In-situ measurements: neutron and synchrotron x-ray

17th June 2009

NPL's experiences

Mark Stewart, Markys Cain, Mike Reece

and

Beam-line scientists!!

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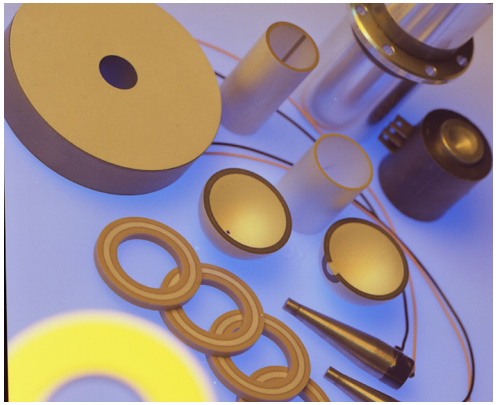
National Physical Laboratory

Multifunctional Materials research at NPL

- Metrology – science of measurement
- Characterisation and performance of:
  - Piezoelectrics
  - Pyroelectrics
  - Multiferroics - - magnetoelectrics
- Scanning imaging methods based on AFM, Laser modulation and interferometry developed
- Real operating environments factored into our work



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Ferroelectric Materials Polarisation Field Measurements: The Anatomy of a PE Loop

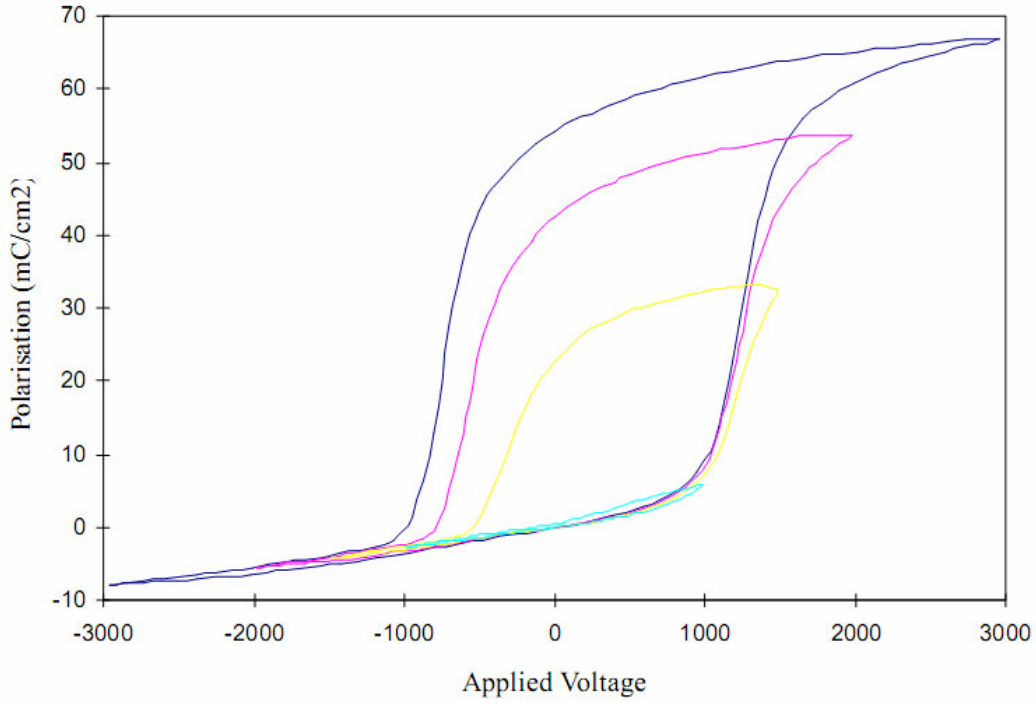


Figure 6 Polarisation measurements for a hard PZT with the polarisation offsets added.

Ferroelectric or Not?

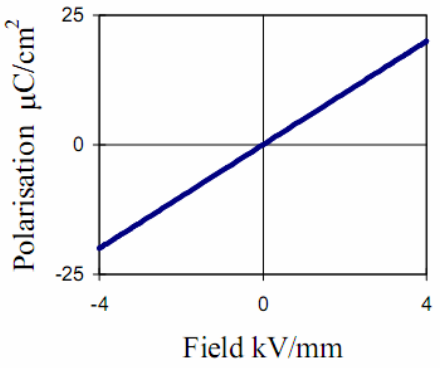


Figure 1a): Ideal linear capacitor response

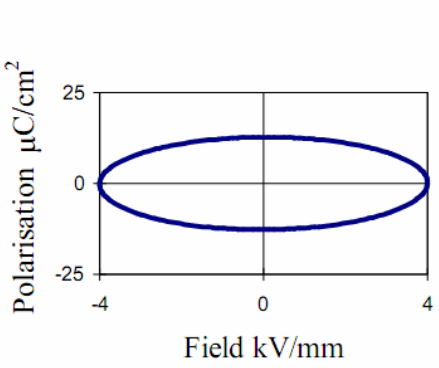
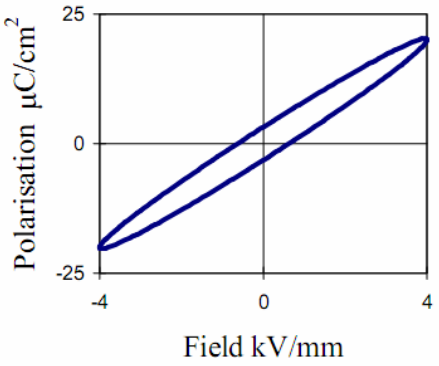
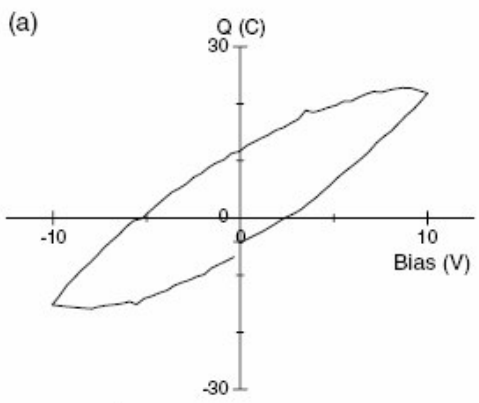


Figure 1b) Ideal resistor response





Scott, J. F. Ferroelectrics go bananas
Journal of Physics: Condensed Matter, **2008**, *20*, 021001

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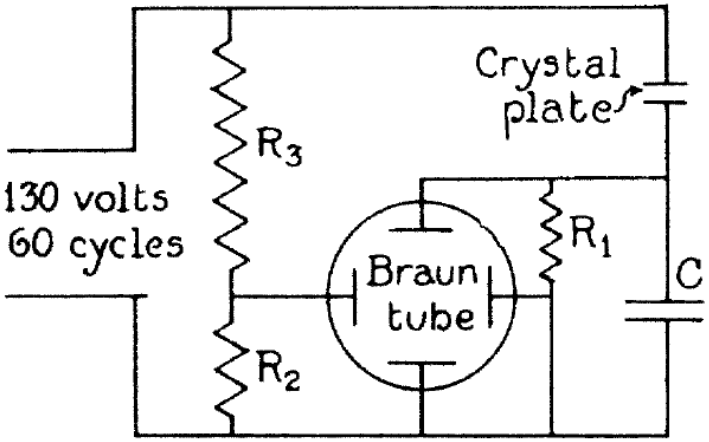
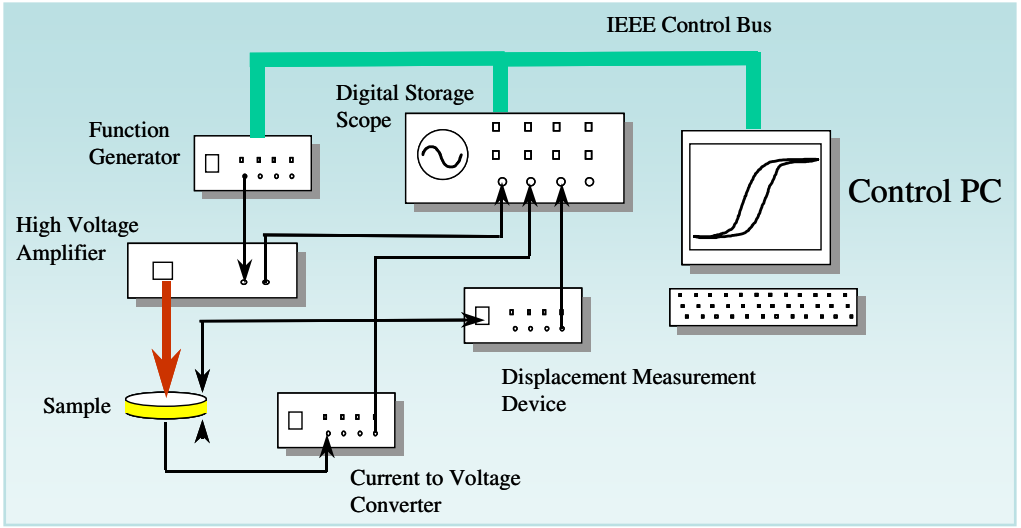
How to do PE Loop Measurement



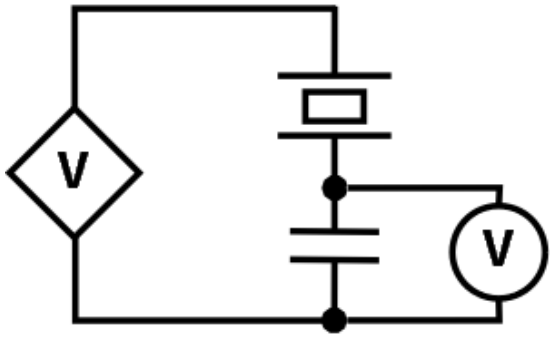


Sawyer, C. B. & Tower, C. H.
Rochelle Salt as a Dielectric
Phys. Rev., American Physical Society, **1930**, *35*, 269-273

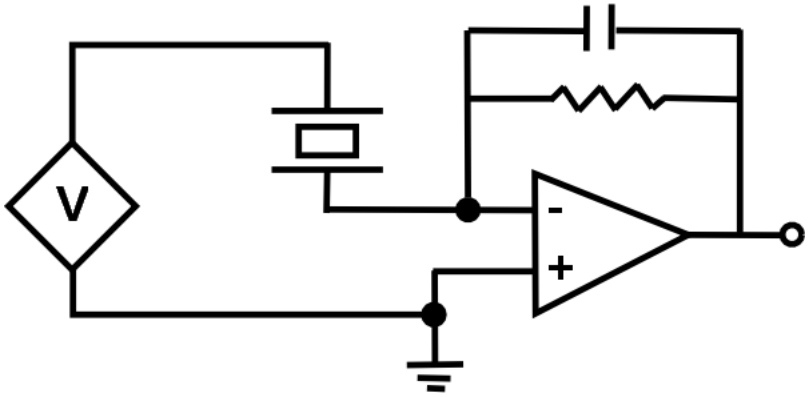
2009

Polarisation Measurement in detail

Low Frequency application

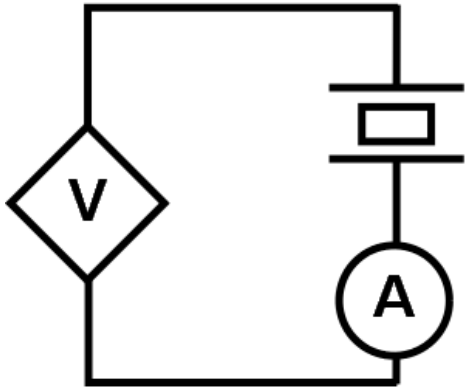


Shunt Capacitor



Charge Amplifier

High Frequency application



Current to Voltage Converter

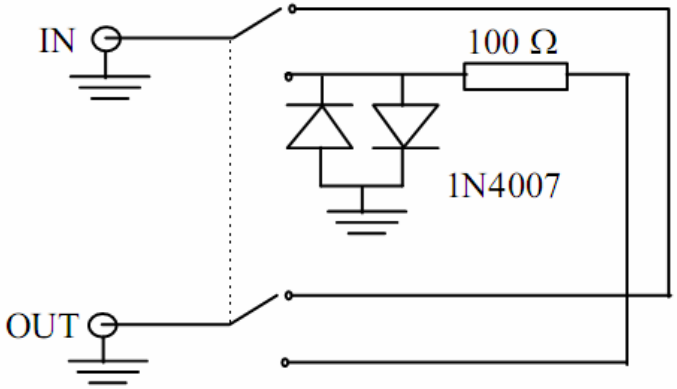
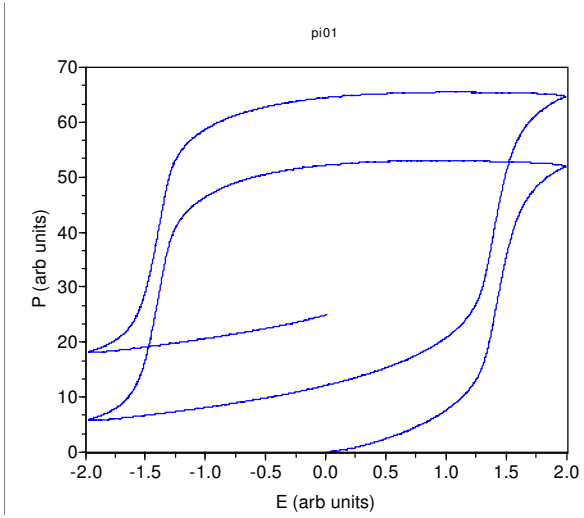


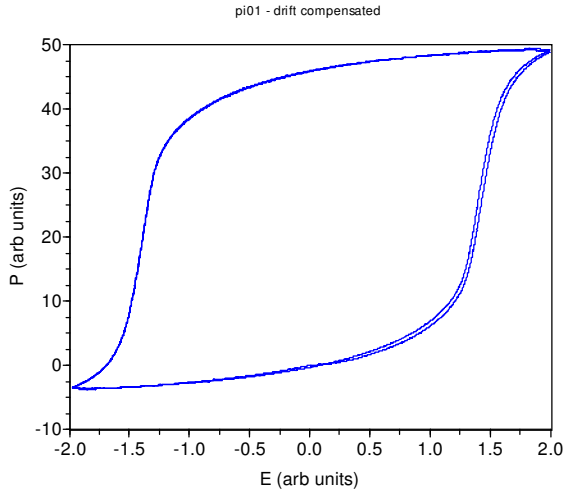
Figure 8: In-line circuit protection

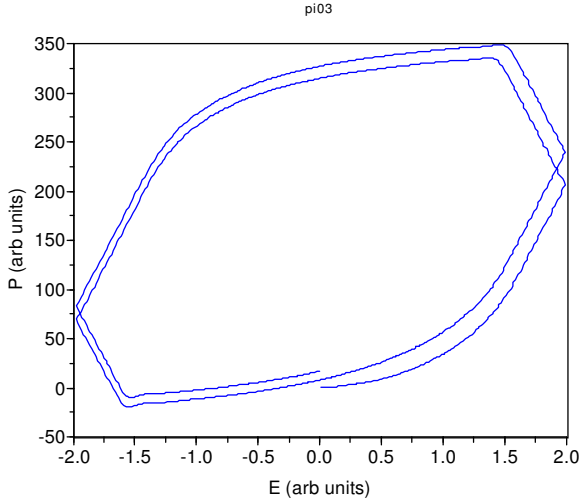
Artefacts

Drift

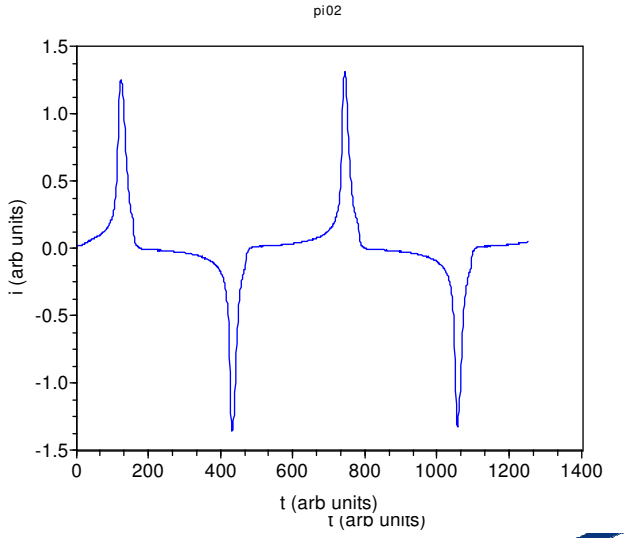


Compensate ->



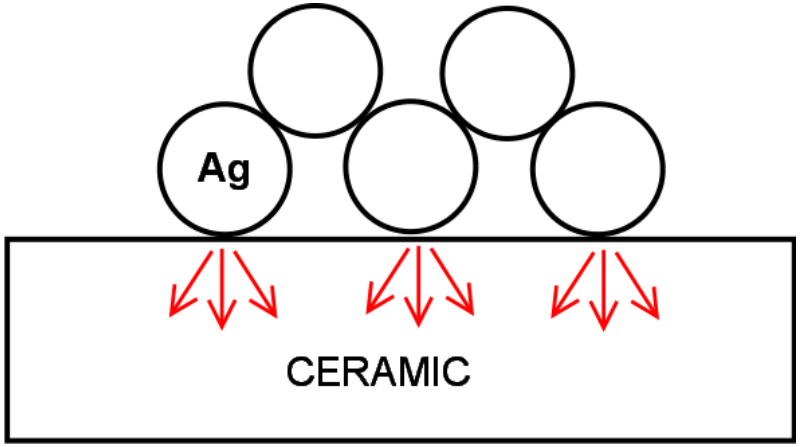


Clipping

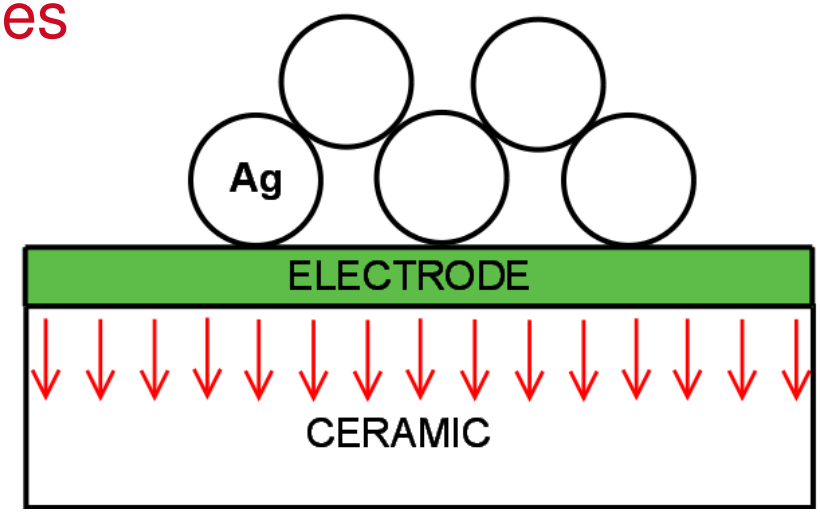


Current vs time

ELECTRODES

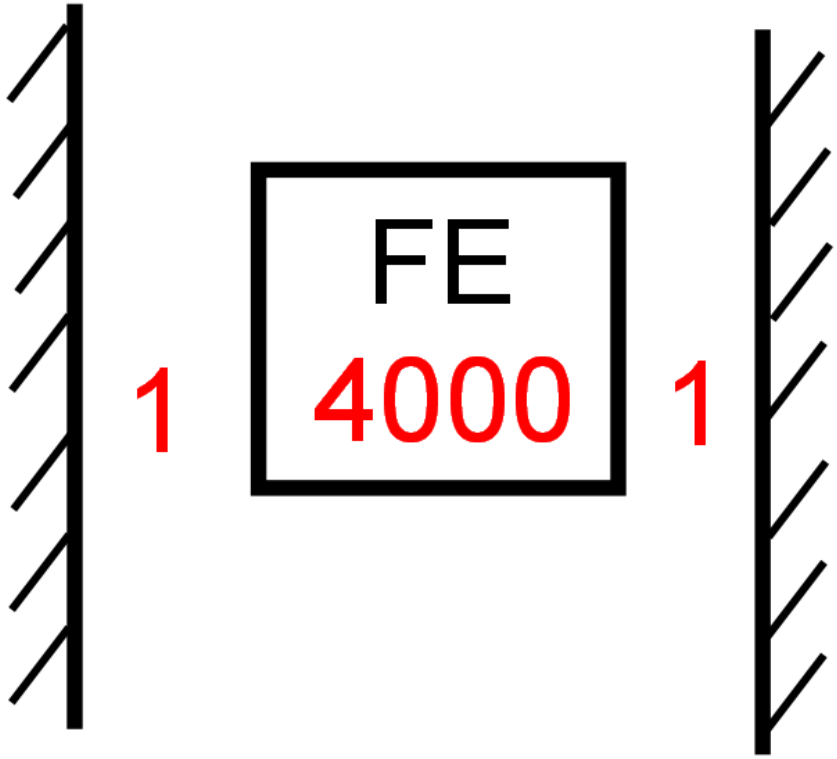


Electric field lines



Uniformity of field and voltage drop

PERMITTIVITY



Do you really know what voltage your sample sees?
Air gaps etc drop the field across the sample

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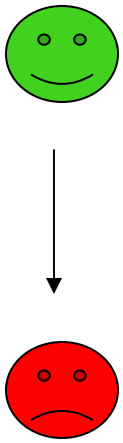
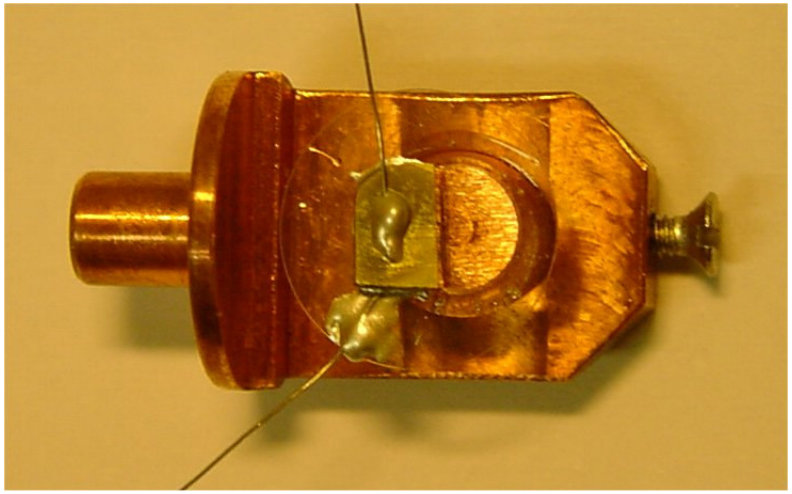
Materials



- Conductors
  - Fired silver electrodes
  - Sputtered Electrodes
  - Silver loaded epoxy
  - Silver/Carbon DAG
  - Self adhesive copper tape
- Join using
  - soldering
  - wire Bonding
  - mechanical

- Insulators
  - Glass
  - Macor
  - Sapphire
  - PTFE
  - Varnish



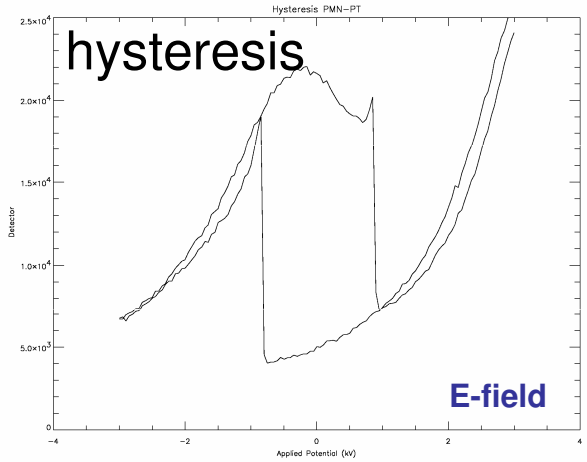
Exploring in-situ electric field induced phase changes for the first time with x-rays in single crystal piezo material

XMaS the UK-CRG

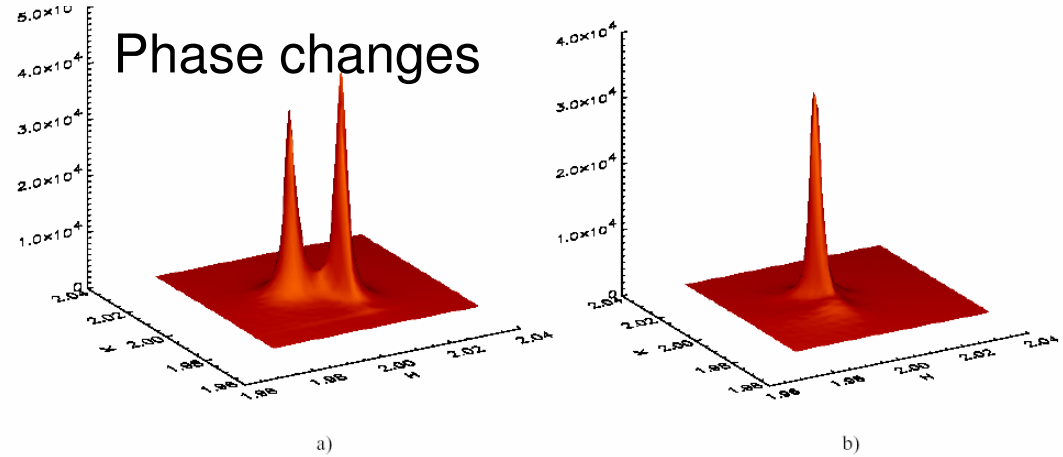
Safety

Electric Field Cell

- ± 3 kV
- Peltier Temperature Control



Hysteresis observed on PMN-PT during the electric field induced rhombohedral to orthorhombic phase transition. The detector signal is from the pseudo-cubic (222) peak.



(a) rhombohedral variants at +3kV and (b) orthorhombic variants at -3kV.

M.C. Cain M. Reece (QMUL) NPL Functional Material Group,BSSM 2008

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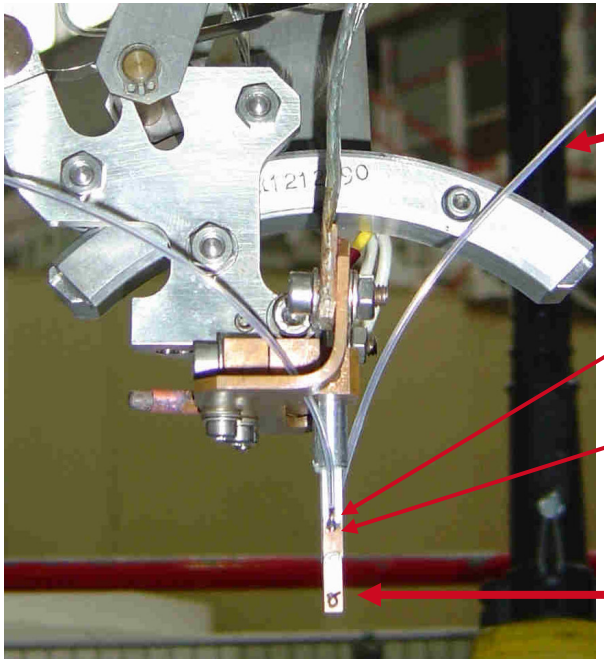
Neutron – ISIS: Single crystal piezo (SXD)

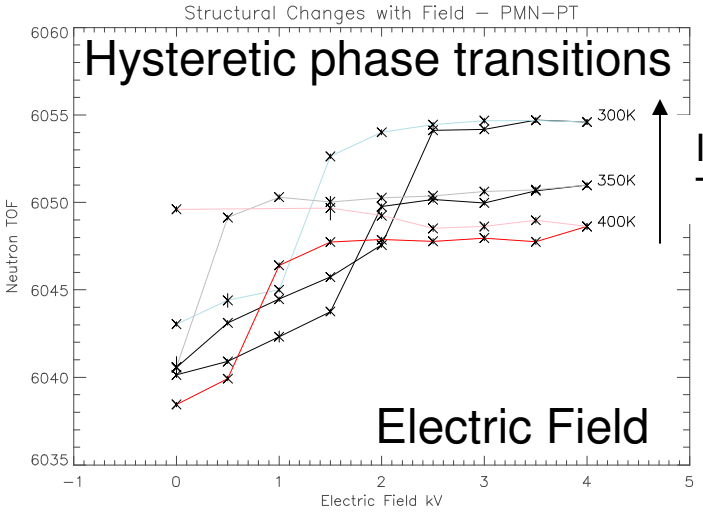


PTFE insulation

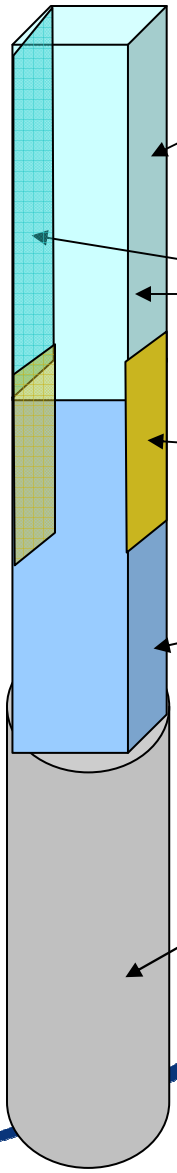
Strain relief pad

Soldered wires to copper shim

Silver solder wire to gold evap sample or silver frit electrodes



Gold evaporation: stencil mask on sample



PMN-PT piezoelectric square section sample: 3mm by 3mm by 9mm long

Gold evaporated electroded opposite surfaces

Copper sheet electrodes

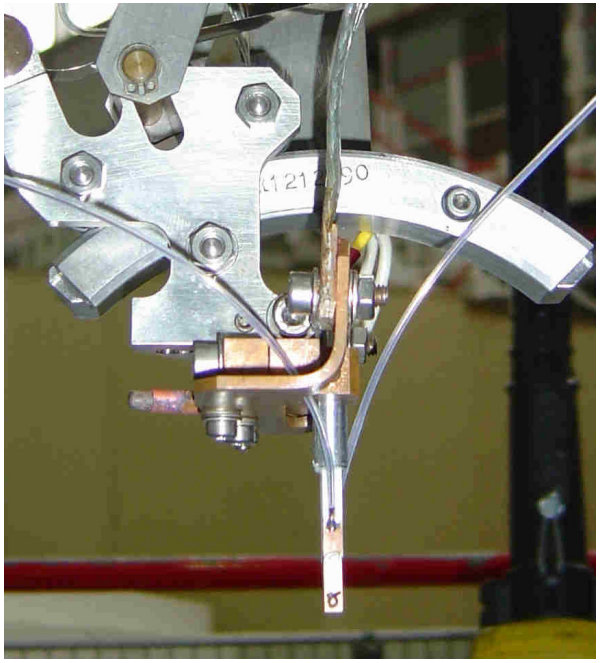
Insulating ceramic square stub: Al2O3

All pieces superglue

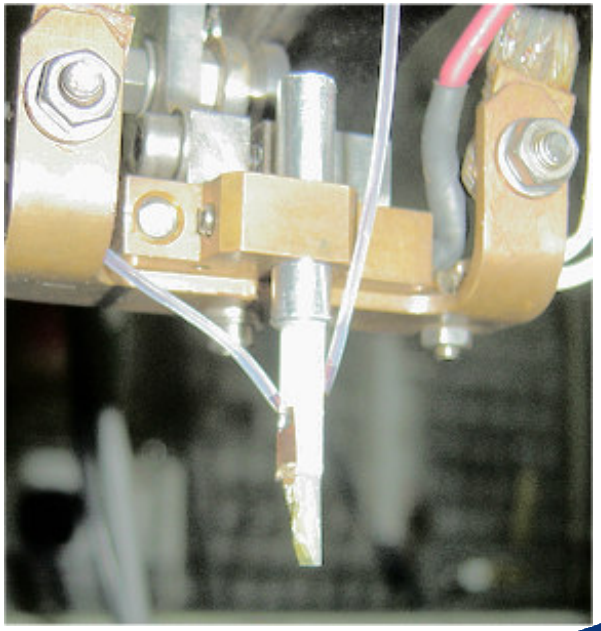
Aluminium round mounting stub – 28mm long.

…is the sample still OK?

- Get out the Multimeter



- Short Circuit 
- Open Circuit 
- Capacitance (1kHz) 



Way forward

- Thank you for all your contributions
- NPL and XMaS will develop a suite of web pages that describe: [Sept 09]
  - in-situ measurement issues
    - electroding, wiring, electronics, instrumentation issues and more
  - NPL PE-Hyst software code access
  - The presentations from today – with permission
  - Open discussion comments
  - How to access our facilities

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