Nanoscience Research at the National Synchrotron Light Source

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Office of Science
U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

VUV Storage Ring:

51 m circumference

E = 0.808 GeV

I= 1.0 A

7 bunches

Orbital Period = 170 ns

Pulse Width = 320 ps

X-Ray Storage Ring:

170 m circumference

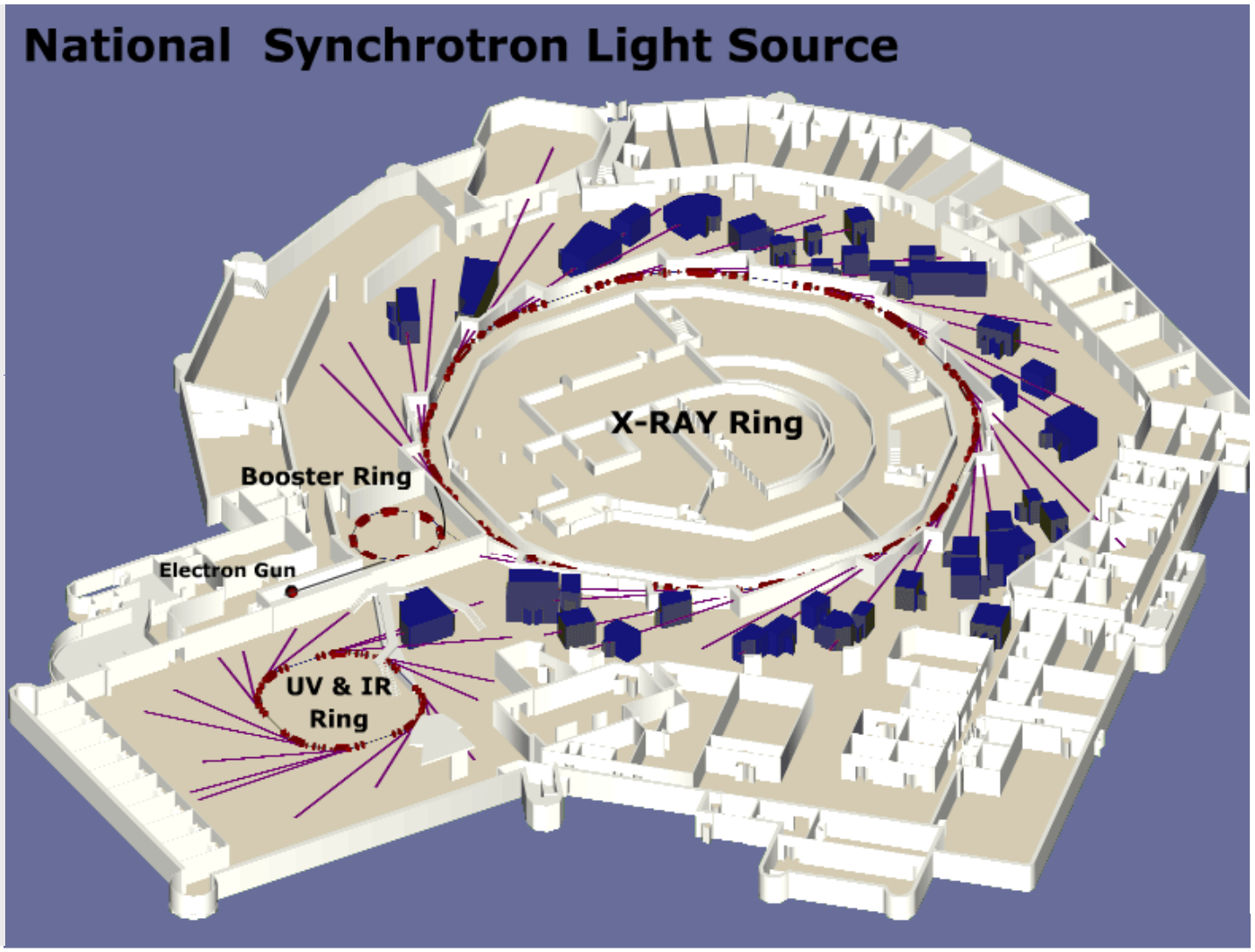
E = 2.8 GeV

I = 280 mA

25 bunches

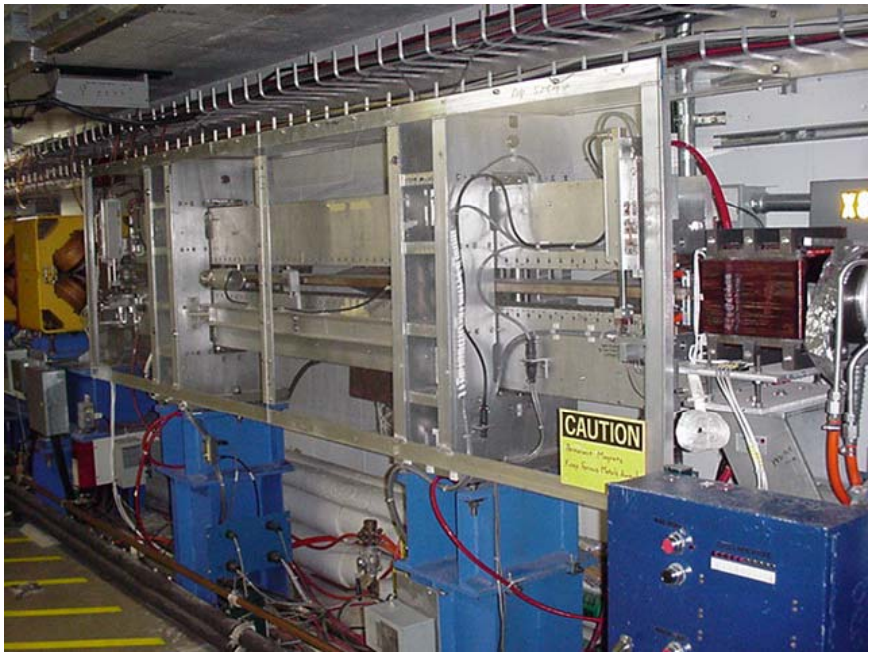
Orbital Period = 567 ns

Pulse Width = 290 ps

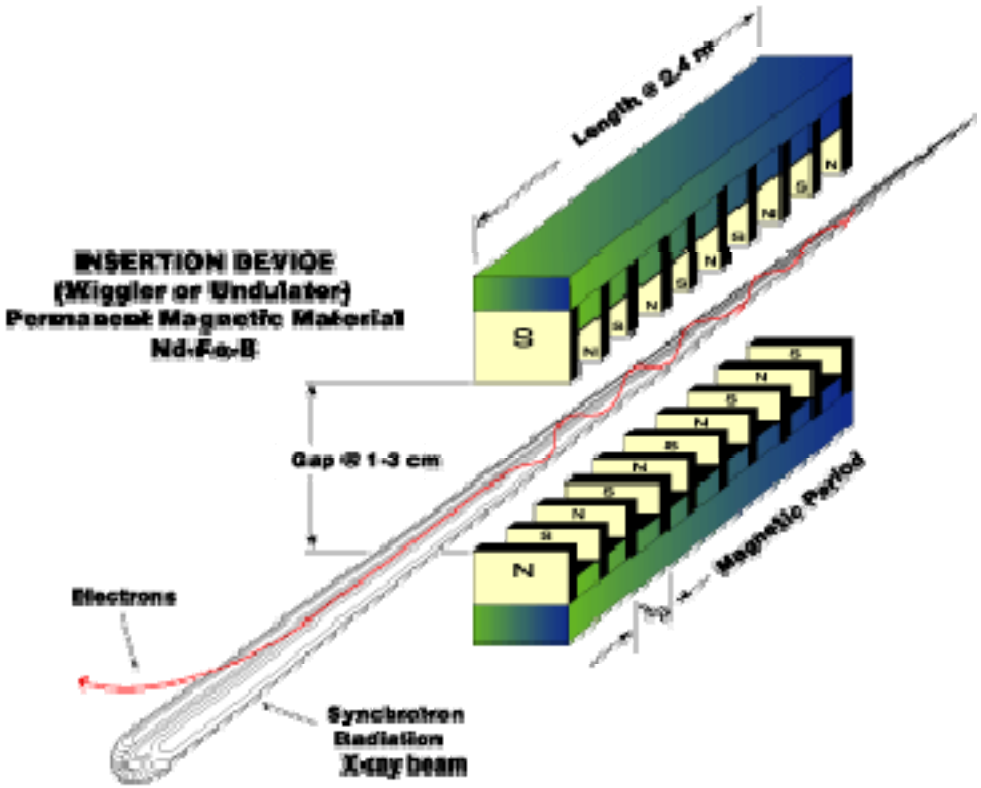


65 Operational Beamlines

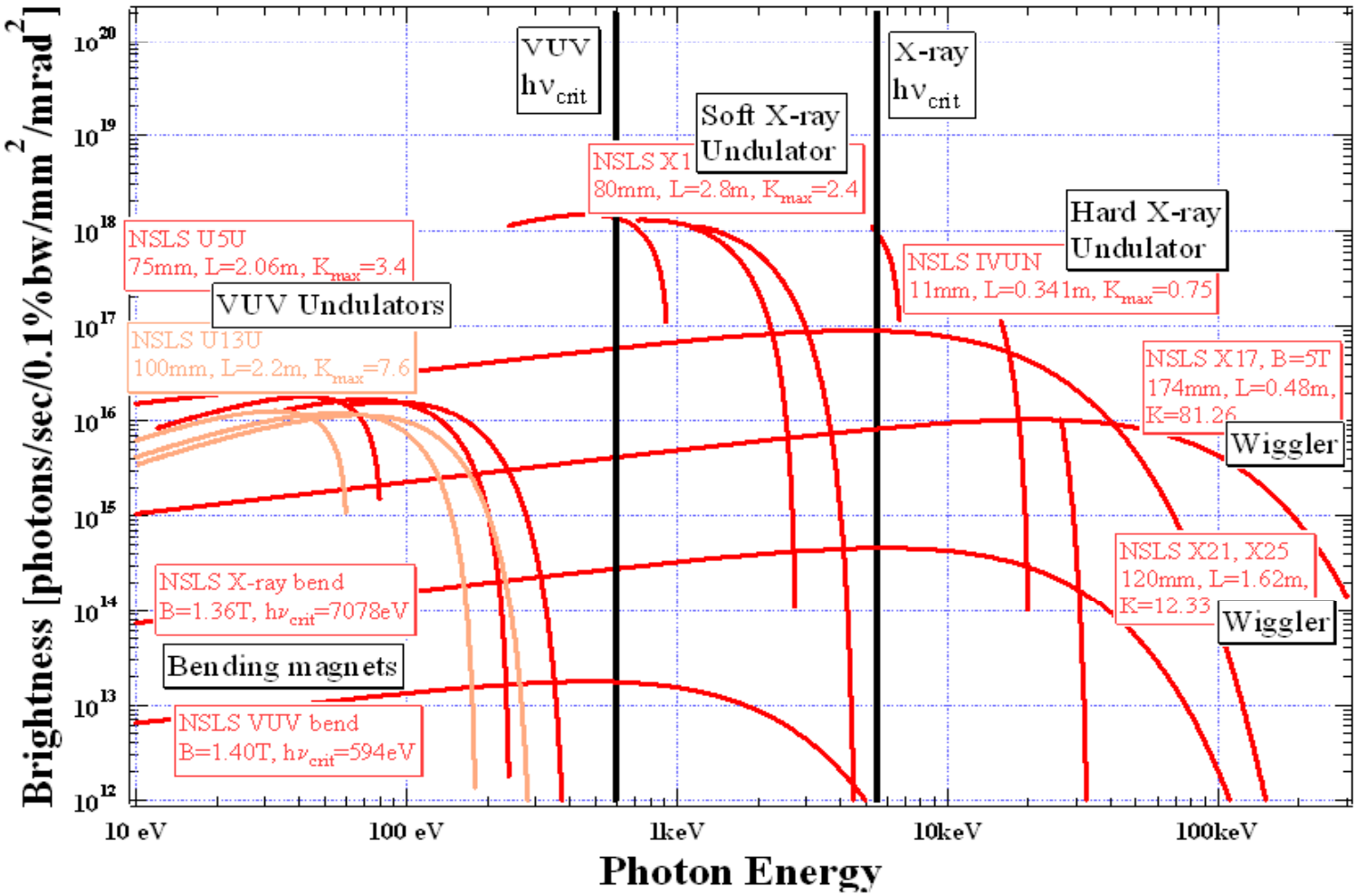
Undulator Insertion Device



Soft X-Ray Undulator – X1

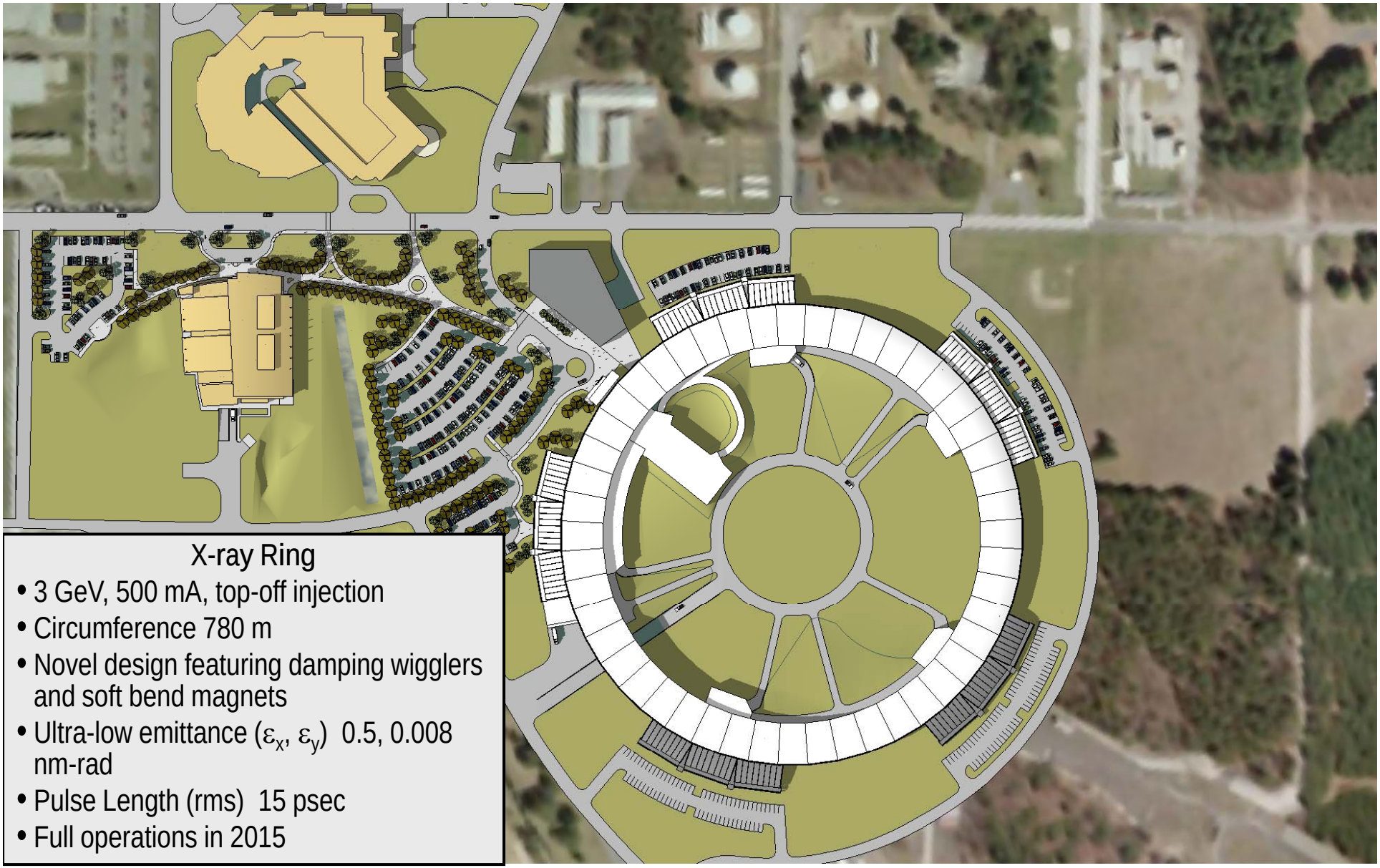


Brightness Spectra of the NSLS Sources



In these units, the brightness of an in-house rotating-anode x-ray source is 10^8 while the diffraction Limit is 10^{23}

NSLS-II Site Plan

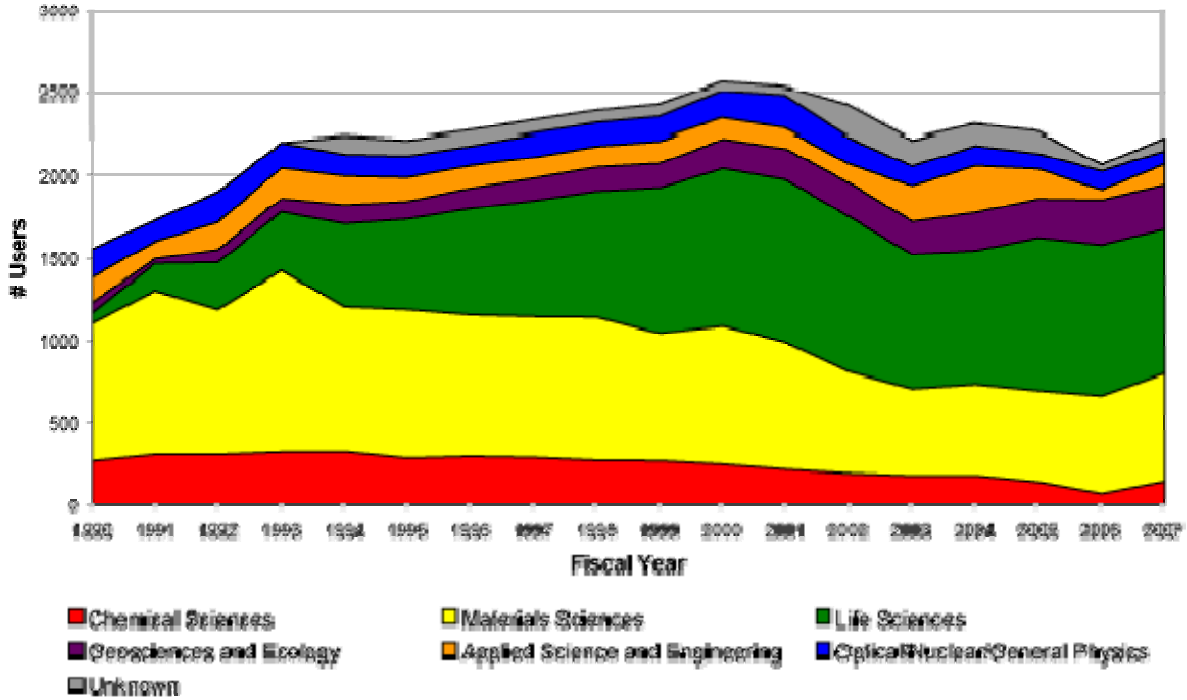


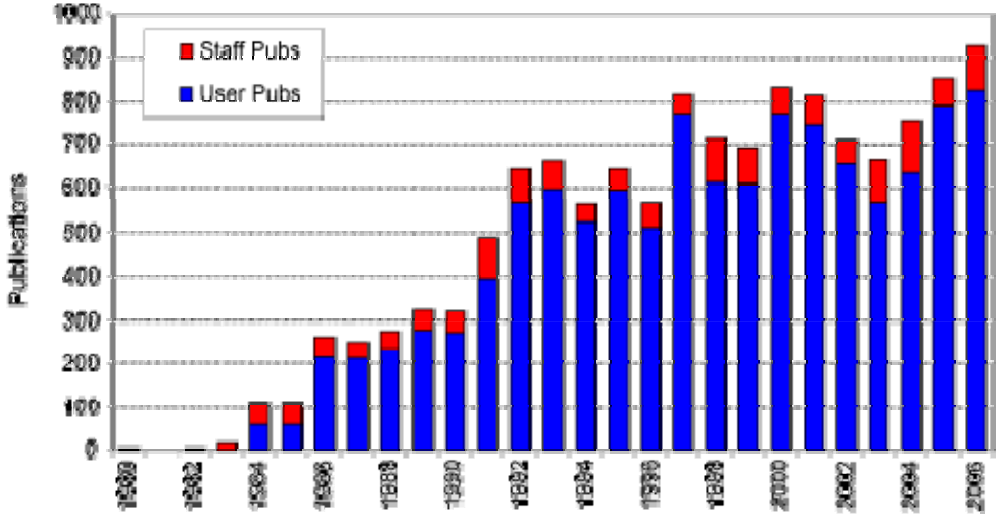
X-ray Ring

- 3 GeV, 500 mA, top-off injection
- Circumference 780 m
- Novel design featuring damping wigglers and soft bend magnets
- Ultra-low emittance (ε_x, ε_y) 0.5, 0.008 nm-rad
- Pulse Length (rms) 15 psec
- Full operations in 2015

Since 1993 the NSLS has supported over 2000 users/year

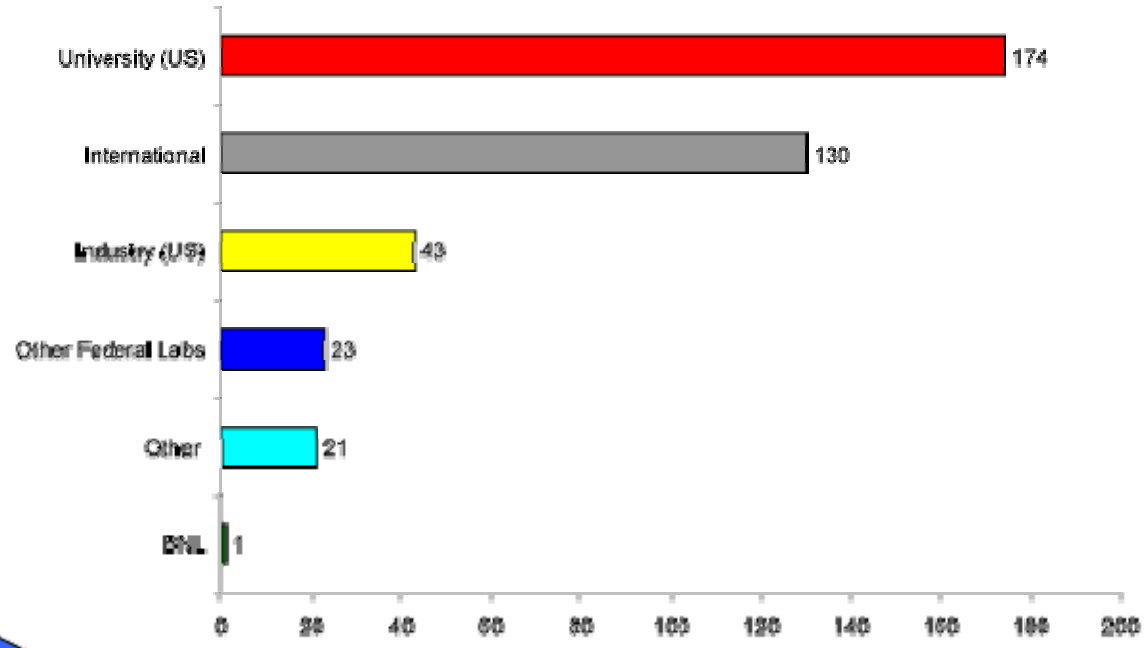
Users are selected by a peer-review process using an on-line proposal system

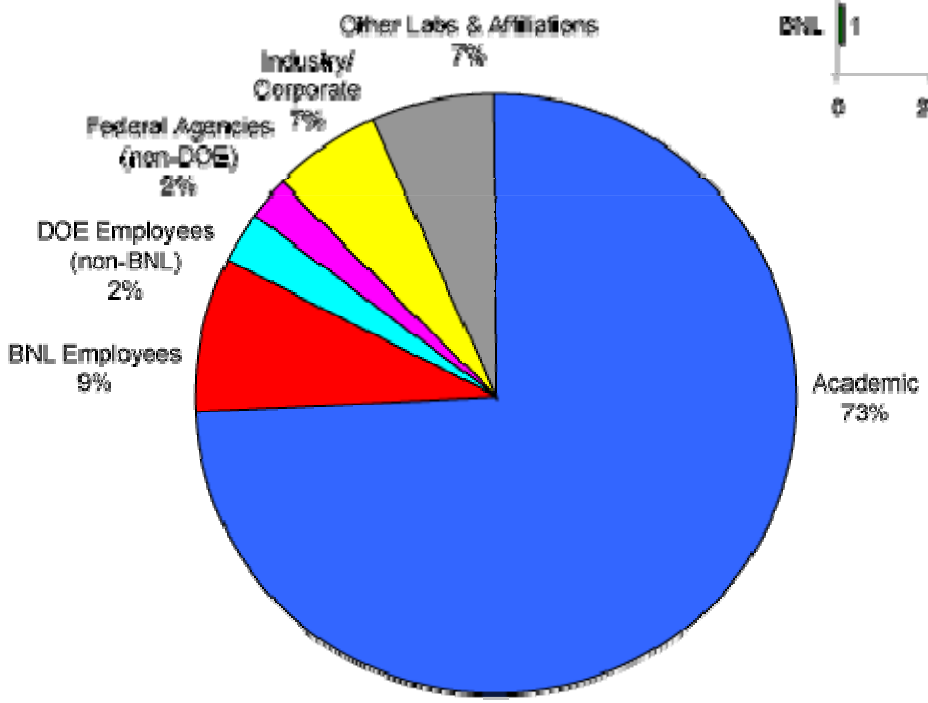




The scientific productivity has steadily increased to over 900 pubs/year

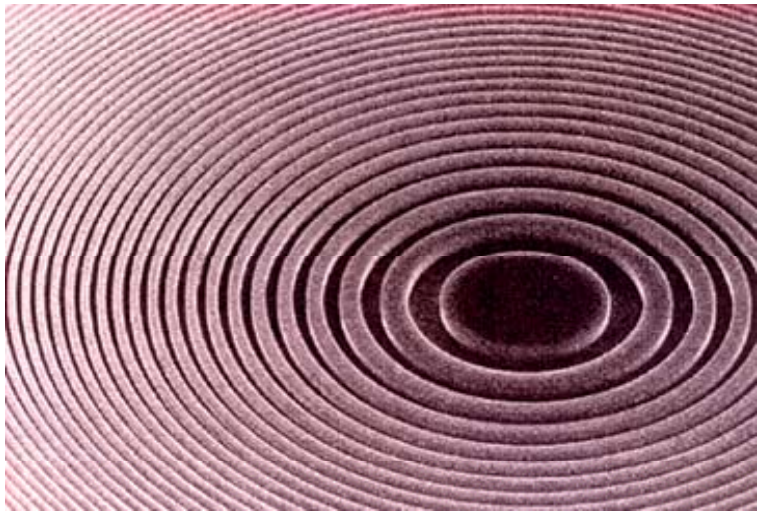
43 different U.S. industrial research labs utilize the NSLS

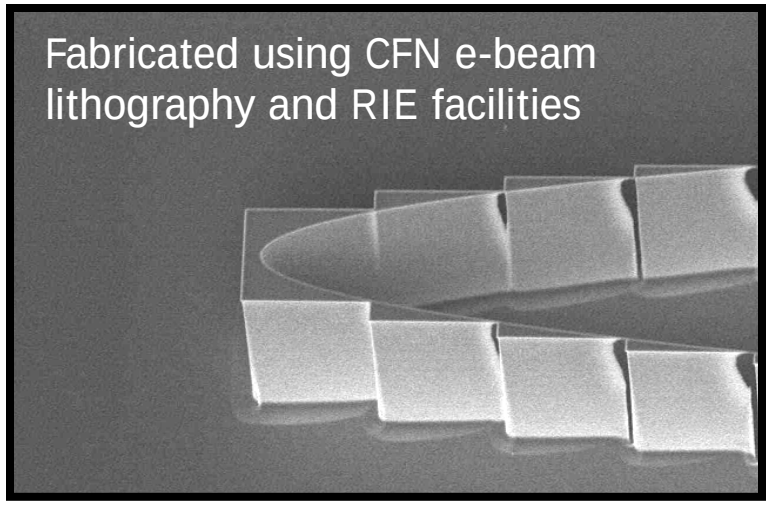


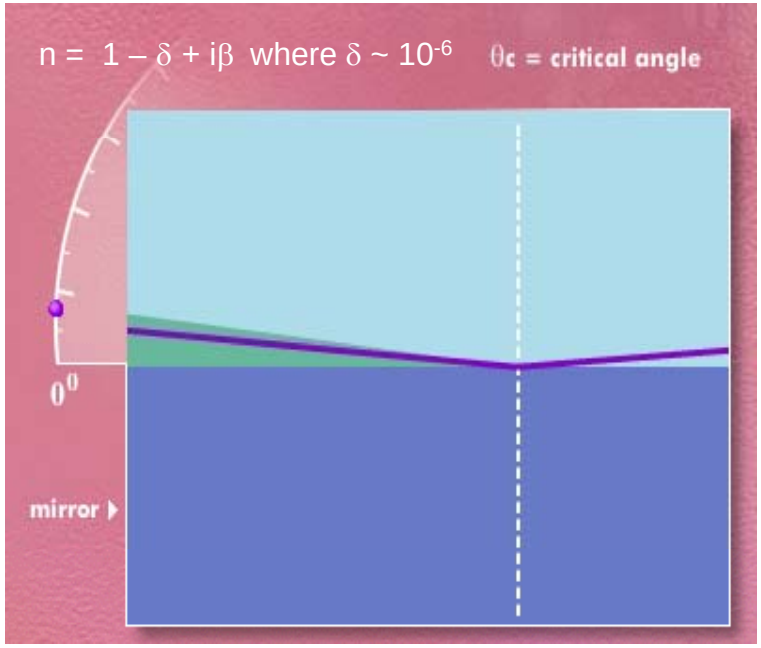


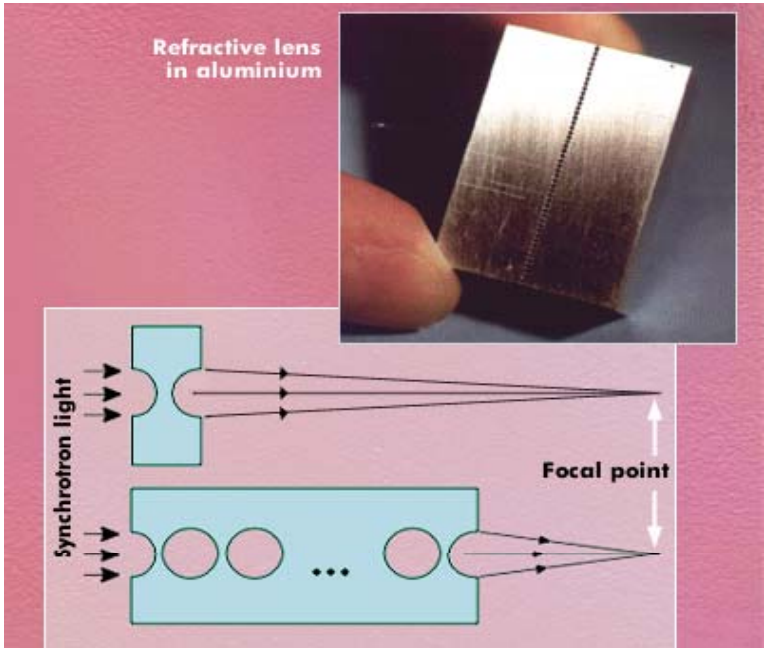
Industrial users represent 7% of the user population

Focusing X-Rays









At NSLS focus hard x-rays to a few microns, at NSLS-II to a few nm

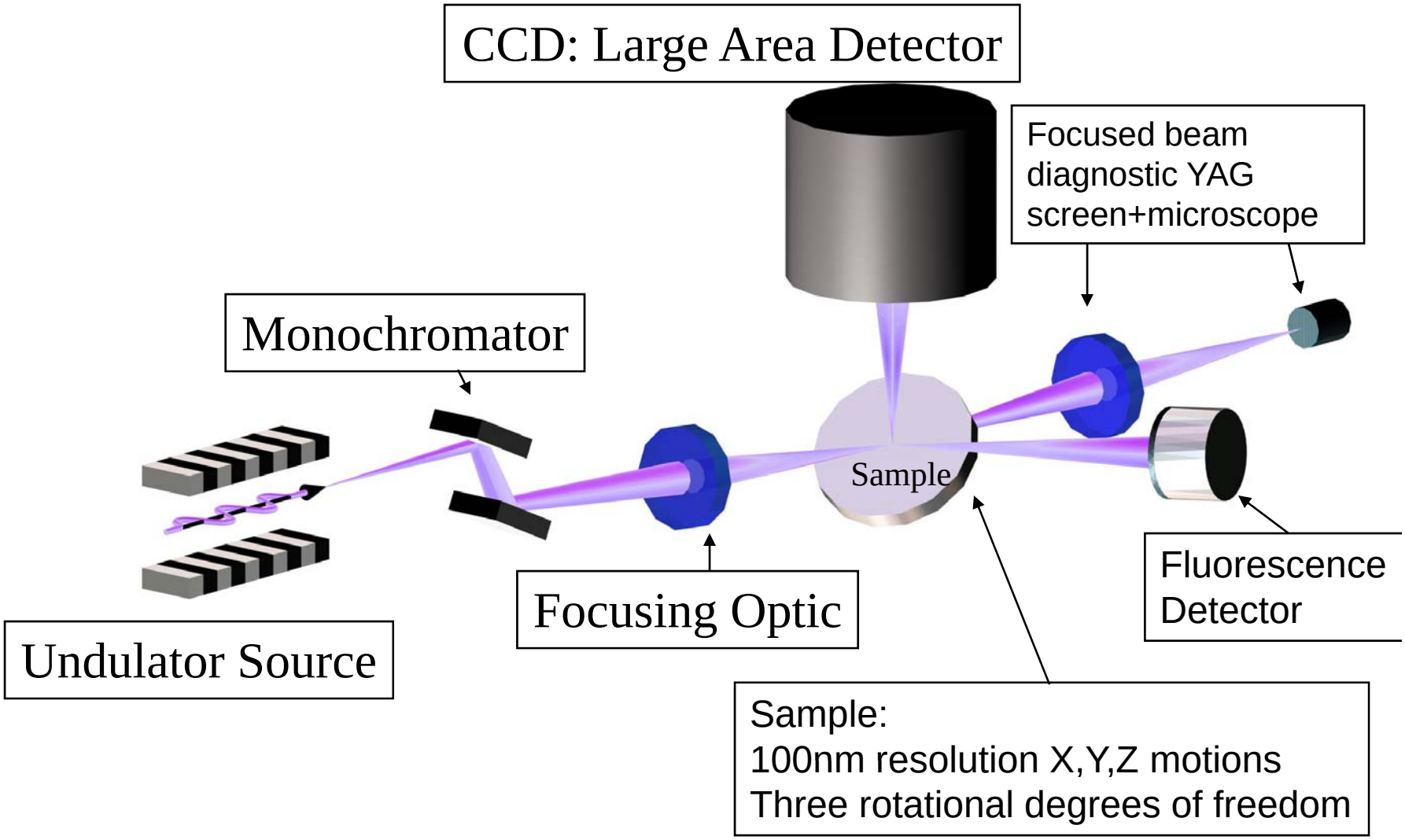
Examples of Nanoscience Research at the NSLS

http://www.nsls.bnl.gov/newsroom/science/

2007

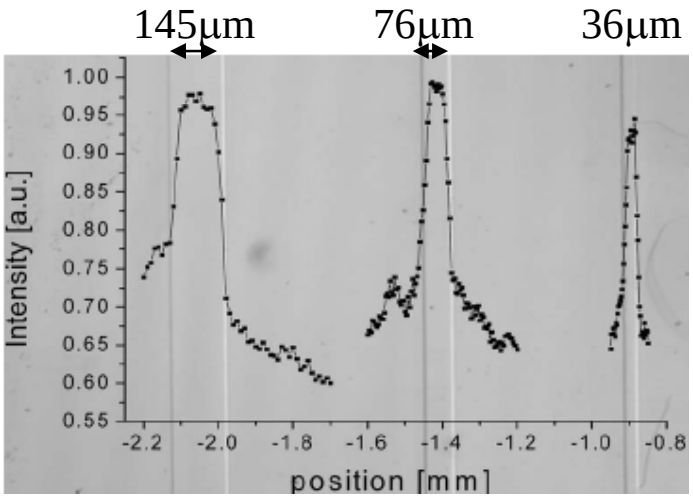
- Progress in Self-Assembly: High Resolution NEXAFS Study of the Wave-like Growth of Self-Assembling SAM Layers on a Silicon Substrate
- New DNA-Based Technique For Assembly of Nano- and Micro-sized Particles
- Quantitative Characterization of the Morphology of Multiwall Carbon Nanotube Films by Small-Angle X-ray Scattering
- Tiny Tubes and Rods Show Promise as Catalysts, Sunscreen
- Platonic Solids on the Nanoscale
- Solution-Phase Deposition and Nanopatterning of GeSbSe Phase-Change Materials
- SEMATECH and NIST Collaborate on Chemical Analysis of Advanced Gate Dielectrics

The X13B Micro - Diffraction Instrument



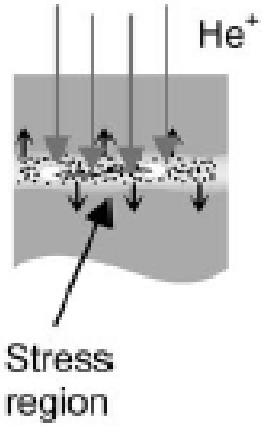
X-Ray Microdiffraction Strain Mapping

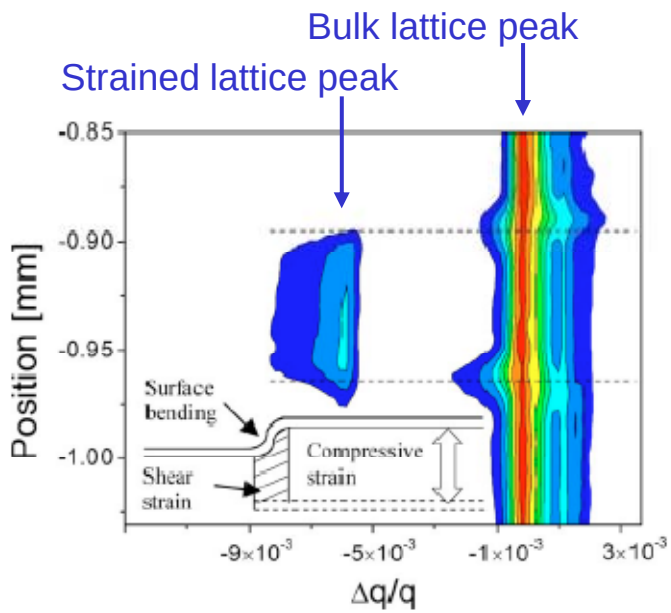
$LiNbO_3$ sample with He^+ implanted channels (depth ~1.6mm)



Superposition of Nomarski micrograph and diffracted intensity at strained lattice d-spacing.

X-ray beam was 1μm (scan direction) x 4μm

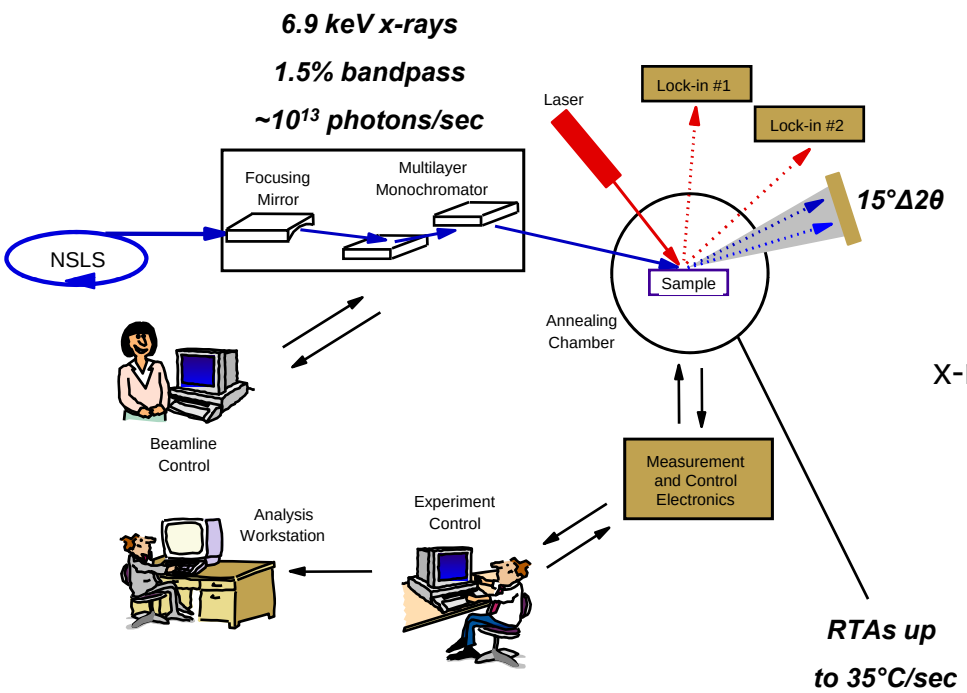


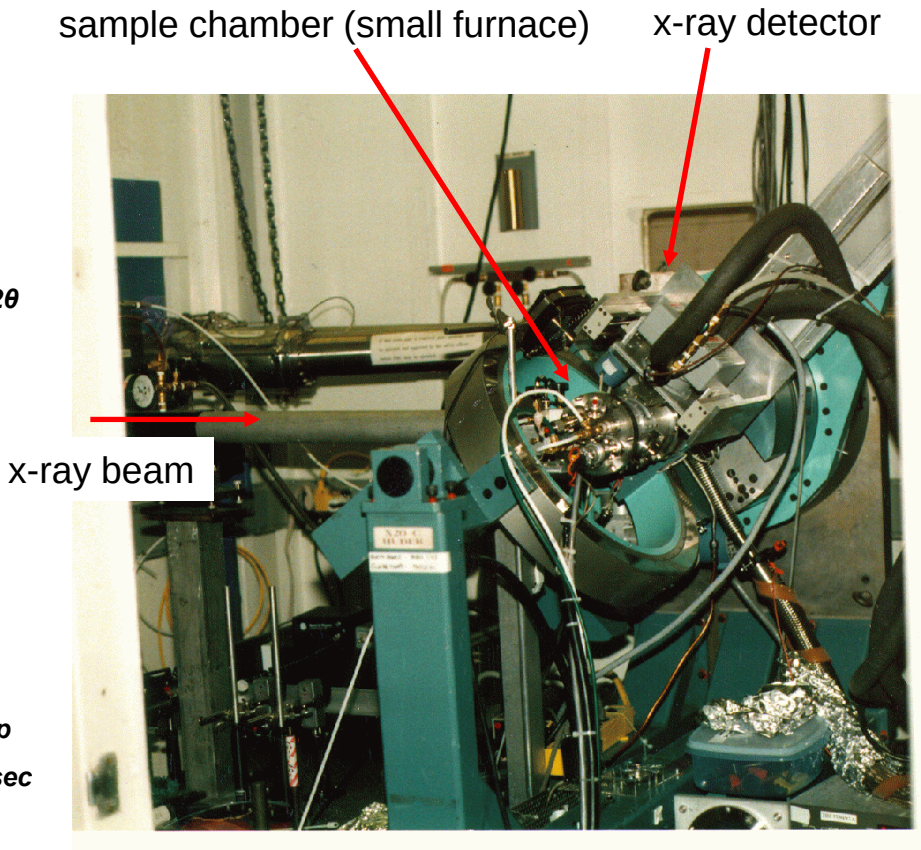


Dashed-lines are channel boundaries, color scale indicates x-ray intensity

D. Djukic, R. Roth, R. Osgood, K. Evans-Lutterodt, H. Bakhru, S. Bakhru, Appl. Phys. Lett. **91**, 112908 (2007).

Time Resolved X-ray Measurements at NSLS X20C





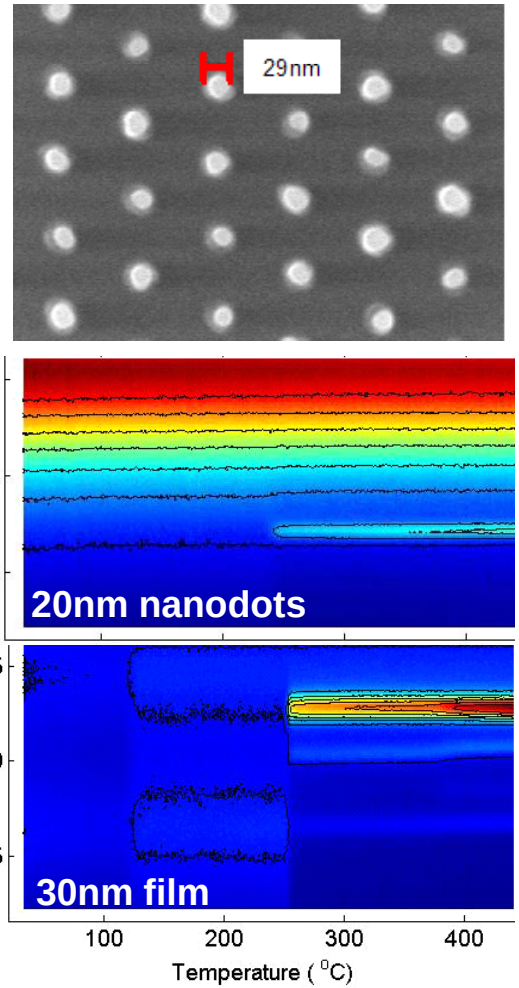
- Rapid, in-situ characterization of materials- either blanket film or structures (nanodots!)
  - X-ray Diffraction - phase transformations
  - Resistivity- phase change, agglomeration, or intermixing
  - Light scattering- surface morphology changes

IBM

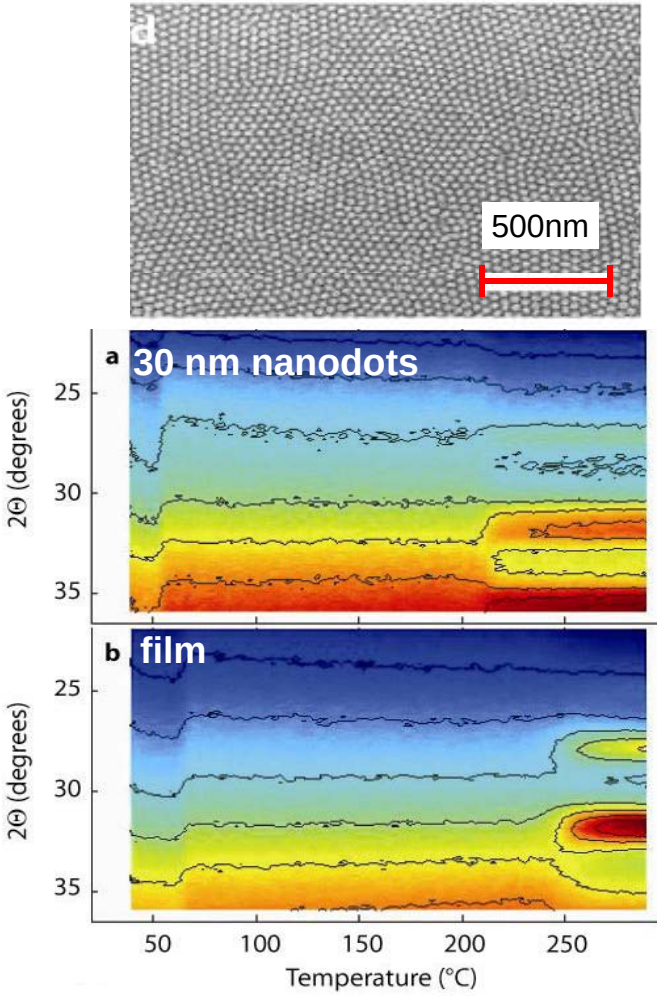
Nanodot Arrays to Test New Solid State Memory Materials

(to see if scaling retains desired crystallization behavior)

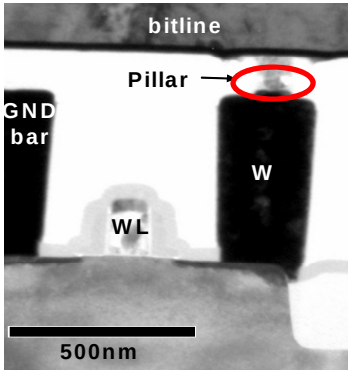
GeSb nanodots made by e-beam lithography, covered with 10nm Al_2O_3



30 nm GeSbSe nanodots made by spinning solution into self-assembled polymer template



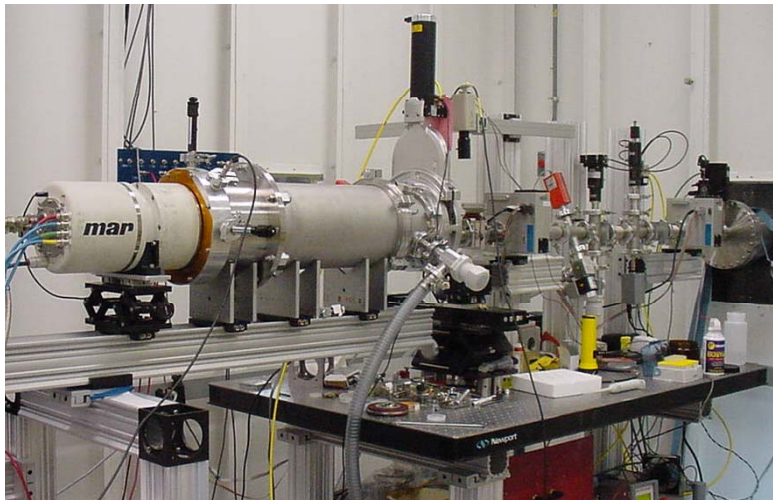
Phase-Change Memory!
faster, cheaper,
smaller, lower power than flash, DRAM, etc.

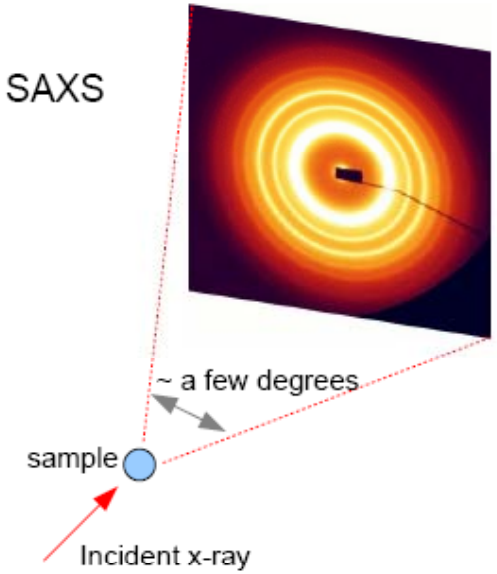


IBM

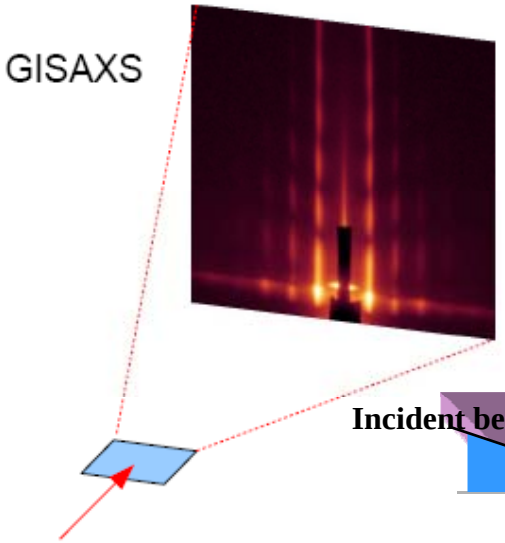
D. Milliron, S. Raoux, R. Shelby, and J. Jordan-Sweet. "Solution-Phase Deposition and Nanopatterning of GeSbSe Phase-Change Materials,"*Nat. Mater.* **6,** 352-356 (2007).

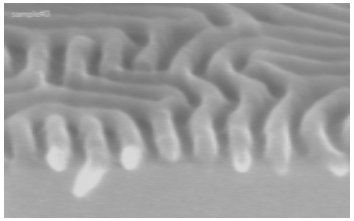
Studying Nanoparticles and Nanopatterns with SAXS / GISAXS

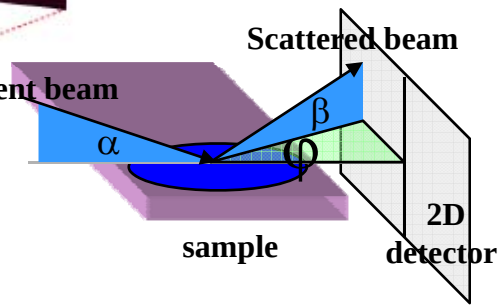




Powder/solution samples are measured in the transmission geometry. The scattering pattern is isotropic.





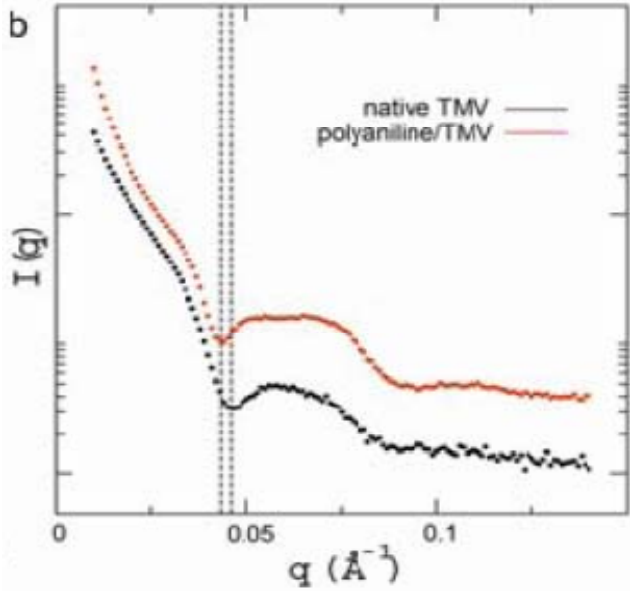


Thin film samples are measured in the grazing incidence geometry. The structure along the film normal can also be studied with X-ray reflectivity.

SAXS Characterization of Single Particles

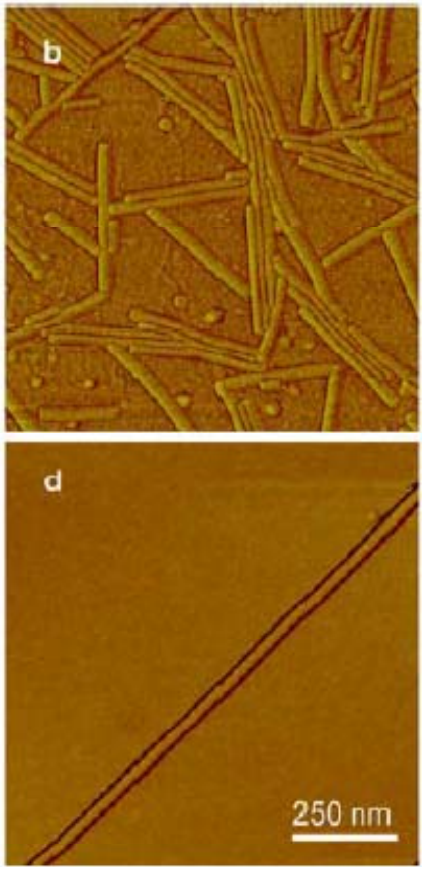
TMV Nanofibers

The coating on TMV nanofibers is not discernible in EM or solution scattering, but causes difference in scattering pattern when embedded in a dense medium (silica gel).



Z. Niu, M. Bruckman, V. Kotakadi, J. He, T. Emrick, T. Russel, L. Yang, and Q. Wang, *Chem. Commun.* 3019 (2006)

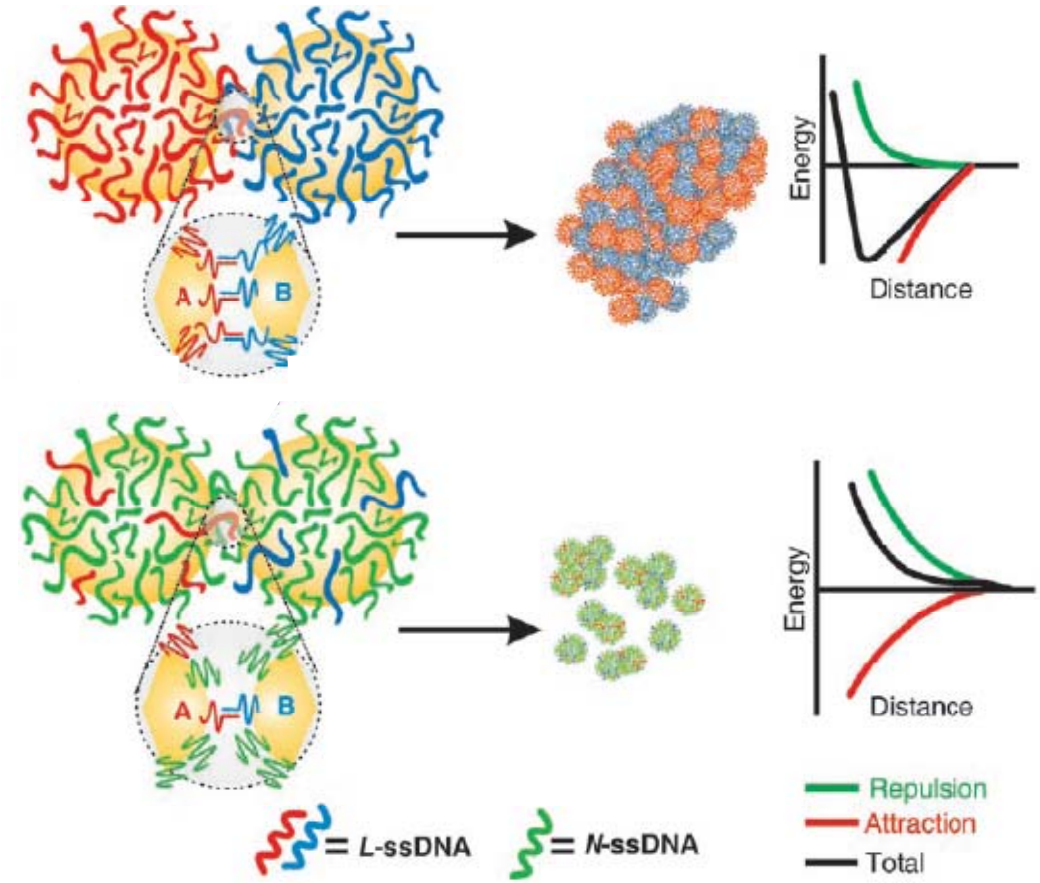
Native TMV



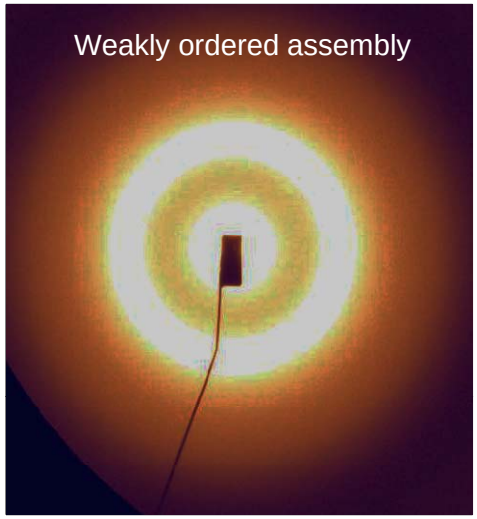
TMV nanofiber

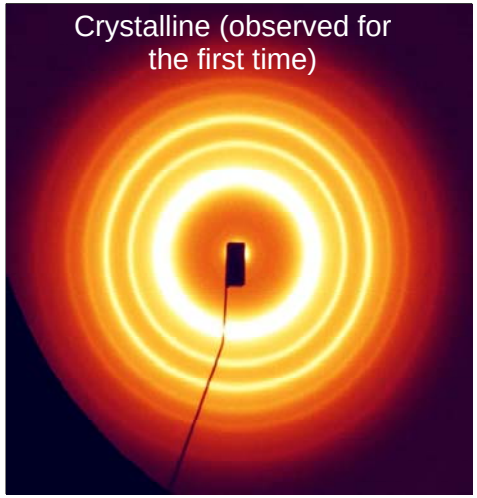
Ordered Structures Assembled in Solution

DNA-mediated assembly of Au or PS nanoparticles



SAXS Patterns

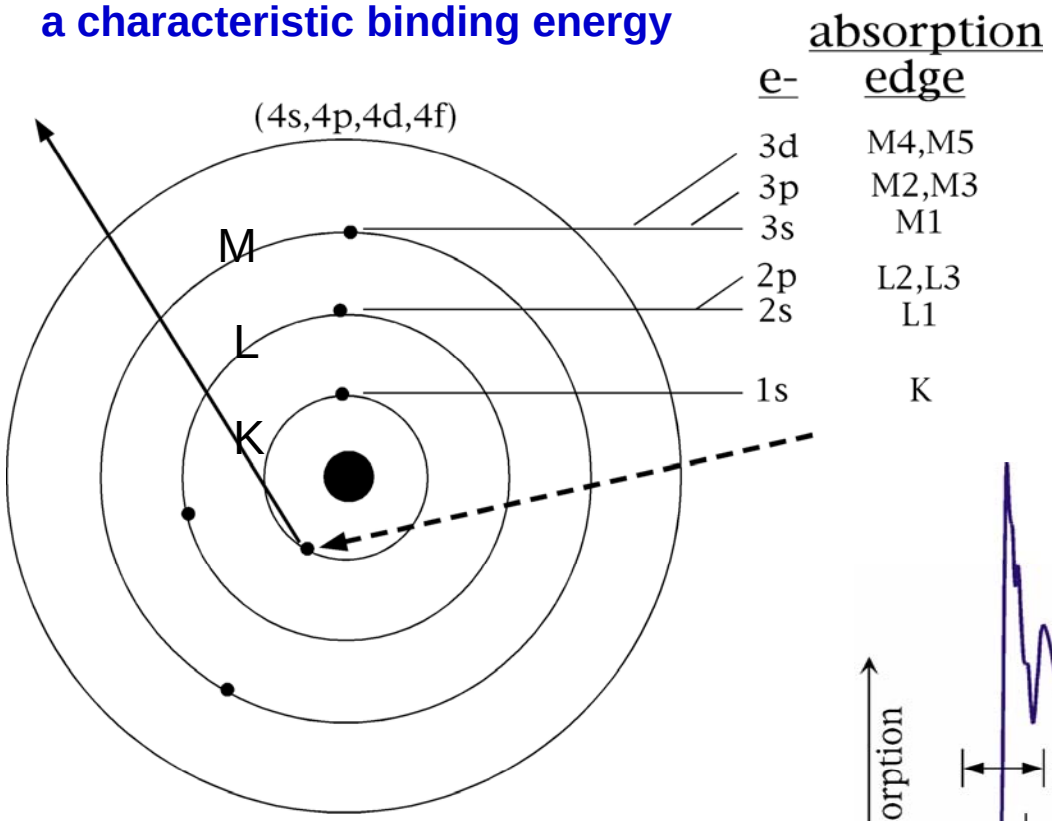


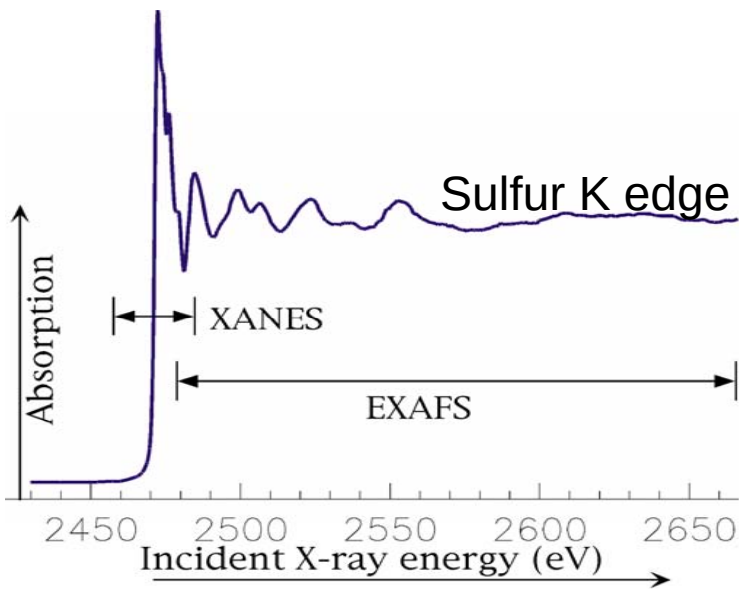


M. Maye, D. Nykypanchuk, D. van der Lelie, and O. Gang, *Small* **3**, 1678 (2007).

X-Ray Absorption Spectroscopy

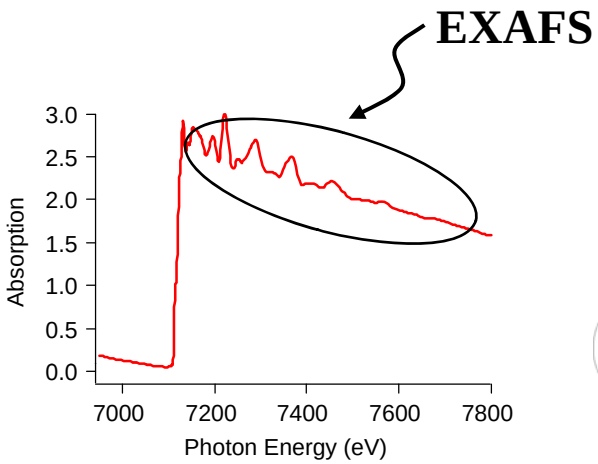
Each edge of each element has a characteristic binding energy

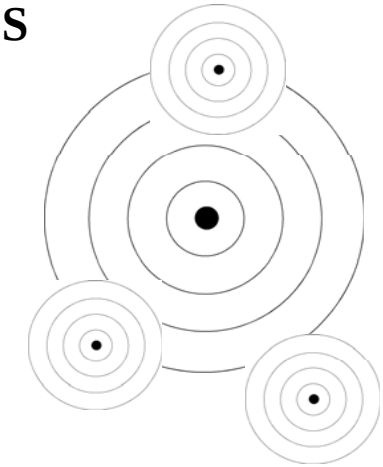


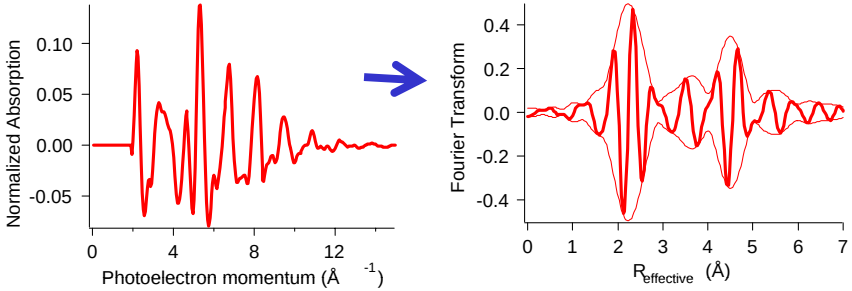


Absorption occurs when the energy of the incident photon is sufficient to eject the electron.

EXAFS from Nanoparticles : Technique

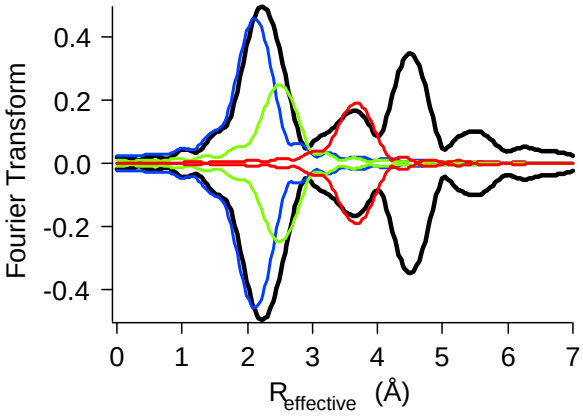


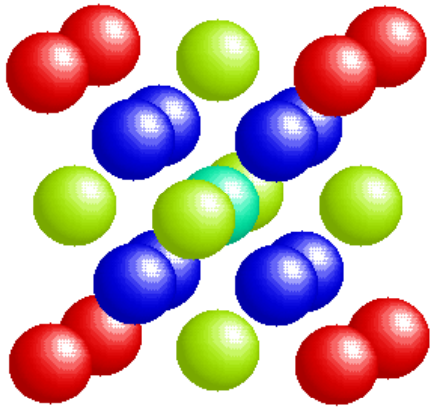




x-ray absorption (XAS) spectra

which, after data reduction and processing.

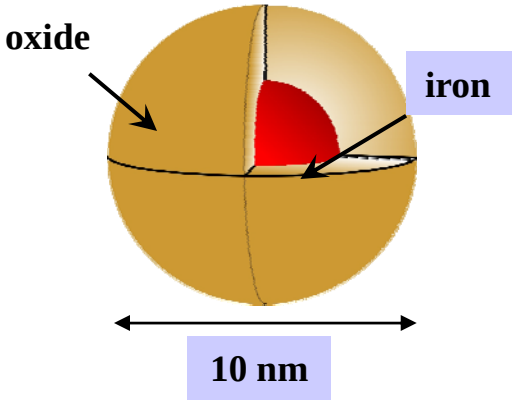




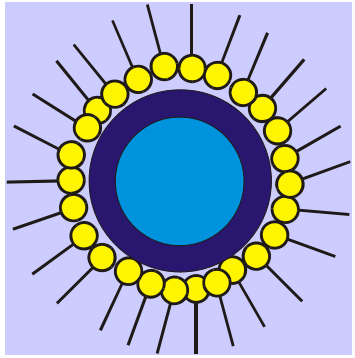
…correspond to atomic structure on a subnanometer scale.

EXAFS from Nanoparticles : Core-Shell Particles

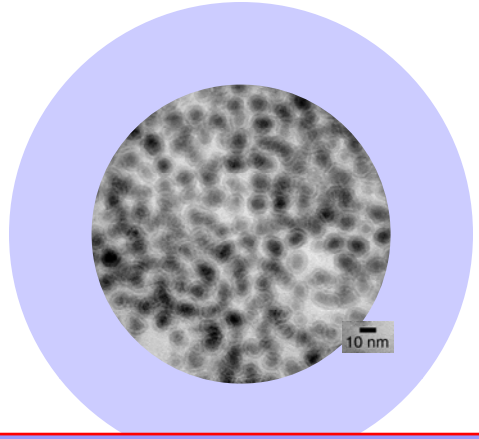
Prof. Scott Calvin, Sarah Lawrence College, Bronxville, NY & collaborators at Naval Research Lab



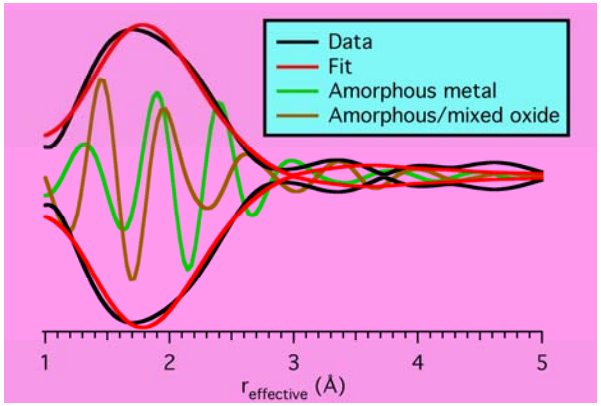
Goal: Air-stable (passivated) nanoparticles with iron cores



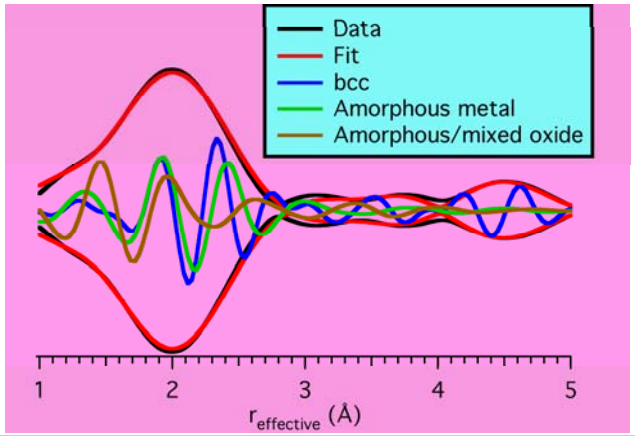
Synthesized using reverse micelles (water in oil emulsions) as nanoreactors



...we obtained core/shell nano-particles of the desired size.



Non-ionic NP synthesis:
EXAFS fits → an amorphous or mixed oxide shell
on an amorphous (nearly close-packed) metallic iron core...



Cationic CTAB synthesis: nanoparticles contain crystallites of bcc iron as well.