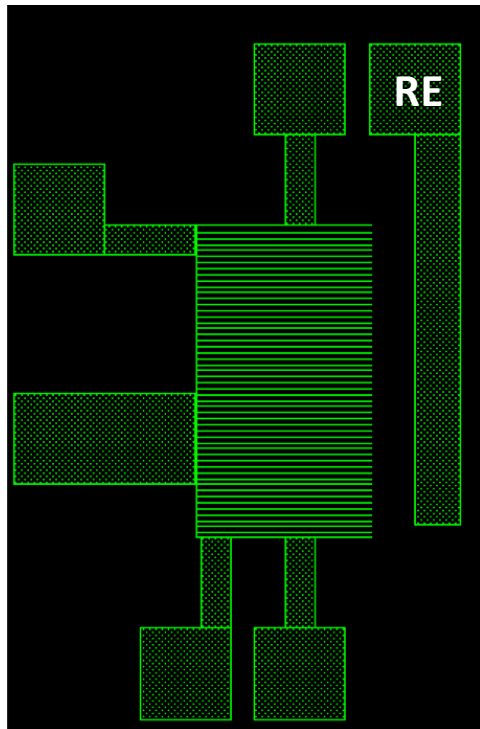


ReResearch Micro-FPE Experiments

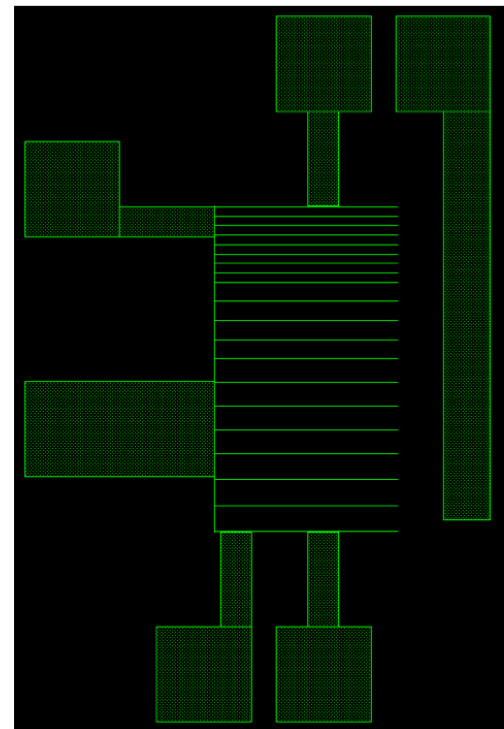
Jan 2015

Micro-FPE Chip

- 100 nm of Pd (or Pt) in 4 patterns:



- Finger pattern
- 20 μm wide lines
- 150 μm spacing
- SA = 0.0422 cm^2

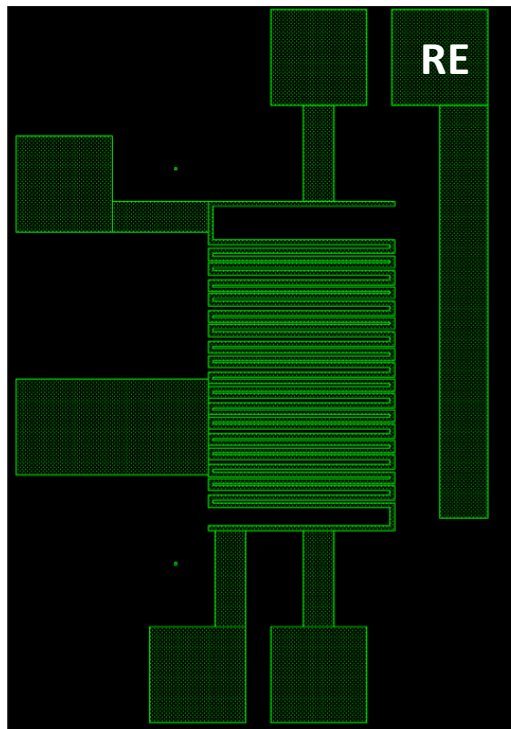


- Gradient finger pattern
- 20 μm wide lines
- Varying spacing
- SA = 0.0168 cm^2

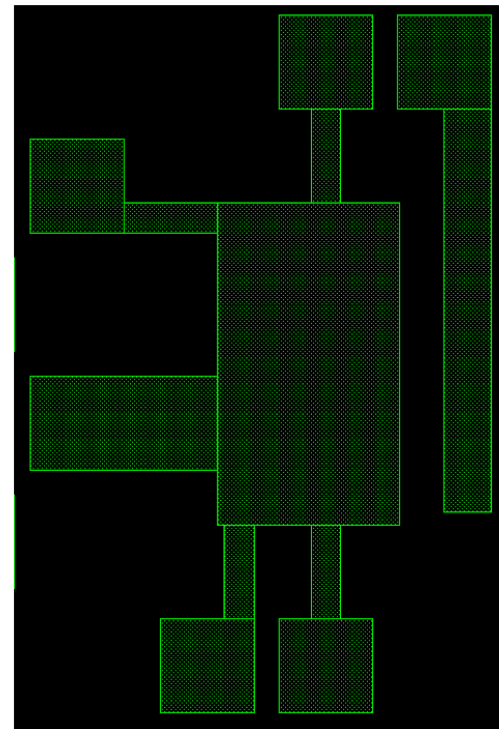
Test of diffusion limited behavior

Micro-FPE Chip

- 100 nm of Pd (or Pt) in 4 patterns:

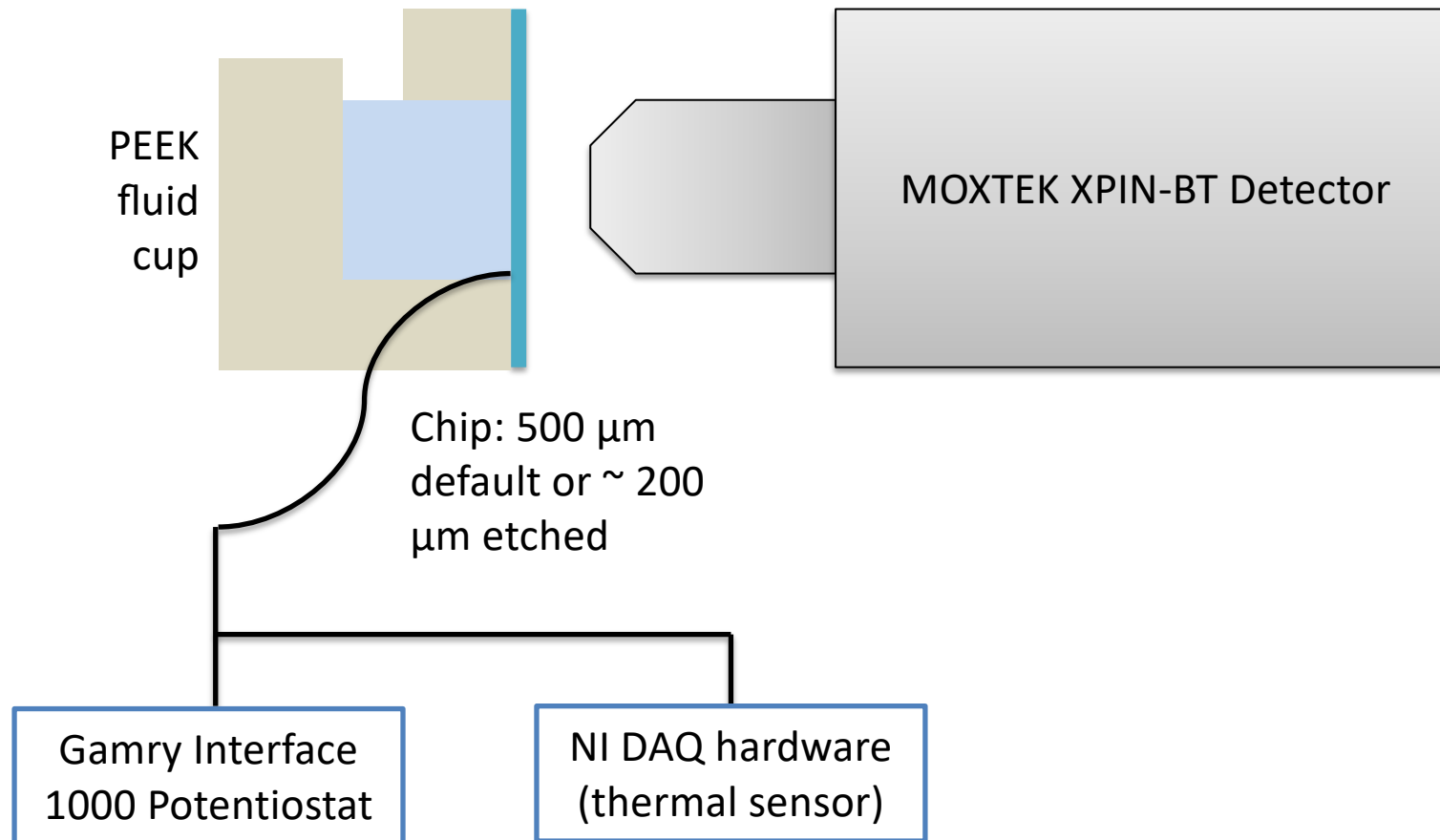


- Serpentine pattern
- 0.145 cm^2



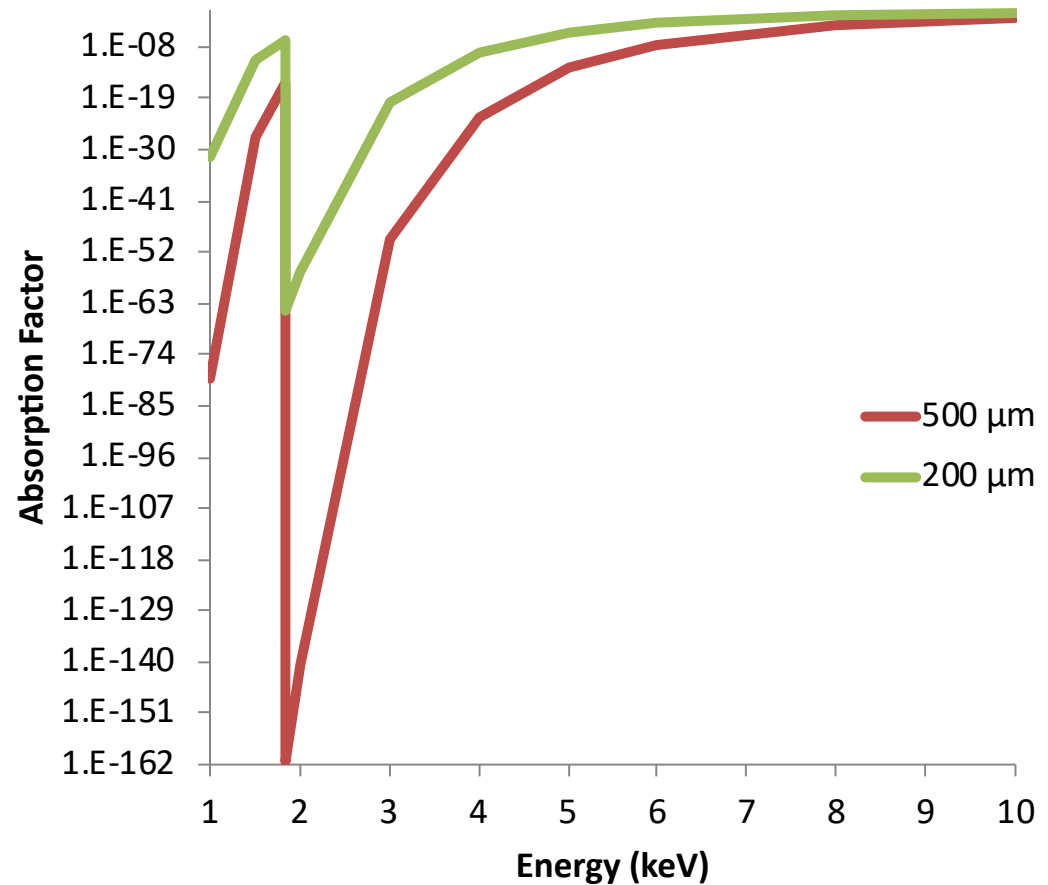
- Pad pattern
- 0.267 cm^2

Experimental Setup (Horiz.)



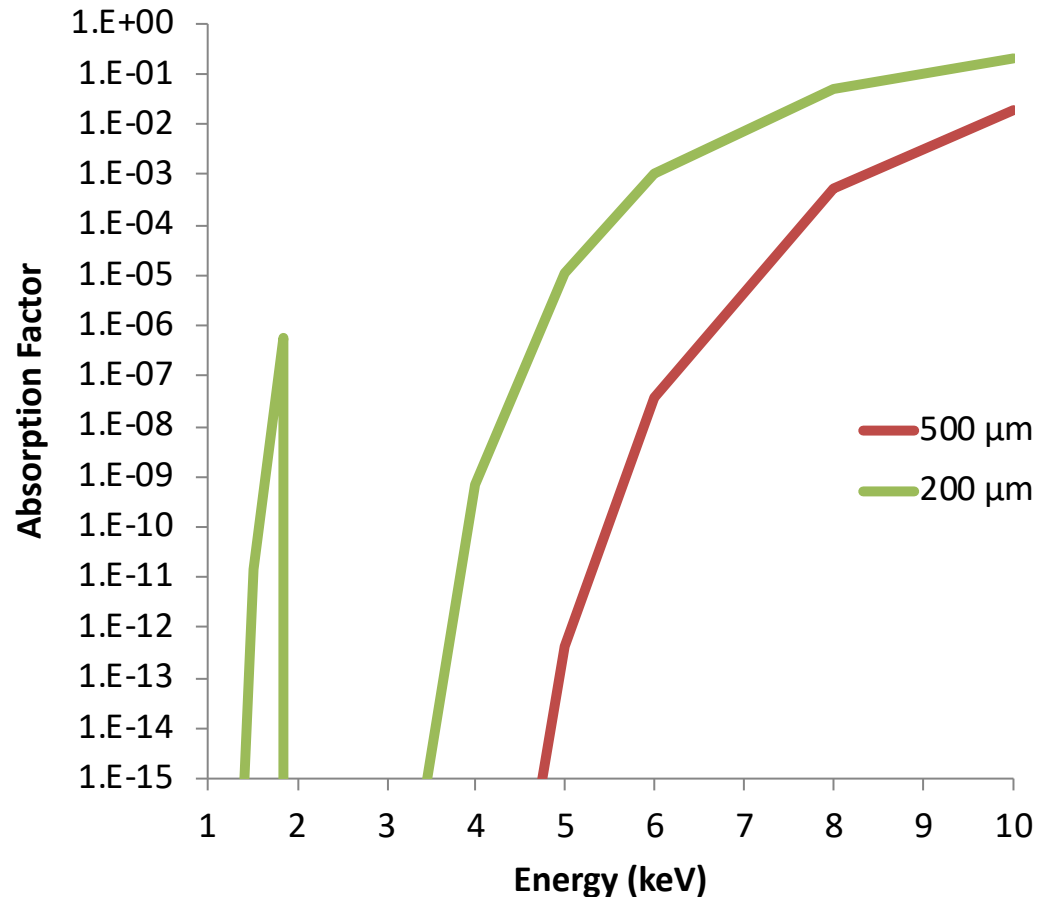
What to expect? Si Absorption

- Need ~ 10 events / minute in a narrow energy window to clearly distinguish from noise



What to expect? Si Absorption

- Events / min needed at 5 keV:
 - 500 μm : 10^{14}
 - ~ 10 nmol
 - 200 μm : 10^5
- 10 keV:
 - 500 μm : 10^3
 - 200 μm : 10
- Order of magnitude correction for detector solid angle



Detection Limits

- Need ~ 100 nmol reactions / min at 5 keV to detect with 500 μm thick silicon
 - Probably on edge of detection window
- Constraint relaxed at 6 keV, 10^5 lower absorption
- Restrict to surface?

Pattern	Pd Amount (nmol)
Finger	48
Grad. Finger	19
Serpentine	164
Pad	302

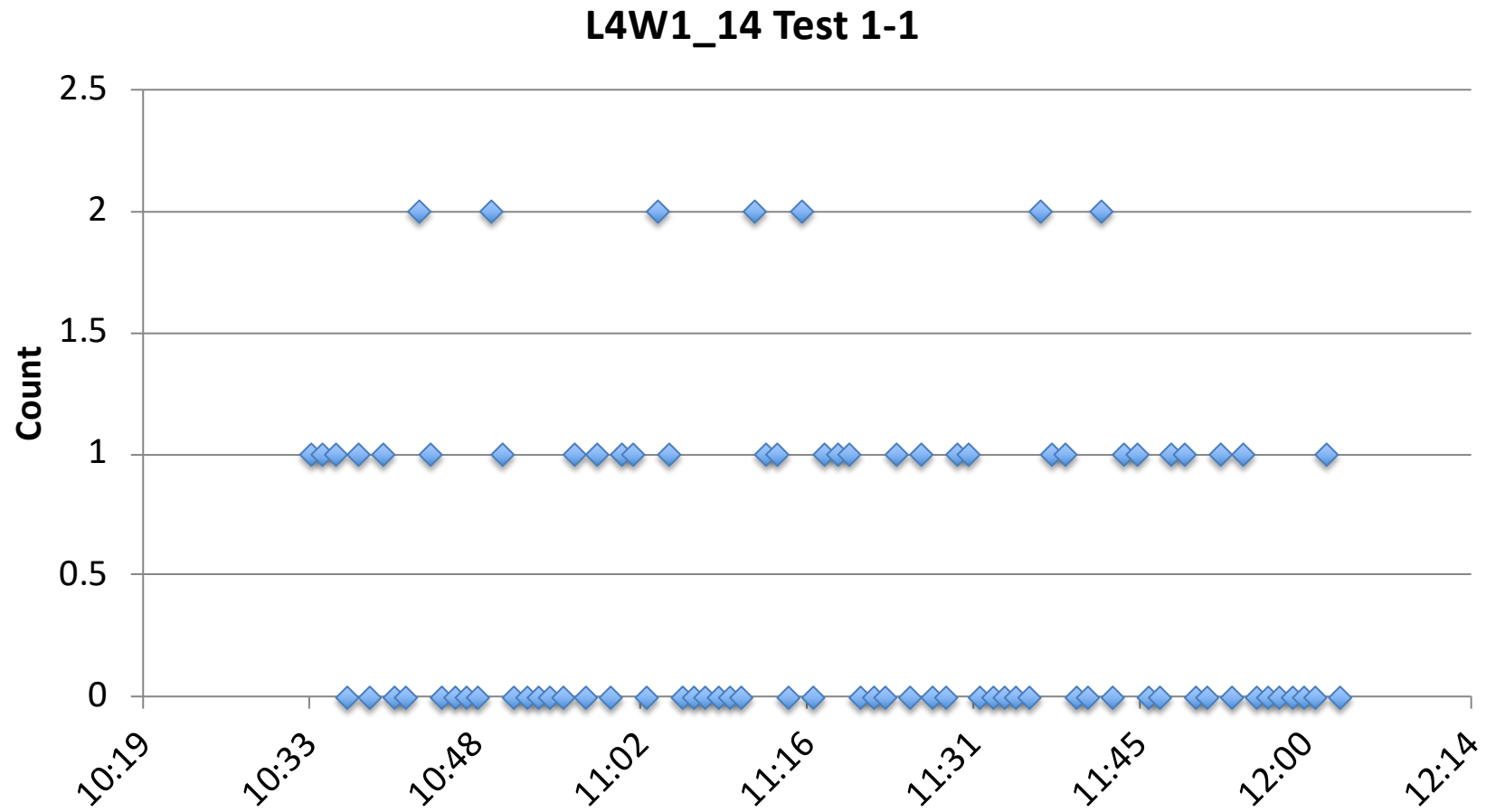
Statistics

- 150+ experiments w/ X-ray detection performed to date
 - ~20 on bare Au film
- Thermal detection with attached thermocouple
 - ~ 30 measurements so far

Conditions Tested So Far

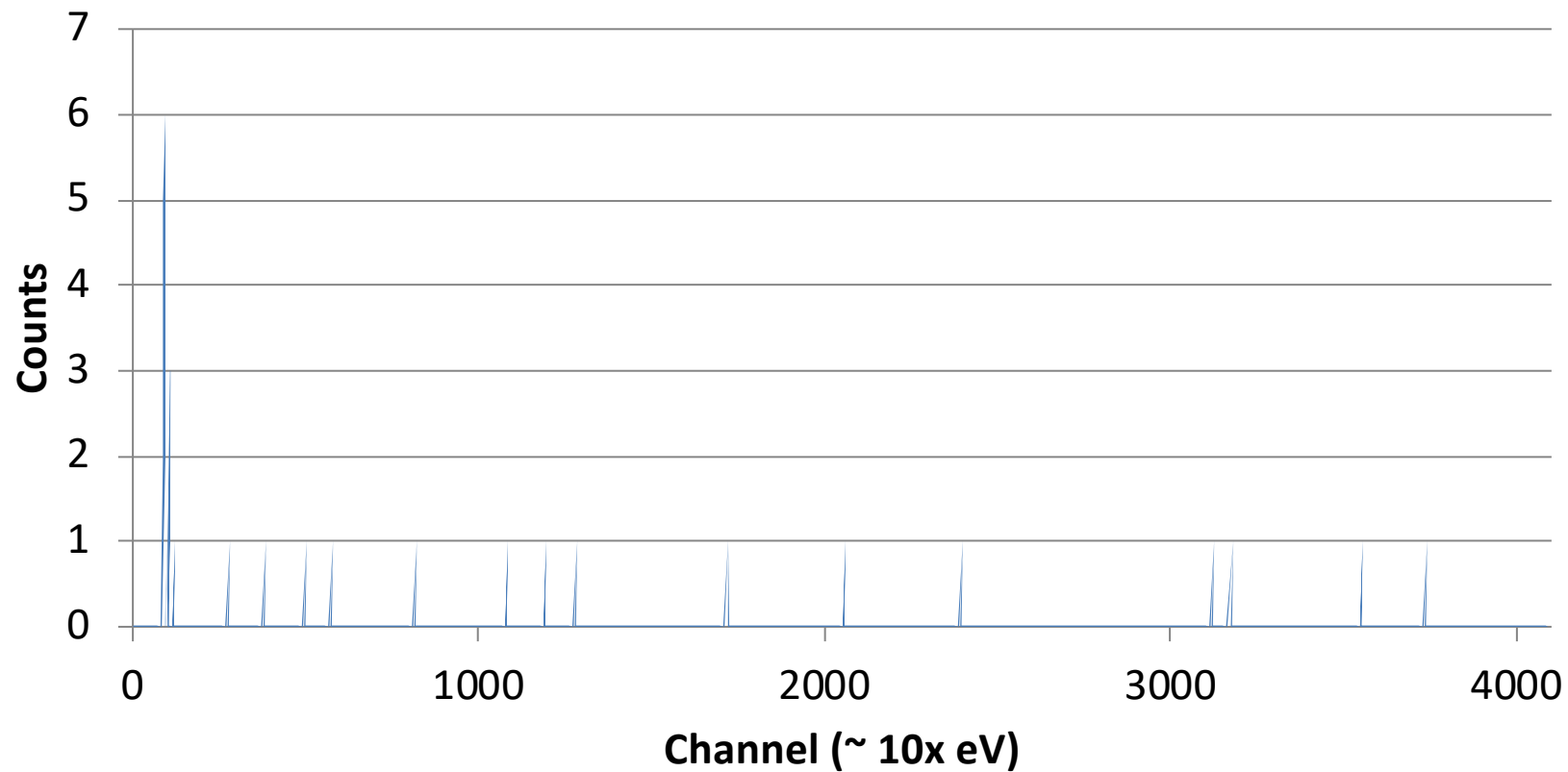
- Voltage / current
 - Bulk (~50) of measurements performed to try to maximize D sorption properties of film
 - For these, voltage mostly restricted to +/- 1.5 V vs. Ag/AgCl
 - Potential range includes D₂ evolution at ~ -1 V
 - Higher currents up to ~ 1 A/cm² tested more recently
- Codeposition from PdCl₂
 - Resulting geometries: From highly periodic, faceted to highly amorphous
- Impurities
 - Au, Fe, Co, Ni, Pd nanocubes, Silica beads, etc (ongoing)

Typical X-Ray Results



Typical Integrated Spectrum

L4W1_14 Test 1-1



Sorption Measurements

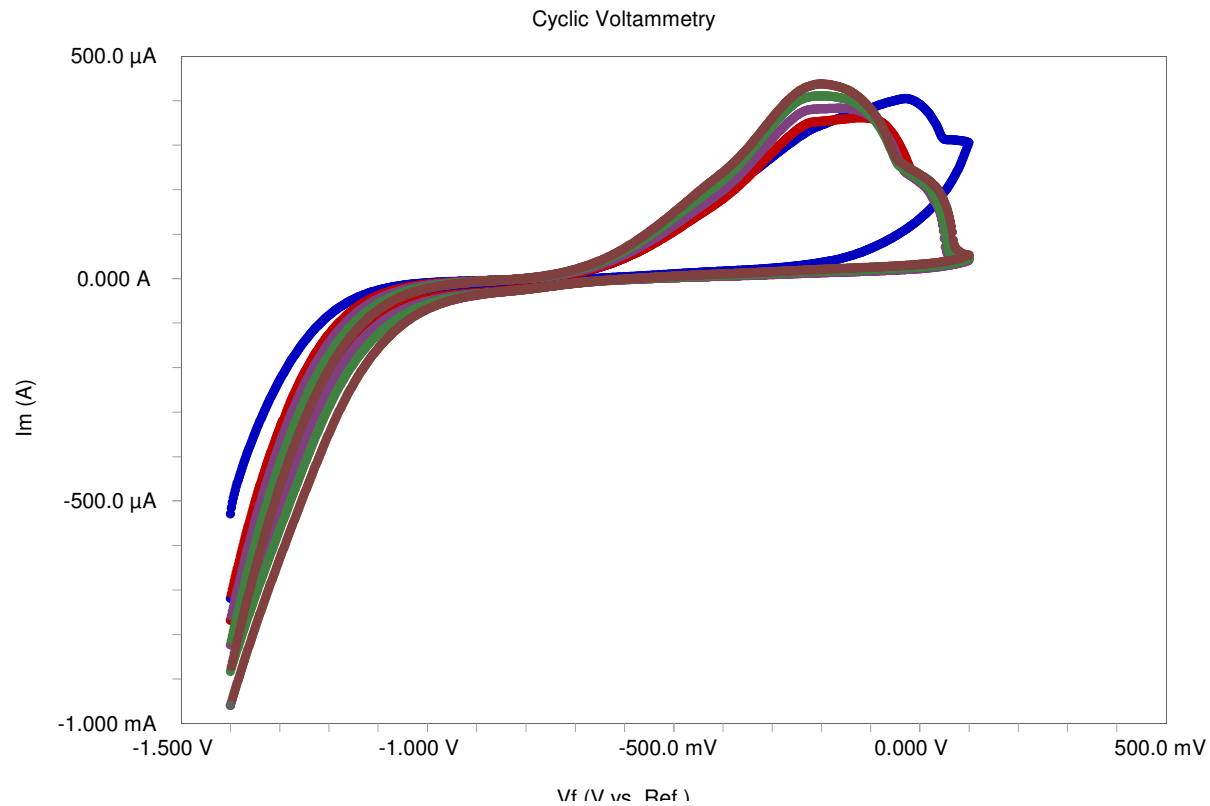
Why our system might be problematic

- Surface-bound film; how can it undergo alpha \rightarrow beta phase transition w/ 10% volume expansion?
 - “...stress-dependent reduction of hydrogen solubility at a given hydrogen pressure.” Wagner, S.; Pundt, A. *Acta Materialia* **2010**, *58*, 1387–1394.

Why it might not

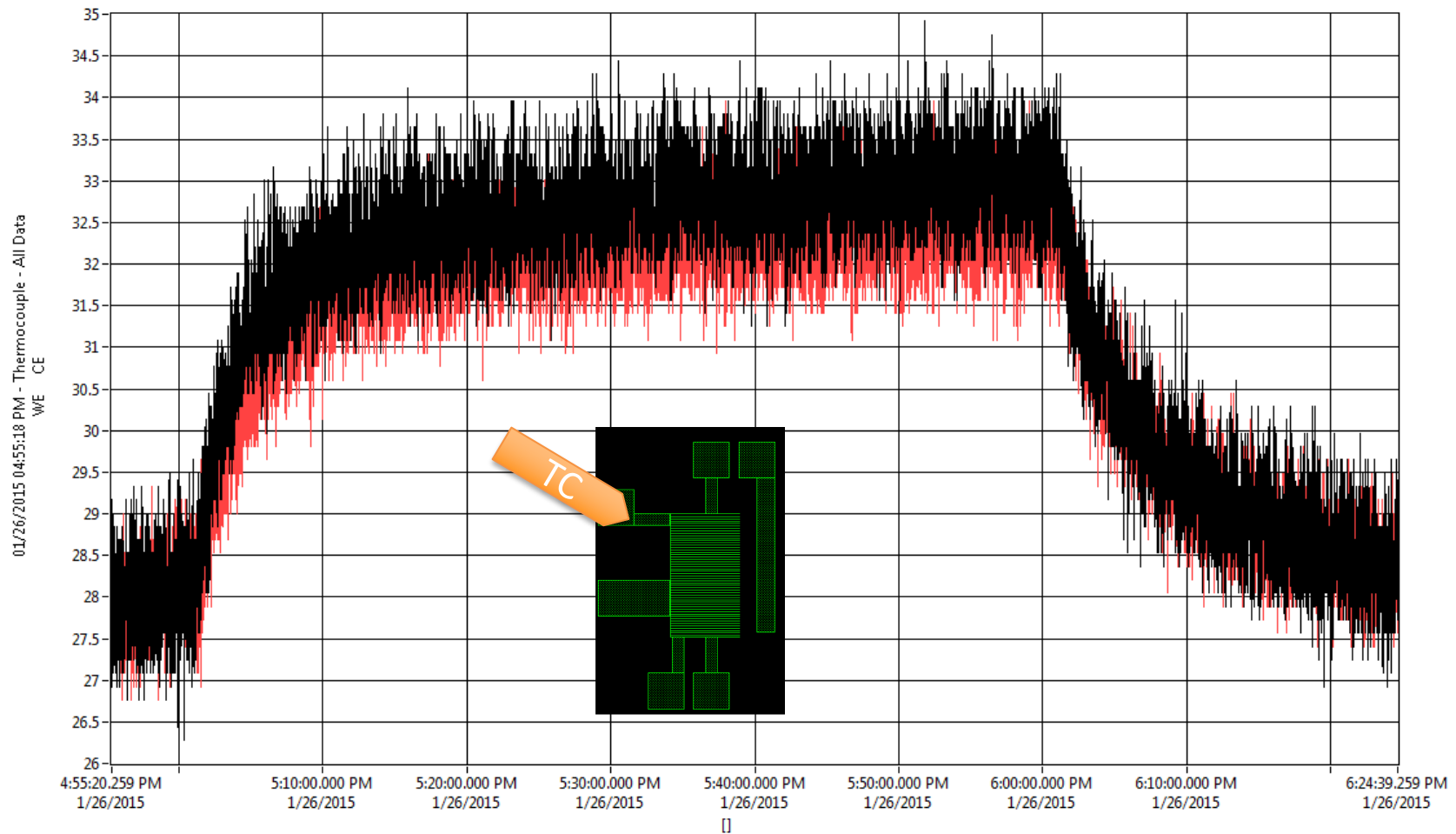
- Lots of work by Czerwiński et. al. on Pd electrodeposited onto Au
 - 0.7 + H/Pd and D/Pd claimed
 - Example: Czerwiński, A.; Kiersztyn, I.; Grdeń, M. *J Electroanal Chem* **2000**, *492*, 128–136.

“Best” Sorption Measurement



- L3W4_8, Serpentine pattern
- Integrated anodic stripping current = 12 mC, 76 % of theoretical max for pattern
- Pad patterns only get ~ 30-40%
- Finger patterns: non-diffusion limited, difficult to assess

“Typical” Thermal response for high current density tests



L4W2_6 Test 3-2, 20 mA