High Voltage Cryogenic Hysteresis Loops

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Summary

- Ferroelectric materials have demonstrated surprising resilience at cryogenic temperatures, so far showing normal polarization hysteresis loops down to 5 K.

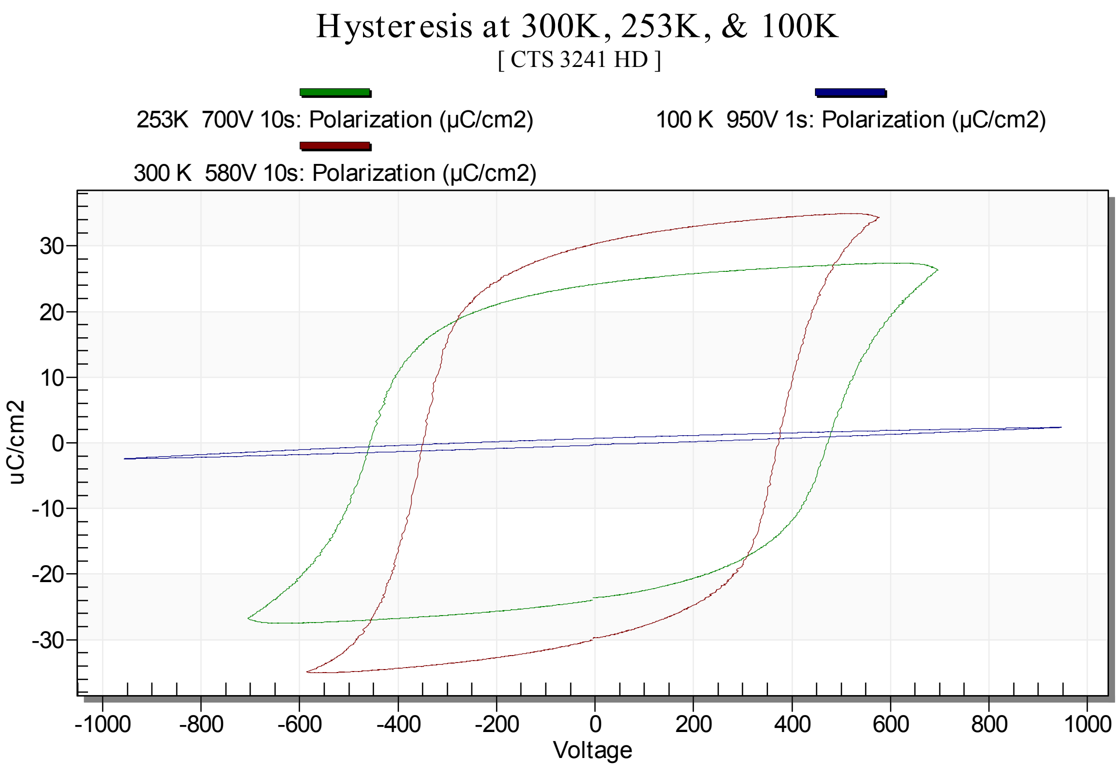
- Hysteresis and other electrical measurements requiring high voltage present special problems in cryogenic chambers with respect to noise, personnel safety, and helium breakdown.

- Successful measurement of non-linear materials at cryogenic temperatures can be accomplished safely through a combination of hardware modifications and changes in chamber operation.

Hysteresis over Temperature

Many surprises await at lower temperatures.

The sample to the right is a commercial piezoelectric PZT from CTS Wireless in Albuquerque measured in a PPMS that indicates a normal hysteresis loop at room temperature but unexpectedly has no remanent polarization at 100 K.



Contents

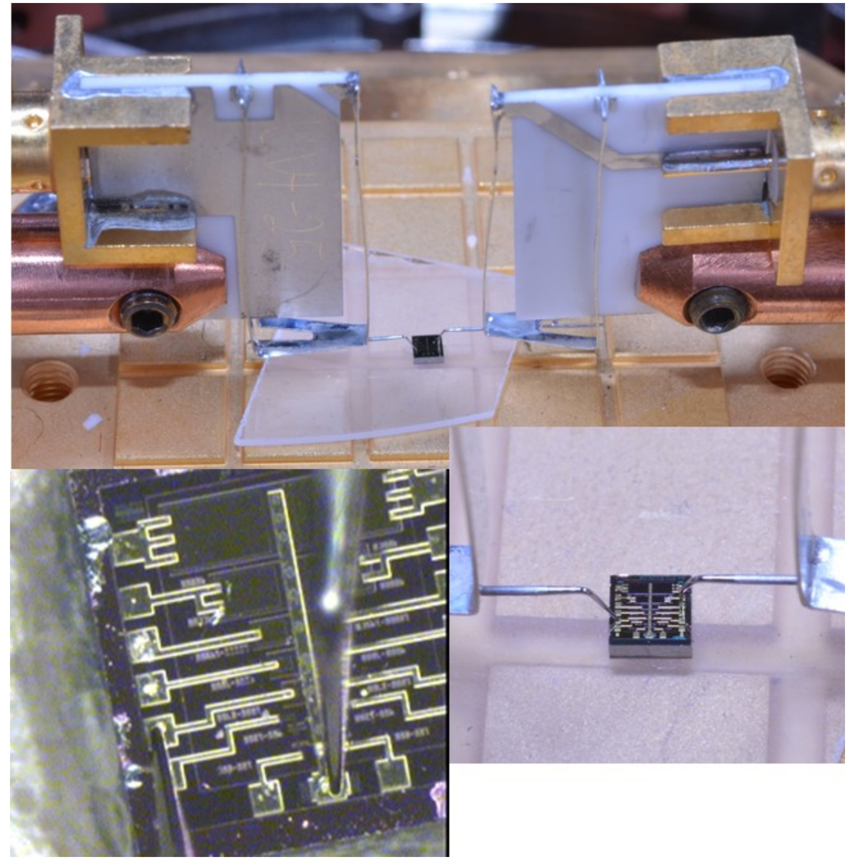
i. Expected material properties at cryogenic temperatures extracted from thin PZT film measurements.

ii. Issues and solutions facing the hardware design of an effective sample holder for the Quantum Design PPMS cryogenic chamber.

iii. Paschen's Law of electrical breakdown.

iv. Recommended chamber operation to prevent inadvertent breakdown at cryogenic temperatures.

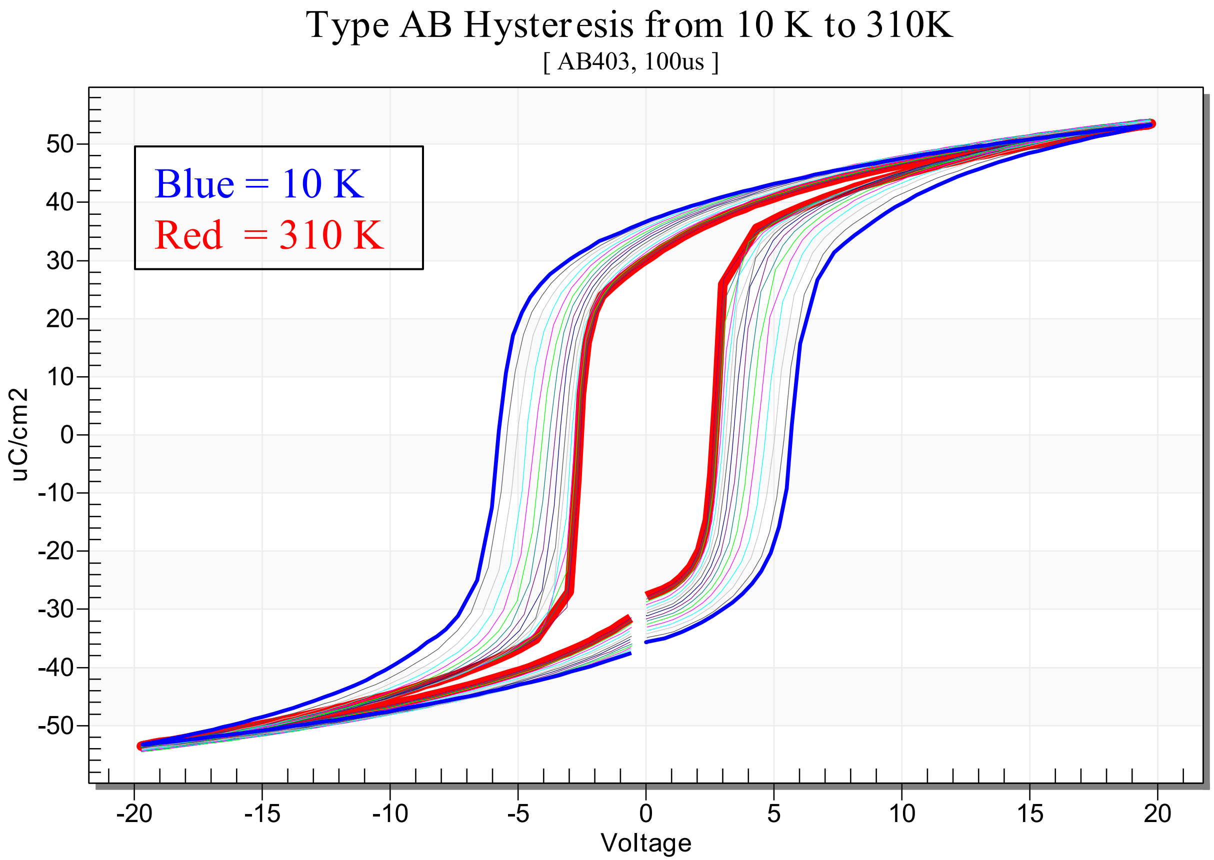
Cryogenic Probe Stations



Cryogenic probe stations such as the Lake Shore Cryotronics Model CRX-4K permit testing of thin ferroelectric films on small substrates from 350 K or higher down to ~6 K.

Hysteresis vs Temperature
40,000 µm^2 0.26m-thick 20/80 PZT

- ±20 volts with 100 microsecond period with temperature steps of 20 K each.

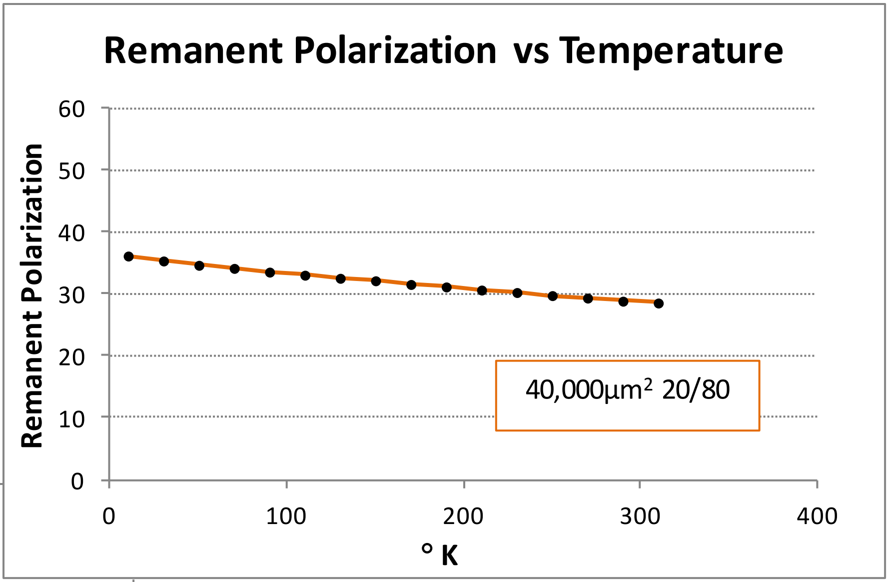


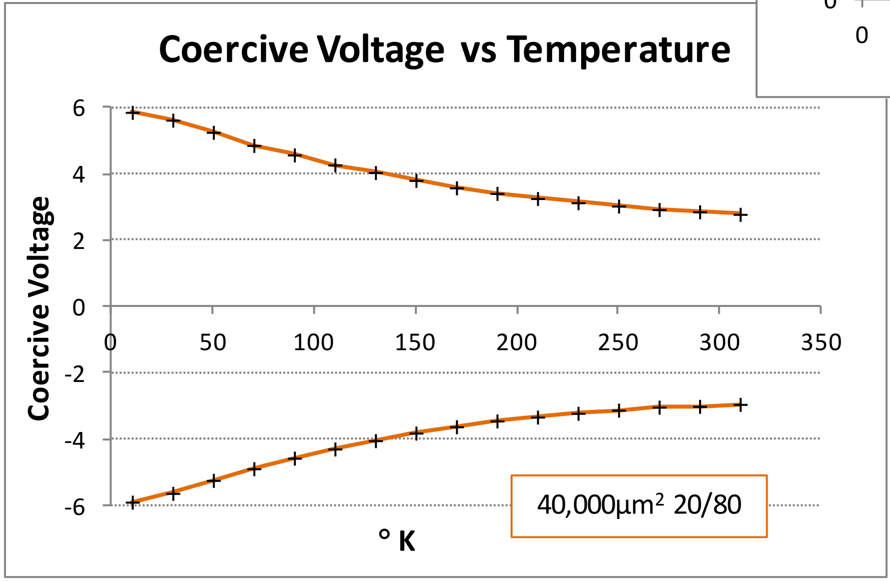
The faster test period of 100 µs prevented breakdown of the sample capacitor at 20 volts at room temperature.

Remanent Hysteresis vs Temperature

40,000 μm² 20/80 PZT

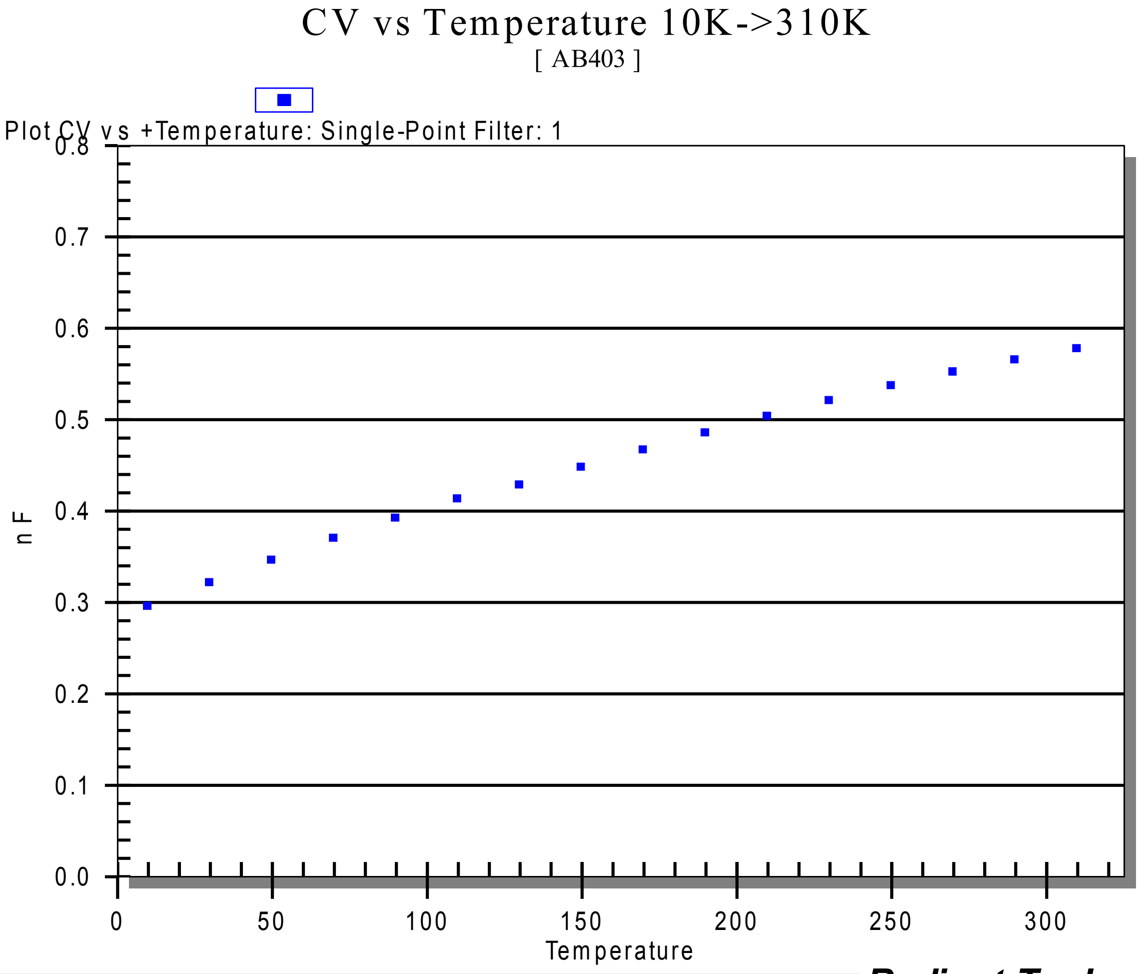
20 volts with 100 microsecond period.





Small Single CV vs Temperature
40,000 μm² 20/80 PZT

- 1 kHz with 0.2 volt amplitude at 0 volts bias.



Issues for Bulk Capacitors

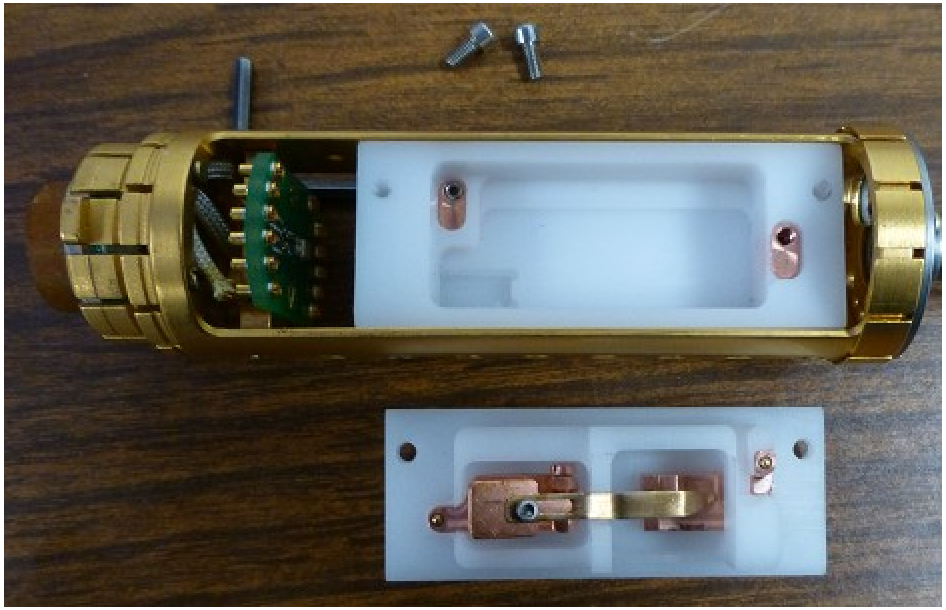
- Bulk capacitors cannot be tested on probe stations, cryogenic or not, due to the high voltages required.

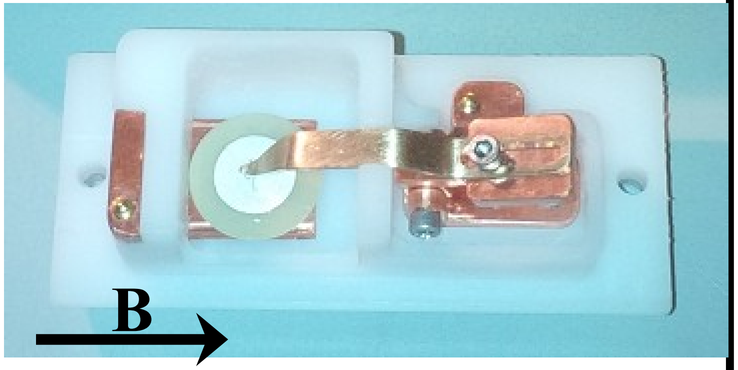
- They can be tested inside columnar chambers with cryogenic wells like the Quantum Design PPMS.

- Issues associated with cryogenic wells:
  - Tight space forces metal connectors into close proximity.
  - The sample must be oriented vertically to align with the magnetic field generated by the chamber for ME tests.
  - A thermometer must be set close to the sample but this exposes external instrument to possible breakdown surges.

Quantum Design PPMS

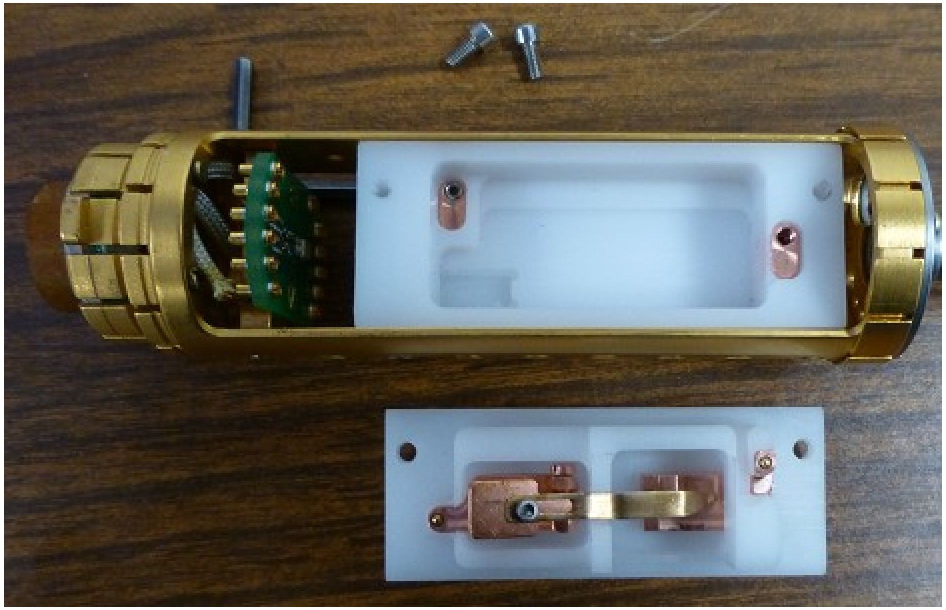
The QD Model P450 Multifunction Probe can be modified to hold a special high voltage fixture at its tip for testing disc-shaped bulk capacitors.





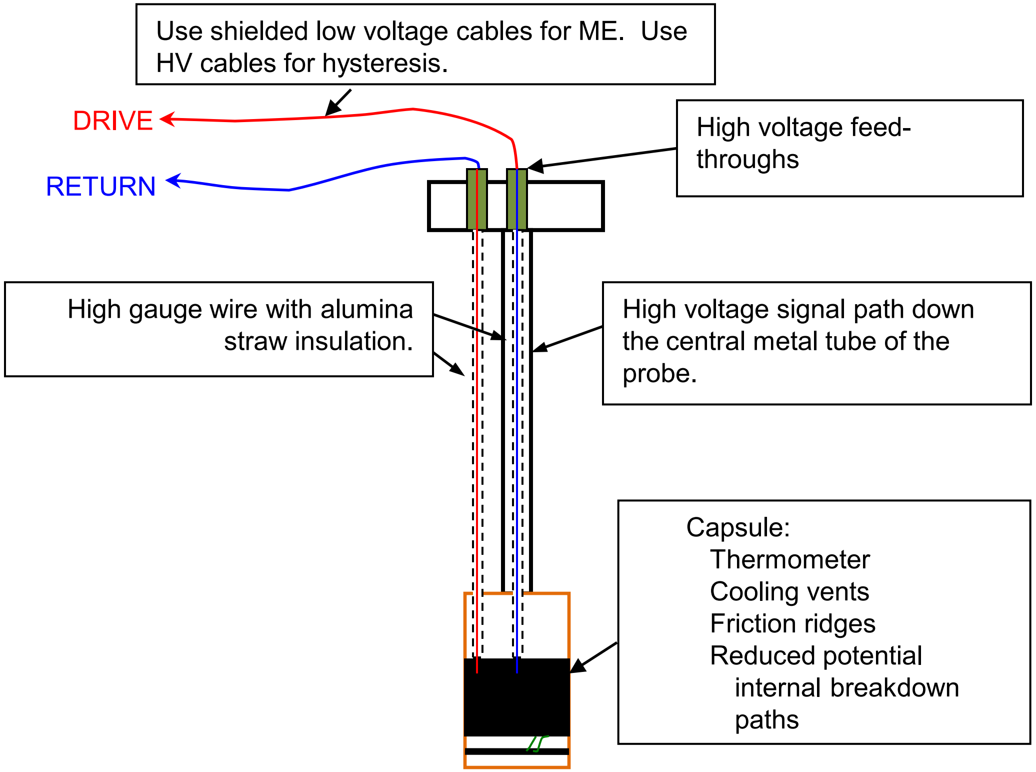
Sample Fixture

The sample holder should be fabricated from a material that provides insulation for the high voltage operation but does not deteriorate from the extreme conditions inside a cryogenic chamber. In this case, we selected Vespel™ as the insulator. It is a NASA approved low-outgassing polyimide suitable for use in extreme environments.



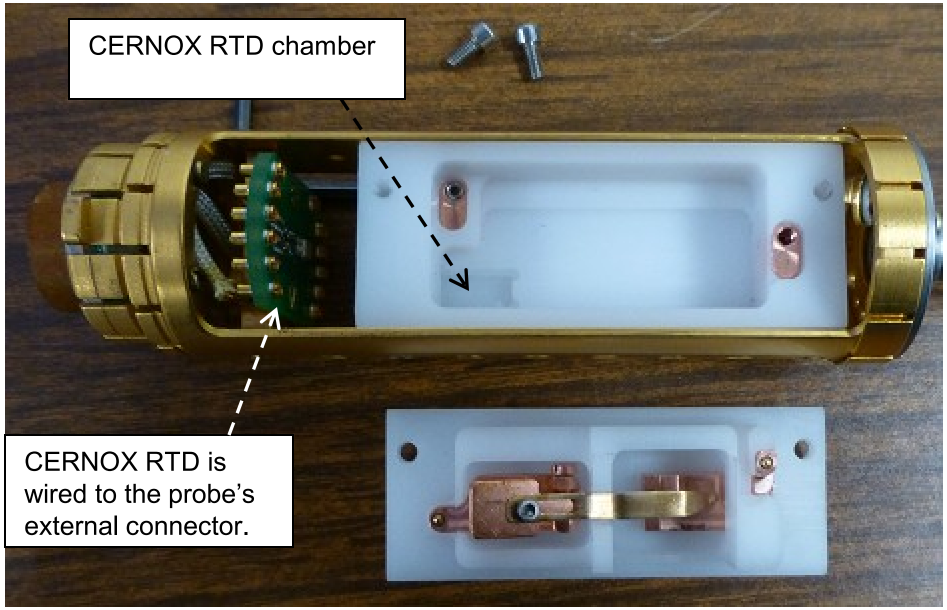
High Voltage Protection

It is desirable to avoid the use of traditional electric insulation material inside the chamber due to possible outgassing. Classic insulation is replaced with bare nickel wire inside alumina tubing. Other physical modifications are necessary.



Embedded Thermometer

A Lakeshore CERNOX thin film RTD thermometer is embedded in the fixture as close as possible to the sample to provide the most accurate indication of the temperature of the sample. However, the RTD is connected electrically directly to external instruments in the PPMS control system so it must sit behind a window insulating it electrically from the sample.



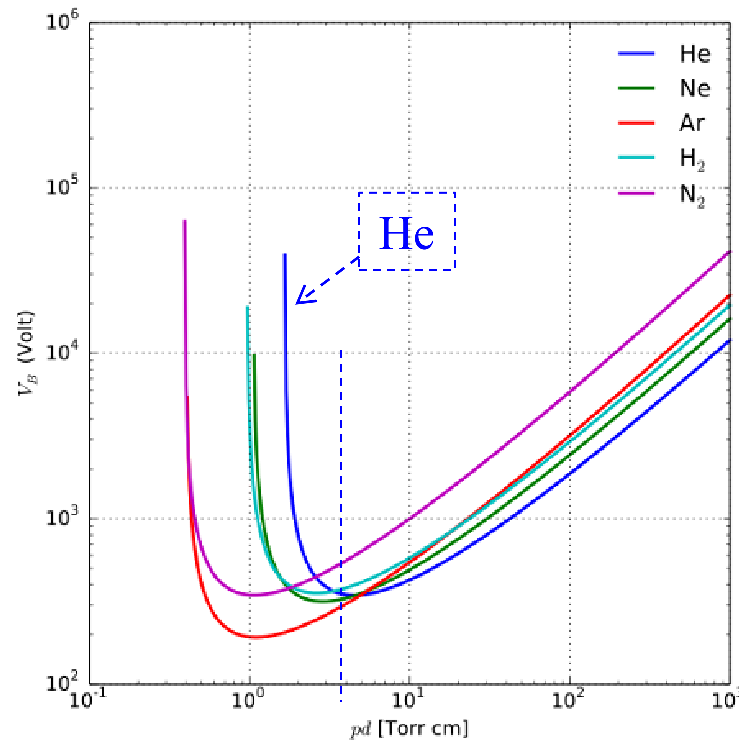
Hanging in air, this fixture easily handles 10,000 volts. No so in the chamber in a helium atmosphere. There the breakdown can be as low as 400 volts!

Paschen's Law

Despite all of the design factors intended to eliminate air-gap breakdown in the cryogenic fixture, it still happens as low as 400 volts when the helium pressure inside the chamber is set to 5 Torr.

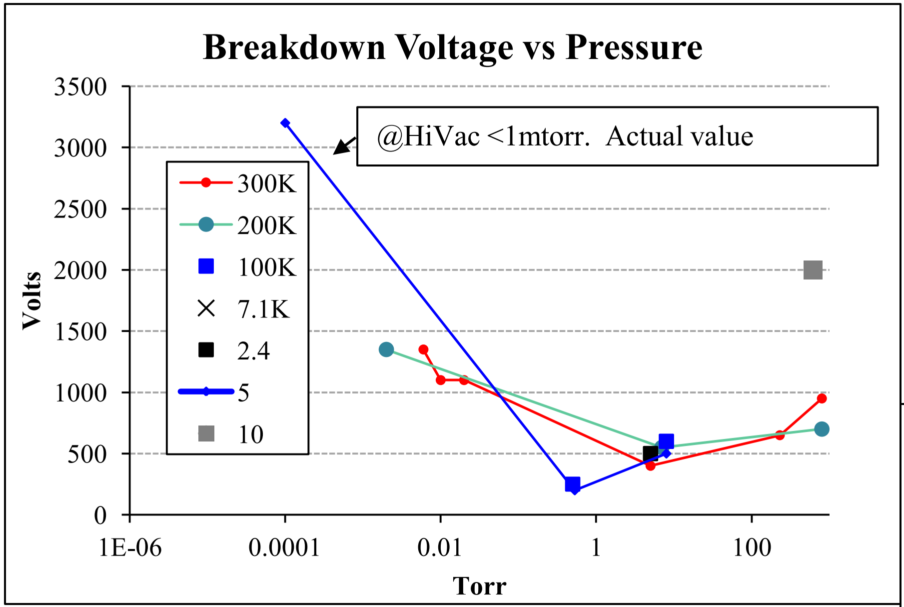
Friedrich Paschen experimented with electrical breakdown of gasses in 1889 and found that electrical breakdown depends upon the gas composition ***and its pressure but not temperature***.

His chart at right shows helium breaking down at ~200 volts at a pressure-distance product of $10^{0.4} = 2.5$. For a 5mm gap, that is 5 Torr.



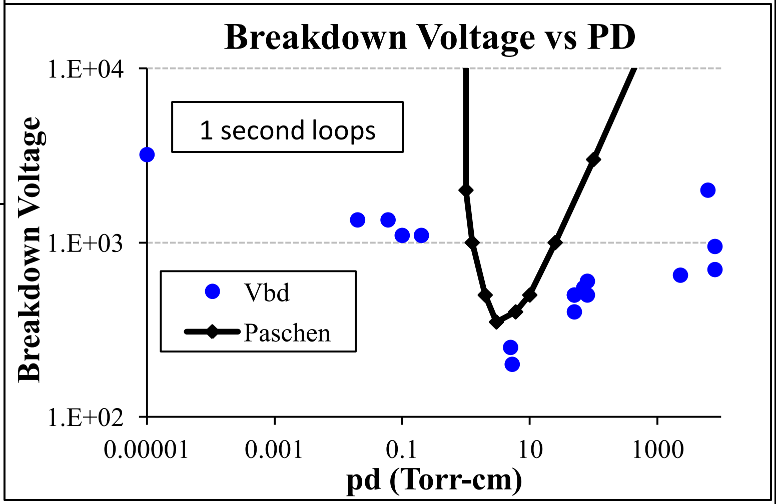
Paschen's Law

5 Torr corresponds to the optimal pressure inside the PPMS cryogenic chamber to maintain best temperature control in helium gas.



In the plot to the left, the lowest breakdown voltage for our tests occurs at roughly the same chamber pressure despite the different temperatures.

A plot of these results compared to Paschen's results indicates the same minimum on the Pressure-Distance plot.



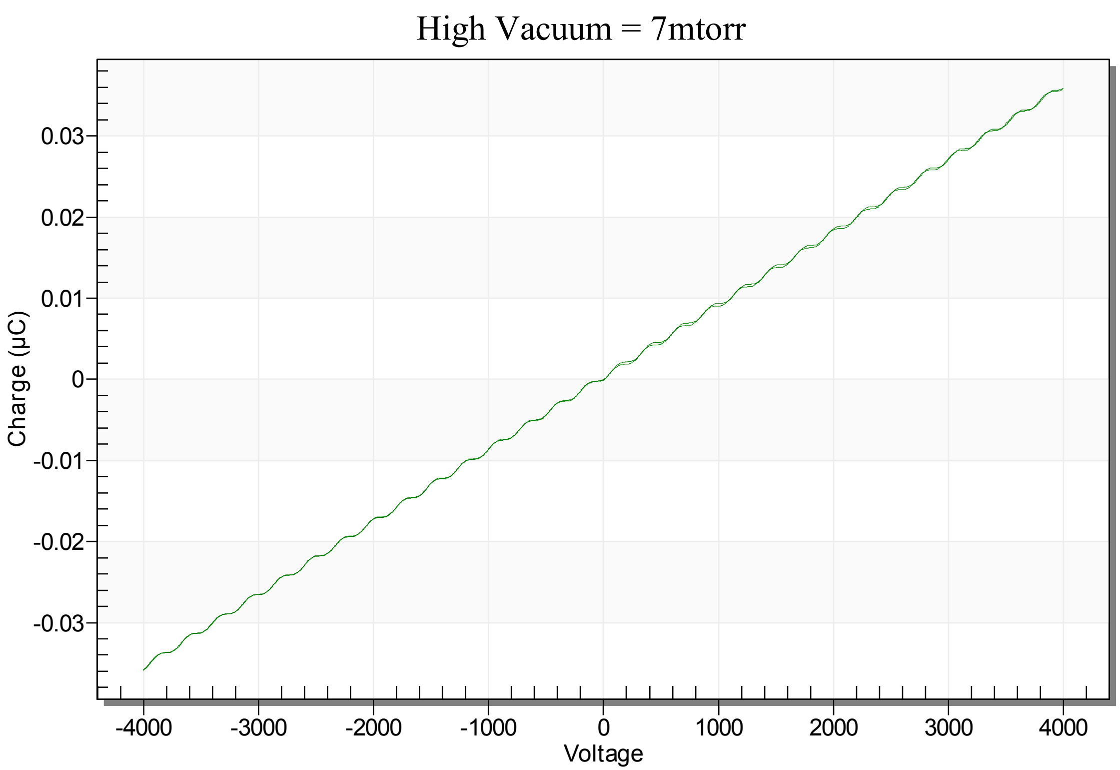
PPMS HV Procedure

- Paschen indicates that the breakdown voltage for any gas soars as the pressure drops below some pressure point of minimum breakdown voltage.

- This property suggests the procedure for measuring high voltage properties in low pressure helium:
  - Set the PPMS to its optimal pressure until the embedded thermometer indicates that the sample is at the selected temperature.
  - Change the PPMS to “High Vacuum” mode, make the HV measurement, and
  - Return the PPMS to its optimal pressure.

PPMS HV Procedure

- The linear capacitor below consists of a bare piece of Teflon placed in the fixture and tested to 4000 volts in the chamber at *7mT*.
- The equivalent capacitance of the dummy sample is ~10pF.



Conclusions

- High voltage electrical measurements of bulk capacitors inside cryogenic chambers pose multiple problems.
- Proper design of the sample fixture can mitigate most of the issues.
- Paschen's Law dictates that high voltage measurements cannot be conducted in helium at the normal operating pressures for most chambers.
- High voltage tests can be successfully conducted after the sample settles at the target temperature if the chamber pressure is reduced to the milliTorr range for the period of the test and is then returned to the optimal chamber pressure.