Radiant Technologies, Inc.
2835B Pan American Freeway NE
Albuquerque, NM 87107
Tel: 505-842-8007
Fax: 505-842-0366
e-mail: radiant@ferrodevices.com
www.ferrodevices.com

User Manual
High Voltage Cryogenic Dielectric Probe
Rev -

Date: March 13, 2018

Author: Joe Evans

Introduction:
The Radiant High Voltage Cryogenic Probe (HVCP) is compatible with Quantum Design PPMS© and DynaCool© cryogenic chambers. The fixture inserts into the Quantum Design system and connects to a Radiant Technologies' Precision Non-linear Materials Tester to allow electrical testing of single crystal or bulk ceramic capacitors in the temperature and magnetic field environments created by the chamber. Electrical measurements up to 1,200 volts may be executed at stabilized temperatures down to 10 Kelvin with DC magnetic fields up to 9 Tesla without electrical breakdown of the internal helium atmosphere that cools the chamber. Measurements above 1,200 volts up to 4,000 volts may be executed without breakdown if the chamber is placed in High Vacuum mode at the sacrifice of temperature stability arising from the lower density of the helium atmosphere.

This manual describes the test fixture and its operating procedures. Warnings and limitations in the use of the fixture are provided. Installation and operation of communication protocols between the tester and the Quantum Design chamber are explained.

Specifications

There are three sources of electrical breakdown for a sample mounted inside this probe.

1) Breakdown of the sample itself.

2) Helium breakdown around the edge of the sample between its electrodes.

3) Breakdown of the helium atmosphere between metal parts of the fixture itself.

Electrical breakdown of a gas is related to its pressure but not its temperature as determined by Friedrich Paschen in the late 1800s. Cryogenic chambers use helium at single-digit Torr pressures. At those pressures samples must use sample geometries that allow all intended measurements to execute below the maximum voltages specified below. The helium atmosphere breakdown limits are not related to the model of tester used or the dielectric material from which the Device Under Test (DUT) is constructed. See the Appendices for a theoretical explanation of the origin of the specifications and test results.

1. **Maximum Sample Dimensions**
   a. Maximum diameter 1cm
   b. Maximum linear dimensions 1cm x 1.3cm

2. **Temperature Limits**
   a. Maximum 380 Kelvin
   b. Minimum 10 Kelvin

3. **Maximum breakdown voltage to Earth Ground inside Sample Holder.**
   a. For sample electrode maximum helium-gap <1mm

In air	9,000V
At 760 Torr in helium	4,500V
~1 Torr to 10 Torr in helium	1,200V
<1 Torr in helium	4,000V

NOTE: Electrical measurements inside the chamber must never be conducted unless the chamber metal is firmly connected to the facility earth ground.

4. **Maximum breakdown voltage sample-electrode to sample-electrode.**
   a. For sample electrode maximum helium-gap <1mm - Unlikely

   b. For sample electrode maximum helium-gap >1mm
      i. ~1 Torr to 10 Torr in helium 400V

5. **Minimum Chamber Pressure**
   a. The HVCP properly seals the PPMS/DynaCool chambers so the user may operate the PPMS at any pressure allowed by the chamber.

Warnings

1. **The Quantum Design PPMS or DynaCool chamber must be connected to earth ground.**

   If the helium breaks down and high voltage arcs to the chamber metal, it must have a path to earth ground. The Quantum Design PPMS or DynaCool chamber is normally connected to earth ground by a copper braid attached to the vacuum pump on first installation. ***Always verify that the chamber metal is indeed connected directly to earth ground.***

   If a *sample* breaks down inside the chamber during high voltage actuation by a Radiant tester, that current spike will be carried back to the Radiant Precision High Voltage Interface along the insulated HV RETURN cable to the Precision HVI which will route the current spike to the tester's own earth ground connection.

2. **Do not operate the fixture when it is outside the chamber.**

   When inserted inside the chamber, the grounded metal of the chamber acts as a shield between the user and the fixture

3. **Use only high voltage cables supplied by Radiant Technologies.**

4. **Contact Radiant Technologies should the HVCP or the high voltage sample capsule appear damaged.**

5. **Never use metal screws to close the Capsule or mount the Capsule.**

   Contact Radiant for replacement PEEK screws.

6. **Do not apply high voltage to the sample for more than 30 seconds.**

   High Voltage DC operation is not specified.

7. **Never disassemble the probe.**

   Its enclosed components are permanently mounted and sealed so it cannot be re-assembled.

8. **Never operate the probe with the top plate of the head removed.**

   High voltage wire will be exposed.

9. **Never connect an external High Voltage Amplifier to the DRIVE path between the tester and the HVCP without using a Precision High Voltage Interface and high voltage rated cables from Radiant.**

   The HVI protects the tester as well as personnel in the area.

10. **Always ensure that the HV DRIVE from the Radiant High Voltage Interface is connected to the DRIVE LEMO connection on the HVCP head.**

Description of the Fixture

The Radiant High Voltage Cryogenic Probe is a modification of the Quantum Design P450 Multifunction Probe. Radiant has

1) Machined the head to add LEMO high voltage sockets for DRIVE and RETURN entry.

2) Modified the interior of the head to extend the high voltage lines down the probe to the sample Capsule.

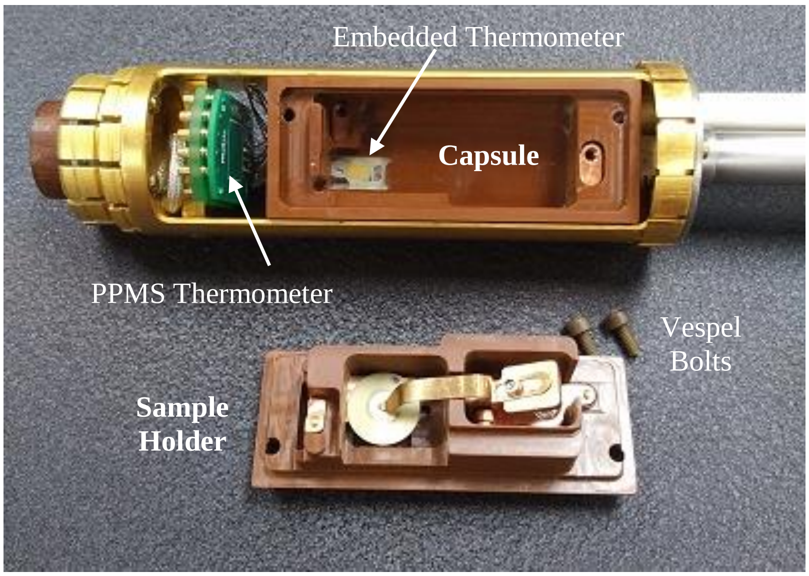
3) Encased the DRIVE and RETURN lines in alumina straws that traverse the length of the probe to the sample holder.

4) Mounted the sample inside a Vespel Capsule capable of withstanding high voltage at cryogenic temperatures and does not outgas to the chamber.

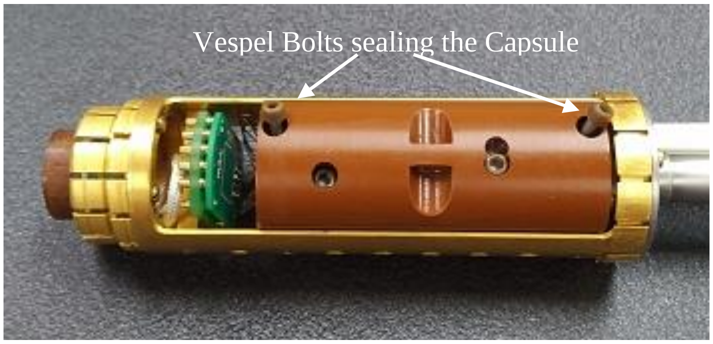
5) Embedded a thermometer inside the capsule to provide an accurate temperature reading at the sample and compensate for the thermal insulation function caused by the Vespel.

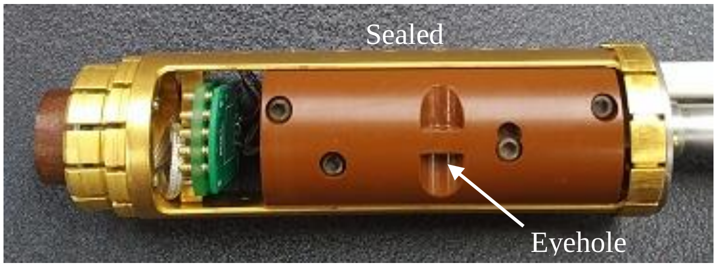
<u>Sample Capsule</u>

The high voltage sample fixture is fabricated with Vespel. Vespel is an air-filled glass developed by NASA. It is noteworthy for its high voltage insulation at cryogenic temperatures, its low coefficient of thermal expansion, and its lack of outgassing. Vespel completely encloses the sample and is thicker than the minimum necessary to withstand electrical breakdown up to 10,000 volts in air. The Vespel fixture consists of two halves: 1) the Capsule and 2) the Sample Holder.



High voltage wires from the tester enter the Capsule where they connect with their internal terminals. The capacitor under test clips between a bottom Oxygen-Free High Conductivity (OFHC) copper square and a top spring loaded OFHC clip in the Sample Holder. The Sample Holder inserts into the fixture capsule and is secured with PEEK bolts. Pogo pins in the Sample Holder connect the sample to the high voltage terminals of the Capsule.





> **WARNING**: Metal bolts ***cannot be used*** to secure the Sample Holder to the Capsule as they may reduce the voltage at which arcing occurs inside the fixture in low pressure helium. Please contact Radiant to replace missing PEEK bolts.

During testing, the sample is completely enclosed inside the capsule. The embedded thermometer is protected by a sapphire window to prevent arcing to ground through the thermometer circuits. The window is sealed with Masterbond EP21TCHT-1 epoxy.

An eyehole milled into the outside surface of the Sample Holder allows the user to insert a pin or screw driver to pull the Sample Holder from the Capsule.

Electrical Insulation

High voltage electrical insulation is provided throughout the HVCP.

1. External high voltage insulation is provided by HV rated electrical cables supplied by Radiant. These cables extend from the Precision High Voltage Interface (HVI) to the head of the HVCP. Connectors on both ends of the high voltage cables are rated above the maximum voltages that may be applied to the cables.

   DRIVE always carries all voltages.
   RETURN is always at or near ground potential even during sample breakdown

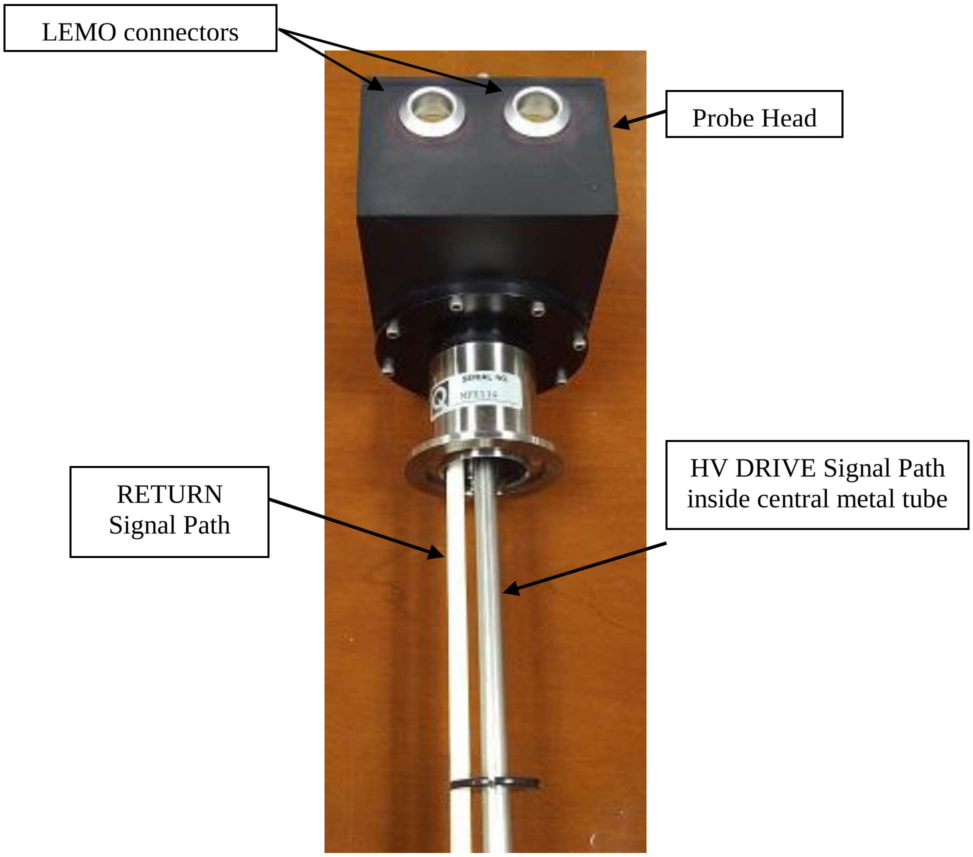
2. Insulation of the wires inside the volume of the head of the probe is provided by natural rubber tubing. The head remains at room temperature during cryogenic operation.

3. The InCoNel wires from the head down to the Capsule are encased in 31.25 mil alumina with a nominal breakdown voltage of 500kV.

4. The alumina straws are sealed with epoxy where they enter the head.

5. The DRIVE signal from the Capsule to the tester is inside an alumina straw which itself is inside the central metal support tube of the probe. This provides an additional layer of safety for electrical arcing. Since the DRIVE signal carries the active voltage, it is the most likely source of arcing during test. If the alumina straw fails to prevent arcing, the arc will ground to the central metal tube which is electrically connected through the chamber to earth ground.

6. The RETURN signal from the Capsule to the tester is inside an alumina tube visible *outside* the central support tube of the probe. See the image below. During normal operation, the DRIVE path carries the active voltage while the RETURN path *is always ground*. Under normal circumstances, the RETURN does not need high voltage insulation. However, if the sample breaks down, the tester RETURN input will hold that wire to1.5 volts above ground potential. If a high voltage amplifier (HVA) is being used in conjunction with a Precision High Voltage Interface, the HVI will hold that RETURN path at 2.1 volts above ground potential. Nevertheless, the RETURN path is insulated by the alumina straw inside the chamber and by high voltage insulated cable outside the chamber.



Both alumina straws enter the Capsule where the DRIVE and RETURN wires connect to the top and bottom OFHC copper components.

The four most likely scenarios for arcing inside the Capsule inside the chamber are:

1. Below 1200 volts in normal cryogenic atmosphere – Through-the-sample breakdown.

2. Below 1200 volts in normal cryogenic atmosphere – Breakdown through the helium atmosphere around the edge of the sample between the top and bottom electrode edges.

3. Near 1200 volts and above in normal cryogenic atmosphere – Breakdown of the helium between OFHC copper components inside the capsule, leaving the sample unharmed.

4. Near 4000 volts and above in high vacuum mode for the chamber - Breakdown of the helium between OFHC copper components inside the capsule or between electrode edges, leaving the sample unharmed.

Thermometers

There are two thermometers in the fixture. One is the standard thermometer chip at the bottom of the P450 probe supplied by Quantum Design. The second is embedded inside the Capsule by Radiant. The Capsule by necessity has a large thermal mass so the temperature inside its interior volume will lag behind that of the exposed PPMS P450 thermometer board at the bottom of the fixture during commanded changes. Both may be monitored simultaneously using PPMS MultiVu software. The embedded thermometer will lag the PPMS thermometer by approximately 10 minutes and should differ by no more than 0.5 Kelvin once the temperature is stabilized. The embedded thermometer can be assigned in MultiVu to control the chamber temperature.

Installing the Fixture into the Chamber
The HVCP is a highly modified version of the Quantum Design P450 Multifunction Probe. It should be inserted into the PPMS or DynaCool stations according to procedures recommended by Quantum Design.

Connecting the Sample to a Radiant Tester
The High Voltage Cryogenic Probe has two LEMO connectors in its exposed head. There are two sets of cables available from Radiant to use with the probe.

1. High Voltage – The high voltage LEMO cables have a LEMO plug on one end and a 25kV-rated plug on the other that connects to the Precision High Voltage Interface. The Precision HVI is must be installed any time an external amplifier is used in testing. *Always use high voltage cables supplied by Radiant when using an HVI with an external HVA.*

2. Low Voltage – The low voltage LEMO cables have a LEMO plug on one end of a standard coaxial cable with a BNC connector on the other end. This cable is to be used any time the tester is to be connected directly to the sample without an external amplifier. The advantage of the coaxial cable is that its shield braid connects to the tester in a manner that cancels external ambient electrical noise in the room. The chamber itself forms a very large Faraday cage so the entire signal path will be electrically shielded, allowing very small magnitude signals to be detected using the probe. Radiant testers are available with internal amplifiers up to 500 volts. *The coaxial cable is rated to no more than 1000 volts.*

Both types of cables must be acquired from Radiant.

Sending Commands to the PPMS/DynaCool MultiVu Control System from Vision

The Vision Data Management System that operates Radiant Precision testers can communicate with the Quantum Design MultiVu operating system that controls the PPMS or DynaCool chambers. The communications capability was jointly designed by Quantum and Radiant. Through this channel, Vision can remotely set and read the temperature, magnetic field, and/or gas pressure of the Quantum Design chamber. Control is executed by Vision through two Tasks in Vision:

Set QD PPMS/DynaCool
Read QD PPMS/DynaCool.

They are located in the

EXTERNAL INSTRUMENTS sub-folder of the HARDWARE folder

in the Vision Library. They are also found under

External Instrument Tasks

in Vision's QuikLook pull down menu. Detailed documentation for Vision's side of the communications can be examined by opening the menu for the "Set QD PPMS/DynaCool" Task or the "Read QD PPMS/DynaCool" Task and pressing the *Click For Task Instructions* button. Quantum Design's documentation is included in its "*1070-209-VB in MultiVu.pdf*".

Some installation is required in order to open the communications channel. However, once the communications channel is established, the user need only execute either of the "QD PPMS/DynaCool" Tasks for all operations.

Installation: One Time Modification of the MultiVu Host Computer

Before a control macro is first executed, the following steps must be performed:

1. Create the C:**RTIVision** folder *on the "C" drive of the computer running MultiVu.*

2. Give Vision read/write privileges to the **C:\RTIVision** folder on the MultiVu host computer.

3. Copy the file **Vision.BAS** from C:\datasets\documents on the Vision computer to a location on the MultiVu host computer.

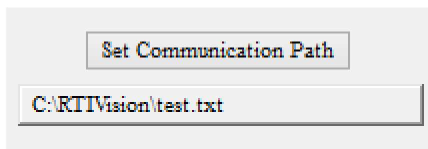
Normal Operation

1. From inside **MultiVu**, execute the macro **Vision.BAS**.

   -If possible, set MultVu to start Vision.BAS on startup as a default condition.

2. Call up and execute “Set QD PPMS/DynaCool” or “Read QD PPMS/DynaCool” from within QuikLook or the Vision Library.

   Vision communicates with MultiVu through the C:\RTIVision folder which must be on the host computer for MultiVu. The “QD PPMS/DynaCool” Tasks must know where to find this folder. There is a window in the lower right-hand corner of the menu for “QD PPMS/DynaCool” showing the pathway to the RTIVision folder on the MultiVu host.



   Use this control to define the path from Vision to the RTIVision folder on the PPMS/DynaCool host computer.

   i. If Vision is on the same computer as MultiVu, then C:\RTIVision will be on that same computer.

   ii. If Vision is on a separate computer from MultiVu, then a LAN channel must be opened between the two computers and C:\RTIVision must be located on the computer with MultiVu.

   iii. Once the pathway is established, Vision will store that path and recall it each time in the future that the “QD PPMS/DynaCool” Task is called.

   Always terminate the path at “test.txt”.

Older Quantum Design Models
Quantum Design PPMS models using an operating system prior to MultiVu can be controlled using GPIB commands. Vision contains several Tasks in its Library to set the temperature and/or magnetic field of a PPMS system via GPIB.

Set Temperature
Read Temperature
Set Field

These Tasks above still work with GPIB-controlled systems but cannot control MulitVu systems. Talk to MulitVu-controlled systems using the “QD PPMS/DynaCool” Tasks.

Enabling the Thermometer Embedded near the Sample
See documentation from Quantum Design.

Operation of Vision with the HVCP
Vision considers the Quantum Design PPMS or DynaCool chamber to be an external instrument that will accept commands from Vision to set the ambient environment of the sample and/or report the values of that environment when requested. Once communication between the PPMS or DynaCool system and Vision has been established, the user need only execute

> “Set QD PPMS/DynaCool”

or

> “Read QD PPMS/DynaCool”

from inside a Test Definition or from QuikLook to set or read the value of the desired environmental parameter. The “Set QD PPMS/DynaCool” Task allows the user to

1. Set the pressure condition inside the chamber from “Seal” to “Purge/Seal” to “High Vacuum”.

2. Set the temperature inside the chamber or

3. Set the magnetic field inside the chamber.

The “Read QD PPMS/DynaCool” Task allows the user to read the value(s) of those three parameters at the time of execution.

There are two factors involving these two Tasks that make their use more complicated but also yield more functionality to the user.

<u>User Variables</u>
Vision maintains a large list of User Variables utilized by all of the tasks. All Program Control and Data Filter tasks can access the User Variable list to use either in plots or to control execution of a Test Definition.

The User Variables associated with the “QD PPMS/DynaCool” Tasks are

> Set QD PPMS/Dynacool: Current Field
> Set QD PPMS/Dynacool: Current Field Ramp Rate
> Set QD PPMS/Dynacool: Current Set Field
> Set QD PPMS/Dynacool: Current Set Temperature
> Set QD PPMS/Dynacool: Current Temperature
> Set QD PPMS/Dynacool: Current Temperature Ramp Rate

Set QD PPMS/Dynacool: Original Field Ramp Rate
Set QD PPMS/Dynacool: Original Set Field Field
Set QD PPMS/Dynacool: Original Set Temperature
Set QD PPMS/Dynacool: Original Temperature Ramp Rate

Read QD PPMS/Dynacool: Current Temperature
Read QD PPMS/Dynacool: Current Field
Read QD PPMS/Dynacool: Temperature Status
Read QD PPMS/Dynacool: Field Status
Read QD PPMS/Dynacool: Chamber Status

Whenever any of the PPMS/DynaCool Tasks is executed, the User Variables above are updated by the called Task prior to exit.

Note the difference between the value of a parameter and the value of its "Set" parameter. For instance, "Current Field" and "Current Set Field" mean two different things.

QD PPMS/DynaCool: **Current Field**:	The last *measured* value of the magnetic field inside the chamber.
QD PPMS/DynaCool: **Current Set Field**:	The last value *assigned* to the magnetic field inside the chamber.

If the value of the magnetic field inside the PPMS or DynaCool chamber is set by a call to "Set QD PPMS/DynaCool" Task, the value of **Current Set Field** will remain constant at that assigned value until the next time the Set Task is called. However, the value of **Current Field** will change every time the field is measured by Vision. Every "Set" call ends with a measurement and an update of both variable types.

Set QD PPMS/DynaCool

The "Set QD PPMS/DynaCool" Task provides the user with two progress options:

1. Assign values to one or more of the three environmental conditions and exit ***immediately***

Or

2. Assign values to one or more of the three environmental conditions and ***monitor the chamber progress*** until the chamber stabilizes at the new value before exiting the Task.

Control of the exit condition is through the sub-window below on the main menu. In the example below, the box for "Do Not Monitor Temperature Progress" is checked. With this control setting, the Task will exit immediately after sending the assigned command to MultiVu and executing a single parameter measurement.

Since the "Set" Task measures the temperature as part of its operation, the two parameters "**Current Set Temperature**" and "**Current Temperature**" for the QD PPMS/DynaCool Task will have different values.

Current Set Temperature — will have the value of the newly assigned temperature.

Current Temperature — will have the value of the temperature in the chamber *at the time of Task execution.*

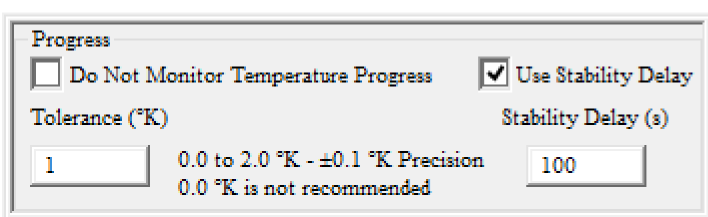
For example, if the chamber is at 300 K and "Set QD PPMS/DynaCool" is called to set the temperature to 10 K with the Progress Monitor is set to "Do Not Monitor Temperature Progress", the Task will exit immediately after sending the new temperature but will make a temperature measurement on exit. The two temperature User Variables will read differently.

Current Set Temperature 10

Current Temperature 300

Since in this case the "Set QD PPMS/DynaCool" task exits immediately, the next task in the Test Definition is called immediately.

If, instead, the task is set to "Use Stability Delay", the task *will not exit* until the stability conditions are met.



With the progress conditions set as in the image above, upon execution the Task will continue until the temperature has changed to the new value and has remained within 1 K of the assigned value for no less than 100 seconds. While waiting, the Task continuously reads and displays the temperature of the chamber. Since the Task does not exit until the newly assigned temperature has stabilized, the two parameters "**Current Set**

Temperature" and "**Current Temperature**" for the QD PPMS/DynaCool Task should have the same or very close to the same values.

Current Set Temperature	will have the value of the newly assigned temperature.
Current Temperature	will have the value of the temperature in the chamber *at the time the* task *exits* after the temperature stabilizes within the assigned tolerance of the assigned temperature.

If the assigned temperature is 10 K, then the Current Temperature parameter will be 10 ±*1.0* on task exit.

<u>Read QD PPMS/DynaCool Task</u>
On execution this task reads the value of the selected parameters and updates the User Variable List. The task offers a variety of conditional methods for acquiring measurements and exiting.

<u>For QuikLook Use:</u>

1. User Demand — For QuikLook use, a read is executed manually from the keyboard by the operator.
2. User Abort — The Task executes continuously until the user manually aborts the task.

<u>For Use Inside Test Definition:</u>

3. Programmed Count — The Task executes the specified number of read operations.
4. Specified Duration — The Task executes continuously for the specified time period.
5. Until Set Point — Same function as "Use Stability Delay".

<u>Using Stability Delay</u>
Whether to "Use Stability Delay" or not depends upon the maximum voltage to be used in the Test Definition.

1. If the maximum voltage is less than the 1200 volt for normal PPMS/DynaCool operation, then the Stability Delay *can be used*. The helium pressure is set for optimal cooling (1 Torr -> 10 Torr). Tests using voltages less than the 1200 volt specification can be used with it unlikely that electrical breakdown will occur through the helium. The Quantum Design chambers will go to the assigned temperature down to 10 K and maintain that temperature precisely for as long as desired. Any temperature within the rating of that

chamber can be reached and stabilized and will be reported accurately to Vision upon request. By invoking Stability Delay, the user is assured that the chamber will reach the assigned temperature and the "Set QD PPMS/DynaCool" Task will not let the Test Definition proceed until the sample is stable at the assigned temperature. Complex temperature profiles may be executed over long periods of time with full confidence that the measurements will occur at the desired temperatures.

2. If the maximum voltage is above 1200 volts, breakdown of the helium in the chamber in the Torr pressure range will occur so the High Vacuum condition must be invoked to avoid breakdown. However, the chamber will be unable to maintain the temperature of the sample at the assigned value in High Vacuum mode. The temperature inside the chamber will begin to climb as soon as High Vacuum is invoked. It will stop at approximately 200 K if High Vacuum is maintained.

   To make a single measurement *near* a single temperature, the user:

   a. Commands the chamber to the target temperature and waits until the temperature has stabilized.
      - **Set QD PPMS/DynaCool** in "Use Stability Delay" mode

   b. Commands the chamber into High Vacuum mode.
      - **Set QD PPMS/DynaCool** in "Do Not Monitor Temperature Progress" mode

   c. Executes the high voltage electrical measurement immediately.

   d. Reads the temperature of the chamber after the test is complete.
      - **Read QD PPMS/DynaCool** in "Do Not Monitor Temperature Progress" mode

   e. Returns the chamber to normal pressure.
      - **Set QD PPMS/DynaCool** in "Do Not Monitor Temperature Progress" mode

   The user may loop around these five steps to create a temperature profile.

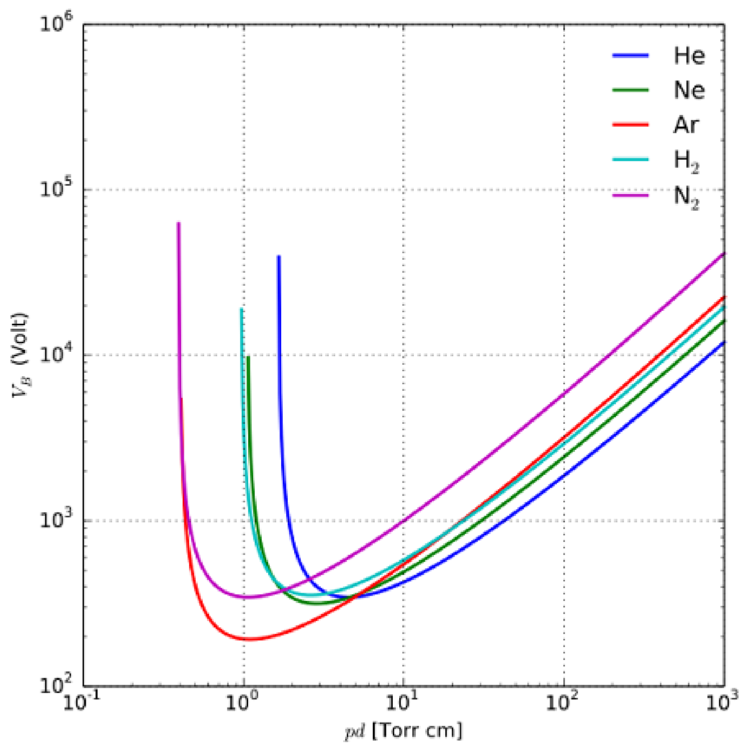
Appendix A

Theory of Voltage Breakdown of Helium Gas

Friedrich Paschen published a paper in 1889 describing the measurements he had performed on electrical breakdown of various gasses. [*Annalen der Physik* V273, Issue 5, 1889 pp69-96] Paschen found that the breakdown voltage is independent of temperature but does depend on the pressure of the gas, the type of gas, and the distance between the electrified metal plates in the gas. Below is a plot of his results copied from Wikipedia Commons under the Creative Commons License.

["Paschen curves" by Krishnavedala - Own work. Licensed under CC BY-SA 4.0 via Wikimedia Commons - http://commons.wikimedia.org/wiki/File:Paschen_curves.svg#mediaviewer/File:Paschen_curves.svg]

Paschen's work was so thorough that even today university engineering or science students are provided the necessary equipment and tasked to reproduce Paschen's results as a lab exercise. It is a difficult experiment to conduct accurately so Paschen's results still stand.



Reproduction of Paschen's Curves

Normal isotope-mixed helium condenses at 4.2 Kelvin at one atmosphere. Quantum Design uses helium in its PPMS and DynaCool systems to cool the sample and its chamber to as low as 1.9 Kelvin but must resort to special techniques below 10 Kelvin. Since the complexity of operating the PPMS or DynaCool chamber increases significantly below 10 Kelvin, Radiant has established 10 Kelvin as the lower limit for use of the High Voltage Cryogenic Probe.

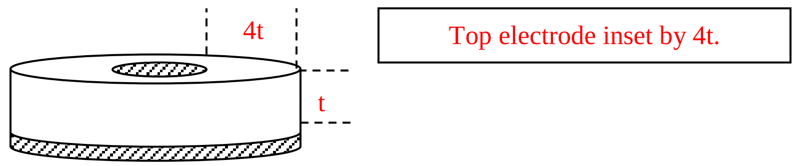
The Quantum Design chambers operate at a few Torr of pressure, well below that of ambient room pressure. This pressure places operating limitations on the operating points of the Sample Holder and the sample to prevent electric breakdown of the helium atmosphere. Helium is the blue curve of the Paschen measurements plotted above. The X-axis of that plot is in units of Pressure x Distance (between electrified plates). The helium trace hits its lowest value at roughly 3 or 4 *Torr-centimeters*. The breakdown voltage skyrockets if the pressure-distance goes much lower. At values above the minimum pressure-distance, breakdown voltage in helium increases at roughly a log/linear pace. This shape of Paschen's helium breakdown curve creates surprising limitations for the sample and the test fixture.

<u>Electrode-to-Electrode Distance</u>

Most samples to be measured in a Quantum Design chamber will have sample thicknesses on the order of 0.01cm to 0.1cm. If the electrodes of the disk capacitor extend to the top and bottom edges for the disk, the electrode separation through helium will be the same as the thickness of the dielectric material. Dividing the minimum breakdown voltage for helium at 4 Torr-cm by the sample thickness indicates that the lowest electrical breakdown voltage through helium *around the edge of such a disk capacitor* would occur between 40 Torr to 400 Torr of ambient helium pressure. It is unlikely that electrical measurements of the sample will be executed with the PPMS or DynaCool chamber at these pressure levels. When the HVCP is first inserted into the chamber, it can be tested at room pressure near 760 Torr. During cryogenic cooling with helium, the Quantum Design chambers operate between 1 and 10 Torr. Calculating the operating points on Paschen's helium curve for 7 Torr pressure with sample thickness between 0.0 cm and 0.1 cm yields *breakdown voltages above 10,000 volts.*

$$\text{Operating Point (100µm thickness)} = 0.01\text{cm} \times 7\text{ Torr} = 0.07\ Torr - cm$$
$$\text{Operating Point (1mm thickness)} = 0.1\text{cm} \times 7\text{ Torr} = 0.7\ Torr - cm$$

Note that the operating points calculated above are determined *by the distance between the electrodes,* not by the sample thickness. The helium-gap is defined here as the minimum distance through helium between the sample electrodes. If the sample's electrodes do not extend to the disk edge, the helium-gap *increases*. This geometry actually *decreases* the helium breakdown voltage. For instance, suppose a *1mm-thick* capacitor is electroded with a smaller electrode diameter on one side in an attempt to raise the helium-gap breakdown voltage. The geometry is graphed below.



For this electrode geometry, the helium-gap between electrode edges is 0.5 cm. The operating point becomes

$$\text{Operating Point (100μm thickness)} = 5 * 0.1\text{cm} \times 7\text{ Torr} = 3.5\, Torr - cm$$

This 3.5 Torr-cm operating point is actually near the point of the lowest breakdown voltage for helium at that pressure, around 400 volts. Even though the helium-gap between electrode edges is made *longer* by insetting the top electrode, the breakdown for this sample is *lower* than the same sample with its electrodes extending *to the edge* on both surfaces!

The worst case electrode edge separation between 1 and 10 Torr of helium pressure is 1 centimeter. The sample diameter is limited by the space available inside the Sample Holder to approximately 1 centimeter. Electrode edges on a sample that fits in the sample holder can therefore never be further apart than 1 centimeter. Thinner samples electroded to their edges are best.

Sample Holder Breakdown Limits

The HVCP fixture has electrified metal inside the Capsule during testing. The metal electrodes inside the HVCP Sample holder generally sit 1 to 3 centimeters apart, putting their breakdown operating point in the Torr range. The interior volume of the Capsule was designed with baffles and fences to increase the path distances between electrified metals. If the design and operating limits of the DUT and its electrodes are above those of the Capsule, breakdown through helium between metal parts inside the Capsule set the maximum test voltages allowed in the fixture specifications.

Conclusions from Paschen's Curves

1. Cryogenic chambers tend to operate at the helium pressure with the lowest breakdown voltage for a 1 centimeter distance between electrified metal.

2. Larger gap distances increase the breakdown voltage exponentially. Gap distances of 0.1cm or smaller *will not breakdown* below 10,000 volts at the helium pressure for optimal cooling used in the Quantum Design chambers.

3. The metal parts inside the small HVCP Capsule are no more than 1 to 2 centimeters apart so they set the absolute maximum operating voltage of the fixture above which helium breakdown between metal components *of the fixture* will always occur independent of what the sample does.

4. The helium-gap distance between electrodes on any sample must be 1 millimeter or less in order to exceed the helium-gap breakdown limits of the Capsule.

5. If the helium-gap distance between electrodes on any sample is greater than 1 millimeter, the voltage specification for the fixture will decreases to approximately 400 volts.

6. Samples can suffer through-the-dielectric-material breakdown independent of the helium-gap distance for its electrodes or any specification for the fixture.

Appendix B

Recognizing Breakdown Events

If a helium-gap or a sample breakdown occurs during a test, unique features are introduced into the plot of the test results. This section describes how to recognize breakdown events in measurements.

For all examples in Appendix B, an inert “no-load” sample was tested at voltage in the fixture. A “no-load” sample is a piece of plastic or Teflon with no electrodes. Only a tiny capacitance is formed between the two copper contacts of the Sample Holder which are held separated by the no-load sample. Since the no-load sample itself will not break down electrically, there are only two paths for breakdown to occur. The first is by conduction along the surface of the sample from the bottom fixture electrode around the edge of the sample and back to the top fixture electrode. In a dry environment, this conduction will be nearly invisible but its occurrence can increase dramatically if liquid “helium dew” condenses on the sample or Capsule surface. The second source of breakdown is by helium gas ionization from the “hot” electrode to a grounded piece of metal.

Normally, the bottom electrode of the fixture is at high voltage while the top electrode is at virtual ground. When high voltage is applied to the bottom electrode of the fixture, only that circuit is charged *while all other metal pieces in the fixture and the chamber will be at ground potential*. Therefore, a breakdown does not necessarily have to be only between the top and bottom electrical contacts of the no-load sample. The PPMS chamber and all metal parts of the HVCP are connected to earth ground so there is no danger of injury to the operator if a high voltage breakdown occurs from the fixture to the chamber. Nevertheless, the voltage at which such events occur will limit the maximum voltage that may be applied across the sample for a successful measurement.

Regardless of the *path* of the breakdown, there are two *types* of breakdowns through the helium evidenced in the measurement by a Radiant tester. The first type acts like a wire. When breakdown occurs, the ionized helium will conduct more current through the breakdown path than can be supplied by the output amplifier of the test instrumentation. Such a high current flow will pull the output voltage of the amplifier towards ground and appear as a zigzag in the data. Therefore, any sudden deviation of the output voltage of the tester or its high voltage amplifier *from the assigned waveform* should be considered indication of a breakdown. These deviations fall into two categories:

1. Hard Breakdown: The ionization path for the breakdown is so strong that it remains ionized for long fractions of the stimulus profile. Or, the ionization path evaporates, the amplifier returns to the breakdown voltage, and the breakdown occurs again immediately. This type of breakdown is evidenced by severe distortion of the output waveform during the test accompanied by multiple spikes in the voltage profile.

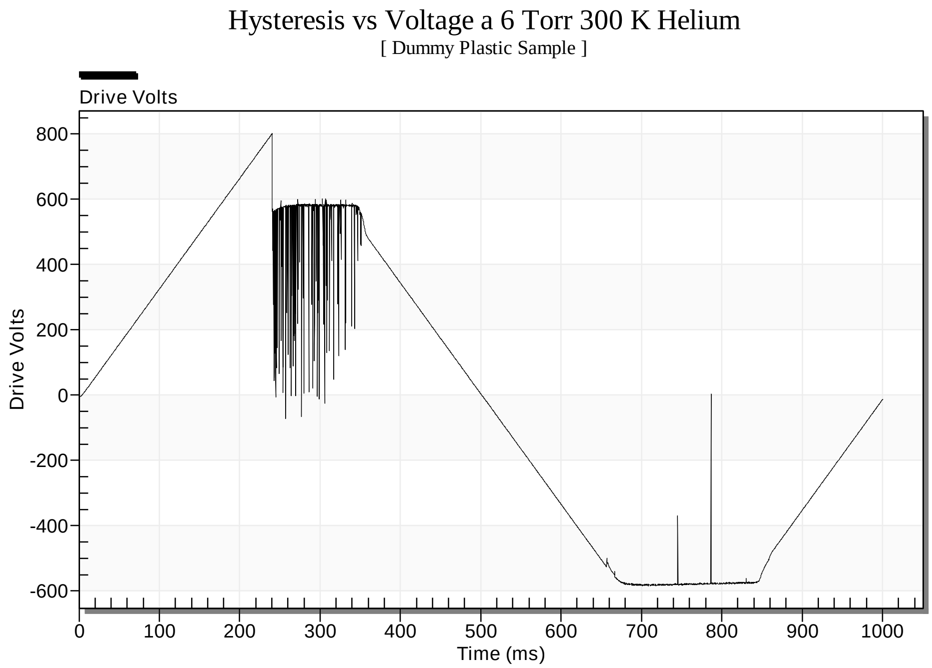
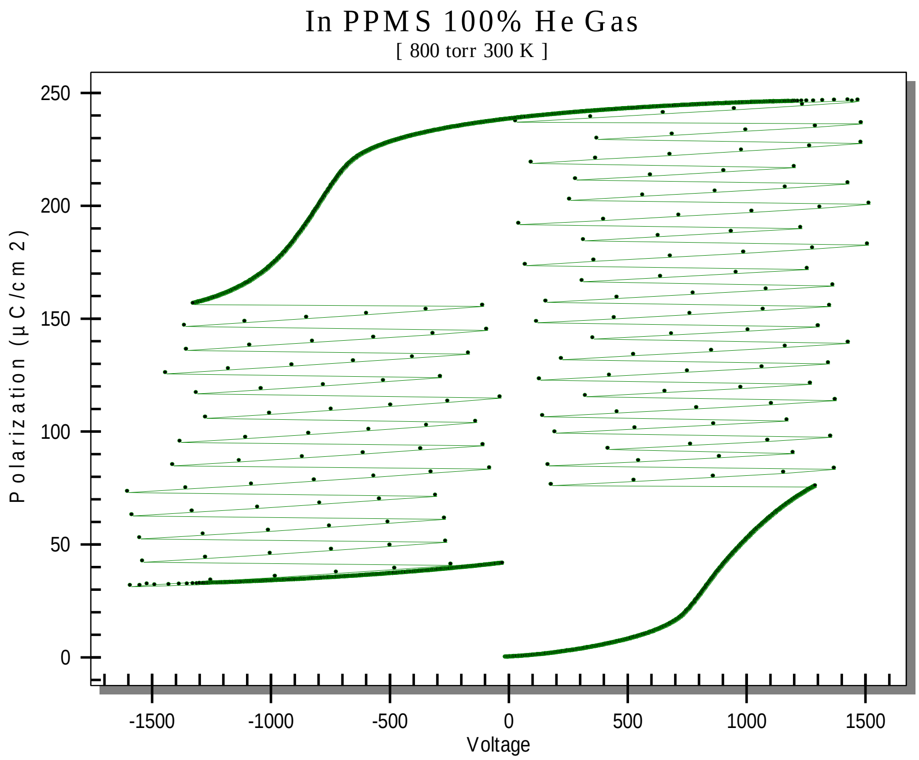


Fig. 1: Hard breakdown in 6 Torr 300 K helium
(*Quantum Design PPMS Verification 02_28_2017:::Hyst vs Voltage @ 760 Torr 300 K Helium 4000V 100V steps:0*)

A ferroelectric hysteresis beset by a hard breakdown is a sight to behold.



Note that the breakdowns in the plot above *where through the helium.* The sample remained undamaged despite the dramatic appearance of the plot. The voltage assigned for the test was simply above that which the helium could handle.

2. Soft Breakdown: The ionization path is catalyzed by a foreign material that itself is vaporized by the breakdown plasma. Soft breakdowns are fast and end immediately, allowing the high voltage amplifier to return its output to the assigned voltage without further problems. Soft breakdowns occur *below* the hard breakdown limit of the gas or the sample. A soft breakdown appears as a single spike or a few spikes in the voltage profile and usually does not re-appear during a repeat of the same test. See the three sequential tests below for an example.

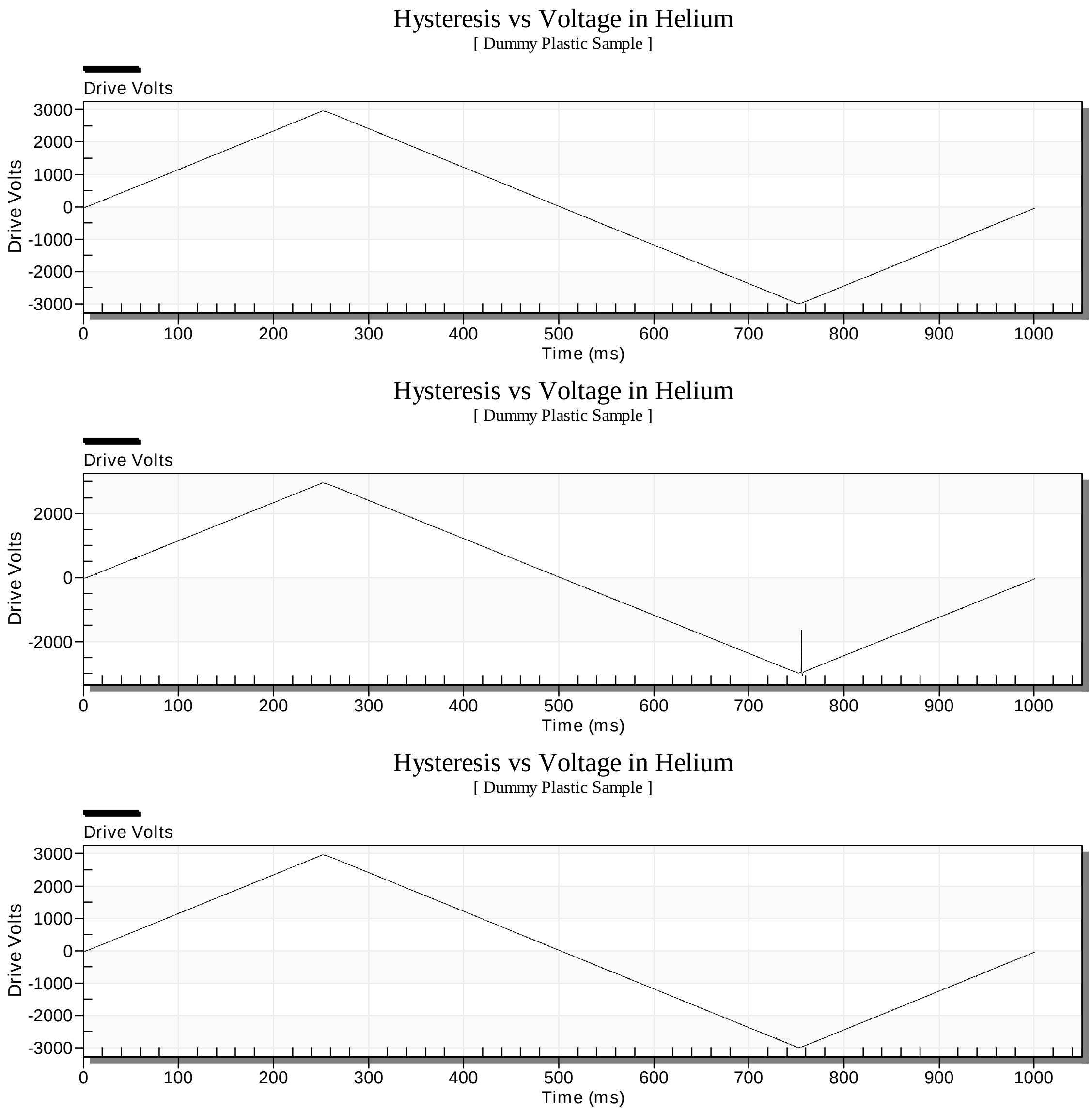


Fig. 2: One soft breakdown out of three tries in 1mTorr 26.4 K helium.
(Quantum *Design PPMS Verification 02_28_2017::: Hyst vs Voltage @ 1m Torr 24.6K Helium 3.0kV 1s:0*)

Inside the PPMS chamber, the helium gas should have no moisture content but itself can condense on the sample or fixture surface under certain operating conditions. If this happens, breakdown voltages will plunge but they should be soft breakdowns. The heat carried away from the sample fixture by the hot helium gas by such breakdown events will appear on the temperature monitor of the chamber.

Given the nature of electrical breakdowns expected inside the PPMS chamber, there will be three voltage regions for operating the fixture:

1. The clean operating area having low breakdown probability.

2. A gray band between the clean operating area and the hard breakdown limit where soft spike breakdowns occur sporadically but not repeatably on the same test.

3. The hard breakdown limit.

Paschen found that breakdown varies qualitatively by the composition of the gas as well as by pressure. Breakdown voltage will be lowest in the Torr range of pressure but increase dramatically at pressures either higher or lower than the Torr range. These conditions create multiple operating envelopes for the fixture specification based on pressure.

1. Dry air at 760 Torr

2. Helium at 760 Torr (300 K – equivalent to loading the fixture into the chamber and flooding it with helium.)

3. Helium at the optimal cooling pressure for the chamber (~5 Torr)

4. Helium in high vacuum. (<1 Torr)

Real Sample Breakdowns

With a real sample in place, the breakdown environment becomes more complex. There are new possibilities:

1. Soft breakdown of the helium.
2. Hard breakdown of the helium.
3. Soft breakdown *through* the sample.
4. Hard breakdown *through* the sample.

Through-the-sample breakdowns generally follow the same rules but their appearance, speed, and current densities are different than for helium because the conducting medium is a plasma *of the constituents of the dielectric material and the electrodes.* The question during testing becomes:

> *Was that breakdown through the sample or across a helium-gap*?

In actual samples, hard breakdowns burn through the sample while depositing melted electrode metal along the way and rendering the sample unusable. Soft breakdowns are real breakdowns through the sample but they heal themselves so the sample is still functional after the test but is microscopically reduced in area. Typically, both types of breakdowns occur through pores in the material and appear as black spots on the sample after they have burned themselves out. The appearance of soft breakdowns with real samples, however, is an indication that the hard breakdown voltage for that sample is being approached and could happen on the next test if the voltage is not lowered.

Through-the-sample breakdowns are a function of the sample so the fixture cannot prevent it from happening. However, it is one of the objectives of the HVCP Fixture to prevent damage to the PPMS/DynaCool chamber, damage to the tester, or injury to the operator should a sample burn up during a test. If the HV Sample Fixture can prevent damage to the cryogenic chamber from hard *gas ionization* breakdowns of the no-load sample, it will do the same for hard breakdowns of real samples by the same protection mechanisms already discussed. Radiant's testers have extensive internal protection against damage from hard sample breakdowns but cannot handle every possible situation. Sometimes a breakdown can damage a tester. In that case the customer must return the tester to Radiant for repair.

It is important to note that it is not possible to distinguish between soft breakdowns due to condensation on the sample and soft breakdowns *through* the sample. The operator will have to develop a sense of which is which from experience with multiple examples of that sample type. Hard breakdowns can be distinguished. After hard breakdown due to gas ionization, the sample will still be functional at the same voltages. A hard breakdown *through the sample* always results in a shorted sample.

<u>Breakdown Effects on Polarization</u>

A breakdown transfers a large pulse of charge along the breakdown path. This pulse of charge may or may not show up in the polarization results depending on the type of breakdown. The RETURN input of the tester simply counts the electrons coming and going. It cannot see any charge that does not enter the RETURN.

1. Gas ionization between an electrode of the fixture or the sample and earth ground.
    a. The DRIVE voltage drops due to the high current flow but polarization is unaffected because no electrons carried by the arc go into the RETURN input of the tester.

2. Gas ionization between the two electrodes of the sample.
    a. This helium-gap gas ionization creates a wire between the DRIVE and RETURN terminals of the tester, delivering a huge number of electrons into the RETURN. This population of charge will almost certainly saturate the measurement circuitry of the RETURN input and cause a Vision error.
        i. If in Autoamplification, Vision will repeat the test at various amplification levels until it achieves a valid measurement.
        ii. If in Autoamplification and the breakdown occurs on every test, Vision will continue to lower the amplification level of the tester and repeat the test until it reaches the lowest amplification level and report an error.
        iii. If Autoamplification is turned off, Vision will return with an error stating that the sample is too large.

3. The sample breaks down soft through its thickness between electrodes
    a. The breakdown creates a high resistance wire between the electrodes. Current flow through the plasma will heat it to the point that dielectric material and electrode evaporate, leaving a non-conductive hole in the capacitor area.

    b. Current flow through the plasma will be restricted significantly compared to a helium-gap breakdown *but will flow into the RETURN input*. The test is likely to complete but with a *step* somewhere in the hysteresis curve representing the count of the temporary surge of electrons into the RETURN.
        i. There will be one "step" in the hysteresis curve for each soft breakdown.
        ii. The sample will remain usable after a soft breakdown but the test voltage may need to be lowered to prevent more from occurring.

4. The sample breaks down hard through its thickness between electrodes
    a. The breakdown creates a low resistance wire between the electrodes. Current flow through the plasma melts the electrodes so they flow through the hole, leaving a permanent short.
        i. If in Autoamplification, Vision will continue to lower the amplification level of the tester and repeat the test until it reaches the lowest amplification level and report an error because the sample is hard shorted.
        ii. If Autoamplification is turned off, Vision will return with an error stating that the sample is too large.
        iii. The sample will remain unusable after the breakdown.

Appendix C

Example Test Results

In Air
The fixture was tested in air at Radiant in Albuquerque, NM up to 10,000 volts with a no-load sample with no hard breakdowns occurring. Albuquerque typically has 15% relative humidity (RH) during that time of the year. Testing for hard breakdown resistance by the fixture in the dry air of New Mexico verified the fundamental design of the fixture and the insulated paths to/from the fixture through the top head of the HVCP. Because of the properties of helium, hard gas breakdown in helium should be no better than half the voltage achieved in dry air.

Breakdown tests in the humid (55% RH) air of Southern California might cause soft breakdowns below 10,000 volts. Soft breakdowns did occur in San Diego in air at 9,500 volts and 10,000 volts using the no-load sample. No breakdowns of either type occurred at 9,000 volts or lower. (See *Quantum Design PPMS Verification 02_28_2017.dst*)

In Helium
After hanging the fixture in air for testing, the HV Sample fixture was inserted into the PPMS and tested for breakdown at various pressures and temperatures, all in pure helium. The data below were taken are in *Quantum Design PPMS Verification 02_28_2017.dst*.

If all breakdowns that occurred with the no-load sample are plotted as a function of temperature, no pattern arises in the results.

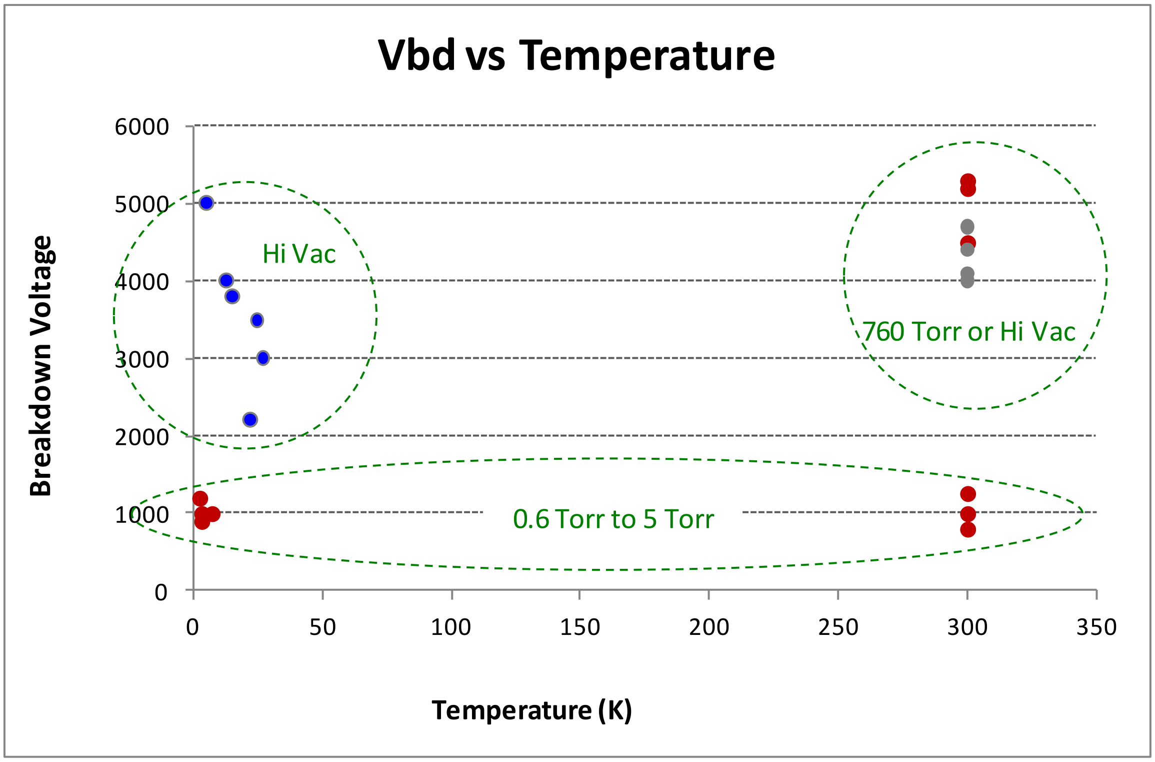


Fig. 3: All breakdowns in helium as a function of temperature.

The **red** points in Figure 3 are hard breakdowns. The **gray** points are soft breakdowns. The **blue** points are also soft breakdowns but at cryogenic temperatures. The wide distribution of hard breakdown events in Figure 3 is in keeping with Paschen's prediction that breakdown voltage is not a function of temperature.

The **blue** points are interesting. They occurred during a short period towards the end of the second day of testing when the sample temperature was rising from 2.2K in High Vacuum mode. With such a low pressure of helium, there was not enough cooling power to maintain the cryogenic temperatures against heat leakage down the fixture from its head sitting in the ambient atmosphere above the PPMS. Measurements continued every few degrees as the temperature rose on its own. The soft breakdowns associated with this one test condition were at far lower voltages than any that had occurred up to that point. It is possible that the higher temperature of the helium gas against the cooler sample caused helium condensation on the sample and fixture or otherwise modified the local surfaces to increase the possibility of a soft electrical breakdown at lower voltages. Removing those soft blue breakdowns in Figure 4 modifies the plot to show that at high vacuum or at high pressure the hard breakdown specification for the fixture should be ~4,500 volts while the specification at the optimal cooling pressure in the Torr range should range between 800V and 1200V. Soft breakdowns can occur in a small range below the hard limits.

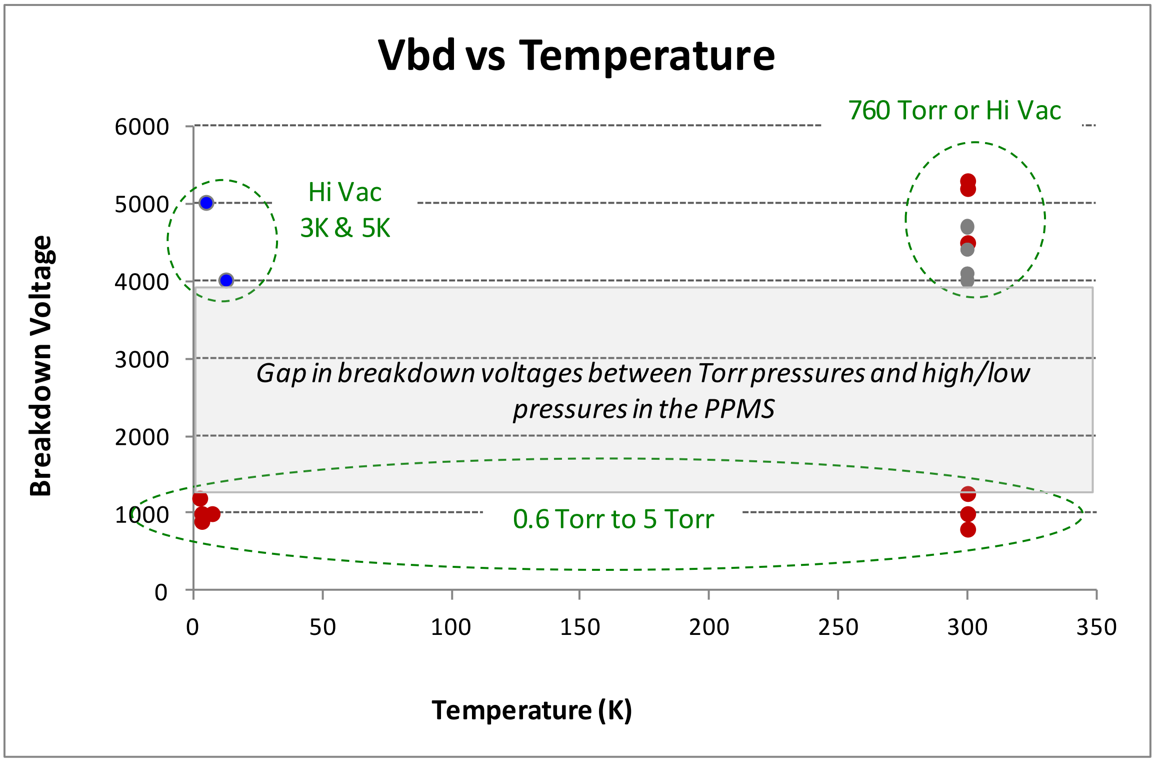


Fig. 4: All breakdowns in helium not associated with a rising sample temperature in high vacuum.

Re-plotting the same data in Figure 3 as a function of pressure shows a breakdown function geometry predicted by Paschen's Law.

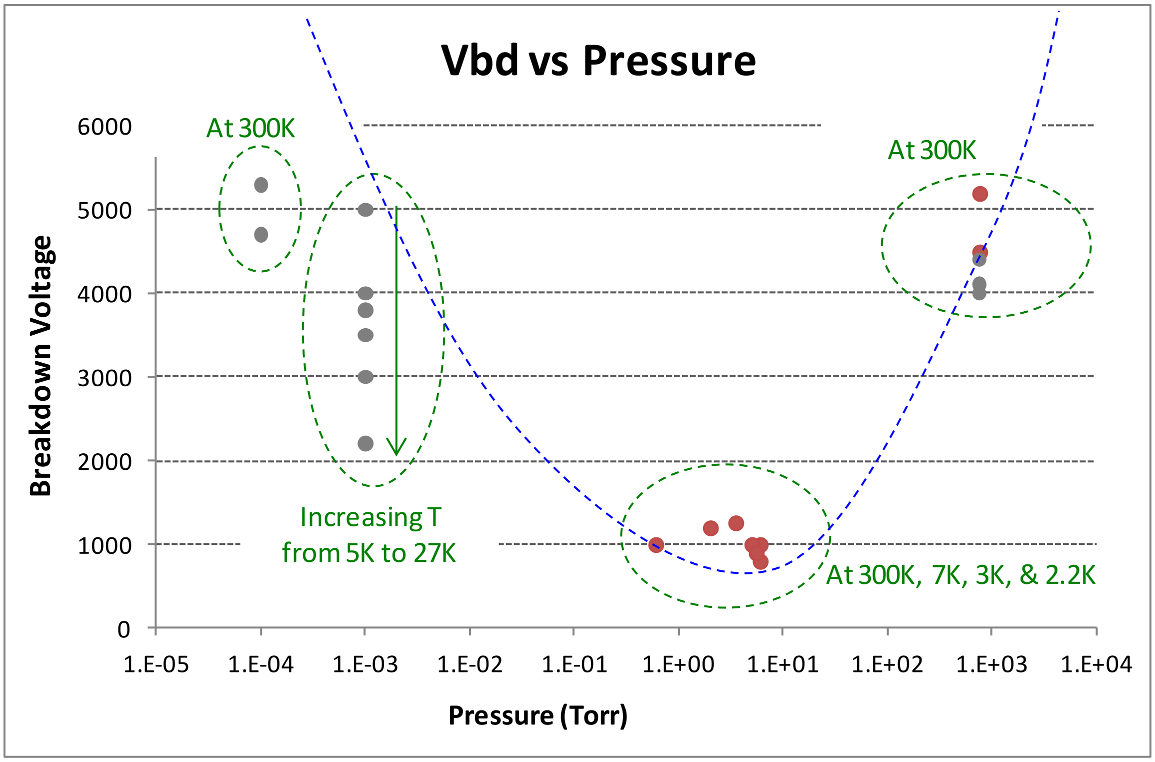


Fig. 5: All breakdowns in helium as a function of pressure.

Hard breakdowns reach a minimum voltage around 5 Torr but that voltage limit increases dramatically at atmospheric pressure. Only soft breakdowns occurred during testing in the high vacuum mode up to 5300 volts. The **blue** dashed line is only a hand-drawn *estimate*Paschen's Law and is not calculated by the Law. These results allow a prediction of the operating envelope for the fixture in Figure 6.

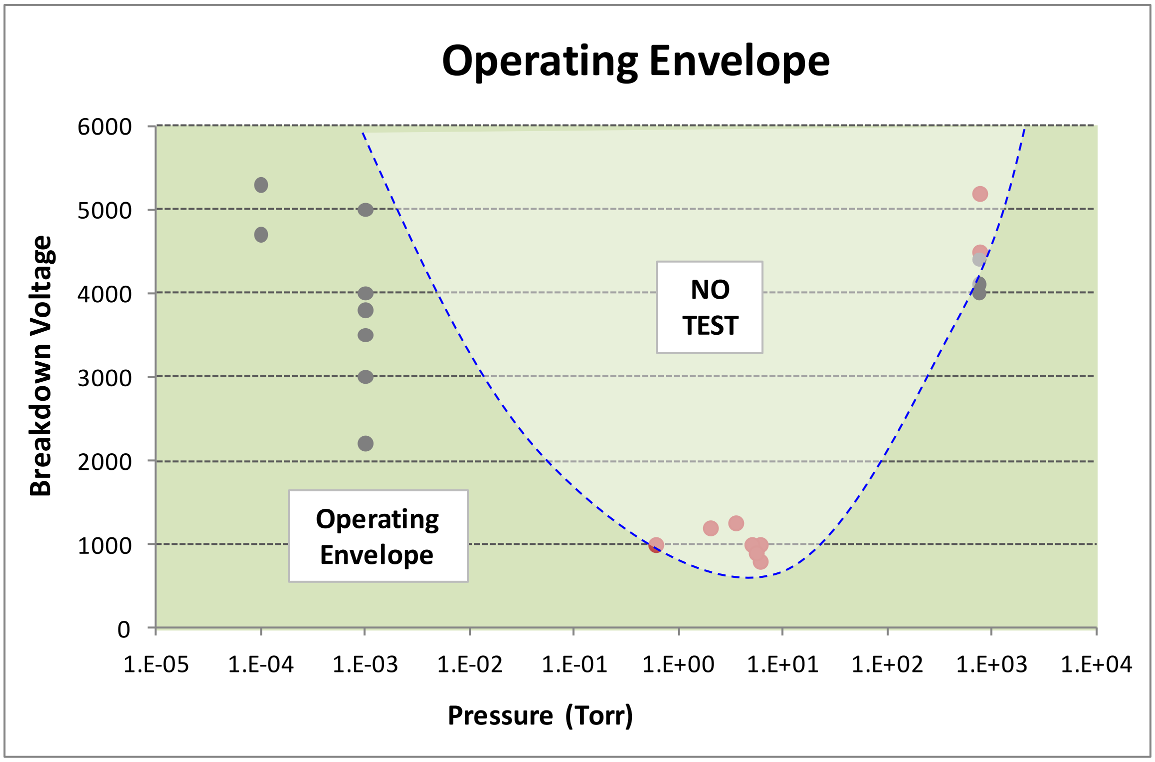


Fig. 6: Predicted Voltage-Pressure envelope for successful high voltage testing in the PPMS.

The bottom of the **No Test** region will vary in voltage according to the helium-gap characteristics of the sample and the fixture. Consistent operation up to 1200 volts can be achieved by careful design of the sample.

CTS HD3241 Ceramic Piezoelectric Actuator

On March 3, 2017, a PPMS chamber was used to measure a 3241HD piezoelectric sample type from CTS Wireless. Another capacitor fabricated in the same batch had been measured previously by Radiant in a PPMS in October 2015. The measurements from 2015 ranged from 100 K up to 300 K. Two additional measurements were acquired from the second sample in 2017 at 396 K and 73 K. Those two measurements combined with the original 2015 measures are plotted below in Figure 9.

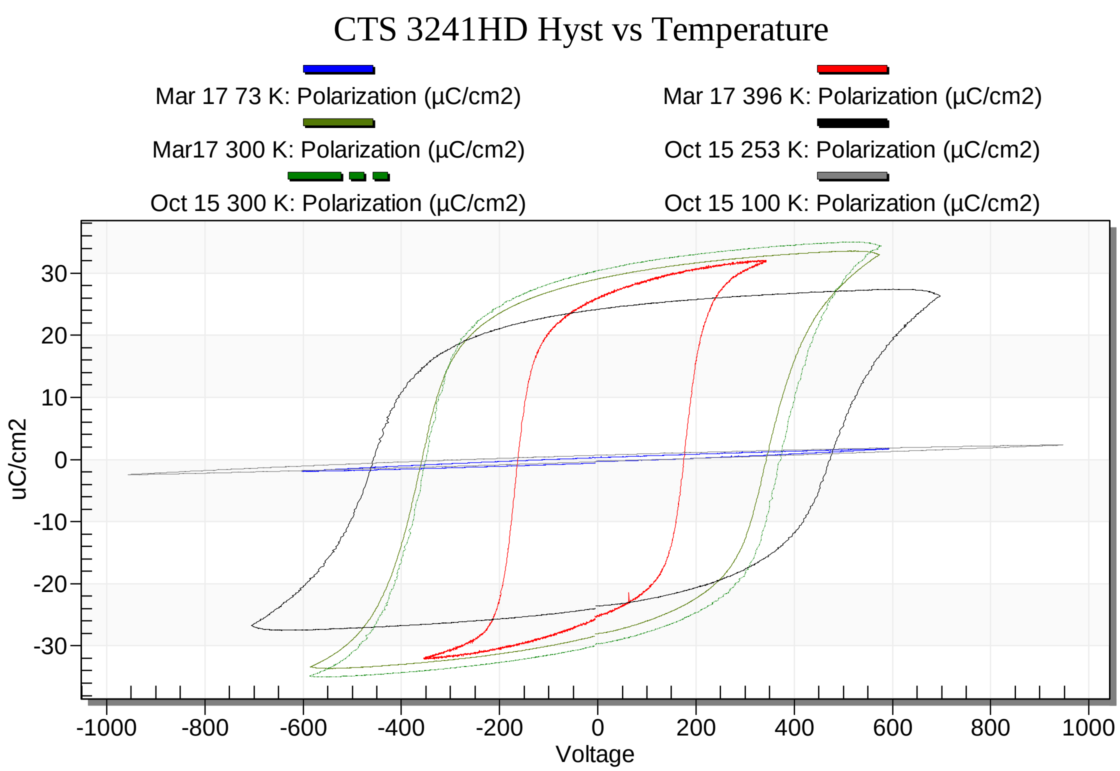


Fig. 9: Hysteresis measurement of 3241HD from 2015 and 2017.

There were slight differences between the two samples. The **green** traces in Figure 9 were taken at 300 K on both samples in helium. The solid green hysteresis loop is the most recent. The dashed loop is from 2015. There is a difference between the two loops but it is slight. This is typical for ceramic capacitors from the same batch to vary by this amount. The other difference between the samples is highlighted by the **blue** trace. It is the 73 K measurement made in 2017 at 600V. The **gray** trace in the plot is of the 100 K measurement at 950V from 2015. The two overlay as they should. However, a 950V test of the newer sample caused a hard breakdown *of the sample* and it could not be tested further. That hard failure might have been caused by a defect in that sample or it might have been an effect of the lower temperature, 73 K versus 100 K. Paschen's Law dictates that the lower breakdown voltage did not occur due to helium ionization.

There are two interesting observations about the physics of the 3241HD samples. First, notice the soft breakdown in the sample at 396 K (in **red**). This single spike might have been the sample but it resembles all of the soft breakdowns seen with the no-load sample. Was it the helium or the sample? Only more measurements of more samples will tell the tale.

The second observation is about the lack of ferroelectric hysteresis at 100 K and 73 K for this PZT sample which is optimized for piezoelectric properties. The disappearance of the hysteresis loops at low temperature could be due to a phase change in the material. However, notice how quickly the coercive voltage of the sample hysteresis increases with decreasing temperature as predicted by a linear fit in Figure 10.

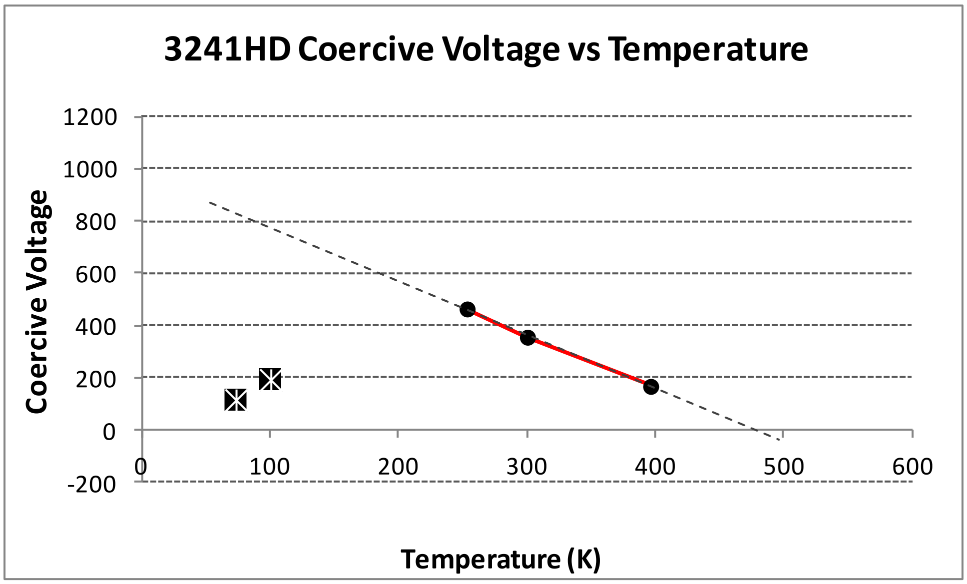


Fig. 10: Projected Hysteresis Coercive Voltage vs Temperature.

The two measurement points at 100 K and 73 K were well below the voltage predicted to be required to open the hysteresis loop for this material. The 800 volt test amplitude necessary to achieve hysteresis in the 3241HD sample is now within reach of the HVCP. Nevertheless, material properties of the sample may prevent reaching those voltages at those temperatures.

<u>Comparison to Thin Films</u>
Radiant has fabricated 20/80 PZT *thin films* and measured them down to cryogenic temperatures. It is much easier to measure these capacitors at cryogenic temperatures because of their low operating voltages. As well, Radiant uses a glass passivation atop the capacitors which prevents gas ionization above the capacitor top electrodes. The only breakdown path is through the thickness of the thin PZT.

The nested hysteresis loops for this material measured down to 10 K show an increase in coercive voltage but the rate of growth is different than for 3241HD.

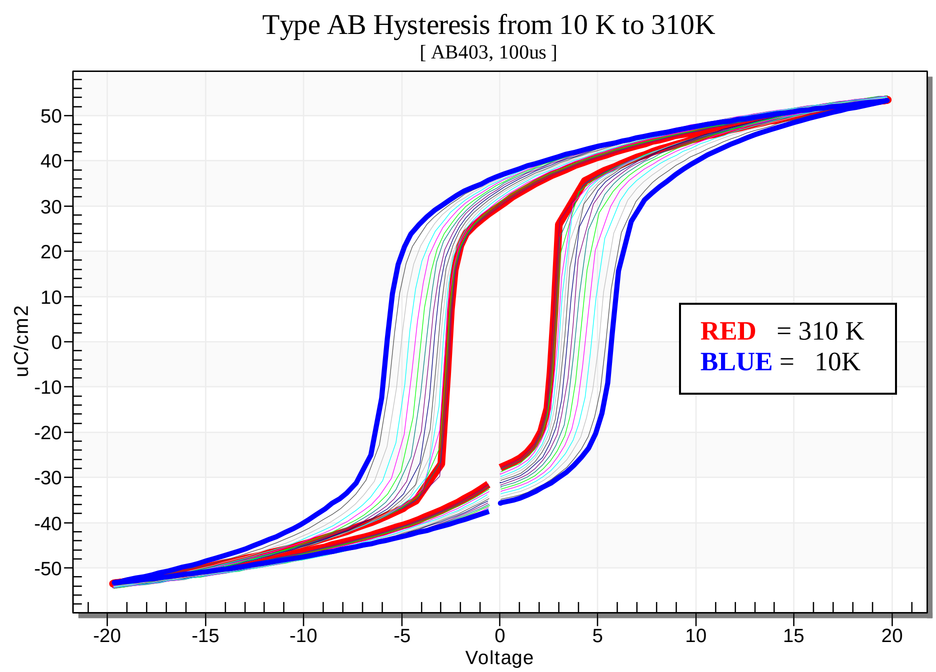


Fig. 11: 20/80 PZT Hysteresis vs Temperature.

Plotting Electric Field instead of Voltage versus Temperature allows the comparison of the thin film capacitor of one composition to the bulk ceramic capacitor of another composition. Both the 3241HD and thin PZT coercive field data are fit with a third order polynomial.

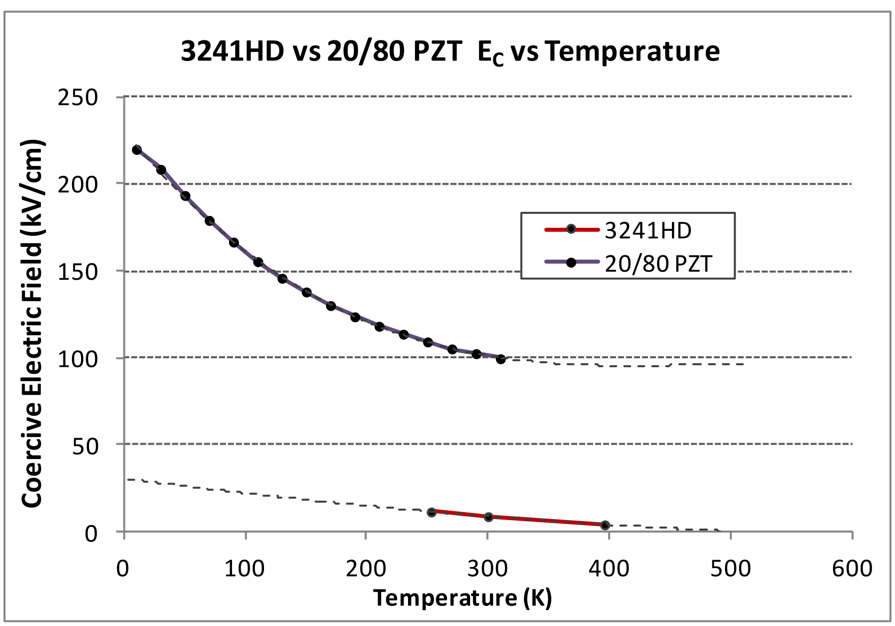


Fig. 12: Thin 20/80 PZT versus bulk 3241HD ceramic.

The polynomial fit predicts that very thin 3241HD samples will need to be fabricated in order to achieve hysteresis loops for this composition near 10 Kelvin without exceeding the voltage specification of the HVCP.

Finally, notice the large difference in coercive electric fields for the thin film capacitor versus bulk ceramic capacitor. This is true all for all thin films and bulk capacitors even of the same composition. This ambiguity in coercive electric field for bulk vs thin film ferroelectric material remains unexplained. This type of study is exactly the type of research made possible by the HV Cryogenic Probe.