Electrical Properties of 20/80 PZT and 3/20/80 PNZT from 5 K to Room Temperature

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Summary

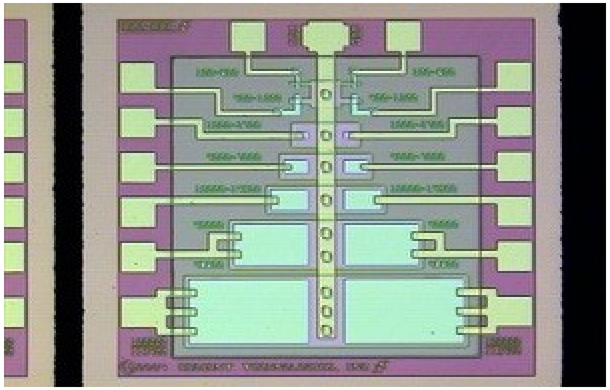
- Lake Shore Cryotronics and Radiant Technologies measured electrical properties of 20/80 PZT and 3/20/80 PNbZT thin ferroelectric film capacitors from 5 K up to 300 K.

- Lake Shore has specialized electrical probe tips for contacting samples in its cryogenic chambers that will compensate for thermal expansion of the probe arms in order to maintain electrical contact with the sample over temperature,

- The temperature-compensating probe tips allowed us to implement *automated* data acquisition over a large temperature range under the control of Radiant's Vision data acquisition program without manual intervention.

Summary

- The 20/80 capacitor under test had the following properties:
  - Thickness = 2,600Å
  - Platinum top and bottom electrodes
  - Full integration with glass passivation above the capacitor
  - Chrome/Gold probe pads and traces



- The 3/20/80 capacitor under test had the following properties:
  - Thickness = 1,500Å
  - Platinum top and bottom electrodes
  - Full integration with glass passivation above the capacitor
  - Chrome/Gold probe pads and traces

- Both samples used the same mask set.

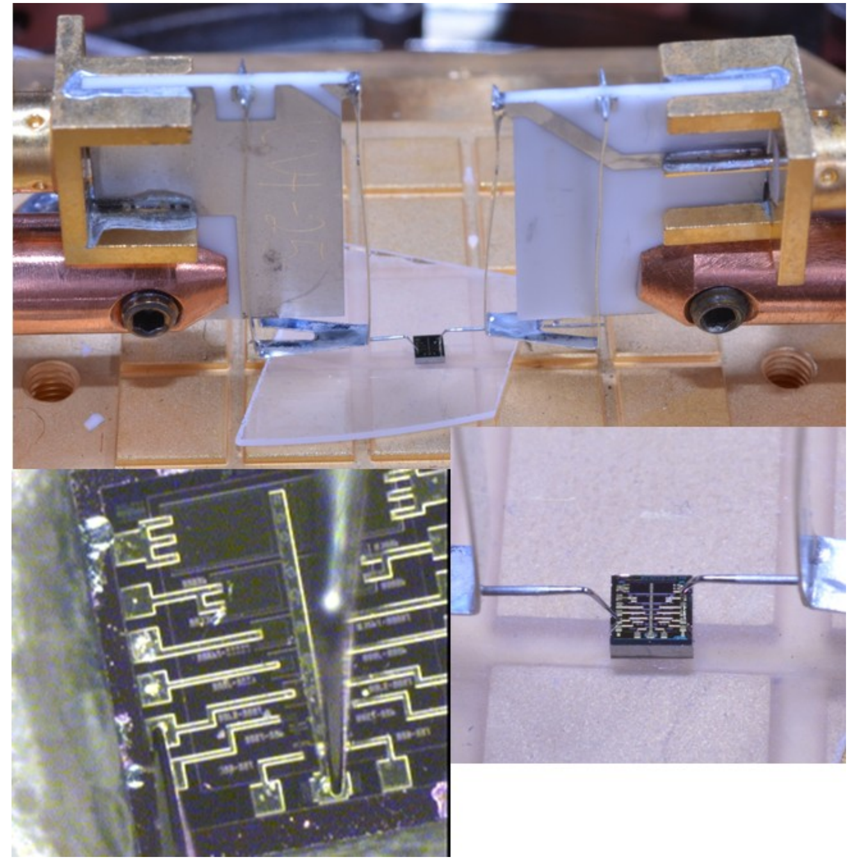
Test Voltage vs Temperature

- The coercive and saturation voltages for hysteresis loops of PZT decrease dramatically from cryogenic temperatures up to the Curie Temperature.
- The tests, to be useful, should be executed with voltages well above the saturation voltage of the hysteresis loop *for all loops*.
- The Vmax selected for this testing ensured saturated loops at the coldest temperature.
- Since the working voltage for the capacitor goes down as temperature goes up, the proper test voltage for the cryogenic temperatures may be too much for the same capacitor at room temperature or higher.
- To ensure the capacitors would not break down at higher temperatures, very fast measurements were used.

Test Voltage vs Temperature

- The small signal capacitance and leakage tests were executed at low voltages so they were not subject to the test period limitation at higher temperatures.
- The first test was done at 20 volts on 2600Å 20/80 PZT. It functioned up to 300 K for a 1 millisecond test but broke down for the tests at 340 K.
- Subsequent 20/80 tests were limited to 310 K.
- The 1500Å 4/20/80 PNZT capacitor was tested at 12 volts up to 250°K.
- *The test voltage vs temperature vs frequency envelope must be evaluated before starting long automated tests.*

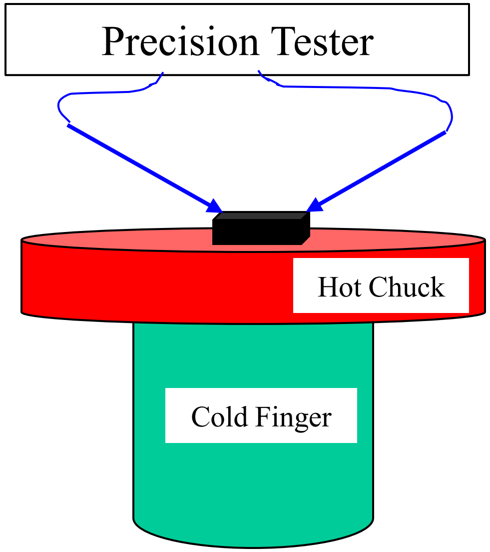
Lake Shore Cryogenic Chamber



The Lake Shore Cryotronics CRX-4K chamber is capable of 350 K down to 4.5 K. The chamber has up to six micropositioners with which to contact the sample. Lake Shore's patented temperature-compensating CVT probe tips were used to connect to the test dice.

Lake Shore Cryogenic Chamber

- The Lake Shore Cryotronics CRX-4K chamber has a hot chuck placed above a cold finger.
- The cold finger was first dropped to 5.0 K while the hot chuck was maintained at room temperature.
- The hot chuck was then set to the first temperature of the test profile and testing began.
- The initial drop to 5.0 K by the cold finger took about two hours.
- For temperature changes, the controller was commanded to use a ramp rate of 3°K per minute and then to soak the sample at the new temperature for 10 minutes before conducting tests.



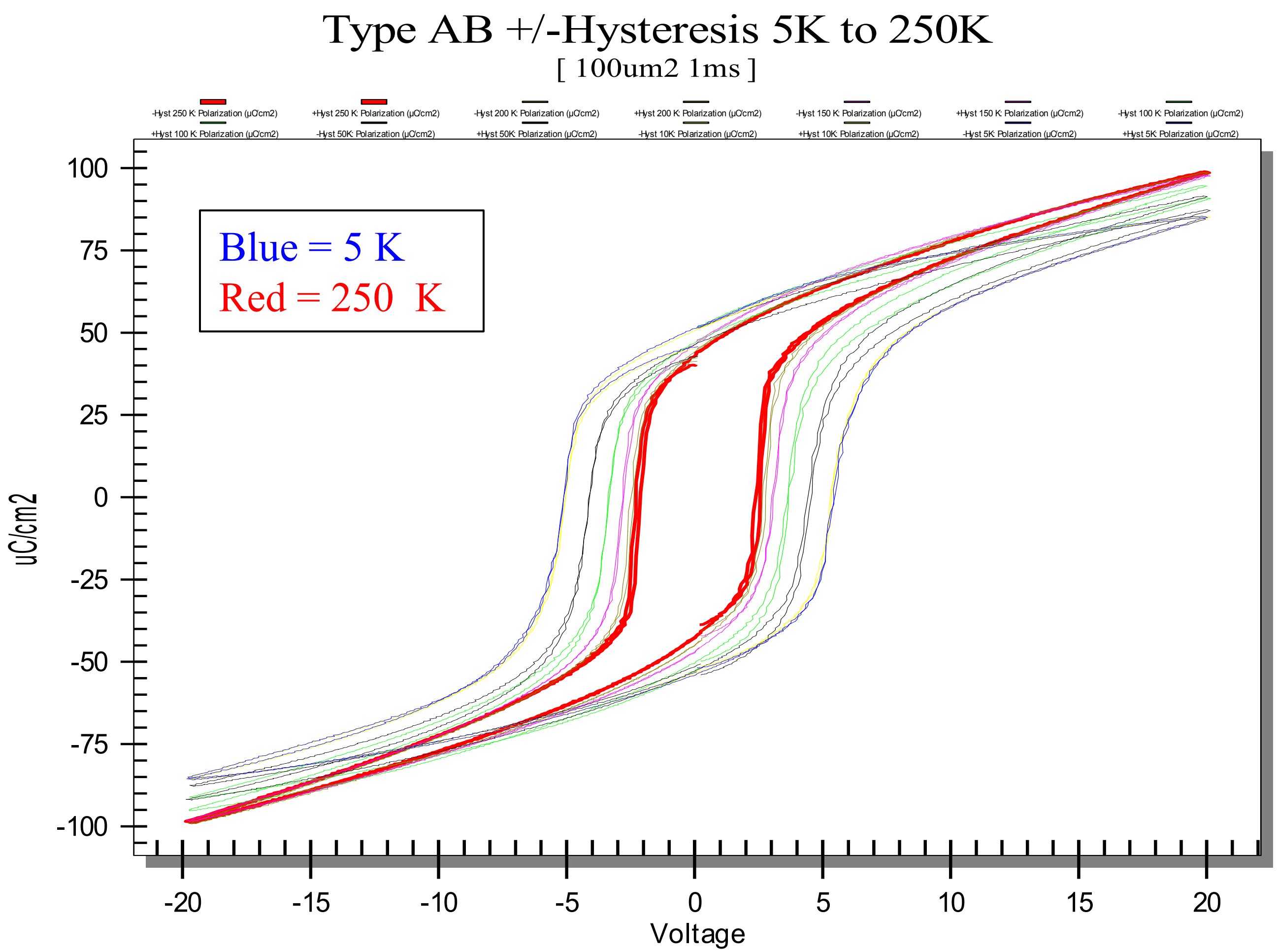
Test #1

- 100µm^2 20/80 PZT Capacitor
  - 5 K then 10 K, 50 K , 100 K , 150 K , 200 K , and 250 K.
  - Type AB — Thickness = 0.26m
    Vsat = 9 volts @ room temperature
  - Hysteresis — 20 volts with a 1 millisecond period
  - Remanent Hysteresis — 20 volts with a 1 millisecond period
  - Switching Speed — 9.9 volts from 1µs to 100ms

Hysteresis vs Temperature

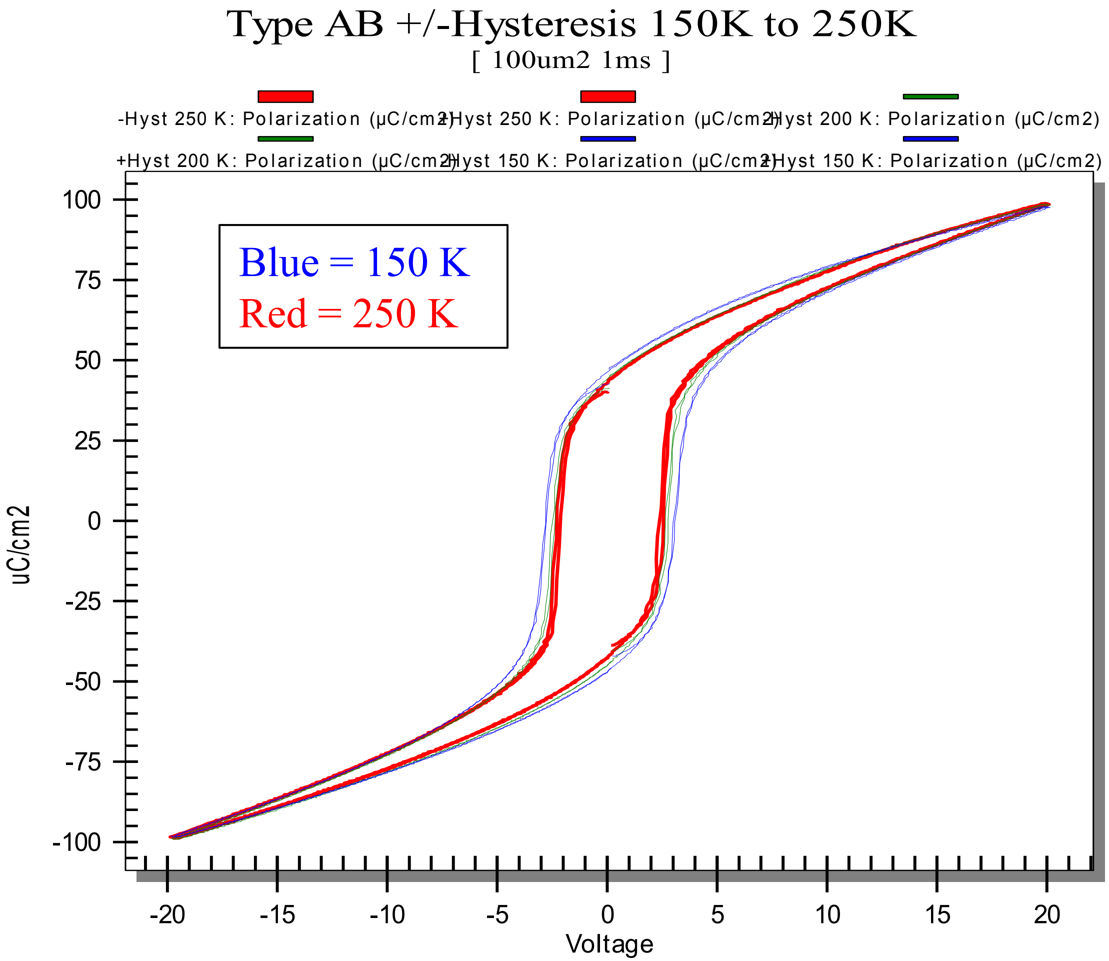
100 µm² 20/80 PZT

- ±20 volts with 1 millisecond period.



Hysteresis vs Temperature
100 µm^2 20/80 PZT

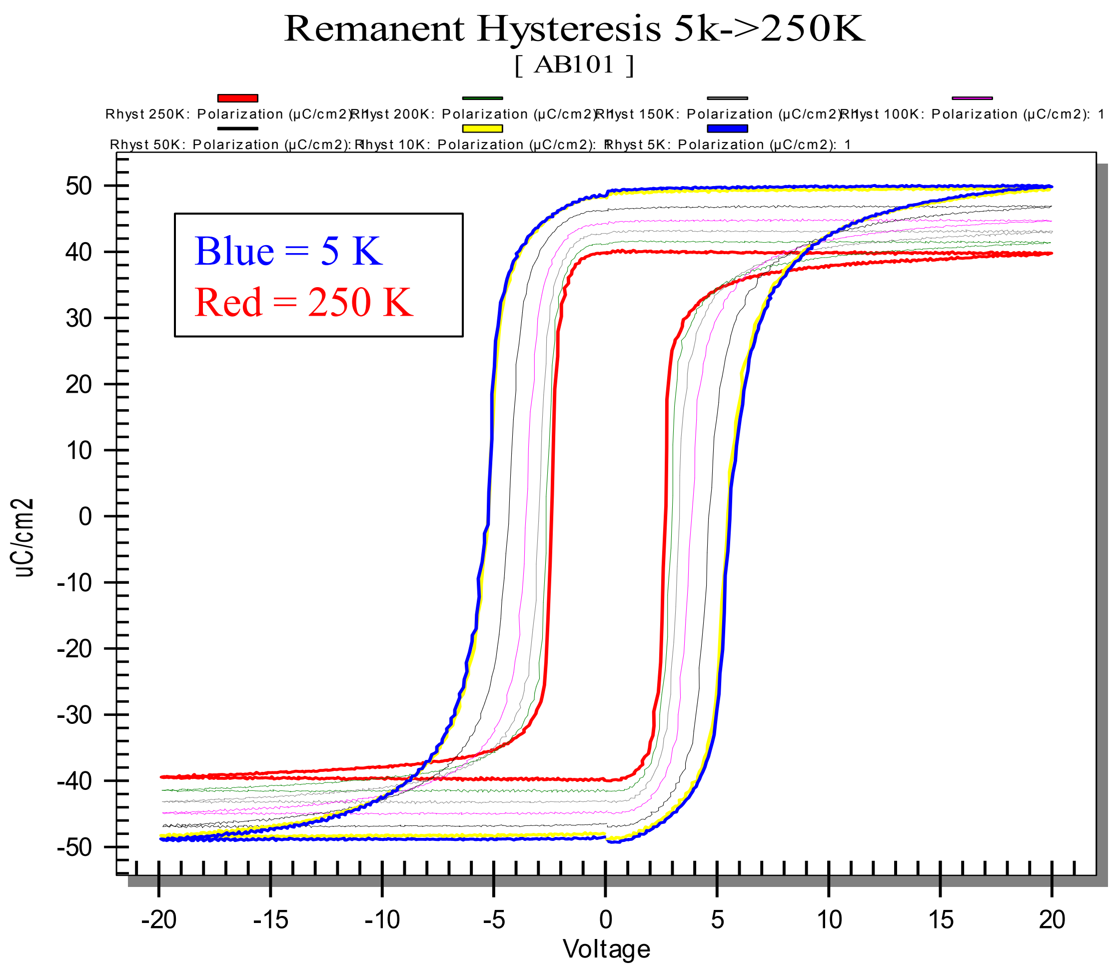
- ±20 volts with 1 millisecond period.



Remanent Hysteresis vs Temperature

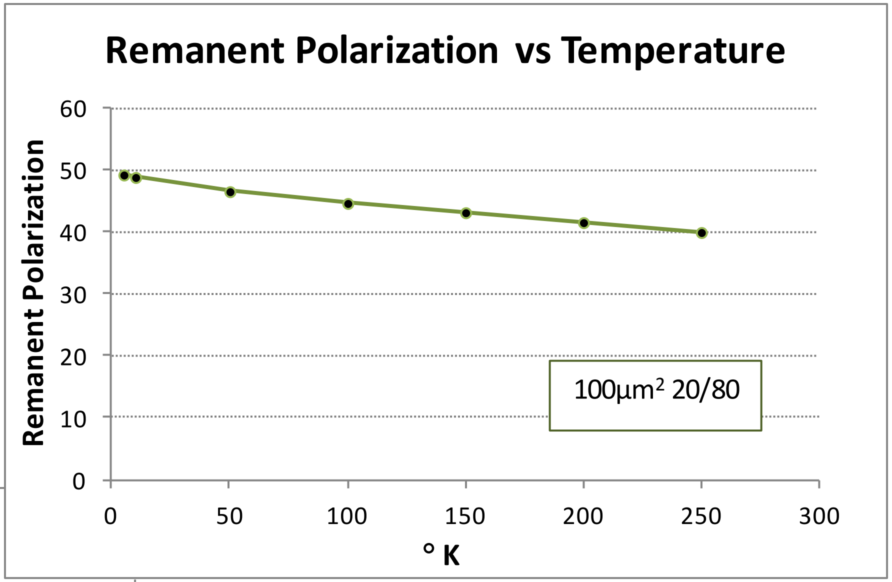
100 µm² 20/80 PZT

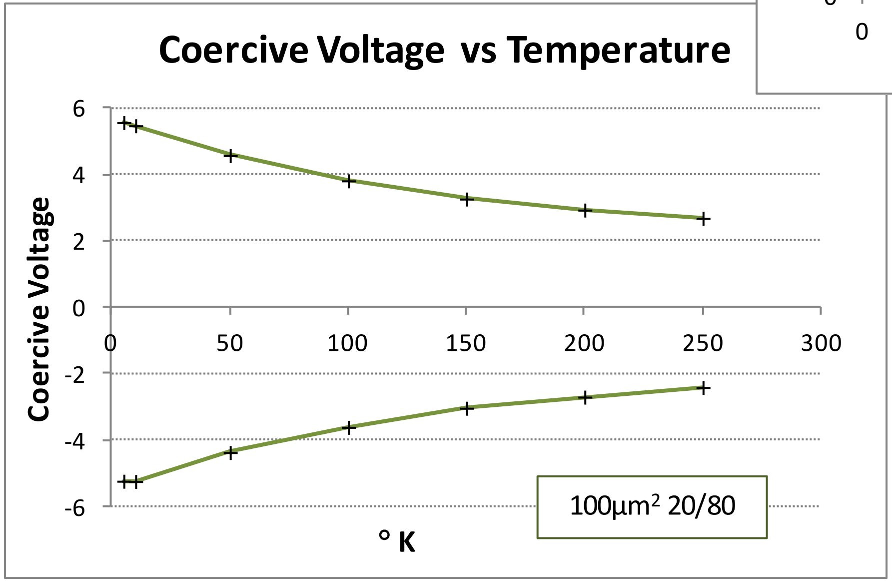
- 20 volts with 1 millisecond period.



Remanent Hysteresis vs Temperature
100 μm² 20/80 PZT

- 20 volts with 1 millisecond period.

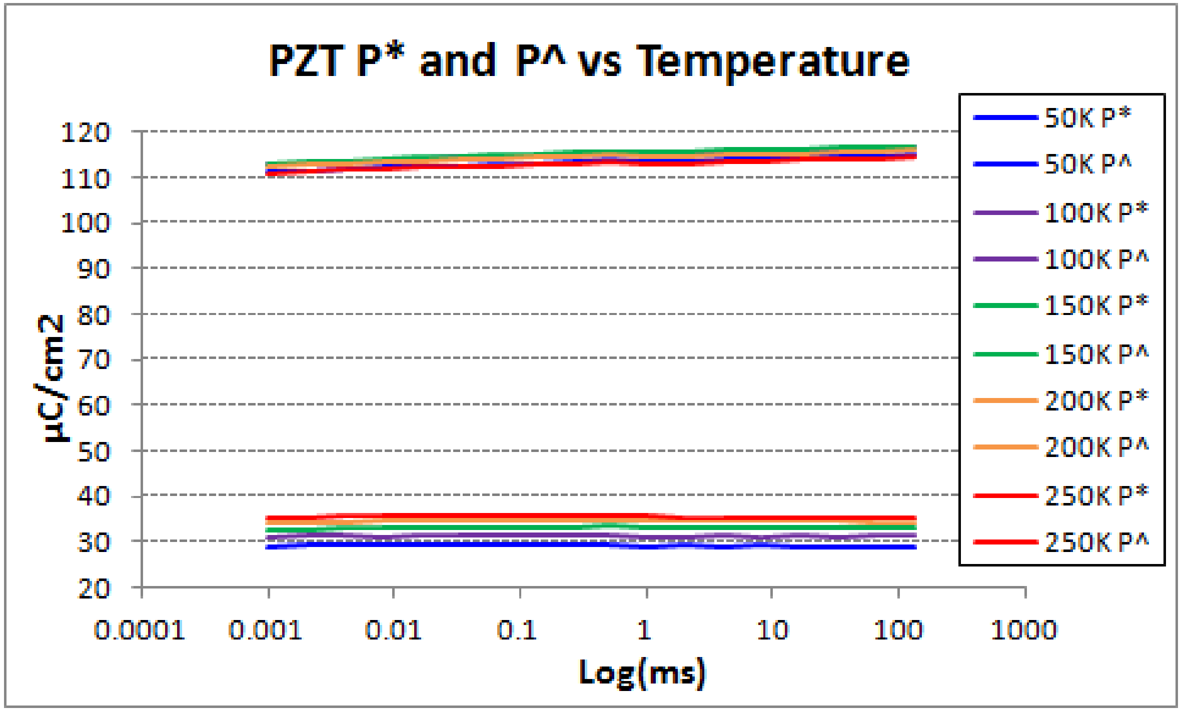




PUND vs Temperature

100 µm² 20/80 PZT

- 9.9 volts from 1µs pulse width to 131ms pulse width.
- Definitions:
  - P* switching pulse
  - P^ non-switching pulse



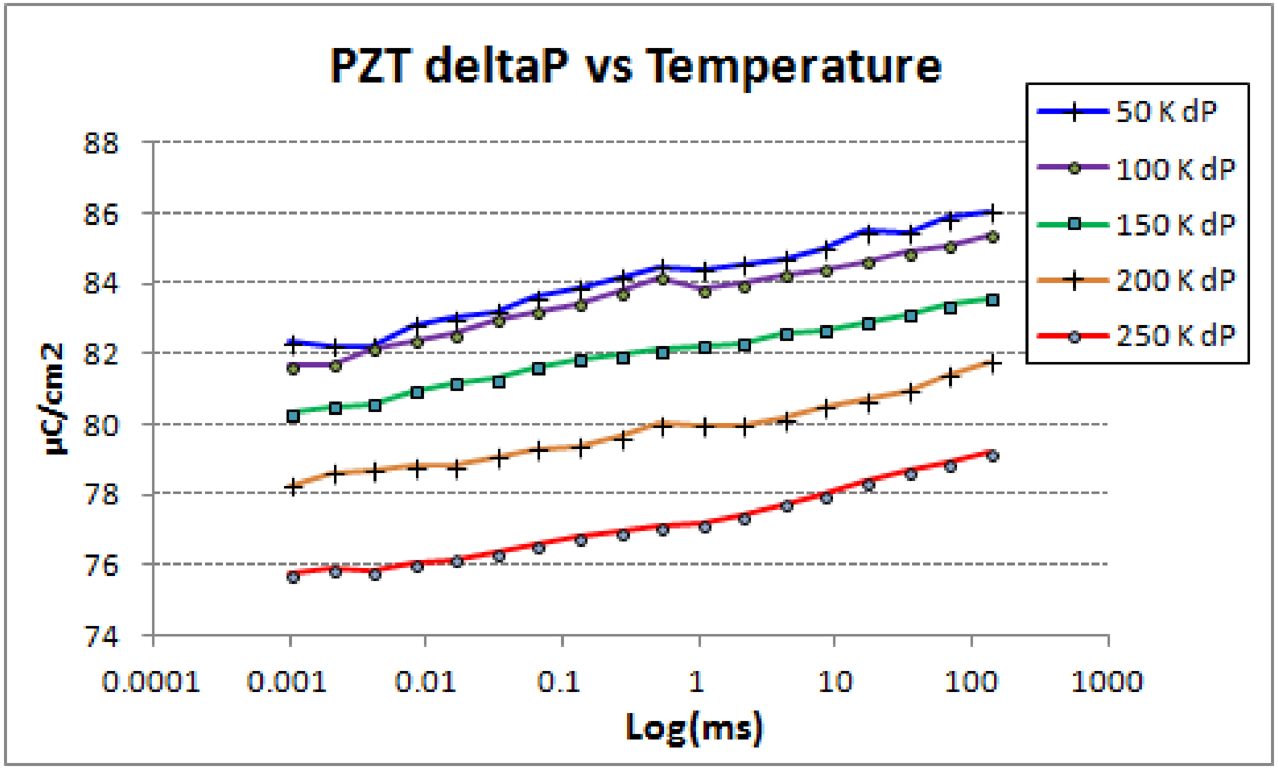
It appears that as the temperature increases from 50 K to 250 K, the P, or switching polarization, decreases slightly while the non-switching component increases more.*

NOTE: The switching pulse is the sum of the non-switching pulse response and the remanent polarization.

PUND vs Temperature

100 μm² 20/80 PZT

- dP = P* - P^ = 2 x remanent polarization



- The remanent polarization decreases its magnitude with *increasing* temperature.
- The remanent polarization decreases in magnitude with *decreasing* pulse width.
- The switching speed vs pulse width slope remains constant down to 50 K.

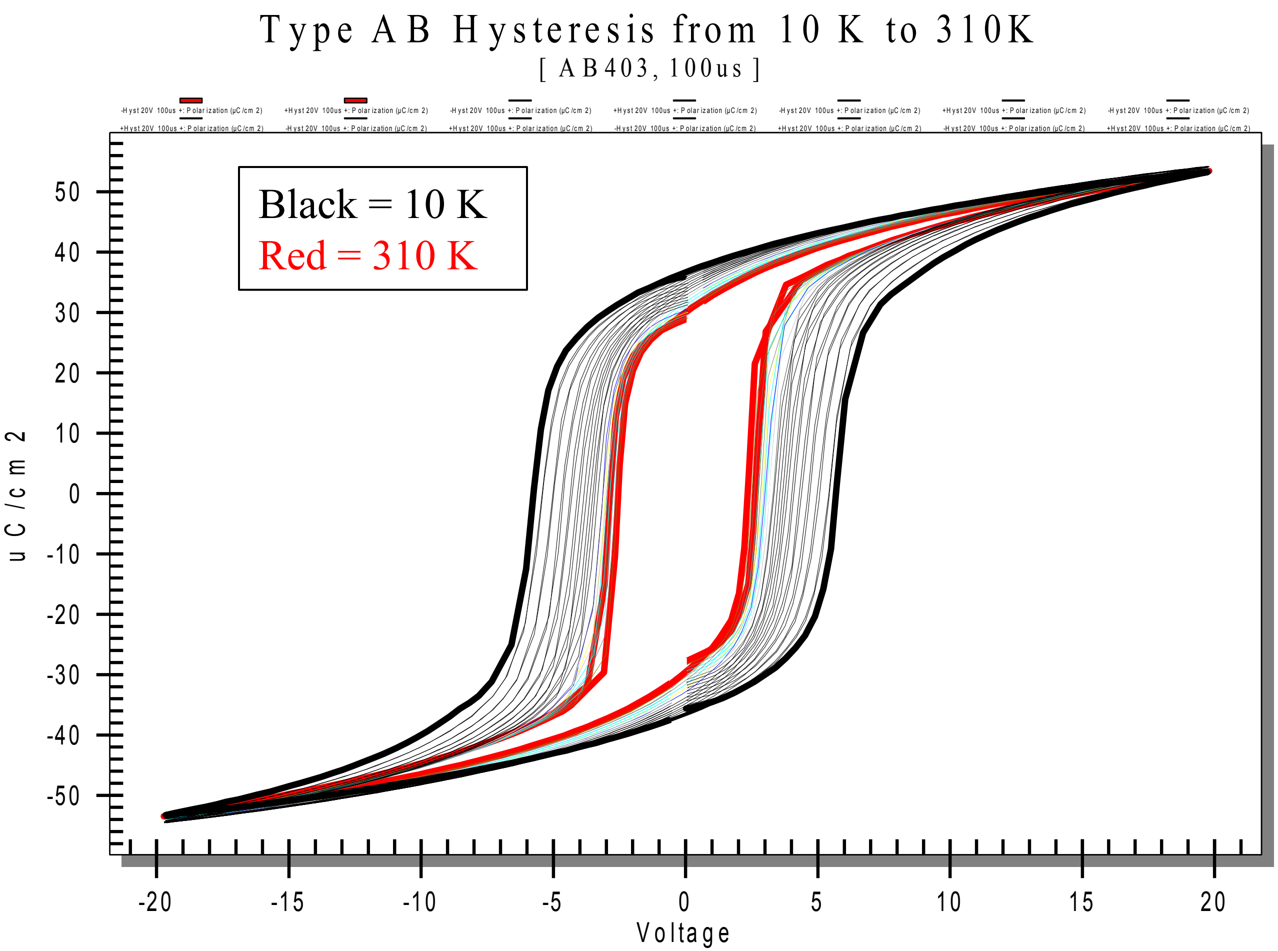
Test #2

- 40,000μm^2 20/80 PZT Capacitor
  - 10 K to 310 K in 20 K steps
  - Type AB — Thickness = 0.26m
Vsat = 9 volts @ room temperature
  - Hysteresis — 20 volts with a 100 microsecond period
  - Remanent Hysteresis — 20 volts with a 100 microsecond period
  - Small Signal Capacitance — 1 kHz 200mV with 0 volt bias
  - Leakage — 1 volt over 1 second with 1 second soak

Hysteresis vs Temperature

40,000 µm^2 20/80 PZT

- ±20 volts with 100 microsecond period.



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

100μm^2 vs 40,000μm^2

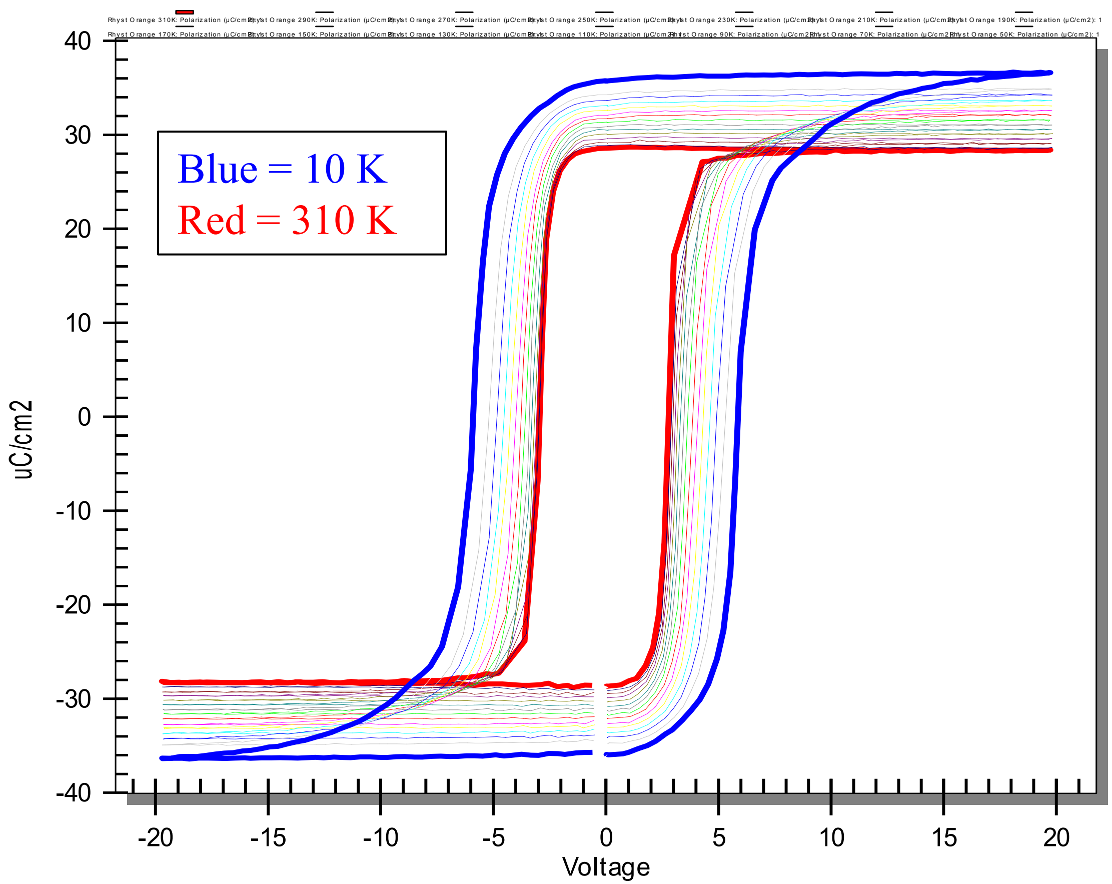
- The small 100μm^2 Type AB capacitor showed changes in its Pmax values at temperatures below 150 K.
- The much larger 40,000μm^2 Type AB capacitor showed no change in Pmax from the cold 10 K all the way to the warm 310 K.
- This difference in behavior is most likely due to the small size of the 100μm^2 capacitor.
  - The 100μm^2 capacitor has an equivalent capacitance of only 10pF.
  - The 40,000μm^2 capacitor is 400 times larger.
  - Parasitic linear capacitance in parallel to the capacitor under test could modify the shape of the smaller capacitor loop but not the larger capacitor loop.
- That parasitic capacitance most likely changed over the temperature test range, affecting the 100μm^2 capacitor results but not those of the 40,000μm^2 capacitor.

Remanent Hysteresis vs Temperature

40,000 μm^2 20/80 PZT

- 20 volts with 100 microsecond period.

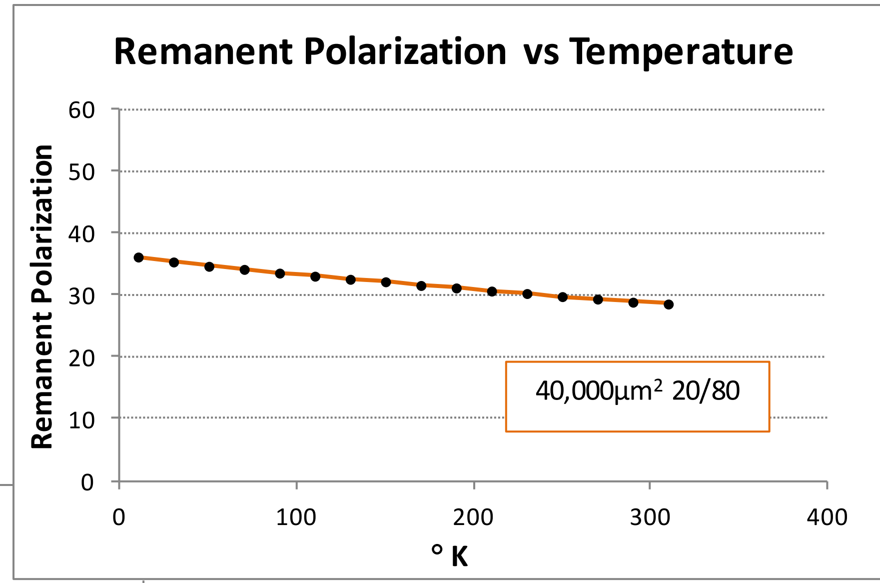
Remanent Hysteresis 10k->310K
[AB403, 100us]

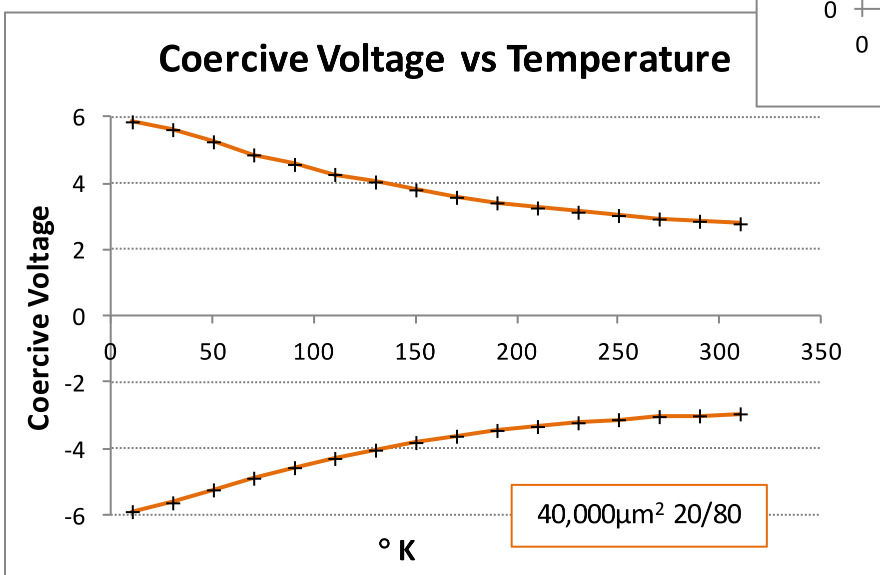


Remanent Hysteresis vs Temperature

40,000 µm² 20/80 PZT

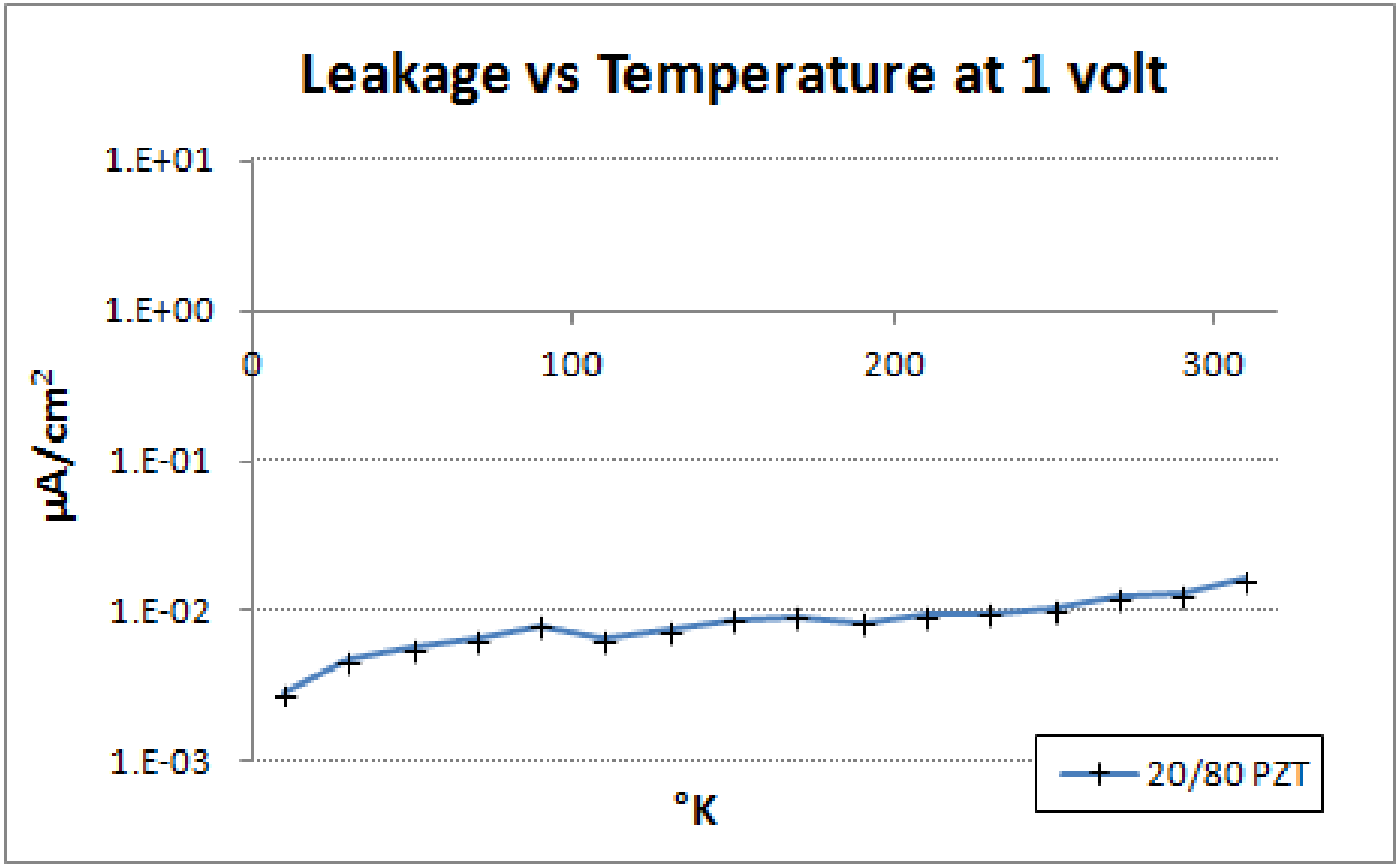
20 volts with 100 microsecond period.





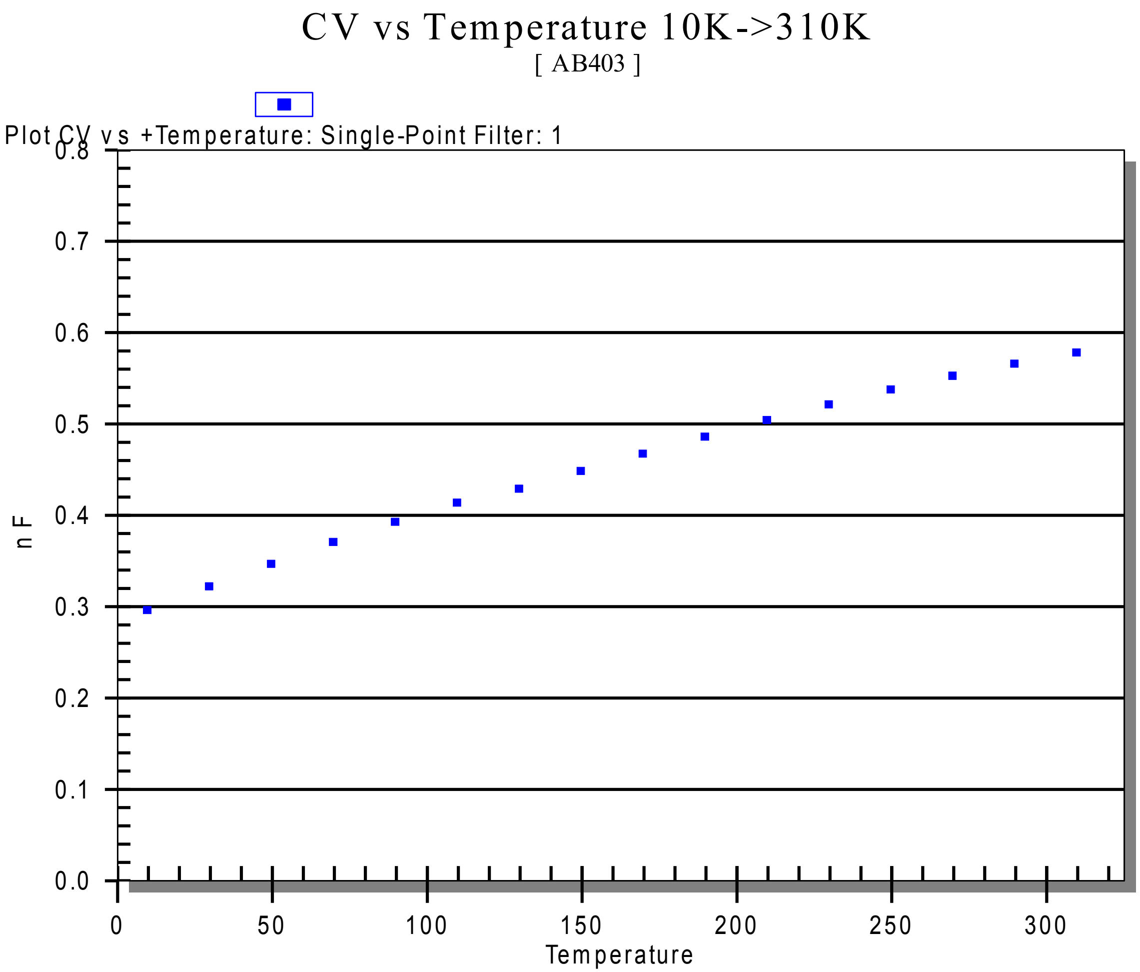
Leakage vs Temperature
40,000 µm² 20/80 PZT

- 1 volt for 1 second after a 1 second soak at 1 volt.



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

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



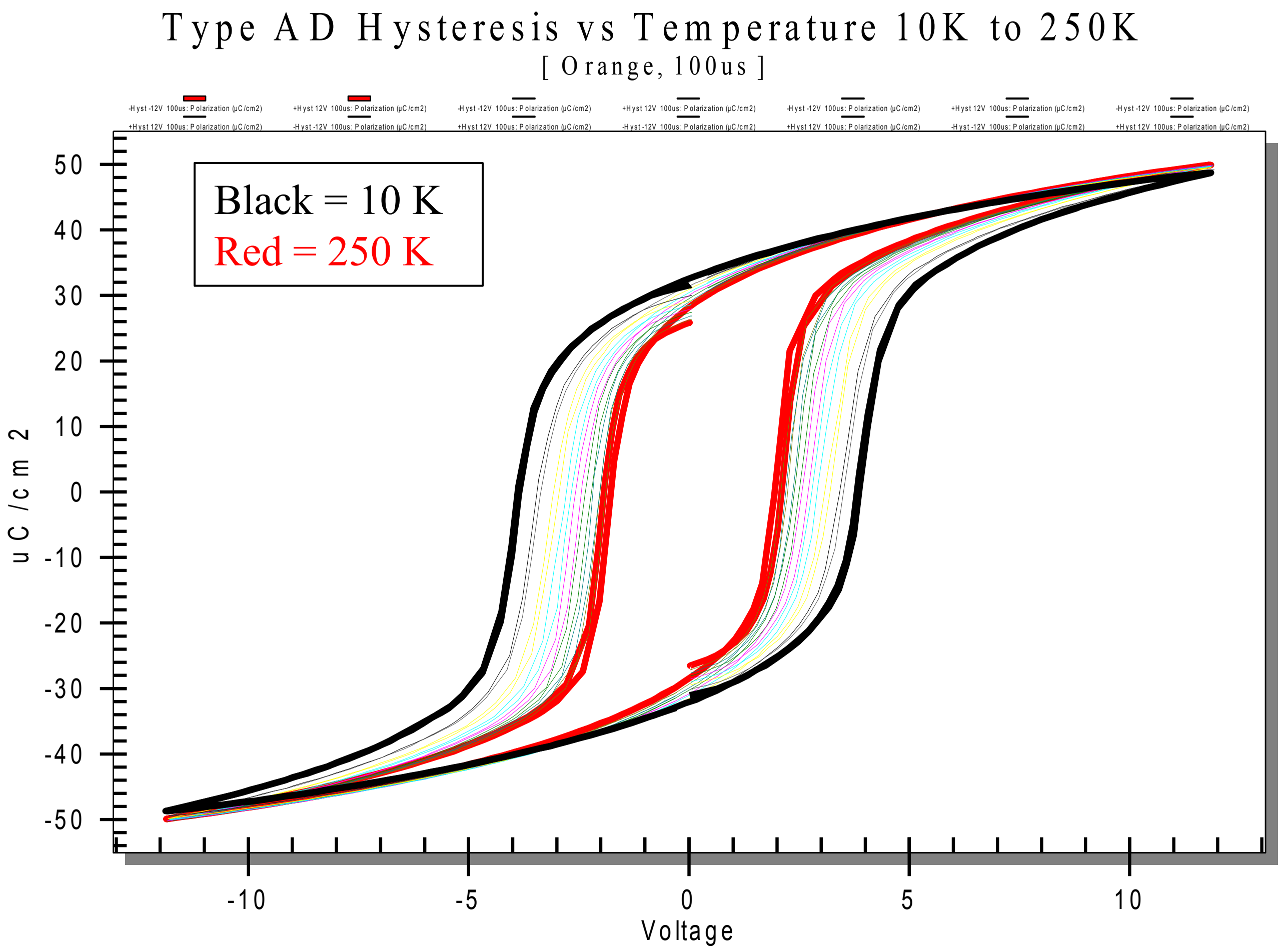
Test #3

- 40,000µm² 3/20/80 PNZT Capacitor (Type AD)
  - 10 K to 250 K in 30 K steps
  - Type AB — Thickness = 0.16m, Vsat = 5 volts
  - Hysteresis — 12 volts with a 100 microsecond period
  - Remanent Hysteresis — 12 volts with a 100 microsecond period
  - Small Signal Capacitance — 1 kHz 200mV with 0 volt bias
  - Leakage — 1 volt over 1 second with 1 second soak
  - Switching Speed — 9.9 volts from 1µs to 65ms

Hysteresis vs Temperature

40,000 µm² 3/20/80 PNZT

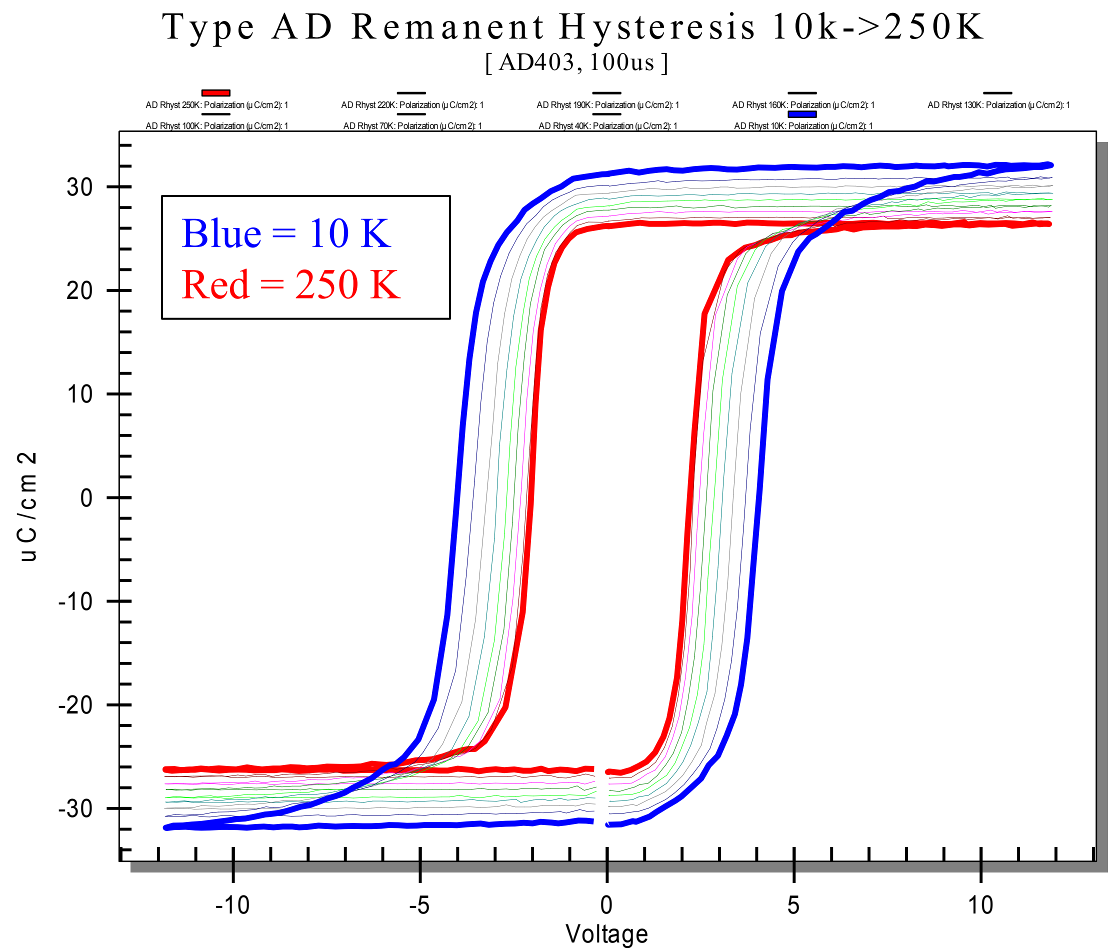
- ±12 volts with 100 microsecond period.



Remanent Hysteresis vs Temperature

40,000 µm^2 3/20/80 PNZT

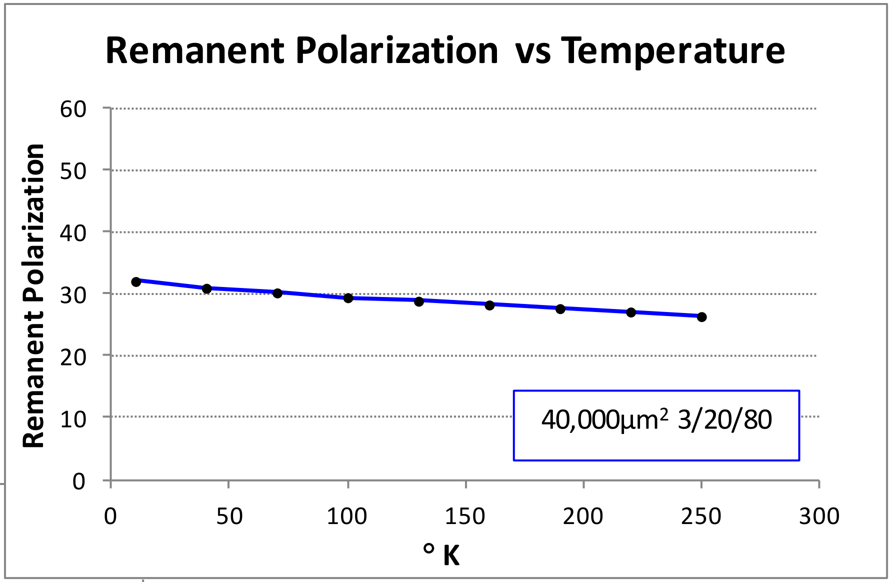
- 12 volts with 100 microsecond period.

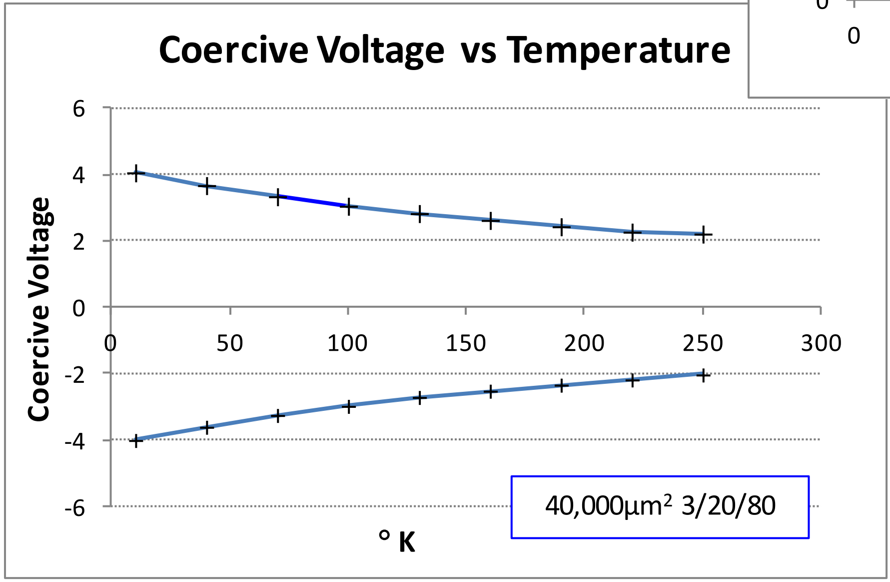


Remanent Hysteresis vs Temperature

40,000 µm² 3/20/80 PNZT

12 volts with 100 microsecond period.

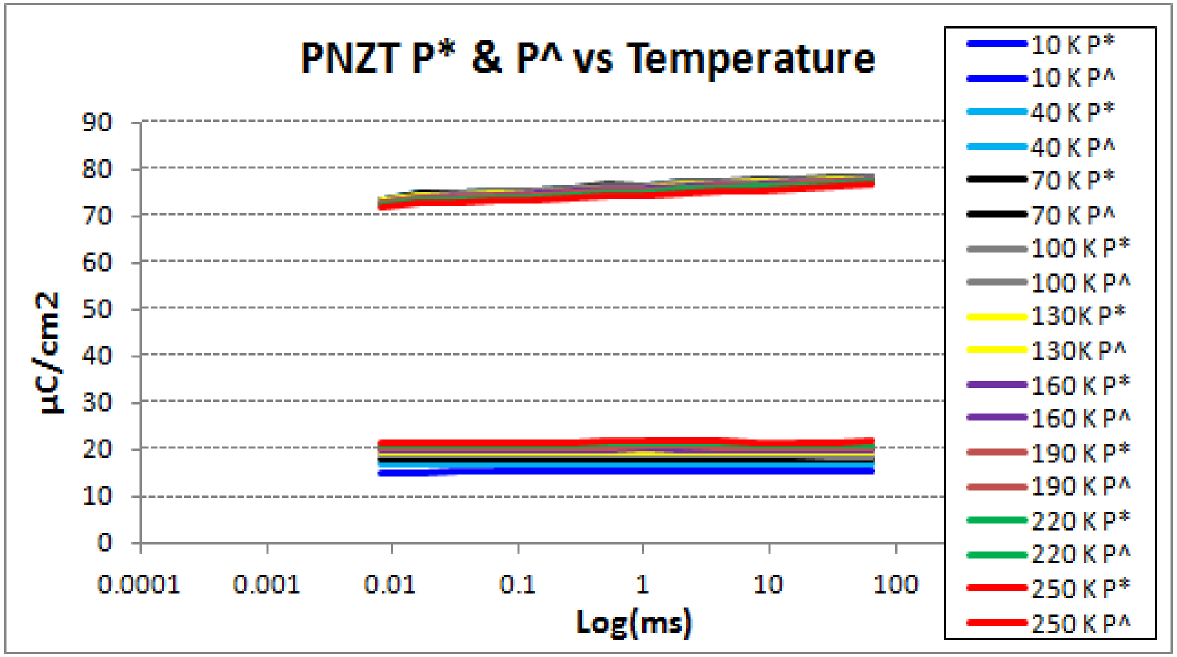




PUND vs Temperature

40,000 μm^2 3/20/80 PNZT

- 9.9 volts from 1μs pulse width to 131ms pulse width.
- Definitions:
  - P* switching pulse
  - P^ non-switching pulse

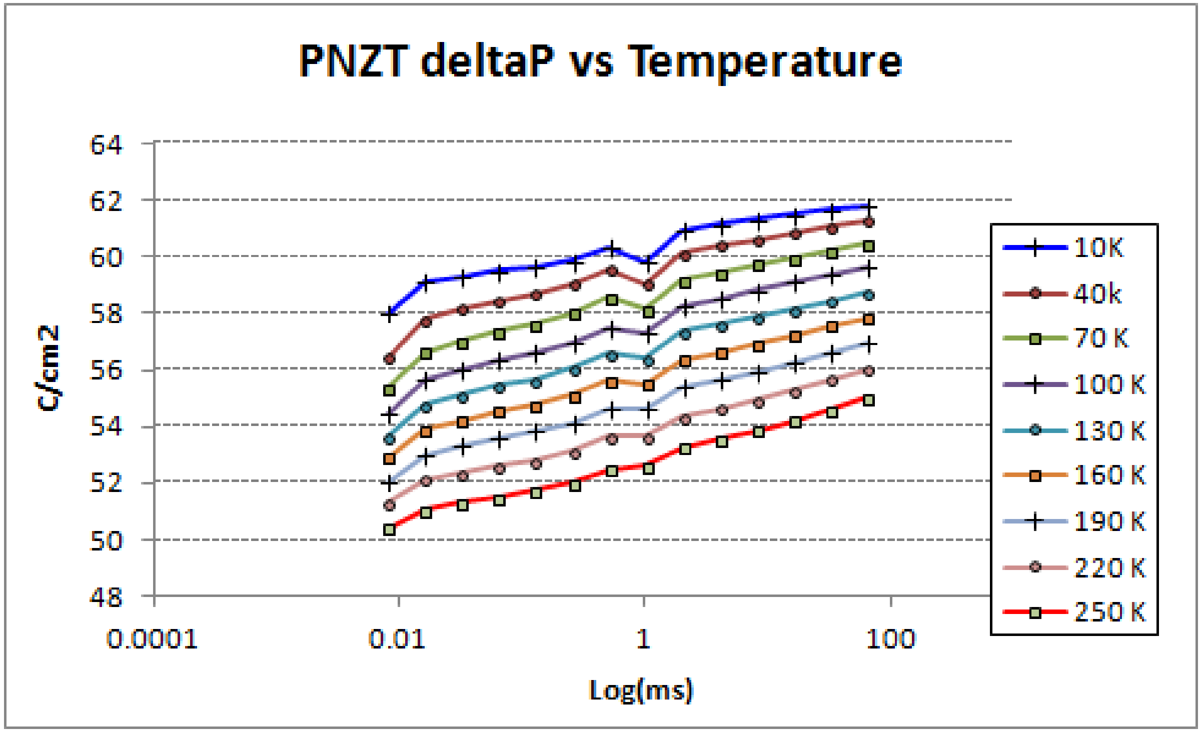


It appears that as the temperature increases from 50 K to 250 K, the P, or switching polarization, remains essentially constant while the non-switching component increases.*

NOTE: The switching pulse is the sum of the non-switching pulse response and the remanent polarization.

PUND vs Temperature
40,000 μm² 3/20/80 PNZT

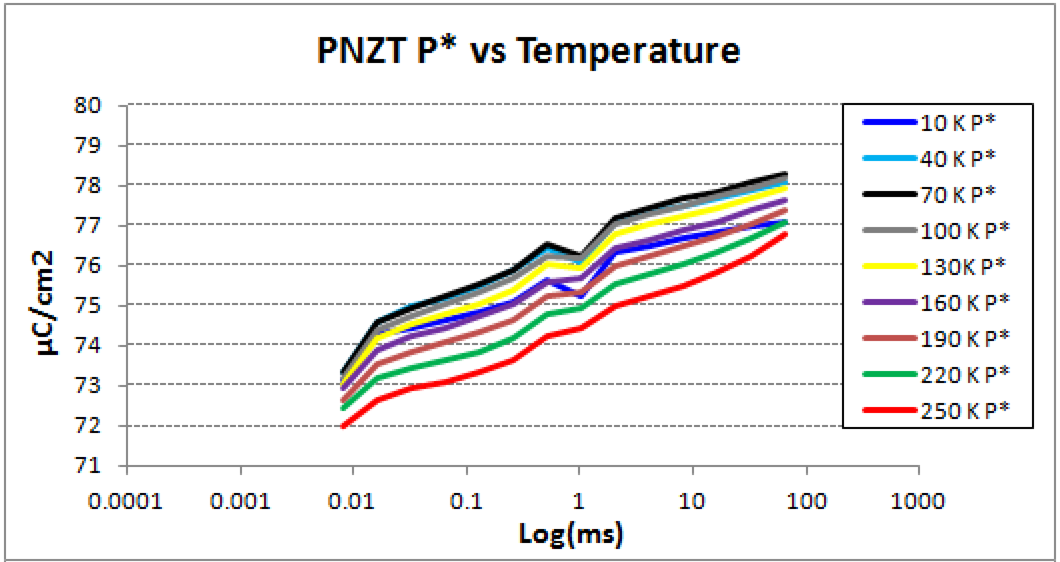
- dP = P* - P^ = 2 x remanent polarization

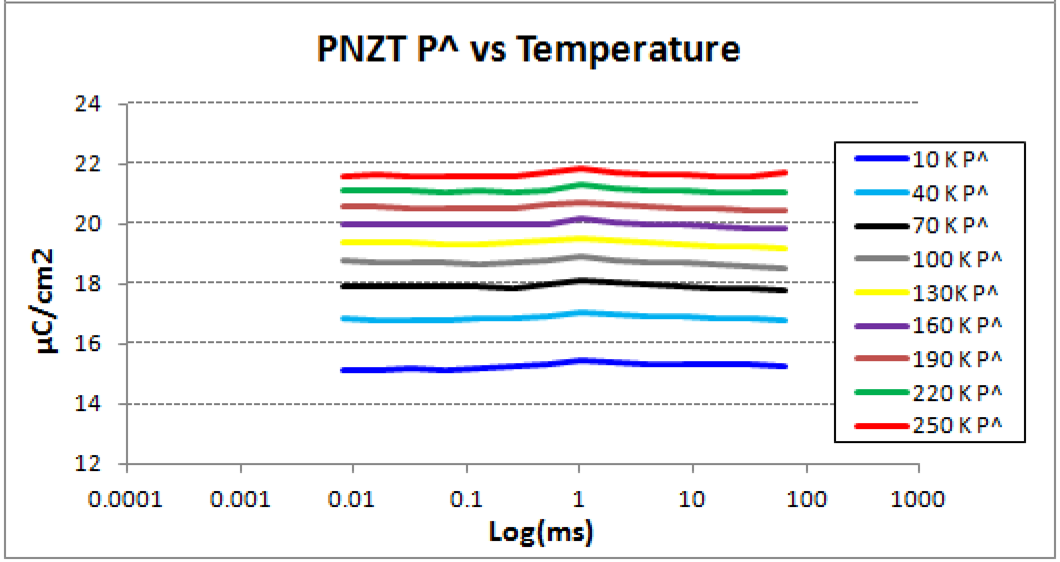


- The remanent polarization decreases its magnitude with *increasing* temperature.
- The remanent polarization decreases in magnitude with *decreasing* pulse width.
- The switching speed vs pulse width slope remains constant down to 50 K.

PUND vs Temperature

40,000 µm² 3/20/80 PNZT



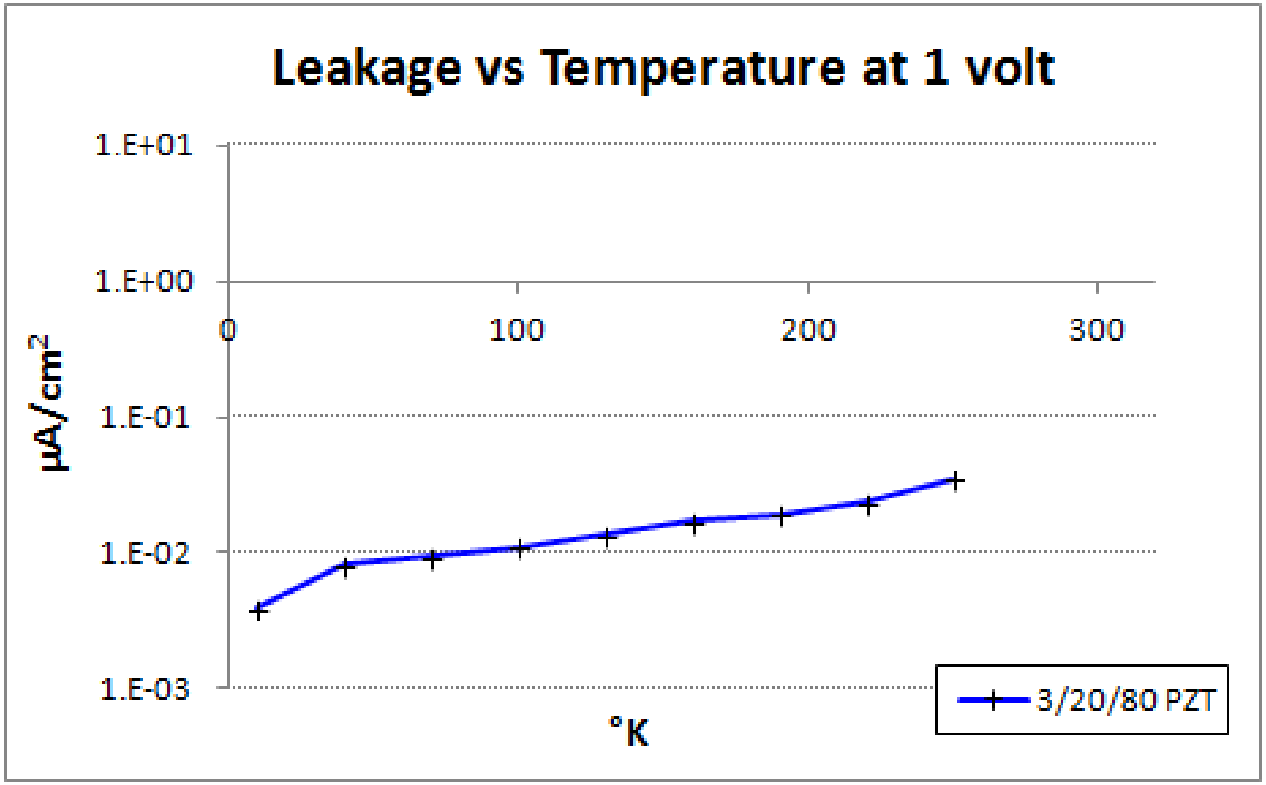


As with 20/80 PZT, when temperature increases for PNZT the P* decreases while the P^ increases at a greater rate.

Leakage vs Temperature

40,000 μm² 3/20/80 PNZT

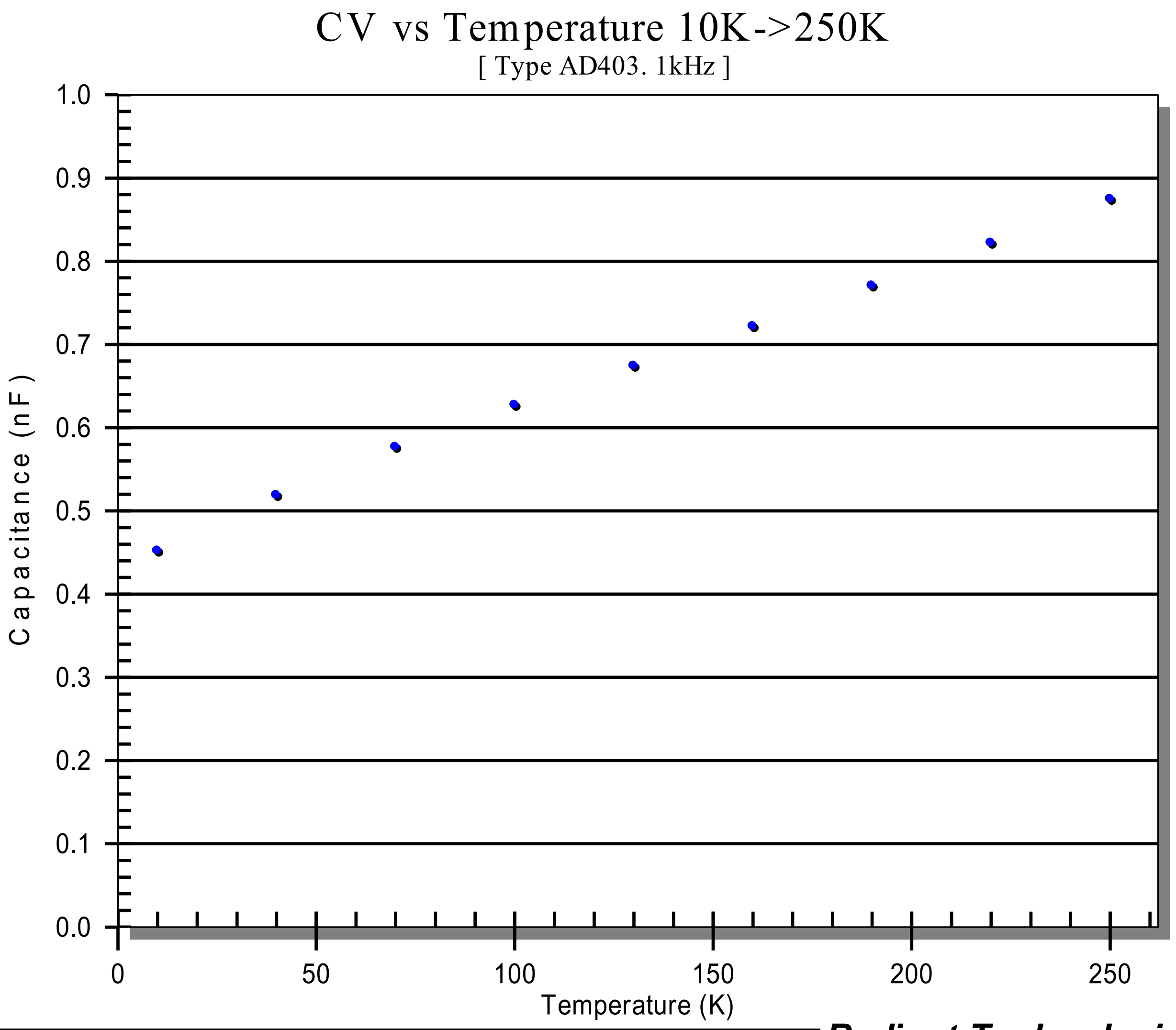
- 1 volt for 1 second after a 1 second soak at 1 volt.



Small Single CV vs Temperature

40,000 μm^2 3/20/80 PNZT

- 1 kHz

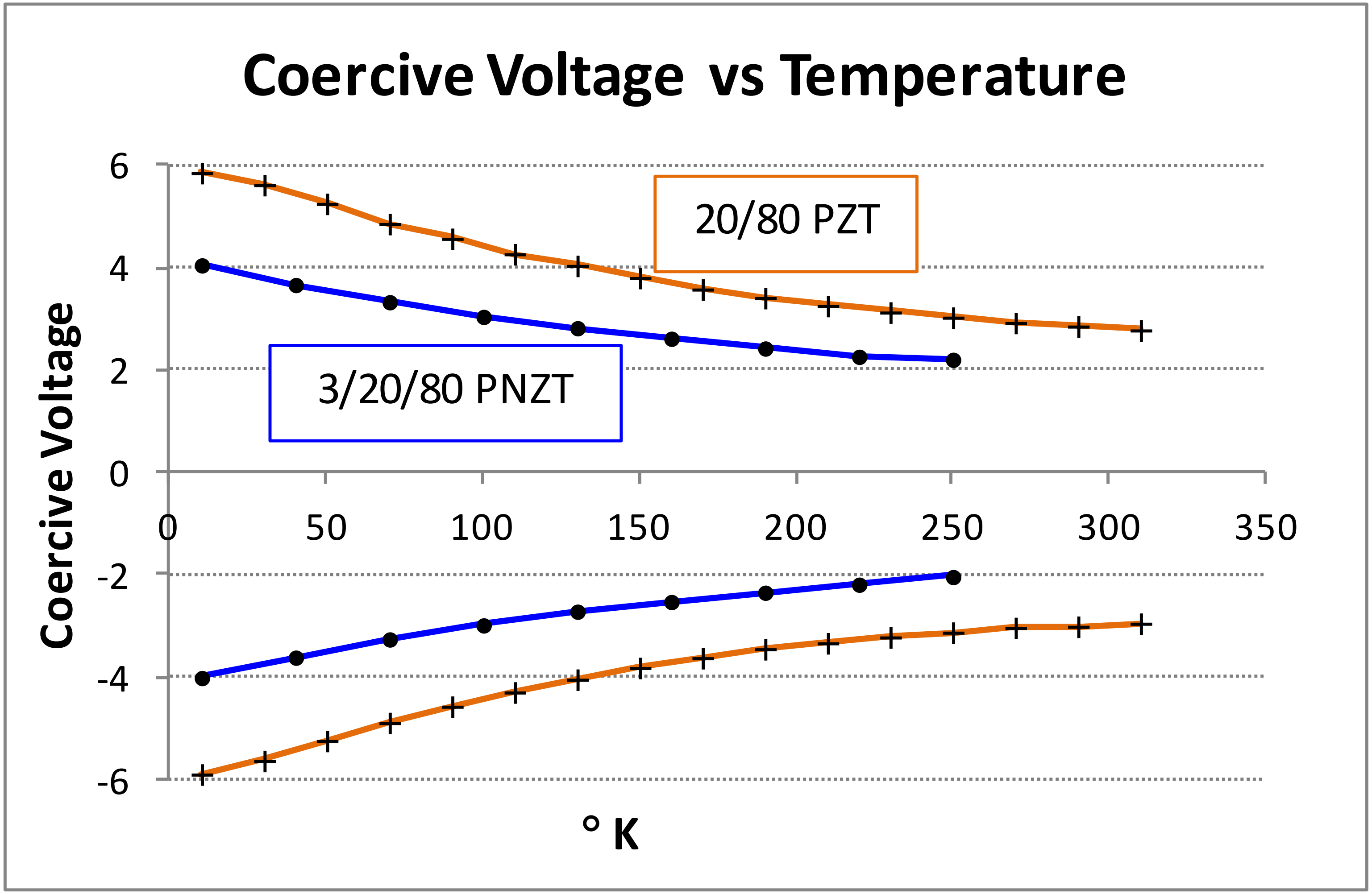


COMPARE 20/80 –> 3/20/80

- In the next few slides, I compare the results for the 20/80 PZT vs the 3/20/80 niobium doped PZT.
- Thicknesses:
  - 20/80 2600Å
  - 3/20/80 1600Å
- Areas: Both 40,000μm^2

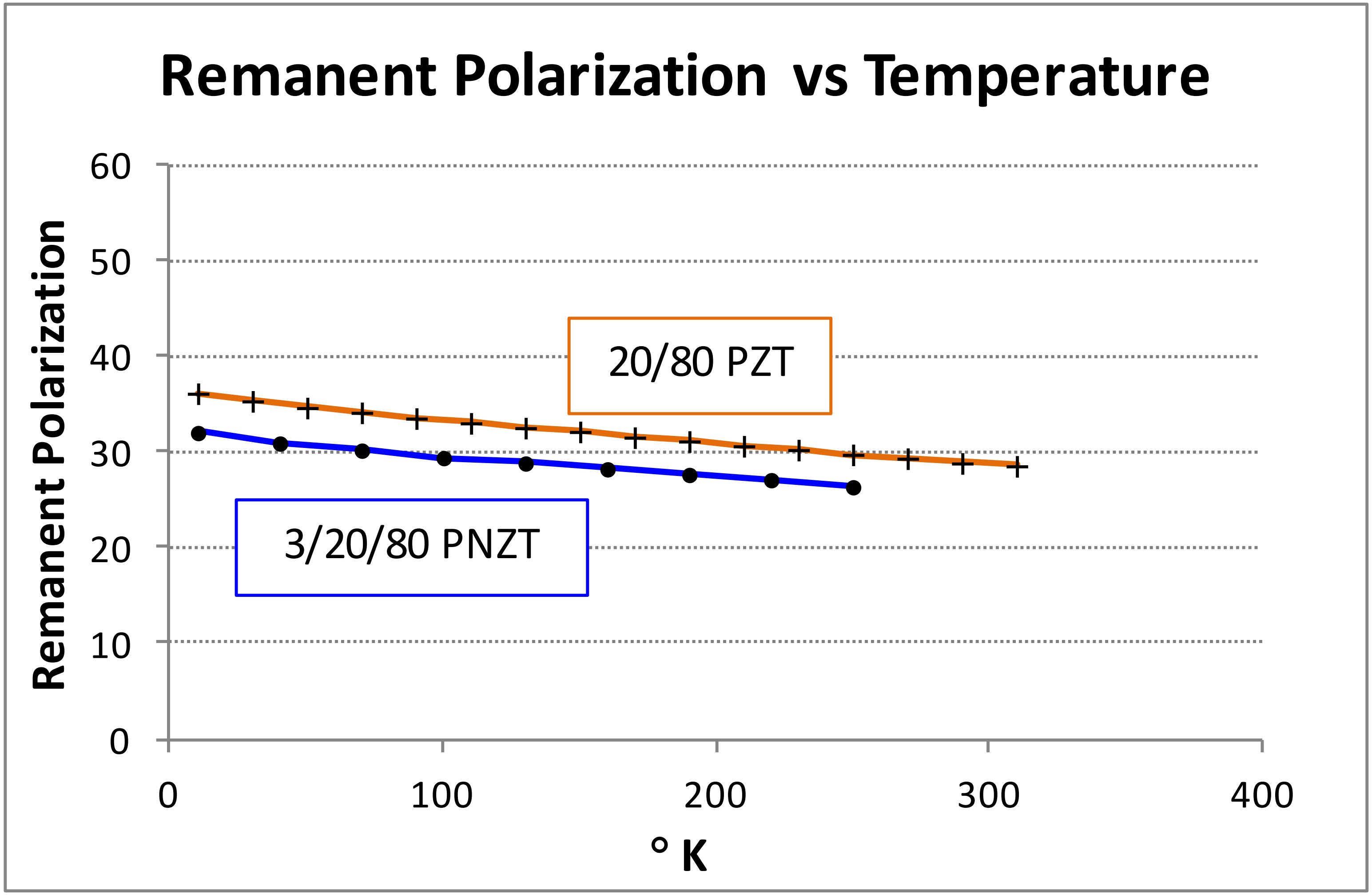
Coercive Voltage vs Temperature

40,000 µm² 3/20/80 PNZT vs 20/80 PNZT



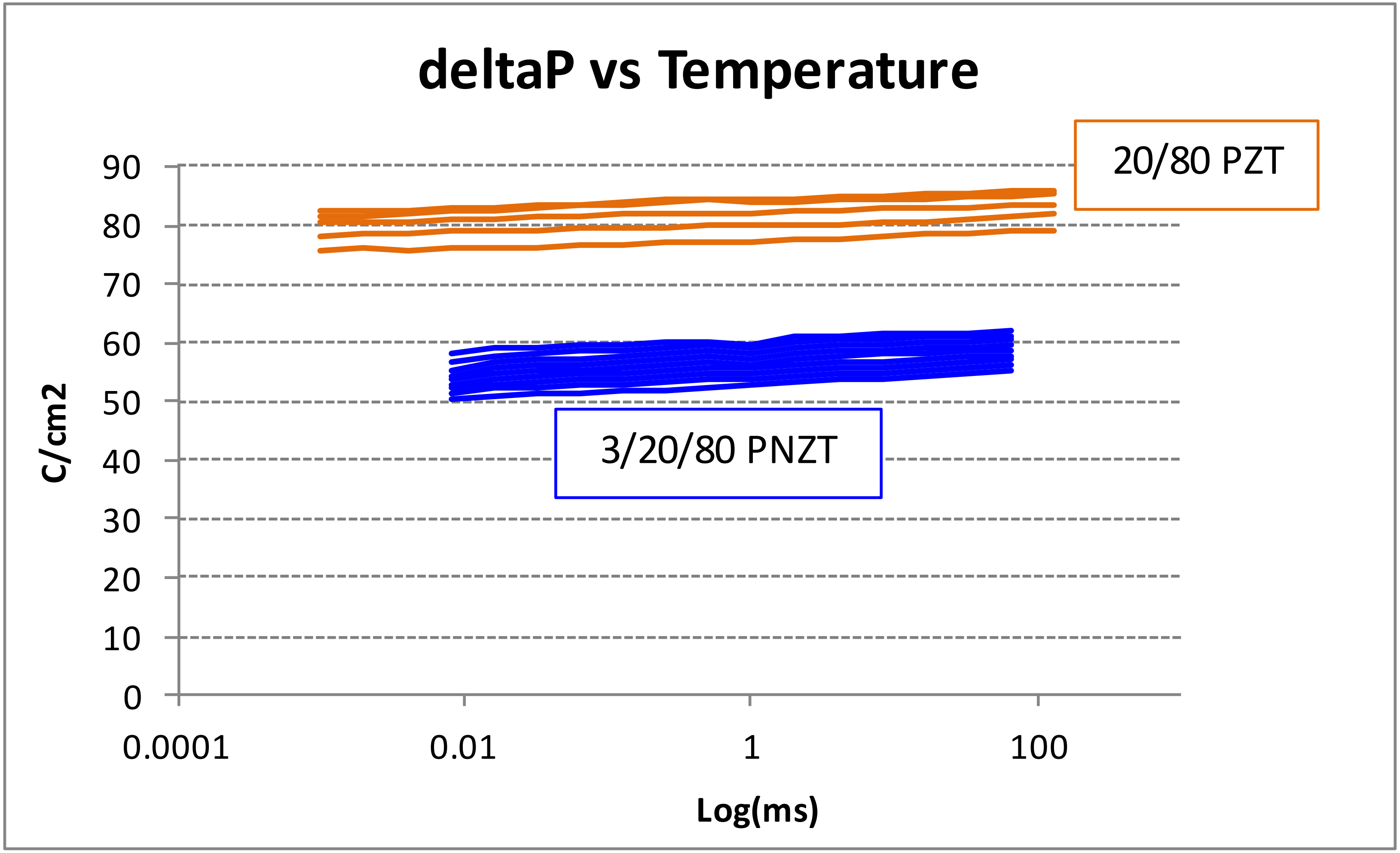
Remanent Polarization vs Temperature

40,000 µm² 3/20/80 PNZT vs 20/80 PNZT



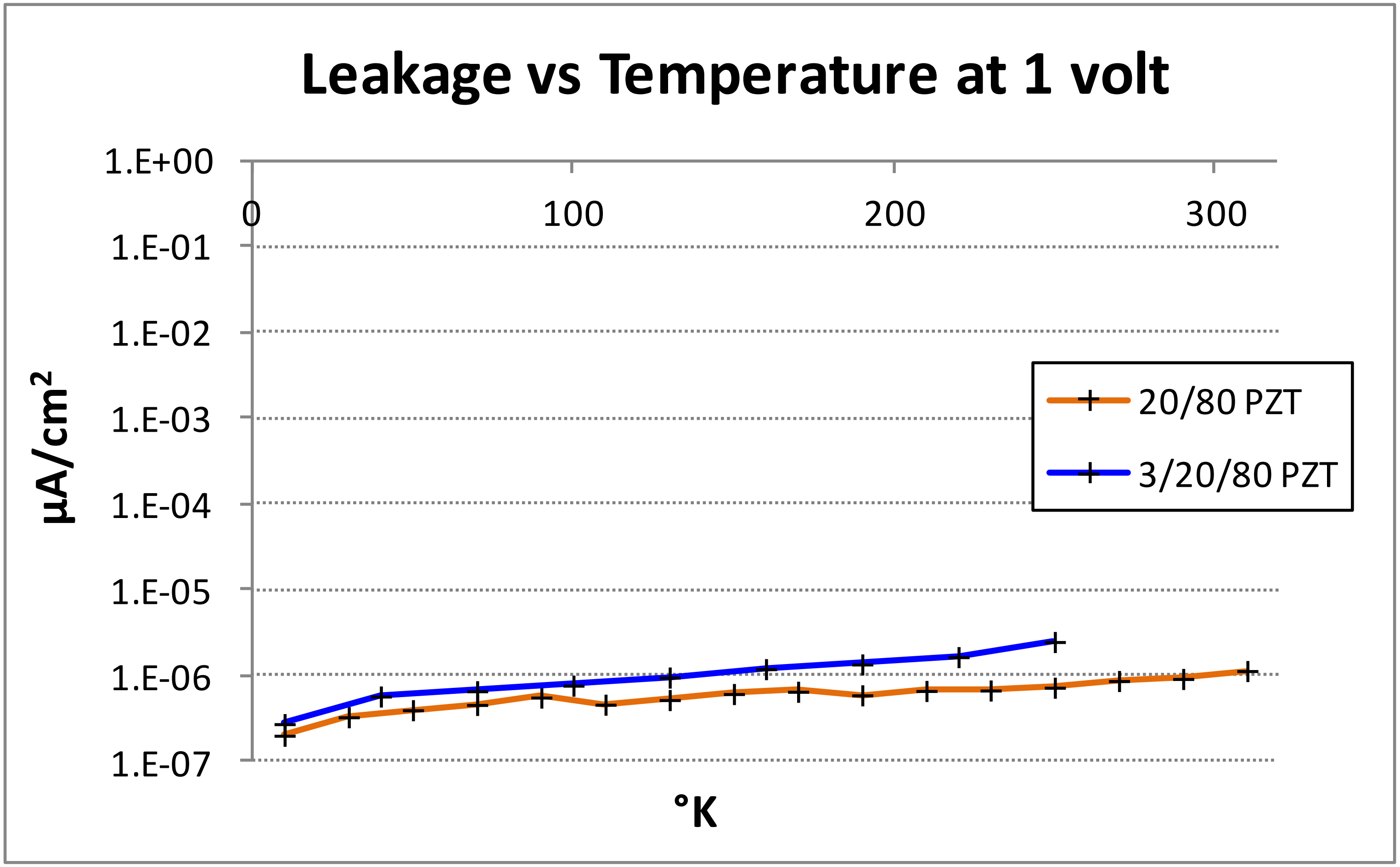
deltaP vs Temperature

40,000 μm^2 3/20/80 PNZT vs 20/80 PNZT



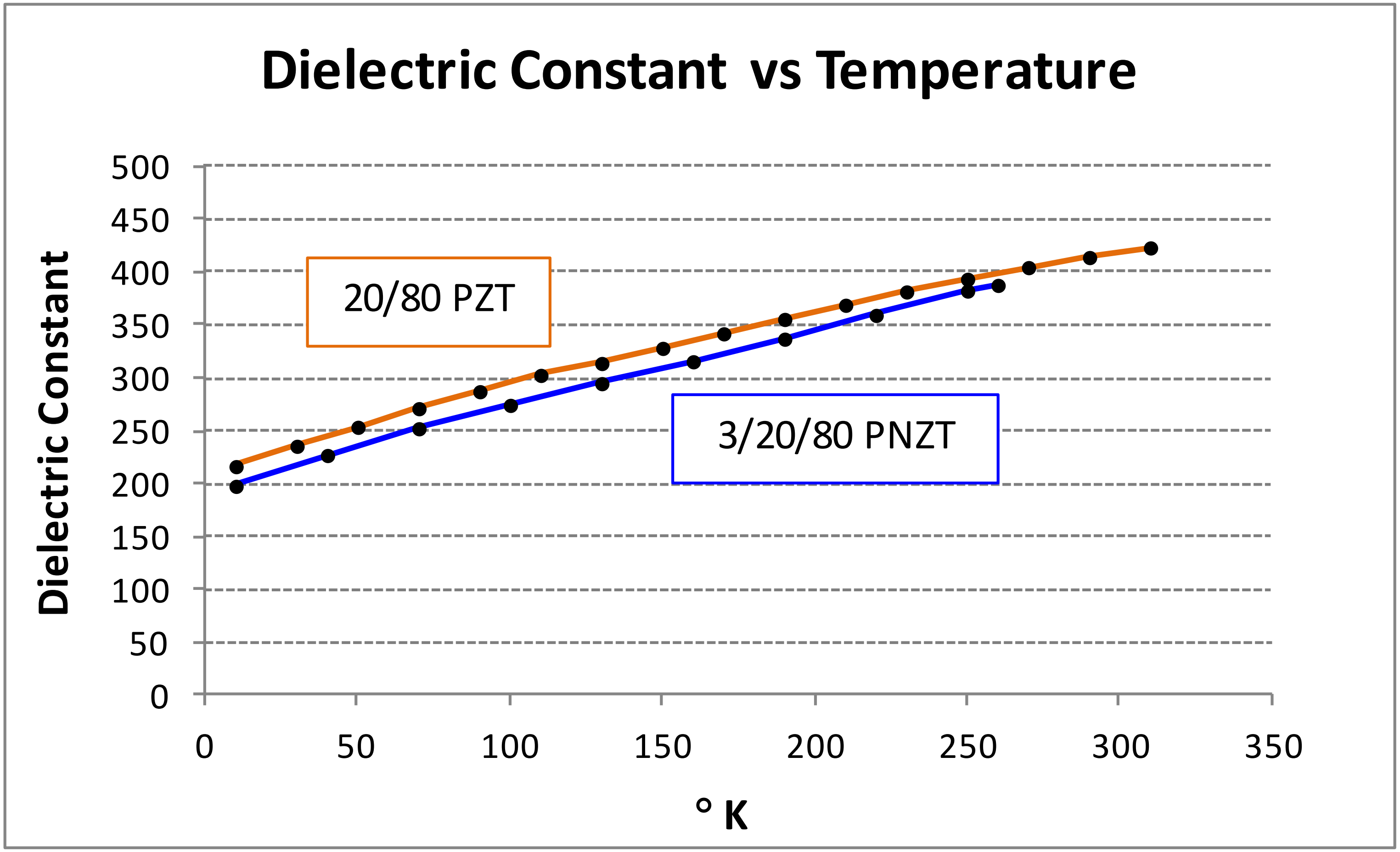
Leakage vs Temperature

40,000 µm² 3/20/80 PNZT vs 20/80 PNZT



Dielectric Constant vs Temperature

40,000 μm² 3/20/80 PNZT vs 20/80 PNZT



Conclusions

- It appears that tetragonal PZT *does not have a phase boundary* from room temperature down to 5 K.
- Of the parameters of the hysteresis loop for both undoped and niobium-doped PZT, *only the coercive voltages change with temperature.*
- Switching speed for both compositions *is unaffected by temperature.*
- Leakage *decreases as temperatures decrease.*

Conclusions

- Dielectric constant *decreases as temperature decreases*.
- Remanent polarization *increases as the temperature decreases*.
  - Switched polarization (P*) *increases* as temperature goes down.
  - Unswitched polarization (P^) *decreases* as temperature goes down.

20/80 PZT and its niobium-doped cousins appear to remain fully functional as memory devices down to 5 K.

Conclusions

- Lake Shore's temperature compensated probes on its cryogenic probe stations combined with Radiant's Vision data acquisition programming language running a Radiant tester make possible automated characterization of ferroelectric thin films properties over extremely wide temperature ranges.

- It is possible to measure and plot hysteresis, remanent polarization, leakage, and small signal capacitance over a large temperature range in a single pass on a single sample.

- Extremely consistent results can be achieved if *a single capacitor is measured over the entire temperature profile*.