Characterizing x-ray mirrors in reciprocal space

Preliminary results from the NIST
X-ray Optics Evaluation Double-Crystal Diffractometer

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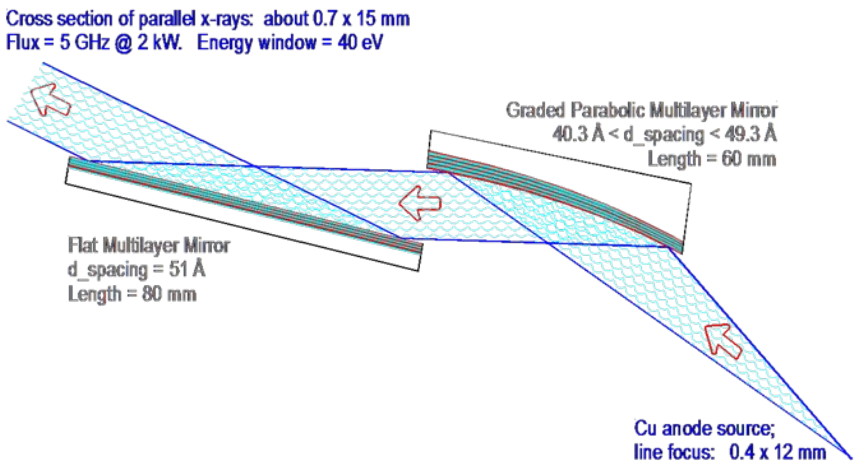
Introduction

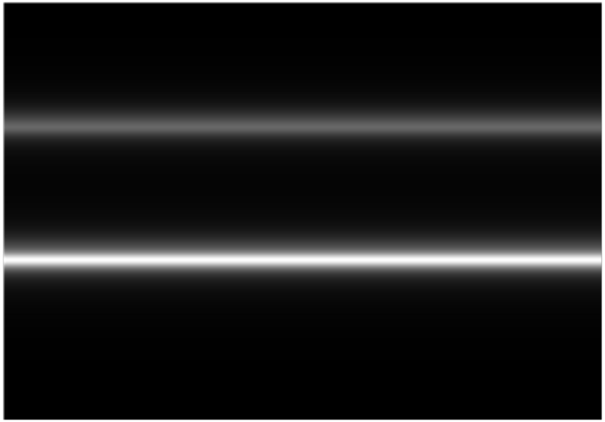
Old NIST PBPD instrument

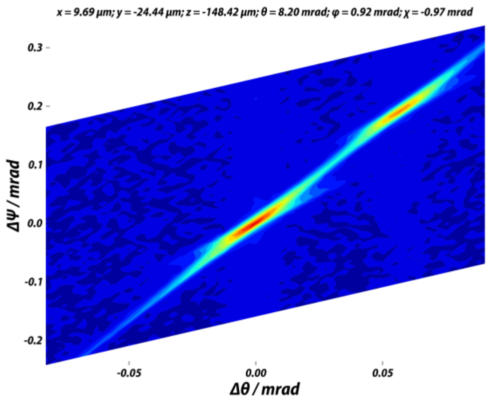
- Deslattes et al. demonstrated the usefulness of PGM optics for high-accuracy PD lattice parameter measurement
- Certification of SRMs 640c and 660a
- Difficulties in modeling due to unknown beam character



Character of parabolic graded multilayer mirrors in reciprocal space

- Character of mirror source is very different from bare anodes
  - **PGMs provide high reflectivity by diffraction**
  - **Diffraction implies dispersion**
- Wavelength/divergence character needed for data analysis





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Questions for using PGMs for x-ray metrology applications

- Many questions
  - **When is a PGM aligned “well”?**
  - **What do you need to do to align a PGM?**
    - Typical approach is purely intensity based
    - Does maximizing intensity uniquely specify wavelength/divergence character?
  - **How sensitive are PGMs to drift?**
    - Stability
    - Cold alignment
- The question: What is the wavelength/divergence character of the beam produced by PGM as alignment is varied?

The question for using PGMs for x-ray metrology

- What is the wavelength/divergence character of the beam produced by PGM as alignment is varied?
- Previous studies
  - **Most work on graded multilayer optics has concerned spatial characteristics (usually for a single wavelength)**
  - **Toraya and Hibino (2000) performed very useful single-crystal study of PGMs**
- How to answer the question?
  - **An instrument that can position a PGM in its six DOF and measure its wavelength/divergence character**
  - **Experiment designs to explore eight-dimensional space**

The NIST X-ray Optics Evaluation Double-Crystal Diffractometer

Instrument

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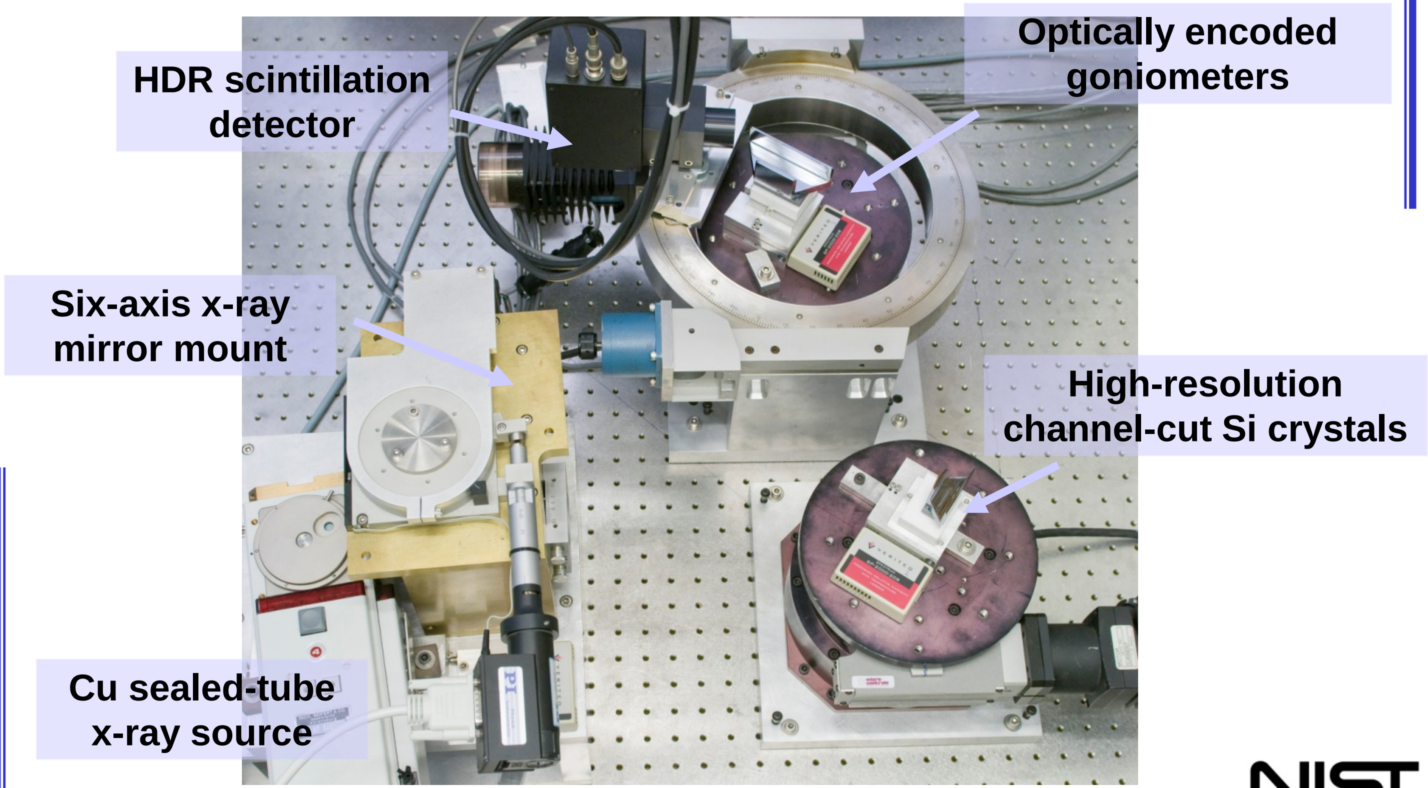
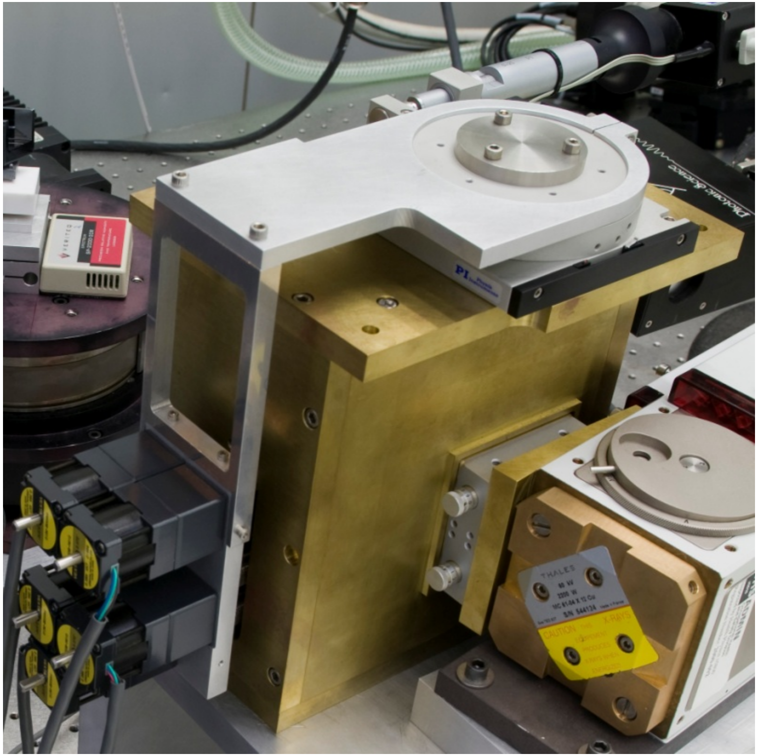
XOEDCD overview picture

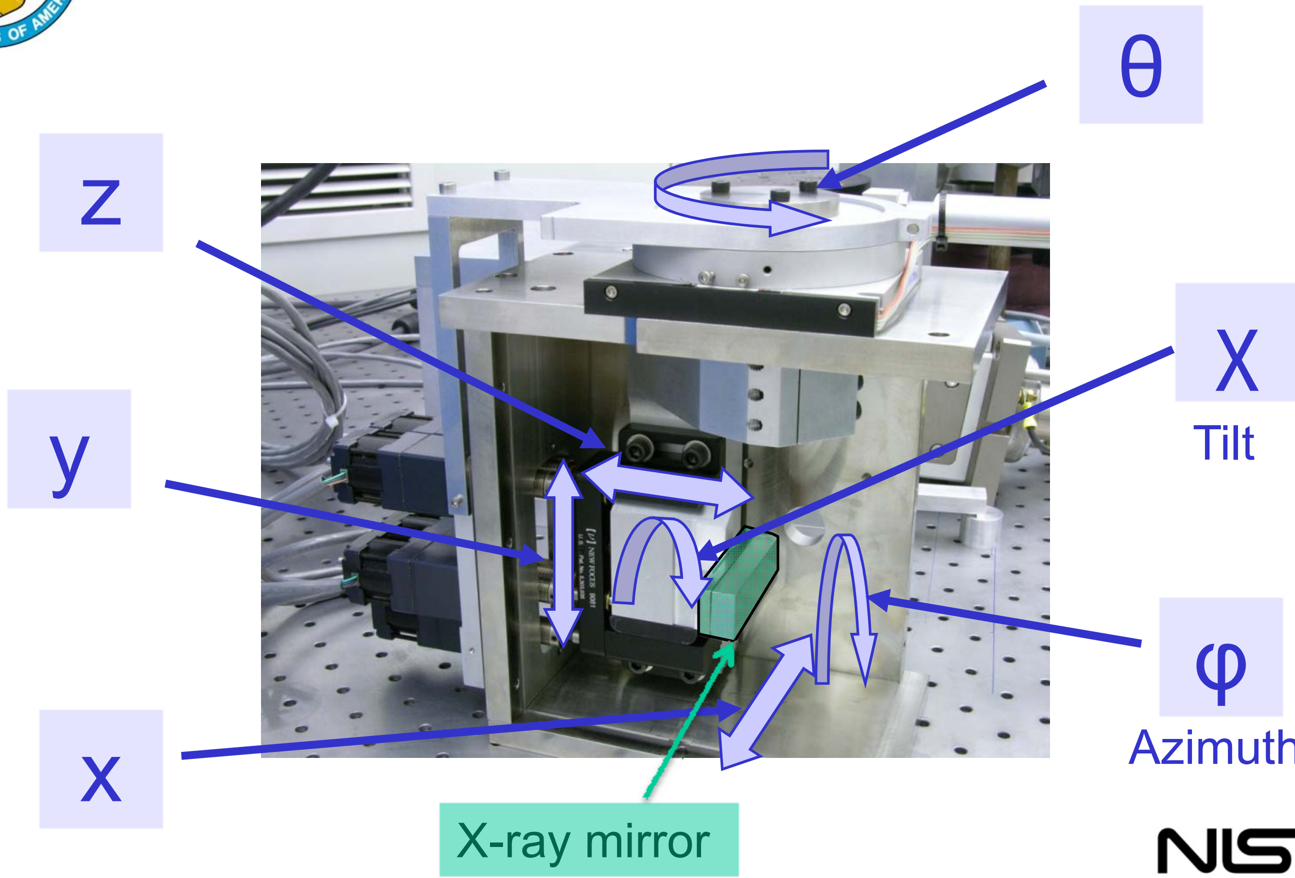


Six-axis mirror mount

- Stepper motor driven New Focus five-axis kinematic alignment stage
- Servo driven Physik Instrumente tangent arm
- Accuracy studied by autocollimator measurements



Six-axis stage directions of motion



Toraya and Hibino diagram

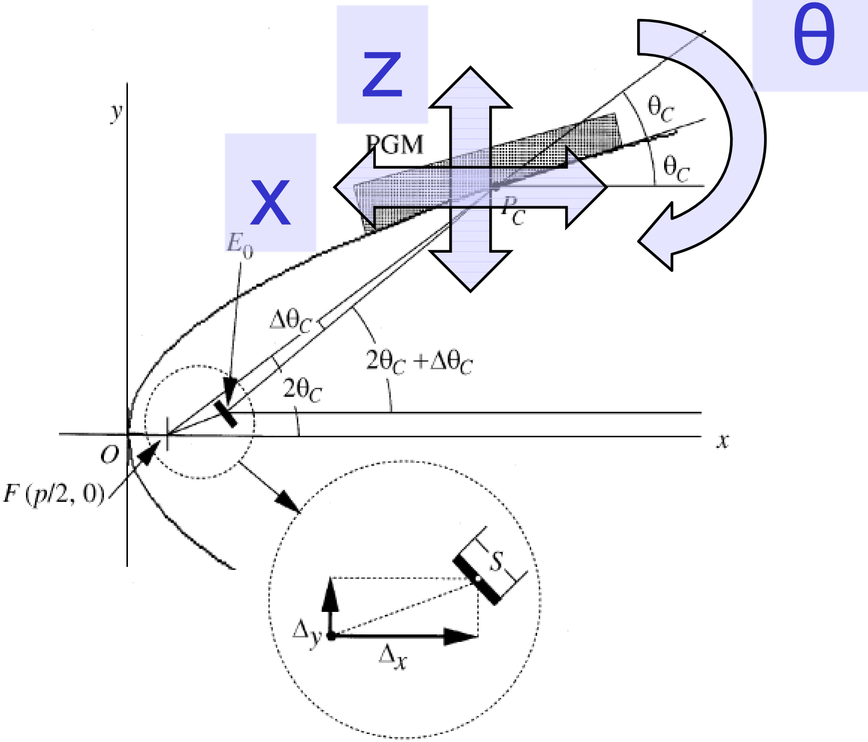


Figure 1
Geometrical arrangement of the X-ray focus and the parabolic graded multilayer (PGM).

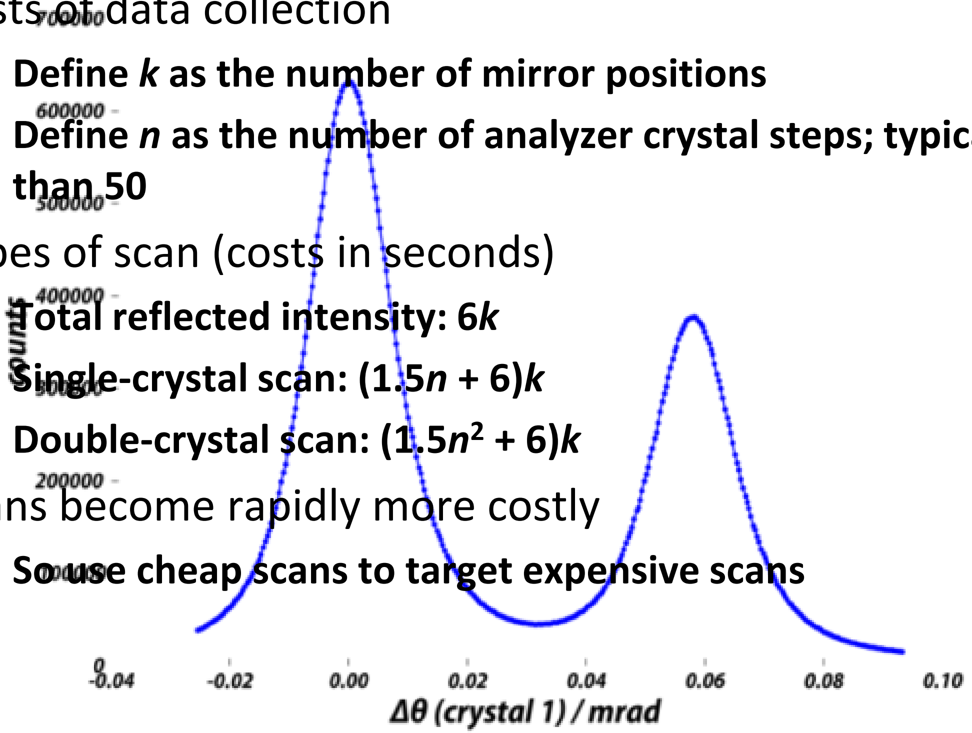
from Toraya and Hibino (2000), 1318

Approaches to exploring the six-dimensional mirror position space

Experiment Design

Approach to data collection

- Costs of data collection
  - **Define *k* as the number of mirror positions**
  - **Define *n* as the number of analyzer crystal steps; typically greater than 50**
- Types of scan (costs in seconds)
  - **Total reflected intensity: 6*k***
  - **Single-crystal scan: $(1.5n + 6)k$**
  - **Double-crystal scan: $(1.5n^2 + 6)k$**
- Scans become rapidly more costly
  - **So use cheap scans to target expensive scans**



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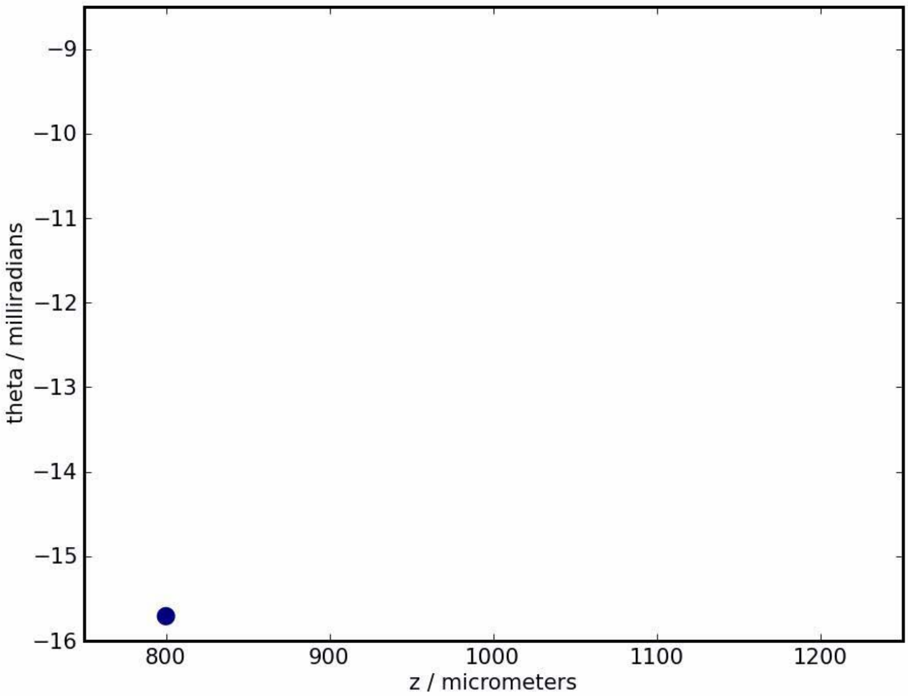
Experiment Designs

- Single-axis variation
  - **If effects of axes are completely uncorrelated, find maximum of intensity and scan each axis separately**
  - **But if there's correlation, this produces wrong estimates of effects**
- Factorial and fractional factorial designs
  - **Effective, easy to interpret conventional statistical designs**
  - **Typically only used for 2, occasionally 3 levels**
- Monte Carlo and Quasi-Monte Carlo methods

Monte Carlo and Quasi-Monte Carlo

- Monte Carlo
  - **Random sampling of space**
  - **Easy method: just pick uniformly distributed random deviates**
- Quasi-Monte Carlo
  - **Low-discrepancy random numbers: fills the space uniformly**
  - **Sequential: can terminate at any time and still have a ‘fair’ sample**

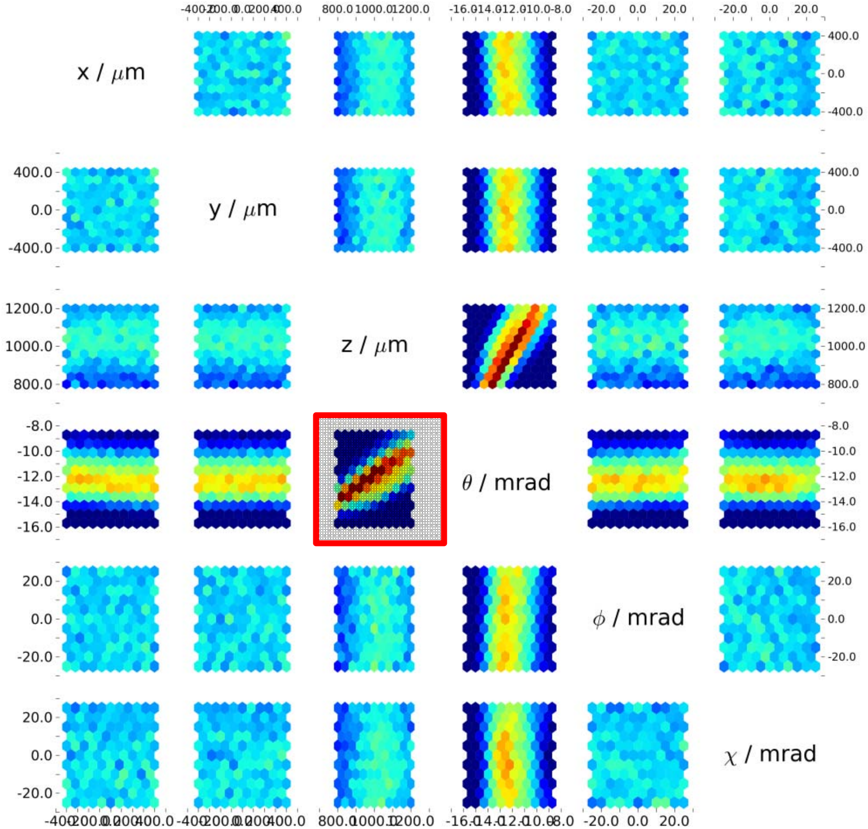
Theta angle versus z position scatter plot movie



Results from Quasi-Monte Carlo study of the variation of total reflected intensity with mirror position

Data

Two-dimensional hexagonal bin plots



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Toraya and Hibino: Theta angle and z position

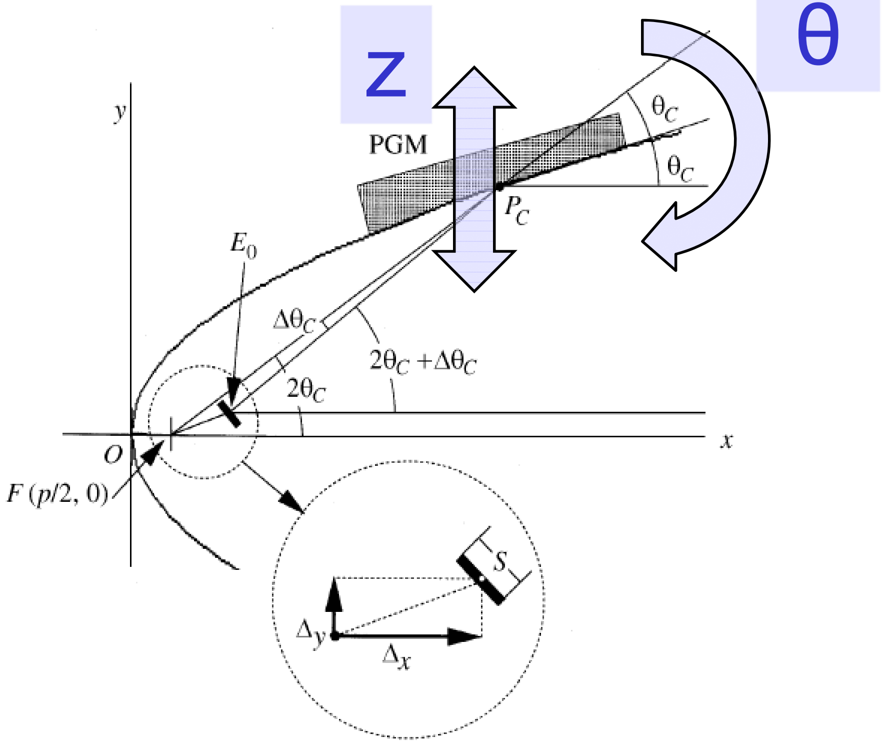
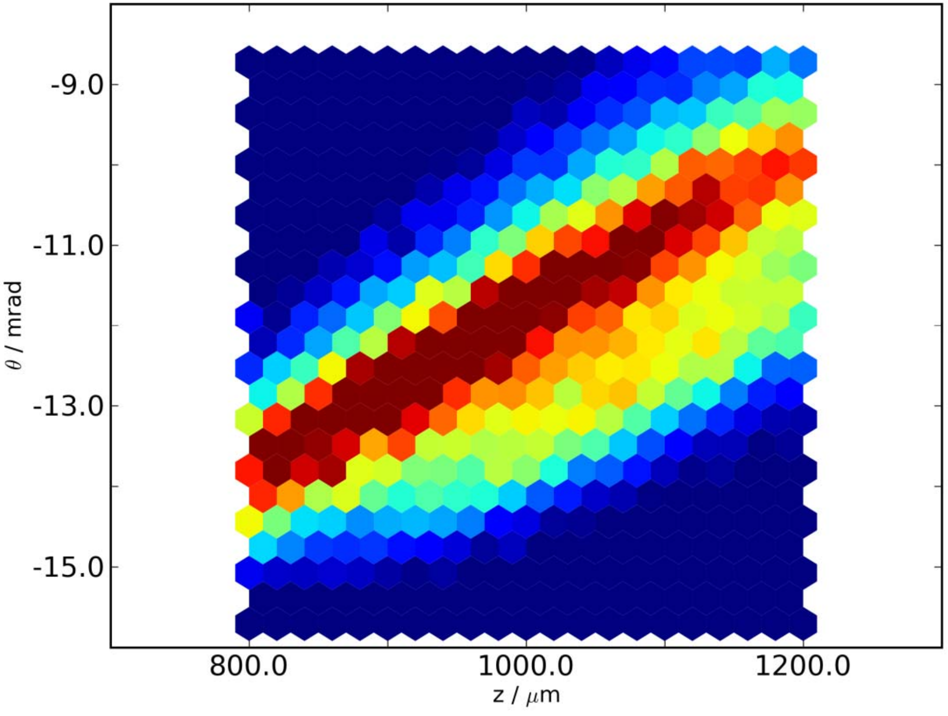


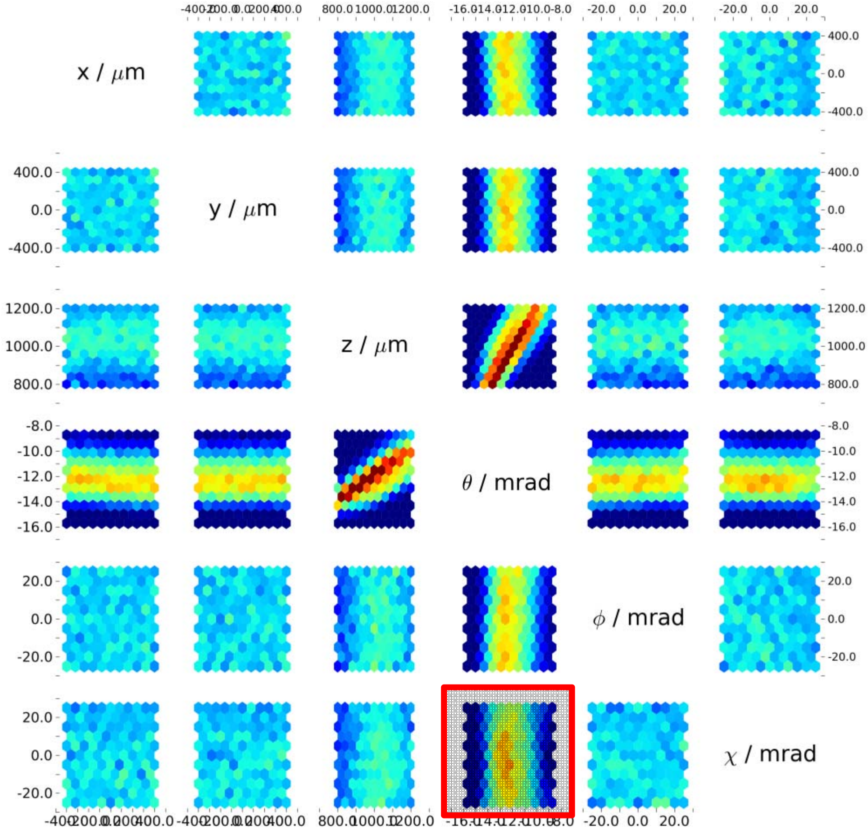
Figure 1
Geometrical arrangement of the X-ray focus and the parabolic graded multilayer (PGM).

from Toraya and Hibino (2000), 1318

Theta angle versus z position

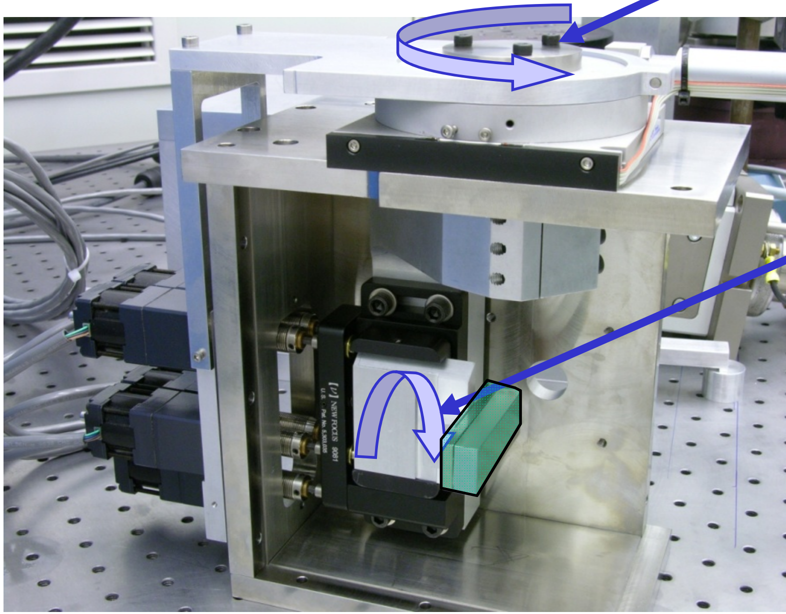


Two-dimensional hexagonal bin plots



Six-axis stage directions of motion: theta angle and chi angle

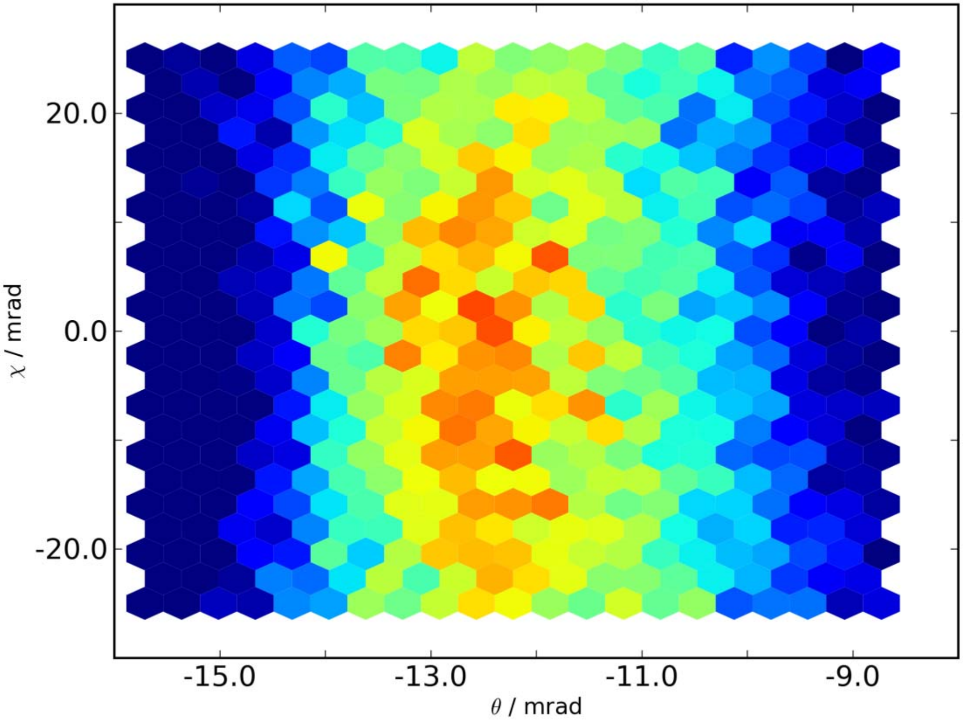
θ



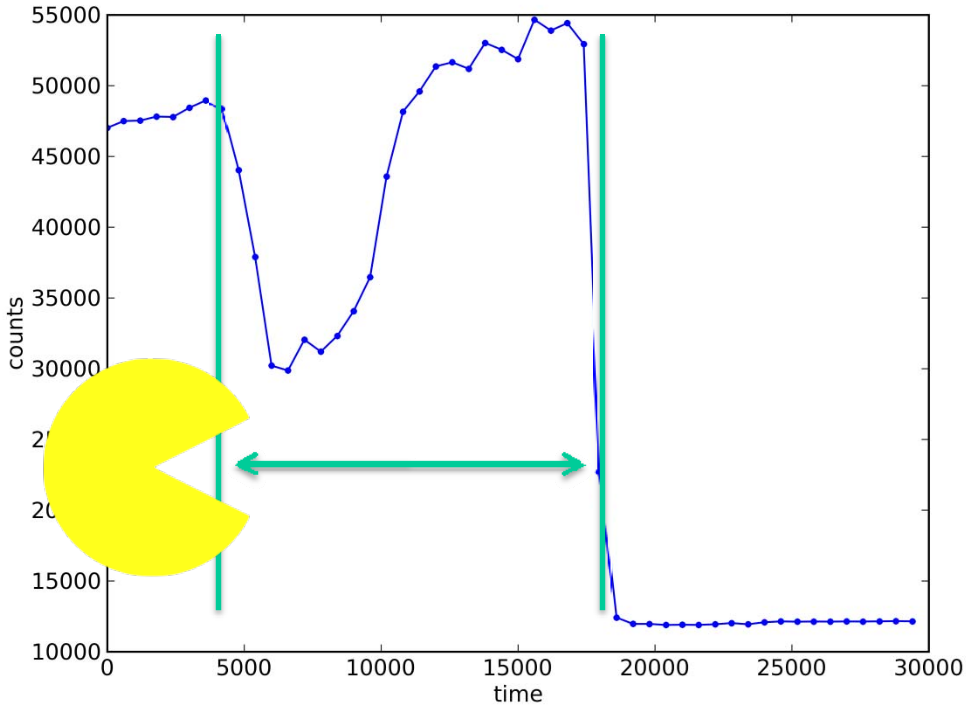
χ

Tilt

Chi angle versus theta angle



Counts over time



Aug. 2008 intensity collection

Reciprocal space maps of wavelength/divergence character

Double-crystal data

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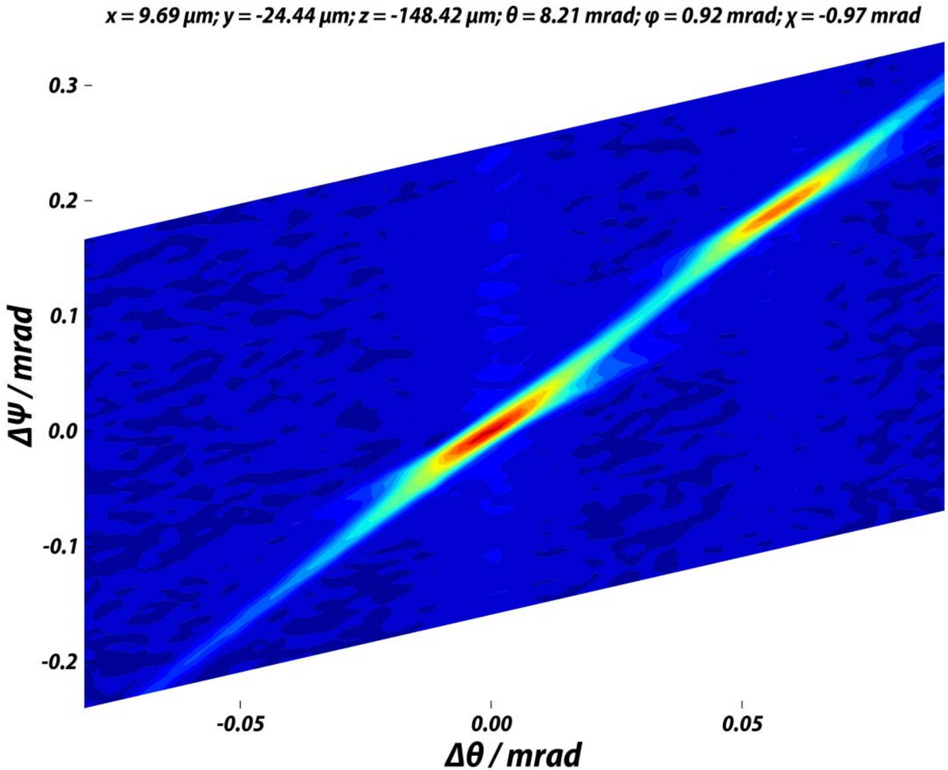
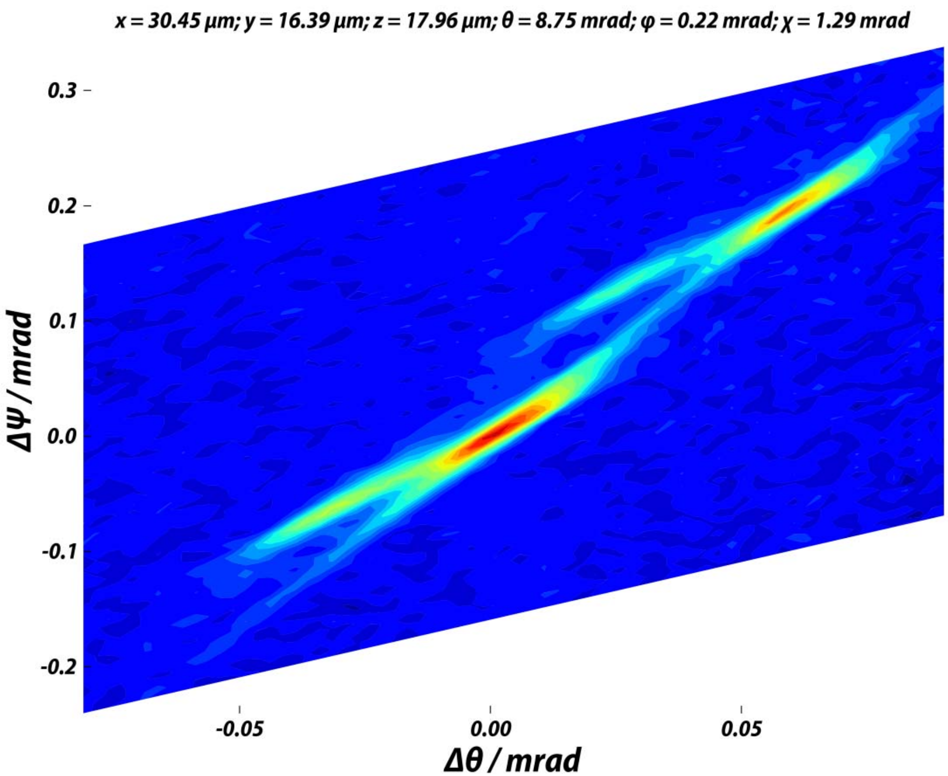
RSM: well-aligned



RSM: misaligned



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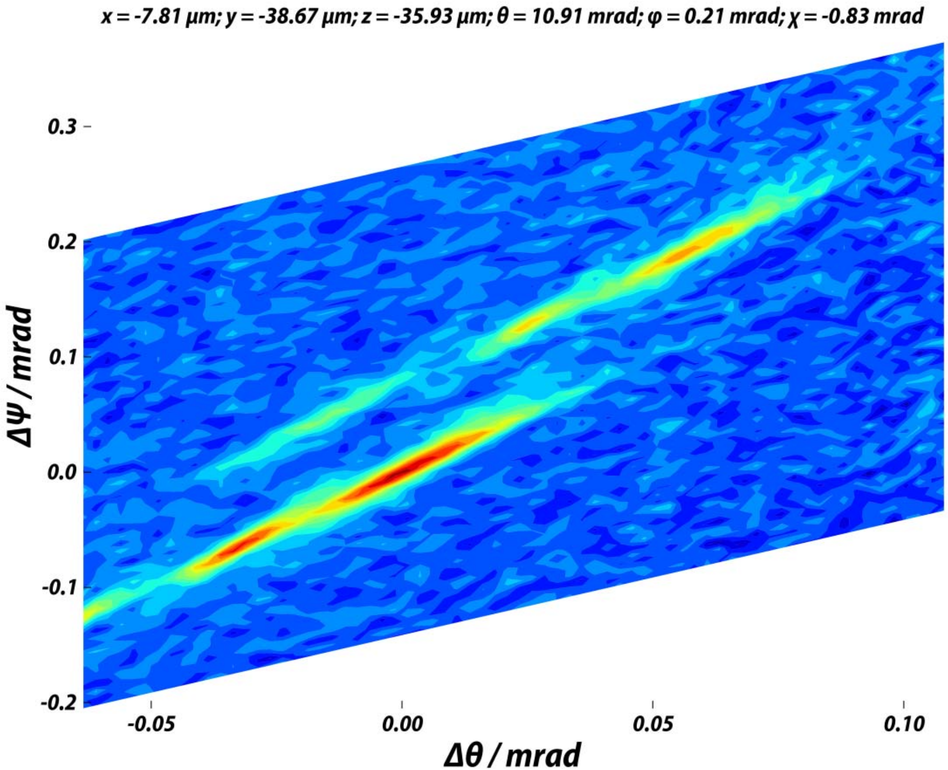
RSM: very misaligned



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Conclusions

Conclusions and plans

- Conclusions
  - **QMC is an easy way to sample a high-dimensional alignment space**
  - **Results can reveal correlations between parameters, or drift over time**
  - **Initial collections of double-crystal data show interesting structure**
- Future plans
  - **Collection of representative set of RSMs**
  - **Quantitative analysis of RSMs**

Contributions

- Donald Windover, co-leader of the XRM project, performed an initial demonstration of the instrument in 2003 and assisted in the alignment for the data presented here;
- Albert Henins designed and built the DCD, and cut the silicon channels;
- and Jim Cline, co-leader of the XRM project, developed the DCD strategy in collaboration with Dick Deslattes.

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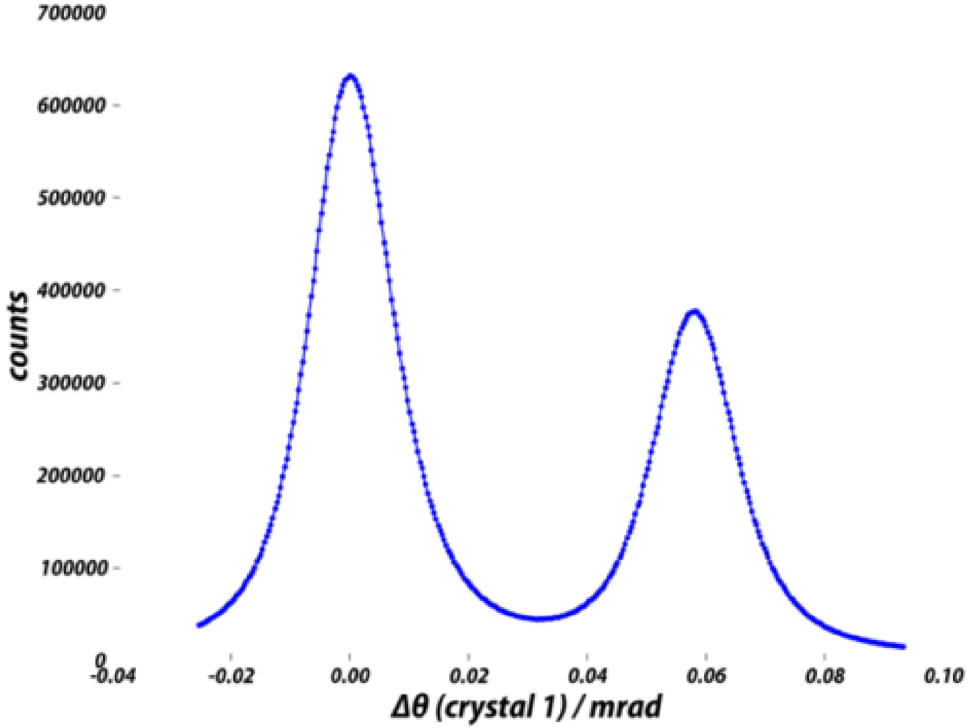
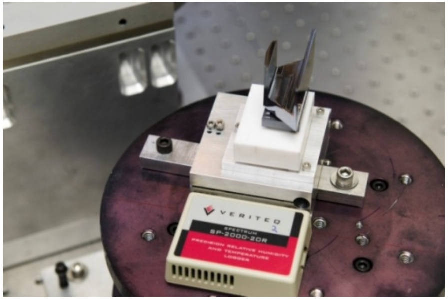
Supplemental Material

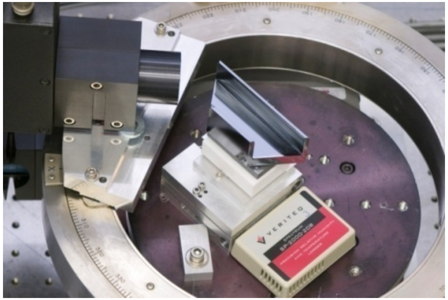
Single crystal rocking curve of first monochromator crystal



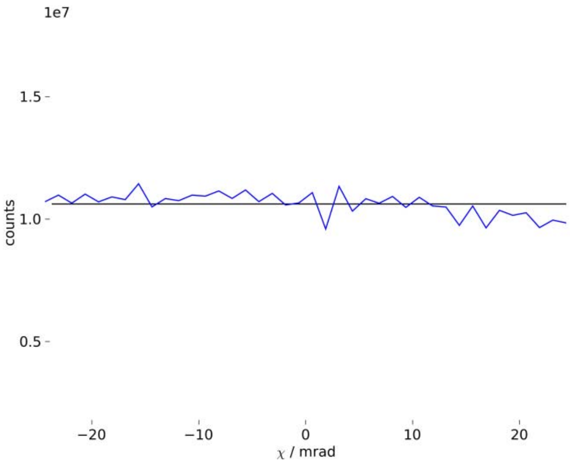
Metrological aspects of high-resolution double crystal spectrometry

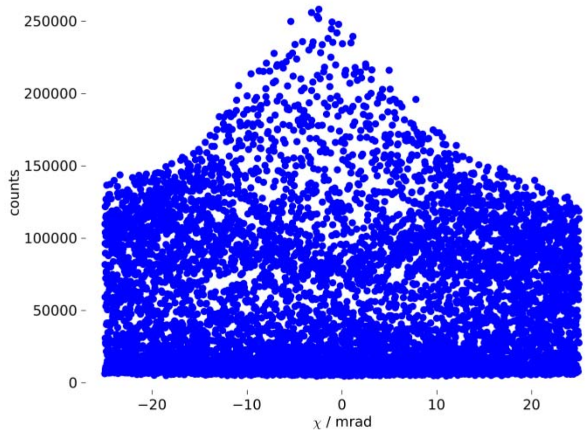
- Goniometers
  - **Micro-Controle stages driven by harmonic drive steppers**
  - **Heidenhain 800 optical encoders**
- Crystals
  - **From 640d boules: d-spacing known**
  - **Si (440) three-bounce channels**
    - High resolution, narrow tails





Chi angle: scatter plot versus weighted histogram plot





Powder diffraction SRMs planned for recertification on CDPBD

Diffraction Application	SRM Number	Composition
Line Position	640d	Silicon Powder
Line Shape	660b	LaB_6 Powder
Instrument Response	1976a	Sintered Alumina Plate
Quantitative Analysis	676a	Alumina (corundum) Powder

Factorial and fractional factorial experiment designs

- Factorial and fractional factorial designs
  - **Effective, easy to interpret conventional statistical designs**
  - **Typically only used for 2, occasionally 3 levels**
- Factorial designs are just multi-dimensional grids

2^3 factorial design:

$2^{(3-1)}$ fractional factorial design:

X1	X2	X3
-1	-1	-1
+1	-1	-1
-1	+1	-1
+1	+1	-1
-1	-1	+1
-1	+1	+1
+1	+1	+1

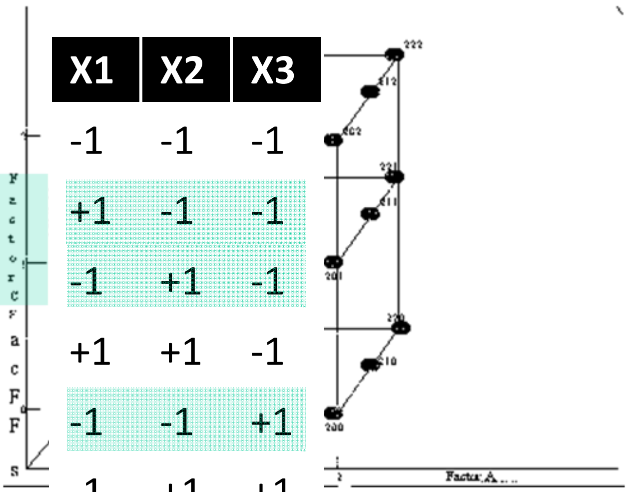


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Diagram of old NIST Parallel Beam Powder Diffractometer

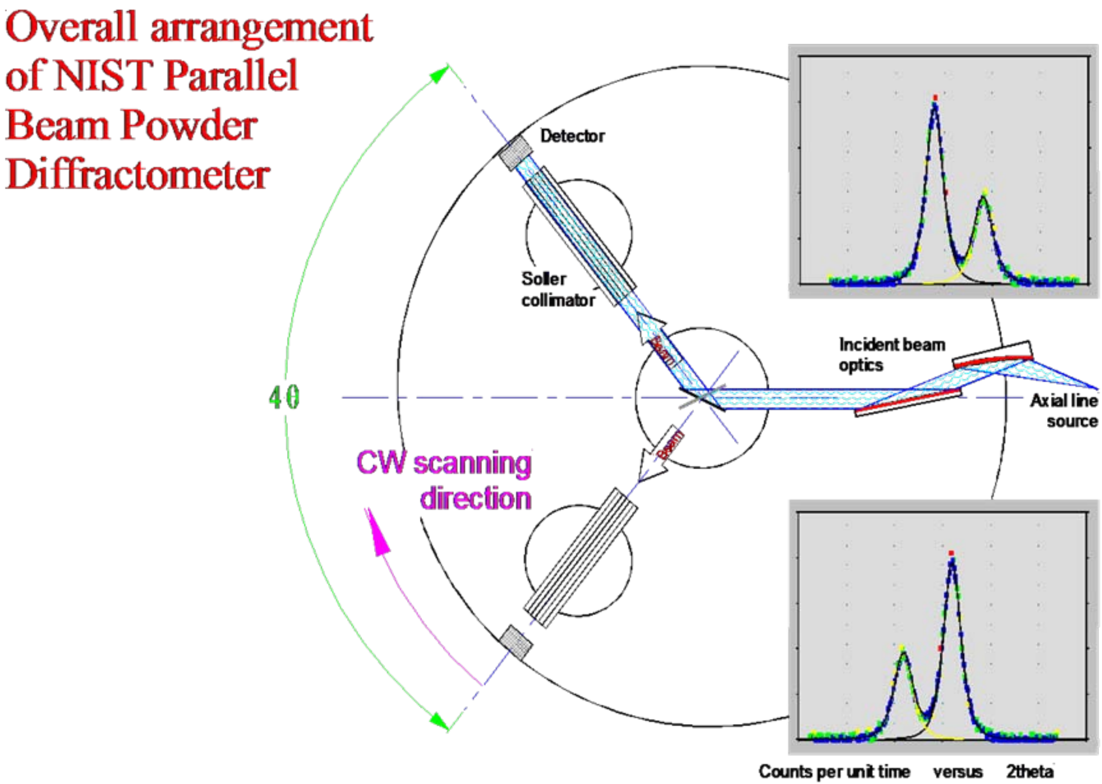

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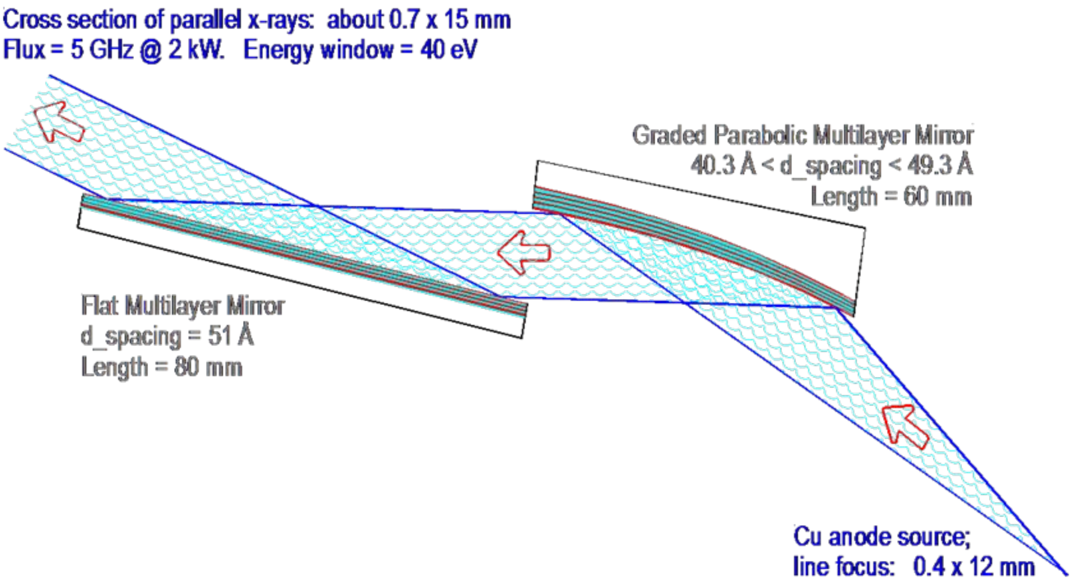
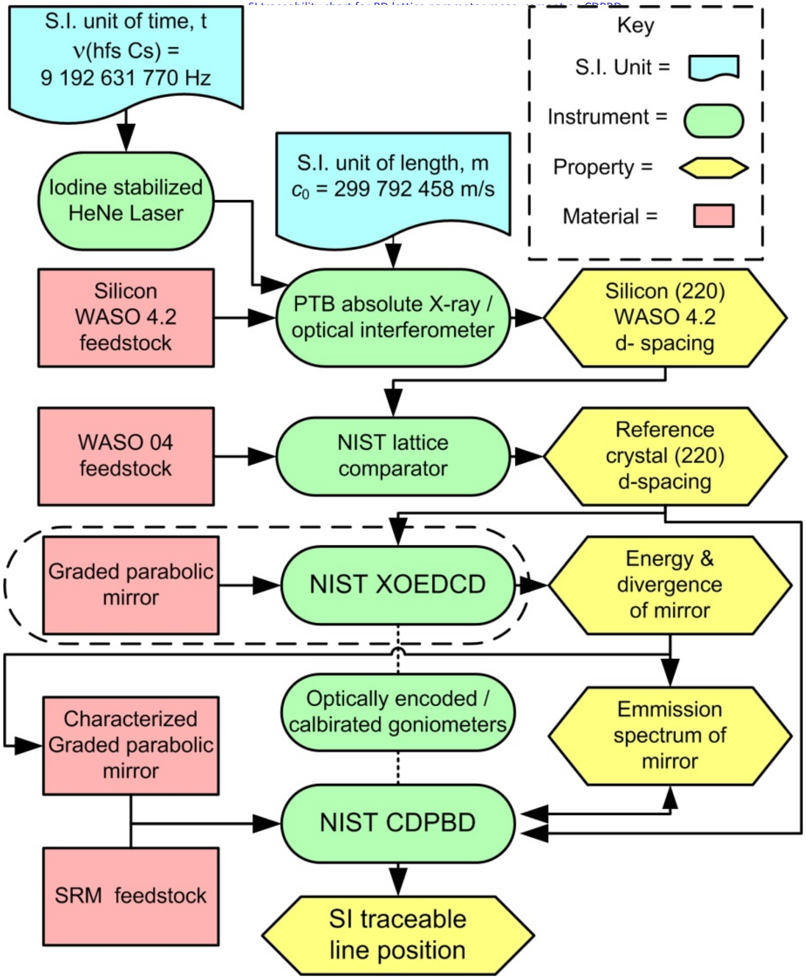
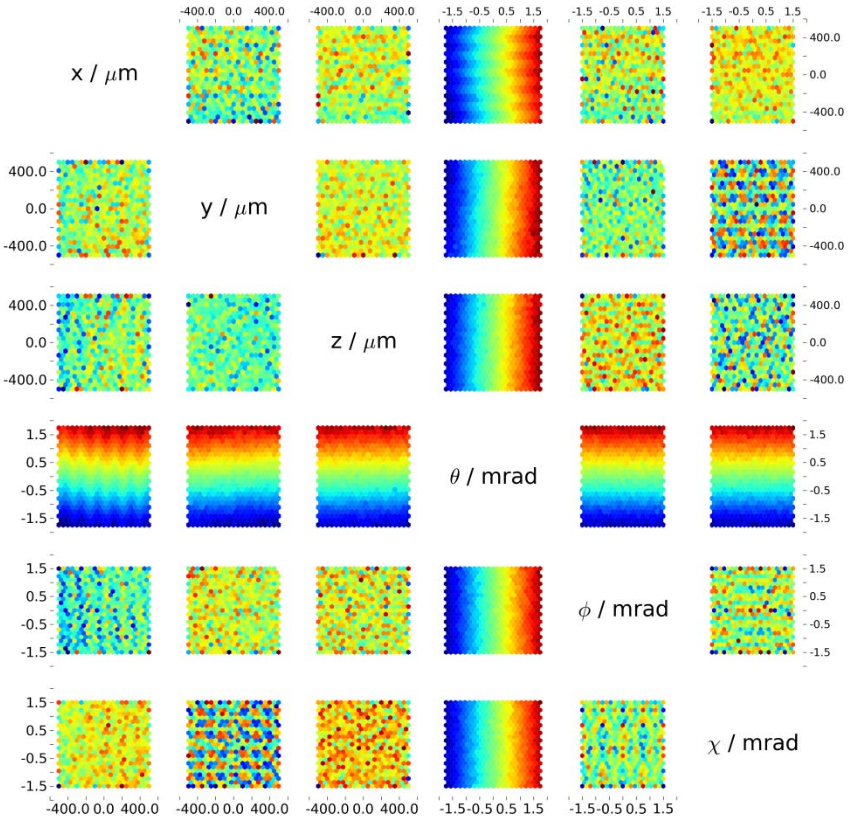
Diagram of old NIST Parallel Beam Powder Diffractometer primary x-ray beam optics

Primary X-ray Beam Optics for NIST Parallel Beam Powder Diffractometer




S.I. unit of time, t
ν(hfs Cs) =
9 192 631 770 Hz
Key
S.I. Unit =
Instrument =
Property =
Material =
Iodine stabilized HeNe Laser
S.I. unit of length, m
c_0 = 299 792 458 m/s
Silicon WASO 4.2 feedstock
PTB absolute X-ray / optical interferometer
Silicon (220) WASO 4.2 d- spacing
WASO 04 feedstock
NIST lattice comparator
Reference crystal (220) d-spacing
Graded parabolic mirror
NIST XOEDCD
Energy & divergence of mirror
Characterized Graded parabolic mirror
Optically encoded / calibrated goniometers
Emmission spectrum of mirror
NIST CDPBD
SRM feedstock
SI traceable line position

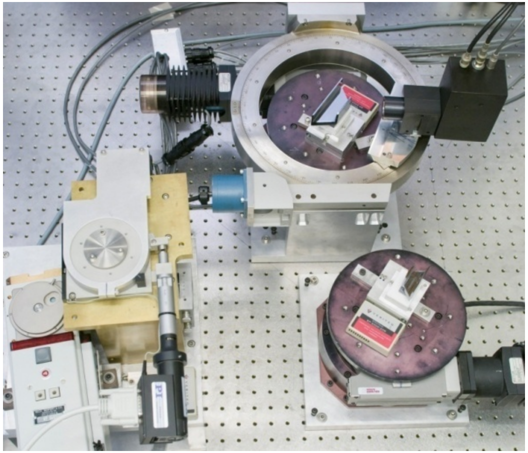
Example autocollimator data

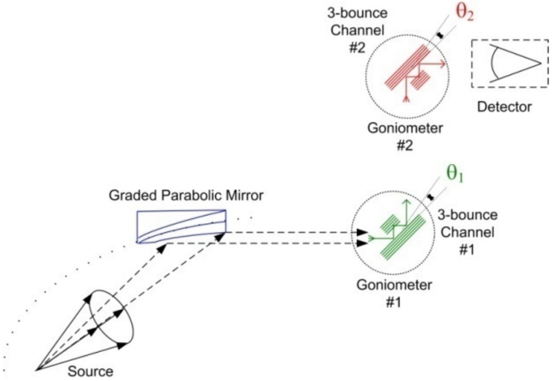


NIST CDPBD

- Rigorously characterized Heidenhain 905s
- Spherical air bearing for alignment
- In AML +-0.01 C space
- Multiple configurations
  - **HRXRD**
  - **XRR**
  - **PD**

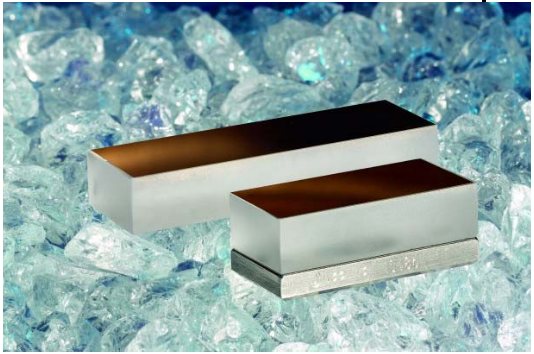
XOEDCD, non-dispersive configuration

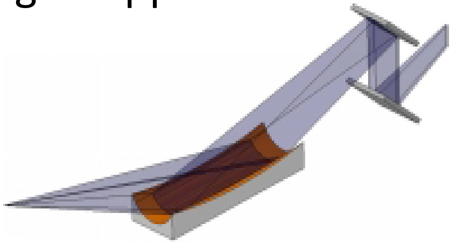




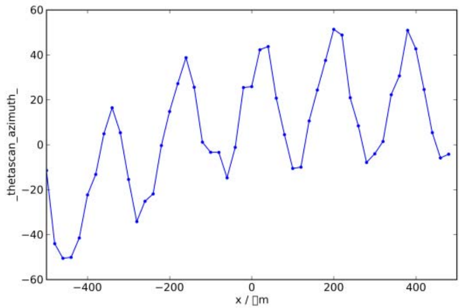
Graded multilayer x-ray optics

- Shaped substrates deposited with periodic multilayer of varying d-spacing for high reflectivity
- Becoming ubiquitous for laboratory x-ray instruments
- Improved source quality over other optics options
  - **Customized divergence and source size**
  - **Somewhat monochromated beam ($\Delta d/d \sim 10\%$)**
- Two-dimensional parabola for beam collimated in one axis
- Three-dimensional optics beginning to appear





Images from http://www.incoatec.de/GoebelMirrors_36.html and http://www.xenocs.com/range-2D-diffraction-optics.htm



60
40
20
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−60
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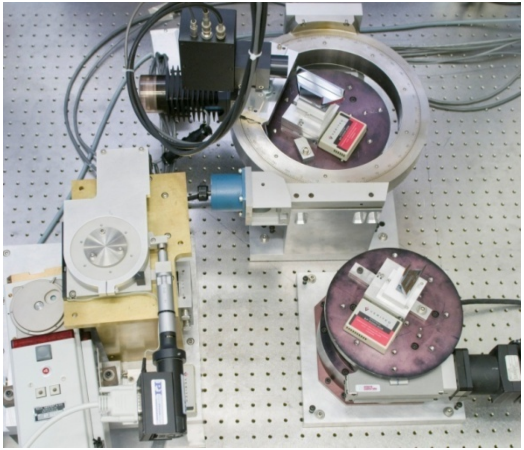
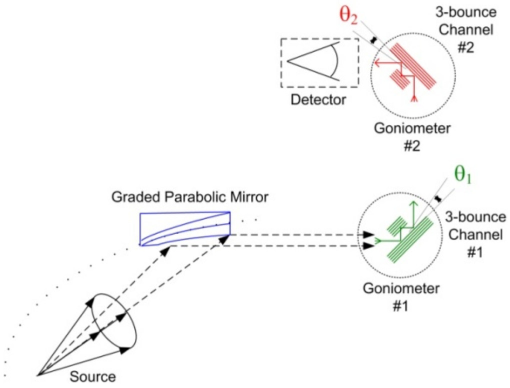
−400
−200
0
200
400
x / m

XOEDCD, dispersive configuration





Parabolic graded multilayer x-ray optics for powder diffraction

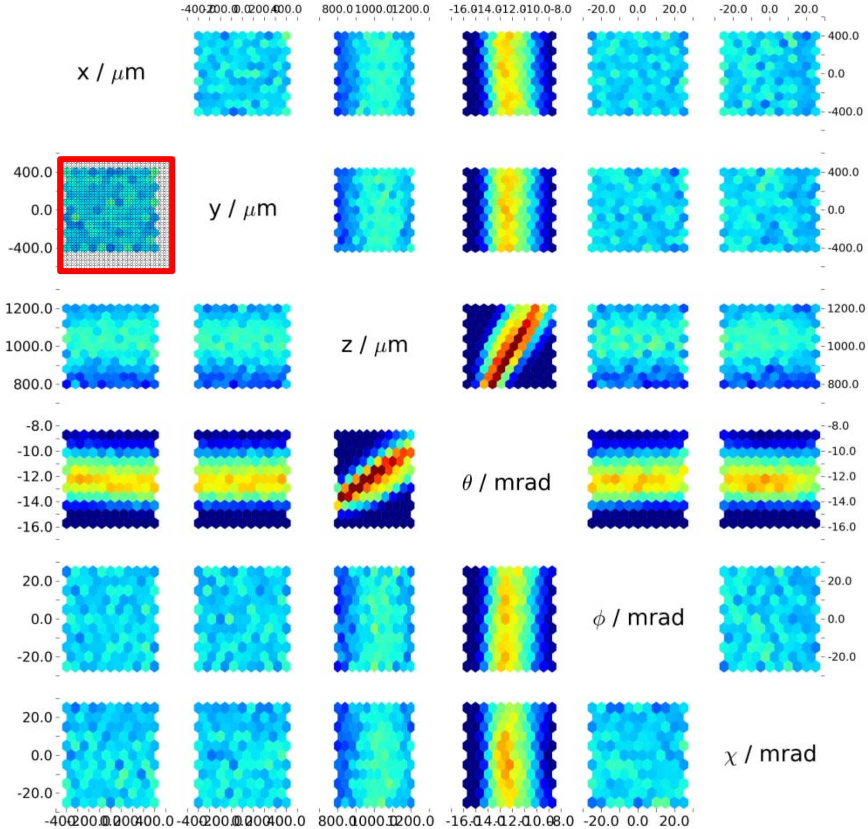
- Conventional powder diffraction optics: focusing
- Parabolic graded multilayers
  - **Low divergence**
  - **Higher resolution**

Study of the variation of total reflected intensity with mirror position

- Detector placed directly in front of six-axis mirror housing
  - **Source at @??**
  - **Attenuating foil used to bring counts down into range of detector**

@ Picture of detector in front of mirror housing

Two-dimensional hexagonal bin plots



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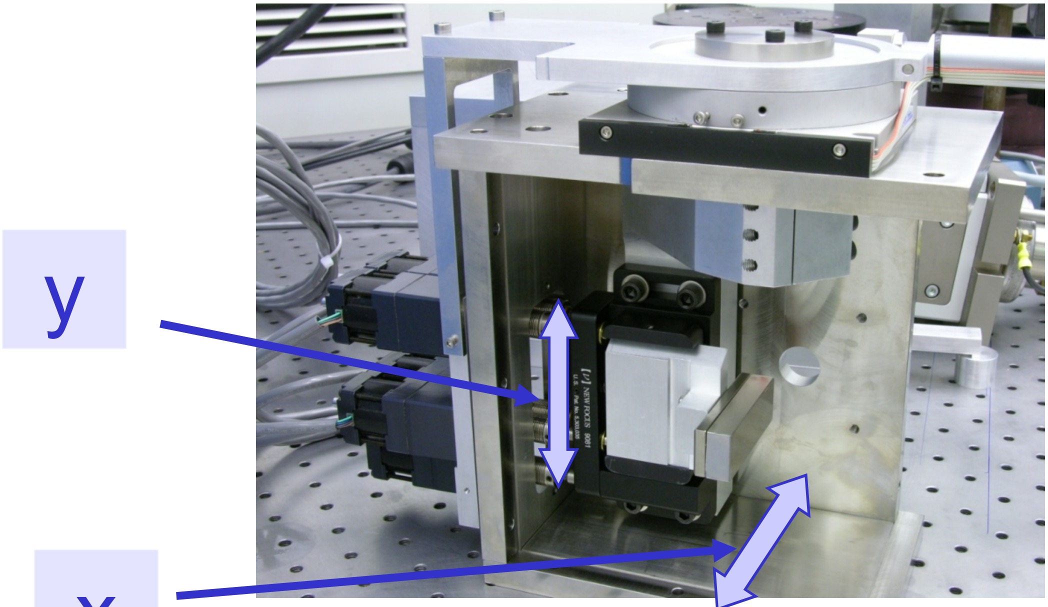
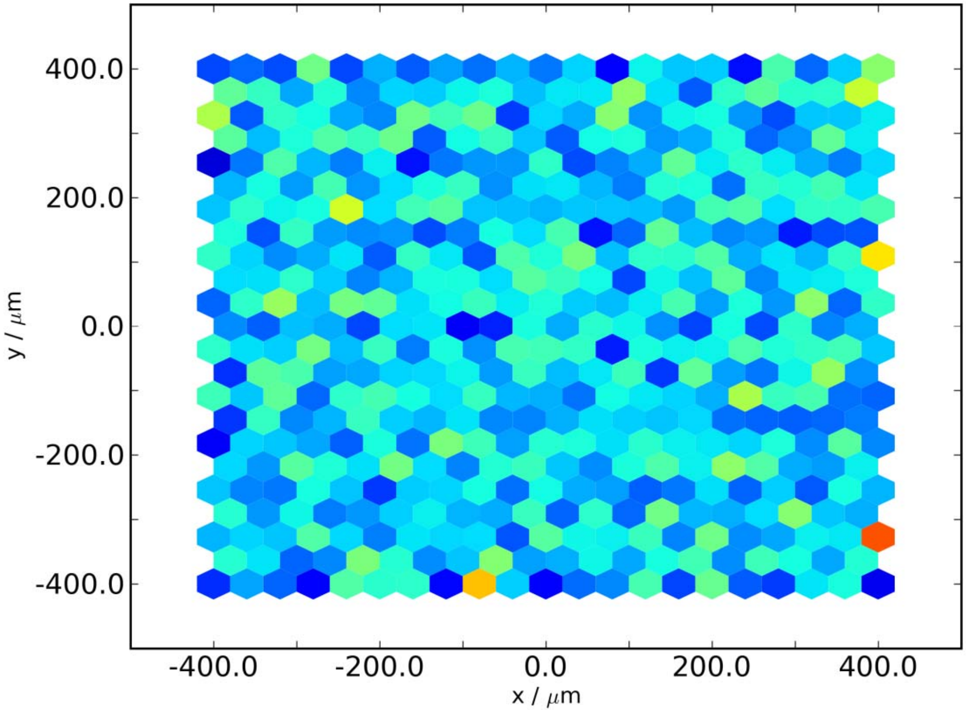
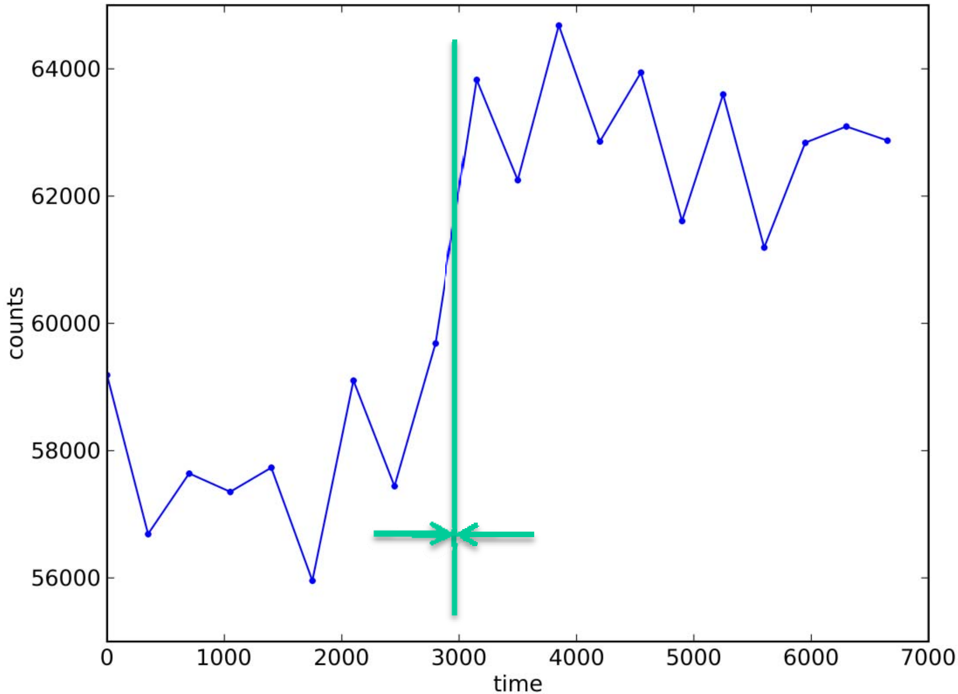
Six-axis stage directions of motion: x position and y position



x position versus y position



Counts over time



Feb. 2009 intensity collection