National Synchrotron Light Source

Ron Pindak

Center for Functional Nanomaterials

Emilio Mendez

Why Synchrotron Measurements Might Benefit Your Research Programs
&
How to Access the NSLS Synchrotron Facilities

Ron Pindak

Physical & Chemical Sciences Division Head

National Synchrotron Light Source

Office of Science
U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
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Useful Web Sites

- Background information on synchrotron techniques as well as descriptions of capabilities, workshops, courses from synchrotron facilities worldwide:

  http://www.lightsources.org/

- Information specifically about the NSLS

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

  - NSLS staff web pages – http://www.nsls.bnl.gov/organization/

    Pindak's web page will contain a copy of this talk

  - NSLS science highlights - http://www.nsls.bnl.gov/newsroom/science/
  - NSLS user access - http://www.nsls.bnl.gov/users/access/
  - NSLS beamline descriptions - http://www.nsls.bnl.gov/beamlines/

- Questions? pindak@bnl.gov

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

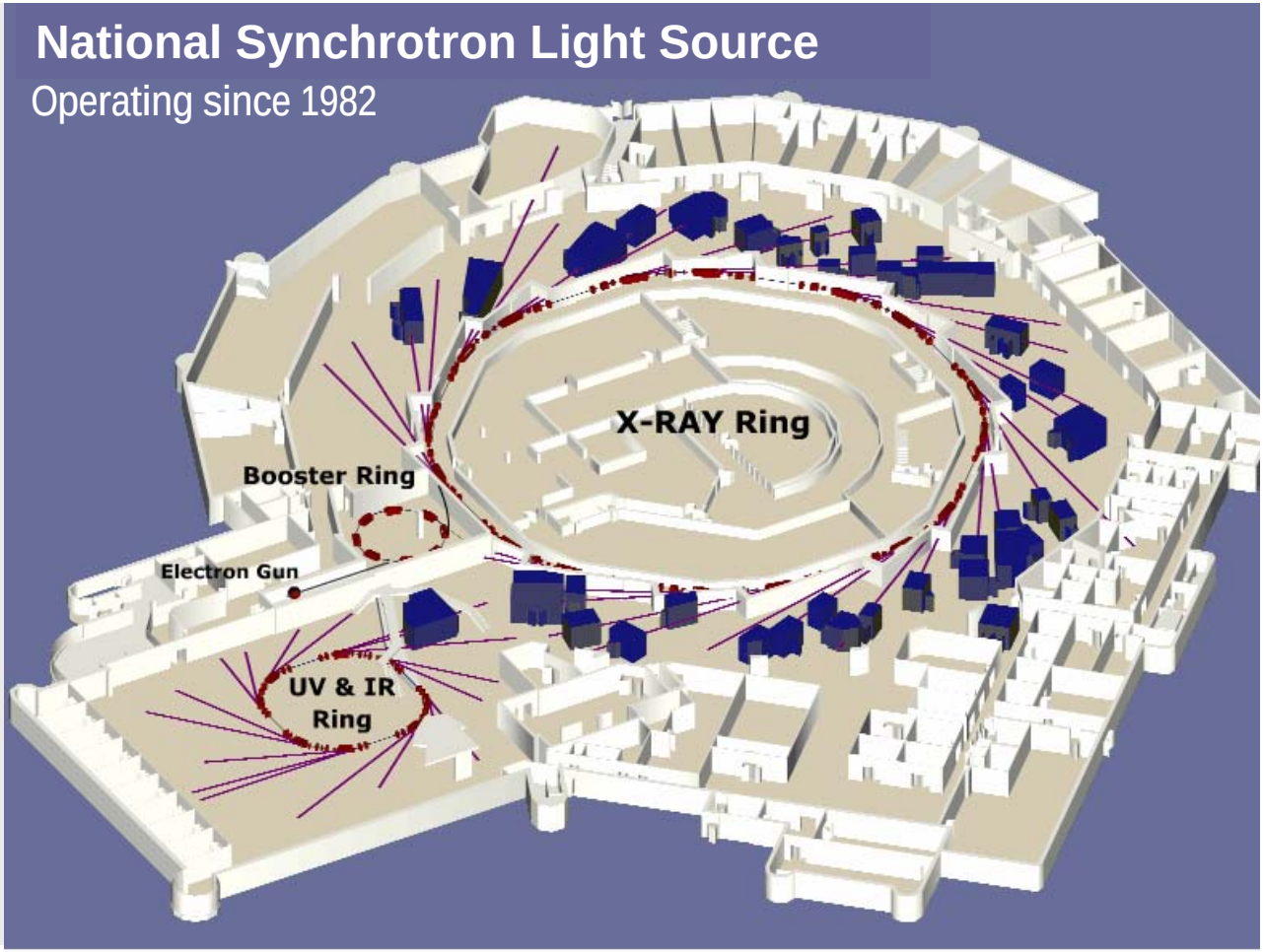
E = 2.8 GeV

I = 280 mA

25 bunches

Orbital Period = 567 ns

Pulse Width = 290 ps

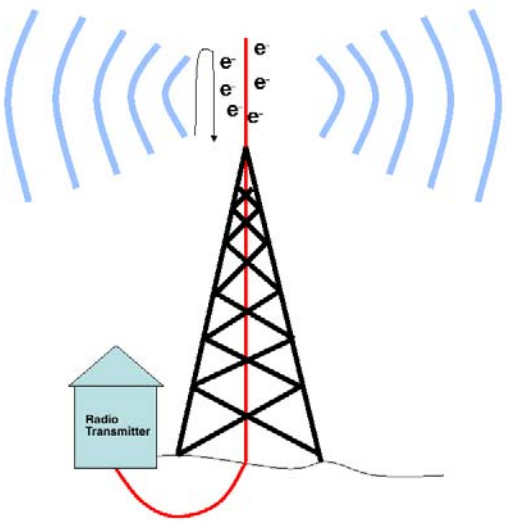


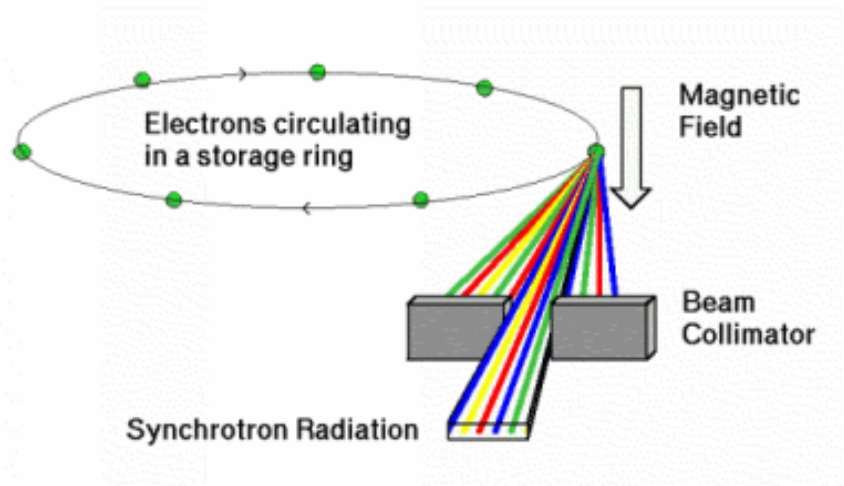
- Facility operates 24 hours/day, 7 days/week, ~10 months/year
- 65 operational beamlines available (>2100 users per year, ~1/3 are new users).
- ~ 60% of the beam time is available to users at no cost via peer-reviewed proposals.

Synchrotron Radiation

Electrons accelerating by running up and down in a radio antenna emit radio waves

Synchrotron radiation is electromagnetic radiation emitted when charged particles are radially accelerated (on a curved path)



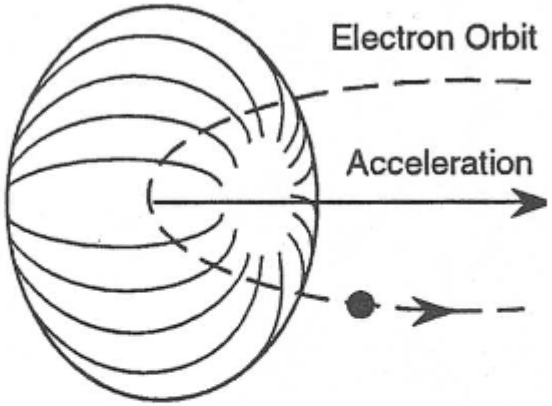


Both cases are manifestation of the same physical phenomenon:

Charged particles radiate when accelerated.

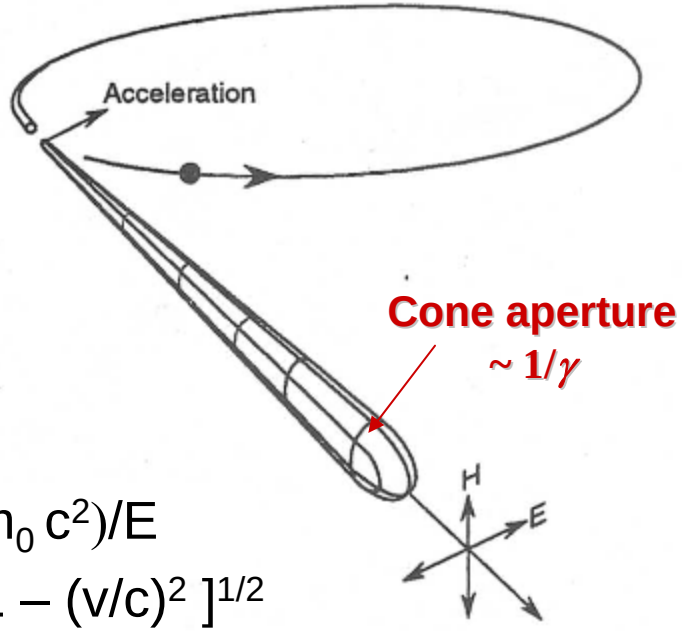
Accelerating Electrons at High Energies

v << c



At low electron velocity (non-relativistic case) the radiation is emitted in a non-directional pattern

v ~ c



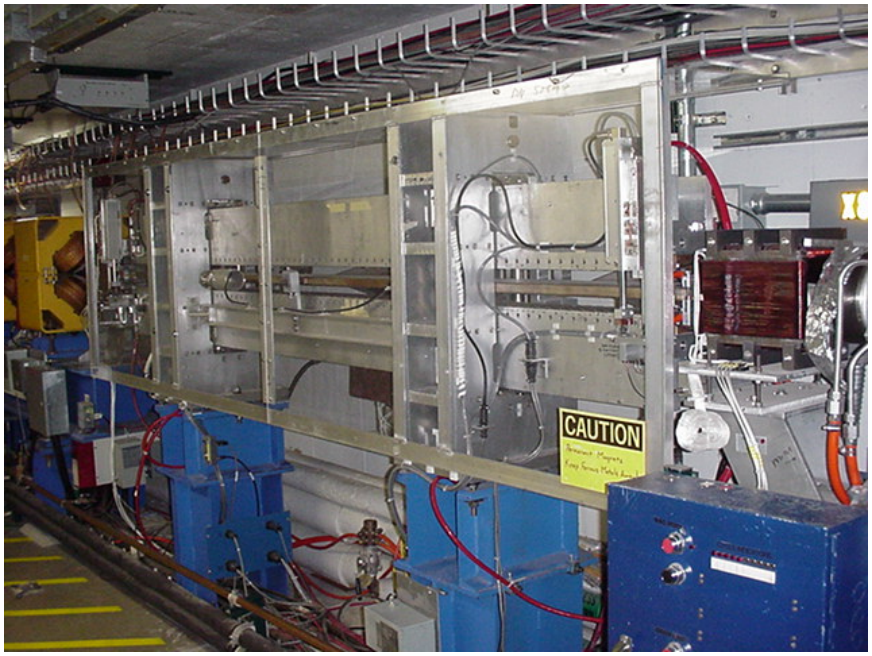
$\gamma^{-1} = (m_0 c^2)/E$

$= [\, 1 - (v/c)^2 \,]^{1/2}$

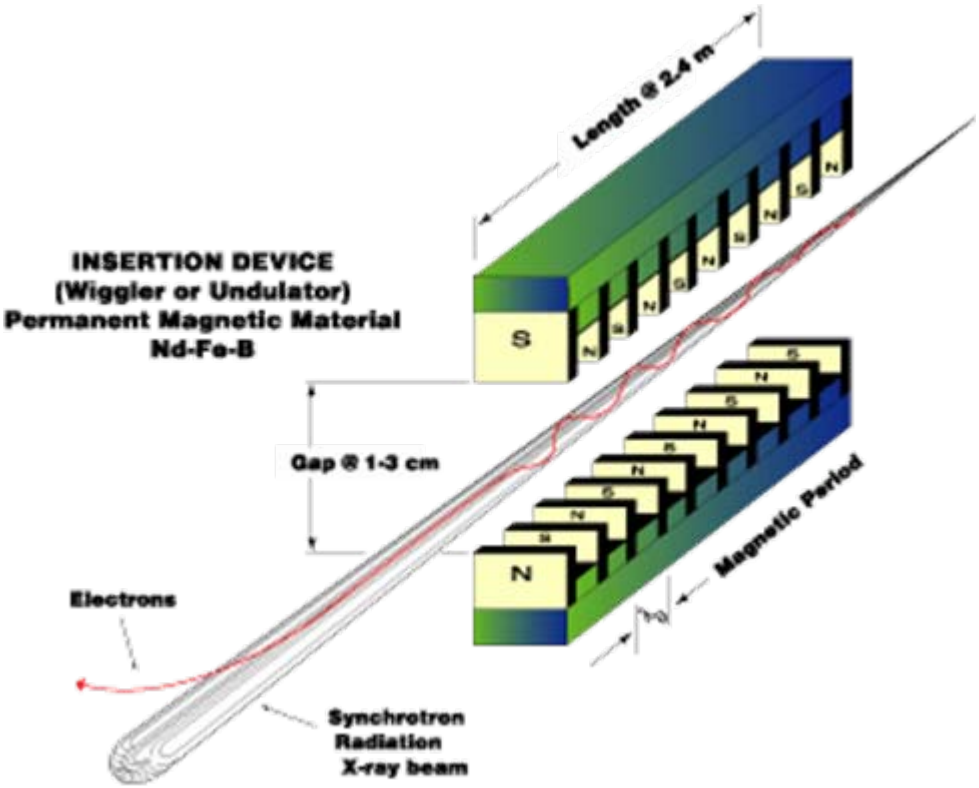
When the electron velocity approaches the velocity of light, the emission pattern is folded sharply forward.

Radiation becomes more focused at higher energies (velocities)

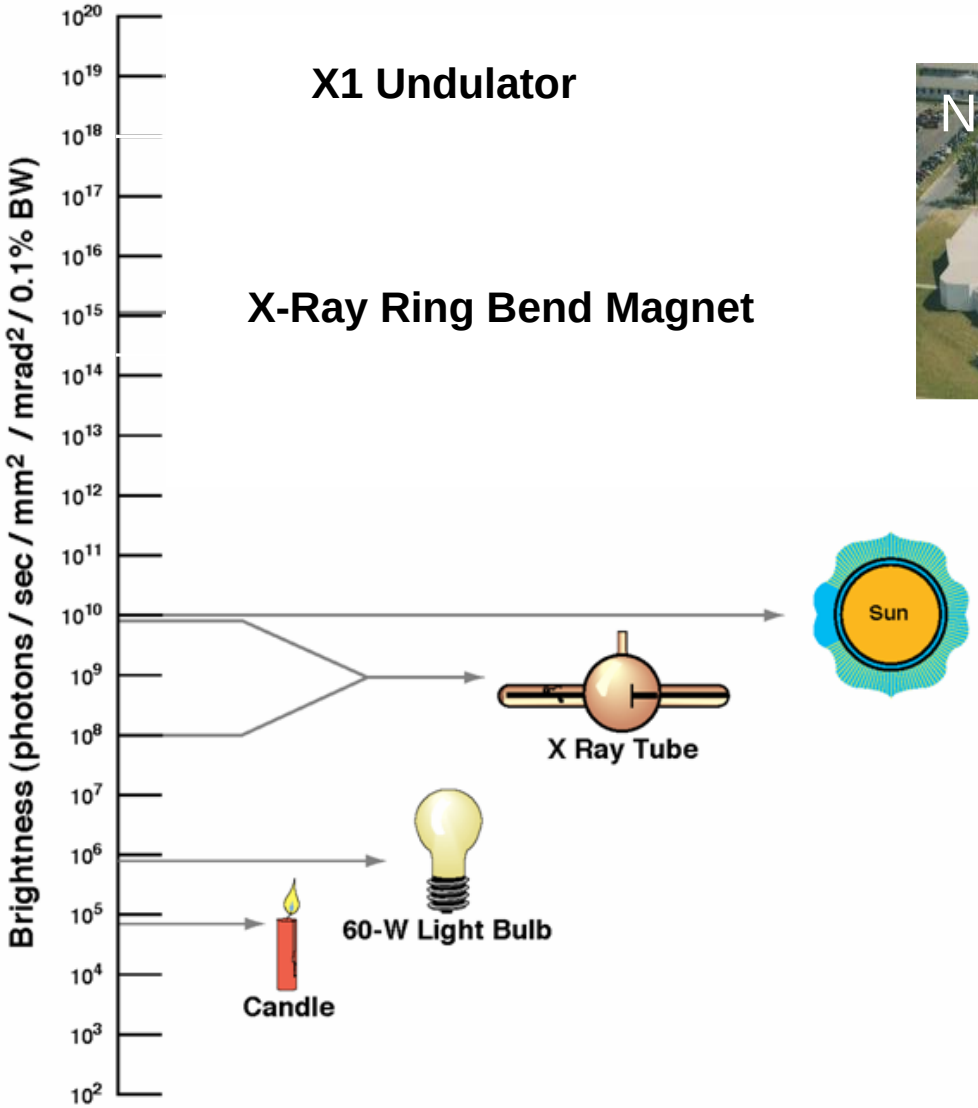
Undulator Insertion Device



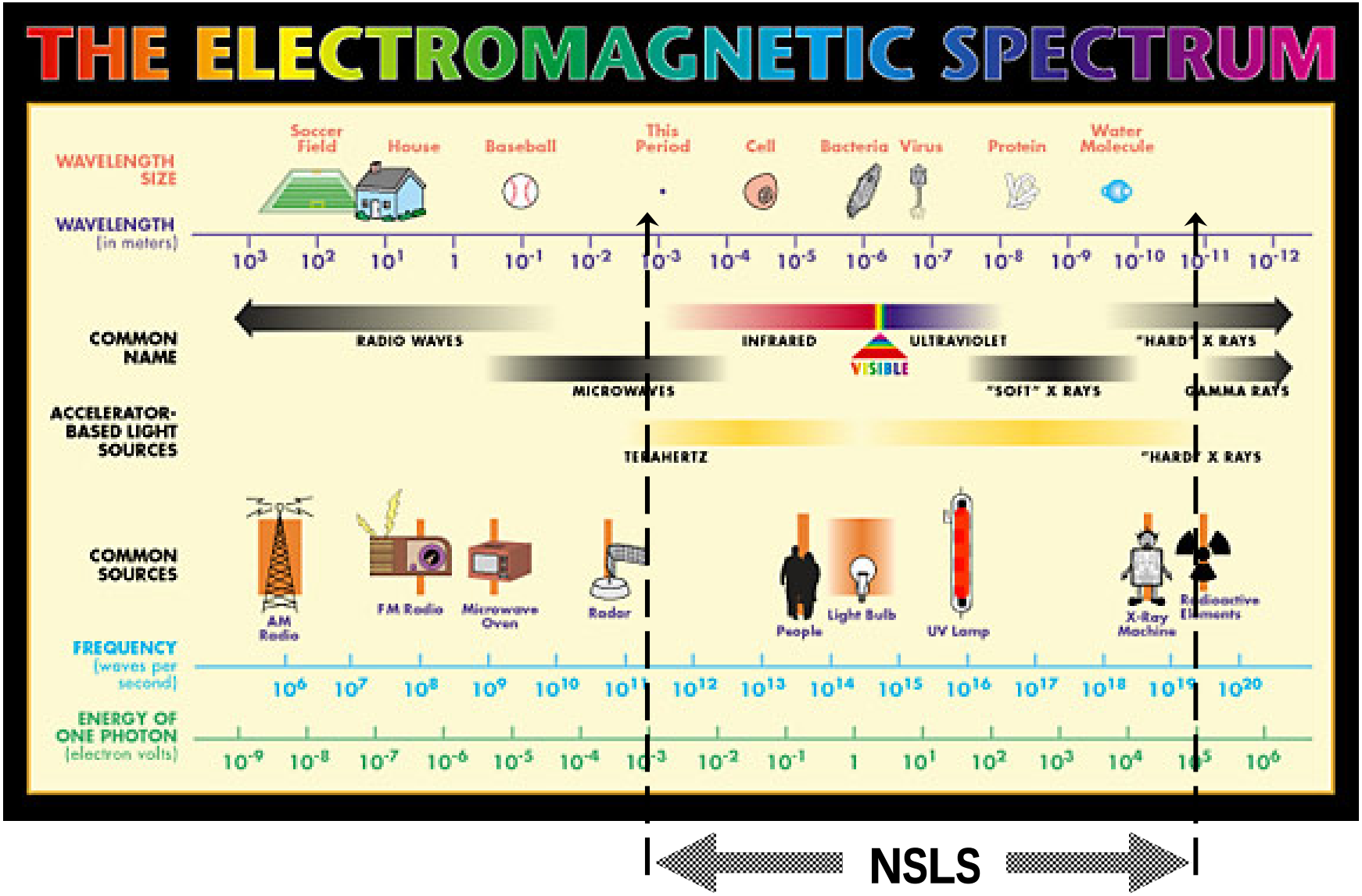
Soft X-Ray Undulator – X1



Synchrotron Brightness



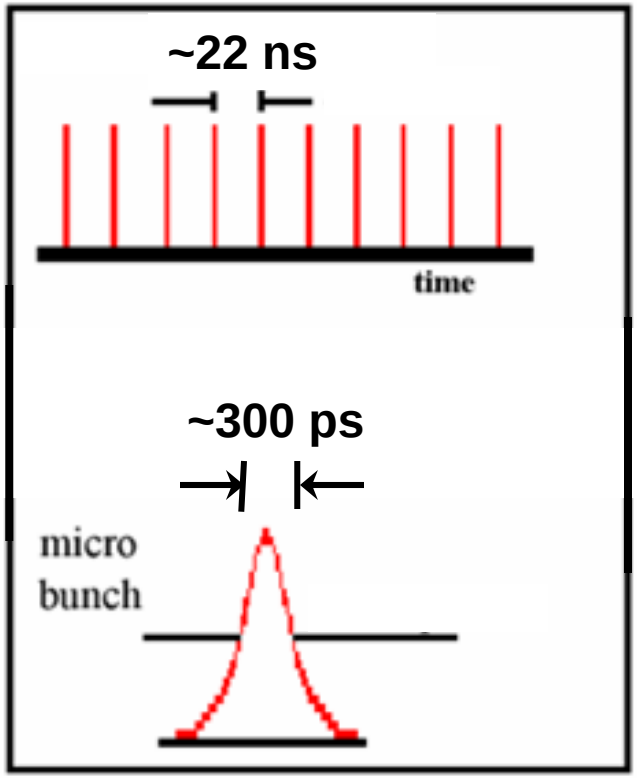
NSLS Beamlines Cover 8 Decades of the EM Spectrum
Individual Beamlines Optimized & Tunable for a Select Range



Properties of the NSLS Synchrotron Source

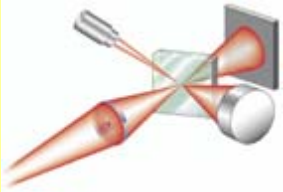
- High brightness and flux
- Very broad energy spectrum
- Highly polarized and short pulses
- Partial coherence
- High stability

Time structure

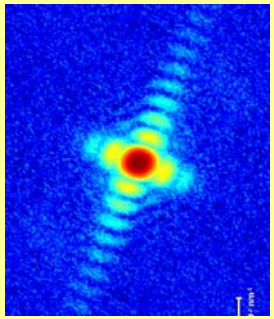


NSLS-II
The Future National Synchrotron Light Source

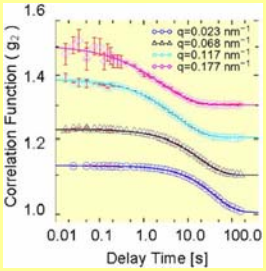
New Capabilities



Nanoprobes



Diffraction Imaging



Coherent Dynamics

Ultra-Bright Medium Energy Storage Ring

Medium Energy (3 GeV) Large Current (500 mA)

792 m in Circumference (>4x present NSLS x-ray ring)

Brightness ~ 10^{21} ph/s/0.1%/mm^2/mrad^2 (> 10^4 x present NSLS)

Flux ~ 10^{16} ph/s/0.1% (>20x present NSLS)

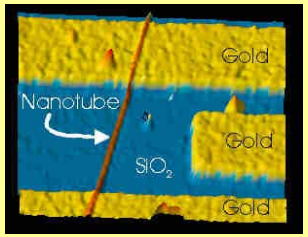
21 Insertion Devices (2.3x number of IDs at the present NSLS)

10 psec pulses (30x shorter than present NSLS)

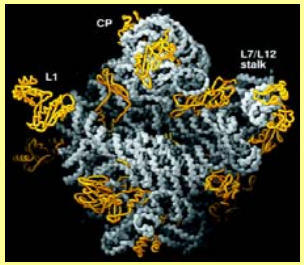
Project Goals:

- **Spatial resolution of 1nm**
- **Energy resolution of 0.1 meV**

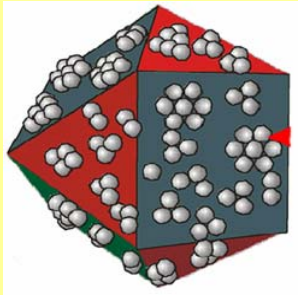
New Science



Nanoscience



Life Science



Nanocatalysis

Proposed to begin operations in 2014

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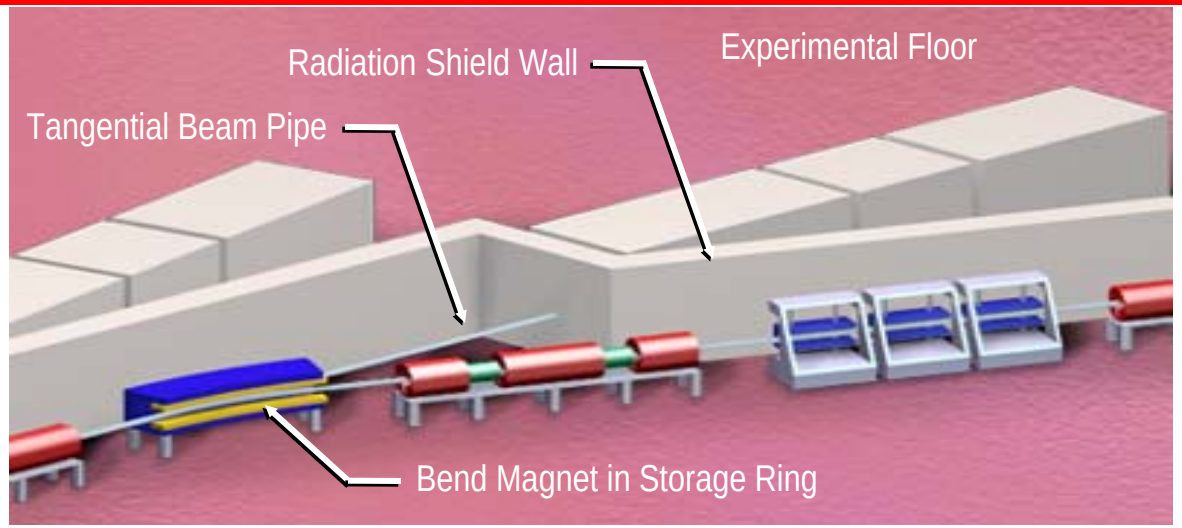
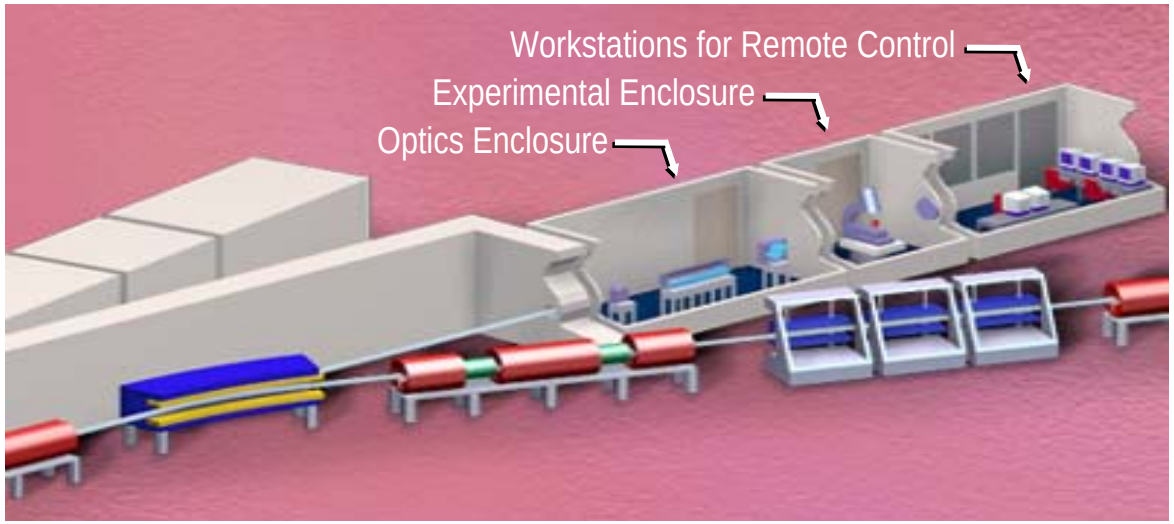
Extracting X-Ray Radiation from the Storage Ring





Major Synchrotron Techniques

SPECTROSCOPY

- Infrared spectroscopy
- Photoelectron spectroscopy
- X-ray absorption spectroscopy
- X-ray emission spectroscopy

DIFFRACTION/SCATTERING

- Protein crystallography
- Small molecule crystallography
- Powder diffraction
- Small-angle x-ray scattering
- X-ray microdiffraction
- High momentum resolution x-ray scattering

IMAGING

- Infrared microspectroscopy
- Soft X-ray scanning microscopy
- Hard X-ray microprobe
- X-ray microtomography
- Diffraction-enhance imaging

Distribution of Operating Instruments

- Macromolecular Crystallography
  X3A, X4A, X4C, X6A, X8C, X12B, X12C, X25, X26C, X29, X28C

- Imaging and Microprobes
  U2B, U10B, X1A1, X1A2, X2B, X13B, X15A, X26A, X27A

- X-Ray Scattering & Diffraction
  X1B, X6B, X9, X10A, X10B, X13A, X20A, X20C, X21, X22A, X22B, X22C, X27C

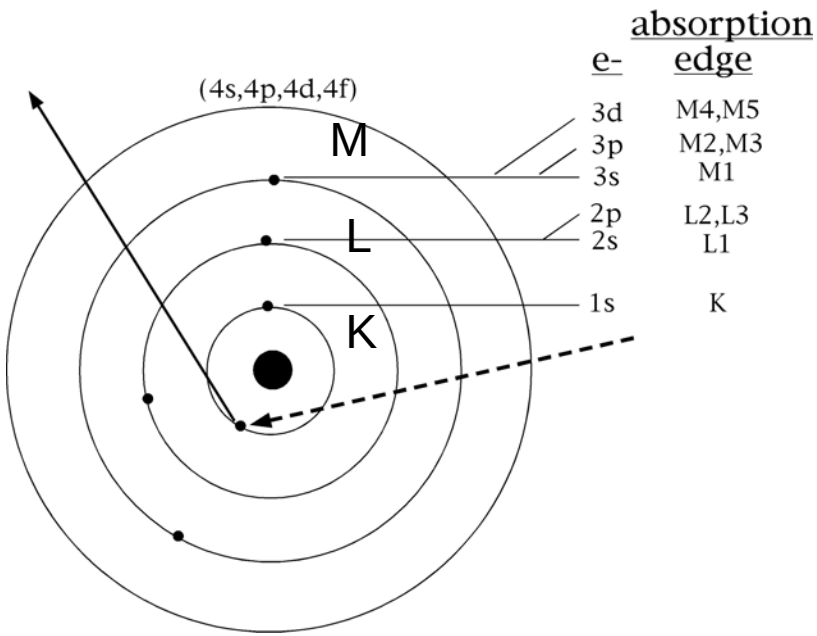
- Powder/Single Crystal Diffraction & High Pressure
  U2A, X7B, X14A, X16C, X17B1, X17B2, X17B3, X17C, X18A, X19C

- X-ray Spectroscopy
  X3B, X10C, X11A, X11B, X15B, X18B, X19A, X23A2, X23B, X24A, X27B

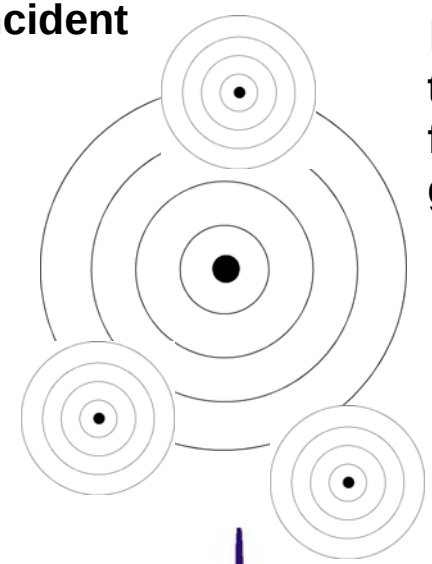
- IR, VUV and Soft X-ray Spectroscopy
  U1A, U3C, U4A, U4B, U5UA, U7A, U9B, U11, U12A, U12IR, U13UB, X8A, X24C

X-Ray Absorption Spectroscopy

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

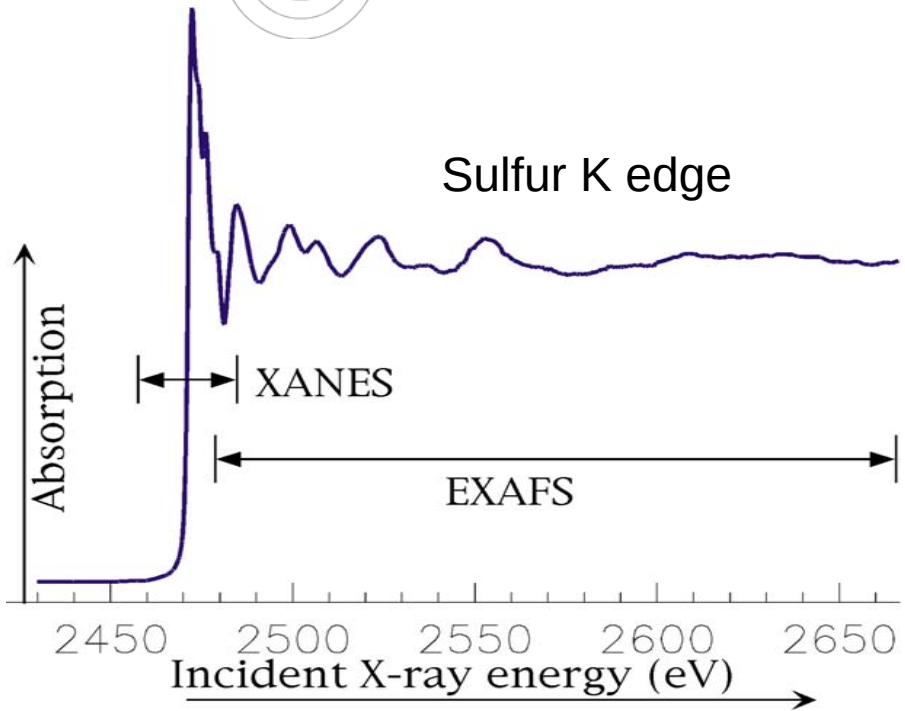


Each edge of each element has a characteristic binding energy

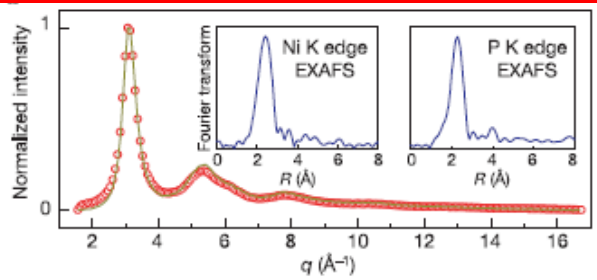


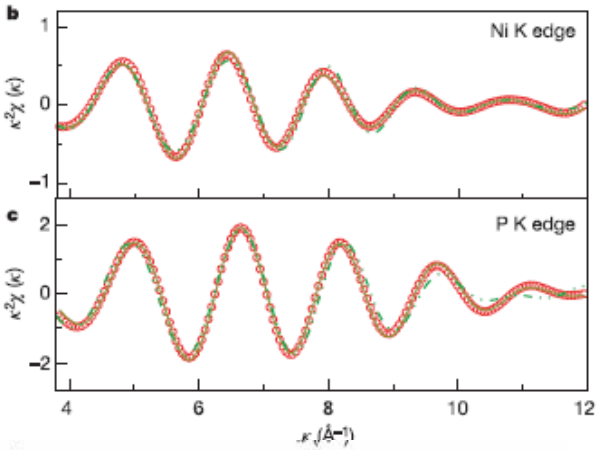
Extended oscillations due to backscatter of electron from neighboring atoms giving information on:

- **Distance**
- **What element (size)**
- **Coordination number**
- **positional disorder**

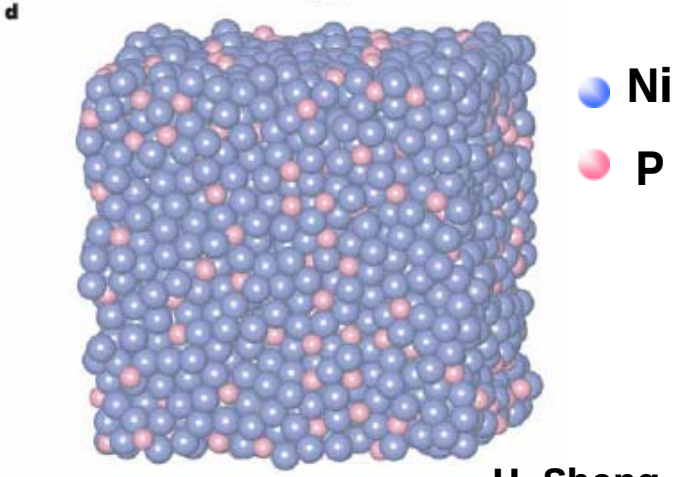


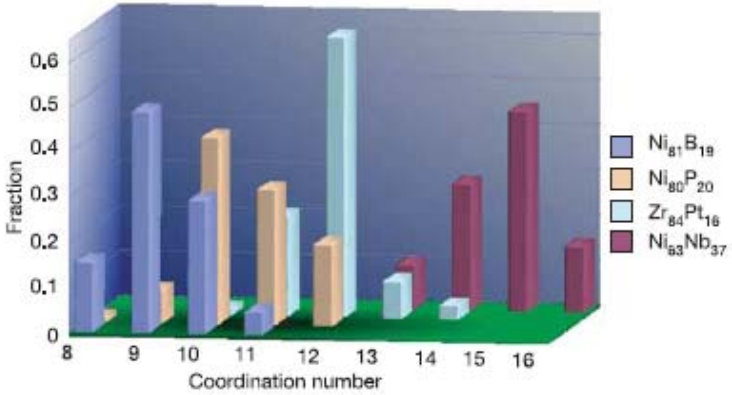
EXAFS: Short-to-Medium-Range Order in Metallic Galasses

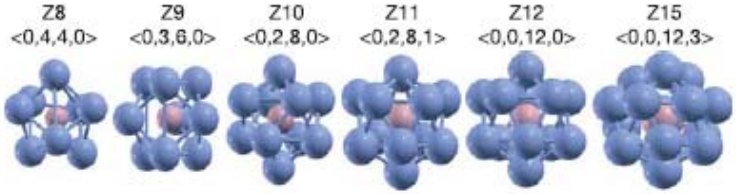




Transmission-mode Ni k-edge EXAFS and fluorescence-mode P k-edge EXAFS combined with X-Ray Diffraction and Reverse Monte-Carlo Modeling to determine ordering in a $Ni_{80}P_{20}$ metallic glass



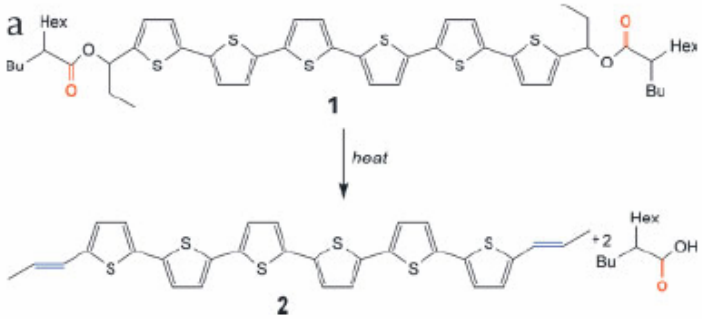


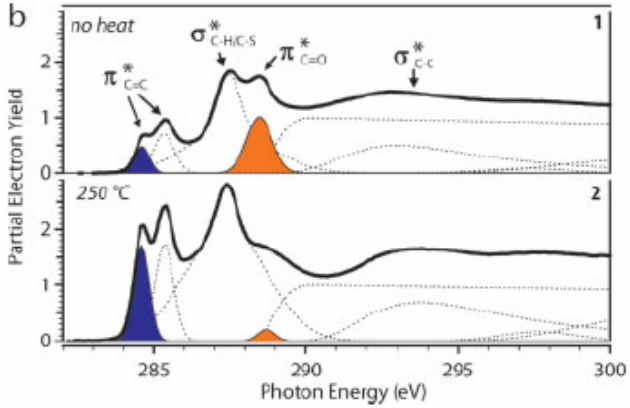


Distribution of different Kasper polyhedra

H. Sheng, W. Luo, F. Alamgir, J. Bai & E. Ma, Nature 439, 419 (2006)

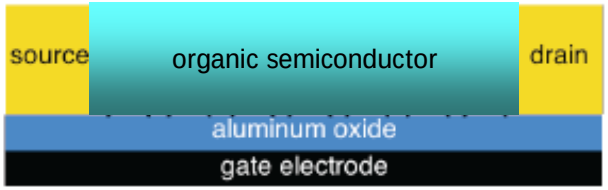
Soft X-Ray NEXAFS: Direct Correlation of Organic Semiconductor Structure to Field-Effect Mobility

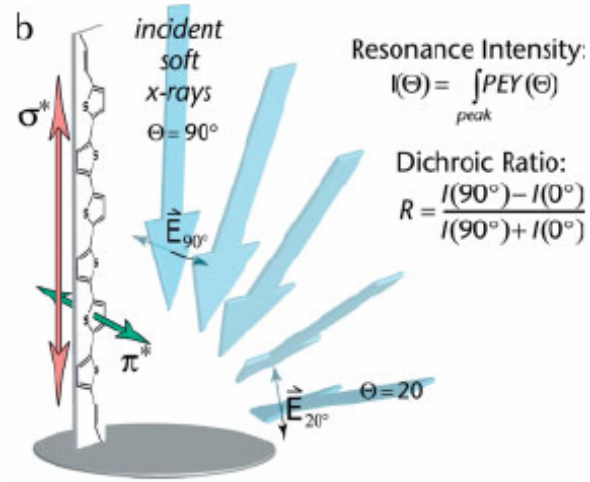


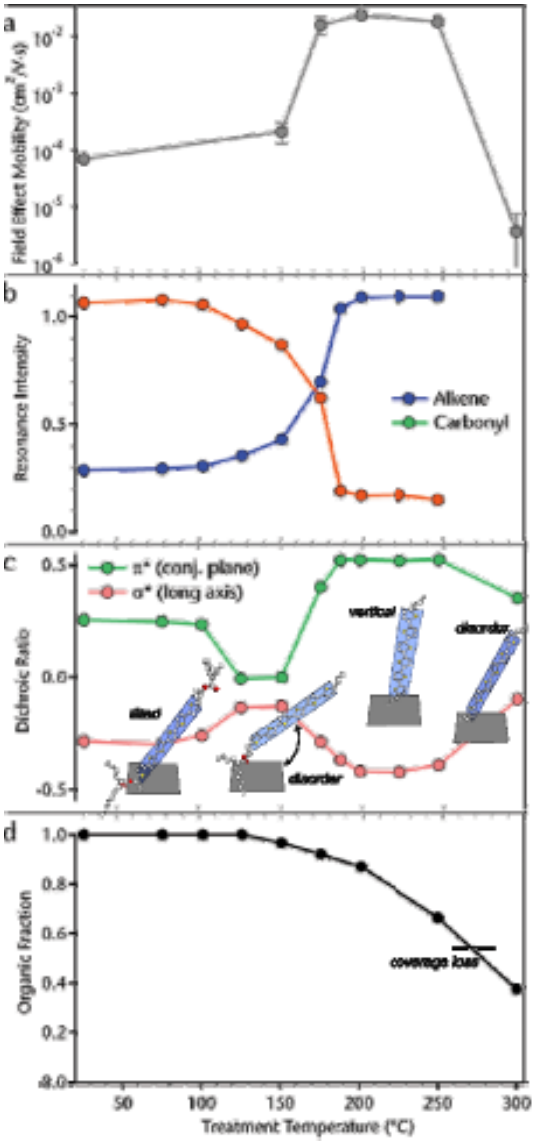


NEXAFS spectra for soluble precursor and thermal annealed organic semiconductor

Organic Field-Effect Transistor

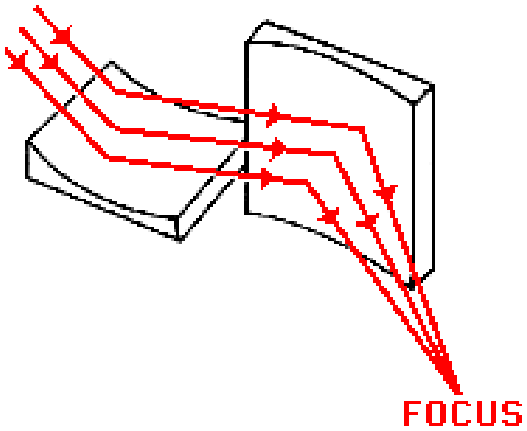


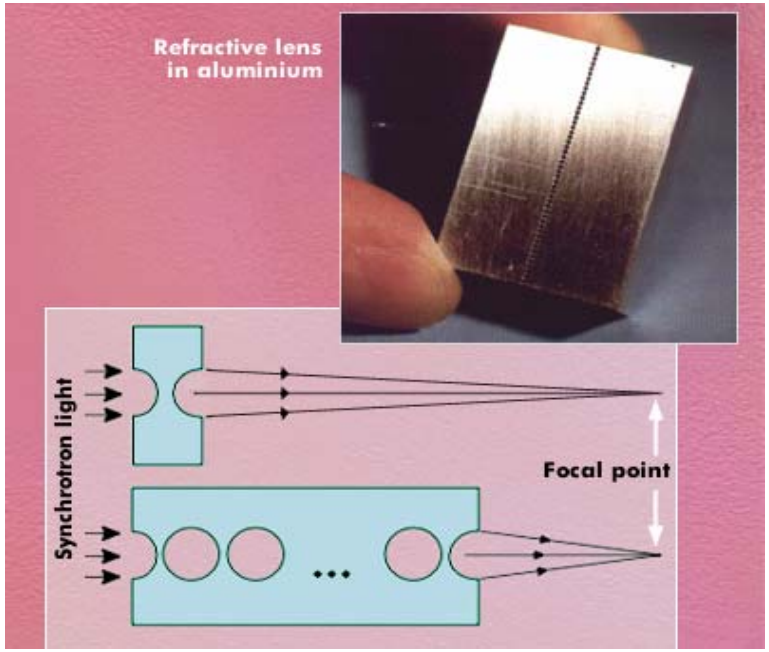


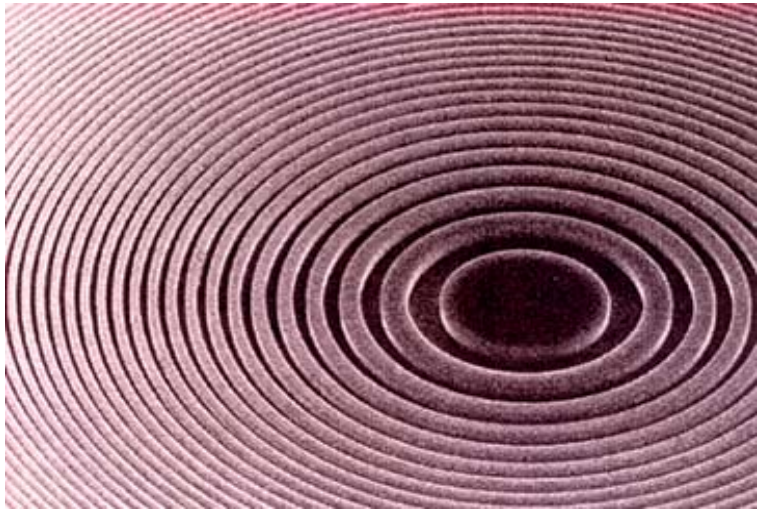


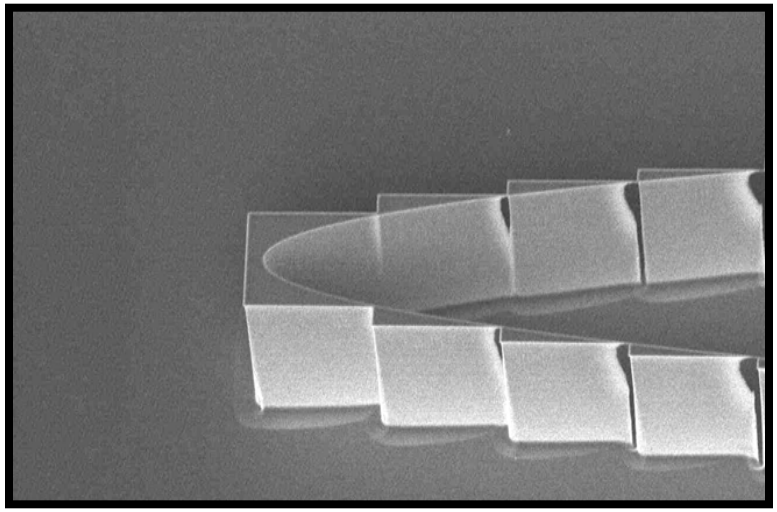
D. DeLongchamp, et al., Advanced Materials 17, 2340 (2005)

Focusing X-Rays

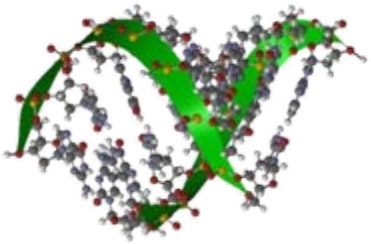






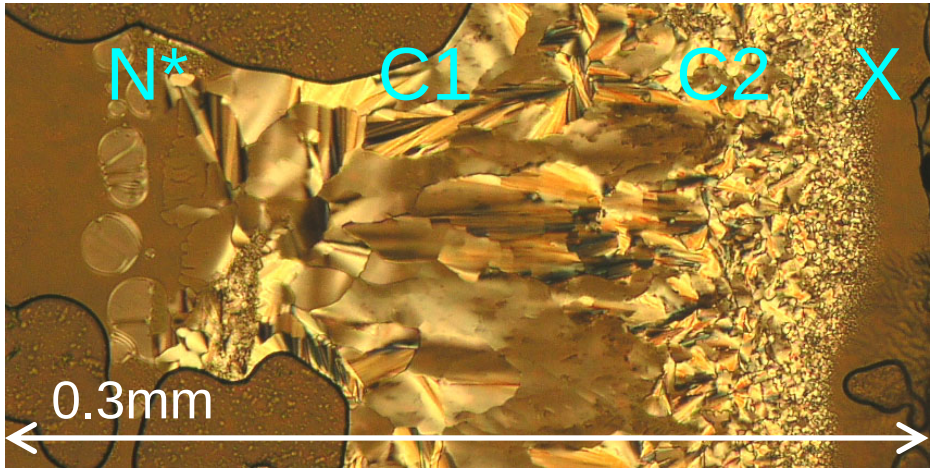


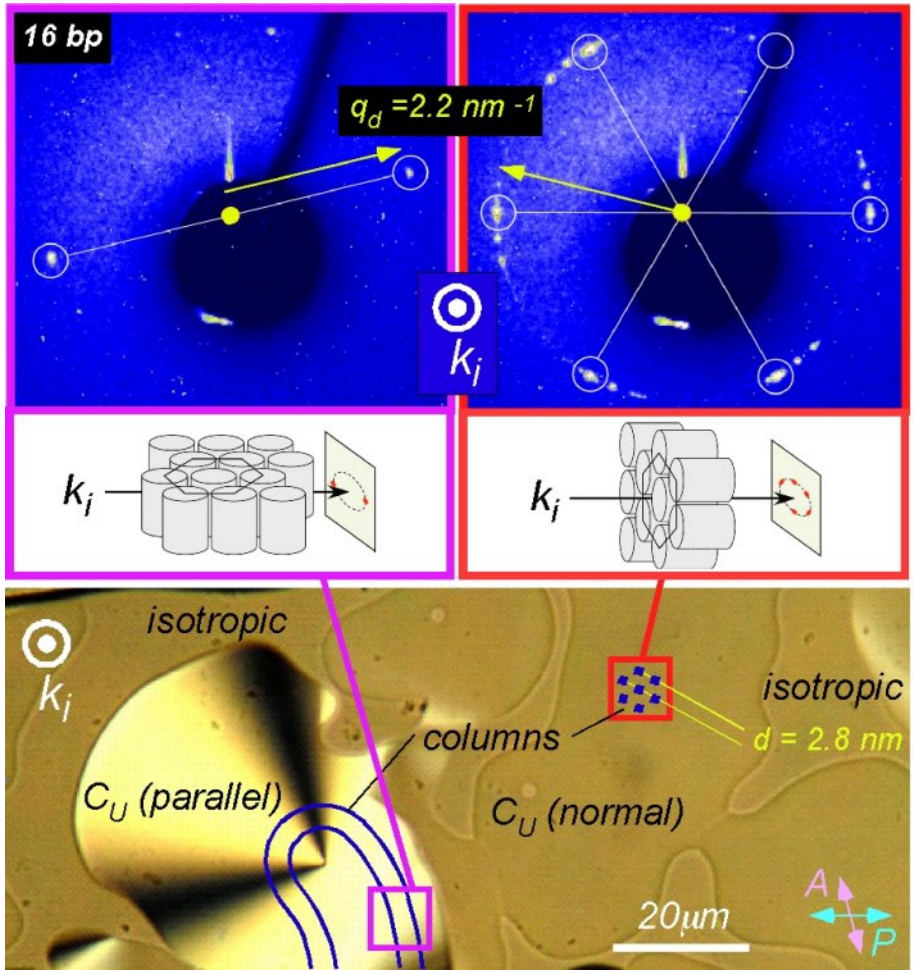
Microbeam Diffraction: DNA Oligomers



L/D~1.4

- Samples are costly, by scanning a micro-beam across a concentration gradient, a range of concentrations can be studied.
- Samples are difficult to align, but exhibit ~100mm sized domains
- Micro beam → ~ 10x10 μm





M. Nakata, G. Zanchetta, B. D. Chapman, C.D. Jones, R. Pindak, T. Bellini, and N.A. Clark, Science 318, 1276 (2007).

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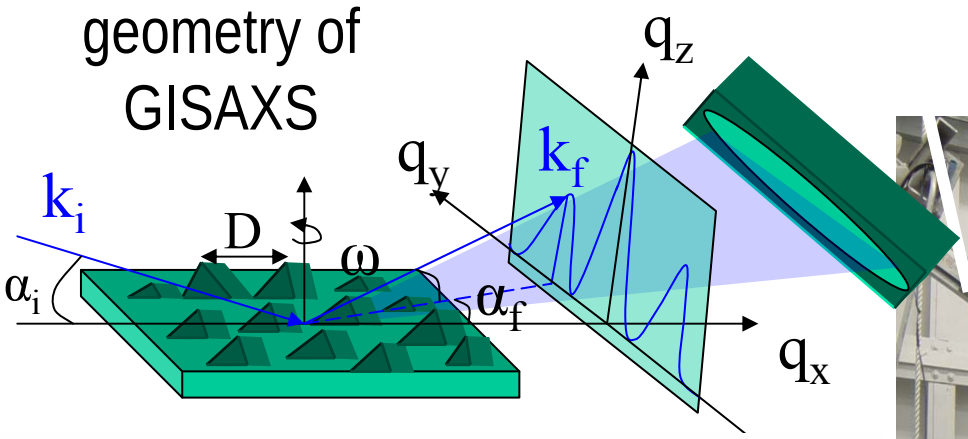
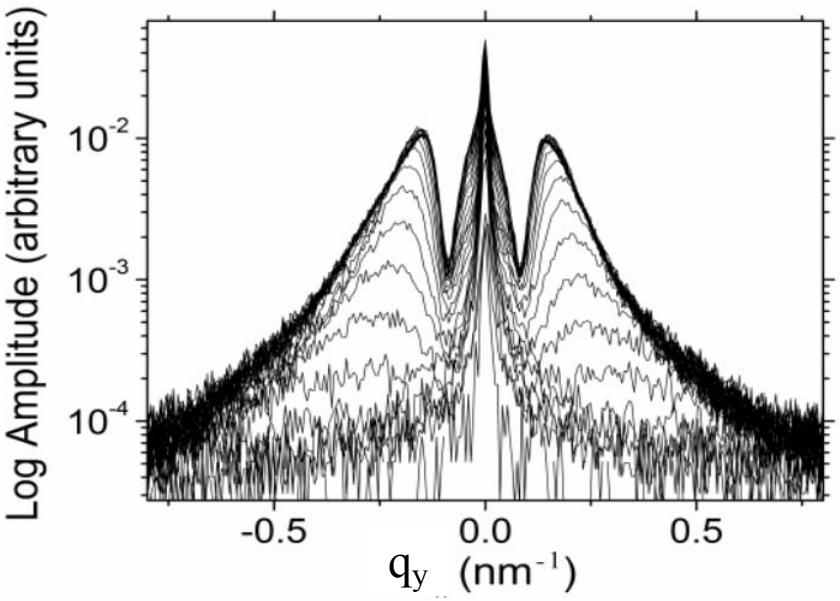
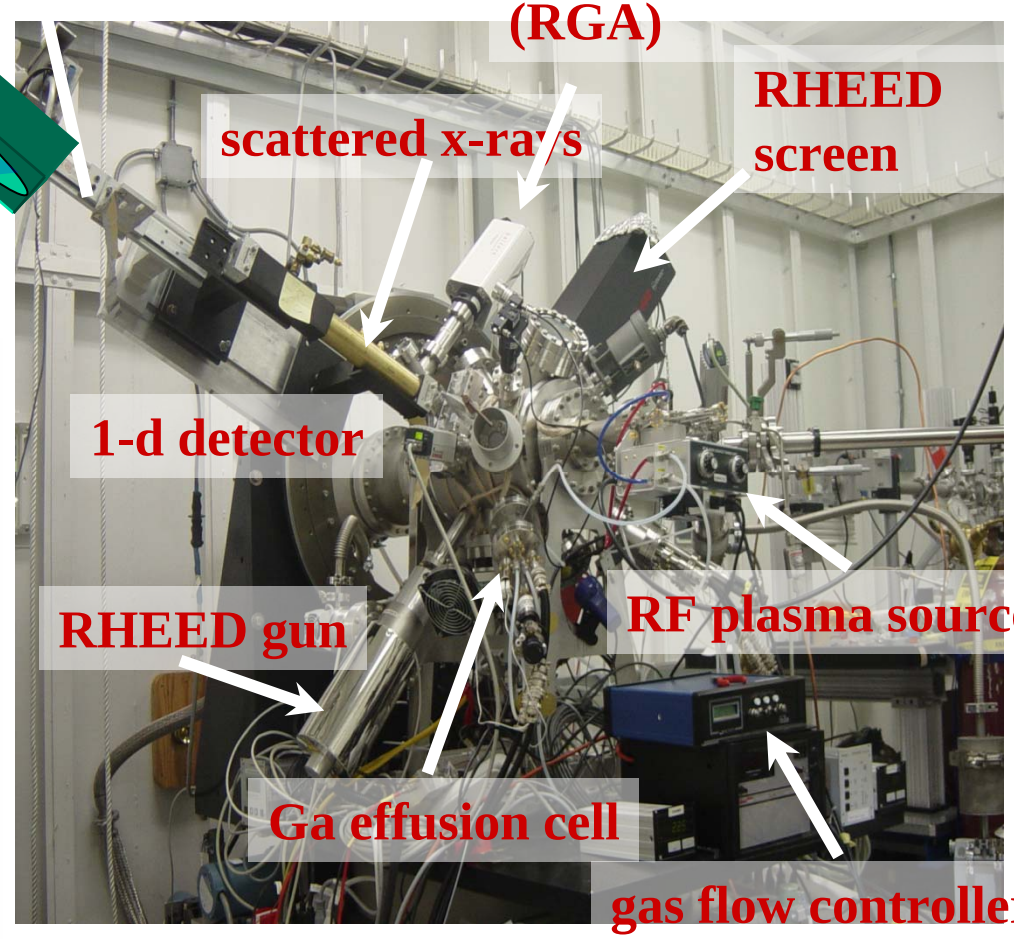
Materials Sciences: Real-Time X-ray Scattering

Scattering geometry of GISAXS





Spontaneous nanodot formation by ion bombardment



Ludwig(BU) and Headrick (U. Vermont), X21

Small Angle X-Ray Scattering: Mapping Ternary Phase Diagrams of Polyelectrolyte Complexes

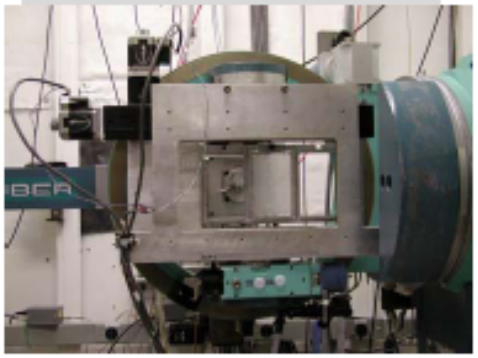
Beamline: X6B

Category of Researcher: GU

Technique: x-ray scattering

Researchers & affiliations:
Helmut H. Strey
Vesna Stanic, Maggie Swierczewska
Alex Bruno, Matt Mancuso
Wasnard Victor, Theresa Cargioli (SBU)

Publication:
in preparation



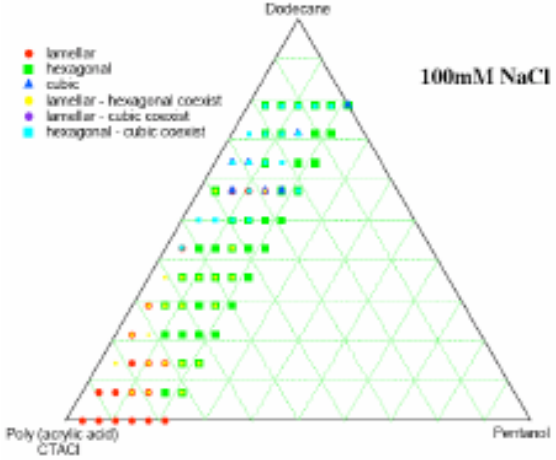
Motivation: There is a need for new. lightweight, strong, porous materials both as structural materials and in applications such as filters. Self-assembled polyelectrolyte-surfactant complexes can provide the structural length scales needed. Since phase diagrams have many components (five to six phases is typical as a minimum), combinatorial materials science methods are needed to discover new regions of phase space.

Results: In collaboration with X6B staff a well plate scanner device has been constructed. In combination with a liquid robotics system, large sample phase space can be explored. A new cubic region has already been identified in the system shown. Automated analysis software is under development by the group.

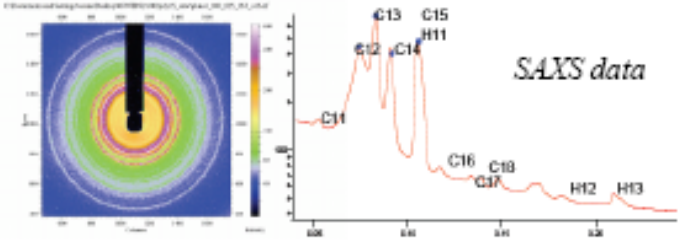
Scanner holds 3 well-plates with 96, 384, or 1536 wells

multi component complex

Ternary phase diagrams



Cubic / Hexagonal coexistence



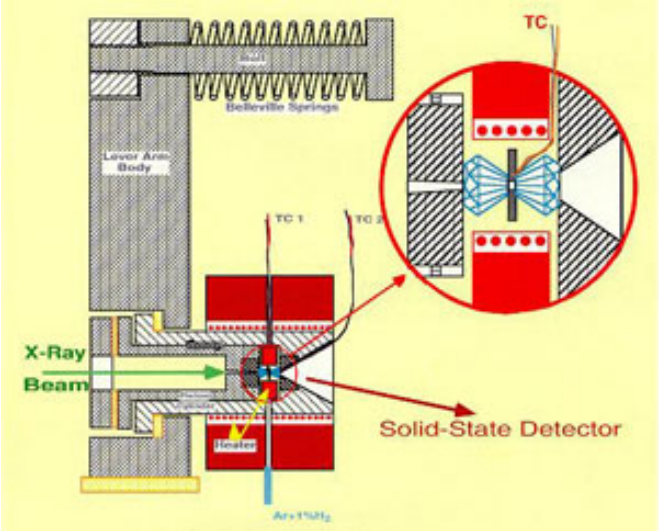
Geoscience: Thermal Expansion of Iron-Rich Alloys and Implications for the Earth's Core

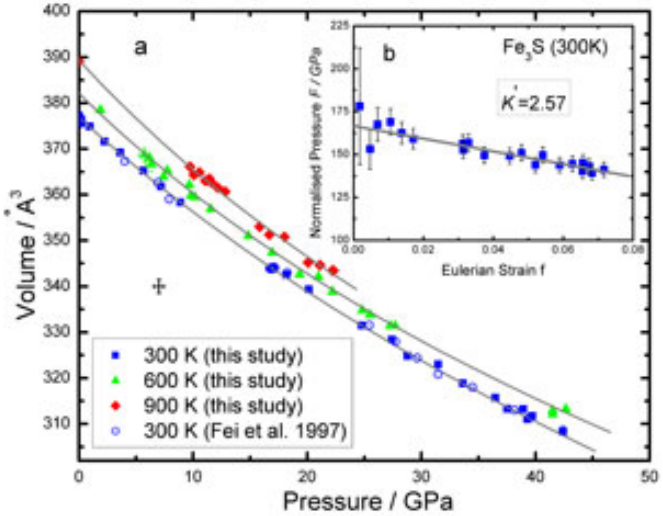
Motivation:

The Earth's core makes up nearly one-third of the planet's mass. Cosmochemical and geochemical analysis suggest that the core is made of Fe-rich alloys containing Ni and one or more lighter elements such as S, Si, C, O and H. A critical test for any candidate core composition model is that it must be able to reproduce the physical properties of the core, such as density profile and velocity profiles of the Earth's interior.

Results:

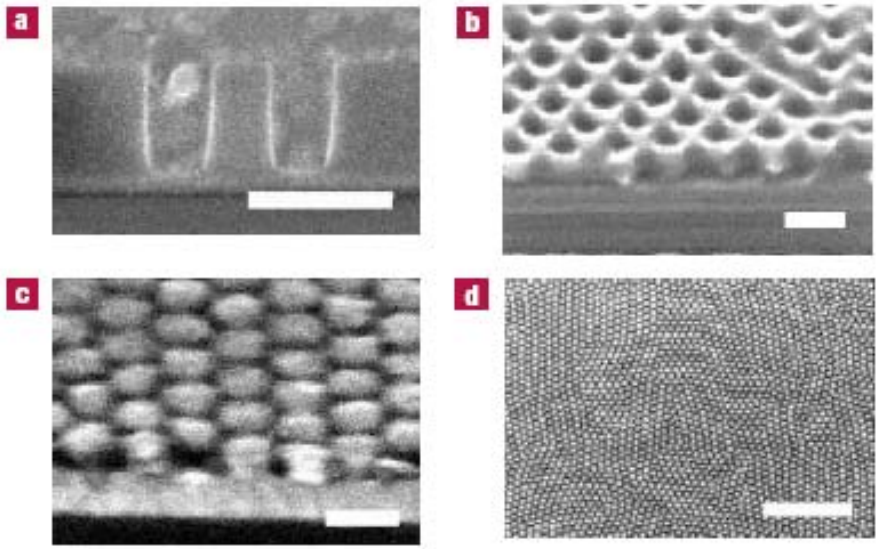
W have determined thermal expansion of the iron–sulfur compound Fe_3S, Fe, and Fe–Si alloys using in-situ synchrotron techniques. The unit-cell parameters and thermal expansivity of Fe_3S have been determined by using synchrotron x-ray diffraction techniques and externally heated diamond–anvil cells at pressures up to 42.5 GPa and temperatures up to 900 K. Our data at 42.5 GPa and 900 K suggest that 2.1 at. % sulfur produces 1% density deficit in iron

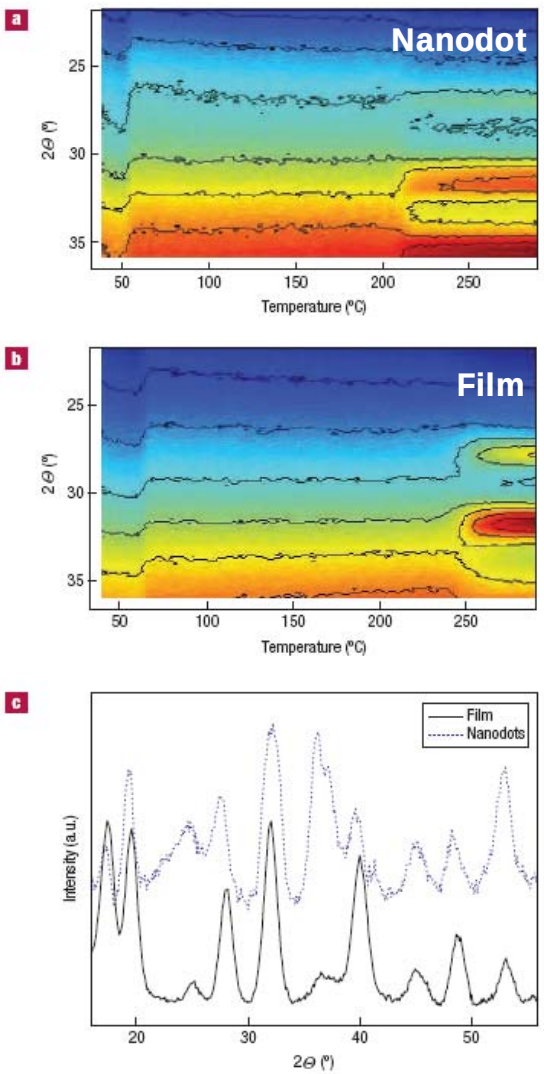




B. Chen et al., PNAS 104, 9162 (2007)

Solution-Phase Deposition and Nanopatterning of GeSbSe Phase-Change Materials



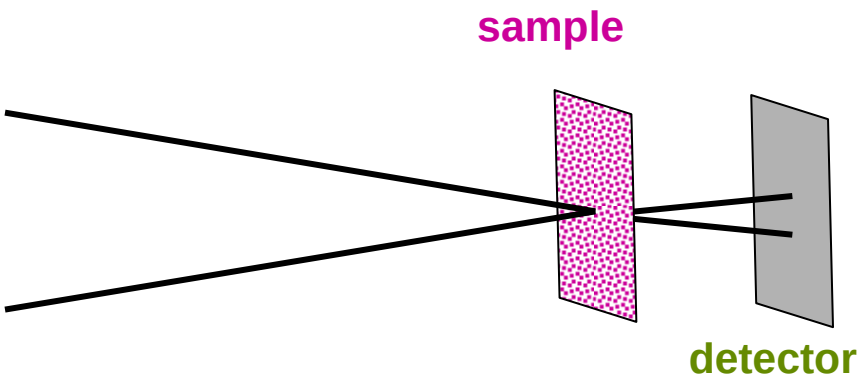


GeSbSe nanodots formed using a PS-PMMA diblock copolymer template

D. Milliron, S. Raoux, R. Shelby, J. Jordan-Sweet, Nature 6, 352 (2007)

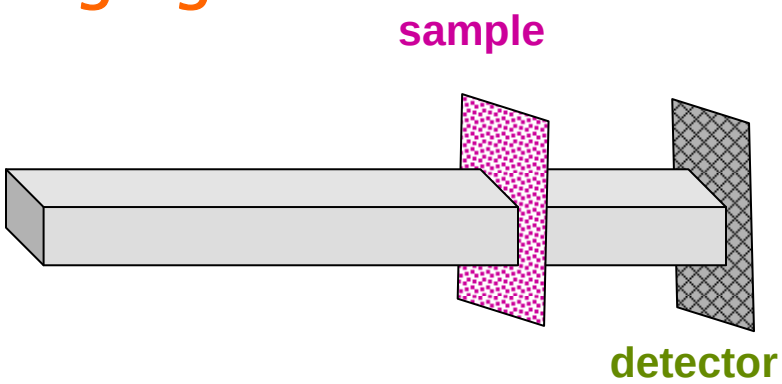
Microprobe vs. Imaging

Microprobe



- Focus beam to small spot on sample
- Raster scan sample through beam

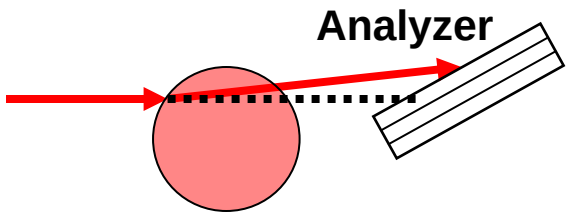
Imaging

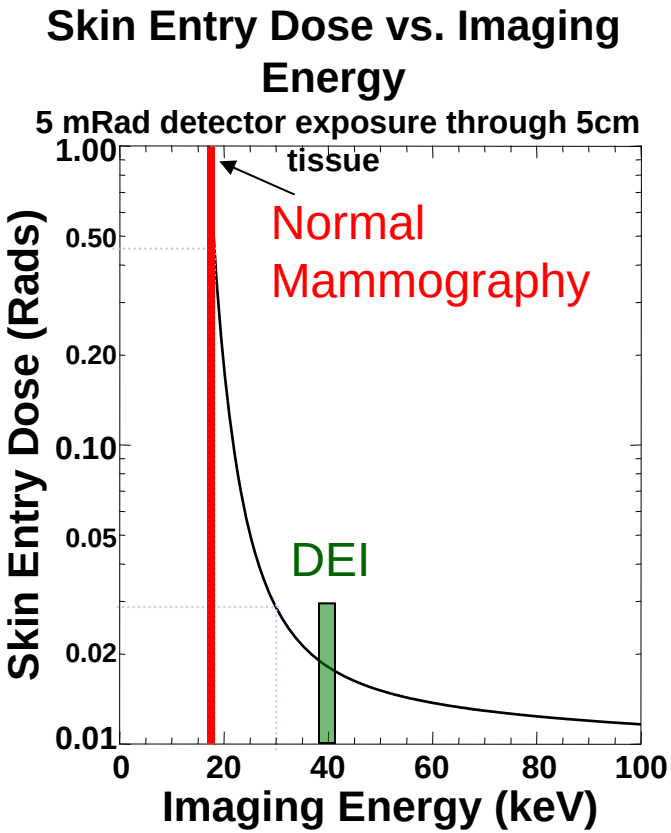


- Illuminate whole sample with big beam
- Use pixel array detector for spatial resolution

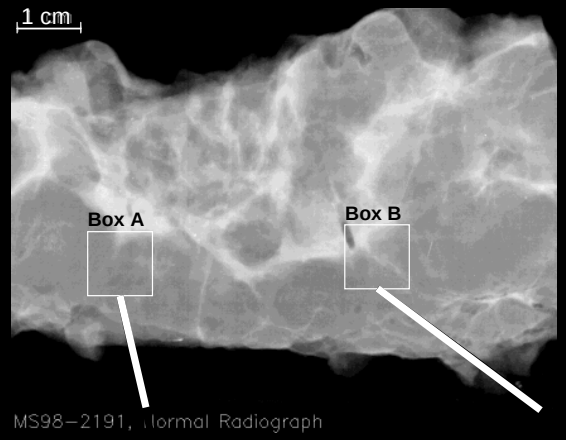
Medical Imaging – Diffraction Enhanced Imaging

Refraction contrast

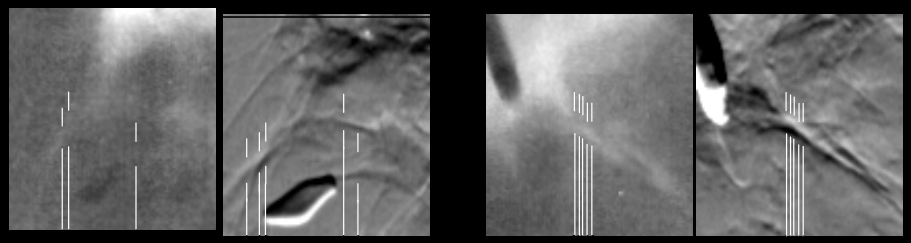




Breast Cancer Imaging with DEI



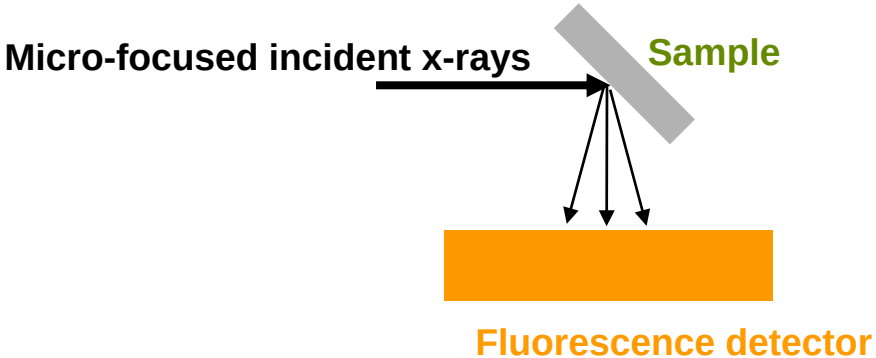
- Spiculations are due to response of the host to the cancer
- DEI has 8 – 33 times greater contrast compared to digital mammogram

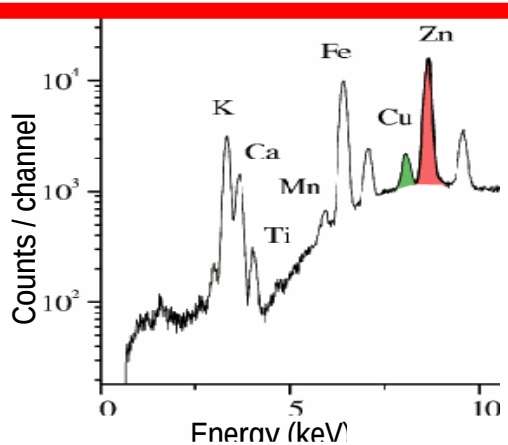


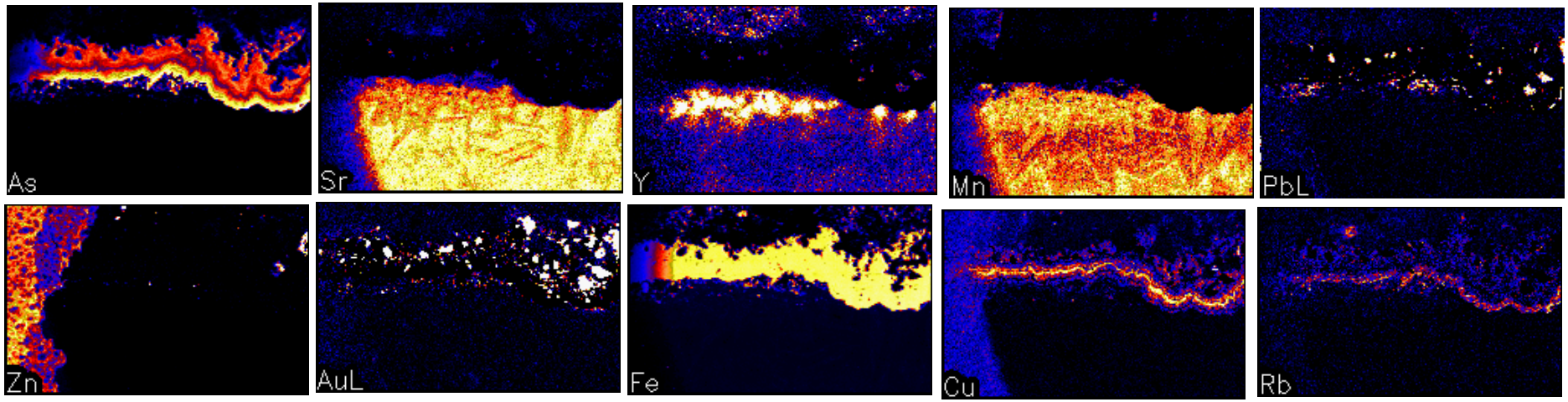
Rad DEI Rad DEI

Hasnah et. al., *Phys. Med. Bio.* 2005

X-Ray Microprobe: Rapid Fluorescence Elemental Mapping







- Hardware: 32-element detector + 2 ASICs + digitizer/processor board.
- Dynamic analysis real-time deconvolution demonstrated at 10^8 events/sec..
- X-ray elemental images of Fiji pyrite collected at NSLS X27A beamline.
- 800x500 pixels of 10um x10um in 5 hr, 20X faster than conventional detector.
- Increase to 400 elements + NSLS-II brightness would give additional $\times 10^4$ gain.

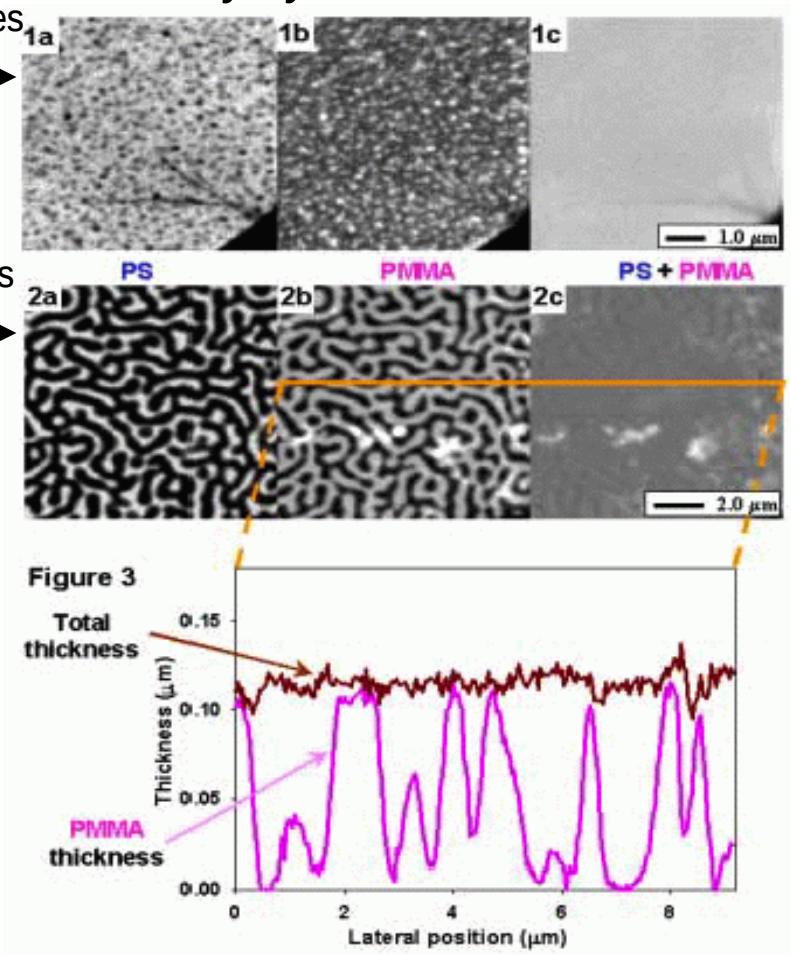
Collaboration between BNL & Australia's Commonwealth Scientific and Industrial Research Organization

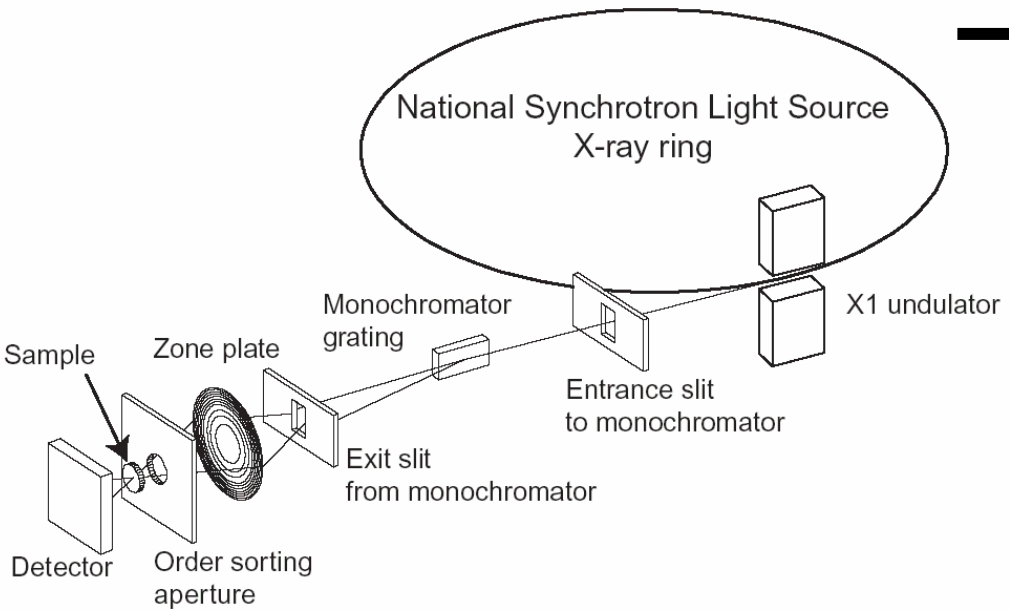
Confinement-Induced Miscibility in Polymer Blends

Polystyrene/PMMA

T Film > D Micelles

T Film < D Micelles



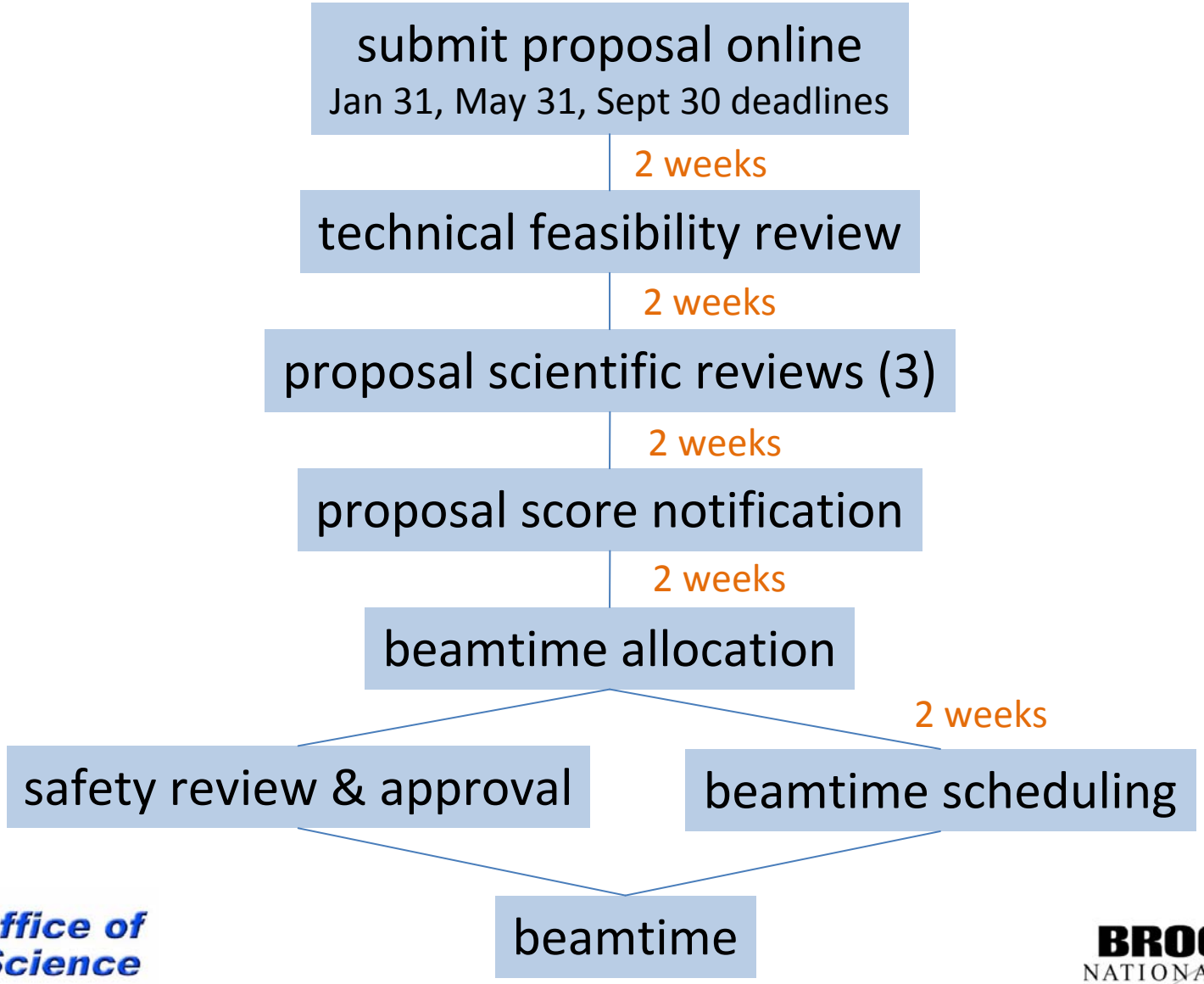


Far-field focus of 35 nm at λ = 2-5 nm

NEXAFS provides sensitivity to functional group

S. Zhu, Y. Liu, M. Rafailovich, J. Sokolov, D. Gersappe, D. Winesett, and H. Ade, Nature 400, 49 (1999)

Proposal Submission Flowchart



Overview of the Proposal System

- 12 Proposal Review Panels (PRPs), categorized first by technique, then by broad science area.
  - Example: *Imaging & Microprobes: Biological & Environmental Sciences*
  - The propose reviewer will most likely only have a *general* knowledge of your specific sub-field.
- Each proposal is reviewed by 3 reviewers who provide a rating between 1 and 5 (1 high). An average proposal rating lower than ~2.5 is required for beam time. Proposals not allocated beam time get a 0.2 rating improvement for next request. Recent rating statistics for each beamline can be found at: **http://www.nsls.bnl.gov/users/usersguide/bt-scoring.asp**
- Proposals can describe a single experiment or a set of experiments that span a period of up to 2 years (6 cycles). Beam Time Request is needed for each cycle.
- There are 3 beam time scheduling cycles.

6 weeks

Scheduling Cycle	Proposal Due Date	Beam Time Allocated
January-April 2007	September 30, 2006	November 11, 2006
May-August 2007	January 31, 2007	March 14, 2007
September-Dec. 2007	May 31, 2007	July 12, 2007

6 weeks

First Step

- Before submitting a proposal you should first identify the beamlines that can be used for your project and contact the Beamline Spokesperson for each beamline.
- Discuss with the Beamline Spokesperson –
  - Does the beamline meet the specifications required for your project?
  - Does your project require additional instrumentation or a non-standard beamline configuration? Can the instrumentation be supplied or the beamline reconfigured?
  - The amount of beam time that would be required to complete your project.
  - Can a preliminary feasibility check be made on your sample?

Physical & Chemical Sciences Division

Ron Pindak, Physicist
Soft Condensed Matter Systems & Interfaces
X-Ray Diffraction, Resonant & Surface XRD

Dario Arena, Physicist
Magnetism & Dynamics of Thin Films
Soft X-Ray Spectroscopy.& Scatt, Time-Resolved

Larry Carr, Physicist
Dynamics of Metals, Superconductors, Semiconductors
Tz, IR spectroscopies, Magnetospect,. Time-Resolved

Lars Ehm, Mineralogist
Materials at Extreme Conditions
X-Ray Powder Diff, X-Ray Total Scatt.

Christie Nelson, Physicist
Strongly Correlated Electron & Mag. Systems
X-Ray Diffraction & Resonant XRD

Cecilia Sanchez-Hanke, Physicist
Magnetic Thin Films & Devices
Soft X-Ray Spectroscopy & Scattering

Elio Vescovo, Physicist
Magnetic Surfaces, Interfaces and Multilayers
Angular- and Spin-resolved Photoemission

Syed Khalid, Physical Chemist (Sci. Assoc.)
Catalytic Systems
XAS, XANES, XRD, Q-EXAFS

Steve LaMarra, Science Associate
Cryogenic Magnets, Detectors, & Displex Support

Life & Environmental Sciences Division

Scientists

Lisa Miller
Biophysical Chemist
Beamline U10B
IR, XRF spectr. & imaging

Marc Allaire
Biochemist
Beamline X6A
MX

Elaine DiMasi
Physicist
Beamline X6B
X-ray scattering

Vivian Stojanoff
Physicist
Beamline X6A
x-ray scattering, MX

Lin Yang
Biophysicist
Beamline X21
X-ray scattering

Science Associates

Randy Smith
Beamline X27A
IR, XRF spectroscopy & Imaging

Qun Kian
Beamline X6A
Computer science

Title and Abstract

- The content of proposals is treated as confidential except for the proposal title and abstract. These are sometimes used in reports to funding agencies.
- Abstract should provide a brief overview of the project without revealing any experimental details that you want to keep private.

Scientific Importance of Proposed Experiment

- Background - provide sufficient background information so the reviewer, who may only have a general knowledge of your subfield, can understand the scientific issues that your project addresses.
- Impact - describe the expected scientific and/or technological and/or educational impact that your results will have.

Why is Synchrotron Radiation Required

- You need to convince the reviewer that your experiment could not be done on an in-house light source.
- Reasons might include:
  - Your experiment requires a unique property of synchrotron radiation such as, high brightness (small beam area), high flux (for weakly scattering samples), or energy tunability (for spectroscopic measurements).
  - Your experiment requires high through-put to characterize a large number of samples.

Describe Work Previously Done at a Synchrotron Facility

- Synchrotron experience is an important criteria used in rating proposals. The proposal reviewers are looking for evidence that you have the experience to carry out a successful experiment.
- New users – It's recommended that new synchrotron users initially collaborate with an experienced user. This mentoring relationship should be described in this section.

List of Three Publications to Assist in Proposal Review

- Reviewers are looking for evidence that your previous use of NSLS beam time resulted in high-impact publications.
- Reviewers are also looking for publications that establish your expertise in the topic of the proposed project.
- Include the title of the publication and a complete list of authors.
- It is best if the listed publications have appeared in print and are readily accessible electronically.

Research Description

- Provide an outline of what experiments you will be doing over (1) all the cycles of the project in <u>moderate</u> detail and (2) the next cycle in <u>complete</u> detail.
- This section serves two purposes:
  - Indicates to Reviewer the likelihood that your experiment will provide the information that you're anticipating and whether the proposed experiments justify the requested amount of beam time.
  - Indicates to the Beamline Local Contact what instrumentation and beamline configuration is needed for your run.
- The research that receives the highest ratings are ground-breaking experiments that do not give the impression of ill-defined 'fishing expeditions' or the next in a 'series' of similar measurements.
- Results using complementary techniques that can identify interesting regions of parameter space are crucial since beam time is limited.
- Figures, which often are more effective than words, should be added to the proposal in a PDF file.

Safety Approval Form

- Each proposal is accompanied by a Safety Approval Form (SAF). In the Safety Approval Form you describe any hazards associated with your proposed experiment (chemical, electrical, environmental). You also provide the names of all the experimenters involved with the project.
- Only a preliminary SAF is required at proposal submission; however, the SAF should be updated when beam time is allocated.
- The SAF is valid for 1 year.

Last Step

- Review by the beamline spokesperson: If you are a new synchrotron user, it is strongly suggested that you ask the beamline spokesperson to review and critique your proposal. But don't send it at the last minute; they get a lot of these requests!

Faculty-Student Research Support Program

ABOUT THE PROGRAM

The Faculty Student Research Support Program is designed to encourage new faculty/student research groups to BNL's National Synchrotron Light Source.

- Faculty/student groups that are new users to the NSLS (within 2 years) are eligible.
- Newly appointed assistant professors (within 2 years of faculty appointment) and their students are also eligible, even if they are current or past NSLS users.
- Faculty must be from a U.S. academic institution of higher education.
- The program covers expenses incurred during exploratory visits to the NSLS and while performing initial experiments. Expenses covered include travel, housing, per diem, and some incidental costs.

PARTICIPATION IN THE PROGRAM

- Based on scheduled General User beam time or PRT time.
- Reimbursement commitments are made for one scheduling cycle at a time.
- Maximum number of participants per request is one faculty member and two students/post docs.
- Faculty member must participate in beamtime with students/postdocs
- Participation in the program is limited to two consecutive years (up to 6 beamtime cycles).

APPLICATION DEADLINES

January – April: November 30; May – August: March 31; September – December: July 31

http://www.nsls.bnl.gov/users/fsrsp/

Office of Science
U.S. DEPARTMENT OF ENERGY

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

Contributing User Programs

Contributing Users are groups who carry out research programs at Facility Beamlines and also enhance beamline capabilities and/or contribute to their operation. In exchange, they receive up to 25% of the available beam time.

Beamline(s)	Contributing User Group	Science Program
U5UA	BNL Center for Functional Nanomaterials	Nano-catalysis: LEEM-XPEEM
X9	BNL Center for Functional Nanomaterials	Soft and Bio Nanoscience: SAXS/GISAXS
X13B	Columbia Univ. & IBM	X-Ray Microbeam Diffraction Analysis
X17B1	Rutgers Univ.	Energy Dispersive X-Ray Diffraction: Strain Field Mapping
U2UA,X17B2, X17B3, X17C	COMPRES (Consortium of 45 U.S. Universities, 3 National Labs, & 28 Foreign Affiliates)	High Pressure X-Ray Studies in Earth Sciences
X18B, X19A	BNL Chemistry, Univ. Delaware, Yeshiva Univ., Oak Ridge Nat. Lab, UOP	Catalysis Consortium: High and Low energy EXAFS
X21	Boston Univ. and Univ. of Vermont	Center for Real-time Studies of Surface Processes
X27A	BNL Environmental Sciences and Stony Brook Univ..Geosciences	Environmental Science Consortium: X-Ray Microprobe Imaging

HBCU Consortium

INCREASE

Interdisciplinary Consortium for Research and Educational Access in Science and Engineering

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News & Events

HBCU Faculty, NSLS Staff Collaborate to Develop Synchrotron Curriculum

Creating an Electron Avalanche without Electronics

NSLS Hosts Historically Black Colleges and Universities Professors' Workshop

Office of Science
U.S. DEPARTMENT OF ENERGY

http://increase.nsls.bnl.gov

BROOKHAVEN NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

Summary

- The NSLS provides spectroscopy, scattering, and imaging techniques for research in a wide range of fields: physics, chemistry, materials science, engineering, biology, biomedicine.
- The NSLS beamline spokespersons are the best resource to answer questions regarding the use of specific facilities
- A faculty-student support program is available for new synchrotron users.
- The construction of NSLS-II, planned for operations in 2014, provides an exciting opportunity for new science, technology, or educational consortia to form for the design of instrumentation or programs for NSLS-II that can be developed at the NSLS.