Development of Diffraction Imaging Methods for Materials Science at NSLS-II

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U.S. DEPARTMENT OF ENERGY

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BROOKHAVEN NATIONAL LABORATORY

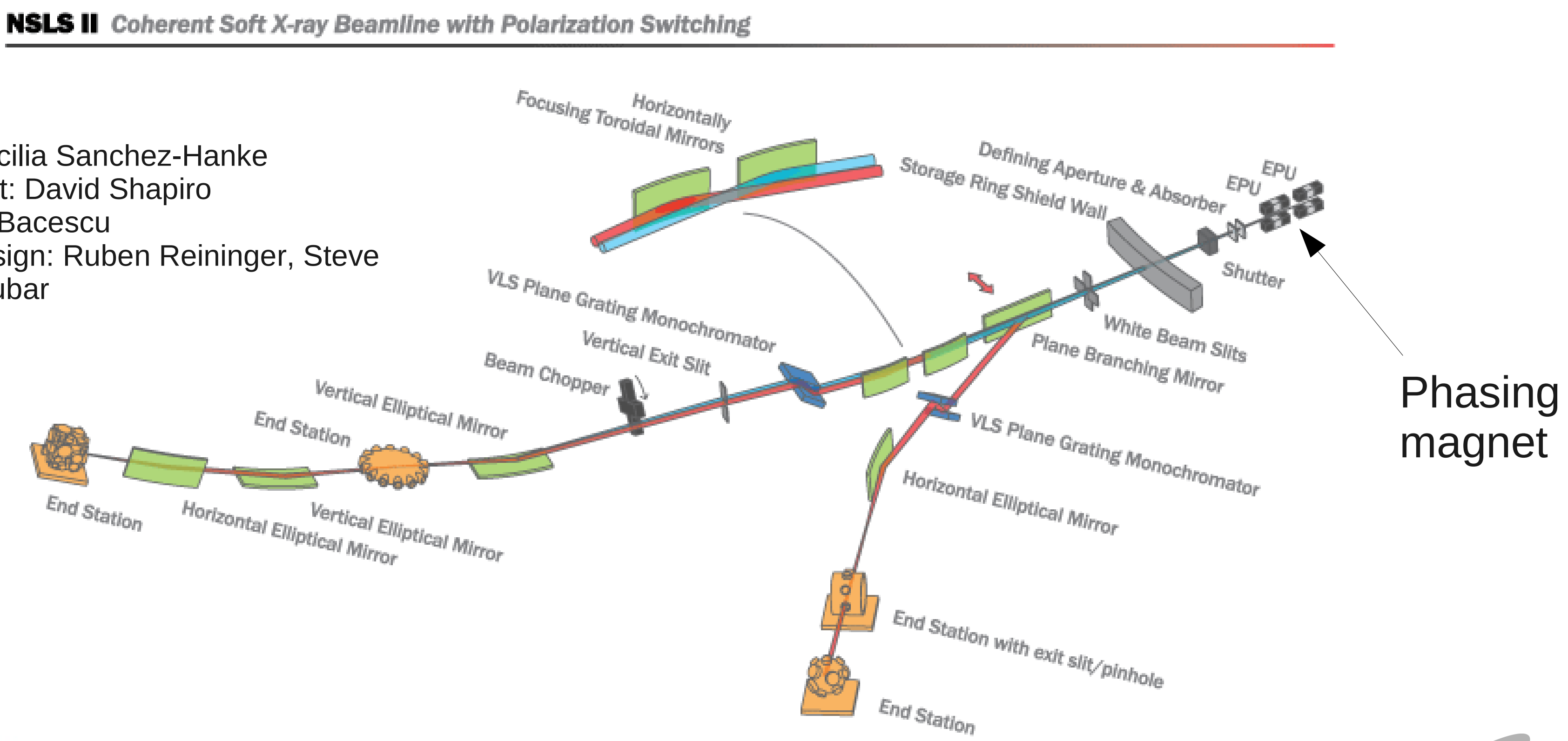
Outline

- The Coherent Soft X-ray Beamline at NSLS-II
- New developments in CDI
- Soft x-ray ptychography at the Canadian Light Source

NSLS-II: Coherent Soft X-ray Beamline

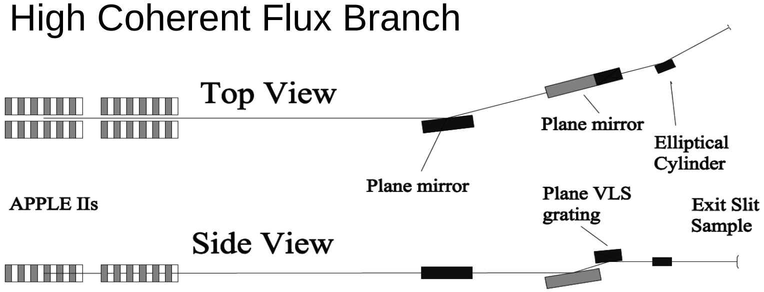
- NSLS-II > half way done
- Beneficial occupancy of first pentant
- 6 “project” beamlines + 10 others day 1
- CSX has 2 branches: full polarization control and high coherent flux
- Focus on magnetic and strongly correlated systems
- Optics procurement under way

Group leader: Cecilia Sanchez-Hanke
Beamline scientist: David Shapiro
Engineer: Daniel Bacescu
Optics/source design: Ruben Reininger, Steve Hulbert, Oleg Chubar

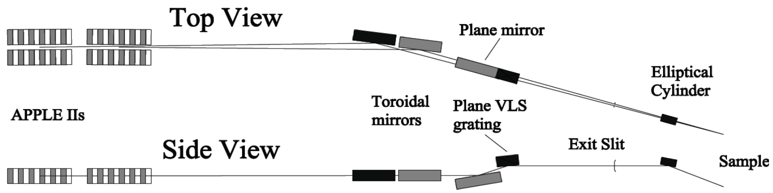


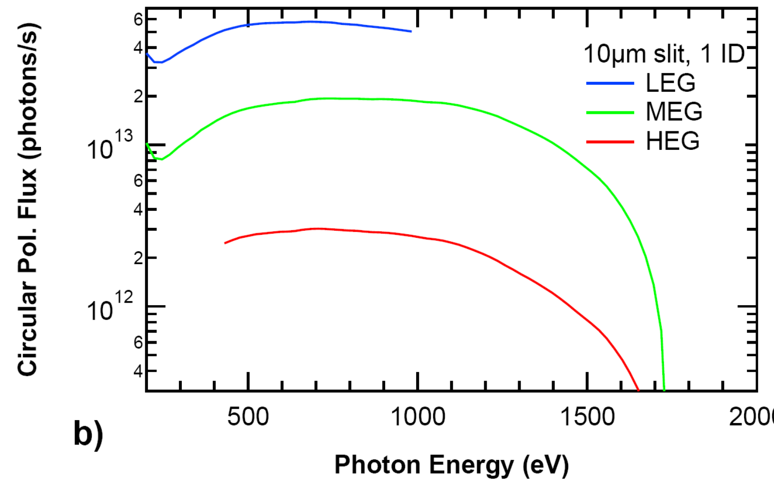
Very high flux enables difficult science

High Coherent Flux Branch

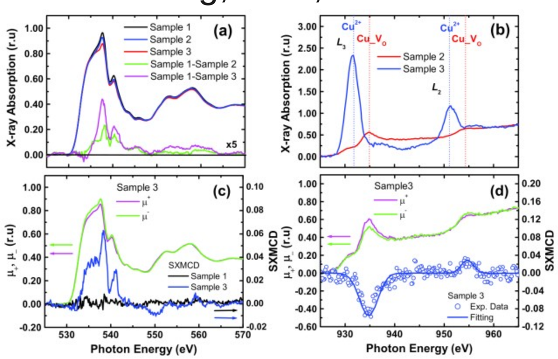


Polarization Control Branch



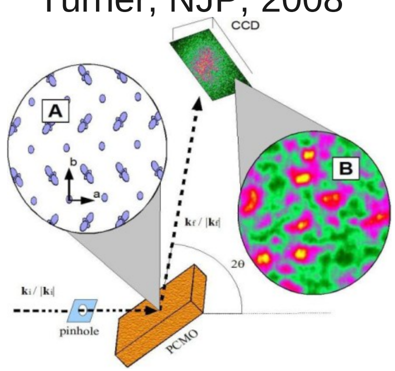


Herng, PRL, 2010

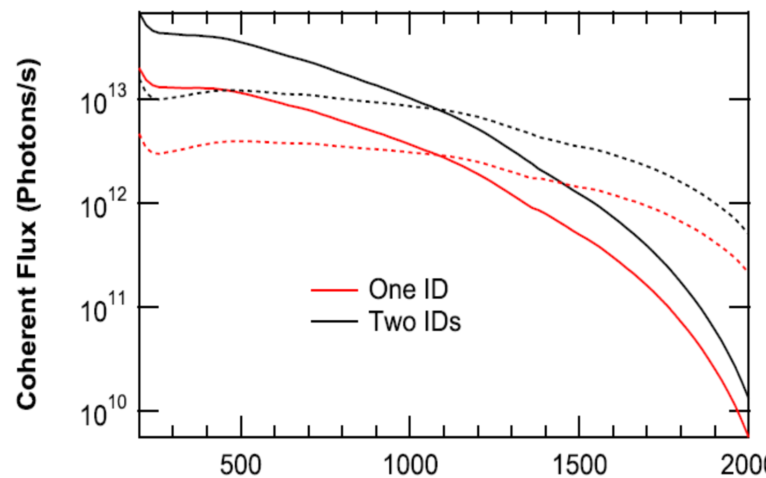


XMCD to probe ferromagnetism in diluted magnetic semiconductors

Turner, NJP, 2008



Coherent diffraction imaging of electronic textures in correlated materials



Ruben Reininger, BNL

TARDIS diffractometer, see talk by S. Wilkins, BNL

More bang for your buck

For a finite aperture, the resonant energy doesn't have the highest brightness

SRW simulated beamline

Resonant energy: 250 eV
Peak brightness: 248 eV

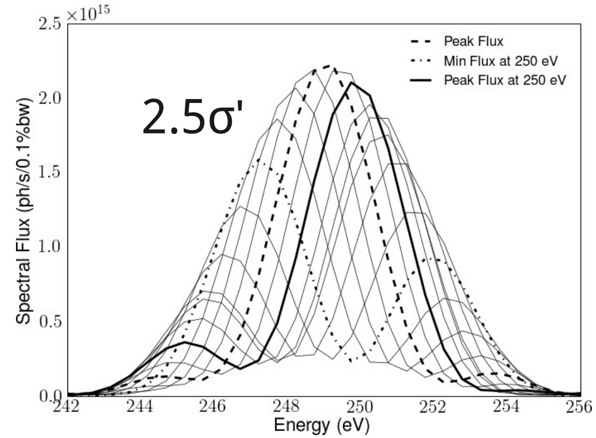
CSX can exploit this by tuning the phasing magnet

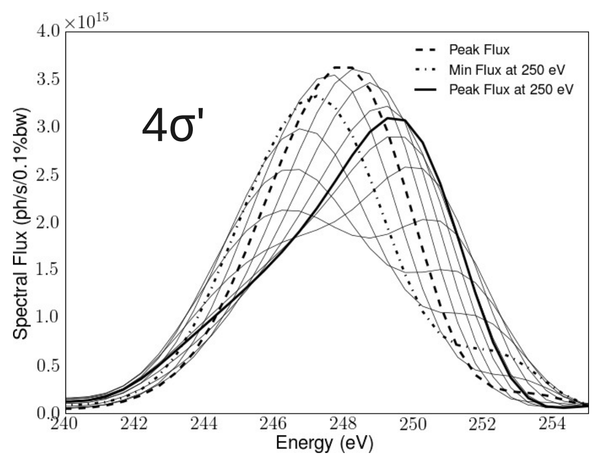
COSMIC can exploit this by detuning the EPU

CSX can accept 4σ'
Limited by shared operation with polarization branch

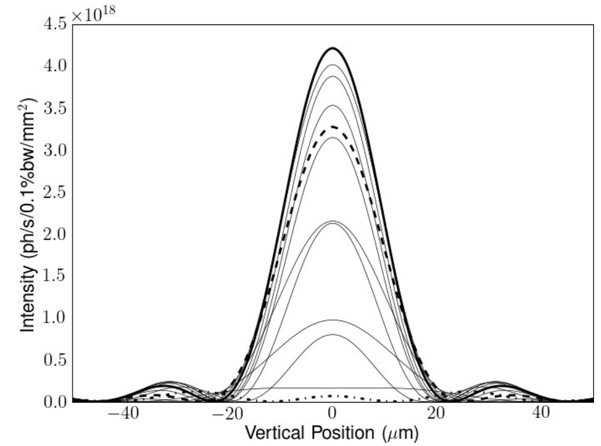
$$\sigma' = \sqrt{\lambda/2L}$$

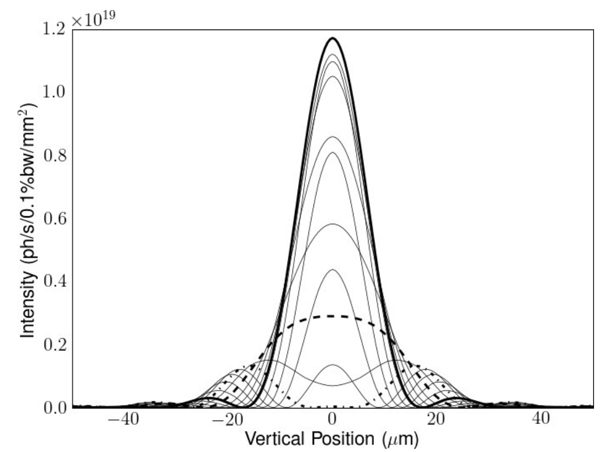
Flux through an aperture





250 eV source image

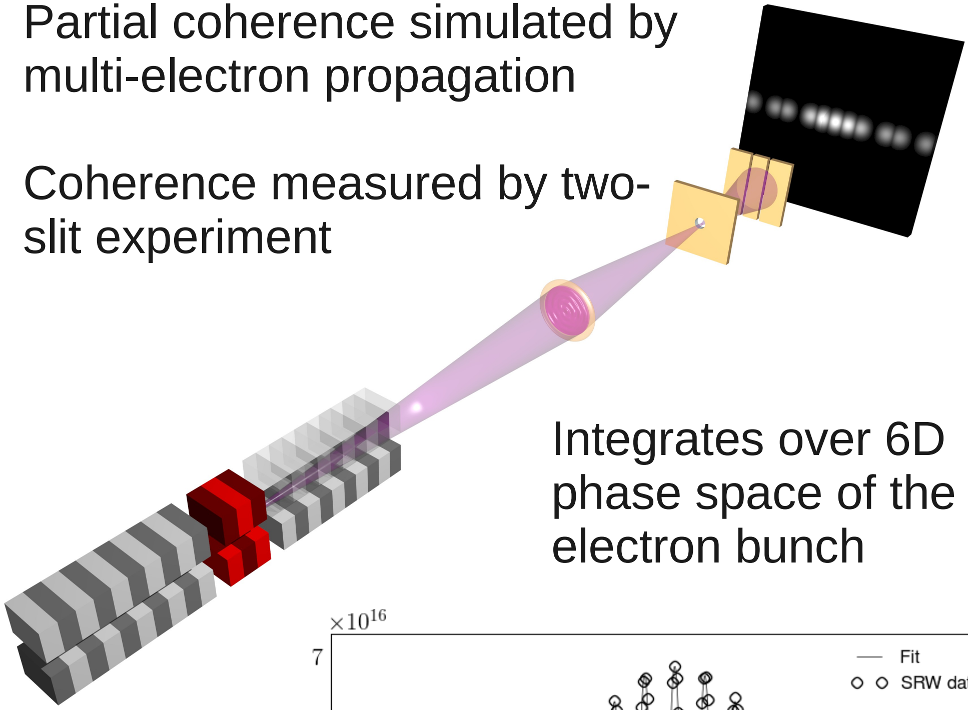




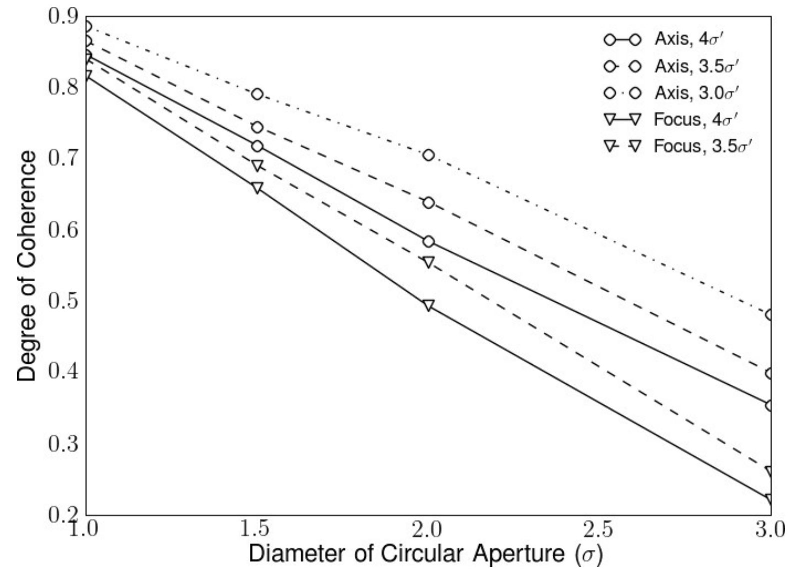
Large aperture: high flux, low coherence

Partial coherence simulated by multi-electron propagation

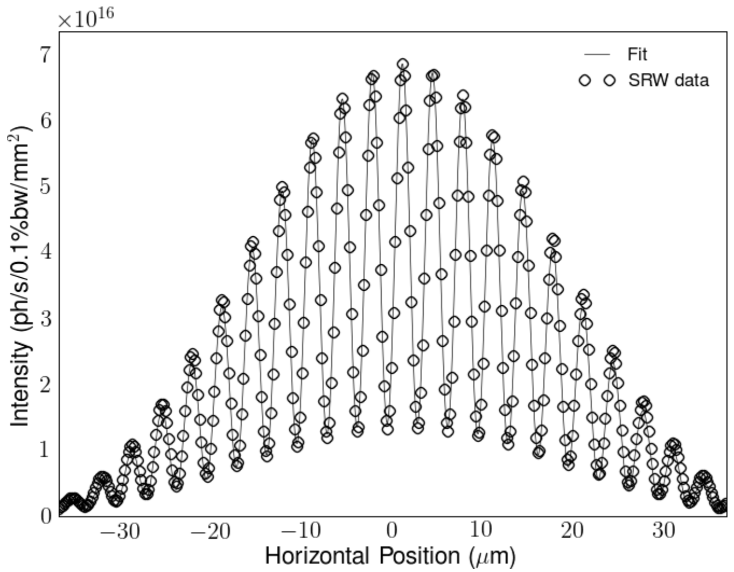
Coherence measured by two-slit experiment

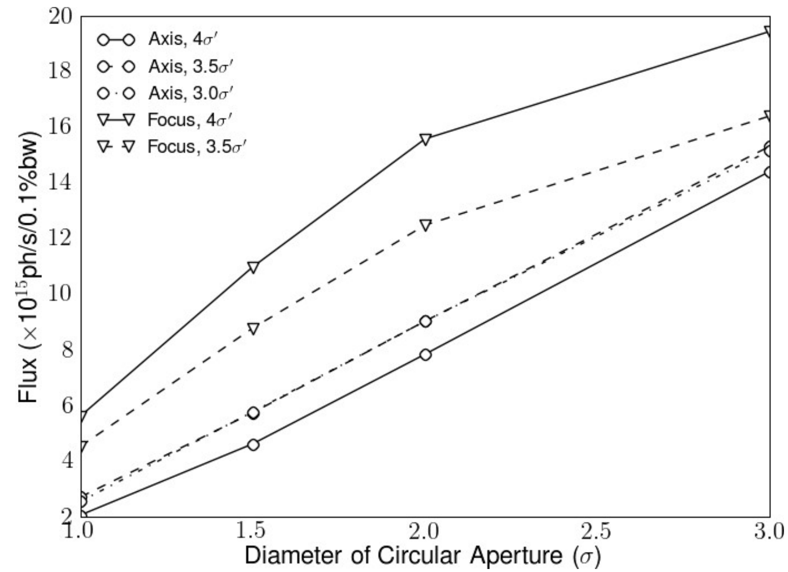


Integrates over 6D phase space of the electron bunch



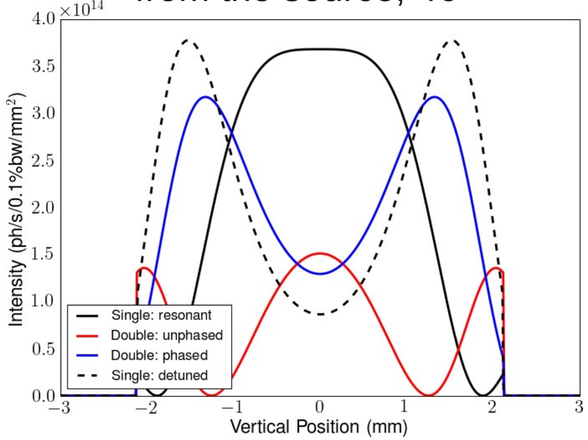
$$\sigma = \frac{1}{2\pi}\sqrt{2L\lambda}$$



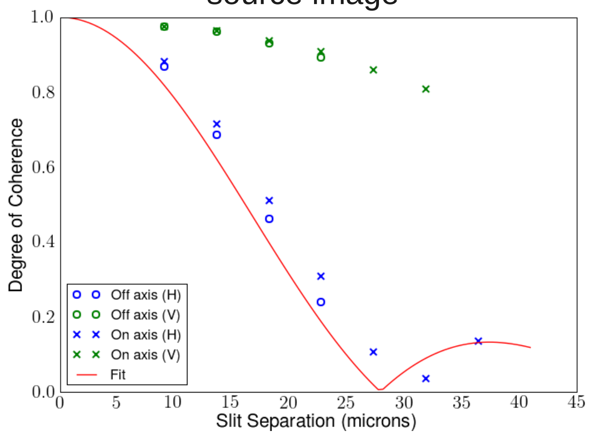


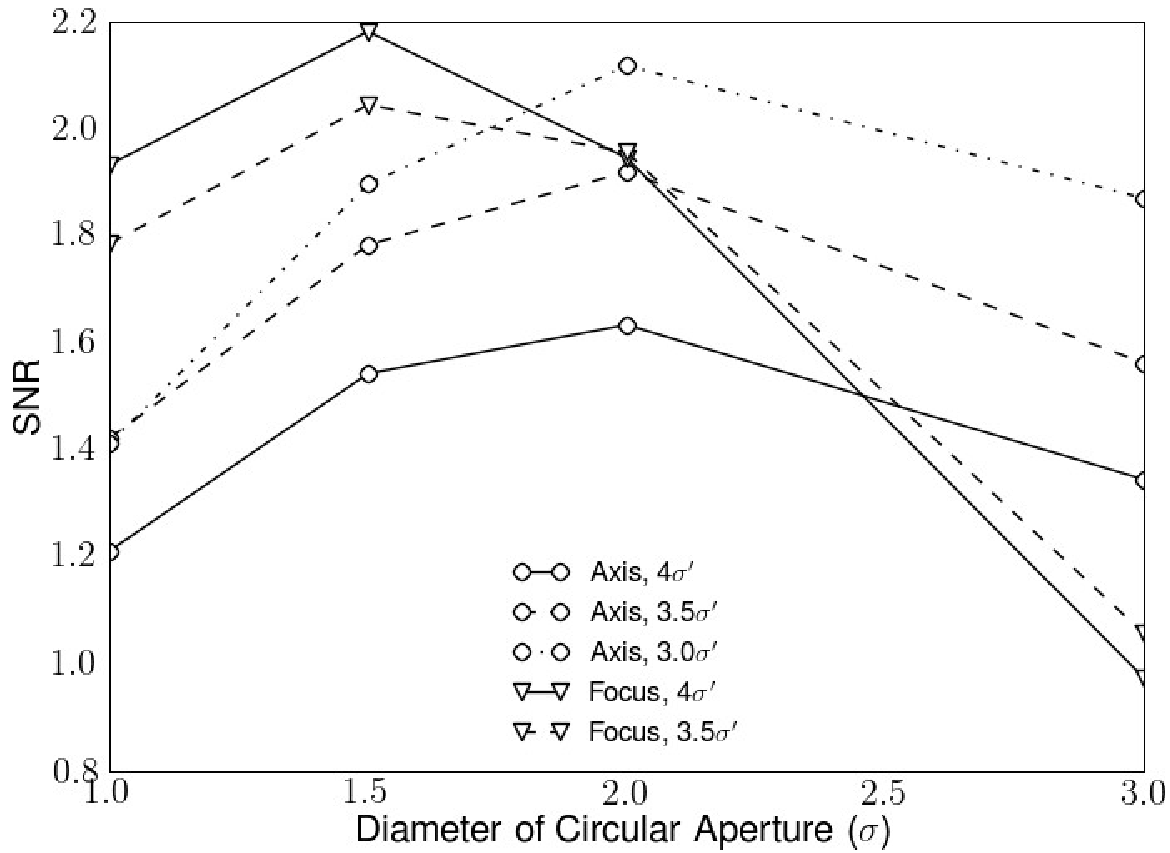
Low coherence, high flux maximizes SNR

250 eV, single electron, 41 m from the source, 4σ'



Degree of coherence at the source image





"Off-axis" radiation has higher flux, higher divergence, smaller source, much higher intensity, somewhat lower coherence

Coherence "figure of merit":

$$SNR = \sqrt{I}\,|\mu|$$

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CDI suffers from the beamstop problem

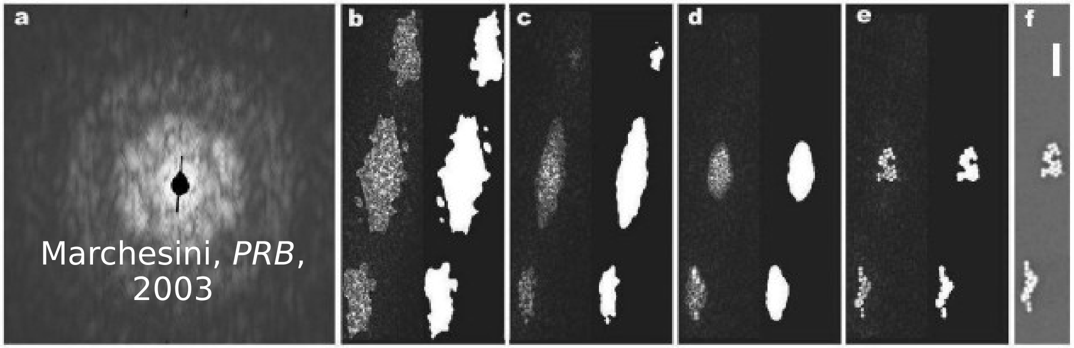
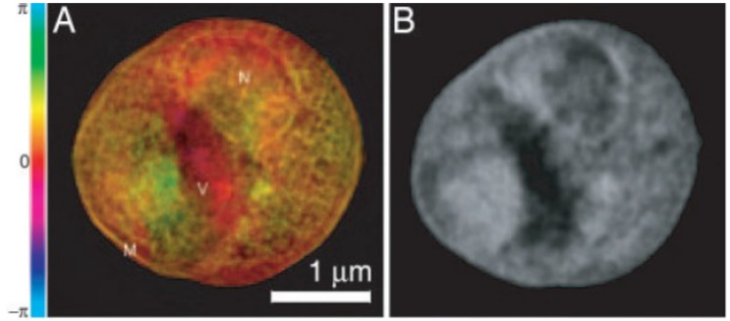


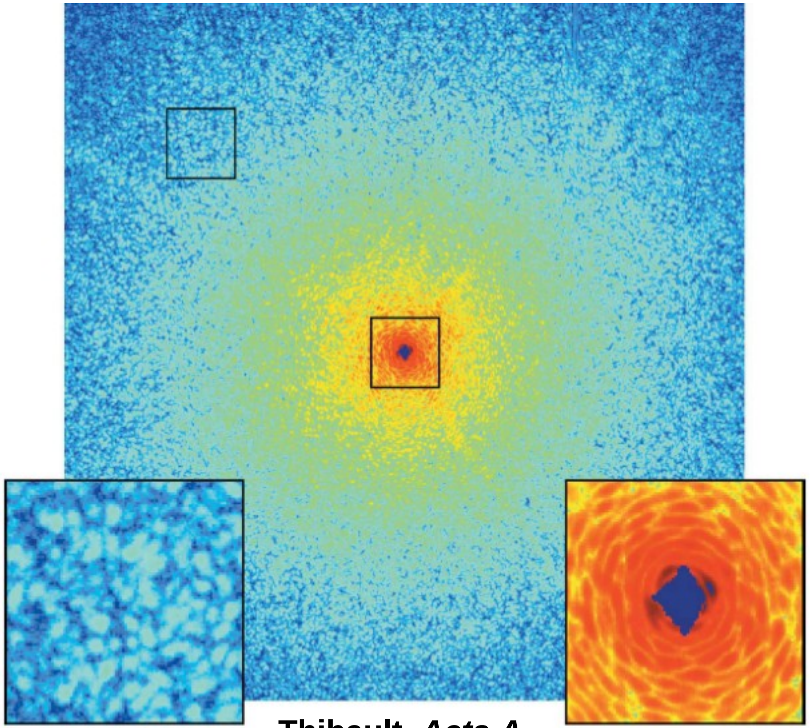
FIG. 1. Image reconstruction from an experimental x-ray-diffraction pattern. (a) X-ray diffraction pattern of a sample of 50-nm colloidal gold particles, recorded at a wavelength of 2 nm. (b–e) shows a sequence of images produced by the algorithm as it converges. Number of iterations: 1 (b), 20 (c), 100 (d), and 1000 (e). The reconstruction progresses from the autocorrelation function in (b) to an image in (e) with a steady improvement of the support boundary (shown at the bottom of each frame). For comparison, a scanning electron micrograph of the object is shown in (f). The scale bar length is 300 nm and the resolution of our reconstructed image is about 20 nm.

Marchesini, *PRB*, 2003

High resolution is easier than low resolution!

- Shrinkwrap works for objects with sharp boundaries
- Biology general requires user intervention for support determination



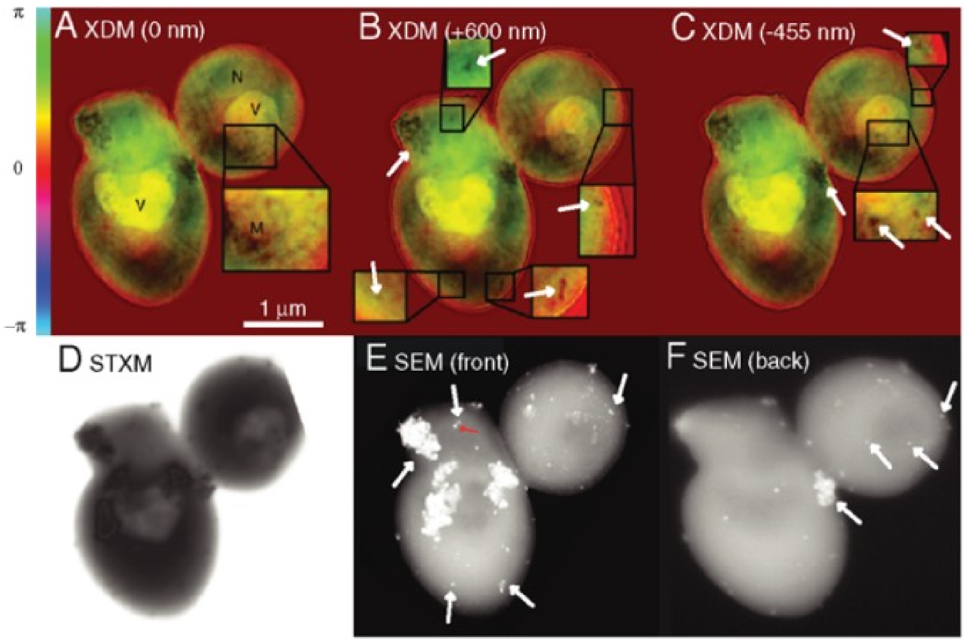


Thibault, *Acta A*, 2005

Figure 1
Soft X-ray ($\lambda = 1.65$ nm) diffraction pattern of a freeze-dried yeast cell on a logarithmic scale (Shapiro *et al.*, 2005). This 1200 × 1200 array extends to $(20.7\ \text{nm})^{-1}$ on the sides, giving the corresponding real-space array 10.3 nm wide pixels. Inset, left: Magnified portion of the diffraction pattern showing the speckles. Inset, right: Magnified central region showing the diamond-shaped missing data region.

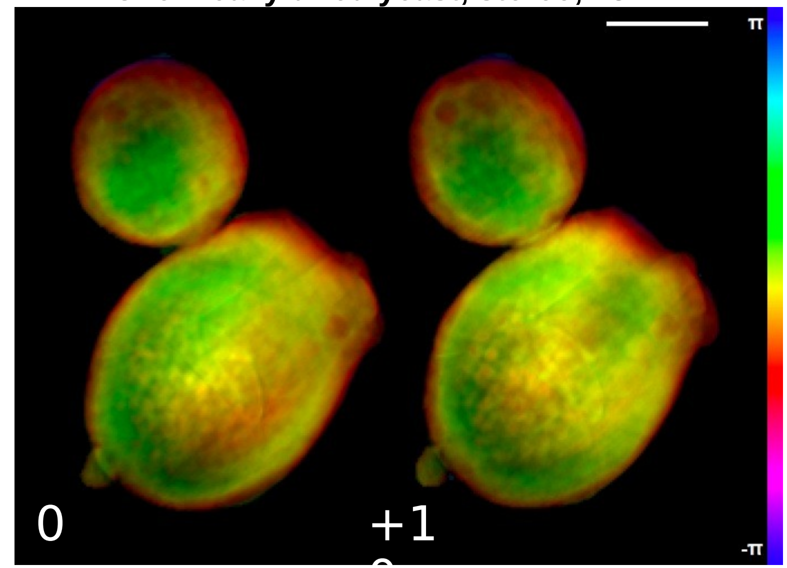
Nevertheless...

Specifically labelled, dried yeast, 13 nm



Nelson, *PNAS*, 2010

Chemically dried yeast, stereo, 13 nm

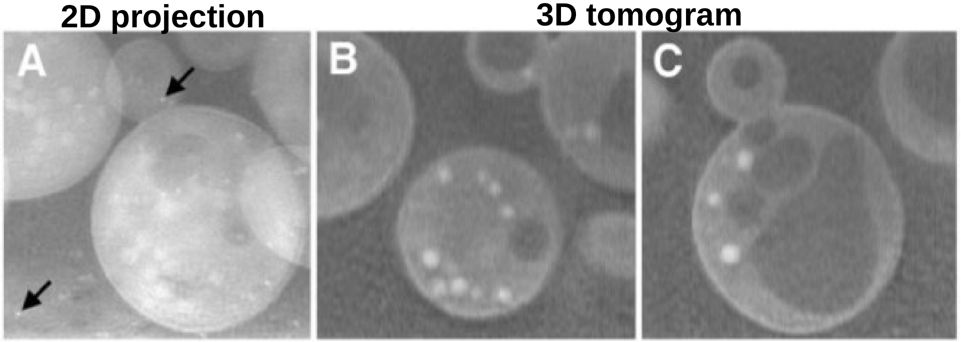


Shapiro, Huang

Comparison with TXM at ~50 nm resolution
3D greatly enhances contrast, cryo needed!

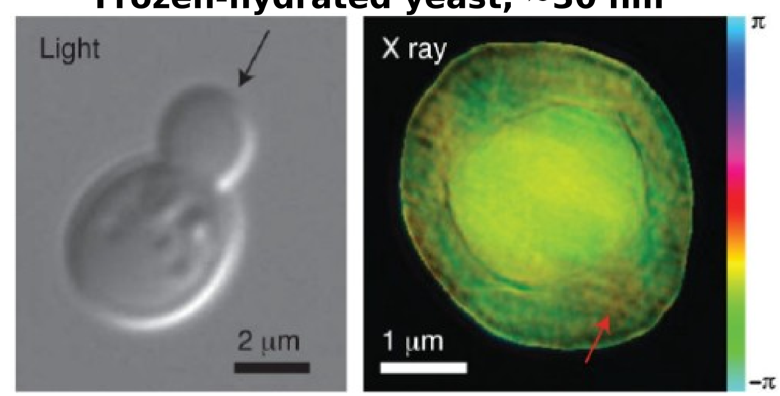
2D projection

3D tomogram



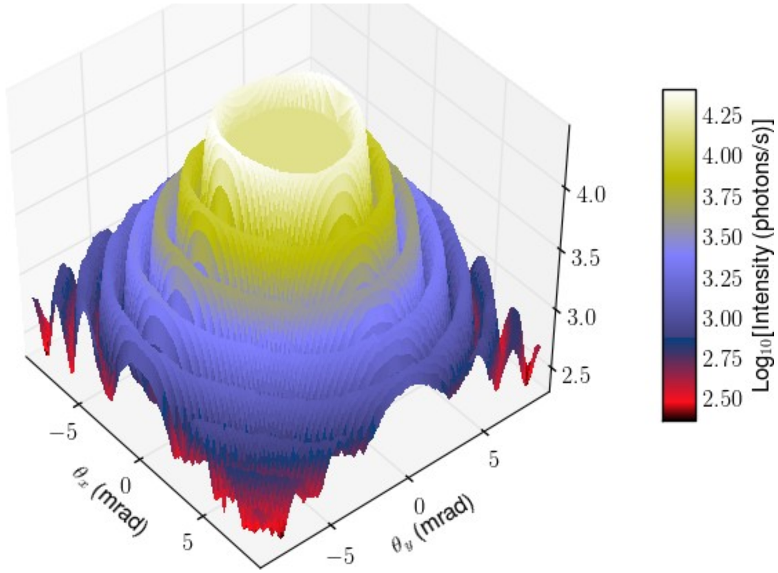
Larabell, *MBC*, 2004

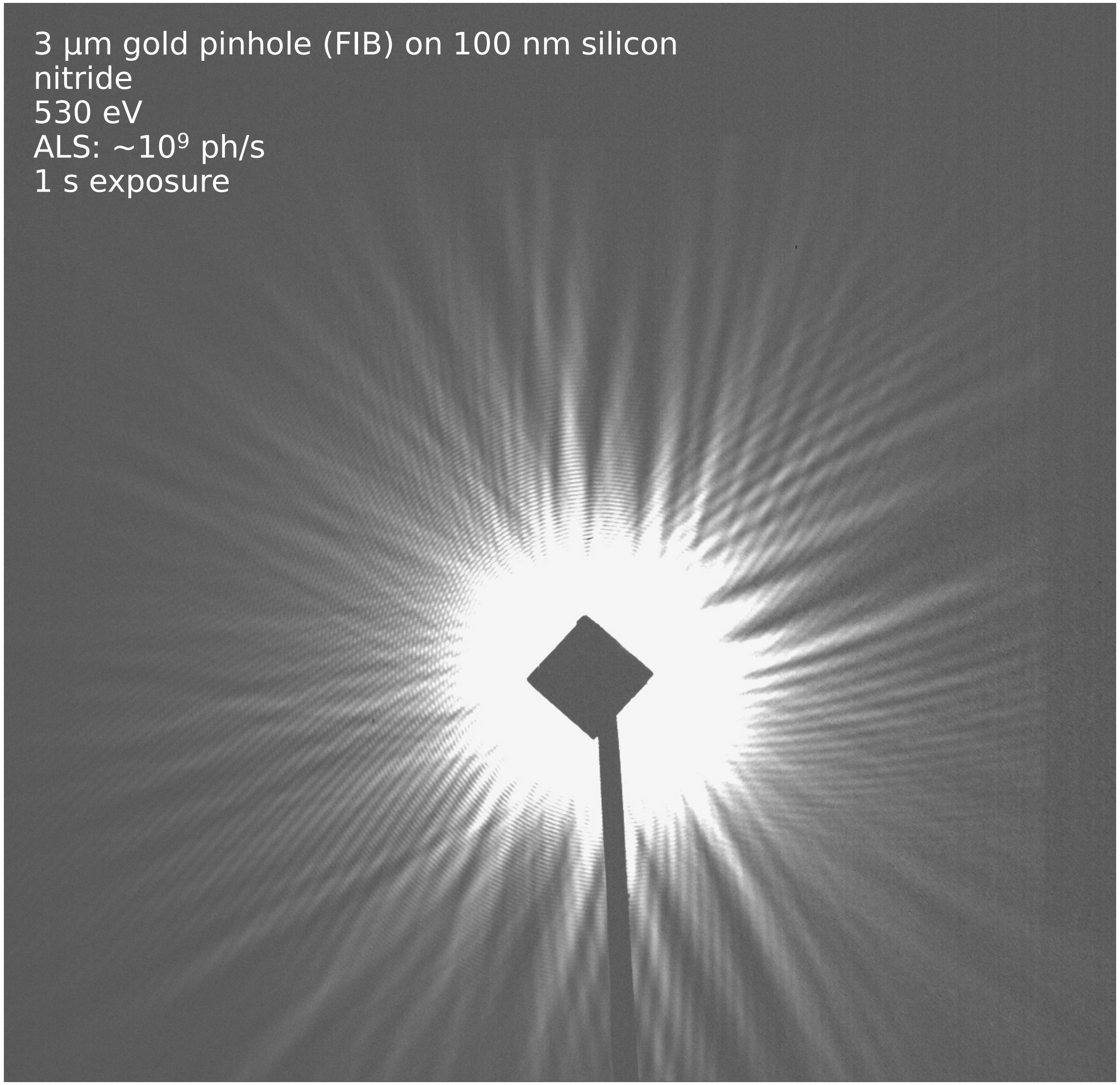
Frozen-hydrated yeast, ~30 nm



Huang, *PRL*, 2010

It’s really hard to make a clean beam





- ALS 9.0.1, 3 micron coherence patch at exit slit
- Metal pinholes select coherent beam
- Airy pattern must be blocked by guard apertures
- Biggest source of systematic noise

A sample is only as clean as its illumination!

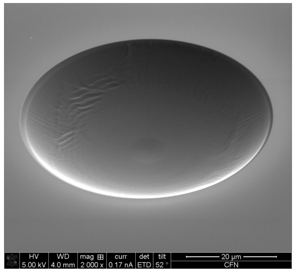
Use a smooth transmission filter instead

A spherical cap transmission profile:

$$T(r < r_0) = R(1 - \sqrt{1 - (\frac{r}{R})^2})$$
$$T(r \geq r_0) = t_0$$

Absorption limits the usable portion to a parabola:

$$T(r) = \frac{r^2}{2R}$$

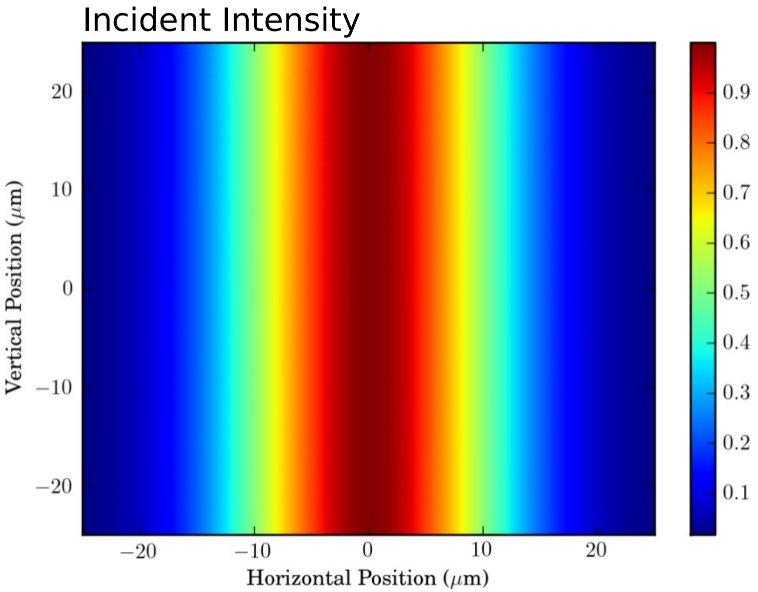


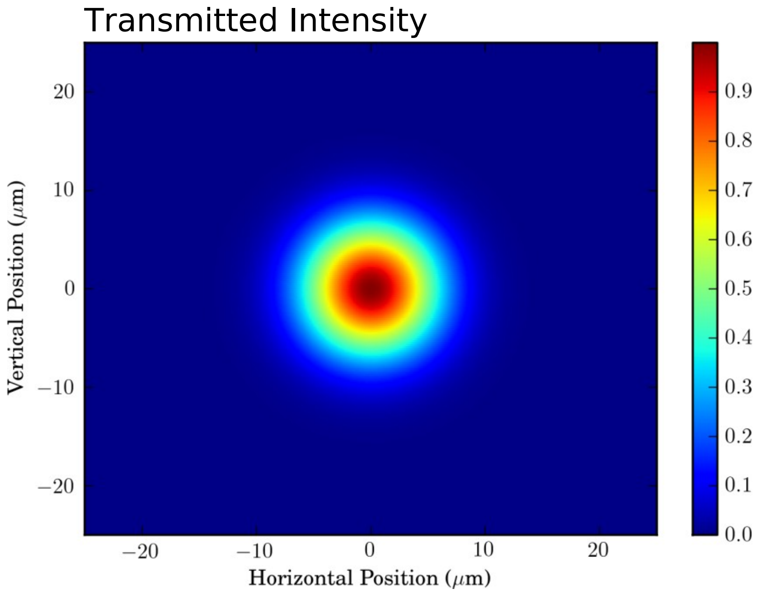
The transmitted intensity has a gaussian profile:

$$I(r) = I_0 e^{-\mu T(r)} = I_0 e^{-\mu r^2/2R}$$

With standard deviation:

$$\sigma_t = \sqrt{\frac{1}{\frac{1}{\sigma i^2} + \frac{\mu}{R}}}$$





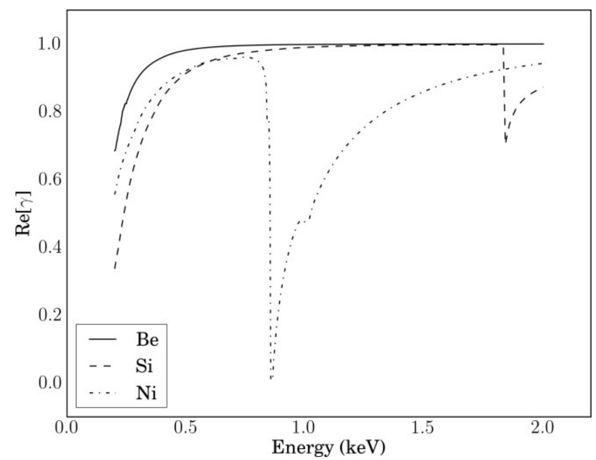
It's also a spherical lens

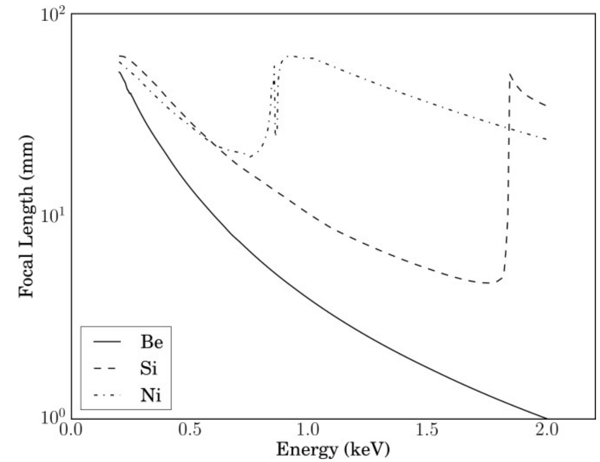
A plano-concave refractive lens has focal length:

$$f = \frac{R}{\delta}\mathrm{Re}[\gamma]$$

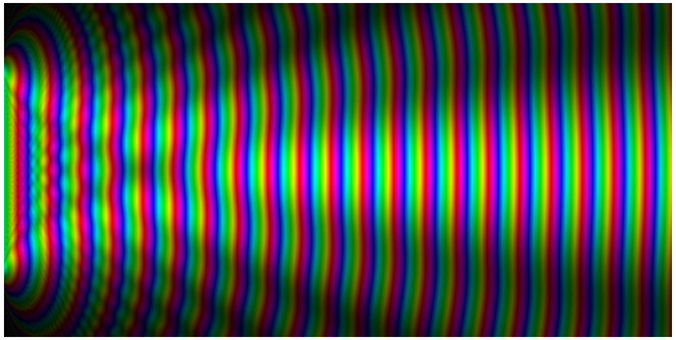
Where:

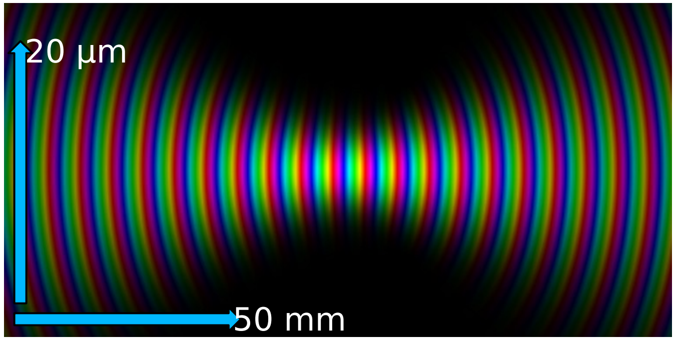
$$\gamma = \frac{\delta}{\delta + i\beta}$$





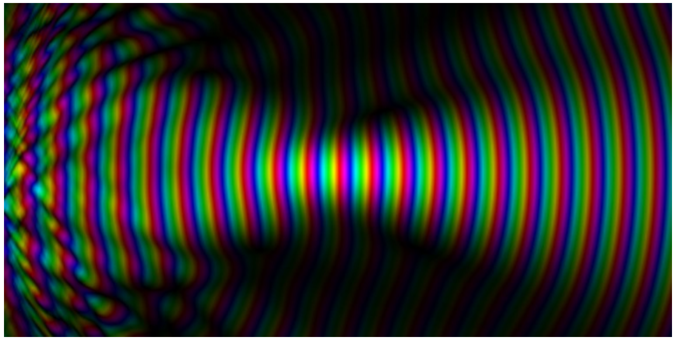
3σ pinhole

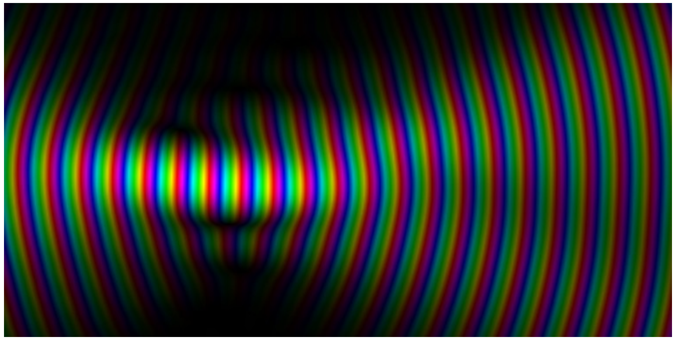




Gaussian filter

Gaussian filter with:
FE = 0.1 μm
$f = 5\ \mu m^{-1}$

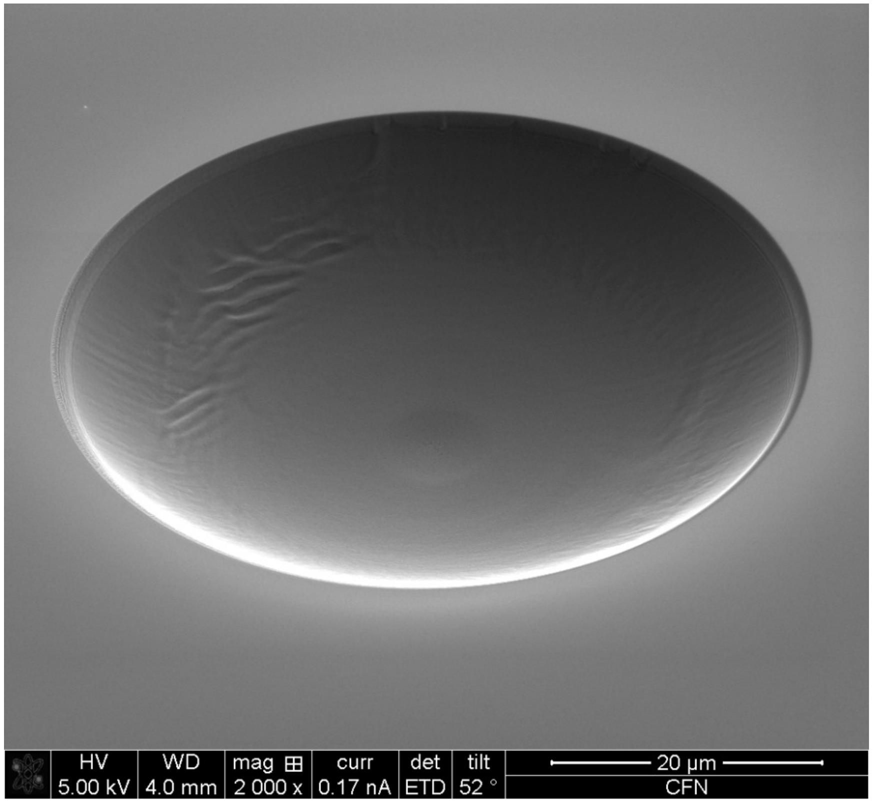


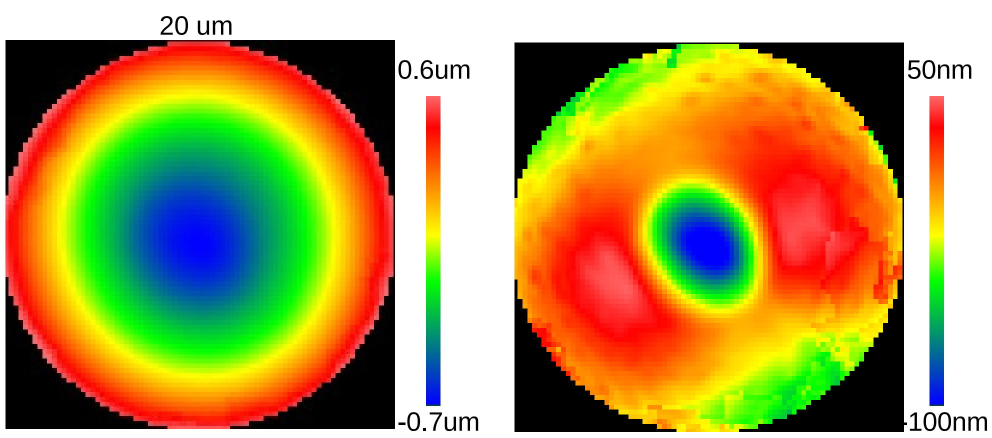


Gaussian filter with:
FE = 1 μm
$f = 0.2\ \mu m^{-1}$

Refractive silicon lens/gaussian filter

- Figured by FIB milling: **Fernando Camino** at the CFN, BNL.
- On-axis thickness: ~350 nm (50% transmission at 500 eV), goal is 100 nm
- SOI wafer device layer thickness: ~10 µm (10^{-9} transmission at 500 eV)
- RMS figure error: ~30 nm
- Peak-Valley figure error: ~165 nm

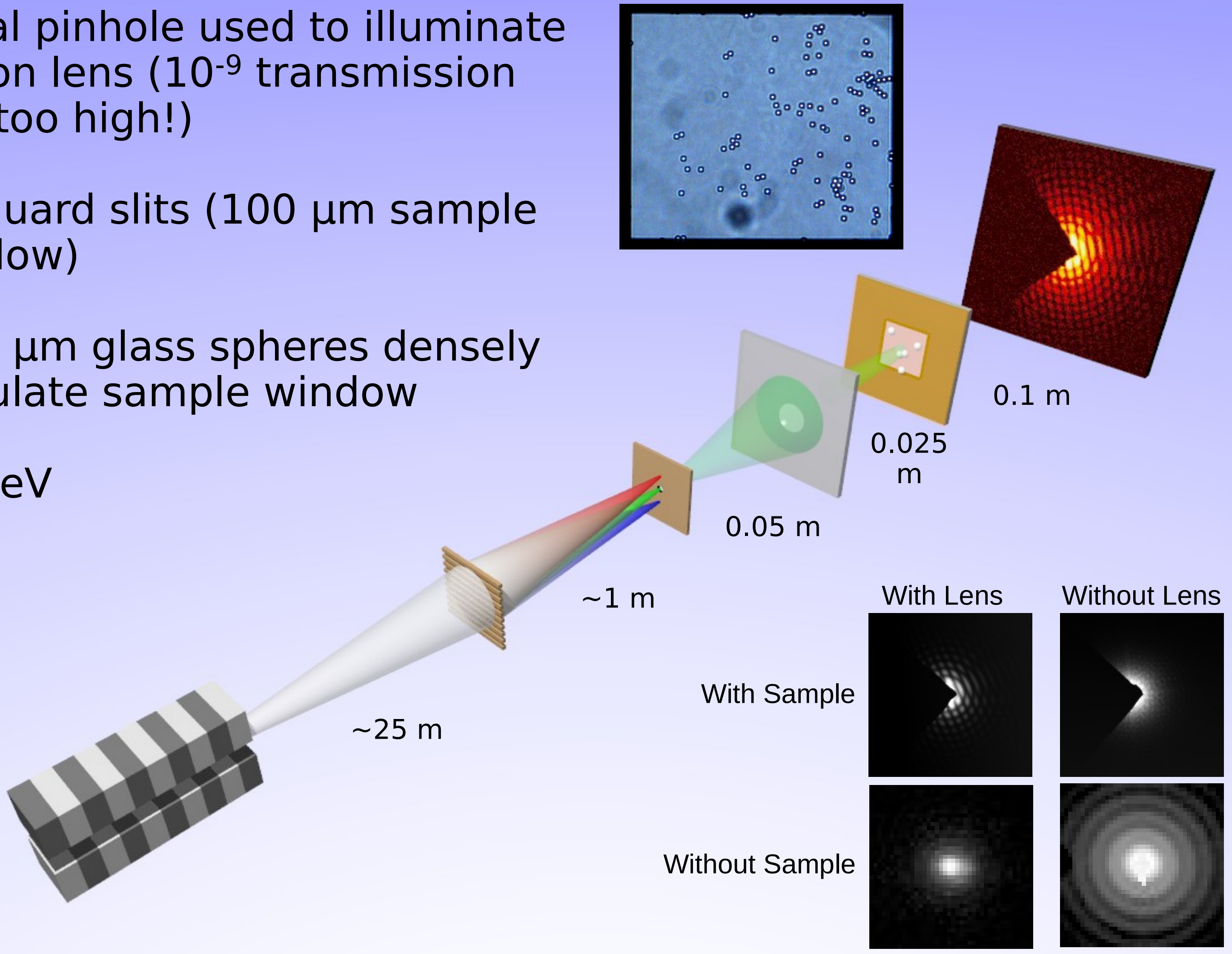




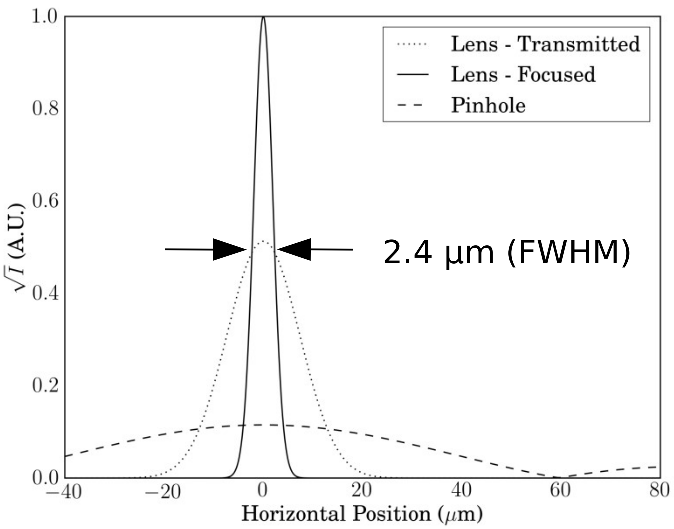
Optical metrology: Konstantine Kaznatcheev

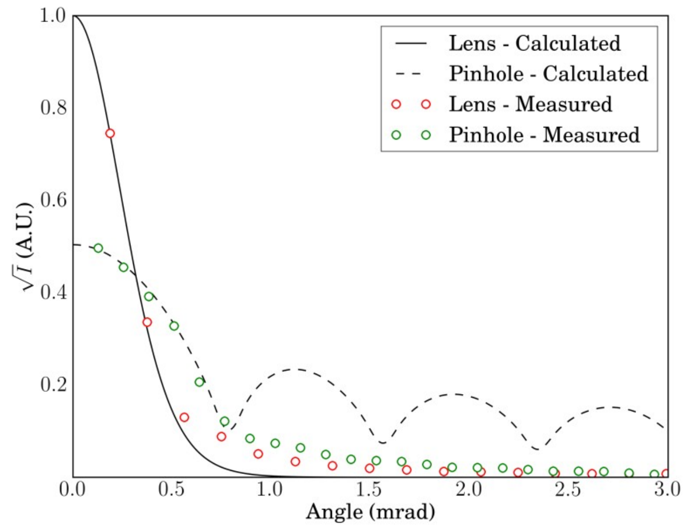
Preliminary tests at the ALS

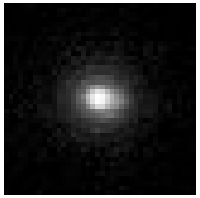
- Metal pinhole used to illuminate silicon lens (10^{-9} transmission still too high!)
- No guard slits (100 µm sample window)
- 1.86 µm glass spheres densely populate sample window
- 530 eV

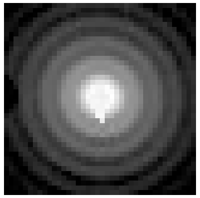


Clean soft x-ray beams

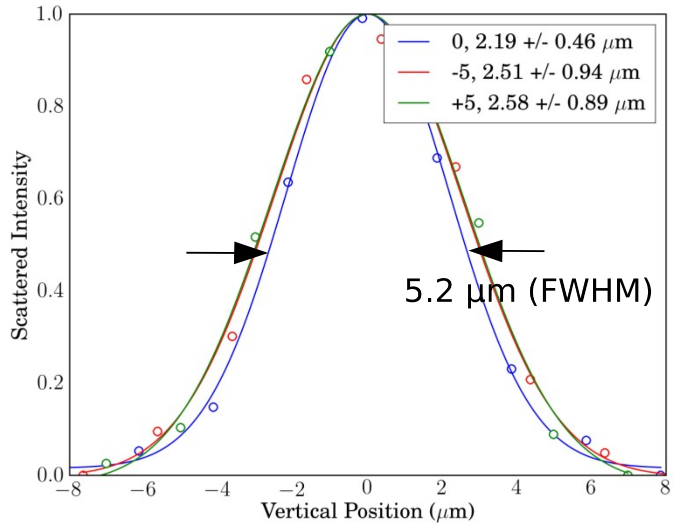


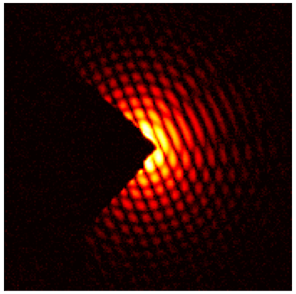






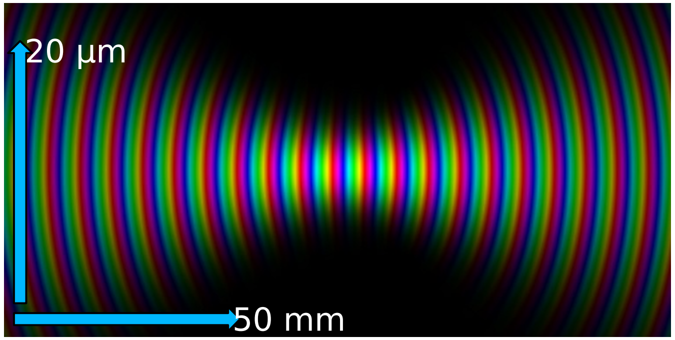
- Predicted width of 2.4 microns (intensity)
- Sample scan gives 5.2 microns
- 3.5 micron sample
- 40 microns on the CCD
- Measured width diverges from focus





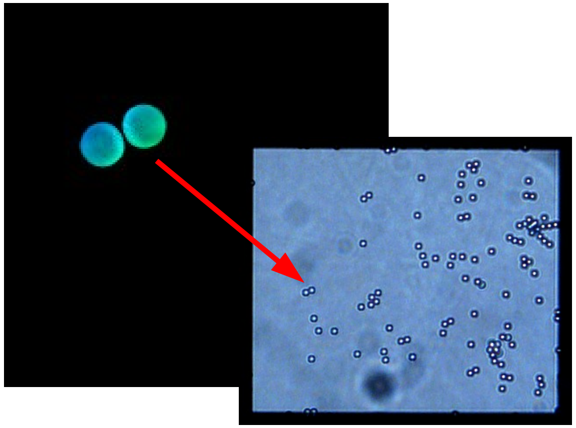
Applications of the Si lens/filter

- Rapidly switch between CDI and ptychography modes
- Coherence is preserved
- Negligible parasitic scattering
- Guard slits are not needed
- Can image much more densely populated samples



Reconstruction of 2 glass beads

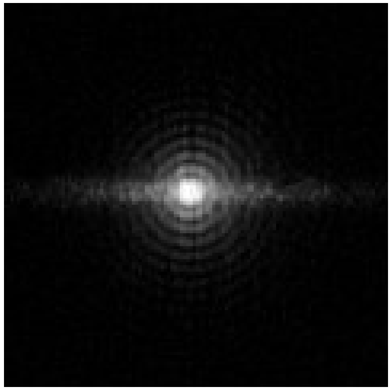
Field of view is 15.6 microns but the nearest neighbor is only 5 microns away!

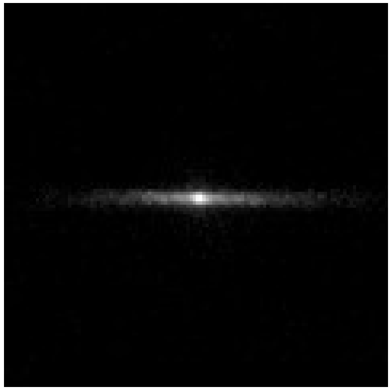


Grazing incidence reflection from a multilayer mirror – ptychography for at-wavelength metrology

Specular and scattered beams overlap

Silicon lens eliminates most of the specular beam (no Airy fringes)





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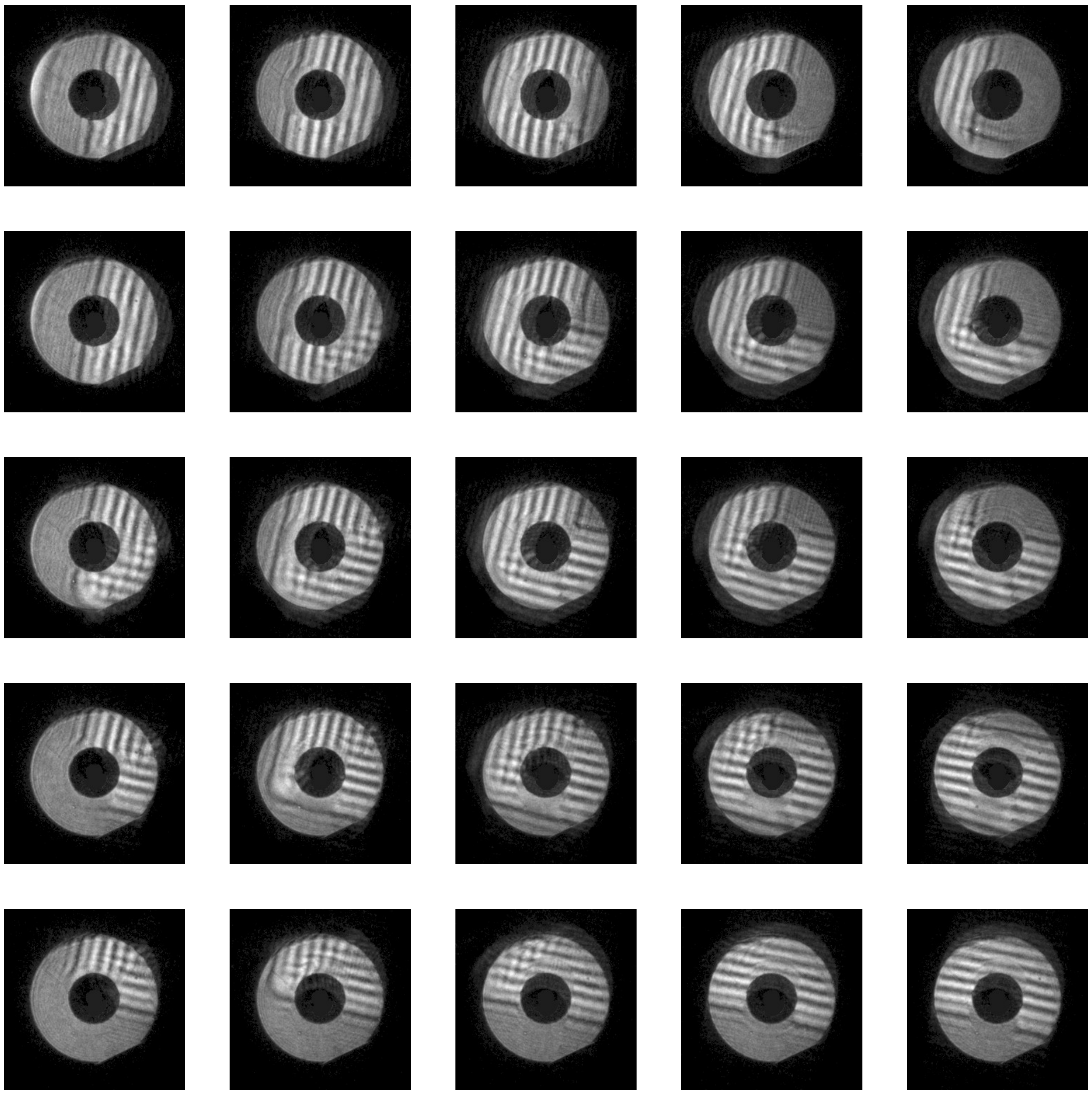
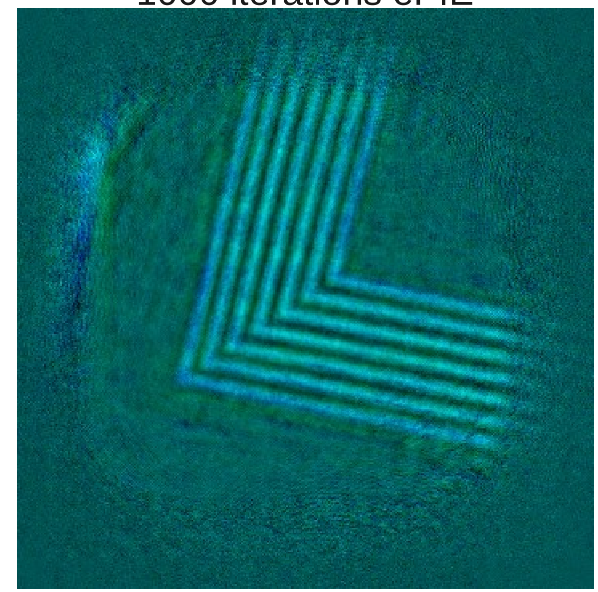
Imaging with a defocused zone plate

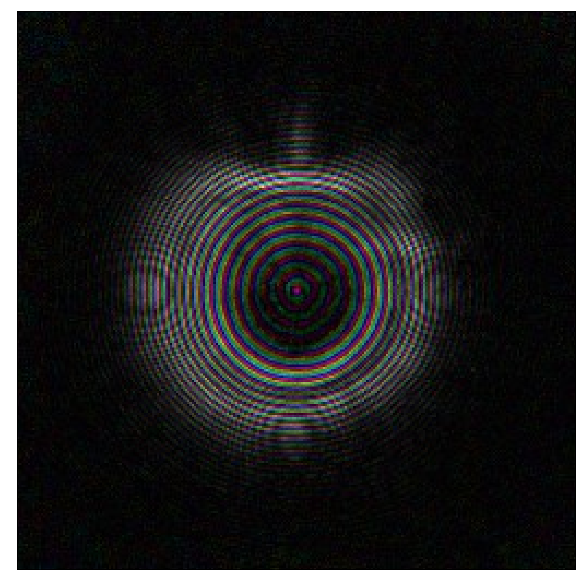
ZP = 240/96/25, E = 709 eV, Andor CCD 1300x1300



Konstantine Kaznatcheev, CLS STXM

1000 iterations ePIE



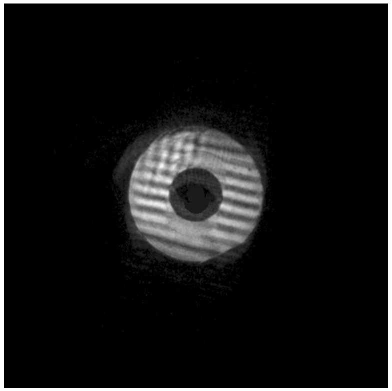


← 4.5 microns →

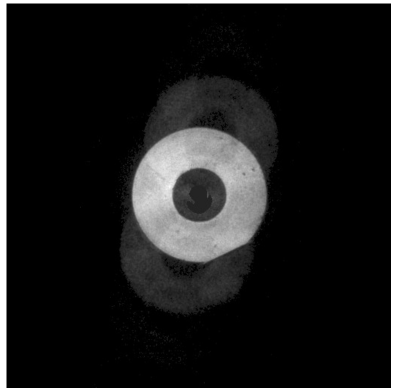
Focusing improves resolution!

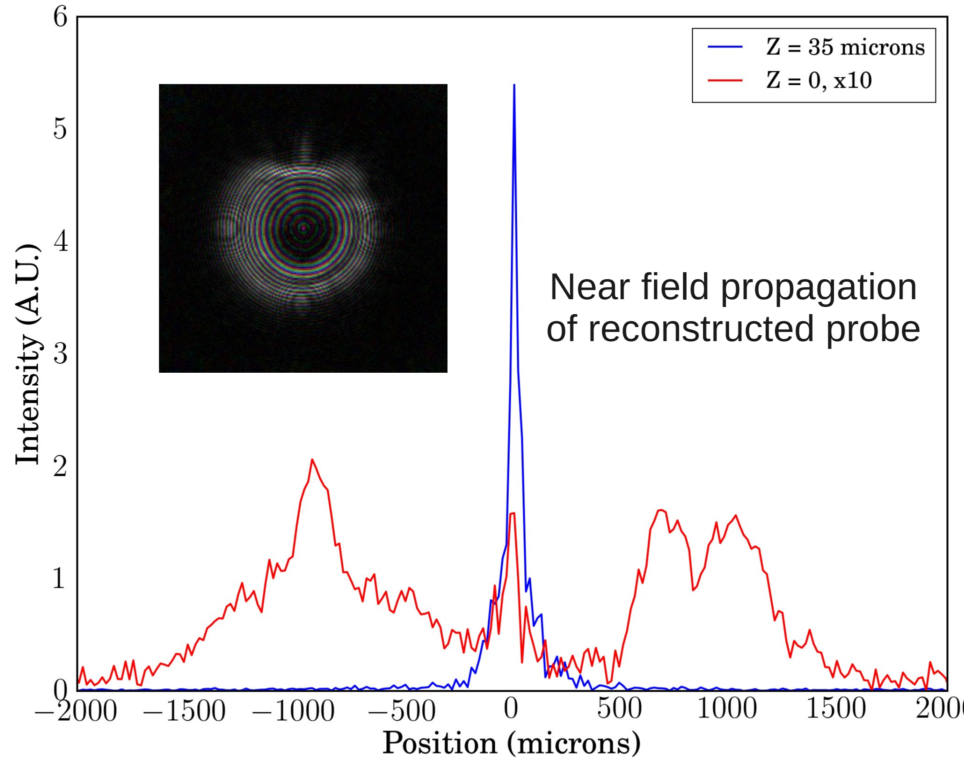
Experiment

35 microns from focus



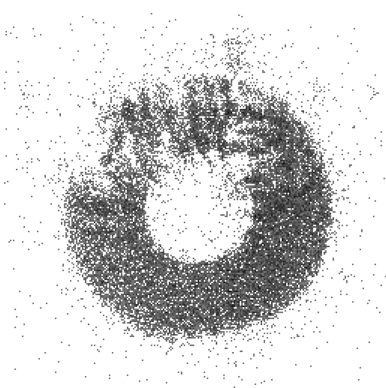
12 microns from focus

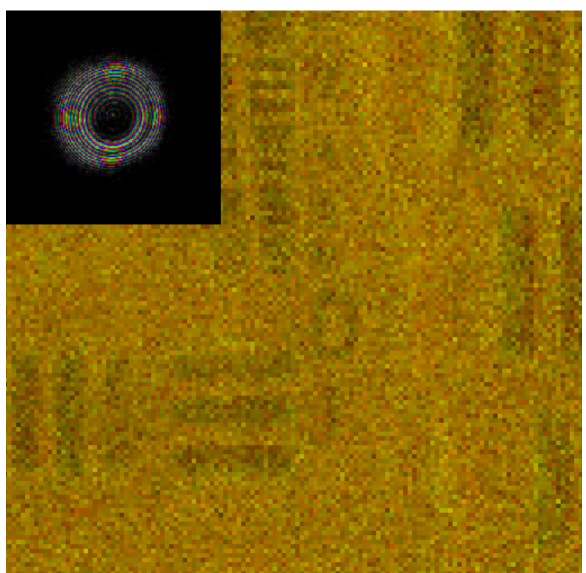


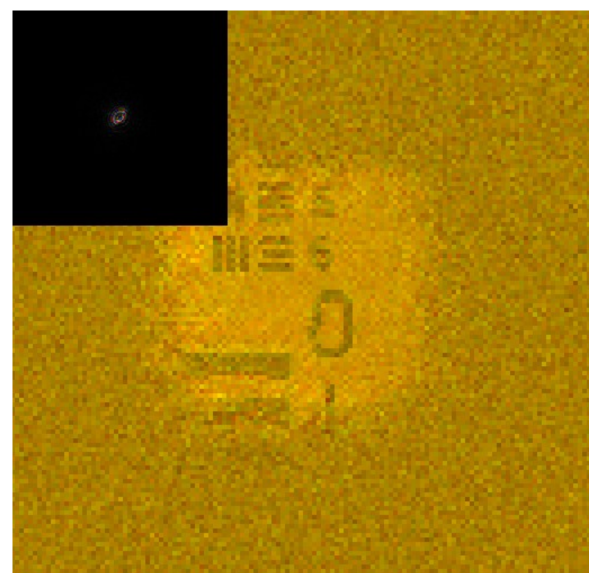


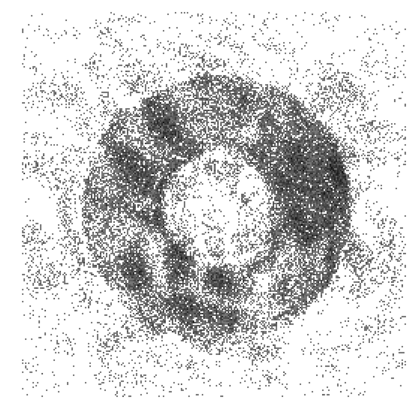
Higher intensity near focus increases resolution with comparable exposure times

Simulation



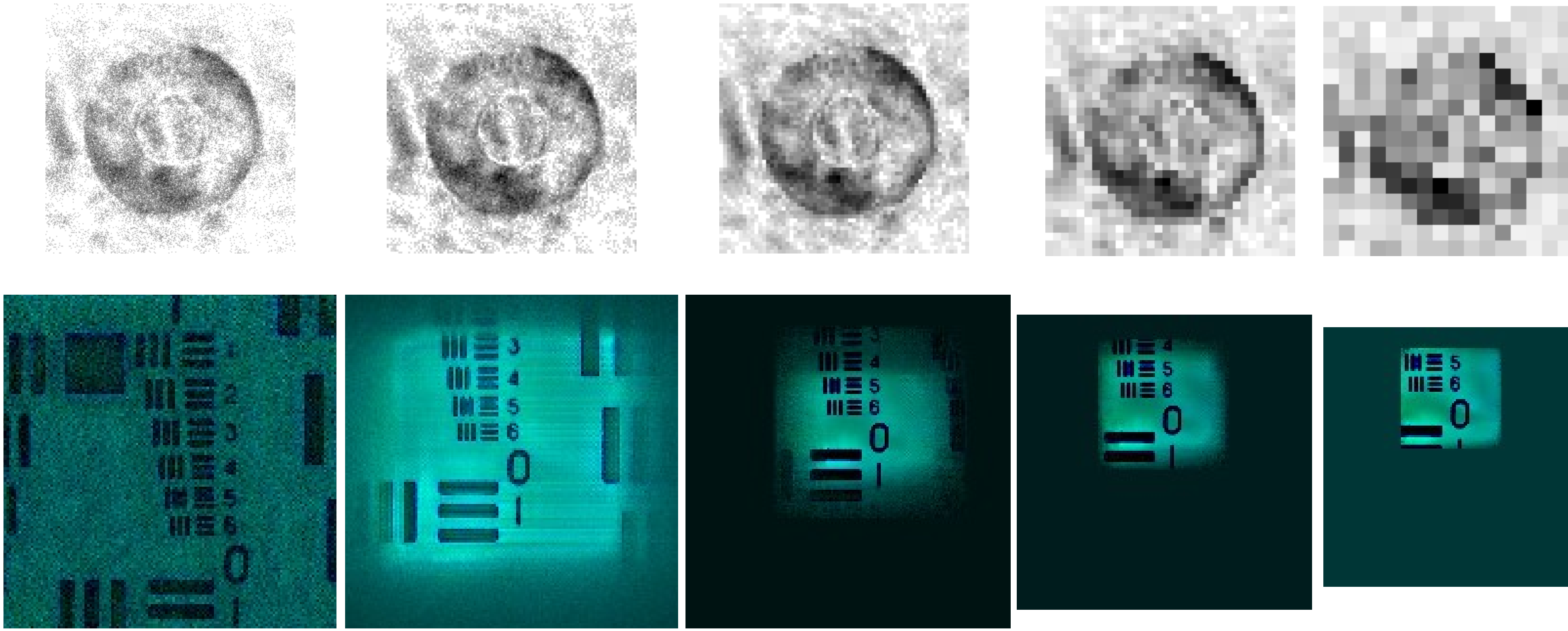






How many samples are needed?

256x256 128x128 64x64 32x32 16x16



Fewer samples:
decreases noise
Speeds up acquisition
Speeds up reconstruction
Decreases storage
Decreases FOV
4x4 failed!

8x8

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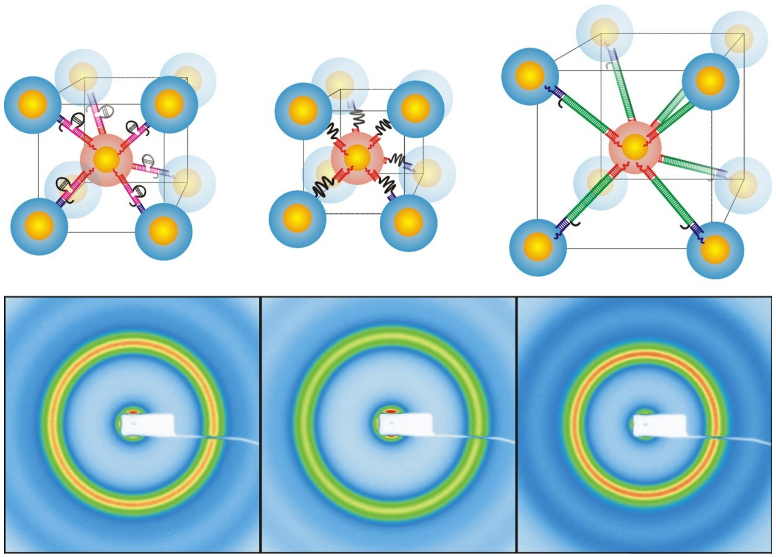
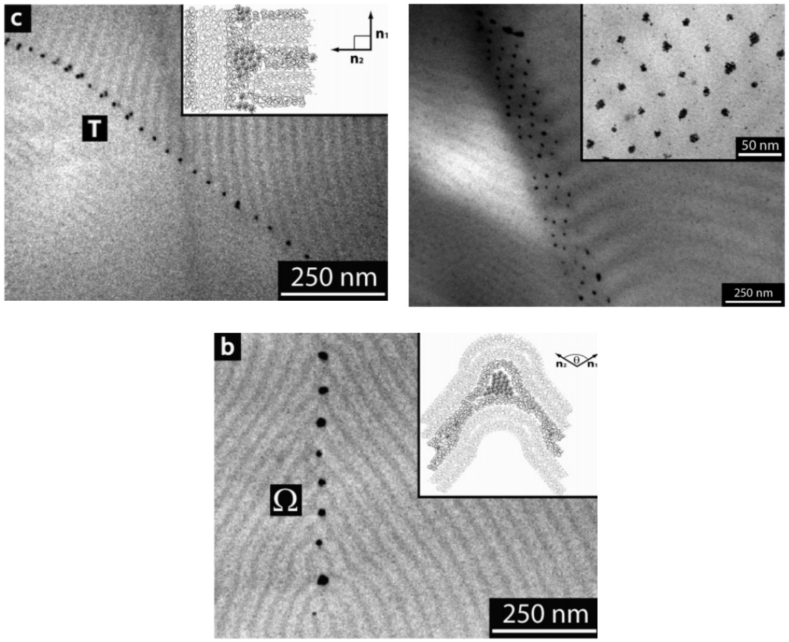
Applications

Center for Functional Nanomaterials:
Self-assembled Au/DNA nano-crystals
•Switchable lattice
•Image DNA scaffold



M. Maye, *et al*, Nature Nanotech., 2010

Block co-polymers:
Nano-particle additives stabilize grain boundaries



Listak, J.; Bockstaller, M.R. Macromolecules ***2006****, 39, 5820.*

Organic materials with controllable nano-structure

In Conclusion

- CSX will deliver very high coherent flux for coherent scattering experiments
- Tuning to the off-axis radiation provides significantly more flux and adequate coherence

- CDI can provide high resolution images but with low throughput
- Clean illumination may alleviate the throughput limiting issues

- Soft x-ray ptychography is under development at the CLS, planned for NSLS-II
- We demonstrate contrast enhancement with focusing and the effect of sampling on image noise

Thank you for your attention!

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and many, many more…