtraction may be executed by the Two-Trace Math Filter from the Vision Library.

Using sufficiently high voltages for the pulses or half-loops in the test above, it is possible to determine the saturated remanent polarization. The test can then be used to determine if remanent polarization may be switched by external magnetic fields. This is done by inserting a “magnetic pulse” in the switching test between the preset pulse and the measurement pulse.

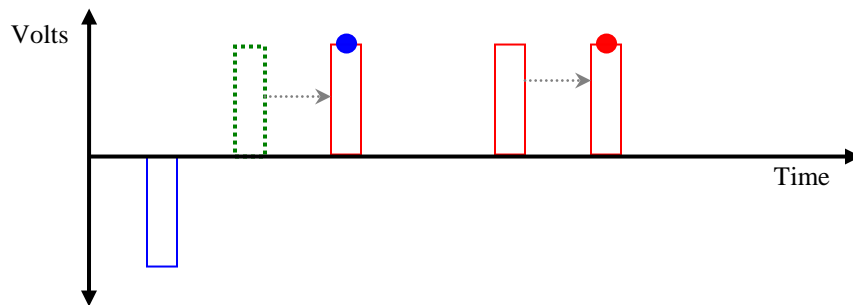





Fig. 4: Classic remanent polarization measurement using pulses.


The first (**blue**) preset pulse ensures that all of the remanent polarization is pointed negative, or DOWN, in the capacitor. The dashed pulse, **green**, represents the application of a magnetic field to the sample. The B-field travels to the assigned amplitude and direction before returning to zero. The third pulse, with the **blue** measurement dot, switches UP any polarization still un-switched by the magnetic field. Subtracting the non-switching value (**red**) from the switched value (**blue**) as before shows how much remanent polarization was still pointing DOWN after the magnetic pulse. If the magnetic field did not switch any polarization or if the magnitude of the magnetic field pulse was zero, the value of remanent polarization should match the saturated value measured in the all-electrical test of Figure 3. If the magnetic field did move remanent polarization, then the remanent polarization measured in Figure 4 will be less than that seen in Figure 3. By executing a series of tests from Figure 4 while varying the amplitude and direction of the magnetic field, the magnetoelectric switching characteristics of the sample can be captured. The Vision program is below.

Task V:4.6.4 9/27/11 Measurement Task 
Task Type: Simple Pulse
Task Name: Preset DOWN -5VVolts: -5.00


Pulse Width (ms): 1.00
Pulse Delay (ms): 1000.00
Delay Volts: 0.00
End Volts: 0.00 Write-Only Pulse


Task V:4.6.3 9/27/11 Hardware Task 
Task Type: DC Magnetic Field
Task Name: Apply B Field 20 Oe
Field: 20.00 (Oe)
Volts-to-Field Ratio (V/Oe): 1.00
I2C Address: 7
Ramp Time (ms): 1000.00
Ramp Steps: 100

Task V:4.6.3 9/27/11 Hardware Task 
Task Type: DC Magnetic Field
Task Name: Zero Magnetic Field
Field: 0.00 (Oe)
Volts-to-Field Ratio (V/Oe): 1.00
I2C Address: 7
Ramp Time (ms): 1000.00
Ramp Steps: 100


Task V:4.6.4 9/27/11 Measurement Task 
Task Type: Simple Pulse
Task Name: Measure DOWN StateVolts: 5.00

Pulse Width (ms): 1.00
Pulse Delay (ms): 1000.00
Delay Volts: 0.00
End Volts: 0.00
Read/Write Pulse


Filter Target 


Task V:4.6.4 9/27/11 Measurement Task 
Task Type: Simple Pulse
Task Name: Preset UP +5VVolts: 5.00

Pulse Width (ms): 1.00
Pulse Delay (ms): 1000.00
Delay Volts: 0.00
End Volts: 0.00 Write-Only Pulse

Task V:4.6.4 9/27/11 Measurement Task 
Task Type: Simple Pulse
Task Name: Measure UP StateVolts: 5.00

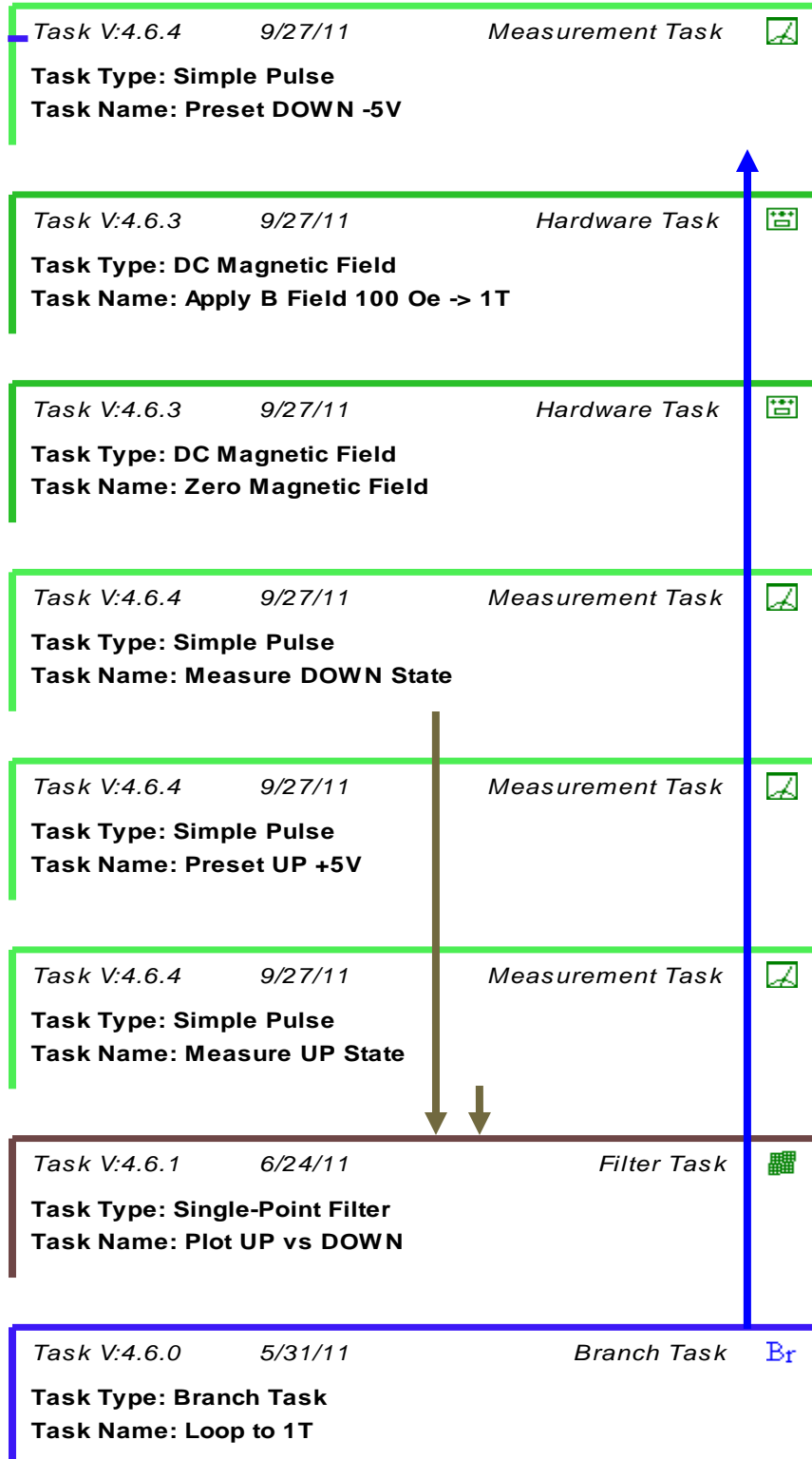
Pulse Width (ms): 1.00
Pulse Delay (ms): 1000.00
Delay Volts: 0.00
End Volts: 0.00
Read/Write Pulse

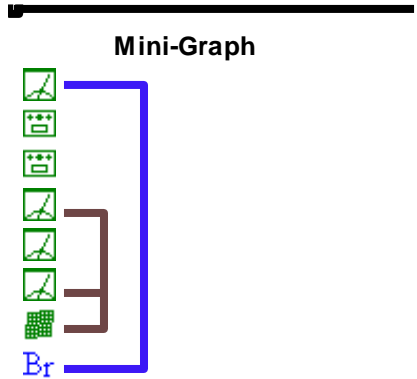
Filter Target 

Task V:4.6.1 6/27/11 Filter Task 
Task Type: Two-Trace Math Filter
Task Name: Calculate Remanent P
Input Data Type: Simple Pulse
Operation: Subtraction
X Data Option: Average A and B Task Data
Input Task Names...
Task 1: Measure DOWN State
Task 2: Measure UP State
X-Axis: Volts

Note the use of the Two Trace Math Filter at the end of the task flow to calculate the difference between the DOWN and UP measurement pulses. As described above, the Single Pulse Tasks may be replaced by Hysteresis Tasks set for monopolar stimulus.

The program can be expanded to sweep through a range of magnetic field strengths. Replace the Two Trace Filter with a Single-Point Filter followed by a Branch Task to force looping. The “Set Adjust Field” button on the DC Magnetic Field Task increases the amplitude of the magnetic field on each loop. The Single-Point Filter task plots the two polarization measurements in each branch loop against the magnetic field amplitude in each loop. A compacted program diagram looks as follows:





The magnetic field should be returned to zero before the electric measurement pulse in case the presence of the magnetic field somehow changes the pulse response of the sample. By returning the magnetic field to zero prior to the polarization measurement, it is assured that only the remanent polarization state after the magnetic field application is measured without complications from a parallel magnetoelectric response to a DC magnetic field.

Test #3: Reliability vs Magnetic Field

The effect of a constant magnetic field on the reliability of a capacitor is straightforward to execute in Vision. The example below shows fatigue in a magnetic field but imprint, retention, or long term dielectric breakdown tests may be substituted for the fatigue test. Each of these reliability tests are standalone tests in the Vision Library.

Task V:4.6.3	9/27/11	Hardware Task	
Task Type: DC Magnetic Field			
Task Name: Apply Magnetic Field			

Task V:4.6.2	9/28/11	Long Duration Task	
Task Type: Fatigue			
Task Name: Fatigue - 3kHz 1E9 cycles			

Task V:4.6.3	9/27/11	Hardware Task	
Task Type: DC Magnetic Field			
Task Name: Zero Magnetic Field			

This test is a straight-through test with no branch looping in the program: 1) set the magnetic field, 2) execute the entire reliability test, and 3) zero the magnetic field.

Conclusion

This application note describes a range of custom tests for magnetoelectric properties that may be executed using Vision combined with a Precision Non-linear Materials Tester. The beauty of Vision is its facility to quickly combine the tasks in the Vision Library in any arbitrary order to create a custom test without the expense and delay of having Radiant write custom programs specifically for the property in which the researcher has an interest. There are a range of useful tests not described above that the user may construct with Vision to characterize ferroelectric or magnetoelectric samples.