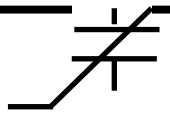

Polarization Properties of 20/80 PZT and 4/20/80 PNZT at Cryogenic Temperatures

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Summary

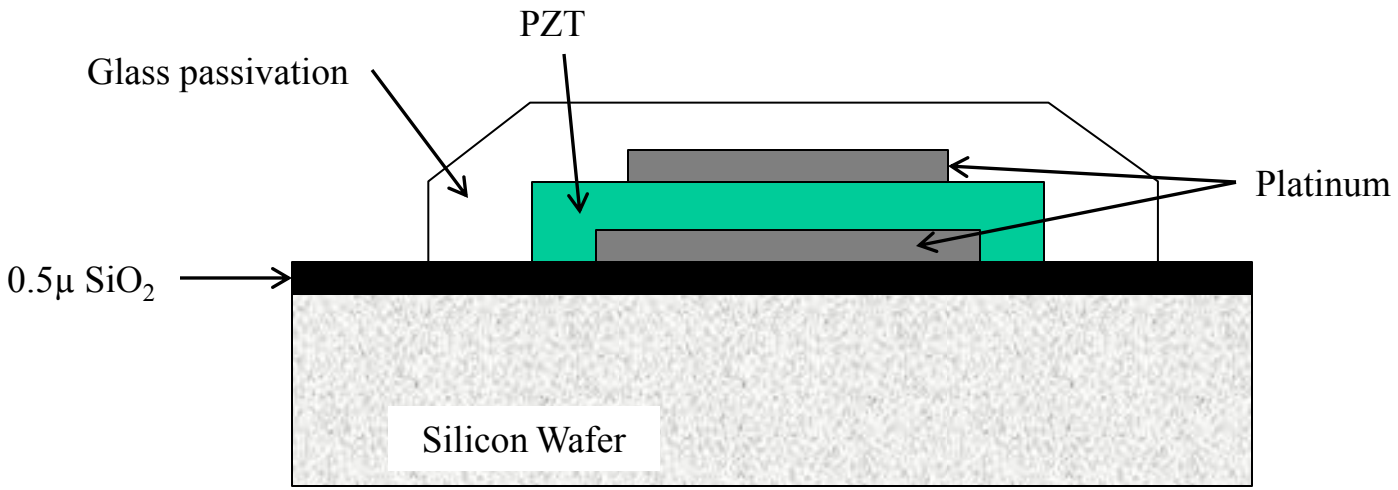
- Working with Lake Shore Cryotronics, Radiant measured the hysteresis loops of 20/80 PZT and 4/20/80 PNbZT thin ferroelectric film capacitors from 300°K down to 6.5°K.

- Both capacitor types functioned properly with little change in electrical properties over the entire temperature change except for an increase in coercive voltage with decreasing temperature.

- Retention tests executed over the change in temperature indicate that there is probably no material phase change from room temperature down to 6.5°K for these compositions.

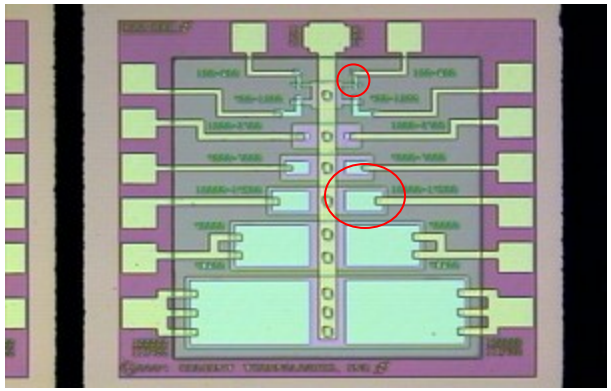
- Initial indications are that fatigue *decreases* in 20/80 PZT with decreasing temperature.

Test Samples

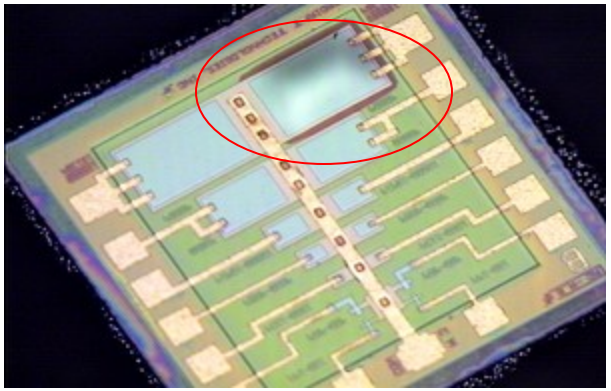


- The thin PZT film capacitors have platinum electrodes, top glass passivation, and chrome/gold metal interconnect (not shown).
- The platinum is patterned by lift-off photolithography.
- PZT is wet etched.

Test Samples

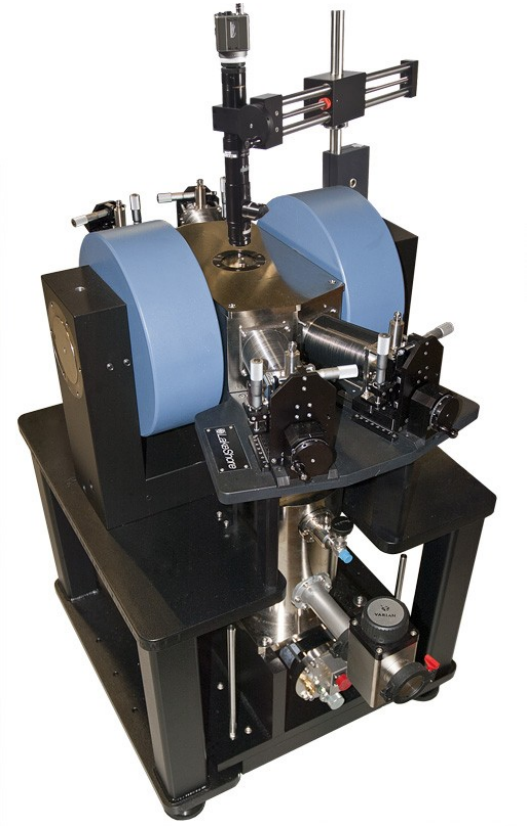


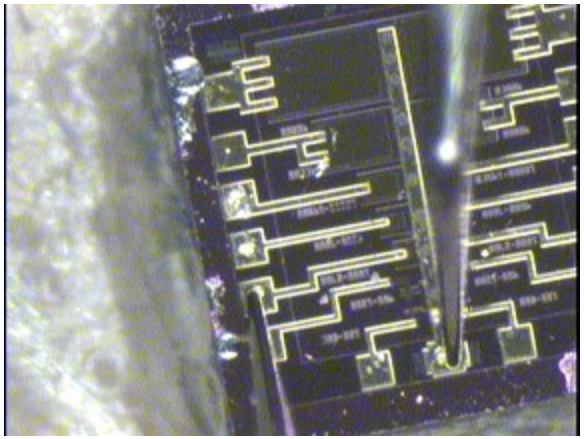
2600Å 20/80 PZT with areas of 10,000μ^2 and 100μ^2. The capacitors are clamped to the substrate surface.



1μ 4% Nb-doped 20/80 PZT with an area of 100,000μ^2. The silicon substrate has been etched out from underneath the capacitor resulting in a 2μ-thick membrane that bows naturally from the die surface.

Lake Shore Cryotronics Probe Station

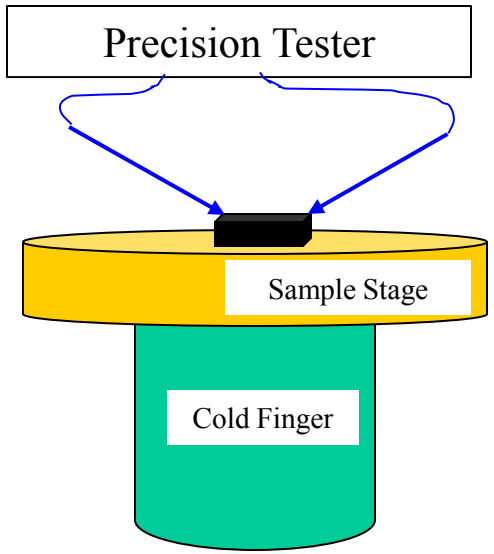




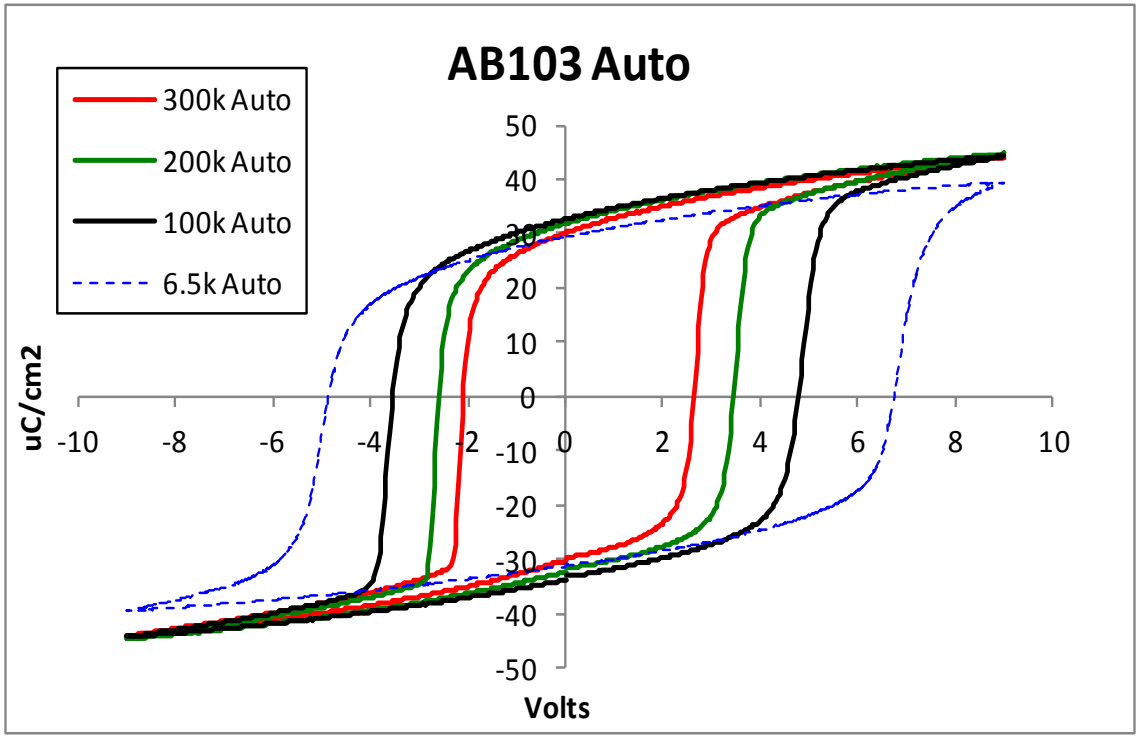
The Lake Shore Cryotronics Model CRX-EM-HF probe station is capable of 400 K down to ~6 K. The chamber has an integrated ±0.6 Tesla electromagnet and up to four micropositioners with which to contact the sample. Lake Shore has special probe tips, not used in this experiment, which can maintain electric contact with the sample while the temperature is changing so the probes do not have to be lifted. Magnetic field was not used for these measurements although will be explored in future measurements.

Lake Shore Cryotronics Probe Station

- The Lake Shore Cryotronics CRX-EM-HF chamber has a sample stage that is thermally connected to the cryocooler cold finger. The stage has an integrated sensor and heater for controlled operation from cryogenic to above room temperature.
- The cold finger was first dropped to base temperature while the sample stage was maintained at room temperature.
- The sample stage was then set to the first test profile temperature and testing began.
- The initial drop to base temperature by the cold finger took about two hours.
- 100 K changes in sample stage temperature once the cold finger stabilized took about 15 minutes including stabilization delay at the new temperature.

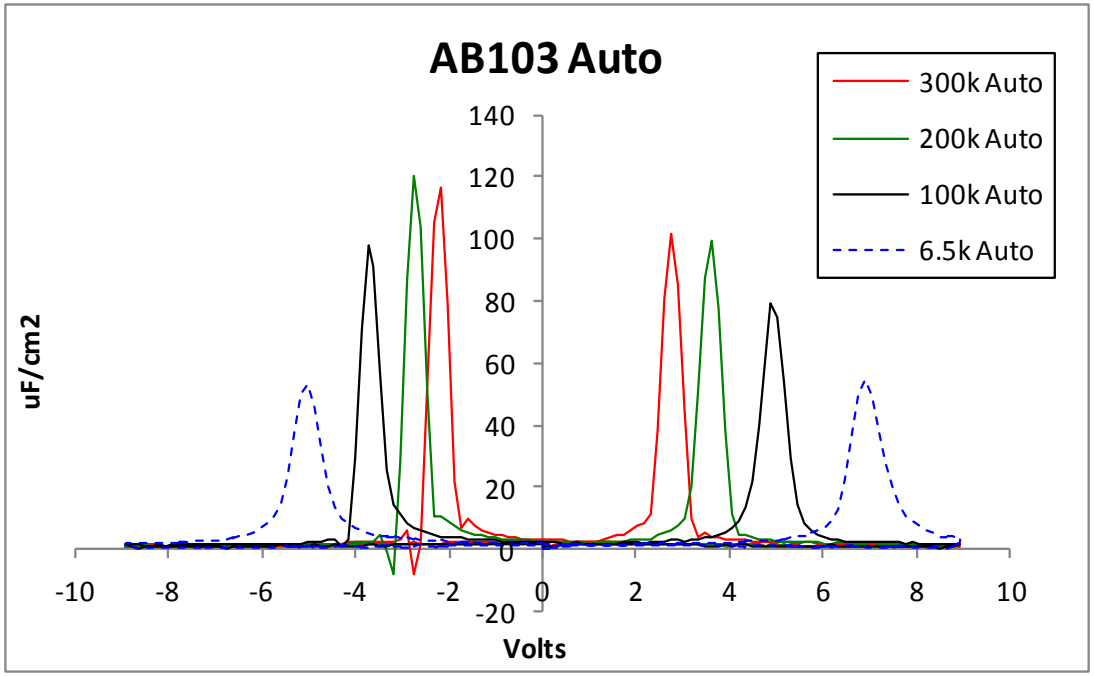


20/80 PZT Hysteresis



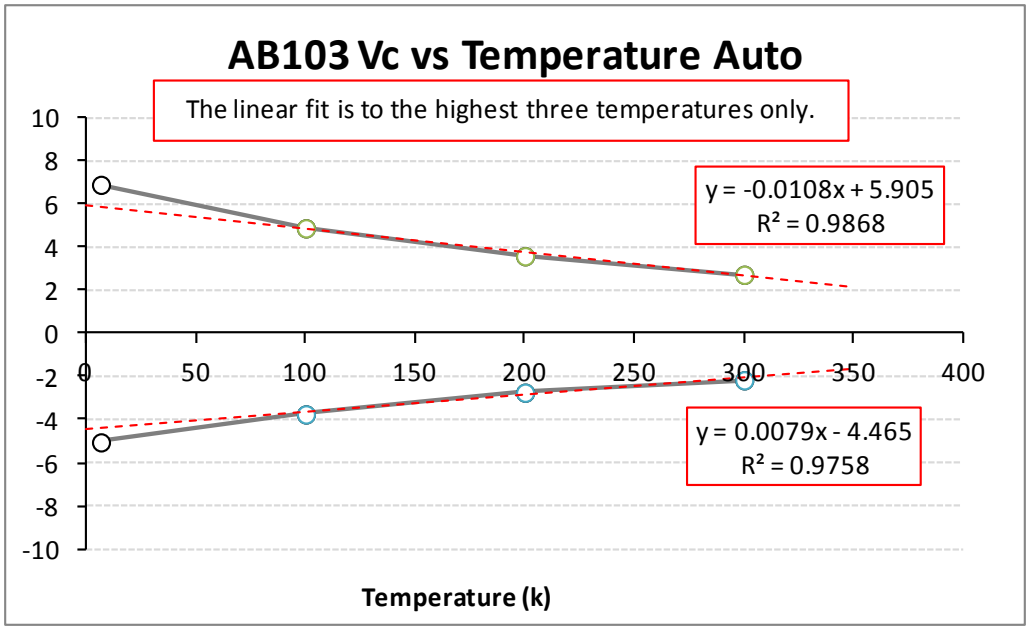
- Hysteresis loops of 2600Å 20/80 PZT overlay exactly except for a change in the coercive voltages from 300°K down to 100°K. The capacitor was fully functional at 6.5°K (blue dashed line) but the loop shape was slightly different, indicating some sort of change in the material properties below 100°K.

20/80 PZT Hysteresis



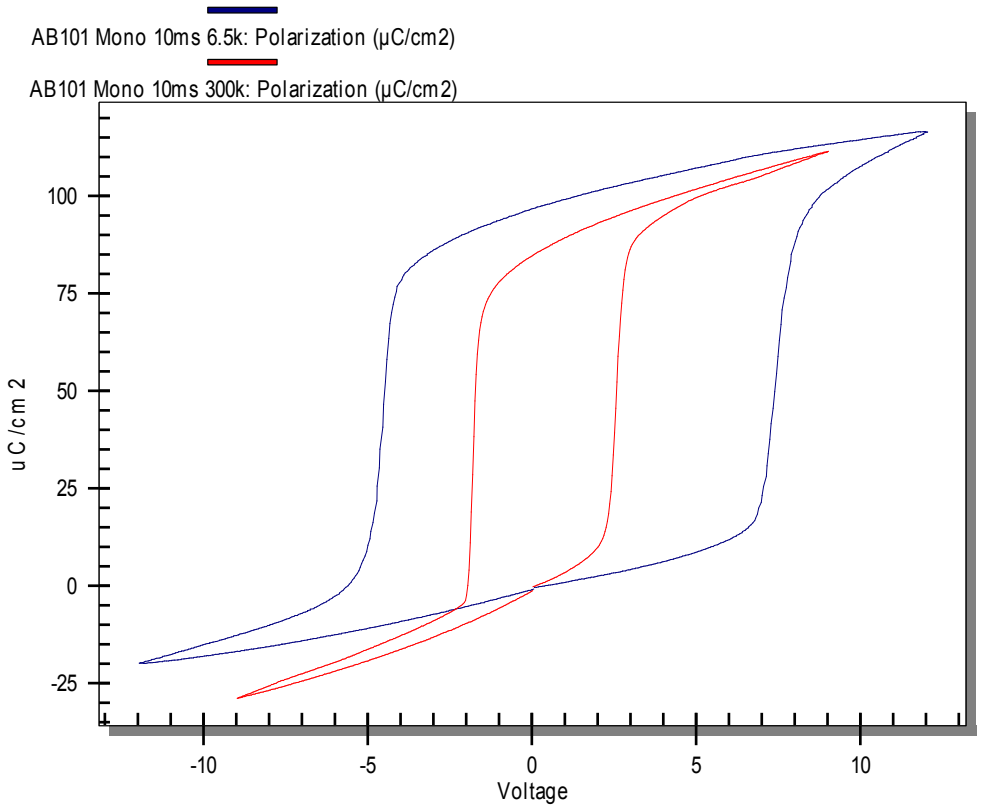
- The normalized CV of the 20/80 loops show the progressively increasing coercive voltages as the temperature dropped followed by a change in the shape of the loop at 6.5°K.

20/80 PZT Coercive Voltage



- The coercive voltage of the 20/80 PZT platinum-electroded capacitor appears to have increased at a rate of -9mV/°K with a 0.98 R^2 fit from 300°K down to 100°K.
- Below 100°K, the coercive voltage changed according to some different function.

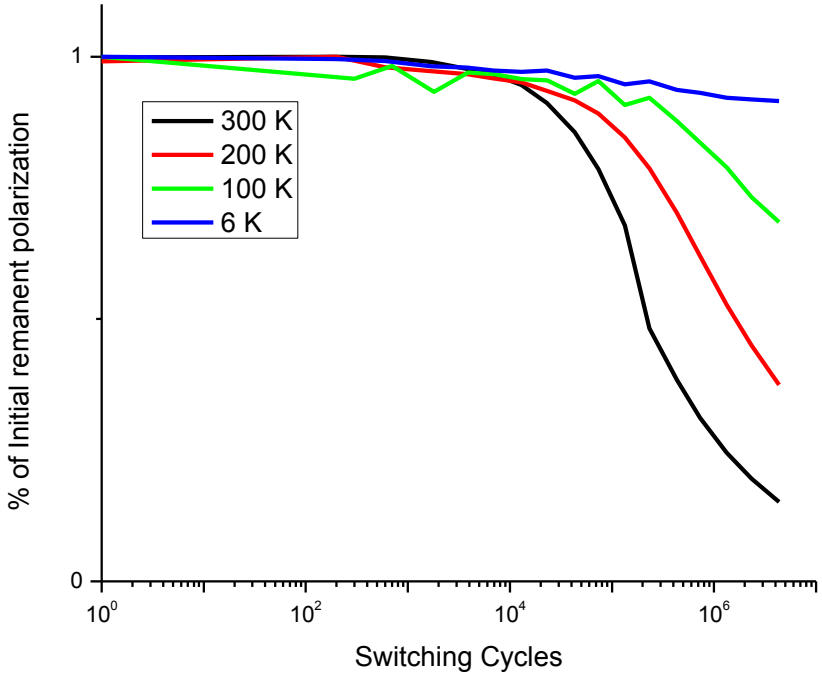
20/80 PZT Retention



- The plot shows retention by a 100µ^2 20/80 PZT capacitor.
- The red loop in the plot was executed at 300°K. The capacitor was untouched and open circuit until a single loop (blue) was executed with no preset at 6.5°K.
- It appears that no phase change occurred from room temperature down to 6.5°K.

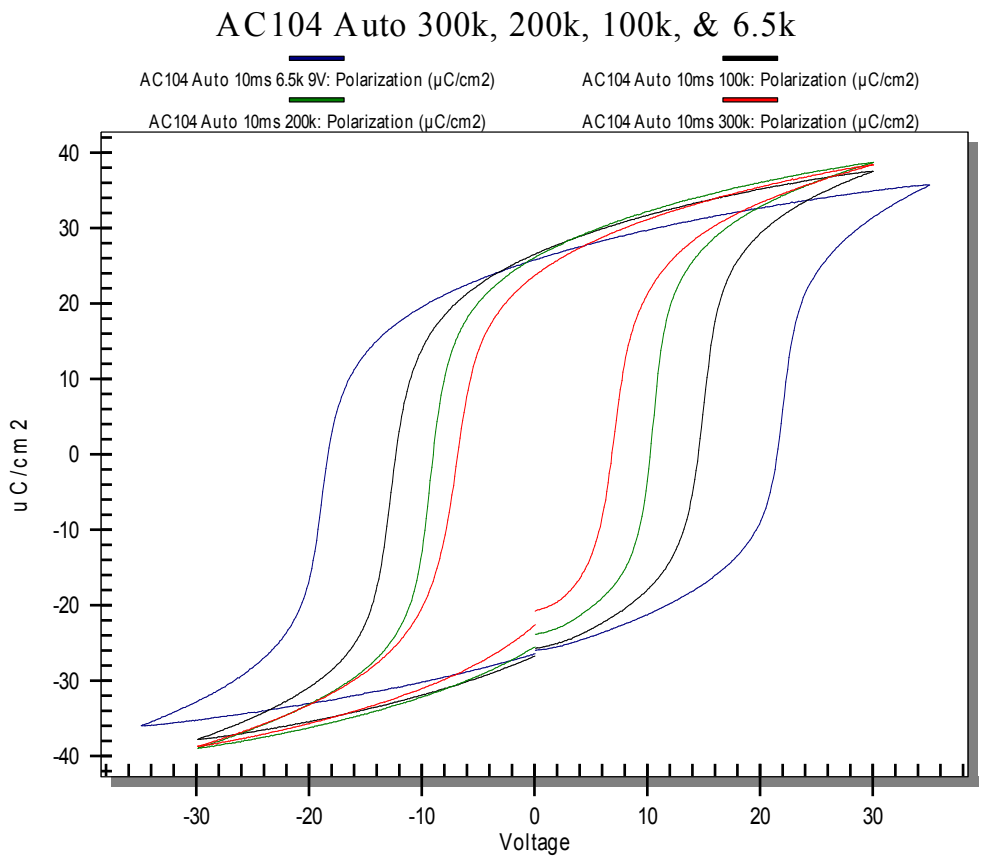
20/80 PZT Fatigue

Data acquired by Dr. David Daughton of Lakeshore Cryotronics and provide to Radiant. Data presented with permission. This data was taken in a Lake Shore Cryotronics CRX-4K *chamber.*



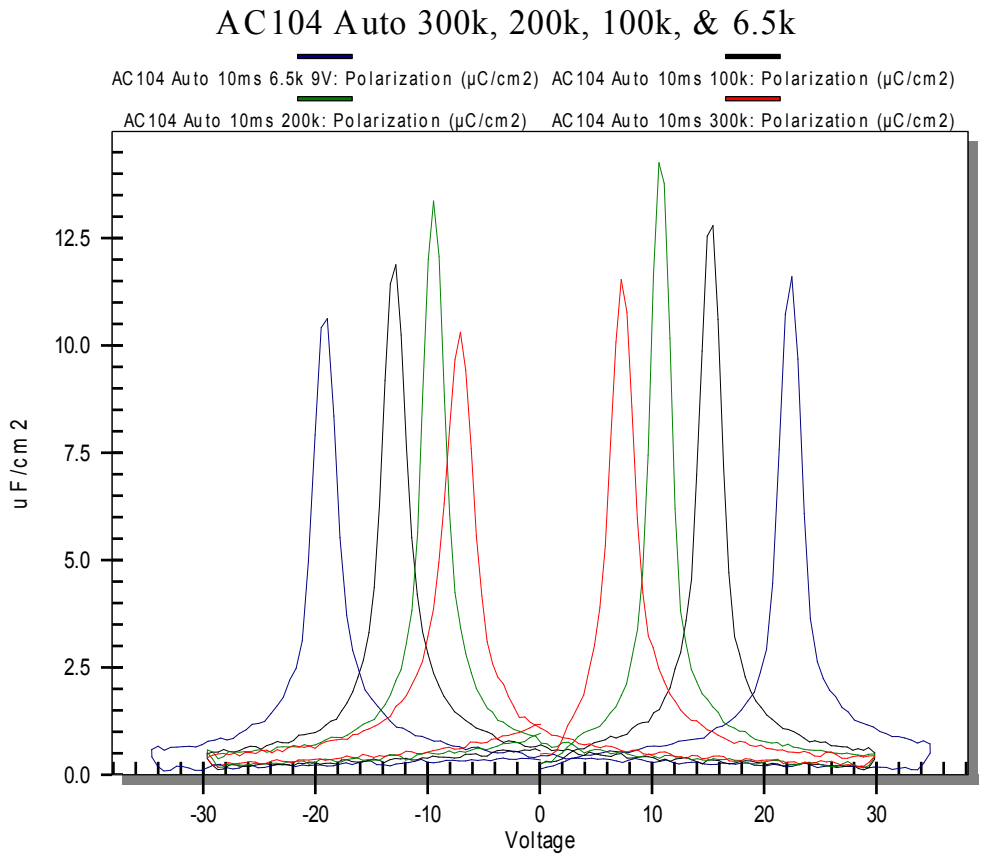
- Fatigue of 20/80 PZT capacitors over the temperature range appeared to decrease as the temperature decreased!
- This experiment was executed with capacitors from a single die but of different areas. The fatigue tests must be repeated on identical capacitors on different dice to verify these initial results.

4/20/80 PZT Membrane Hysteresis



- 4% niobium-doped 20/80 PZT functioned normally over the entire temperature range.

4/20/80 PZT Membrane Hysteresis



- This capacitor is a membrane that physically moves 2µ at room temperature when actuated at 20 volts. Total displacement at 6.5°K has not yet been measured.
- The coercive voltage of the capacitor changed from 7 volts at 300°K to 20 volts at 6.5°K.

Conclusion

- *20/80 thin PZT films remain fully functional at 6.5 °K!*
- The capacitors are ferroelectric over the entire temperature range of 300°K down to 6.5°K.
- The capacitors retain their polarization state in one temperature step from 300°K down to 6.5°K
  - There appears to be no phase change in the PZT over that temperature range.
- The primary effect of reduced temperature is an increase in the coercive voltages.
- Tentative results indicate that fatigue of 20/80 PZT reduces as the temperature goes down.

Acknowledgements

Radiant would like to think Scott Yano and Dr. David Daughton of Lake Shore Cryotronics in Westerville, Ohio for their assistance in this research effort and for the use of their cryogenic chambers.