Stony Brook University

BROOKHAVEN NATIONAL LABORATORY

# Diamond X-Ray Diagnostics
# Building a Better Beam

Using the tools of materials science to improve user facilities from electron sources to beam diagnostics

John Smedley

Brookhaven National Laboratory

# Technologies for NSLS-II and LCLS-II Maybe useful for MaRIE?

- Diamond based x-ray beam diagnostics
  - Theory
  - Flux, position & timing
  - X-ray absorption Spectroscopy
  - Beam imaging!
- Electron Sources
  - Needs for high brighness & role of roughness
  - Source of roughness in alkali antimonides
  - Possible solutions

EPiC

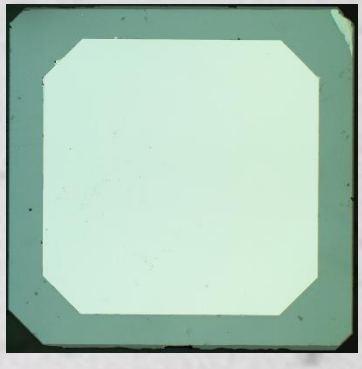
# Diamond as a X-ray sensor

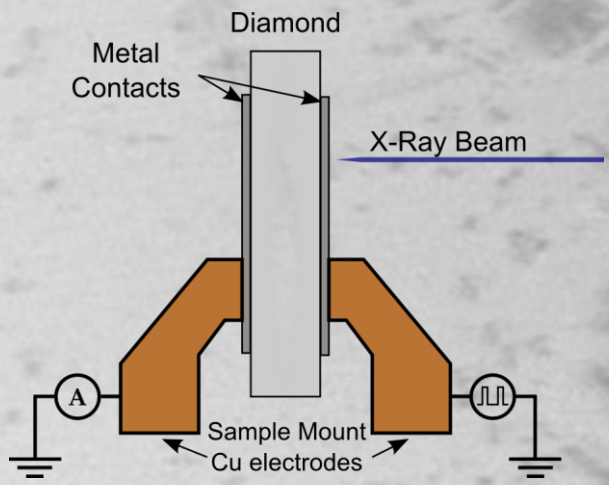
**Diamond Advantages:**

- Low X-ray Absorption
- High Thermal Conductivity
- Mechanical Strength
- Radiation Hardness
- Indirect bandgap

**Sample Information:**

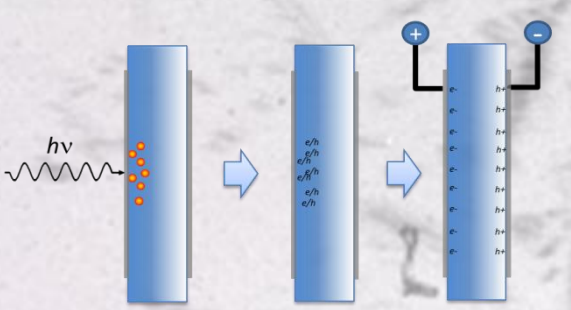
- Electrical grade CVD single crystal diamond
- (100) surface orientation
- ~1 ppb nitrogen impurity
- Typical size: 4mm x 4mm x 50µm





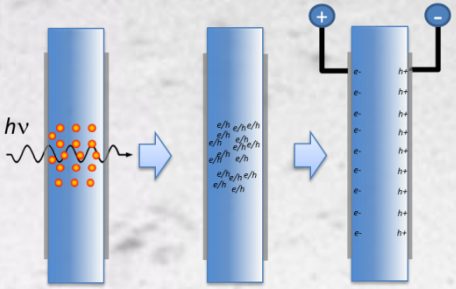

**X-ray generated charge carriers**

Low energy x-ray




Electron hole pairs created near incident electrode: must move entire thickness of the diamond

High energy x-ray




Electron hole pairs created throughout the thickness creating a column of electron-hole pairs

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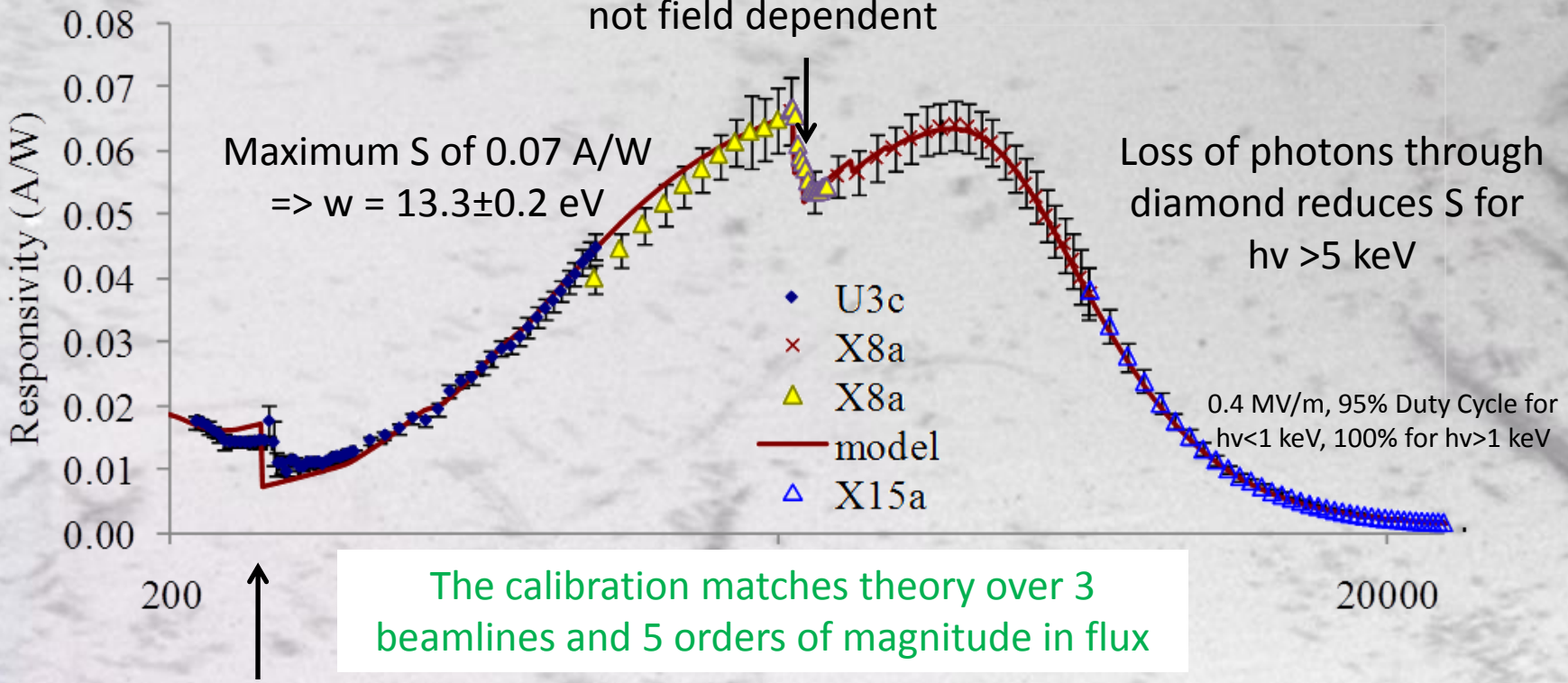
# Responsivity vs Photon Energy

$$S = \frac{1}{w} e^{-t_{metal}/\lambda_{metal}} \left(1 - e^{-t_{dia}/\lambda_{dia}}\right) CE[v, F]$$

Platinum M edge feature due to loss of photons absorbed by incident contact not field dependent




Maximum S of 0.07 A/W
=> w = 13.3±0.2 eV

Loss of photons through diamond reduces S for hv >5 keV

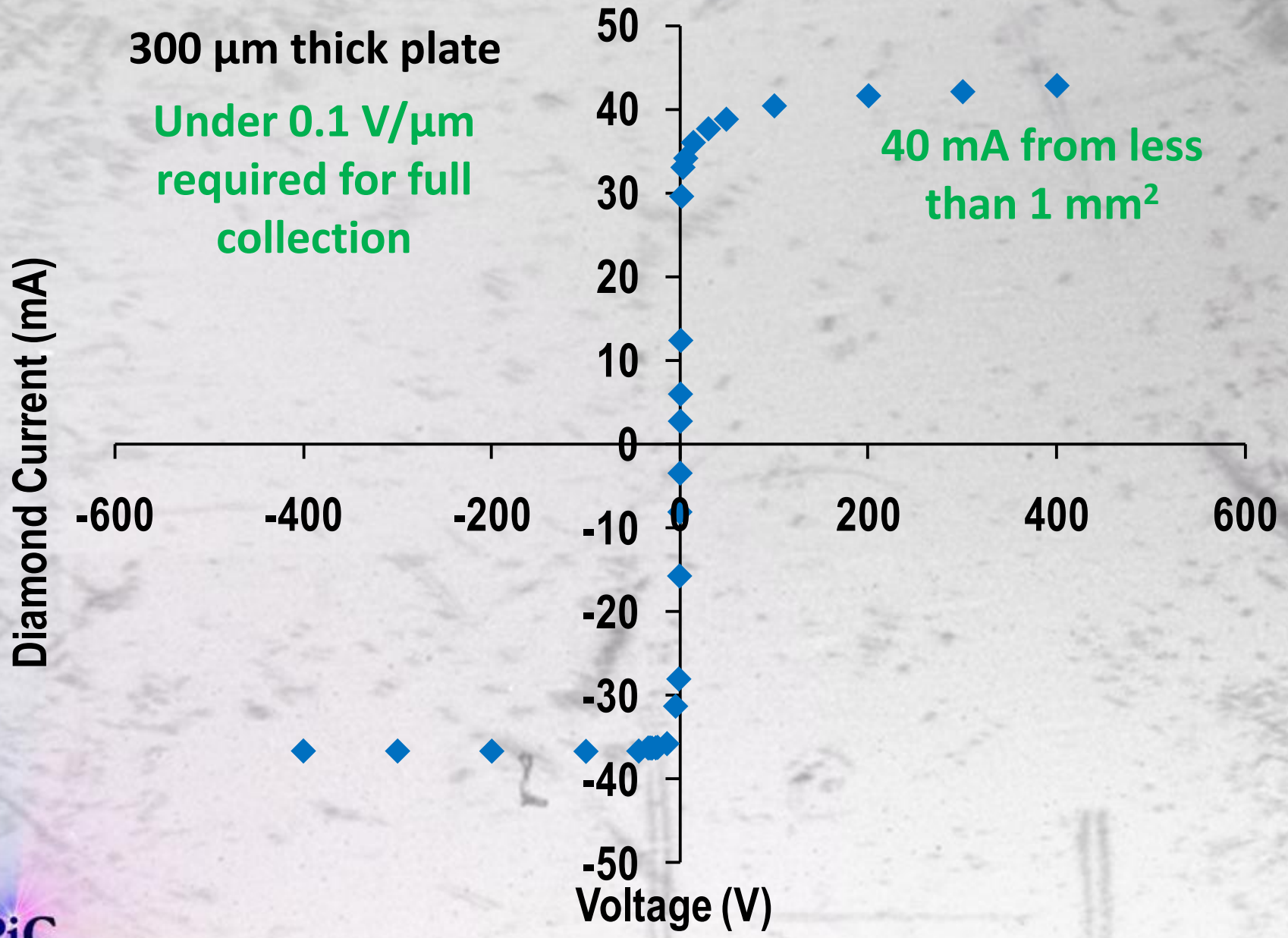
0.4 MV/m, 95% Duty Cycle for hv<1 keV, 100% for hv>1 keV

The calibration matches theory over 3 beamlines and 5 orders of magnitude in flux

C K edge feature is field dependent, caused by incomplete carrier collection for carriers produced near incident electrode – electrons diffuse into incident contact and are lost

J. Keister and J. Smedley, NIM A **606**, (2009), 774

# Response vs Bias

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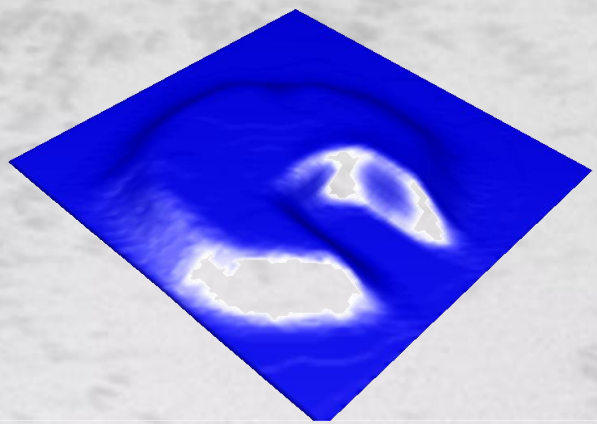
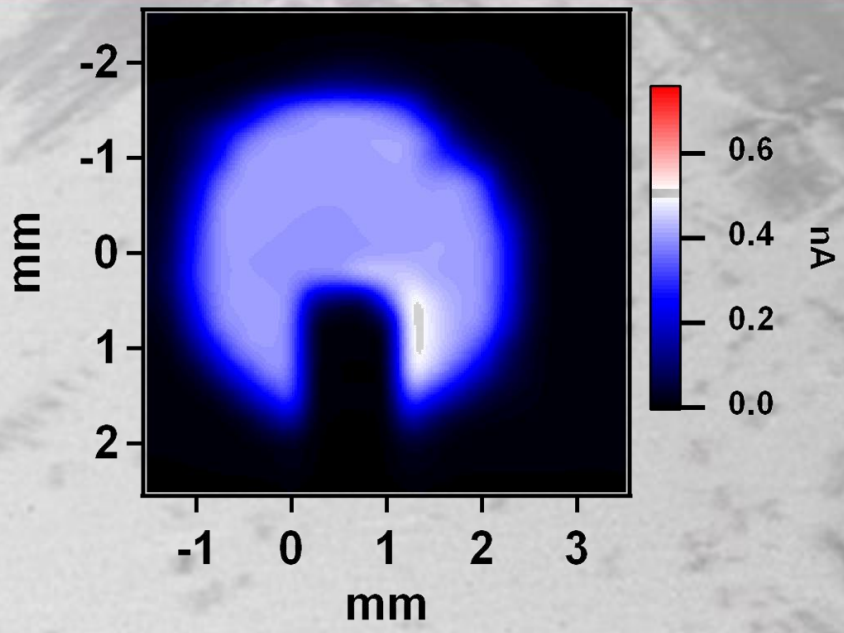
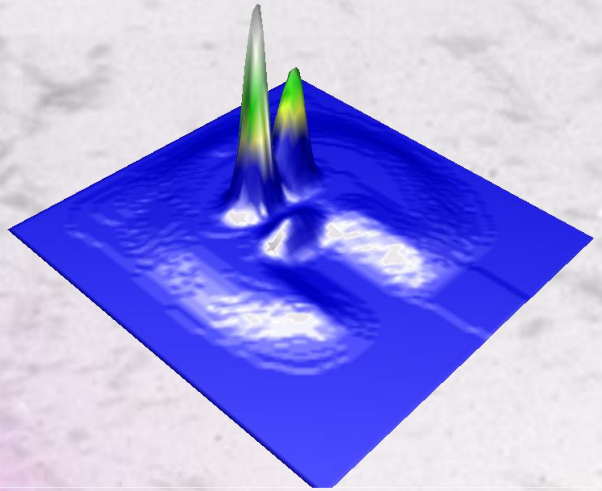
HID14 in X15
(80%, 100V, 1kHz, 19keV)

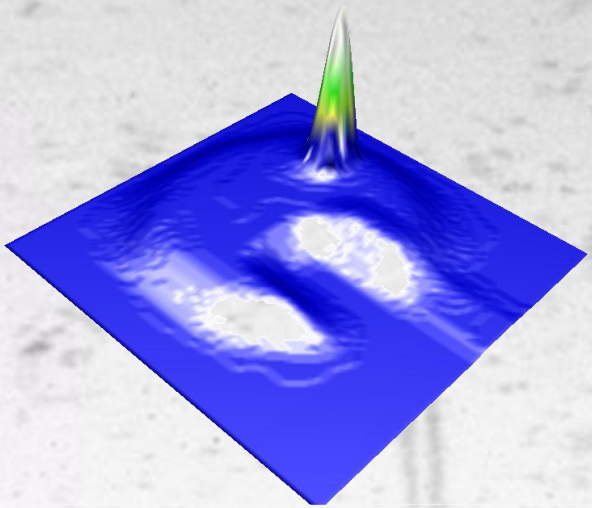





HID18 in X6B
(80% ,-100V, 1kHz, 19keV)



HID18 in X6B
(80% ,+100V, 1kHz, 19keV)



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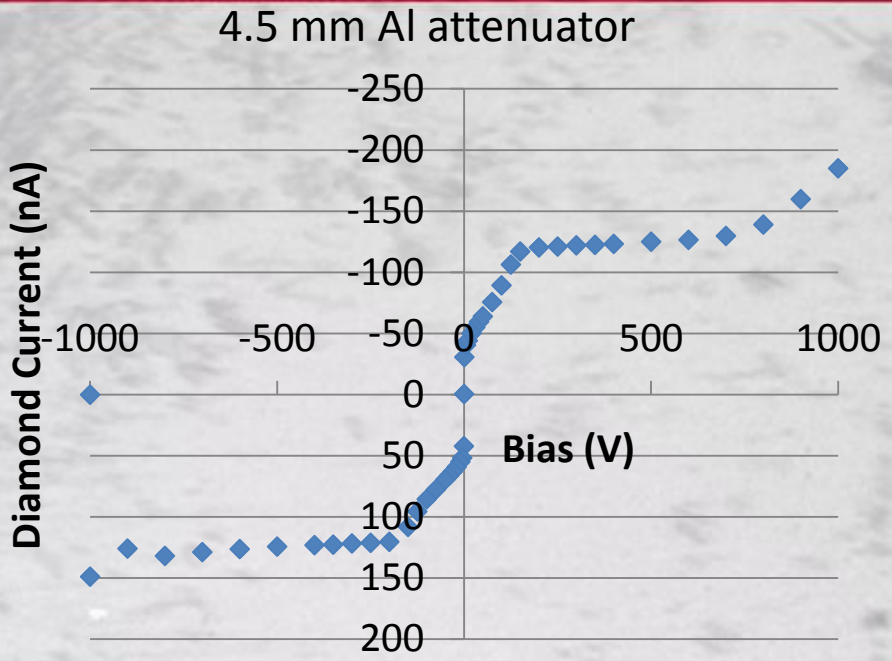
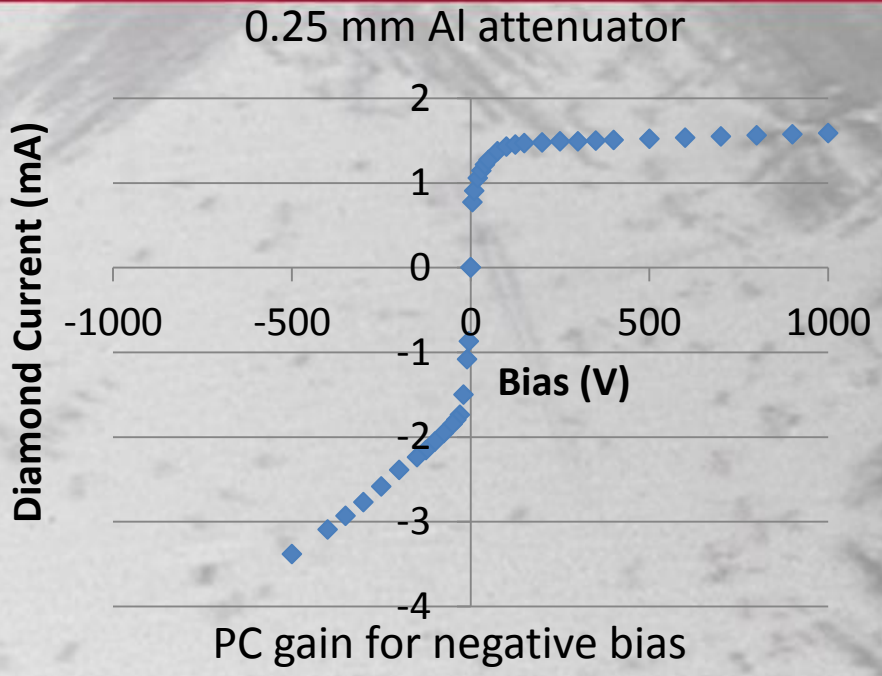
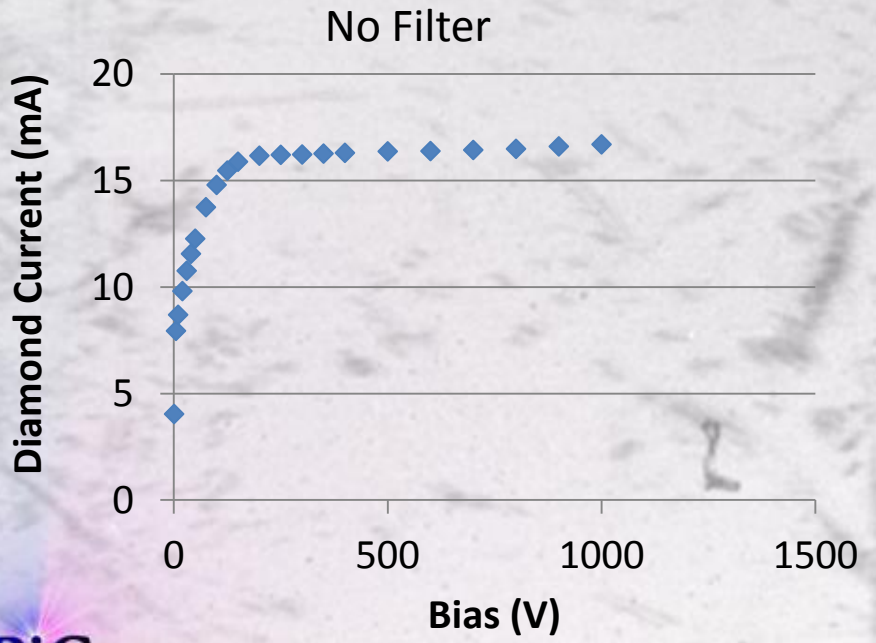
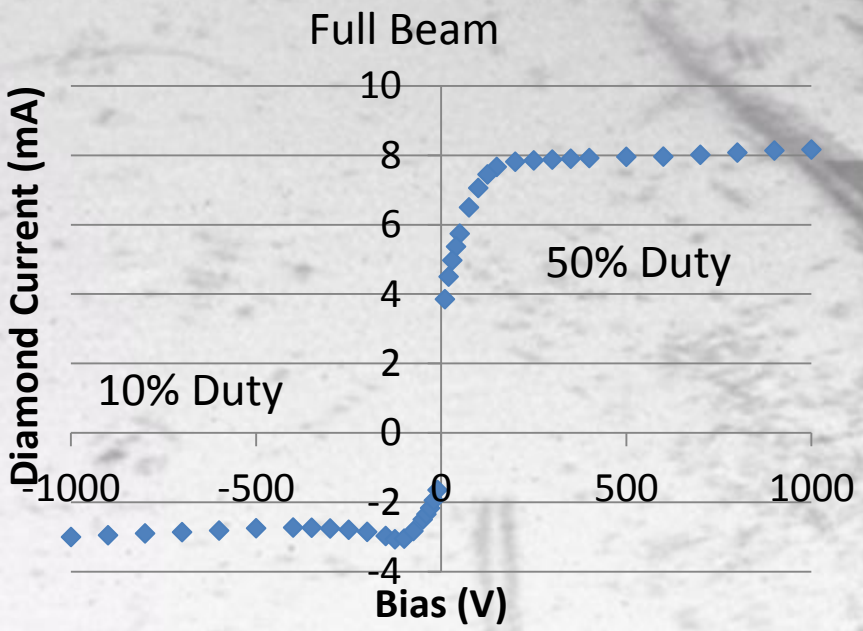



PC gain for negative bias




PC gain removed by operating at low Duty Cycle

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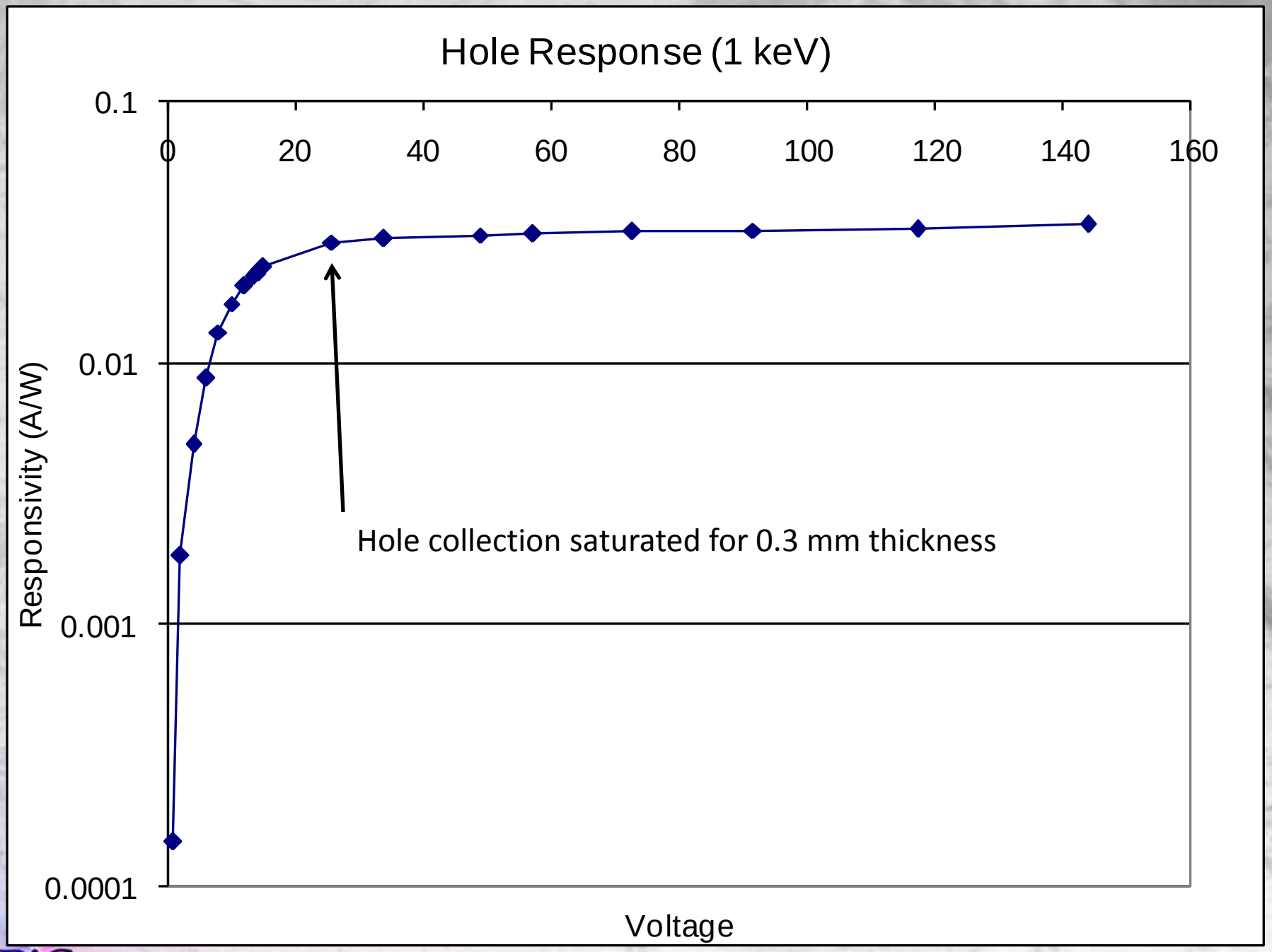

Hole Response (1 keV)
0.1
0.01
0.001
0.0001
0
20
40
60
80
100
120
140
160
Responsivity (A/W)
Voltage
Hole collection saturated for 0.3 mm thickness

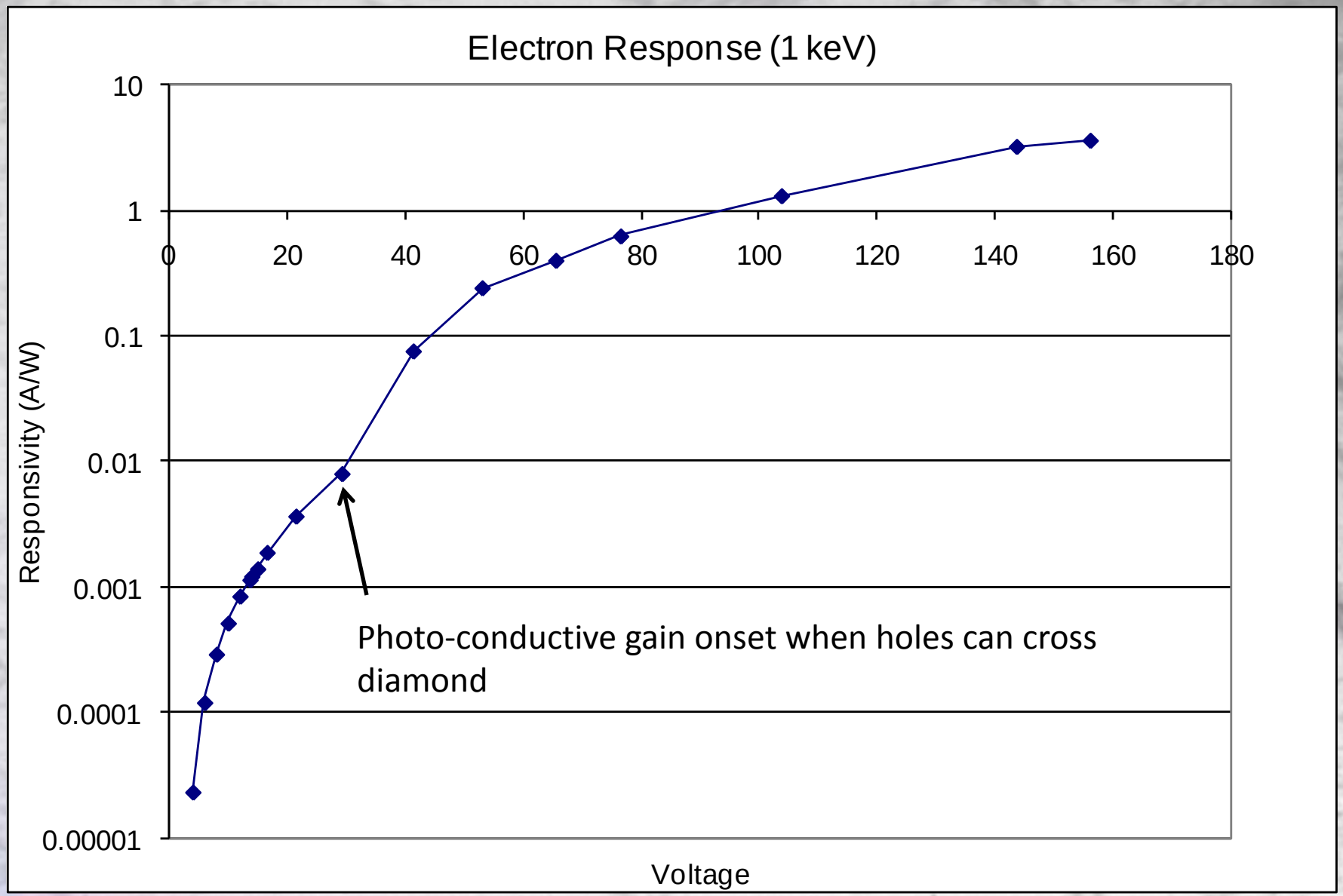
# What about electrons?

- Electron response depends strongly on type of electrical contacts
- For blocking contacts (Pt on O-terminated diamond), electrons exhibit significantly more trapping than holes
  - Lower duty cycle of pulsed bias to avoid signal loss
- For injecting contacts, photoconductive gain is observed
  - Trapped electrons act as effective “doping” of material
  - Holes are injected from opposite electrode

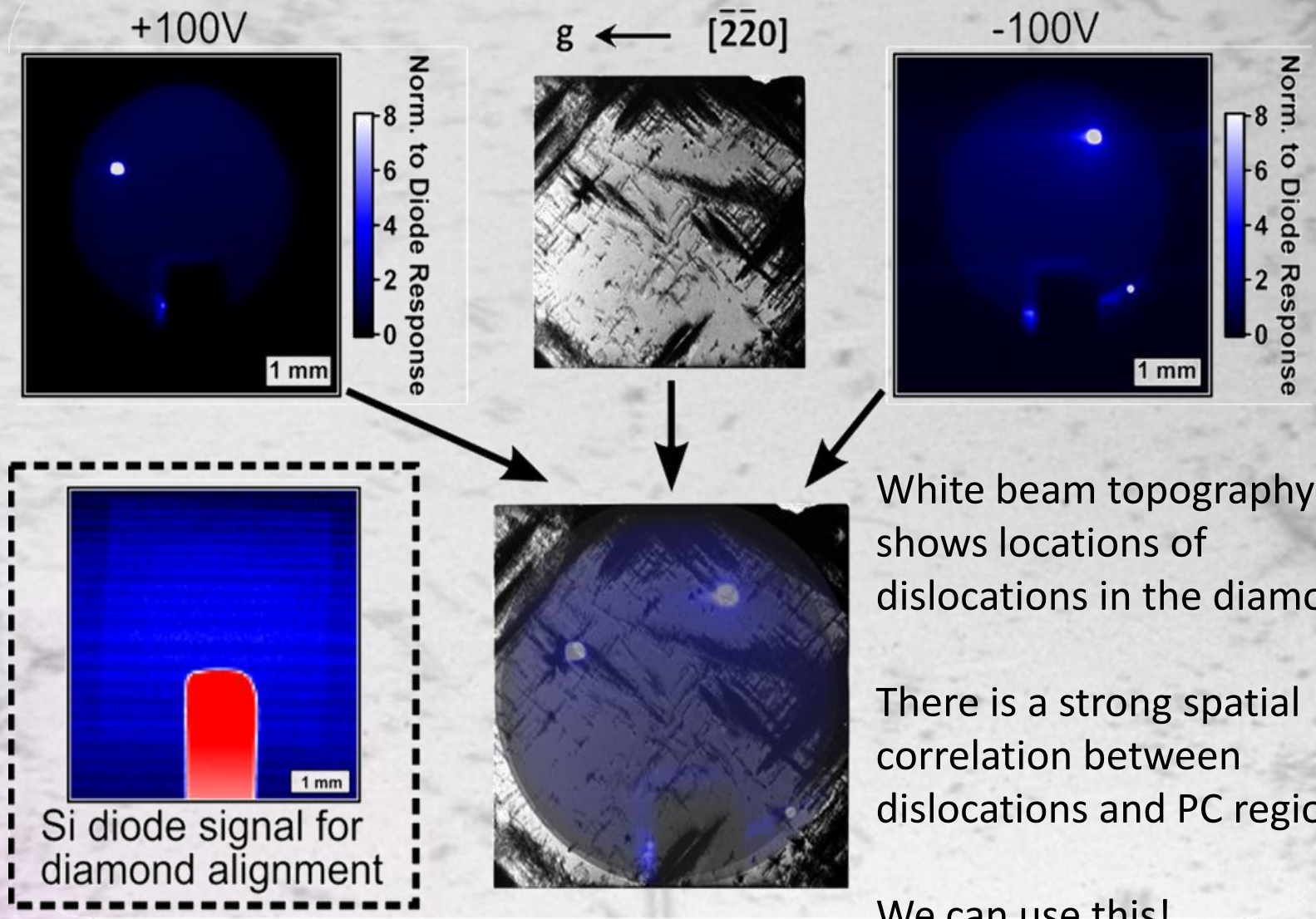
$$Gain = \frac{\tau_{holes}}{t_{holes}}$$

$\tau_{holes}$: Hole lifetime

$t_{holes}$: Hole transit time


Electron Response (1 keV)
10
1
0.1
0.01
0.001
0.0001
0.00001
Responsivity (A/W)
0
20
40
60
80
100
120
140
160
180
Voltage
Photo-conductive gain onset when holes can cross diamond

# Relation to Threading Dislocations




White beam topography shows locations of dislocations in the diamonds.

There is a strong spatial correlation between dislocations and PC regions

We can use this!

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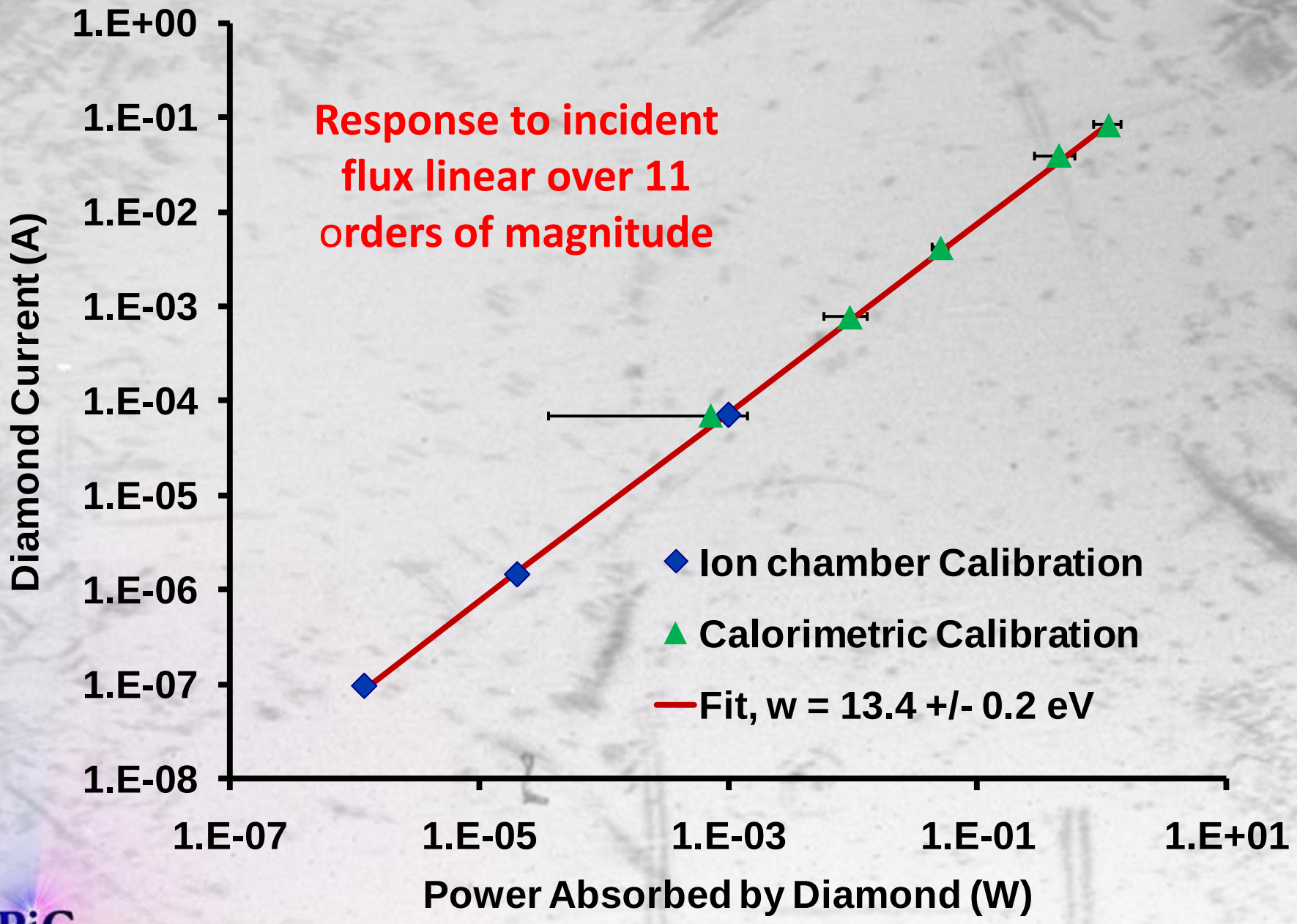
# High Flux Response

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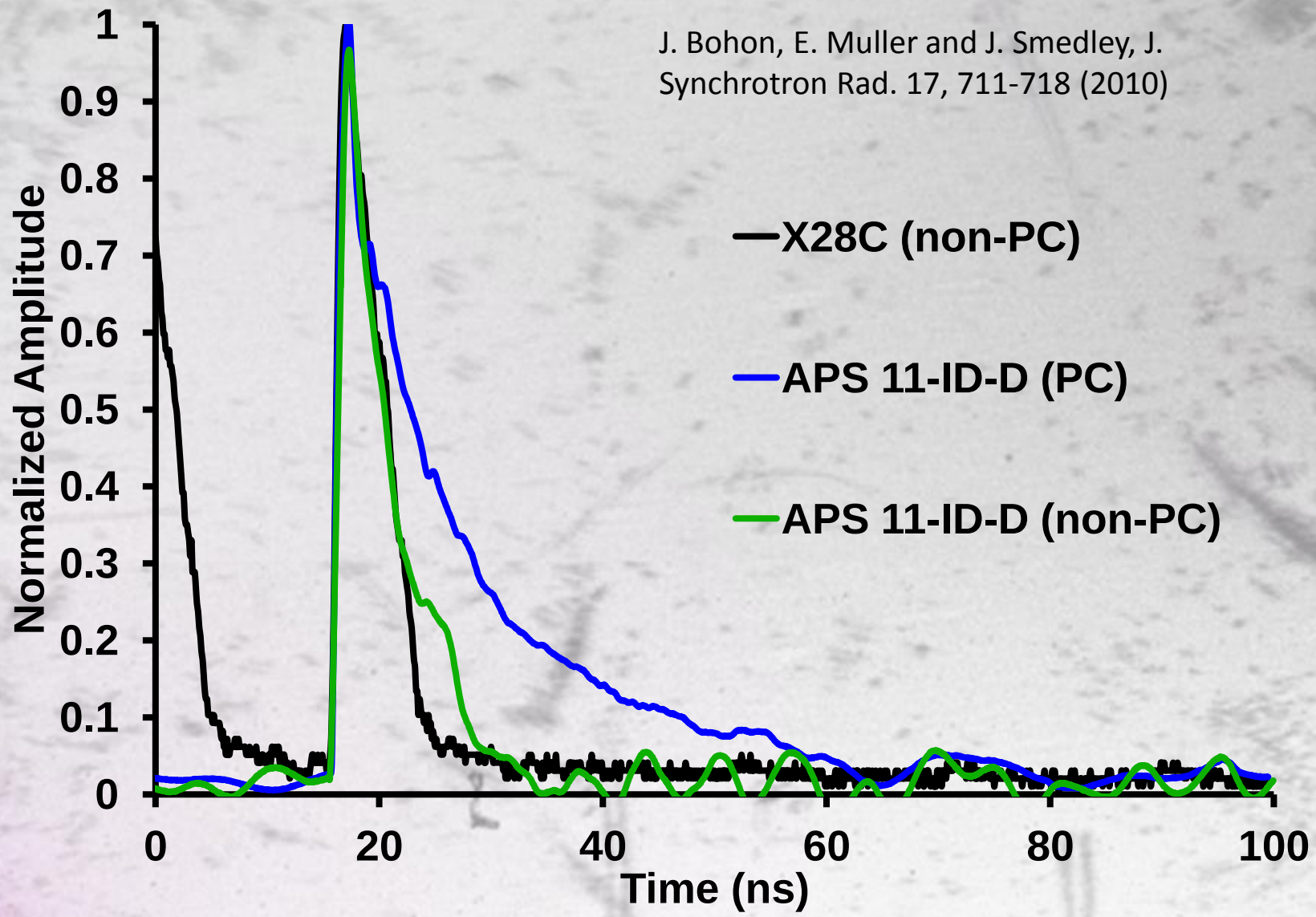
# Temporal Response, Hard X-rays




J. Bohon, E. Muller and J. Smedley, J. Synchrotron Rad. 17, 711-718 (2010)

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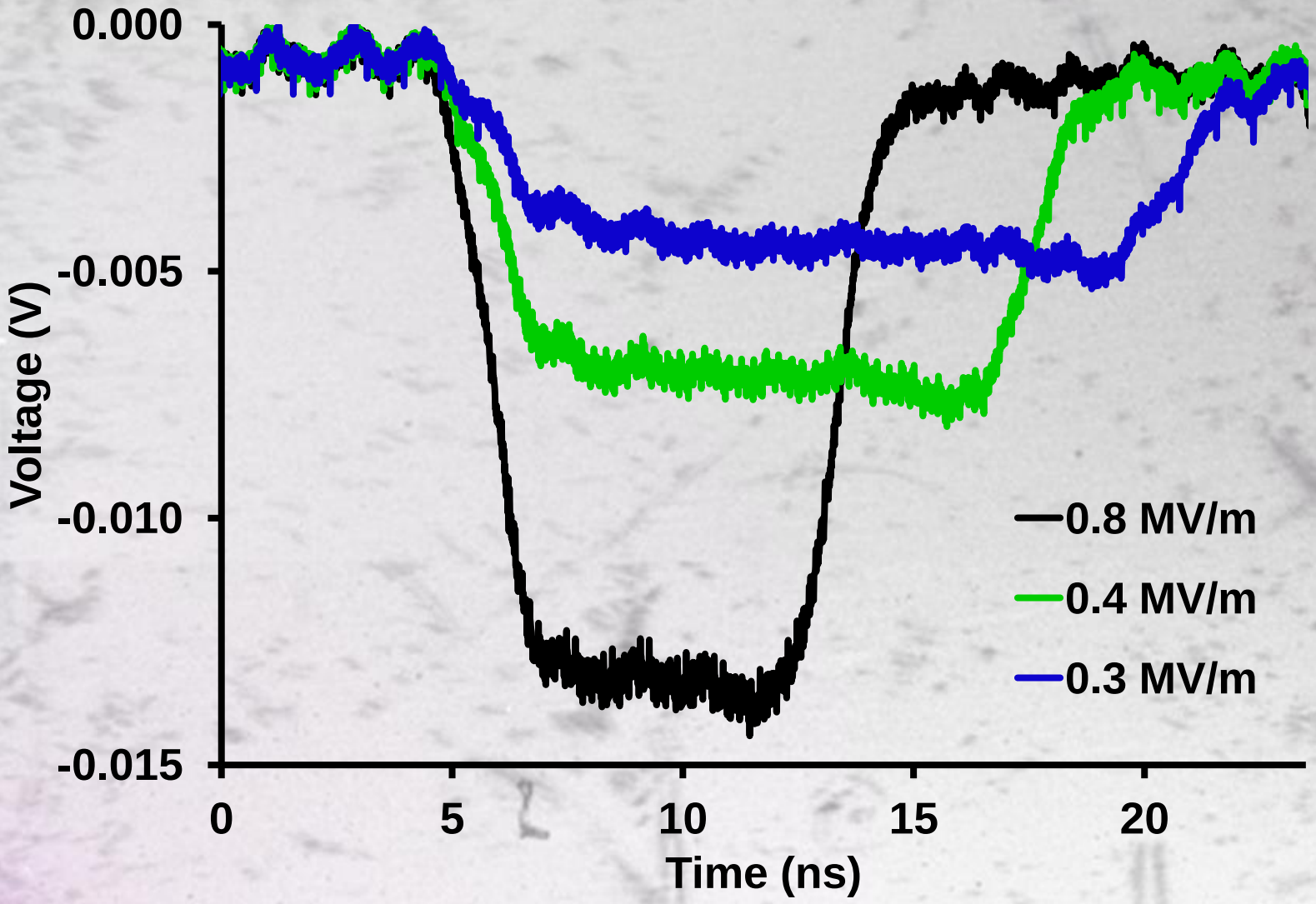
# Temporal Response, Soft X-rays

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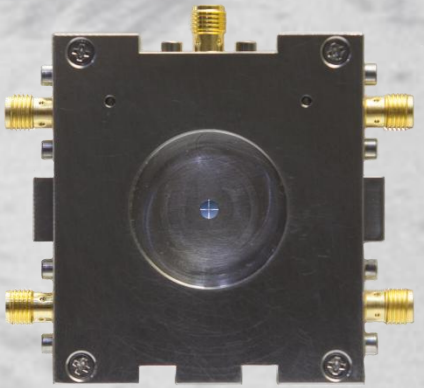
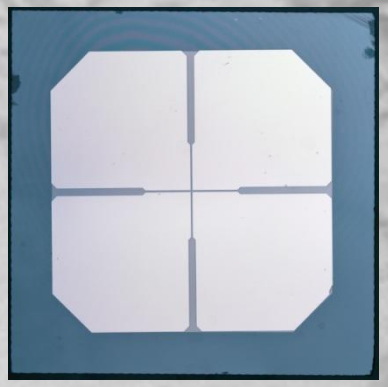
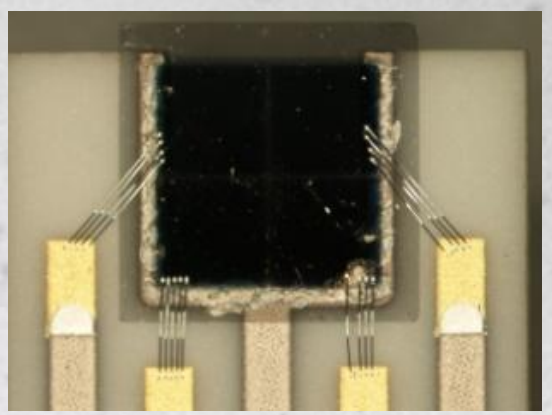
# Diamond Beam Position Monitors

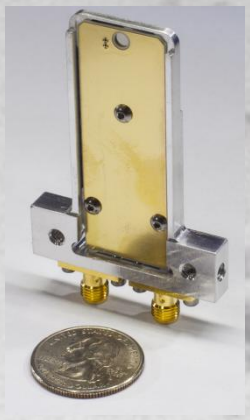
- Circuit Board Mounted
  - Pt metallization
  - wire-bonded electrodes
  - SMA/LEMO connectors





- Application specific –
  X-Ray fluorescence (X27)
  - Ag diamond metallization
  - Ceramic board
  - 1 cm wide (compact)
  - Ag traces





- White BPM (X25)
  - Mini-gap undulator
  - ~100W incident power
  - Large beam

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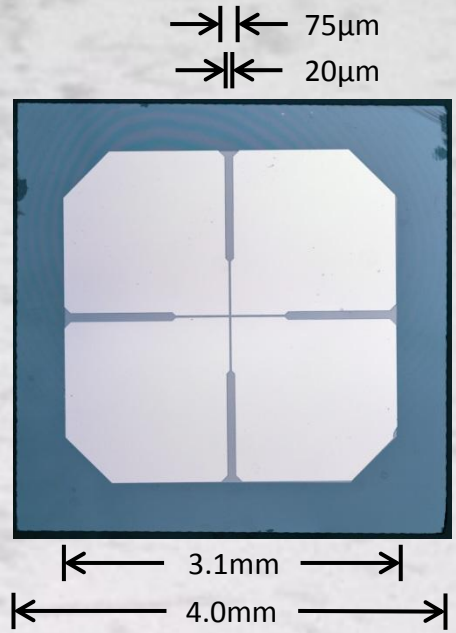
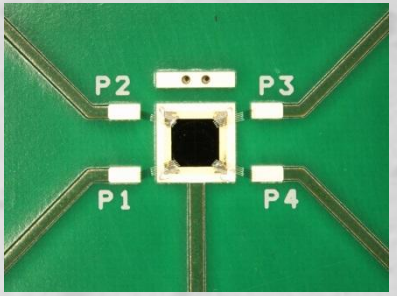
# Fabrication

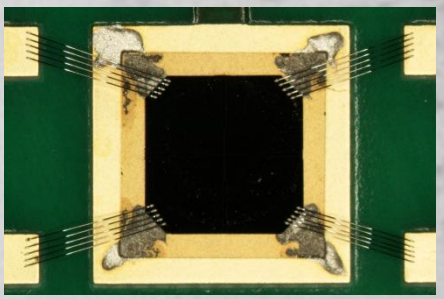
## Lithography @ CFN

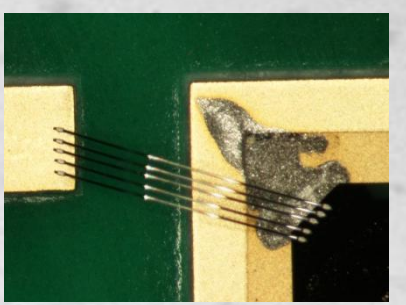



Electronic grade single crystal
(100) diamond
30-50 µm thick
20 µm street over a 1mm center region
Metalization: 25 nm Pt

## Wire Bonding - Instrumentation





25 µm aluminum wirebonds
5 bonds per pad
Conductive epoxy for backside/bias contacts

## Topography – NSLS/CHESS

White beam topography
Prior to slicing

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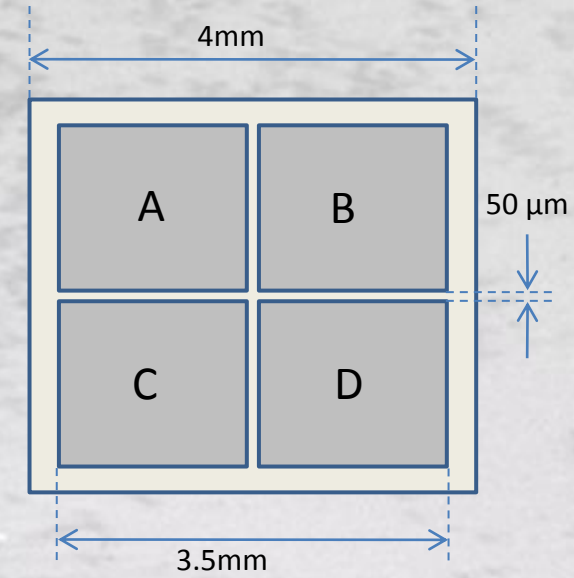
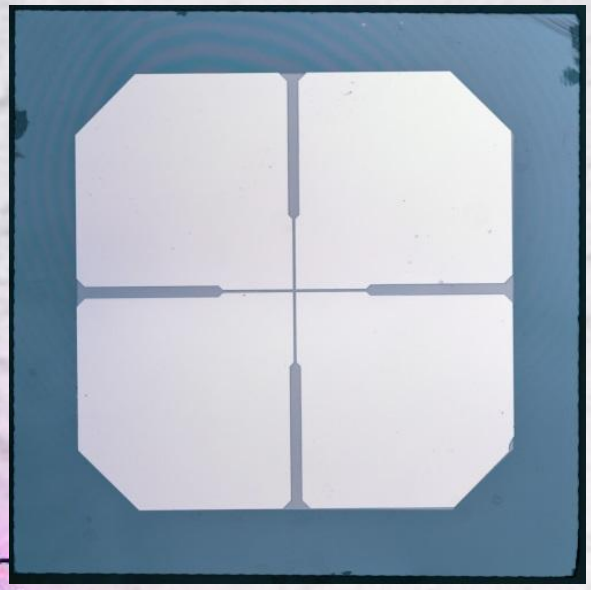
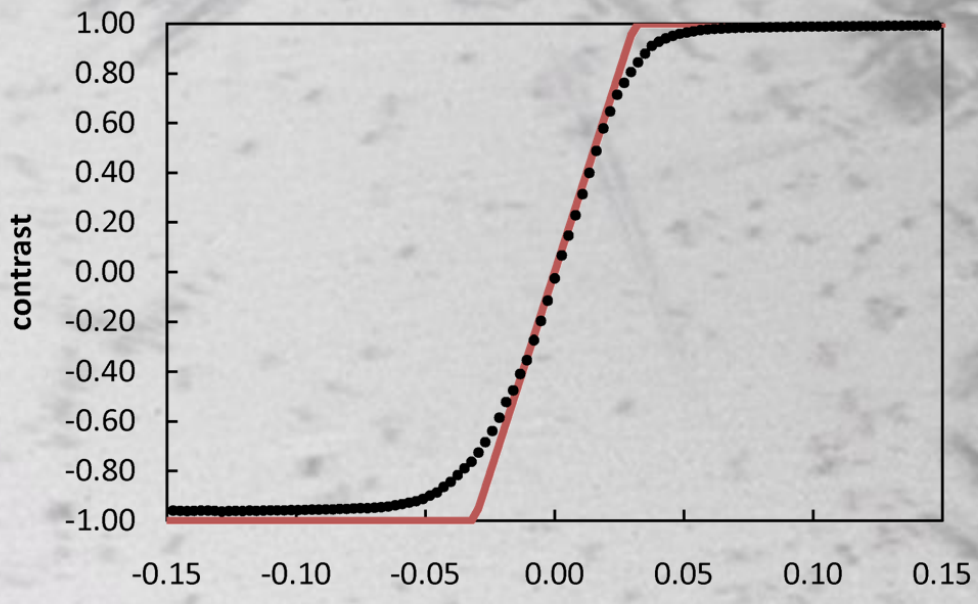
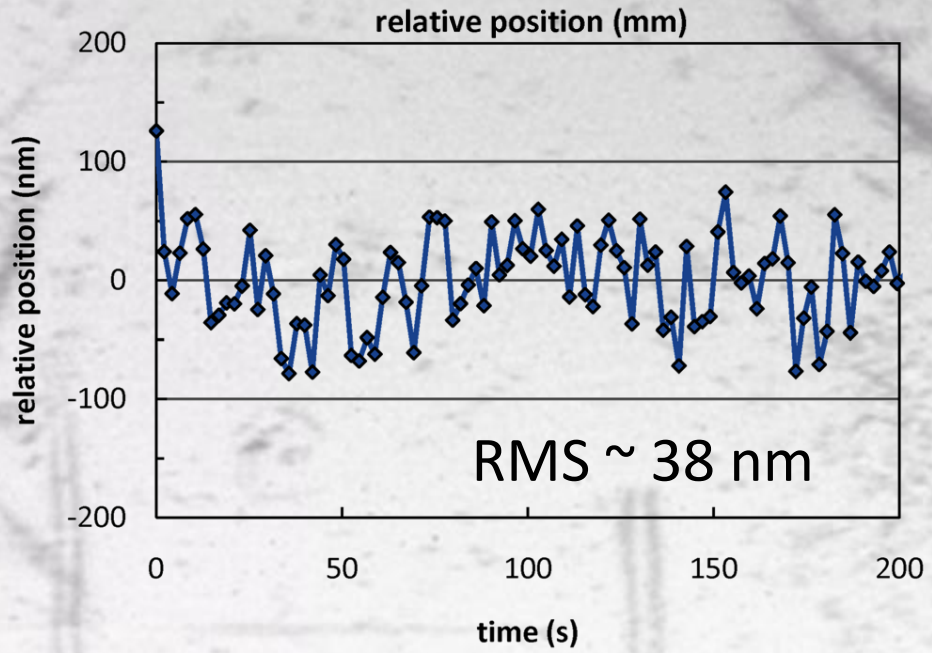
# Beam Position Monitors




$$X = G_x \frac{(I_B + I_D) - (I_A + I_C)}{I_A + I_B + I_C + I_D}$$



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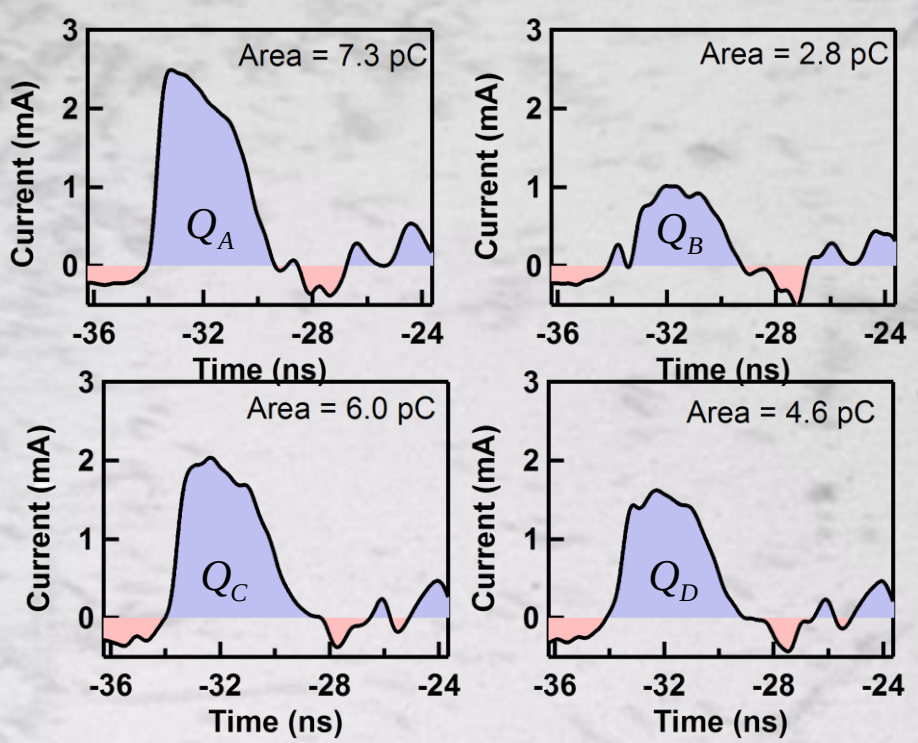
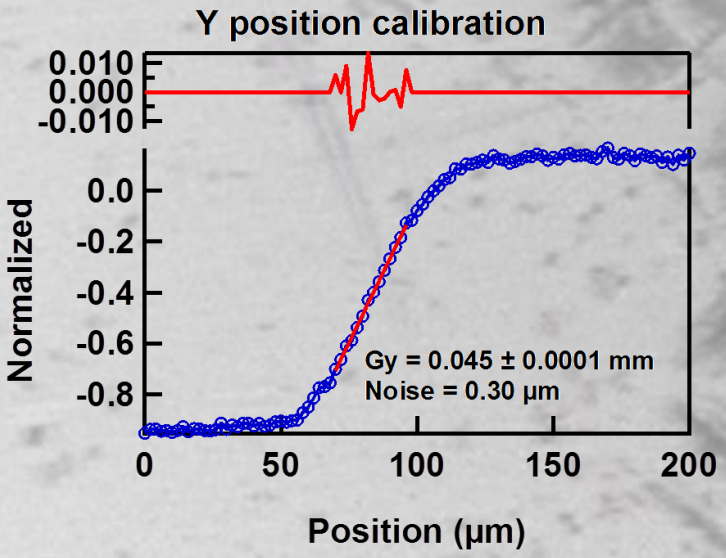
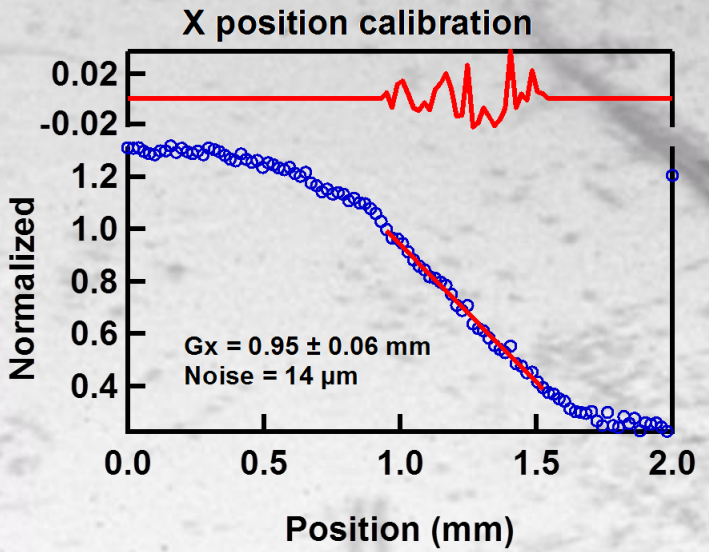
# Pulse Mode Beam Position




- Area under pulse (charge) was used to calculate position
- $G_X$ and $G_Y$ were calculated by using:

$$X = G_x \frac{(Q_B + Q_D) - (Q_A + Q_C)}{Q_A + Q_B + Q_C + Q_D} \qquad Y = G_Y \frac{(Q_A + Q_B) - (Q_C + Q_D)}{Q_A + Q_B + Q_C + Q_D}$$

- Position noise was calculated by taking the standard deviation of the residuals and multiplying by G
  - Includes all noise sources, including actual beam wander

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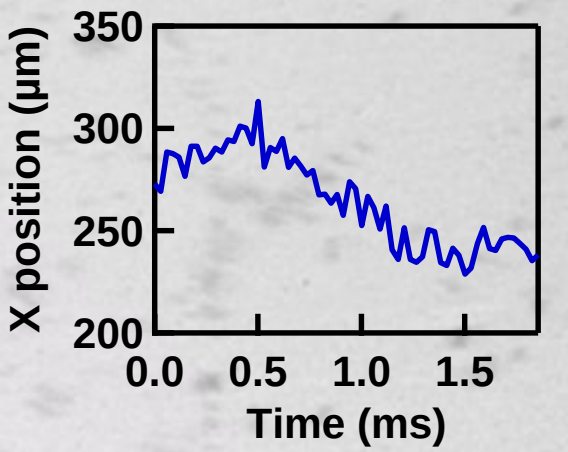
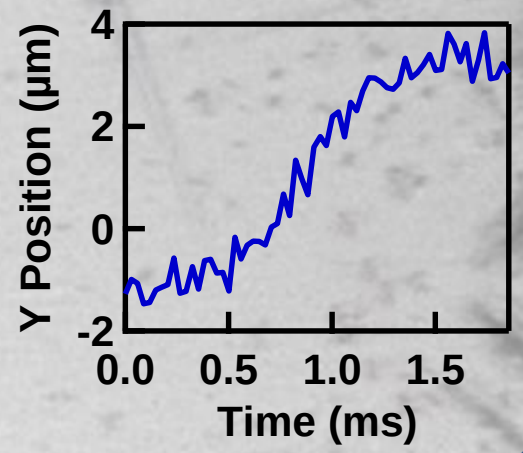
# Position Stability at 11-ID-D, APS

**Ring Structure at APS 11-ID-D**

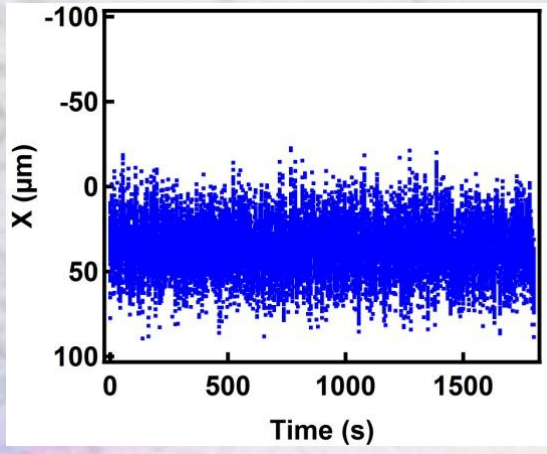
- 24 bunches spaced 153 ns apart
- Takes 3.68 µs to complete one orbit
- Beam size 15 µm x 0.8 mm
- Measured the position of the first bunch continuously
- +400V on quad (~2 MV/m)
- Recently acquired 324 mode

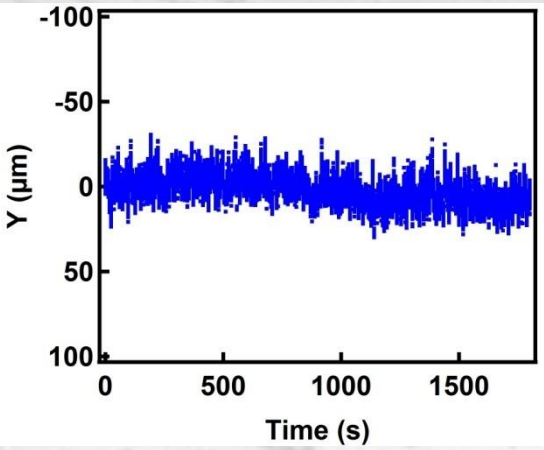
**Short term stability**

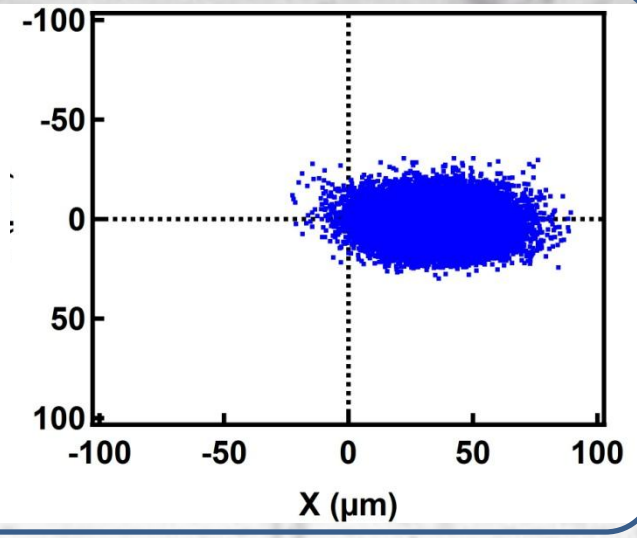



**Long term stability**

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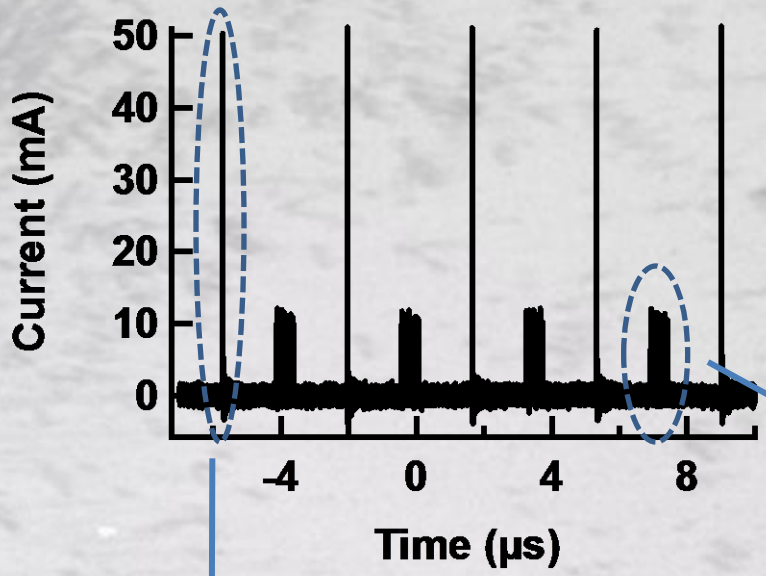
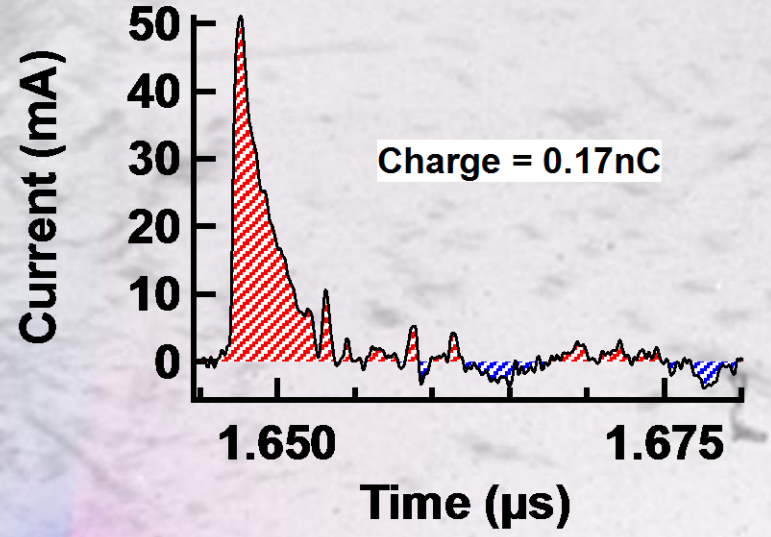
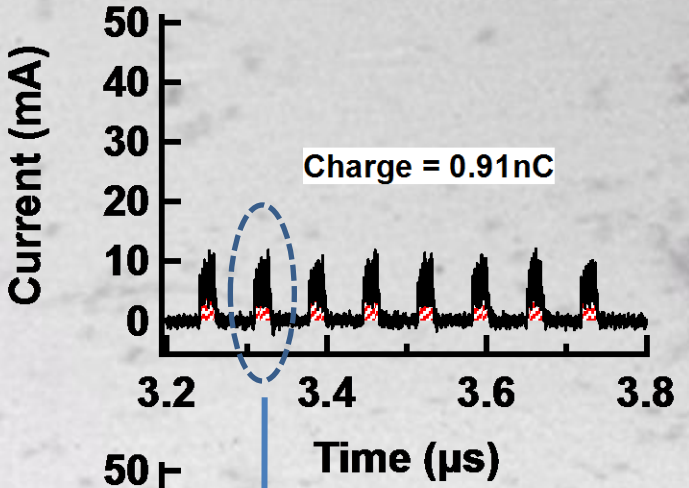
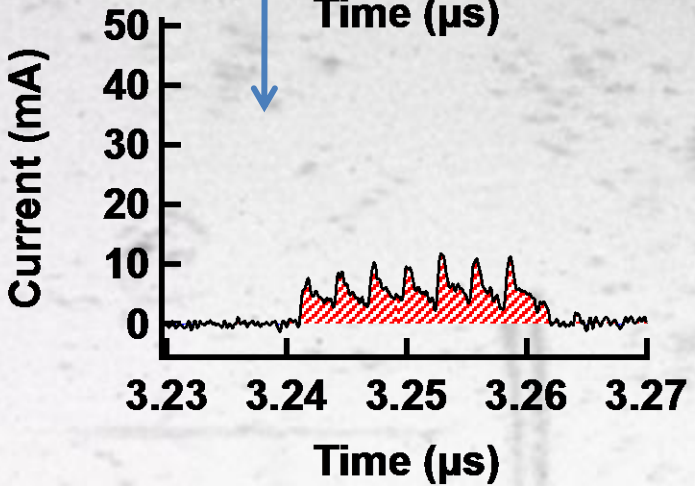
# Pulse Mode Beam Position 11-ID-D, APS




- Ring mode "hybrid fill, top up".
- 102mA total, 16mA in first bunch, 86mA in remaining pulse train.
- Separated by 1.594 μs
- **Ratio of ring currents matches very closely to measured charge ratio**
- ***Current Ratio: 86mA/16mA = 5.38***
- ***Measured Q Ratio: 0.91nC/0.17nC = 5.35***

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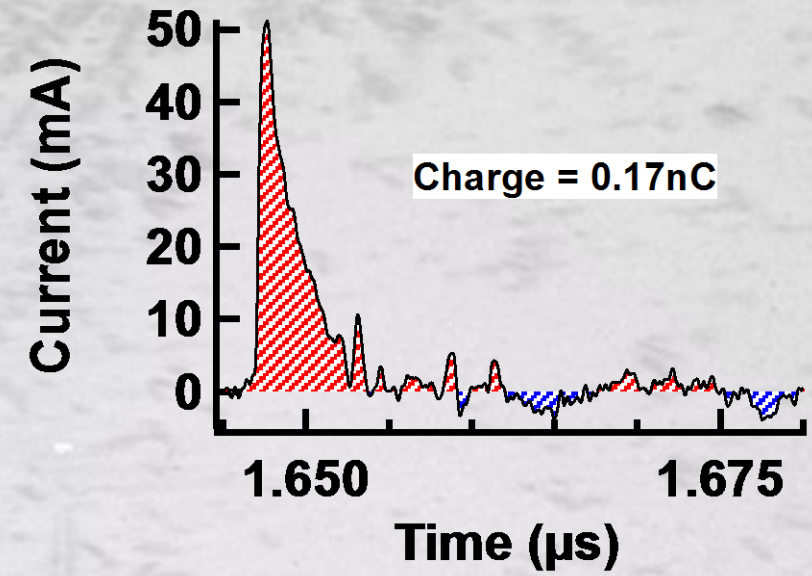
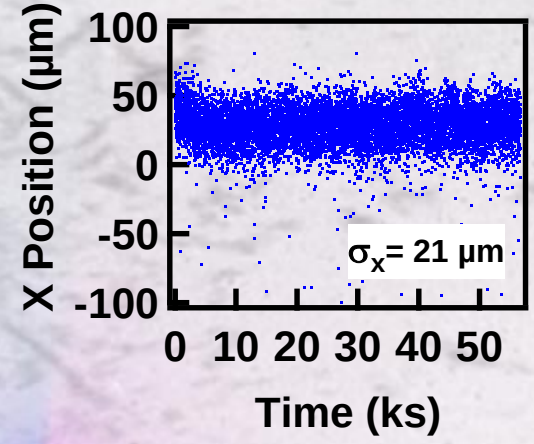
# Pulse Mode Beam Position 11-ID-D, APS

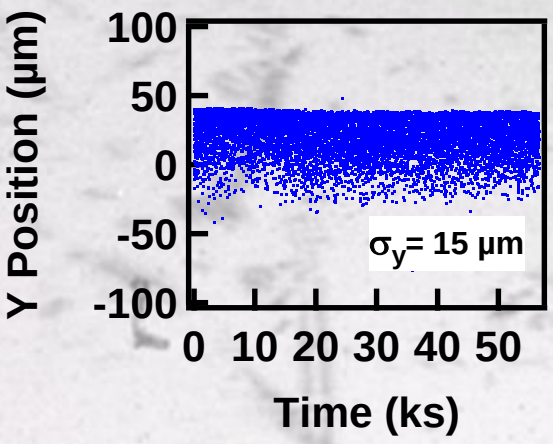


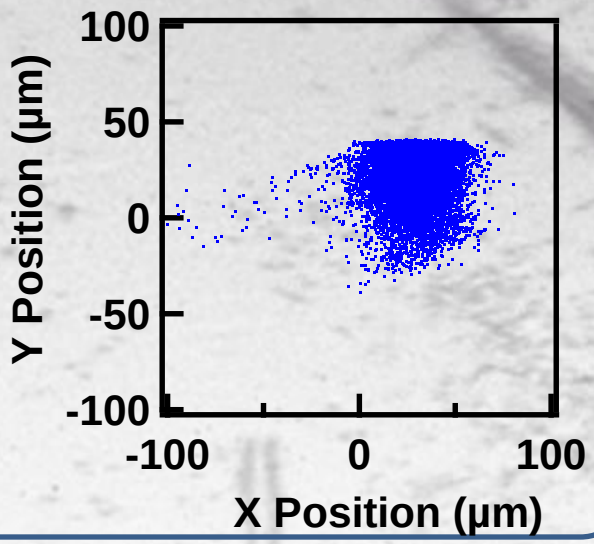

**Ring Structure at APS 11-ID-D**

- Ring mode "hybrid fill, top up".
- Tracked the singlet bunch position every 11 turns (40 μs) for 15 hrs
- Singlet bunch has a peak current density of 200 A/cm$^2$
- ***Traditional alignment feedback works on average current -> looking primarily at pulse train, not at singlet***

**Long term stability**

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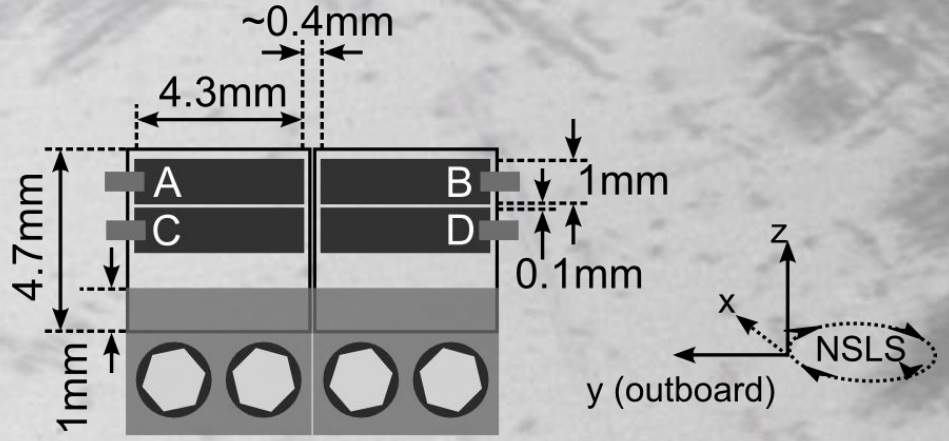
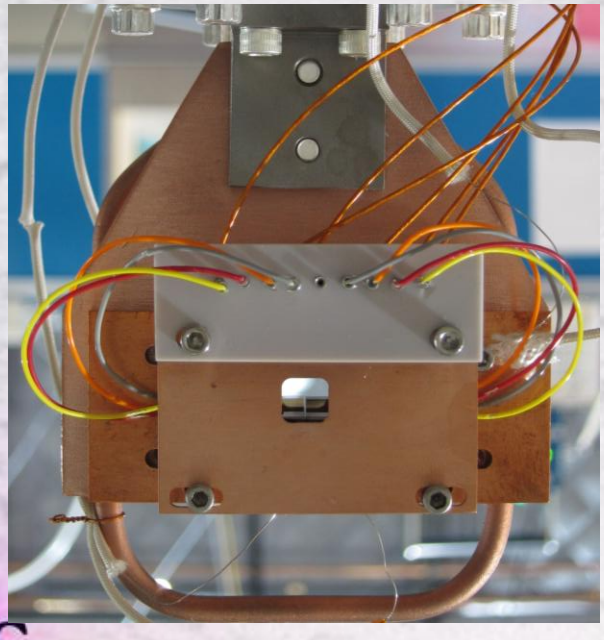
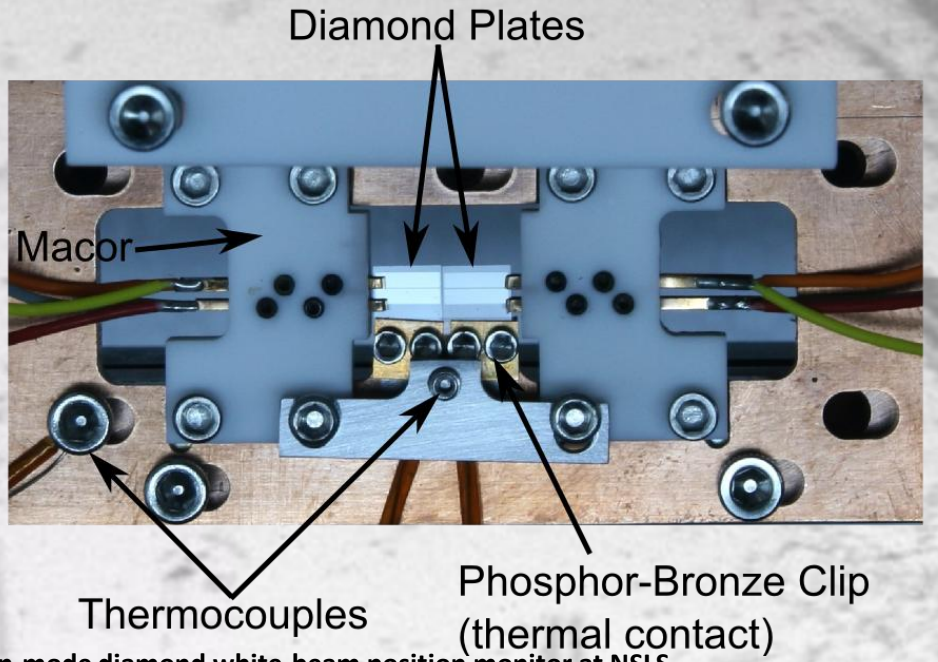
# X25 White Beam Position Monitor

- **Installed 13.6 m from undulator at X25**
- Large (6x1 mm$^2$) beam; up to 100 W, 11W absorbed
- Two 100 µm thick E6/DDL single crystal diamond plates tiled side-by-side
- Selected with topography
- Custom 4-channel current amplifier
- Up to 760 mA observed
- Position noise:

**Better than 0.5 x 0.05 um**






**Transmission-mode diamond white-beam position monitor at NSLS**
E. M. Muller, J. Smedley, J. Bohon, X. Yang, M. Gaowei, J. Skinner, G. De Geronimo, M. Sullivan, M. Allaire, J. W. Keister, L. Berman and A. Héroux
*J. Synchrotron Radiation,* **19**, 381-387 (2012)

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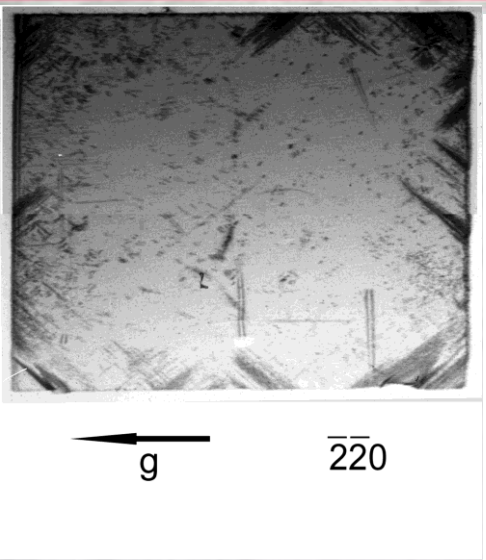
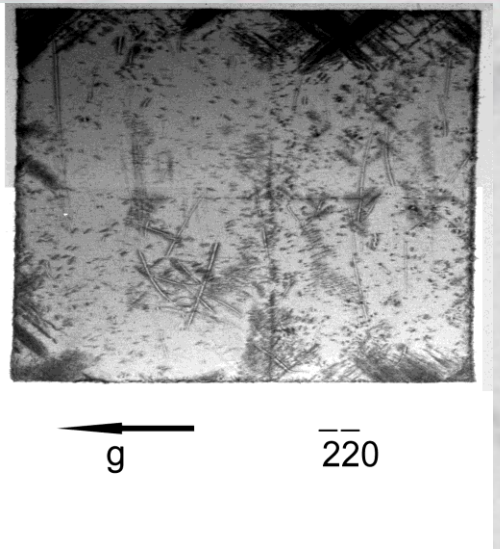
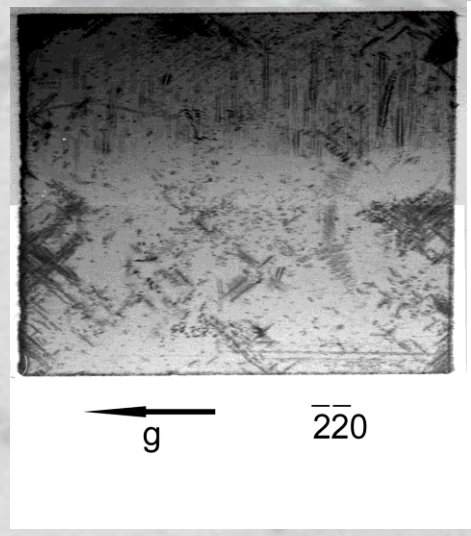



1441B




1443A

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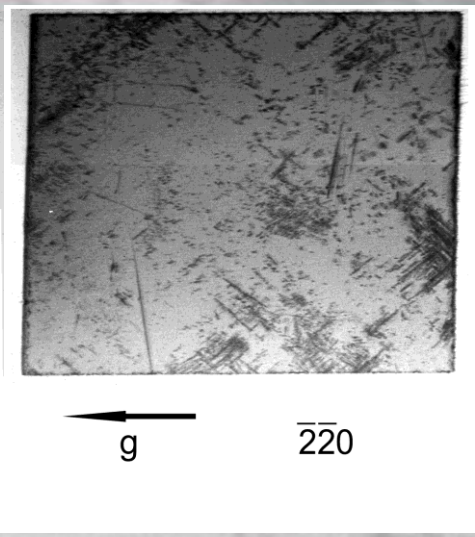
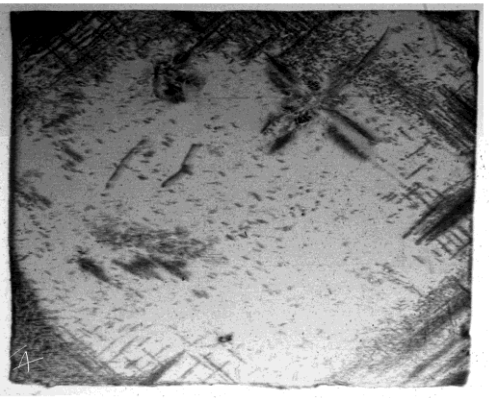
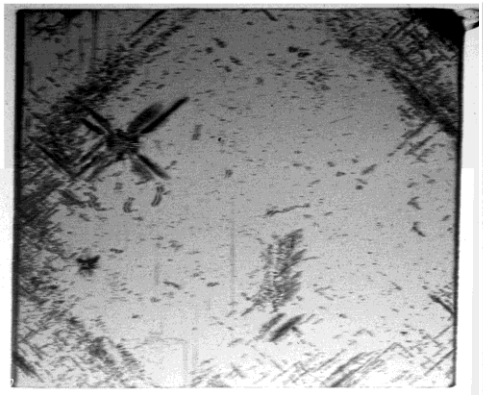



1447B

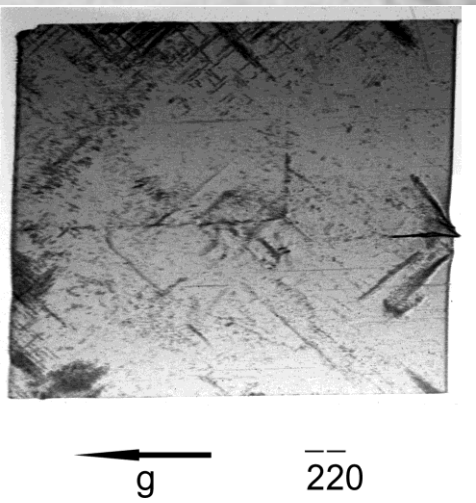



1690A




1690B

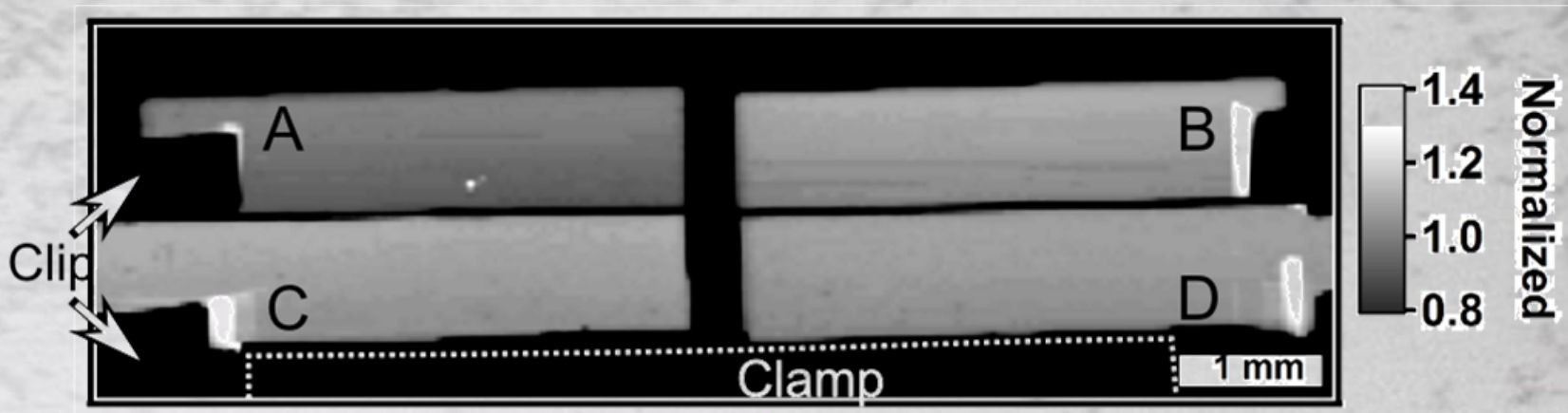

1694A



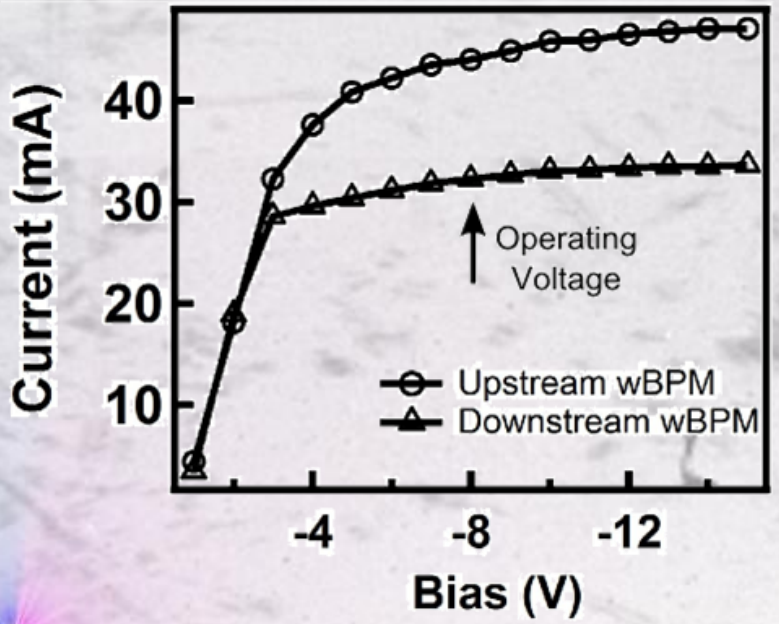

1694B

# Monitor Calibration

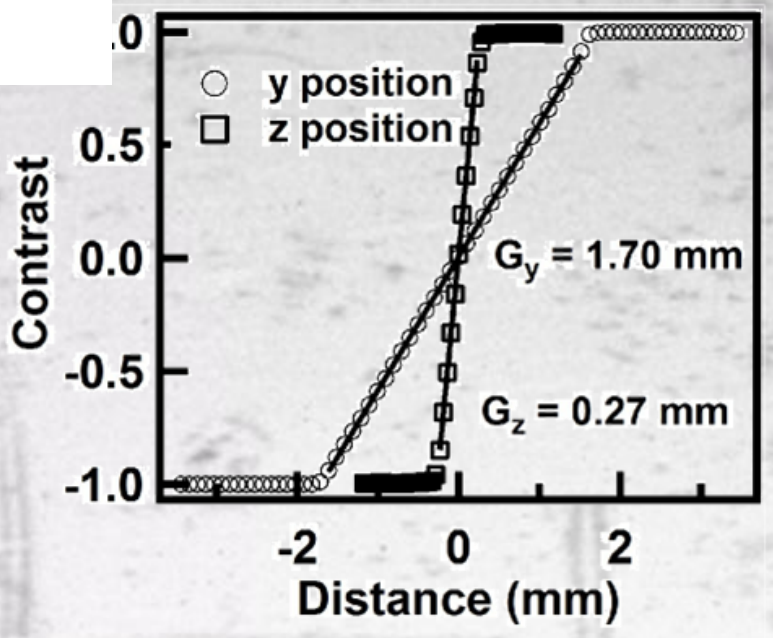
XBIC Map




Bias Calibration




Position Calibration

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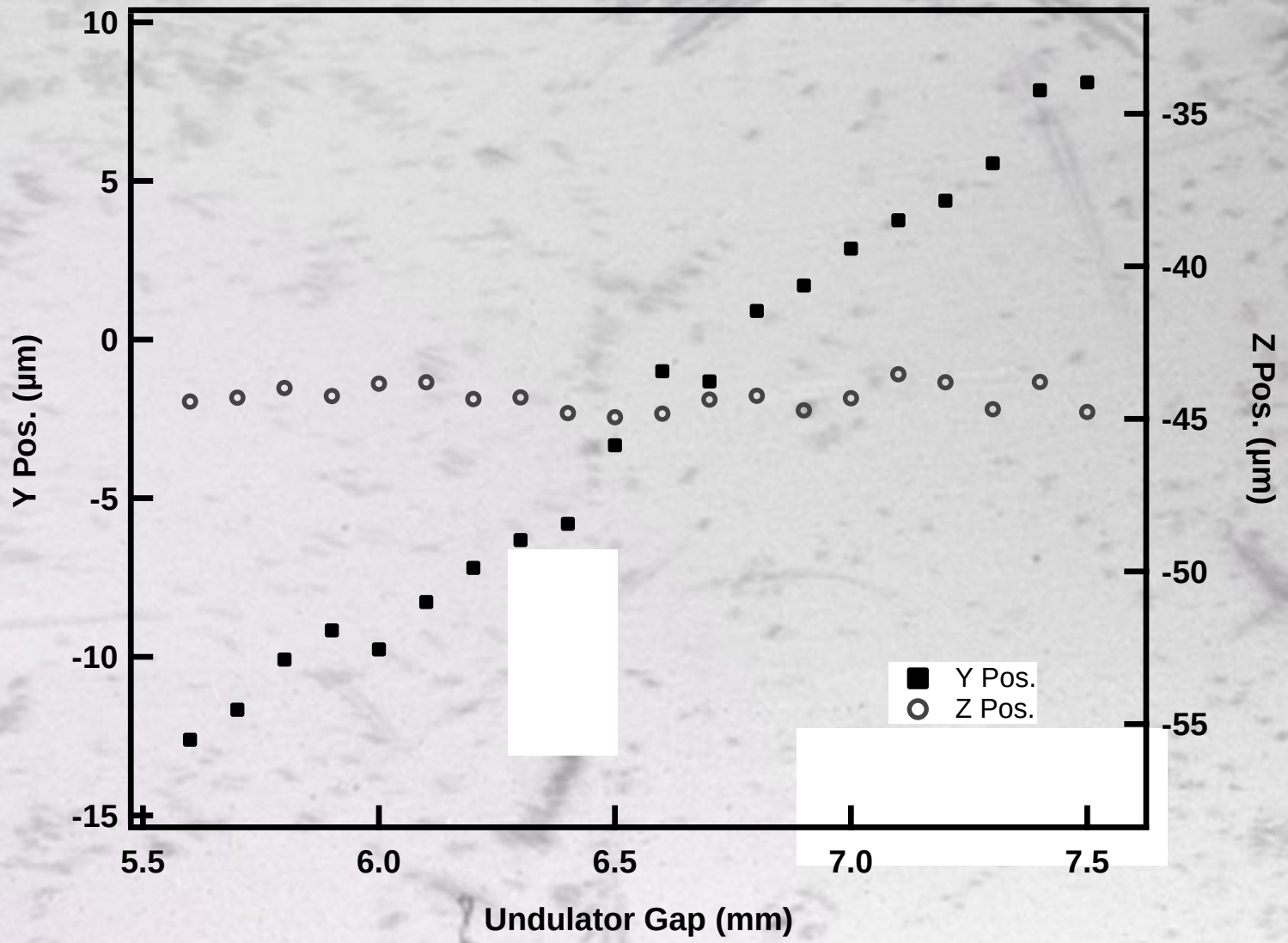
# Monitor Results




Moving electron beam changes photon beam position

**Beam position changes with undulator gap**

Extent of motion is a function of e-beam position

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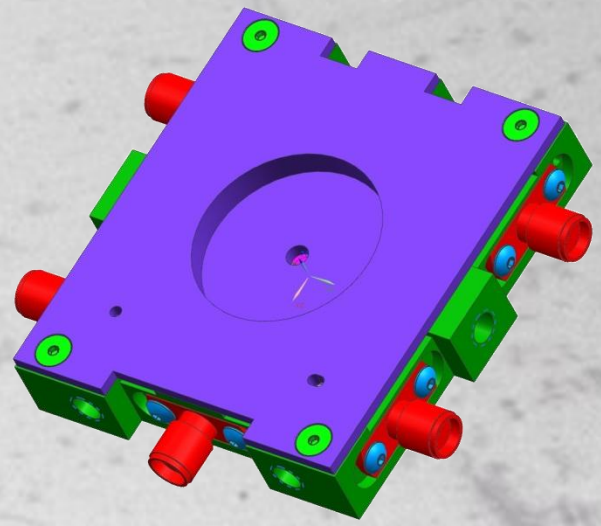
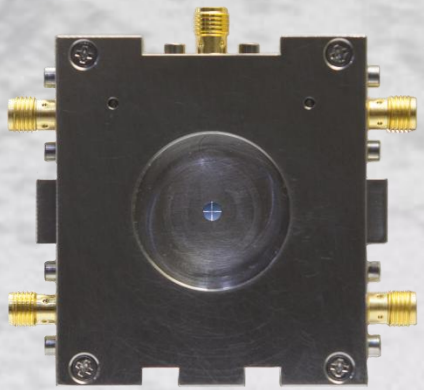
- *Sydor Instruments, LLC* is actively collaborating with BNL in developing the Diamond BPM technology.
- SBIR Phase II awarded for FY2013-2014. Phase IIa just awarded for 2015-16
- Currently developing prototypes for white beam and monochromatic beam applications.
- User requirements is driving commercial development.

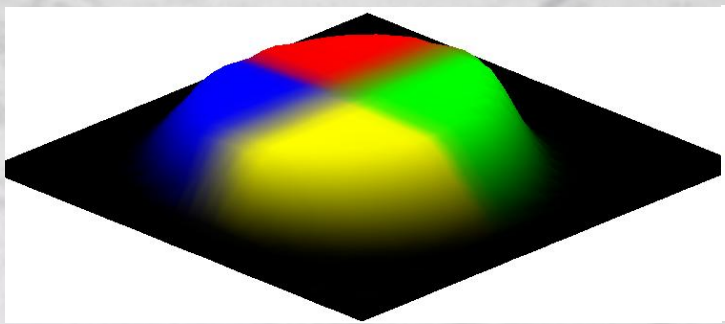


For more information please contact:
Jaime Farrington
jaimef@sydorinstruments.com
585-764-9584

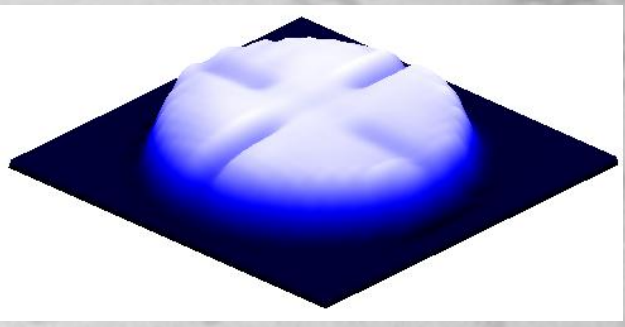
SYDOR INSTRUMENTS, LLC

# Sydor Quad Results



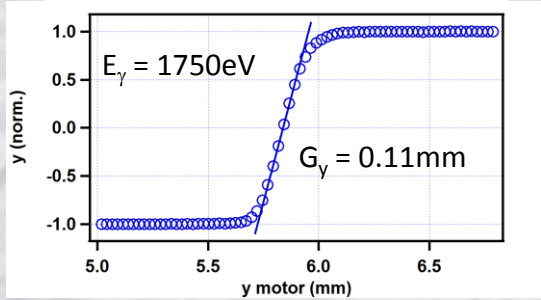


$E_\gamma$ = 4keV, -5V DC



$E_\gamma$ = 3keV, 0.3V 90%

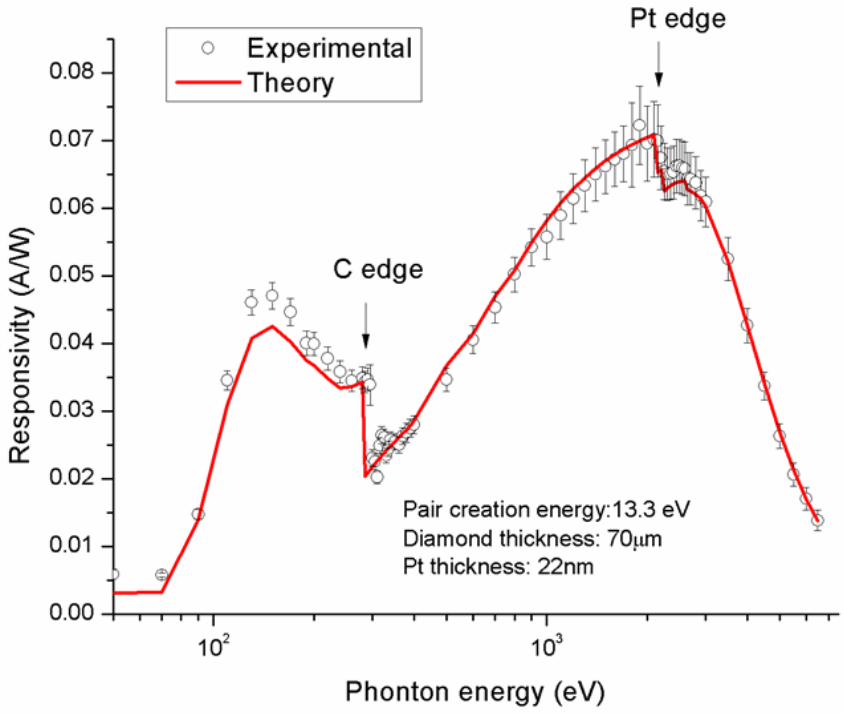
200µm circular beam calibration




| This is for -20V on incident, 90% duty cycle | | |
|---|---|---|
| For 1.6mm beam:<br>Gx=0.74mm<br>Gy=0.76mm | For 0.8mm beam<br>Gx=0.35mm<br>Gy=0.36mm | For 0.4mm beam<br>Gx=0.27mm<br>Gy=0.19mm |

Photon energy for 68µm attenuation length = 3850eV

## Energy Calibration

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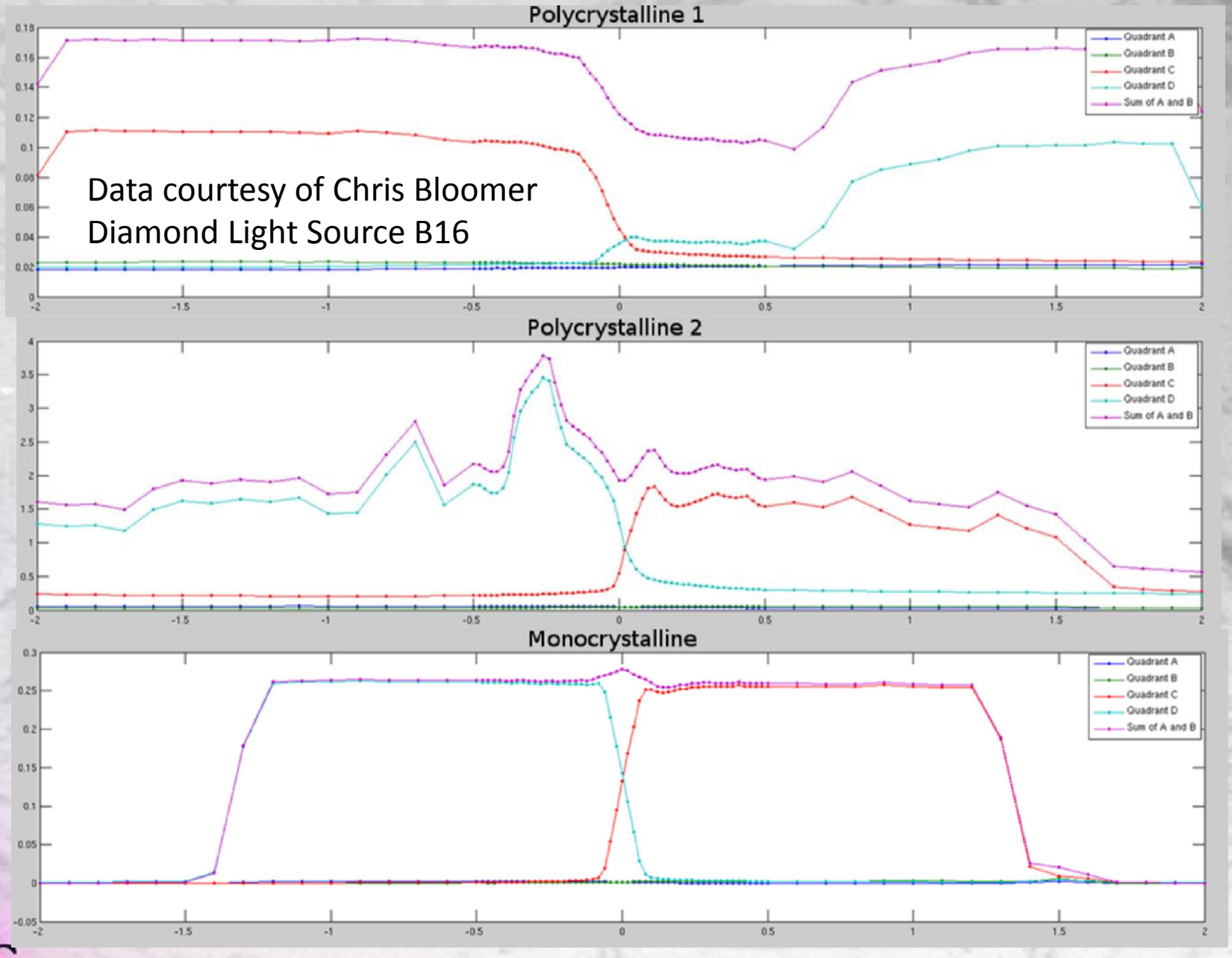
# Comparison to Dectris DBPM

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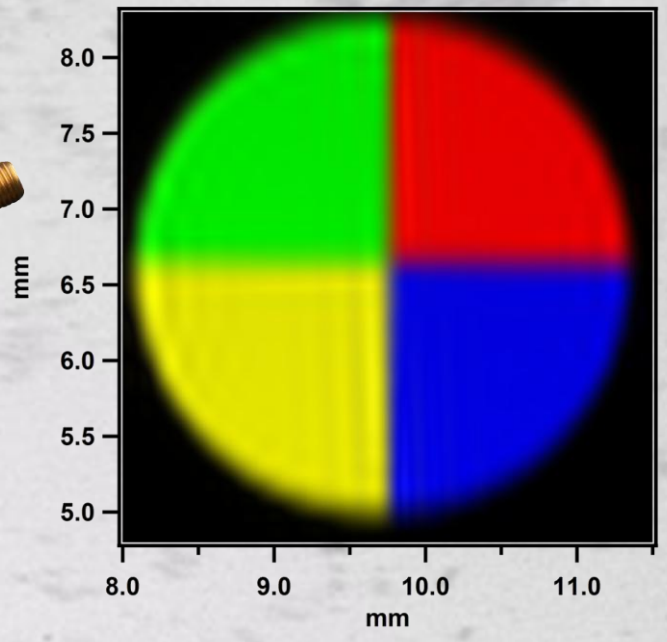
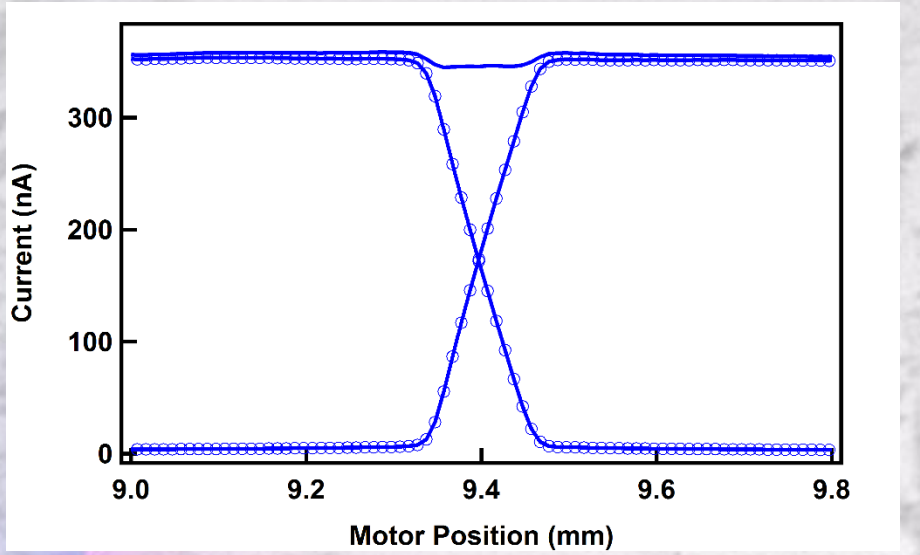
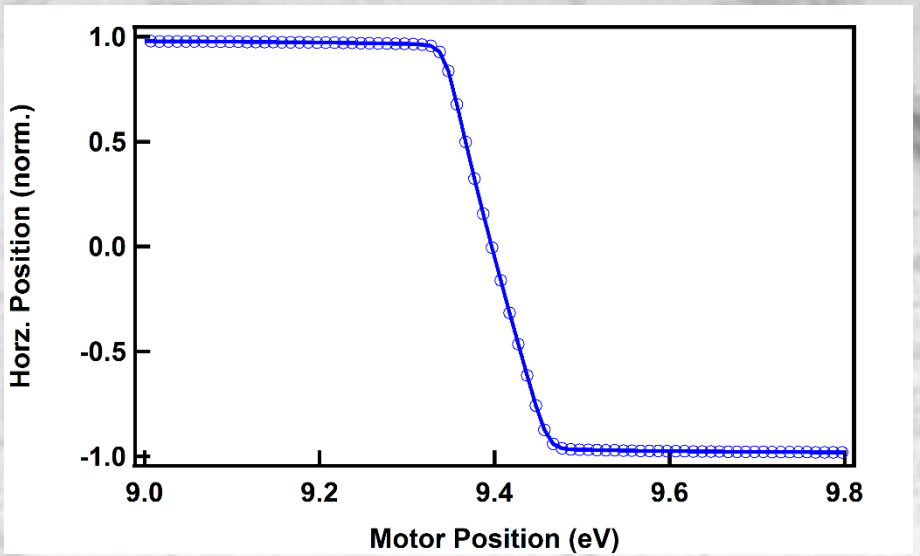



## Quad detectors for NYSBC

- 2 quad detectors (65µm and 80µm thick)
- 3.6mm x 3.6mm x 30nm Pt contacts, 20µm streets
- High thermal conductivity ceramic circuit board (Beryllia)
- Integrated vacuum seal
- Operating voltage 10V.
- Self-aligning defractometer

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# Readout options

- Single channel devices can be read by current amplifiers and digitizers, as ion chambers
- Quadrant devices can be read out several ways
  - Adapting NSLS II eBPM readout
    - Two versions: RF and electrometer
    - RF version measures impulse response rather than current
      - May allow utilization of lower grade of diamond
    - Take advantage of existing NSLS II integration
  - Custom 4-channel current amplifier
  - 4-channel electrometer
  - Sydor marketing these

# Diamond Instrumented Window

**Development of a diamond window which will provide position, flux and morphology of high flux x-ray beams while simultaneously acting as the vacuum-air interface.**

**X-ray footprinting (XFP) at NSLS-II**

- Focused white beam
- Variety of beam sizes/shapes needed
- Feedback and control systems for optical elements or sample positioning stages.

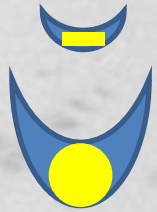
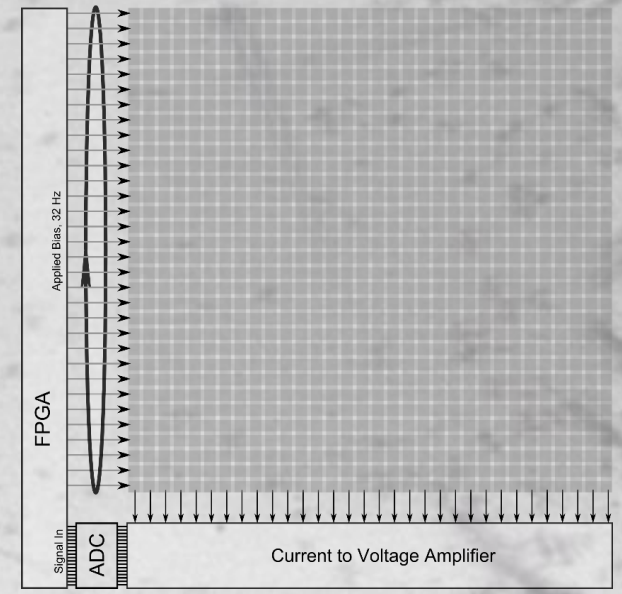
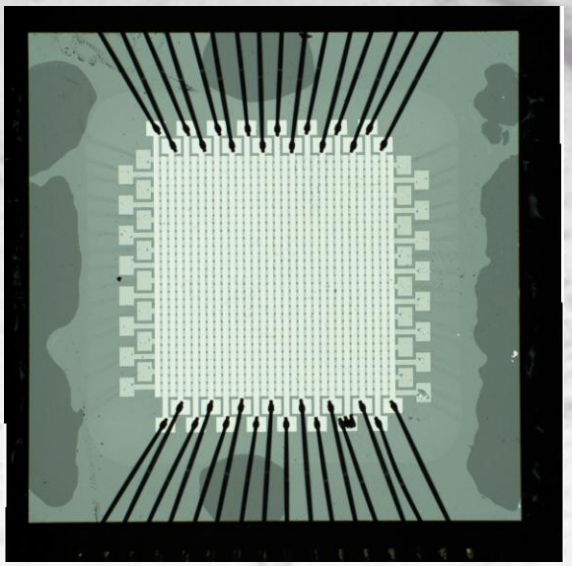


**Image Readout**

- Goal of 32 x 32 stripes, yielding 1024 pixels
- Only one row is active at a time minimizing ohmic heat generation.
- Real-time imaging at 32 Hz
- Allows for feedback control of beamline components.



**Major Challenges for high flux beamlines**

- Heat load management in optics (including beryllium windows)
- Real-time volumetric measurement of beam properties such as flux, position, and morphology.

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# Current and Future Work

**Instrumented window**

- Design, fabricate and test assembled vacuum window
- Increase the dynamic range to extend to lower flux measurement

**SAXS detector**

**XAS flow cell**

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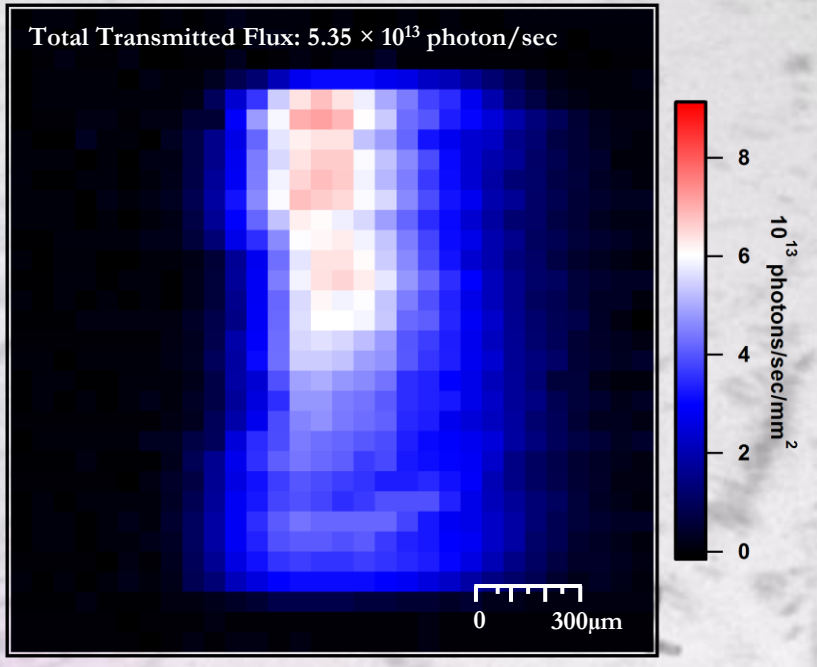
Diamond Imaging Detector @ CHESS, G3




- G-line: Three station beamline with common 49-pole wiggler source
- Can deliver high flux monochromatic beam
- Horizontal and vertical focusing

w/ A. Woll, J. Smedley, M. Gaowei, W. Ding, J. Bohon, T. Zhou, E. Muller

# What do we want out of an electron source?

- The electron beam properties determine the photon beam properties
  - Pulse duration, degree of coherence, flux
- In all light sources through 3rd generation, the phase space is determined by the ring

- In 4th generation sources (LCLS, XFEL, NGLS), this will change – the electron source will determine the beam properties
- The highest brightness sources available are photoinjectors, which use a laser on a photocathode to control the spatial and temporal profile of the emitted electron beam

# What do we want out of photocathode?

- High Brightness: $B = \frac{N_e}{\varepsilon_{nx}\varepsilon_{ny}\varepsilon_{nz}}$
  - large number of electrons in a small volume of phase space
- Low Emittance: $\varepsilon_n = \sigma_x \sqrt{\frac{\hbar\omega - \phi_{eff}}{3mc^2}}$
  - Determines the electron energy required for an X-FEL at a given wavelength $\varepsilon \approx \frac{\lambda}{4\pi} \Rightarrow \frac{\varepsilon_n}{\beta\gamma} \approx \frac{\lambda}{4\pi}$
- High Quantum Efficiency
  - High Average Current
- Long Operational Lifetime
  - Chemical Contamination
  - Ion back bombardment
- Sub-ps response time

*The optimal cathode is still a work in progress*

*It is becoming increasingly clear that material parameters such as texture and surface roughness may play an important role*

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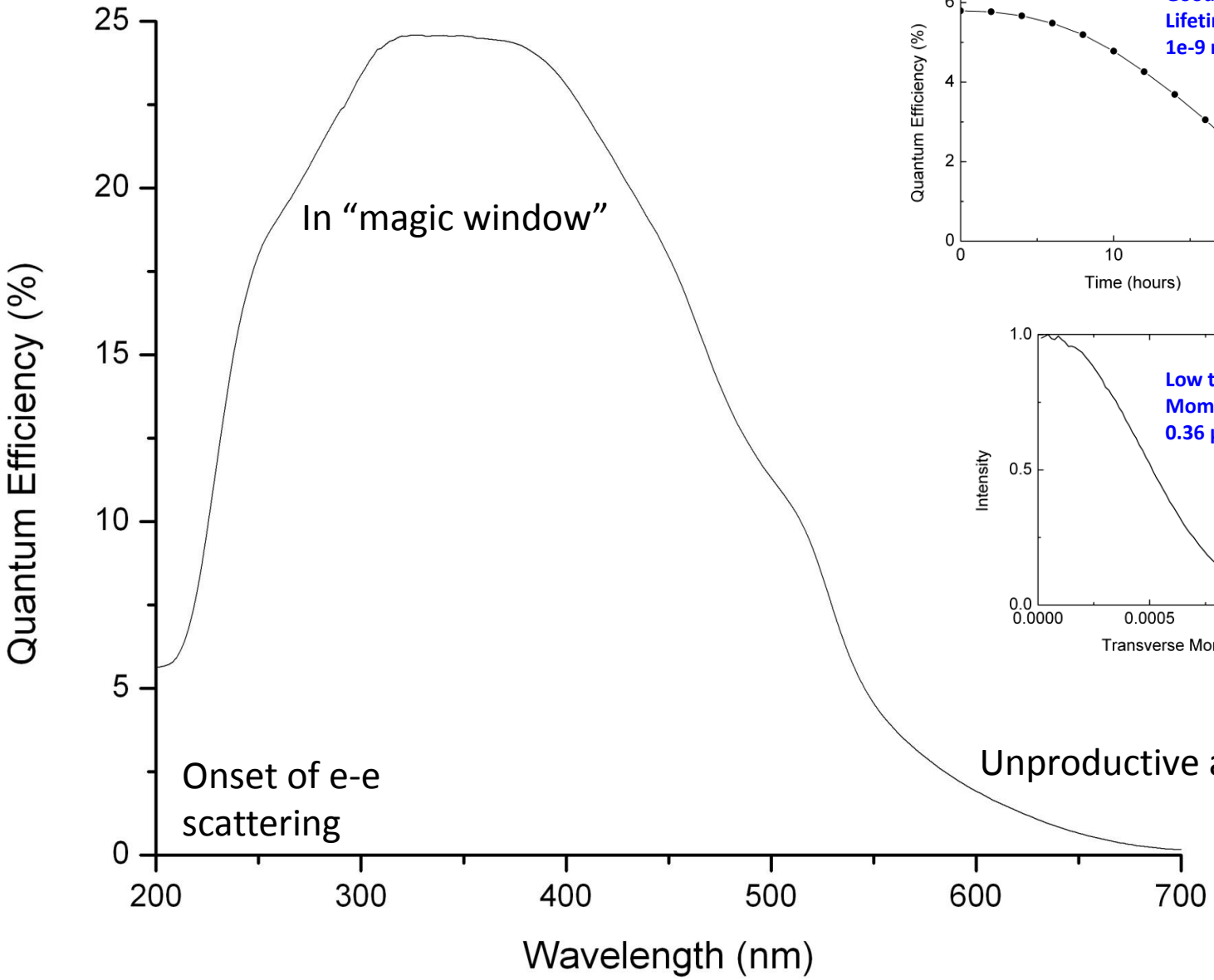
# $K_2CsSb$: A cathode with excellent characteristics for accelerators



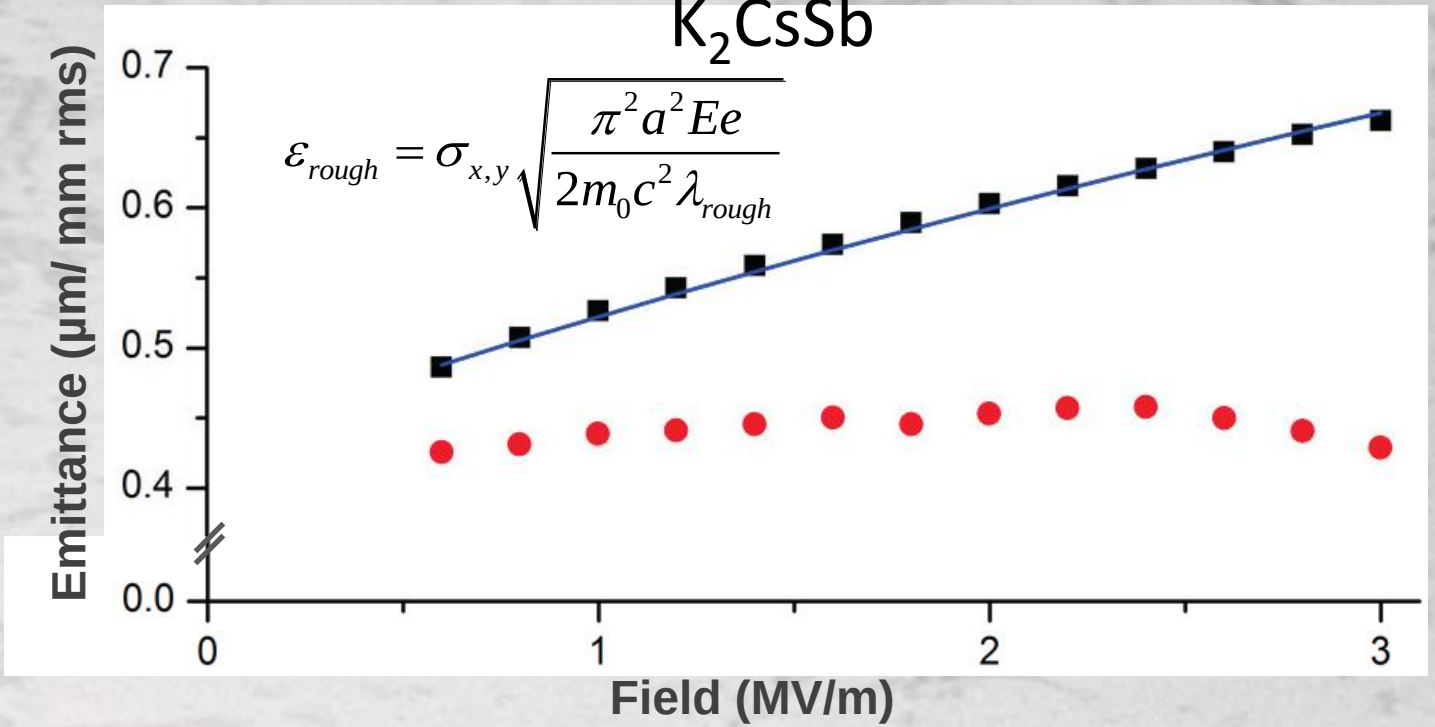







T. Vecchione et al., *Appl. Phys. Lett.* **99**, 034103 (2011)

# Effects of roughness seen in the emittance of thick multilayer $K_2CsSb$




Thin films grown at high rate give ~ expected emittance (very low field dependence)

Films grown in a multilayered manner were shown to give higher QE but showed marked emittance growth with field

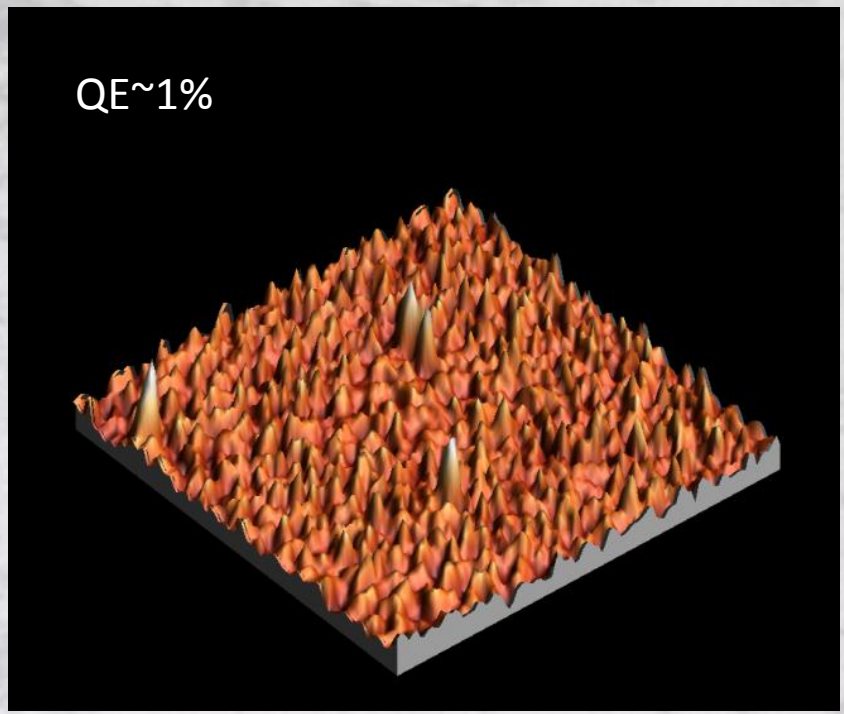
Can be explained by invoking a simple roughness model.

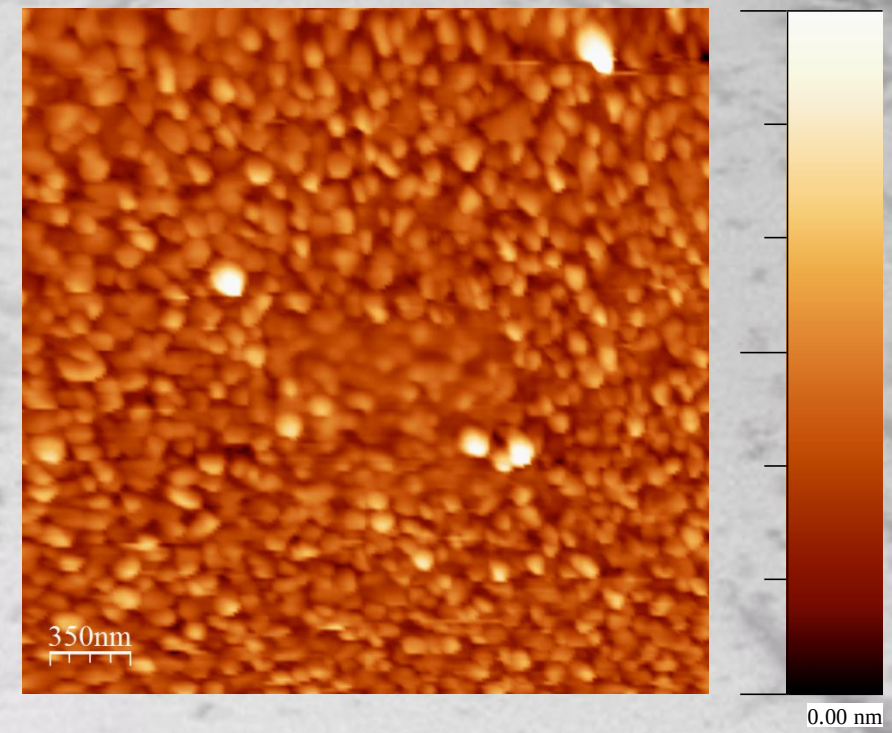
Fitting gave reasonable roughness parameters, confirmed by in vacuum AFM

**Roughness in high gradient guns looks to be an issue based on current in-situ measurements of cathode surfaces**

T Vecchione et al., Proceedings of FEL2011, Shanghai, China 179 (2011)

# *in-situ* AFM on cathode at CFN




10 nm Antimony film evaporated at room temperature

Potassium and Cs added by monitoring QE

Should result in a 50 nm thick final film

Observed **25 nm** RMS roughness, with a 100 nm spatial period

Nano-pillars of uniform height – consistent with XRR and GISAXS

**Likely the source of the Field dependence of the intrinsic emittance**

S. G. Schubert, et al, APL Mater. **1**, 032119 (2013)

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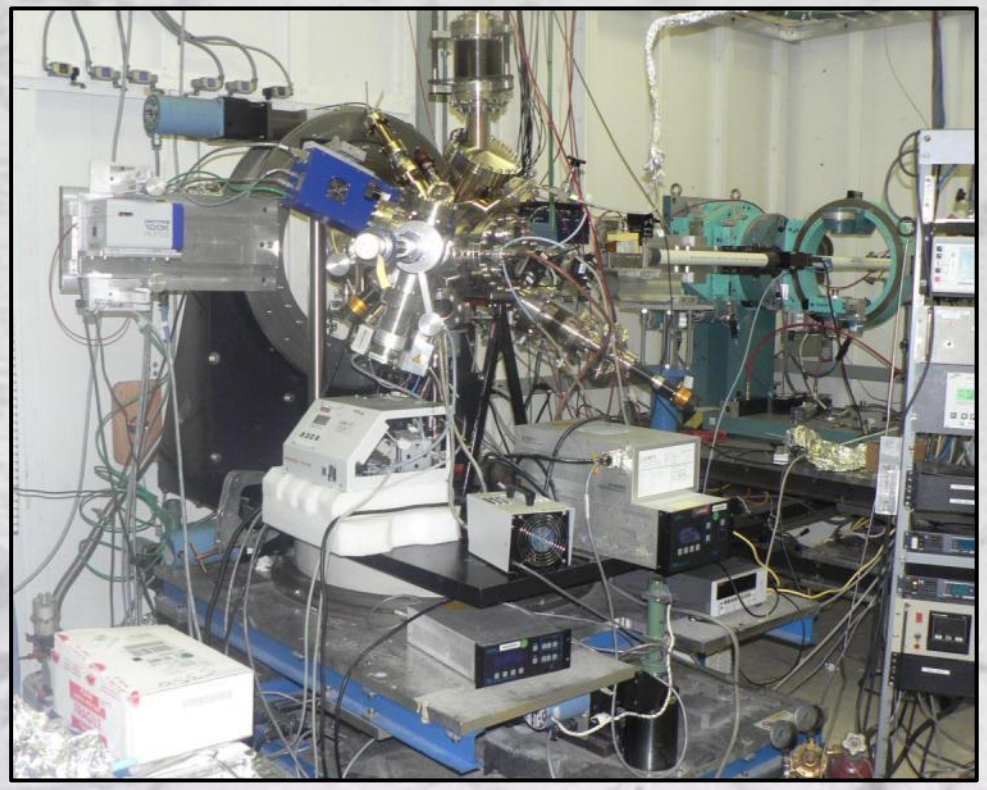
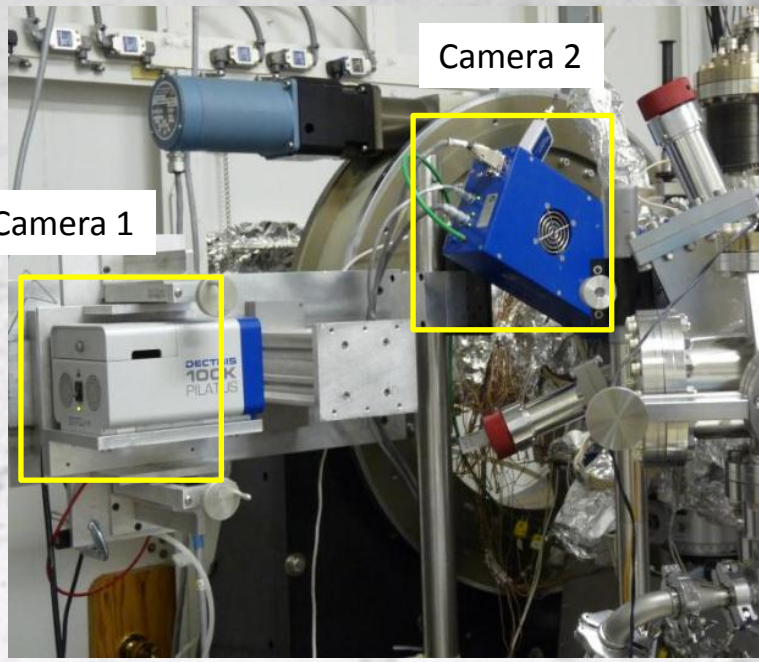
# *In operando* analysis during growth
## (setup at NSLS/X21 & CHESS G3)



- UHV system ( 0.2 nTorr base pressure)
- Residual Gas Analyzer (RGA)
- Heating/cooling substrate/cathode
- Load lock
  - fast exchange of substrates
  - gun transfer
- Horizontal deposition of Sb, K and Cs.

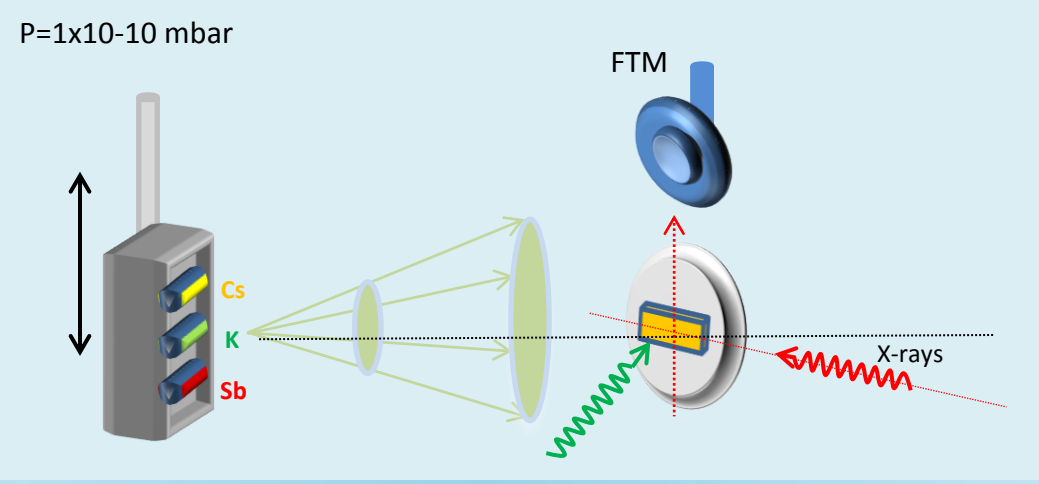



**Two 2D detectors (Pilatus 100K)** ⇨

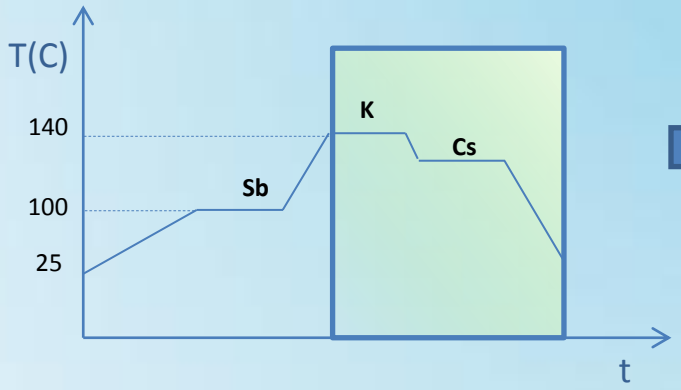
Next Stop: ISR at NSLS-II

# Experimental set up: $K_2CsSb$ cathode growth

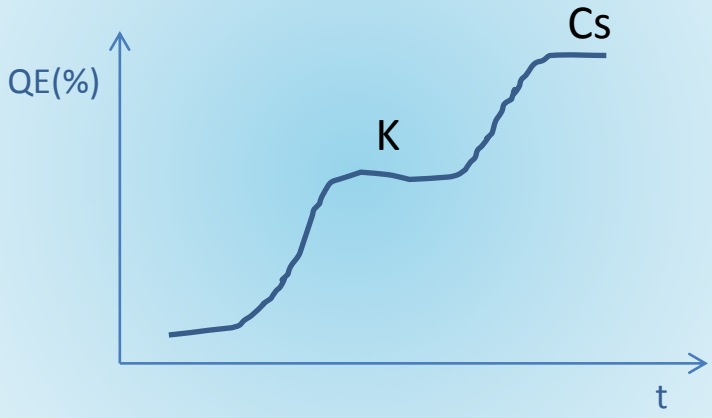
Horizontal evaporation of three sources:




Recipe:




QE during growth (532 nm laser)



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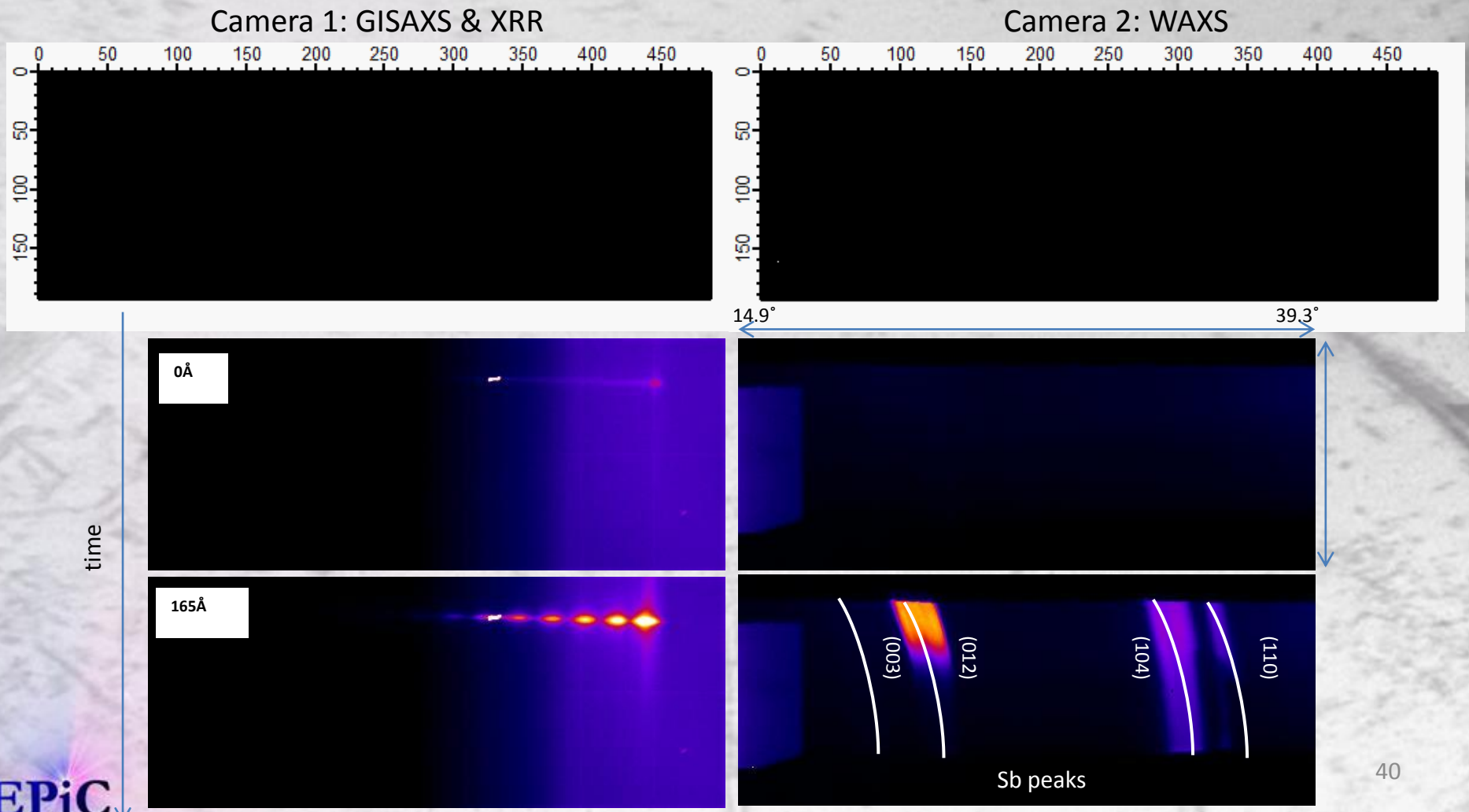
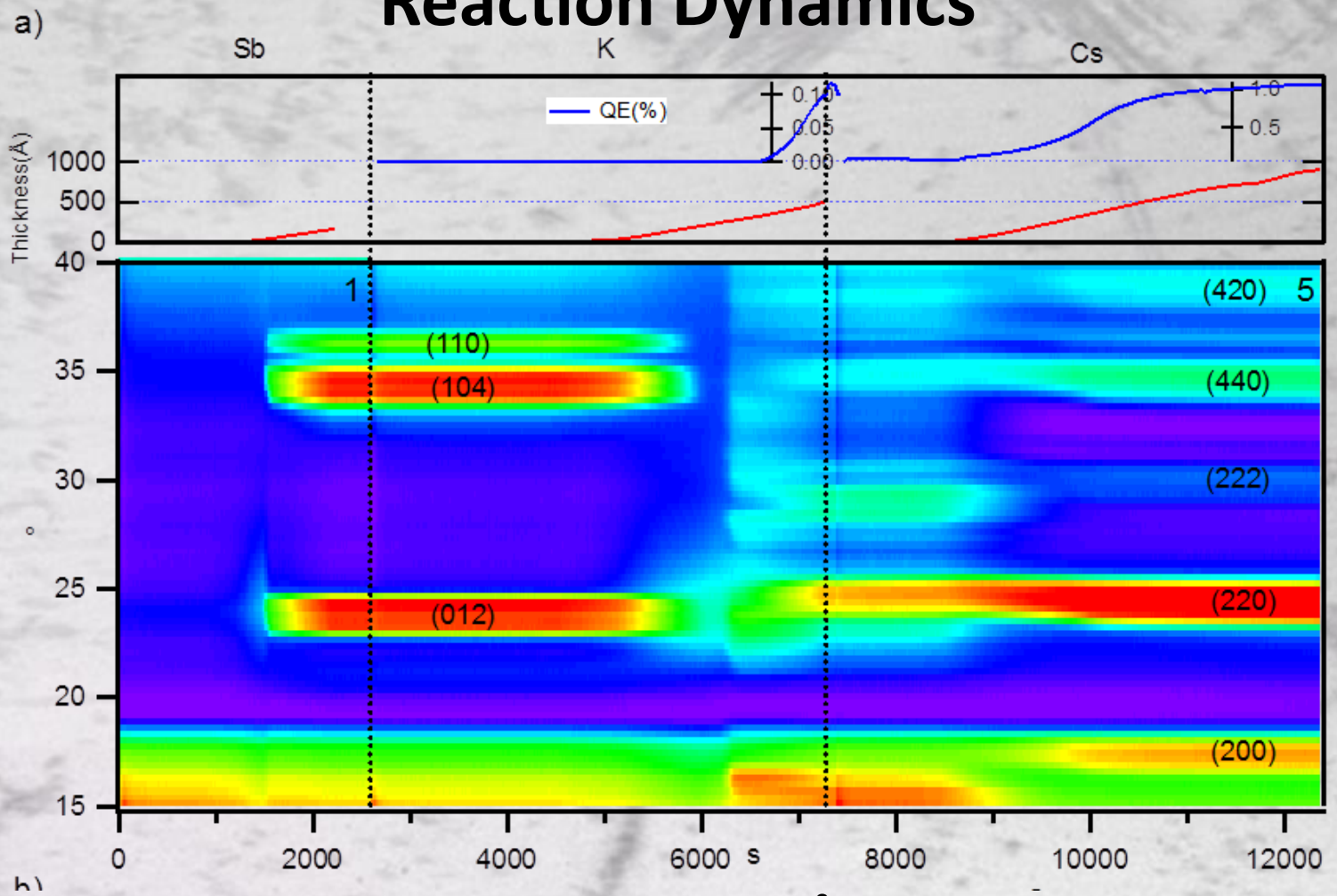
# Simultaneously Acquire XRD and GISAXS

- Understanding reaction dynamics through crystalline phase evolution
- Map the thichness and roughness evolution of the cathode
- Is there a correlation between reactivity, QE and roughness?



# Reaction Dynamics




Antimony evaporated on Si, 0.2 Å/s; crystallize at 4nm

K deposition dissolves Sb layer - This is where roughening occurs!

QE increase corresponds with $K_xSb$ crystallization

Cs increases lattice constant and reduces defects

M. Ruiz-Osés et al., APL Mat. 2, 121101 (2014)

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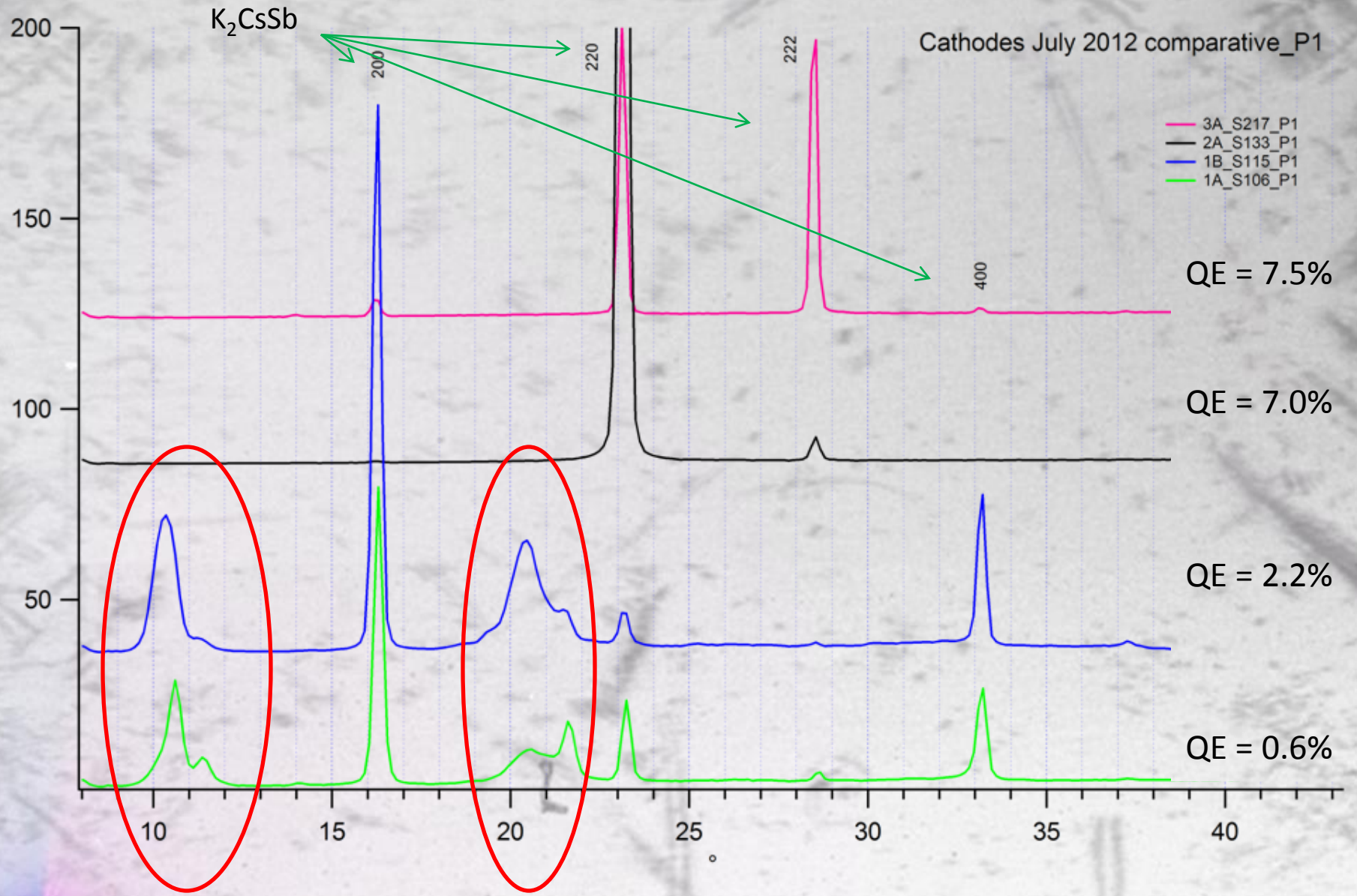
# Comparison of Crystal structure and Final QE



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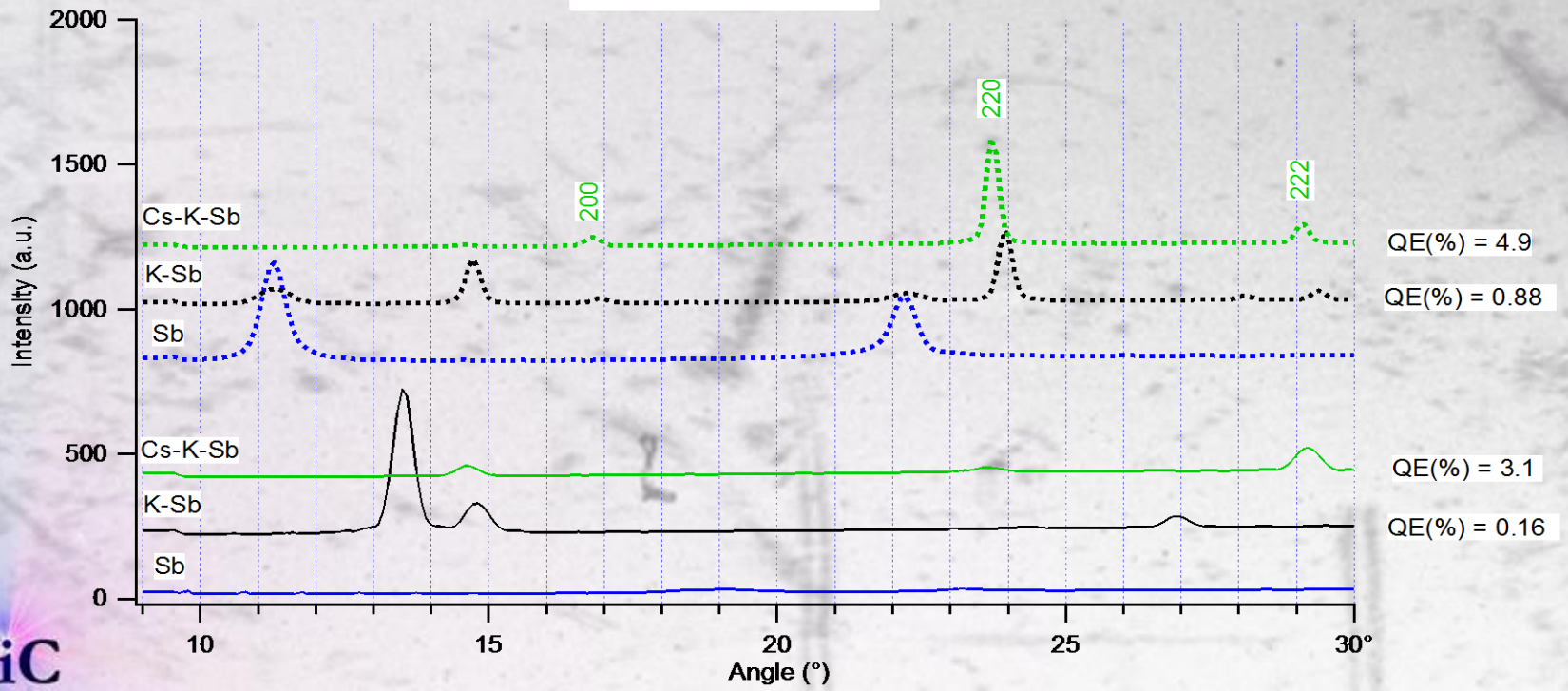
# Engineering a Smoother Cathode

## Idea: Never let Sb crystalize

First layer:
3 nm Sb @ 100C
12 nm K @ 125C QE 0.16 %
42 nm Cs @ 125C QE 3.1 %

Second layer:
5 nm Sb @ 125C
16 nm K @ 125C QE 0.88 %
44 nm Cs @ 125C QE 4.9 %

Second layer on top of first

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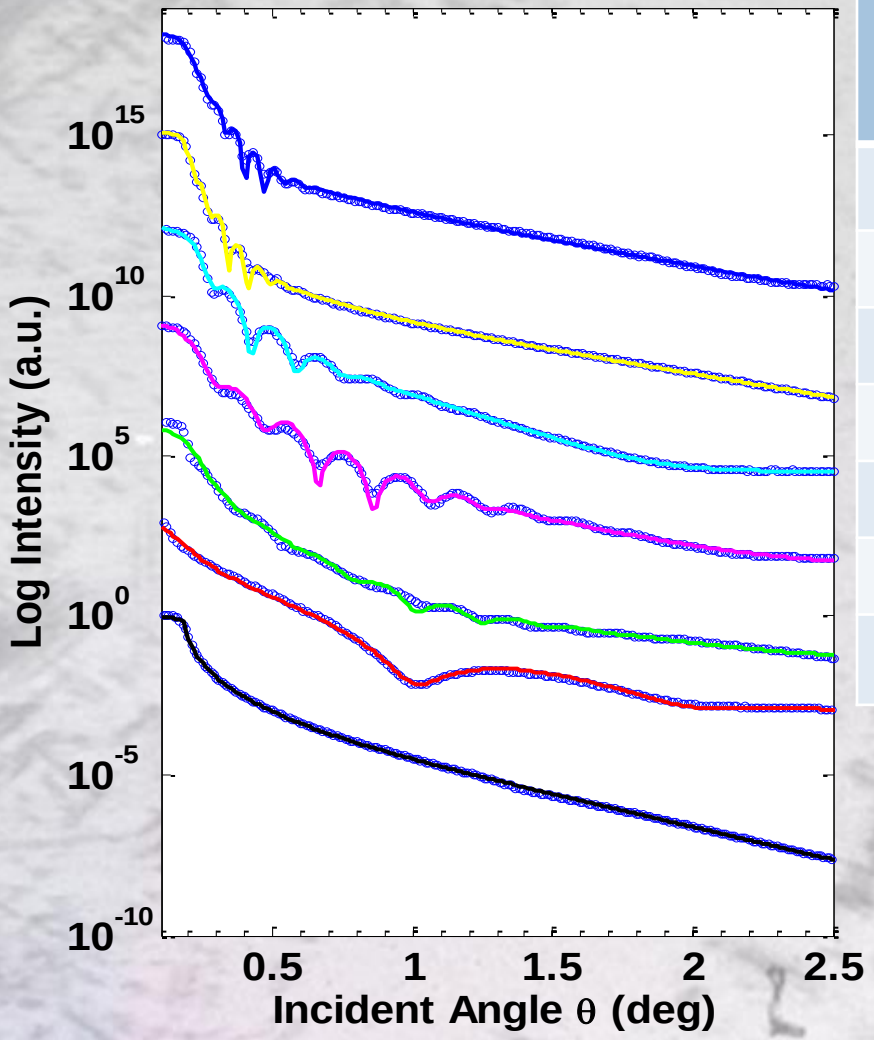
# XRR shows roughness evolution




| Deposited Layers | Total Thickness (Å) | Roughness (Å) |
|---|---|---|
| Cs-K-Sb-Cs-K-Sb/Si | 469 | 32 |
| K-Sb-Cs-K-Sb/Si | 449 | 36 |
| Sb-Cs-K-Sb/Si | 200 | 21.3 |
| Cs-K-Sb/Si | 174 | 13.2 |
| K-Sb/Si | 141 | 10.5 |
| Sb/Si | 35 | 2.9 |
| Si Substrate | - | 3.1 |

The substrate fit includes 1.5 nm of $SiO_2$

Multi-layer subcrystalline film is smoother,
At slight loss of QE

Next Stop: Sputter Deposited Cathodes

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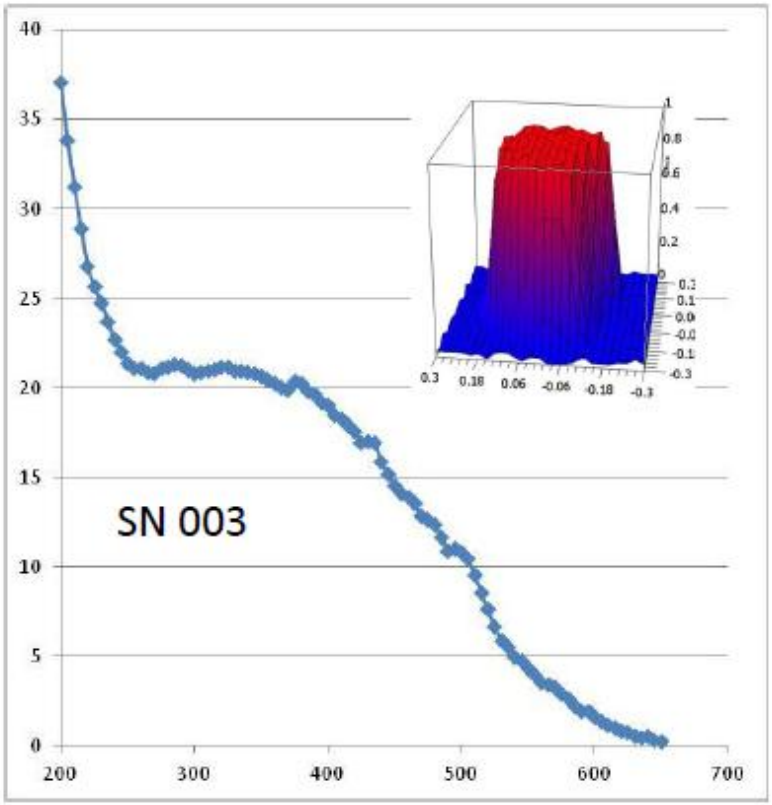
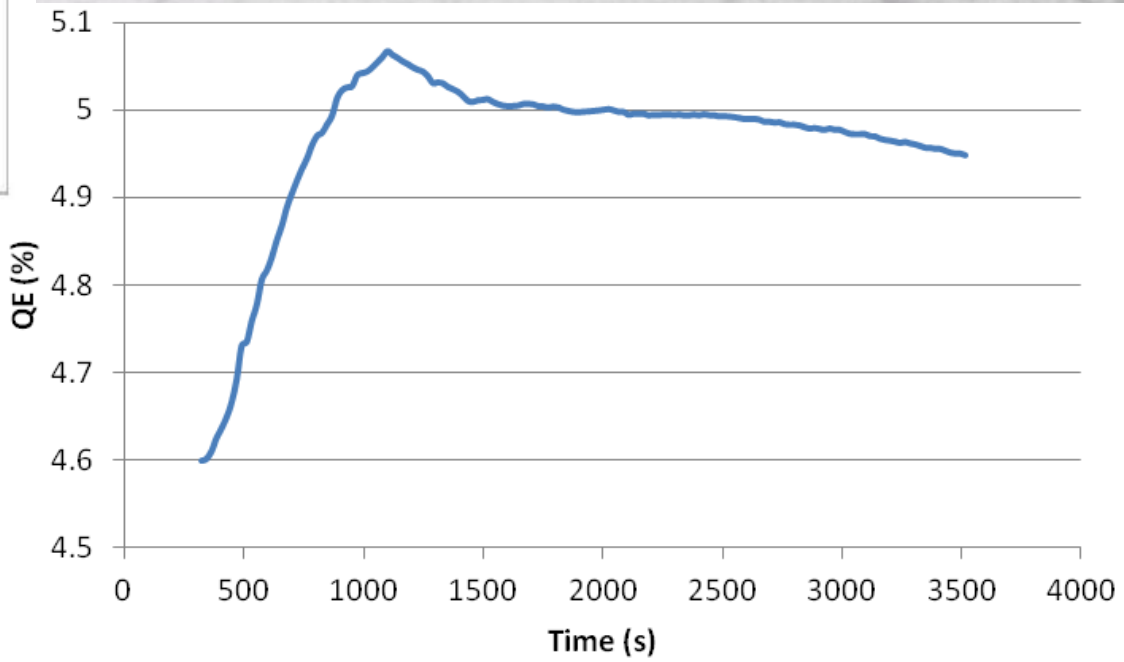
# Sealed Capsule Photocathodes




Photonis USA, using detector growth process

Shelf life of months at least

$NaK_2Sb$ available




QE drops during heating to remove cap, but recovers

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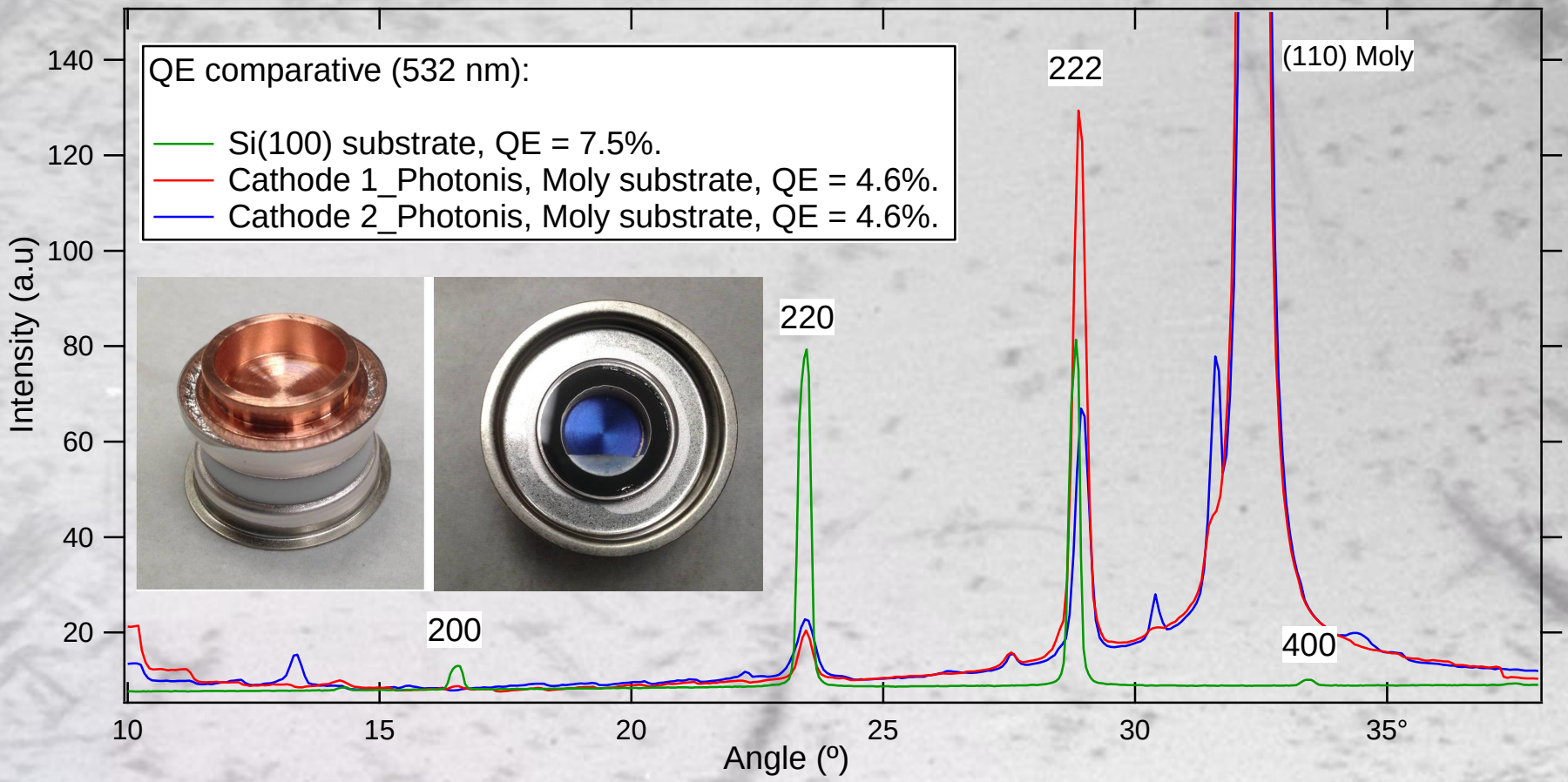
# Sealed Capsule Photocathodes




Comparison to Photonis commercial PMT cathode

- Similar texture (222 surface normal preferred)
- Broader peaks imply smaller grain size
- (50 nm for BNL cathode, 39 nm for Photonis cathode)

# Conclusions

- From Photocahtodes to X-ray diagnostics, we can use our tools to build better ones
- Alkali Antimonide cathodes
  - Peak QE of 35% and a green QE of 7.5% have been achieved
  - We are achieving control of both substrate and cathode crystal structure
  - Traditional cathodes are *very* rough… but we are learning to make them smoother
  - Sealed capsule cathodes may reduce cost and complexity in new user facilities
- Diamond
  - Flux linearity demonstrated over ***11 orders of magnitude***
  - Persistent current/photoconductive gain results from point defects in the material, which can be screened for via topography
  - White beam beam position monitors have been in operation for 2 years
  - Position resolution of better than 50nm, and single bunch flux and position have been achieved
  - 50 devices delivered or on order world wide (APS, CHESS, Diamond, NSLS-II)
  - Pollycappilary lenses solve the diffraction dropout problem for energy scanning applications, and allow high resolution EXAFS mapping
  - 1k Pixel beam imaging system demonstrated for both white and monochromatic beams

**Thank you!**

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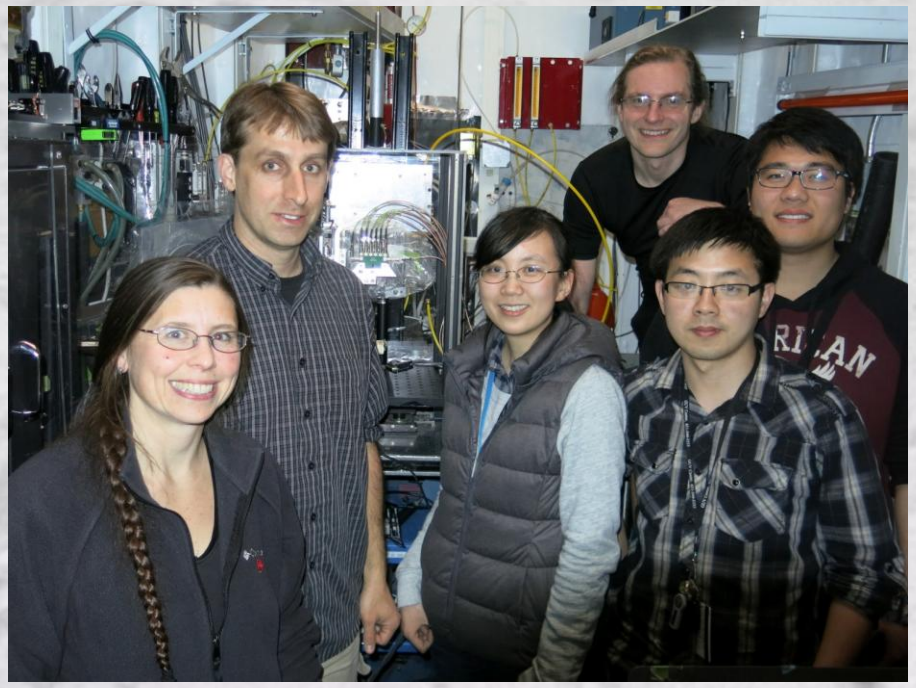
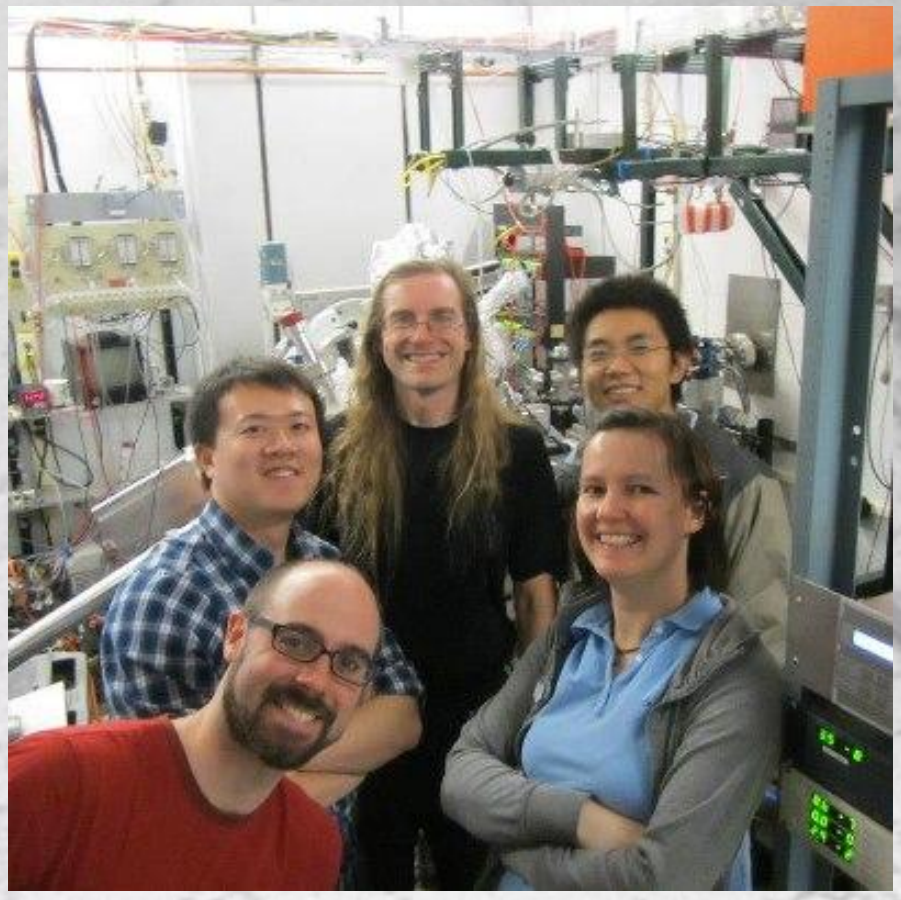
# Electron Photon Instrumentation Center





EPiC Stony Brook University

Berkeley Lab

Brookhaven National Laboratory

Office of Science U.S. Department of Energy

# Thanks for your attention!


- Thanks to K. Attenkofer, J. Mead, W. Ding, T. Zhou, M. Maggipinto, A. Della Penna, T. Rao, S. Schubert, M. Ruiz Oses, J. Xie, J. Wang, H. Padmore, E. M. Muller, J. Bohon, J. Mead, M. Gaowei, A. Héroux, L. Berman, M. Sullivan, R. Beuttenmuller, J. Jordan-Sweet, J. Keister, A. Sumant, E. DiMasi, J. Walsh, B. Raghothamachar, **J. Distel**, K. Vetter, G. DeGeronimo, B. Dong, D. Dimitrov, D. Pinelli, J. Skinner, M. Cowan, S. Ross, R. Tappero, B. Ravel, C. Jaye, D. Fischer, M. Lu, F. Camino, D. Abel, I. Ben-Zvi, T. Vecchione, X. Liang, J. Rameau, P. Johnson
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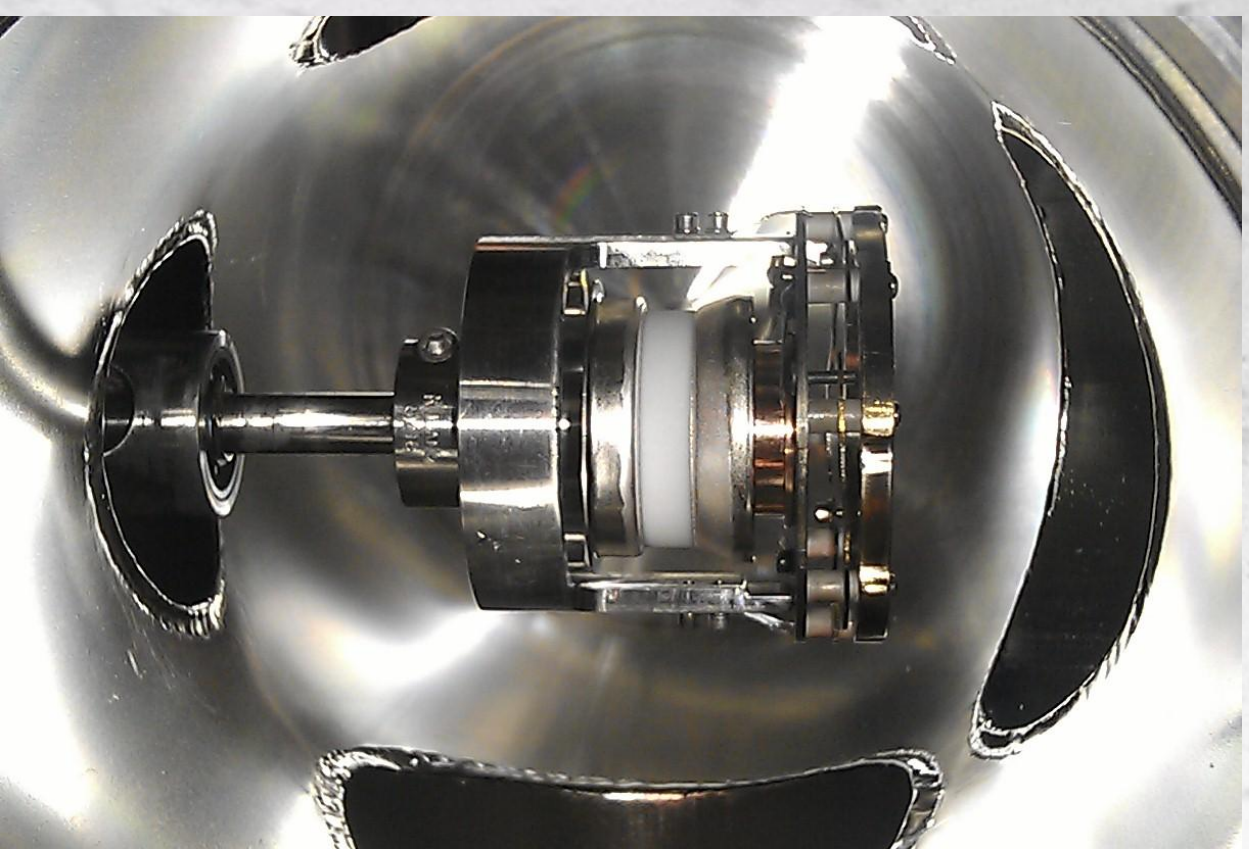

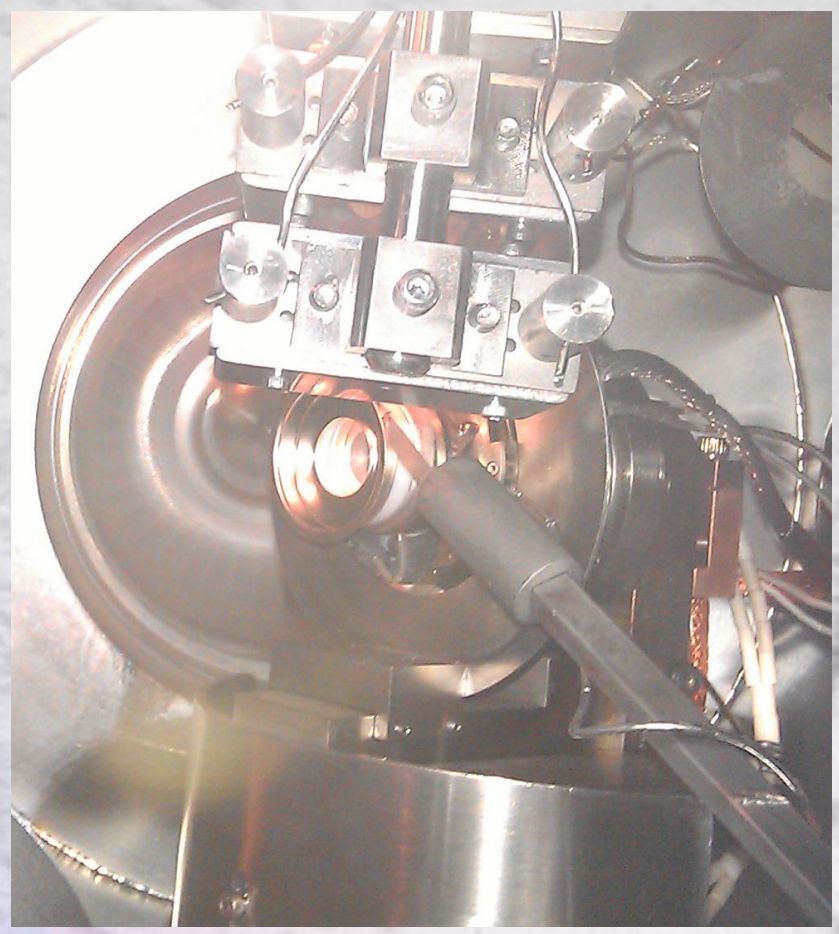

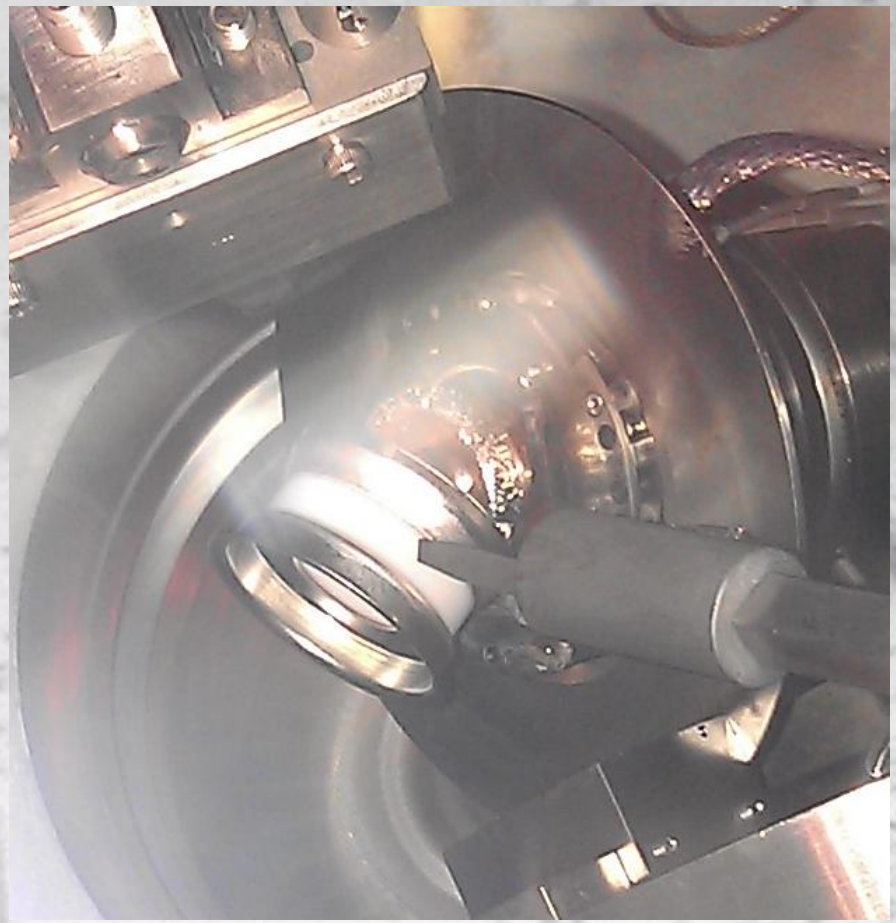

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