Distribution Category: Atomic, Molecular, and Chemical Physics (UC-411)

ANL/APS/TM--11

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ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
Argonne, Illinois 60439

POSTER SESSION:

FIFTH USERS MEETING FOR THE ADVANCED PHOTON SOURCE

Held at Argonne National Laboratory
October 14-15, 1992

November 1992

work sponsored by
U.S. DEPARTMENT OF ENERGY
Office of Energy Research

Contents

POSTER SESSION:
Fifth Users Meeting for the Advanced Photon Source

ABSTRACT

The Advanced Photon Source (APS), which is currently under construction as a national user facility at Argonne National Laboratory is a third-generation synchrotron x-ray source, one of only three in the world. It is expected to produce x-rays that are 10,000 times brighter than any currently produced elsewhere for use in research in a wide range of scientific areas. Users from industry, national laboratories, universities, and business will be able to come to the APS to conduct research either as members of Collaborative Access Teams (CATs) or as Independent Investigators. Principal users will be members of CATs, which will be building and operating all of the beamlines present in the first phase of APS beamline development. The first set of CATs has been selected through a competitive proposal process involving peer scientific review, thorough technical evaluation, and significant management oversight by the APS.

This document is a compilation of posters presented at the Fifth Users Meeting for the Advanced Photon Source, held at Argonne National Laboratory on October 14-15, 1992. All CATs whose scientific cases were approved by the APS Proposal Evaluation Board (which provided scientific peer review) are included. In addition, this document contains a poster from the Center for Synchrotron Radiation and Research and Instrumentation at the Illinois Institute of Technology.

BESSRC CAT

Basic Energy Sciences Synchrotron Radiation Center CAT

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Materials Science Division
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Scientific Program

Time-resolved diffraction in the picosecond domain for monitoring structural changes in fast chemical reactions.

Atomic physics involving x-ray inner-shell excitation and ionization of free atoms and ions.

Investigation of magnetic structures of thin films with tunable polarized radiation from an elliptical multipole wiggler source.

Energy-dispersive XAFS as an analytical tool for research in catalysis and materials science.

BESSRC TECHNICAL FACILITIES

Two sectors — 5 beamlines — 10 experimental stations

FIRST SECTOR

Undulator Beamline: Instrumentation for time-resolved diffraction, anomalous scattering, atomic and molecular physics, small angle scattering and x-ray standing waves.

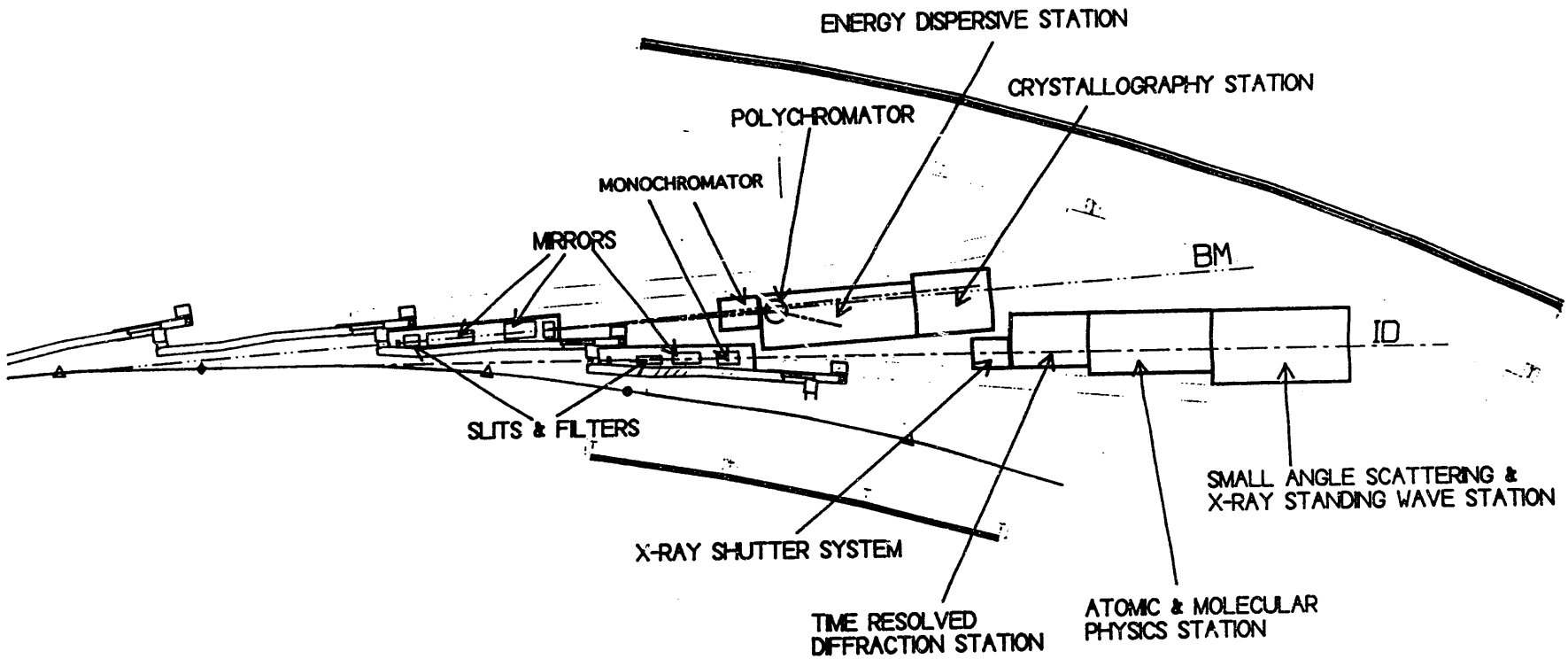
Bending Magnet Beamline: Instrumentation for energy dispersive x-ray absorption and x-ray crystallography.

SECOND SECTOR

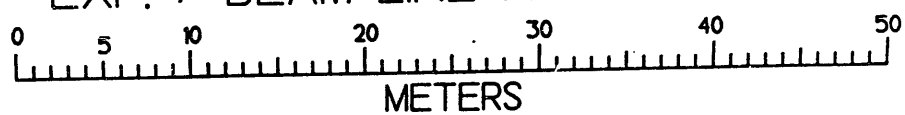
Elliptical Multipole Wiggler Beamline: Instrumentation for magnetic scattering, x-ray absorption, Compton scattering, and surface scattering.

Bending Magnet Beamline: Instrumentation for x-ray powder diffraction and x-ray absorption.

UNDULATOR SECTOR



EXP. / BEAM LINE AREA LAYOUT



Principal Advantages of Undulator-P

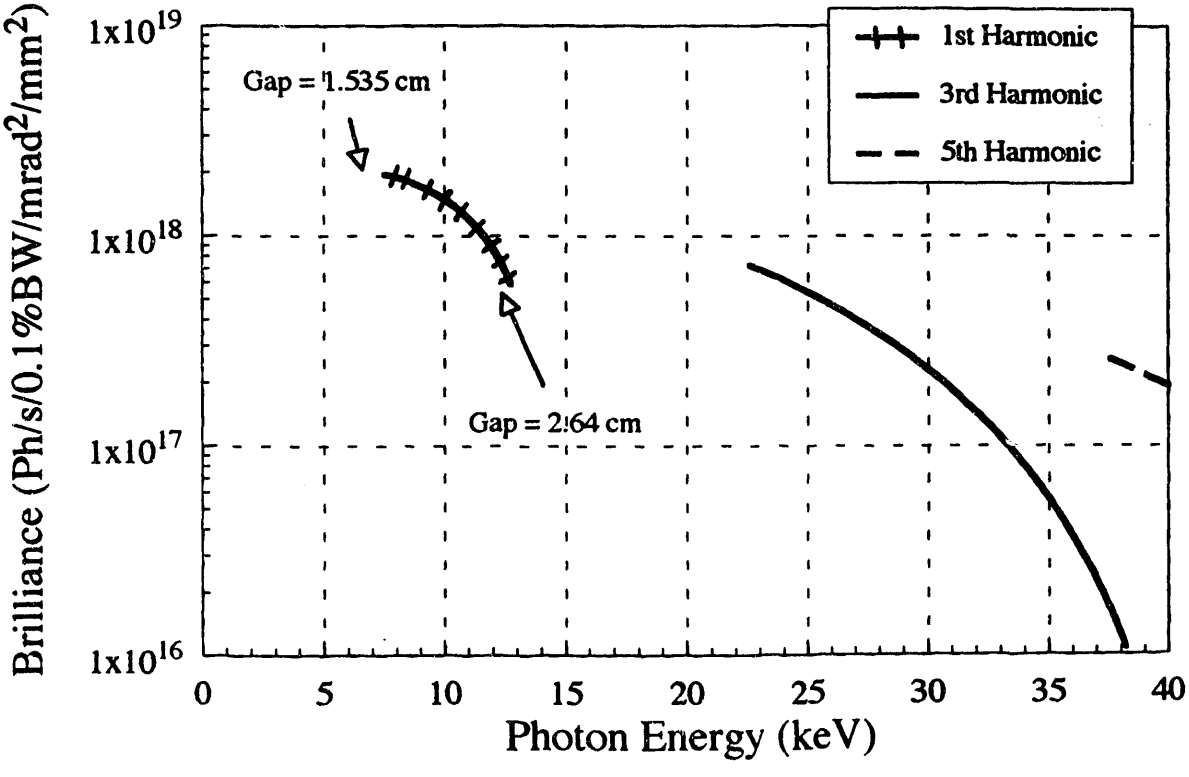
Continuous tunability from 3.1 keV to > 30keV during Phase I using high on-axis brilliance of odd harmonics

This means: Access to K-edges of argon thru iron
Access to K-edges of bromine thru palladium (including Y & Mo)
Access to L_{II}- and L_{III}-edges of ruthenium through samarium
Access to L_{II}- and L_{III}-edges of lead thru uranium (including Bi)
Higher K-values make higher harmonics brighter.
Loss of brilliance less than factor of two in range served by Undulator-A
Higher radiated power density, but this can be suppressed by mirror
No change in divergence of central cone at a given photon energy.
Slight loss of resolving power in energy range where emittance is not limit
No need to collect off-axis radiation from even or odd harmonics

Undulator A (3.3 cm period)

Parameters

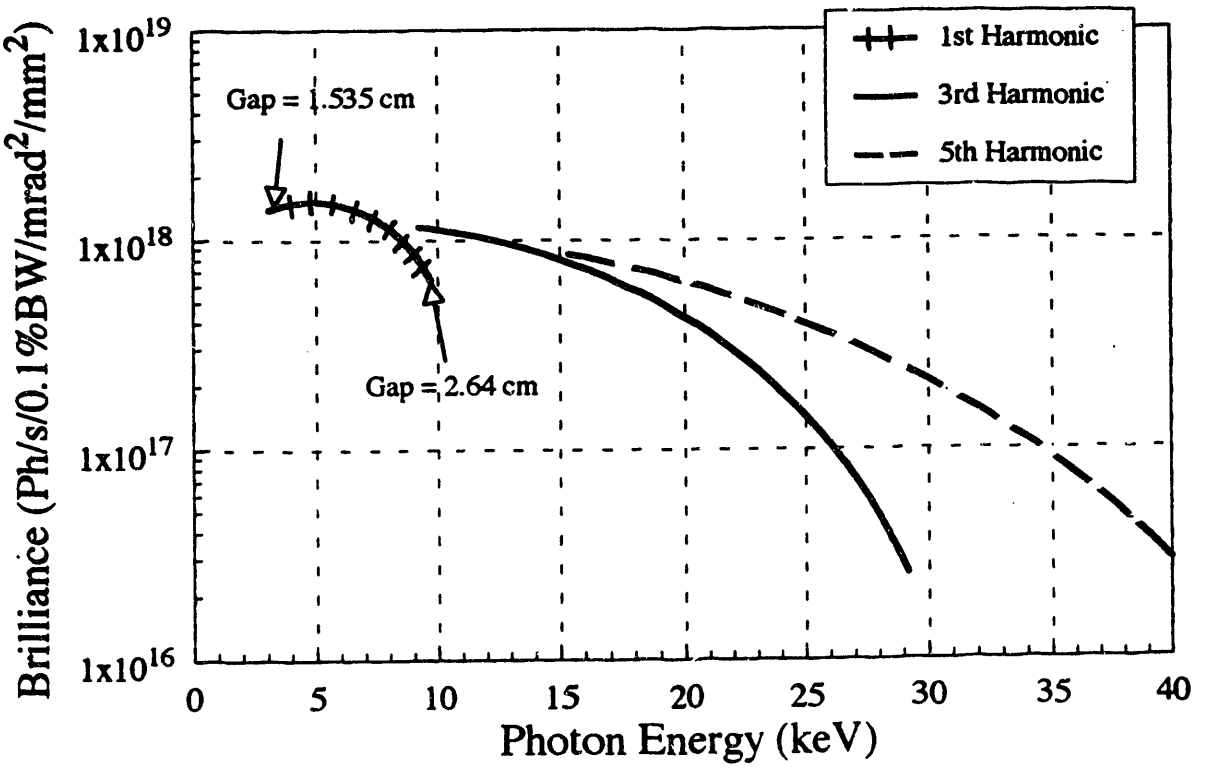
Undulator Period l_0 (cm)	3.3
Magnet Gap Range, G (cm)	1.54-2.44
Peak Field Range on Axis, B_0 (T)	0.15-0.43
Length (m)	2.31
Undulator Periods, N	70



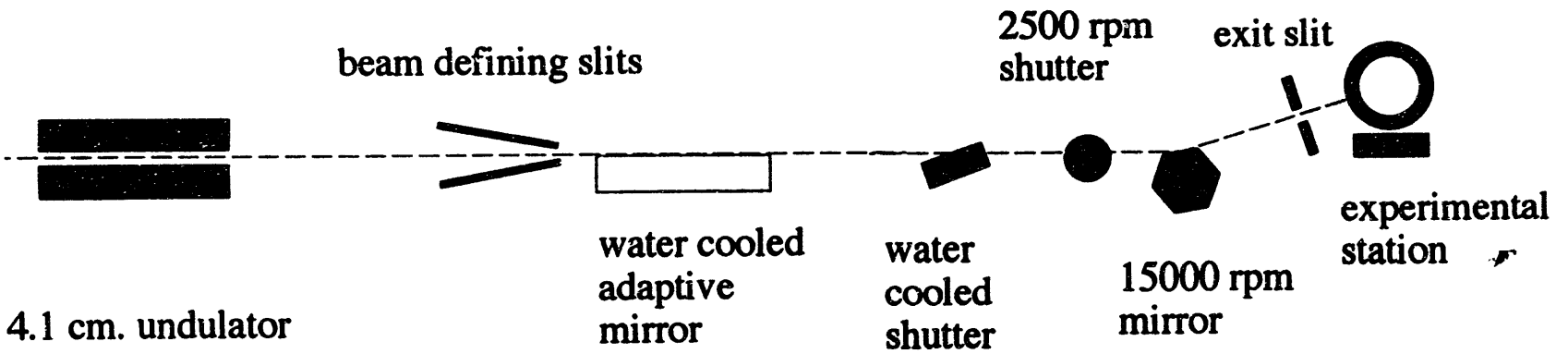
Undulator P (4.1 cm period)

Parameters

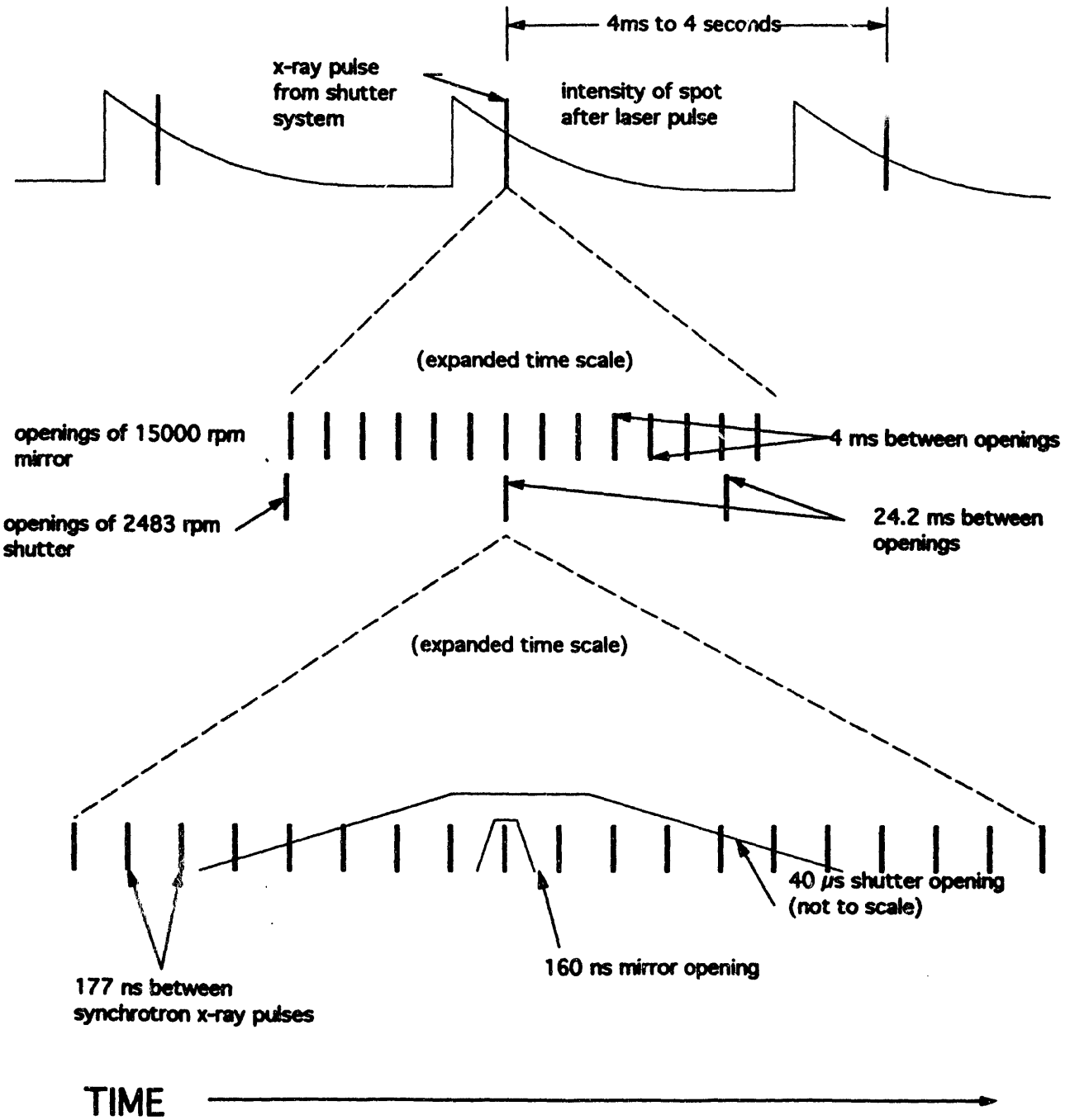
Undulator Period l_0 (cm)	4.1
Magnet Gap Range, G (cm)	1.54-3.28
Peak Field Range on Axis, B_0 (T)	0.15-0.61
Length (m)	2.3
Undulator Periods, N	56



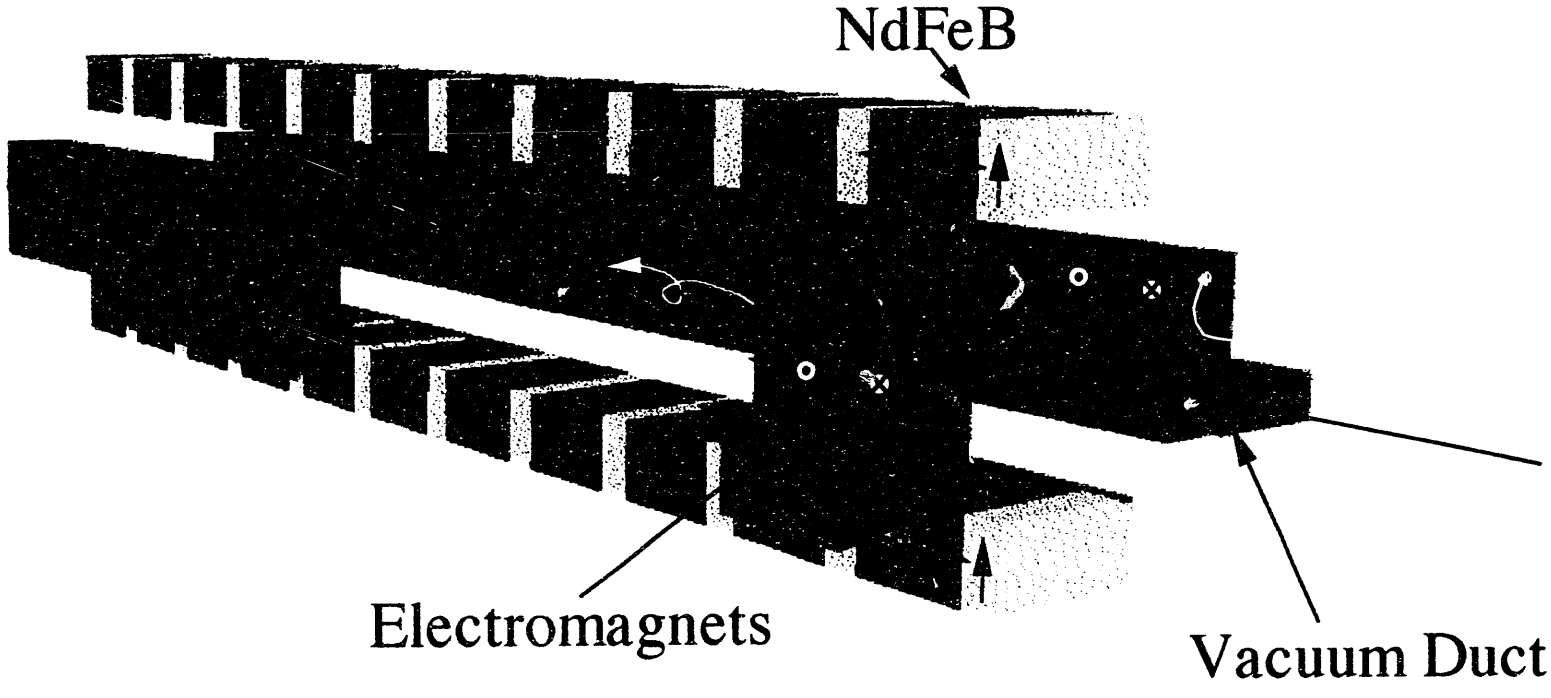
The Key Components of the Undulator Beamline



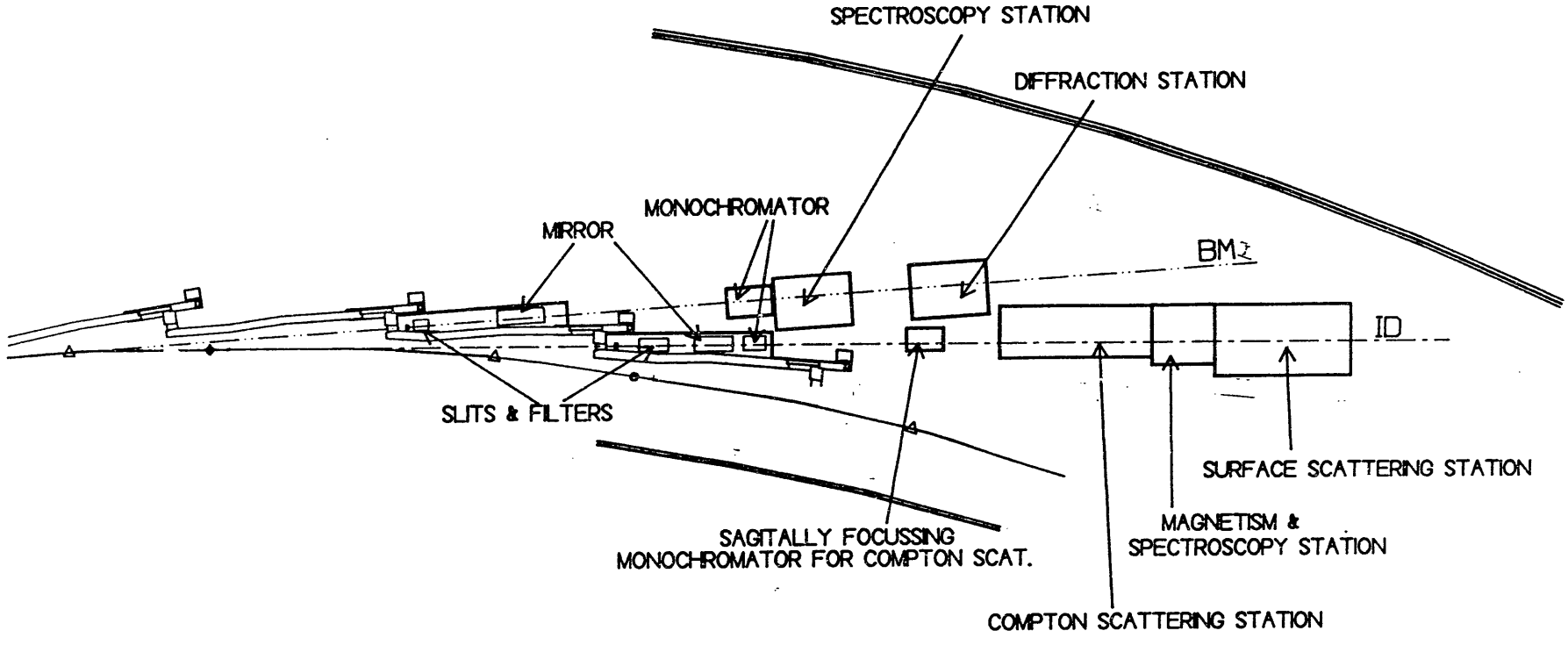
Illustrative example of a possible timing diagram for a time resolved diffraction experiment



Elliptical Multipole Wiggler



ELLIPTICAL MULTIPOLE WIGGLER SECTOR



EXP. / BEAM LINE AREA LAYOUT

0 5 10 20 30 40 50

METERS

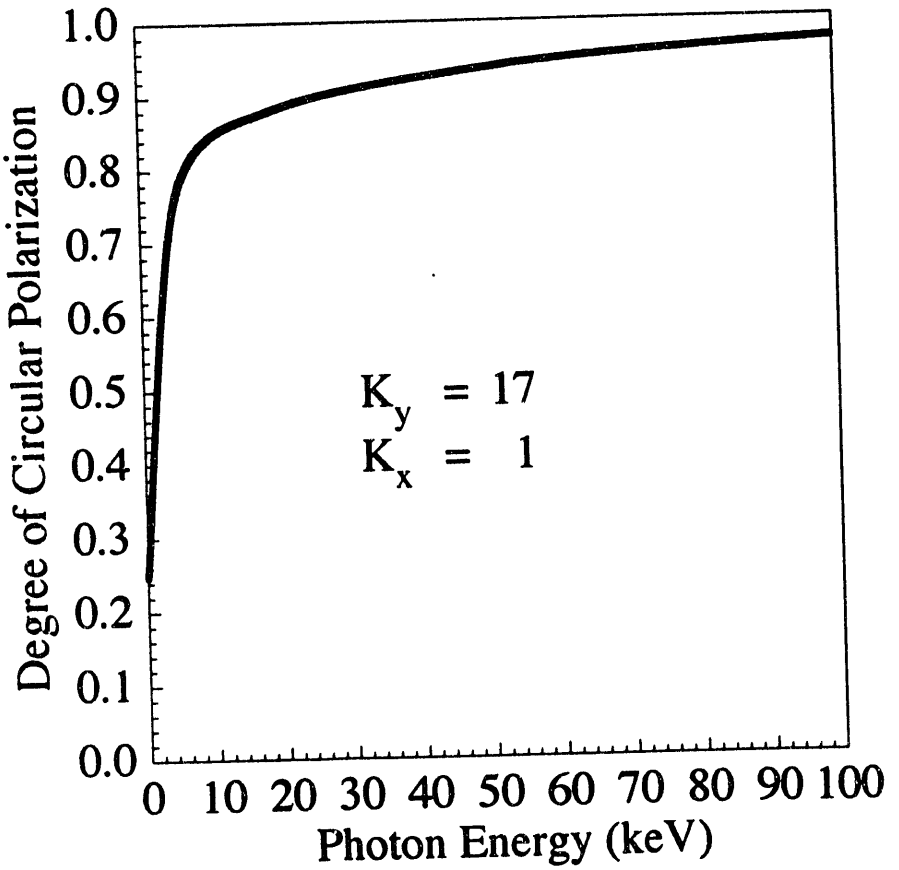
Elliptical Multipole Wiggler (suggested for APS)

E(GeV)= 7 I=100 mA

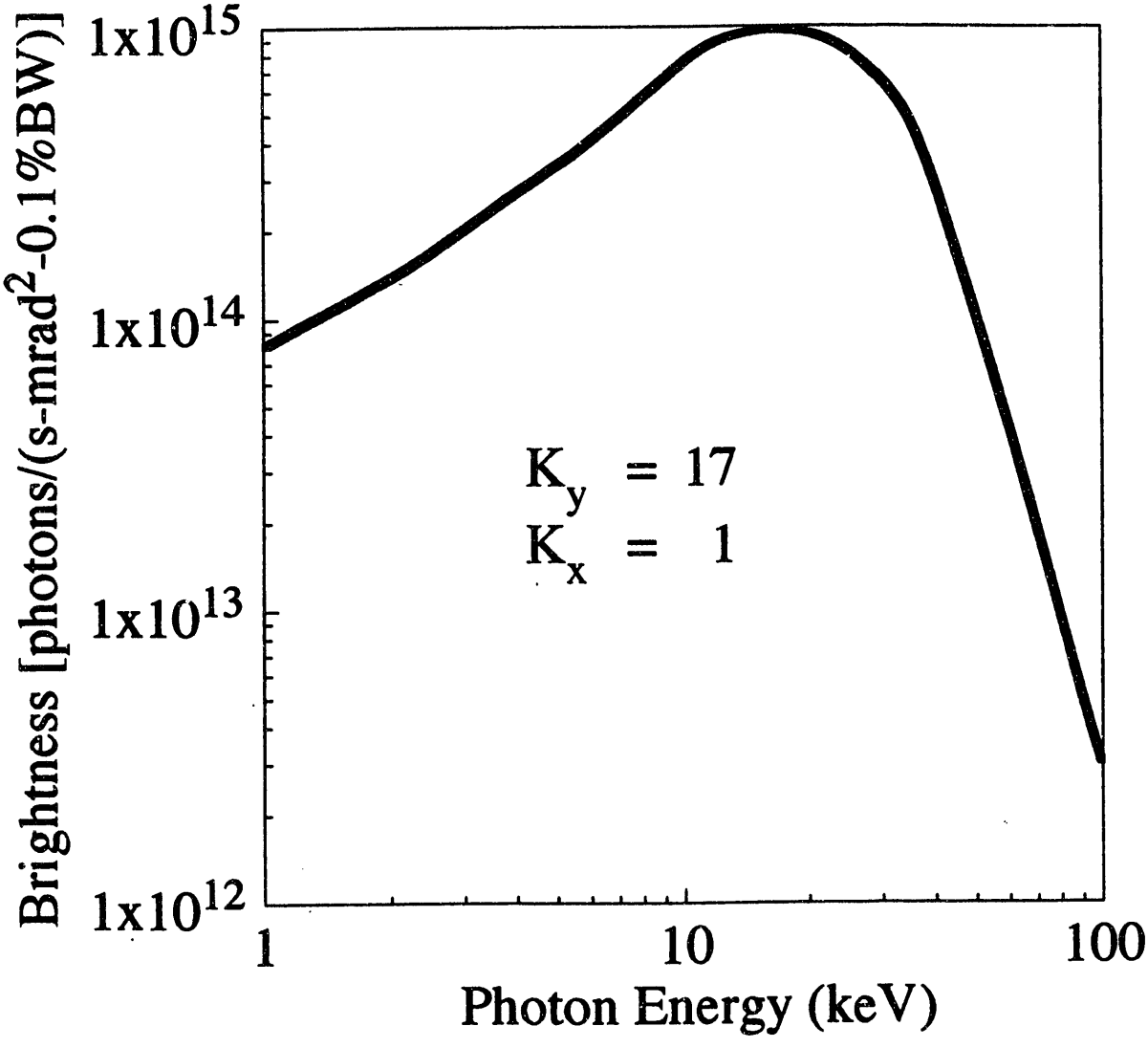
Parameters

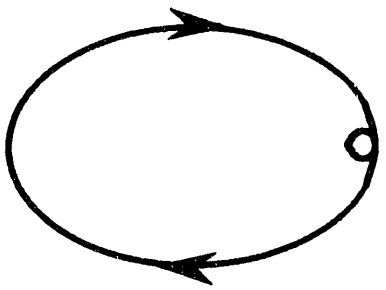
Wiggler Period	15 cm
Number of periods	20
Device length	3 m
Gap range	3-6.2 cm
Field range	By = 0.5-1.2 T
	Bx = 0.05
E_c - range	16-40 keV
K - range	Ky = 1-17
	Kx = 1-3

Elliptical Multipole Wiggler Polarization



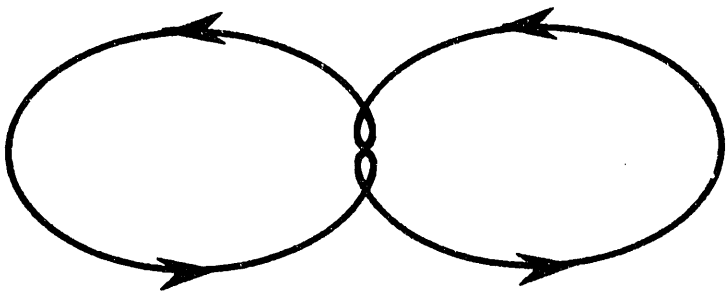
Elliptical Multipole Wiggler Brightness





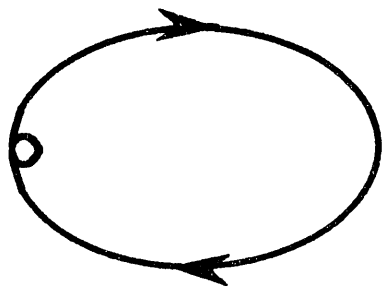
$$\psi = 2K_X\gamma^{-1}$$

$$\psi = K_X\gamma^{-1}$$



$$\psi = 0$$

$$\psi = -K_X\gamma^{-1}$$



$$\psi = -2K_X\gamma^{-1}$$

EMPW

BESSRC

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Deputy Director: G. S. Knapp

Key Personnel: P. Cowan (Phys), M. Bedzyk (MSD/NU), M. Ramanathan (MSD), G. Jennings (MSD), M. Engbretson (MSD), M. Beno (MSD), R. Chiarello (CMT), L. Soderholm (CHM), J. Norris (CHM), M. Bowman (CHM), R. Wynan (CHM), P. Thiyagarajan (IPNS/CHM).

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BioCAT

Biophysics CAT

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Purpose

Develop facilities to study the *structure* and *dynamics* of partially ordered biological materials, such as:

- protein folding intermediates
- biomolecules in solution
- Solvent interactions
- Kinetics, structure/function relationships
- Interactions: protein-protein, protein-nucleic acids
- Glycoproteins
- Multienzyme complexes
- Regulation of activity by subunit association
- Filamentous viruses
- Muscle fibers
- Membranes
- Langmuir Blodgett films, liquid crystals
- Phospholipid phase transitions
- Membrane-bound proteins
- Microtubules and their formation
- Cellular organelles and structures

Motivation

Revolution in modern biology and medicine is driven by molecular& structural biology

Given the success of crystallography, why study *disordered* (non-crystalline) biological materials?

a) Many important biological systems simply are not amenable to crystallographic study

b) Biological molecules are flexible
- motions often are functionally important
- state *in vivo* may differ from that in crystal (evident from XAFS studies)
- microscopic heterogeneity frozen in "conformational substates"

c) Kinetics/time dependence can be studied via XAFS and diffraction

Structure/function correlations
- rapid mixing & stopped flow
- photo-initiation (e.g. caged ATP)
- temperature jump
- pressure jump
- applied electric or magnetic fields.

Techniques

- X-ray small angle scattering
  - anomalous
  - time-resolved

- X-ray Absorption Fine Structure
  - solution
  - oriented samples (polarized)
  - time-resolved

- Novel techniques
  - Diffraction Anomalous Fine Structure
  - High energy resolution fluorescence
  - ...

Philosophy

- Third-party CAT – available to whole biological community
- provide beam time commensurate with projected demand
- complete facilities from sample prep to data analysis
- flexible instrumentation - facilitate novel methods
- easy to use, streamlined setup
- staff on-call 24 hours

Current membership

National consortium of 40+ participants

Funding

Seeking funding as NIH Research Resource
Pilot seed funding provided by Kraft General Foods

Collaborations

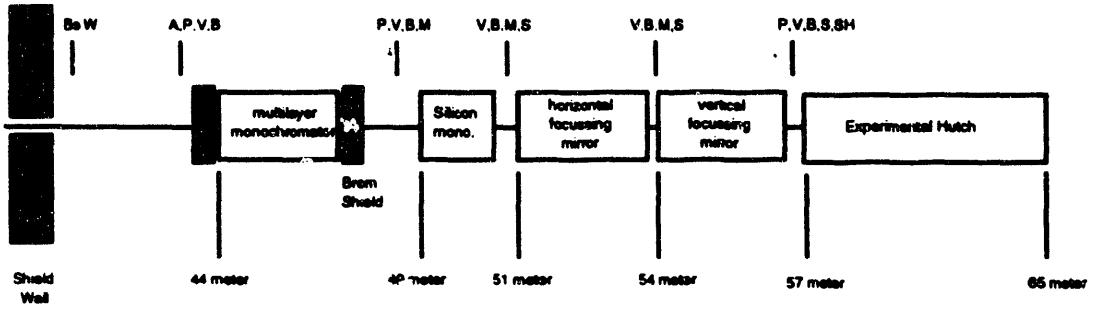
Collaborative instrumentation development with Structural Biology Center CAT and Biostructures PRT (X9) at NSLS

Participant in Center for Synchrotron Radiation Research and Instrumentation (CSRRI) at Illinois Institute of Technology (see poster)

Insertion Device line:

- APS type A taperable undulator, in series with APS type A wiggler
- multilayer premonochromator
- (optional) silicon double crystal monochromator
- (optional) side deflecting horizontal focussing mirror
- (optional) mirror for vertical focussing or harmonic rejection.
- low energy operation down to phosphorus K-edge

Block layout of BioCAT undulator beamline

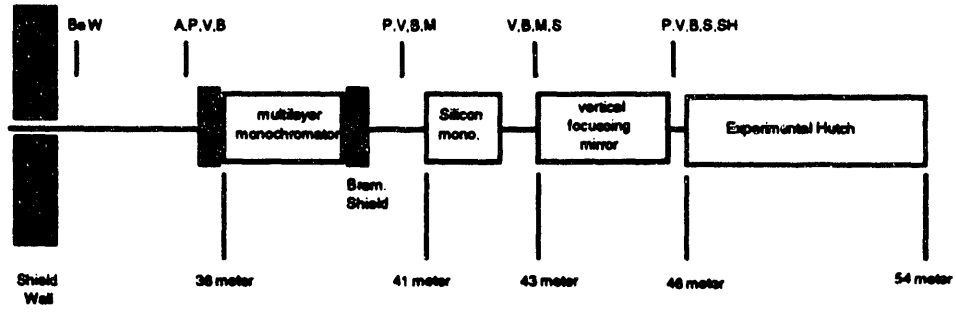


LEGEND
Be W: water cooled beryllium Window
A: water cooled primary aperture
P: pump
V: valve
B: bellows
S: slit
M: beam position monitor
SH: photon shutter

Bending magnet line:

- multilayer premonochromator (optional) horizontal sagittal focus
- (optional) silicon double crystal monochromator, with optional horizontal sagittal focus
- (optional) mirror for vertical focussing or harmonic rejection.

Block layout of BioCAT Bending Magnet beamline



LEGEND
Be W: water cooled beryllium Window
A: water cooled primary aperture
P: pump
V: valve
B: bellows
S: slit
M: beam position monitor
SH: photon shutter

Bending magnet line will be capable of low-energy experiments (down to phosphorus K-edge).

CARS-CAT

Consortium for Advanced Radiation Sources CAT

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*Dr. Keith Moffat became Director of CARS CAT on 1/1/93. Dr. J. V. Smith became Coordinator of Scientific Programs. CARS has relocated at the University of Chicago to 5640 South Ellis Avenue. Phone: 312/702-9951, Fax: 312/702-5454

CARS BOARD OF GOVERNORS

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John Johnson, Vice Chair
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William Bassett
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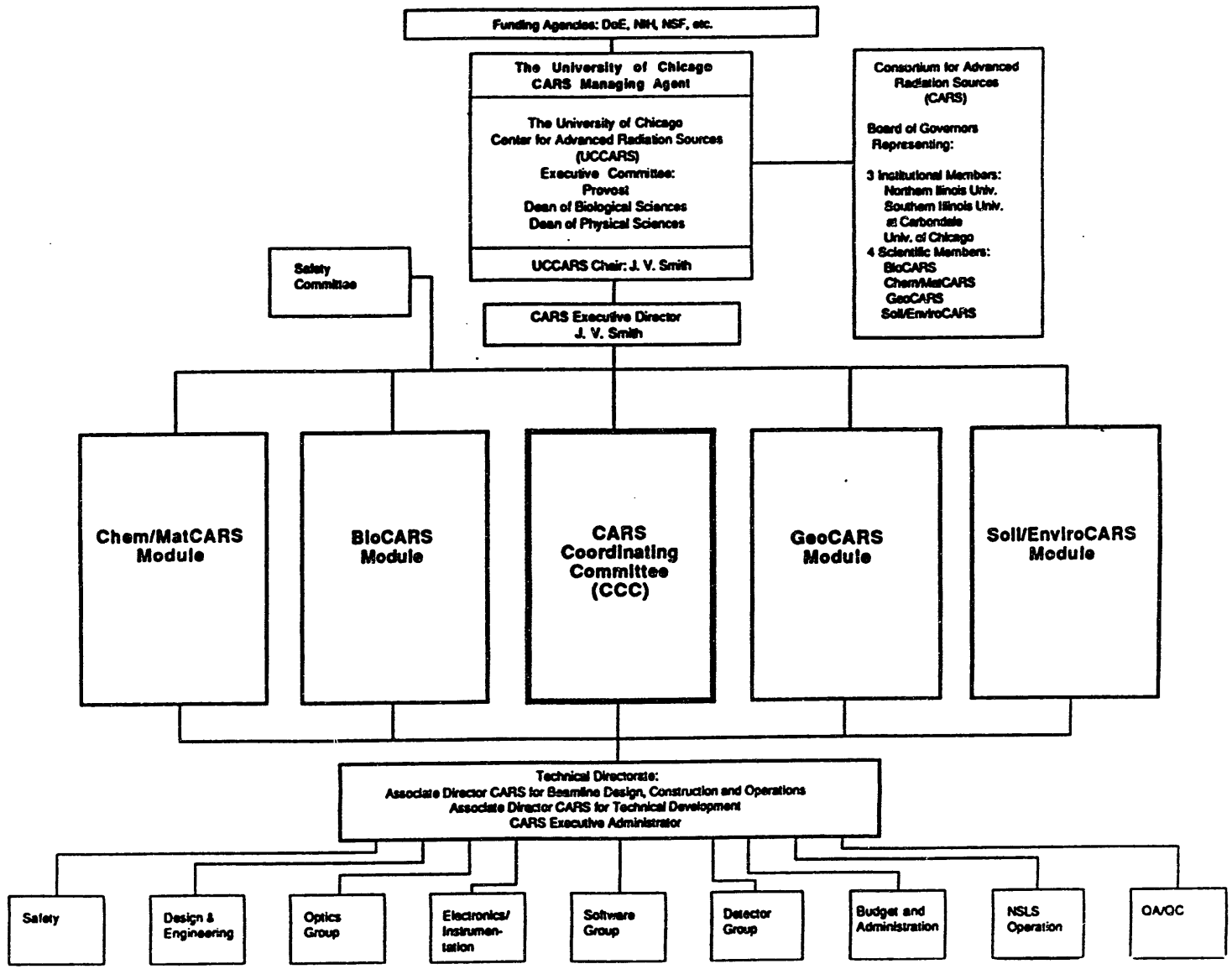
The University of Chicago

CARS ORGANIZATION CHART



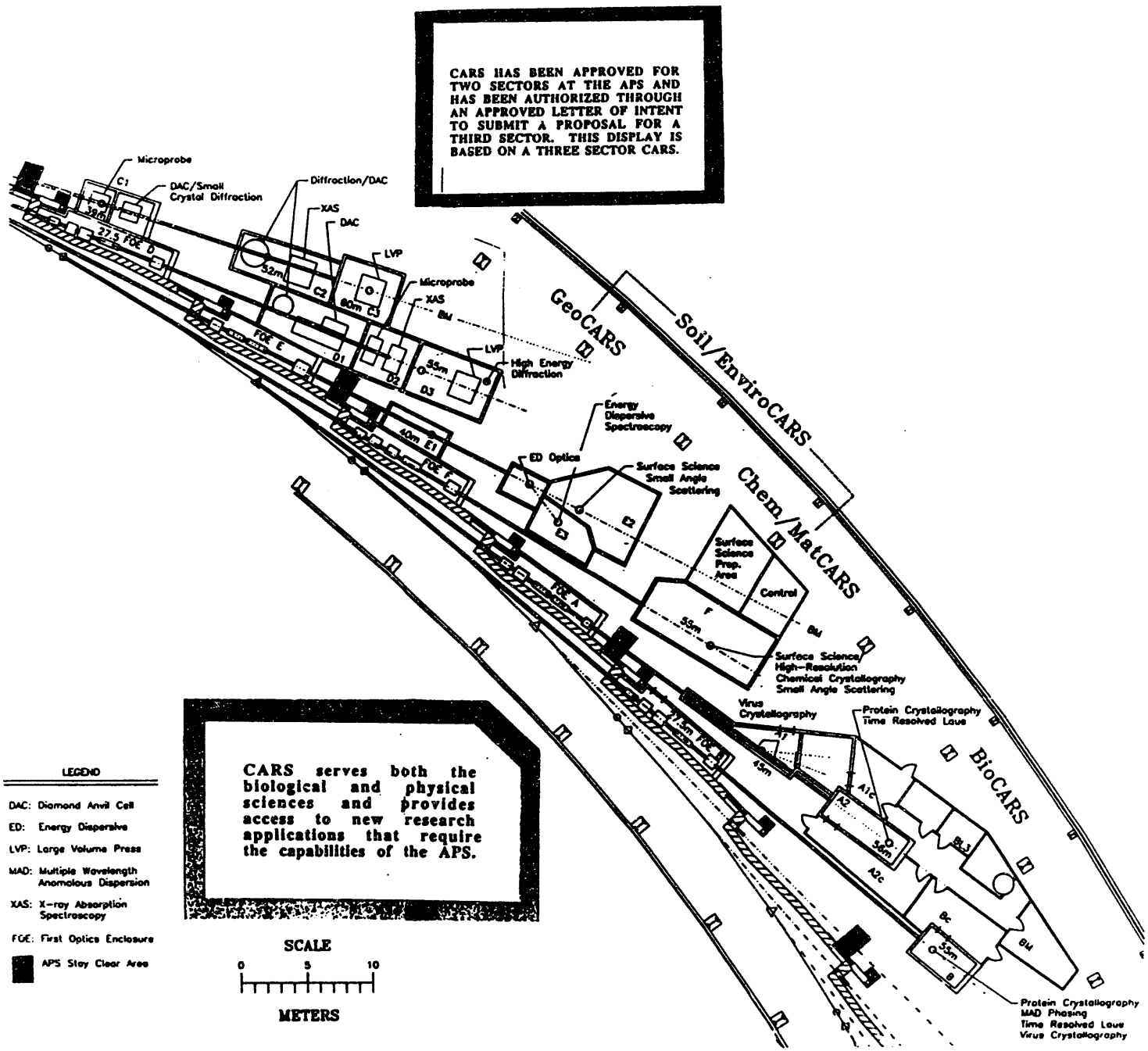
The Consortium for Advanced Radiation Sources consists of three universities:

- The University of Chicago
- Northern Illinois University
- Southern Illinois University at Carbondale

and four discipline-related national groups of scientists who need the capabilities of the APS for their research:

- BioCARS
- Chem/MatCARS
- GeoCARS
- Soil/EnviroCARS

The University of Chicago through the UCCenter for Advanced Radiation Sources is the managing agent for the consortium.


CARS HAS BEEN APPROVED FOR TWO SECTORS AT THE APS AND HAS BEEN AUTHORIZED THROUGH AN APPROVED LETTER OF INTENT TO SUBMIT A PROPOSAL FOR A THIRD SECTOR. THIS DISPLAY IS BASED ON A THREE SECTOR CARS.
Microprobe
DAC/Small Crystal Diffraction
Diffraction/DAC
XAS
DAC
LVP
Microprobe
XAS
LVP
High Energy Diffraction
GeoCARS
Soil/EnviroCARS
Energy Dispersive Spectroscopy
ED Optics
Surface Science Small Angle Scattering
Chem/MatCARS
Surface Science Prep. Area
Control
Surface Science High-Resolution Chemical Crystallography Small Angle Scattering
Virus Crystallography
Protein Crystallography Time Resolved Laue
BioCARS
Protein Crystallography MAD Phasing Time Resolved Laue Virus Crystallography
LEGEND
DAC: Diamond Anvil Cell
ED: Energy Dispersive
LVP: Large Volume Press
MAD: Multiple Wavelength Anomalous Dispersion
XAS: X-ray Absorption Spectroscopy
FOE: First Optics Enclosure
APS Stay Clear Area
CARS serves both the biological and physical sciences and provides access to new research applications that require the capabilities of the APS.
SCALE
0 5 10
METERS

GeoCARS, Soil/EnviroCARS SECTOR

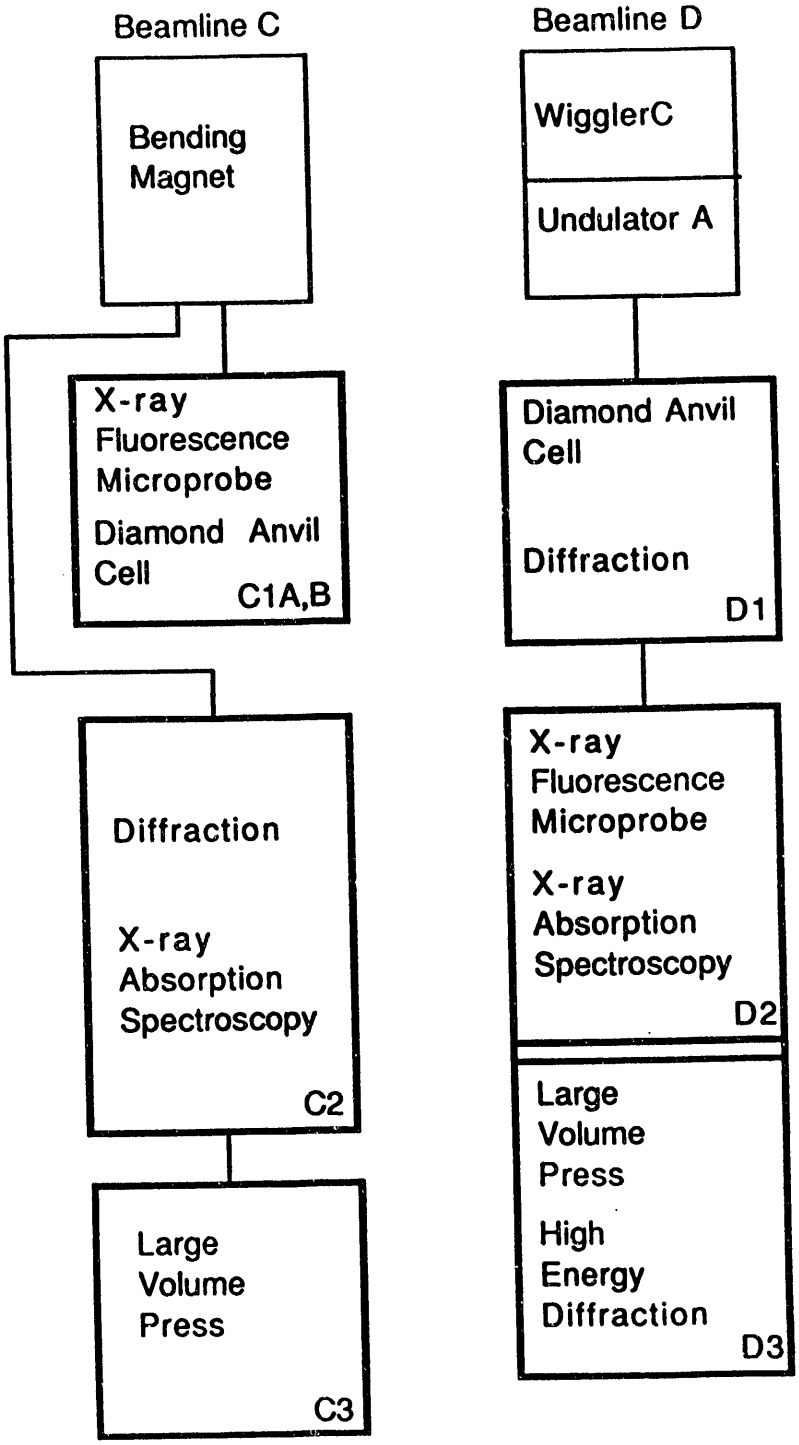
SCIENTIFIC FOCUS: Earth, Planetary, and Soil and Environmental Sciences

- High Pressure Research
  - Structure of Metals, Silicates, Ices, Oxides, Sulfides and Role in Geophysical and Seismic Phenomena
  - Reaction Kinetics and Rheology of Materials Involved in Earthquakes and Convective/Plume Motions
  - Element Partitioning
- Structures of Soil Minerals, "Stardust," Dense and High-Z Minerals, Gels, Liquids, and Surfaces
- Microprobe Trace Element Analysis of Materials Down to Sub-ppm Concentrations and Sub-μm Size
- Microtomography of Materials
- Properties of and Local Structure of Dilute Systems (Upper Mantle Materials, etc.), Gels, and Amorphous Materials and Melts
- Adsorption Complexes on Mineral Surfaces, Low-Temperature Geochemistry
- Biomineralization

PRINCIPAL EXPERIMENTAL TECHNIQUES:

- High Pressure (Diamond Anvil Cell and Large Volume Press)
- Energy/Angle Dispersive Powder and Single-Crystal X-ray Diffraction
- Fluorescence Microprobe
- Macro and Micro X-ray Absorption Spectroscopy
- Microtomography
- Anomalous Scattering
- Small Angle X-ray Scattering

Mainly GeoCARS, Soil/EnviroCARS



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Bingxin Yang
University of Chicago

BioCARS Sector

SCIENTIFIC FOCUS:

- Structure of Weakly Scattering Samples
  - Microcrystals and Single Muscle Fibers
  - Crystals with Very Large Unit Cells (Viruses and Multi-Subunit Complexes
  - Dilute Solutions

- Structure of Strongly Scattering Samples in Short Time Intervals
  - Time Resolved Crystallography
  - Time Resolved Diffraction from Non-Crystalline Samples
  - Single and Multiple Bunch Studies

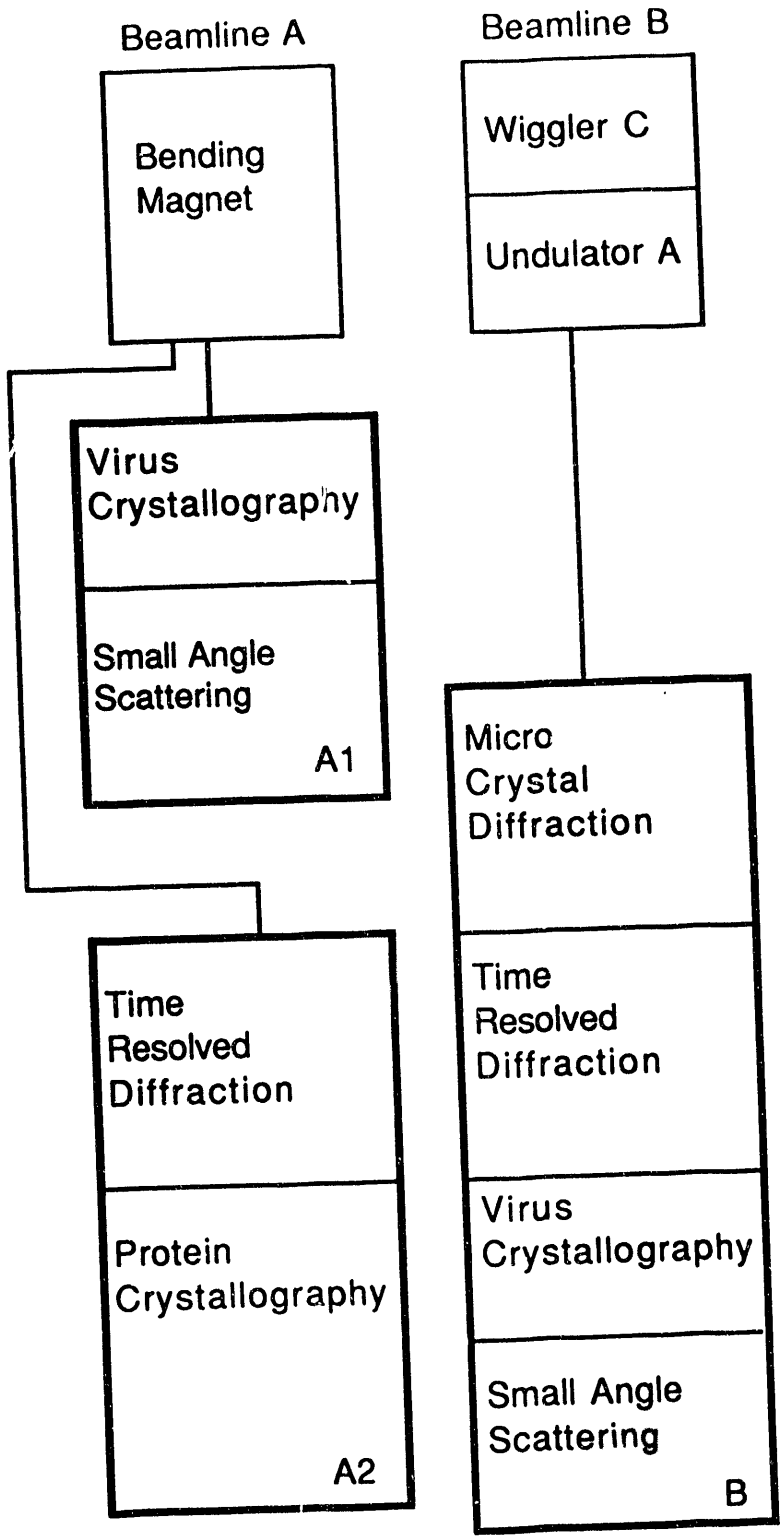
PRINCIPAL EXPERIMENTAL TECHNIQUES:

- Static and Time Resolved Crystallography

- Multiple Wavelength Anomalous Dispersion Phasing

- Static and Time Resolved Small Angle Scattering

Mainly BioCARS



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James Hogle
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GeoCARS has received significant support for design and development from the National Science Foundation, Earth Sciences (Instrumentation) and the Department of Energy, Geosciences.

Major funding for the design, construction, and initial phase operations of the BioCARS sector has been provided through a five-year Cooperative Agreement with the National Center for Research Resources, National Institutes of Health.

Chem/MatCARS SECTOR

SCIENTIFIC FOCUS: Chemical and Material Sciences

- Structure of Liquids, Polymers and Surfaces
- Structure and Properties of Super-Sized Metal Clusters
- Local Structure and Properties of Enzymes
- Crystallography
  - Microcrystallography
  - Anomalous Scattering
  - Time Resolved Studies of Pump-Probe Excited States, Solid State Reactions, Relaxation of Excited States, Response to External Perturbation
- Structure of Microporous and Layered Materials

Chem/MatCARS SECTOR

PRINCIPAL EXPERIMENTAL TECHNIQUES:

- Surface Science
  - Reflectivity
  - Grazing Incidence X-ray Diffraction
  - Surface Diffuse Scattering
  - Anomalous Grazing Incidence Diffraction, Fluorescence, and Reflectivity
  - Surface NEXAFS

- X-ray Absorption Spectroscopy (Static and Time Resolved)

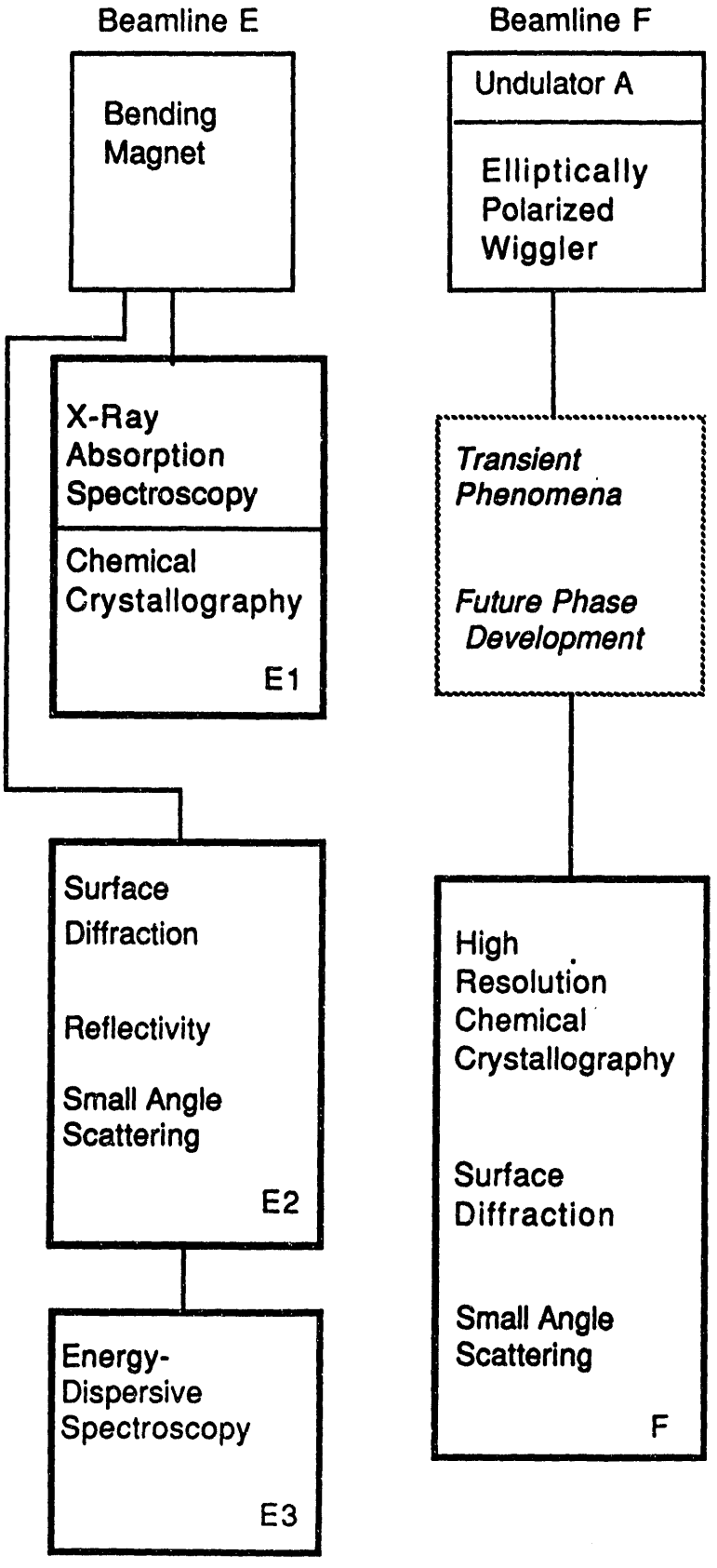
- Anomalous Scattering

- Small Angle Scattering (Static and Time Resolved)

- X-ray Diffraction, Including Energy Dispersive Techniques

- Use of Polarized X-rays

Mainly Chem/MatCARS



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L. D. GIBBS B. M. OCKO	BROOKHAVEN NATIONAL LABORATORY (PHYSICS)
J. K. BLASIE T. EGAMI J. E. FISCHER P. A. HEINEY	UNIV. OF PENNSYLVANIA
C. R. SAFINYA	UNIV. OF CALIFORNIA, SANTA BARBARA
E. W. PLUMMER D. ZEHNER	UNIV. OF TENNESSEE/ OAK RIDGE NATIONAL LABORATORY
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K. L. D'AMICO	X-RAY ANALYTICS, LTD.

CMC SCIENCE

(a) SURFACES, INTERFACES, AND THIN FILMS

Surfaces and Interfaces of Complex Fluids
Ultrathin Organic Films and Biomembranes, Self-Assembling Systems
Surface Roughening and Melting
Alloy Surfaces
Surface Overlayers, Film Growth, and Surface Reactions
Electrochemical Interfaces
Magnetic Scattering from Surfaces and Interfaces

(b) COMPLEX FLUIDS AND SELF-ASSEMBLING SYSTEMS

Structural Studies
Non-Equilibrium Steady-State Structures
X-Ray Correlation Spectroscopy
Kinetics of Phase Transitions in Complex Fluids

CMC SCIENCE (Cont'd)

(c) SOLIDS AND HETEROGENEOUS STRUCTURES

Microcrystallography and Powder Diffraction
Real Space Analysis of Aperiodic Systems with
High Energy Diffraction
Microdiffraction from Conjugated Polymers
Fibrils/Fibers
High Pressure Resolution Studies of Soft Solids

(d) AREA X-RAY DETECTOR DEVELOPMENT

APS UNDULATORS WILL IMPACT SURFACE AND INTERFACE SCIENCE IN FOLLOWING WAYS:

(a) BRIGHTNESS --

- Will Enable Study of 1μm Size Crystal Surfaces for Specular Reflectivity and Truncation Rods.

- Will Enable Diffuse Scattering Study for 1mm Size Surfaces.

- Will Enable Magnetic Scattering Studies of Surfaces.

(b) HIGH ENERGY --

- Will Enable Study of Buried Interfaces (Liquid/Solid, Solid/Solid), e.g., in Polymer Absorption, Electrochemistry, etc.

APS UNDULATORS WILL IMPACT SURFACE AND INTERFACE SCIENCE IN FOLLOWING WAYS (Cont'd.):

(c) TUNABILITY --

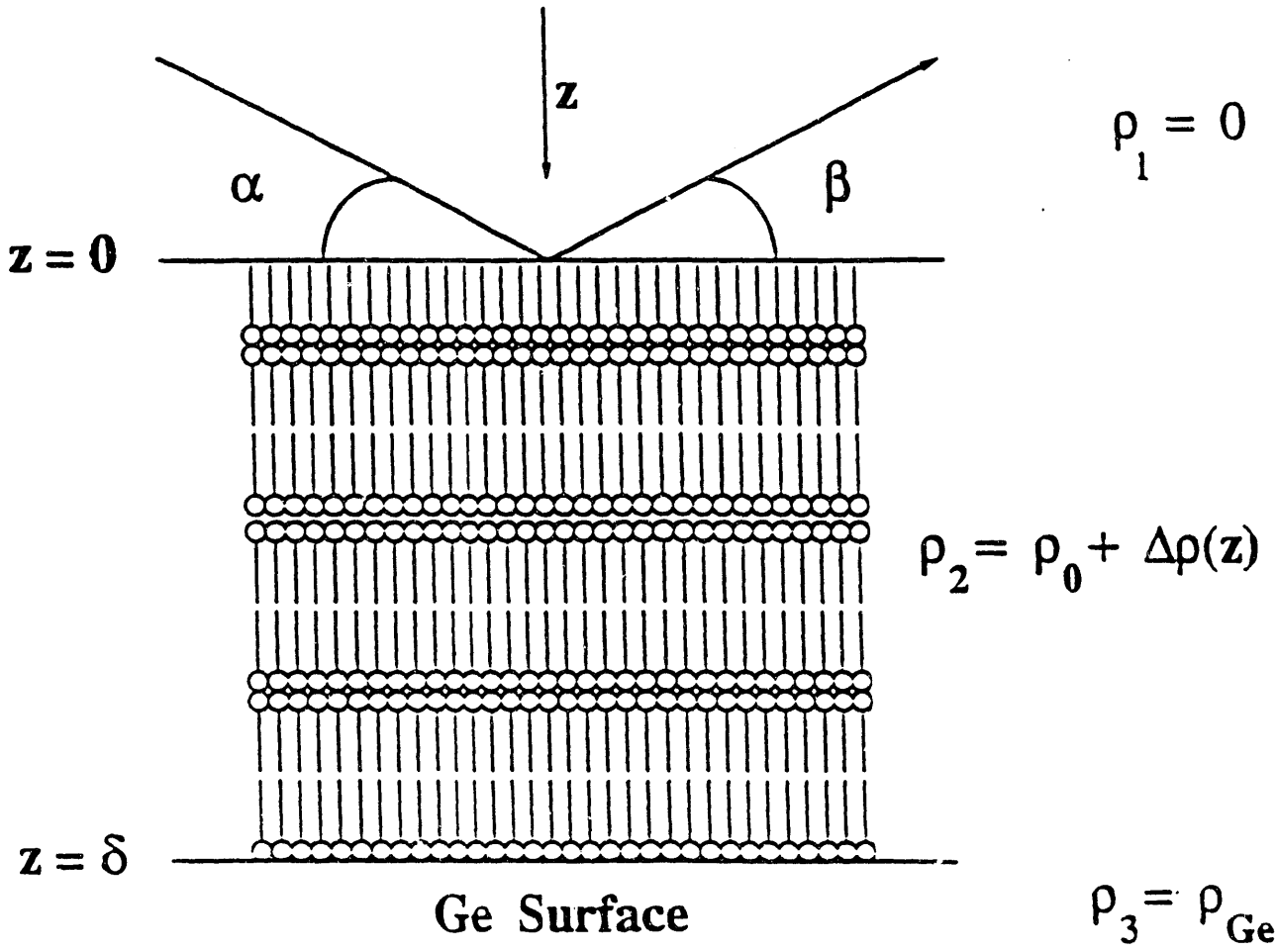
- Will Enable Anomalous Reflectivity and Surface Diffraction Studies.

(d) BRIGHTNESS AND FAST AREA DETECTORS --

- Will Enable Studies of Surface Phase Transitions and Absorption Kinetics.

ANOMALOUS REFLECTIVITY TECHNIQUE

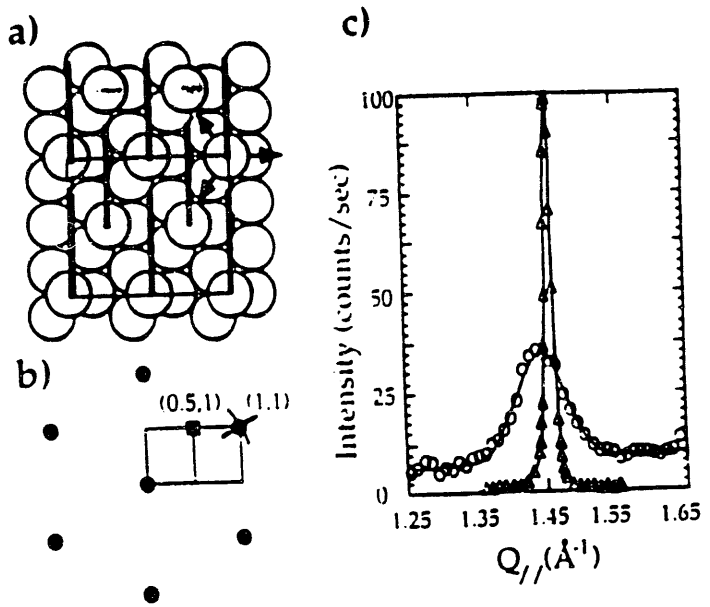
FOR DENSITY PROFILES OF THIN FILMS

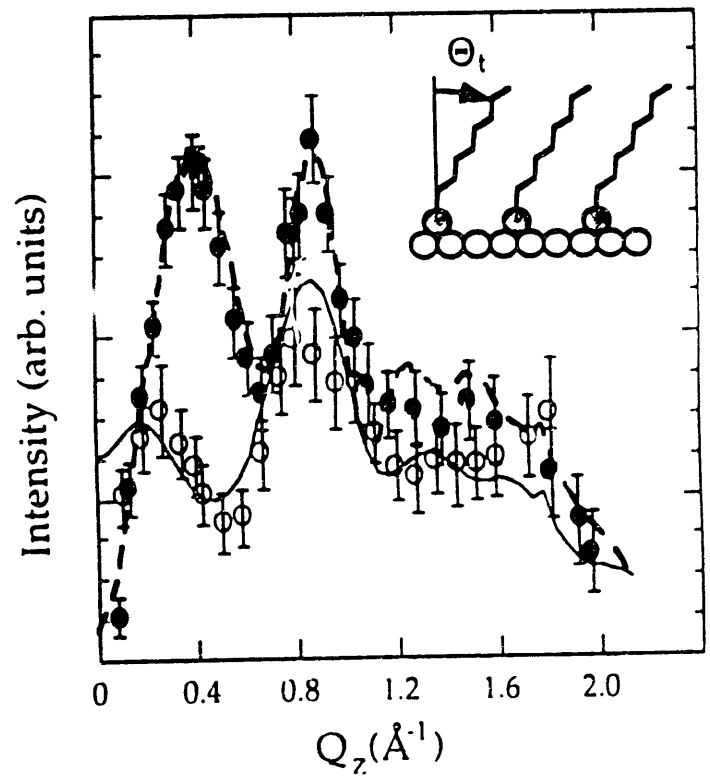


3 1/2 bilayer Cd Arachidate LB film on Ge substrate.

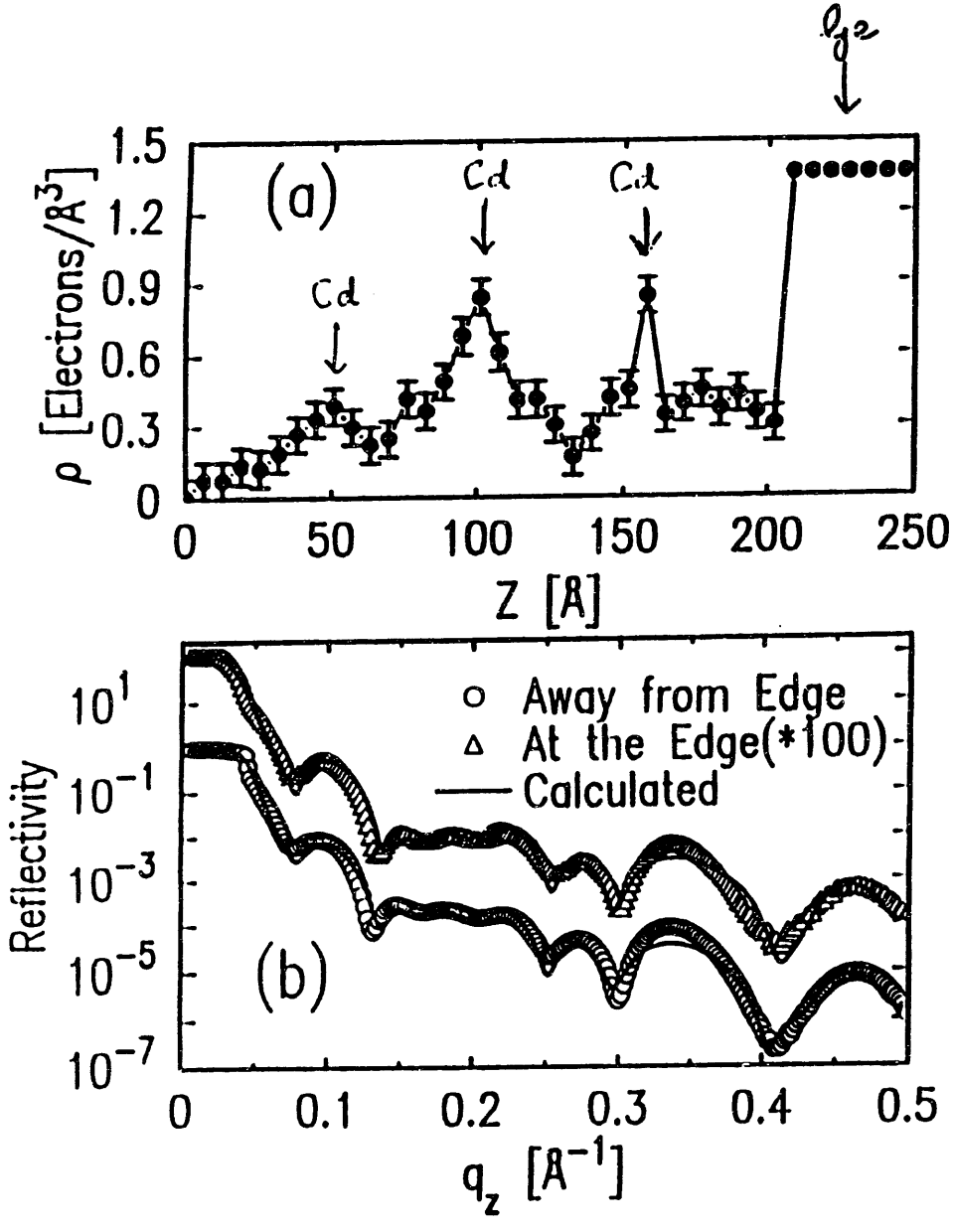
2-DIMENSIONAL SCATTERING

FROM SELF-ASSEMBLED MONOLAYERS





In-plan and rod scans of self-assembled $CH_3(CH_2)_nSH$ monolayers on Au(111) substrate.



Density profile as obtained by anomalous X-ray reflectivity.

Reflectivity profiles at Ge edge (11,103 eV) and below edge as measured and as calculated from inverted profile above.

DUNU CAT*

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* As of December 1992, Dow Chemical Co. joined DUNU CAT, and the name was changed to DND CAT.

SCIENTIFIC PROGRAM

Advanced X-ray structural research on new materials:

- Structure of materials
- Polymer science and technology
- Protein crystallography
- Surface and interface science

Approximately twenty-five principal investigators and fifty other scientists, engineers, students, and post-doctoral fellows currently involved.

Excellent overlap of interests between Du Pont and Northwestern; joint research already under way.

SURFACES, INTERFACES AND THIN FILMS

The structure and composition of surfaces and interfaces control fundamental properties of materials used in many important applications, such as catalysis, corrosion, tribology, crystal growth, electrodeposition, surface epitaxial growth and segregation at grain boundaries. Some specific areas of interest to our CAT are:

- Solid-liquid electrolyte interfaces. Its structure is of fundamental interest in processes involving charge transfer or chemical reactions at the interface (such as electrodeposition, corrosion and power generation and storage).
- Oxide surfaces. Specific areas include supported metal catalysts (in particular strong metal-support interactions) and tribology (high temperature lubricants).
- Intercrystalline interfaces. The structure and chemistry of grain boundaries in engineering materials have a profound effect on their mechanical properties. Model (bicrystal) and natural grain boundaries will be studied.
- *In situ* studies of thin film growth. Ongoing research programs involve diamond and fullerene films, as well as oxide superconductor films.
- Polymer film surfaces. Surface structure of commercial films (Kapton®, PET and Mylar®) greatly affects certain properties, such as adhesion, wettability and abrasion resistance.

We will employ a broad spectrum of x-ray scattering and spectroscopy techniques, including Surface Extended X-ray Absorption Fine Structure spectroscopy (SEXAFS), X-ray absorption near edge spectroscopy (XANES), X-ray standing waves (XSW), X-ray reflectivity and x-ray fluorescence microprobe (XFM). Many members of our CAT have direct and extensive experience with most of these techniques at SR facilities and have been involved in many of the forefront experiments in surface and interface science. For most of these studies, even exploratory experiments on model systems are at the limit of what is possible today.

POLYMER SCIENCE AND ENGINEERING

The second major "thrust" area for our CAT is polymer physics, chemistry and engineering. Areas of interest span the entire spectrum, including solution characterization of the polymers and their precursors, structure formation, structure/property/processing relationships and mechanical properties (fracture, crazing).Highlights of the proposed research include:

- Polymer deformation. "Cold Drawing" is a well-established method for enhancing mechanical properties, particularly stiffness and strength. The structure evolution in the deformation zone remains, however, obscure. Time-resolved microbeam diffraction will be used to study a variety of polymers.
- Crystallization and melting of polymers. Neither mechanism is totally understood. Numerous models for different polymers have been proposed; many cannot be confirmed because existing structure determination methods are not fast enough. SAXS and wide-angle time-resolved studies at the APS will help resolve these issues.
- Polymer deformation and fracture. Craze initiation, growth and breakdown have been studied extensively in glassy, optically transparent amorphous polymers. Semicrystalline polymers or composites, however, are opaque. Microtomography is the only technique that can provide information on internal cracking and damage.

Two techniques crucial to the success of this program will be made substantially available

only through the APS: microbeam diffraction and microtomography. Our CAT will develop or adapt experimental "know how" in both areas.

ATOMIC STRUCTURE OF BULK MATERIALS

X-ray diffraction has provided the bulk of what we now know about atomic arrangements in materials. The APS will allow us to move to the next level of detail in atomic structure. Some of proposed research projects are:

- Arrangements of dopant atoms and other lattice defects in the structures of metals, catalysts and superconductors. Due to the low concentrations and lack of long range order, scattering from dopant atoms and lattice defects is weak and diffuse. Large increases in flux are necessary to study such important materials as steel, engineering aluminum alloys and oxide superconductors.
- *In situ* studies of catalysts. Commercial catalysts operate at elevated temperatures and pressures. Existing x-ray sources do not produce radiation hard enough to penetrate reactor vessels operating under realistic conditions. Catalysts of interest to us include supported metal and bimetallic catalysts (Pd, Pd-Re), zeolites and catalysts for environmentally benign chlorofluorocarbons.

DUNU CAT

PROPOSED RESEARCH BY THE SURFACE SCIENCE GROUP

---

1. Adsorbate structures on semiconductor surfaces

Halogen monolayers on Si and Ge (111)
Chalcogenides on GaAs

Techniques:
(a) X-ray standing wave technique to determine the position and vibration amplitudes of absorbate atoms
(b) SEXAFS to determine local atomic arrangments

PI - Prof. M. Bedzyk, NU

2. Highly oriented thin organic films

- measure arrangments of heavy atoms within highly oriented LB films using the XSW technique

- determine electron density profile

- study ionic gradients at the liquid/solid interface

PI - Prof. M. Bedzyk, NU
Drs. Van Alston & Hsiung, Du Pont

DUNU CAT

PROPOSED RESEARCH BY THE SURFACE SCIENCE GROUP

---

3. Nanostructures - thin films and small particles

surface structure of Bi and Sn thin films on
InSb and CdTe by grazing-incidence XRD

lattice dynamics/melting of small particles
(deposition by a nanosphere or STM technique)

surface structure of oxide surfaces

- molybdenum trioxide as a model
  catalyst and high temperature lubricant

PI: Chung, Cohen, Georgopoulos and Stair, NU
Blackman, Coulman and Firment, Du Pont

4. Other Areas

(a) polymer surfaces

orientation, chain packing and phase separation

possible systems - Kapton and Mylar

PI - Carr and Crist, NU
Van Alsten and Firment, Du Pont

(b) Real catalysts

EXAFS/XANES

PI - Sachtler, NU
Gai, Du Pont

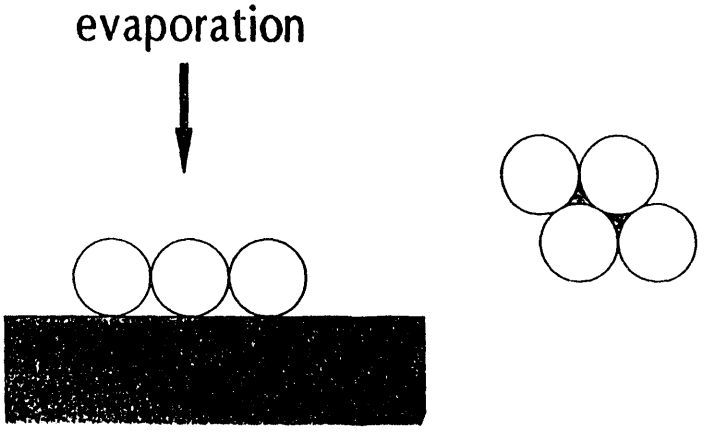
(c) Photolectron microphobe or imaging

PI - Chung and Stair, NU
Blackman, Du Pont

DUNU CAT

Proposed Research by the Surface Science Group

Nanosphere Lithography



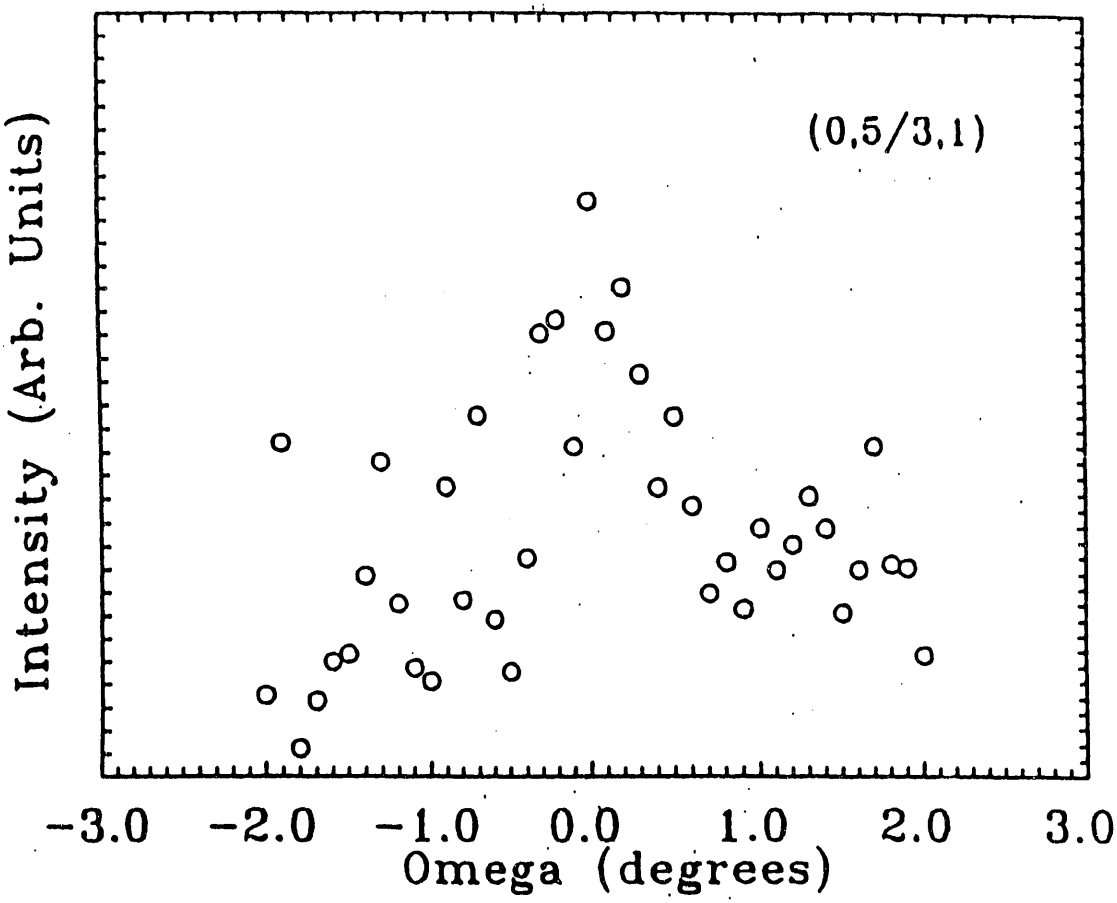
STM Lithography

1. Use the fine electron probe to induce a CVD type reaction,

   e.g. $(CH_3)_3Al + \frac{3}{2}H_2 \longrightarrow 3CH_4 + Al$

2. Field-desorb metal clusters from the STM tip,

   e.g. Au or Sn (low desorption threshold)



Rocking curve of *0,5/3,1* diffraction peak.

TiO_2(100)- 1×3
X18A , λ = 1.3 Å

POLYMER PHYSICS, CHEMISTRY AND ENGINEERING

NORTHWESTERN UNIVERSITY:

Polymer Research Core: 8 Faculty
Allied Research in Polymers: 9 Faculty

This is > 10% of all engineering and physical science faculty.

DuPONT COMPANY:

- One of the world's largest staffs of physical scientists and engineers with responsibility for polymeric products.

- Significant involvement with synchrotron radiation experiments.

POLYMER PHYSICS

CRYSTAL FORMATION

RIGID ROD POLYMERS:

- They form bundle nuclei
- They grow fibrillar crystals, having only low angle grain boundaries in the final solid.

Question: What laws govern solidification and crystal growth?

- Existing bundle nucleation models need to be extended to the case of rigid rod polymers.
- Is phase transformation from liquid to crystal by bundle growth or by bundle accretion?
- How does crystal interconnectivity give rise to observed properties?

CONSEQUENCE OF UNDERSTANDING:

- Development of a structure-based model for the extreme strength and stiffness exhibited by rigid rod polymers.
- Sets foundation for designed creation of multi-functional polymers solids.
- Extends current understanding of behavior of macromolecules to limit of vanishing flexibility.

CRYSTAL FORMATION

FLEXIBLE CHAINS:

- They form lamellar crystals.
- Chains partition defective segments between inside and outside of crystals.

Question: What laws govern this behavior?

- There are intra- and inter-chain kinetic considerations.
- There are intra- and inter-chain entropic considerations.
- There are intra- and inter-crystalline strain energy considerations.

CONSEQUENCE OF UNDERSTANDING:

- Tests of local chain dynamic theories.
- Tests of crystal packing energetics.
- Improved "semicrystalline" plastics.

POLYMER CHEMISTRY

SOLIDIFICATION BY IMIDIZATION

POLYIMIDES:

- Are central to new high-temperature resistant polymers.
- Are central to new materials for electronics packaging.

Question: What laws describe concomitant polymerization and Solidification?

- How do present ideas of chemical reactivity and reaction kinetic change in the case of densely crosslinked polymers?
- How do different chemical compositions affect final order developed in polyimide solids?

CONSEQUENCE OF UNDERSTANDING:

- Development of a chemical structure-based model for outstanding mechanical and dielectric properties.
- Sets foundation for creation of succeeding families of thermosets.
- Extends current understanding of behavior of macromolecules to limit of vanishing flexibility.

ZEOLITES

Extremely important in catalysis.

Need to know atomic configuration in unit cell under catalytic reactor conditions.

Current limitations :

- o Suitably large single crystals cannot always be obtained.
- o In situ experiments difficult with current x-ray sources.

APS benefits:

- o Multifold reduction in minimum sample size (Laue techniques are being considered also).
- o Because of high intensity available at short wavelengths, heavy containment vessels can be used so that catalysts can be studied at pressures more representative of true reactor conditions.

SUPERCONDUCTING OXIDES

Need to obtain the average and local atomic structure in fine detail.

Need to study subtle phase transitions common in these materials and their effect on the superconducting properties.

Current limitations:

- Suitably large single crystals cannot always be obtained.
- Superlattice reflections are very weak and difficult to measure.
- Serious absorption and extinction effects hard to treat effectively.
- Low intensities and a restricted wavelength range limit studies to materials with high average lattice symmetry.

APS benefits:

- Studies of local atomic arrangements in multi-component systems with resonant scattering techniques.

ATOMIC STRUCTURE OF BULK MATERIALS

- Arrangements of dopant atoms and other lattice defects in the structures of metals, catalysts and superconductors. Due to the low concentrations and lack of long range order, scattering from dopant atoms and lattice defects is weak and diffuse. Large increases in flux are necessary to study such important materials as steel, engineering aluminum alloys and oxide superconductors.

- *In situ* studies of catalysts. Commercial catalysts operate at elevated temperatures and pressures. Existing x-ray sources do not produce radiation hard enough to penetrate reactor vessels operating under realistic conditions. Catalysts of interest to us include supported metal and bimetallic catalysts (Pd, Pd-Re), zeolites and catalysts for environmentally benign chlorofluorocarbons.

IMCA CAT

Industrial Macromolecular Crystallography Association CAT

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The Industrial Macromolecular Crystallography Association X-ray Beamlines at the Advanced Photon Source

Noel D. Jones[1], Sorinel Cimpoes[2], Jay Min Lee[2], and Timothy I. Morrison[2]

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X-ray crystallography is the technique of choice for the determination of the structures of biological macromolecules and is playing an increasingly important role in protein engineering and structure-based design of biologically active small molecules. Over the past decade there has been dramatic growth of macromolecular crystallography in industry worldwide (see Fig. 1).

The Industrial Macromolecular Crystallography Association (IMCA) is a consortium of twelve major pharmaceutical and chemical companies, each of which is engaged in macromolecular structure research in the United States.

Abbott Laboratories	Miles, Inc.
Bristol-Myers Squibb	Monsanto Company
E. I. du Pont de Nemours	The Proctor & Gamble Company
Glaxo, Inc.	SmithKline Beecham
Eli Lilly and Company	Sterling Winthrop, Inc.
Merck & Co., Inc.	The Upjohn Company

The association was formed for the purpose of developing beamlines for x-ray diffraction research at the Advanced Photon Source, the 7-GeV synchrotron currently under construction at Argonne National Laboratory near Chicago. Companies which are members of IMCA will share equally in development and operating costs and in access to beamline facilities but *will carry out independent research*. IMCA is a cooperative effort intended to provide, on a shared basis, research facilities which would not be cost effective for individual companies to develop and operate.

Illinois Institute of Technology, through its Center for Synchrotron Radiation Research and Instrumentation, will collaborate with IMCA in the design, construction, and operation of two beamlines at the APS and will share in their use. Our goal is to develop a high throughput, fast access facility. We anticipate making 25% of the beam time available to noncommercial, third party users.

The layout of the two beamlines, one on a bending magnet and the other on a wiggler, is shown in Fig. 2. Each will be configured to permit monochromatic, multiwavelength or wide-bandpass Laue diffraction studies in the 6 to 25 KeV energy range. The optical components for the two beamlines, as shown in Fig. 3, are essentially the same except for their distances from the radiation source. The fixed exit monochromator (G) has two optical elements, the first a flat, gallium-cooled crystal and the second a sagittally bent crystal. Vertical focusing and harmonic rejection for monochromatic radiation will be accomplished by a

rhodium-coated mirror (J) which is dynamically bent in the tangential direction. The monochromator will be quickly tunable for multiwavelength experiments or may be translated out of the beam to obtain white radiation (dotted line, Fig. 3) for Laue diffraction studies. A bent cylindrical (toroidal) mirror (M) coated with either rhodium or platinum will be used for white radiation focusing. No decision has yet been made on the type of goniostat or x-ray detector which will be used except that both will be as automated as possible.

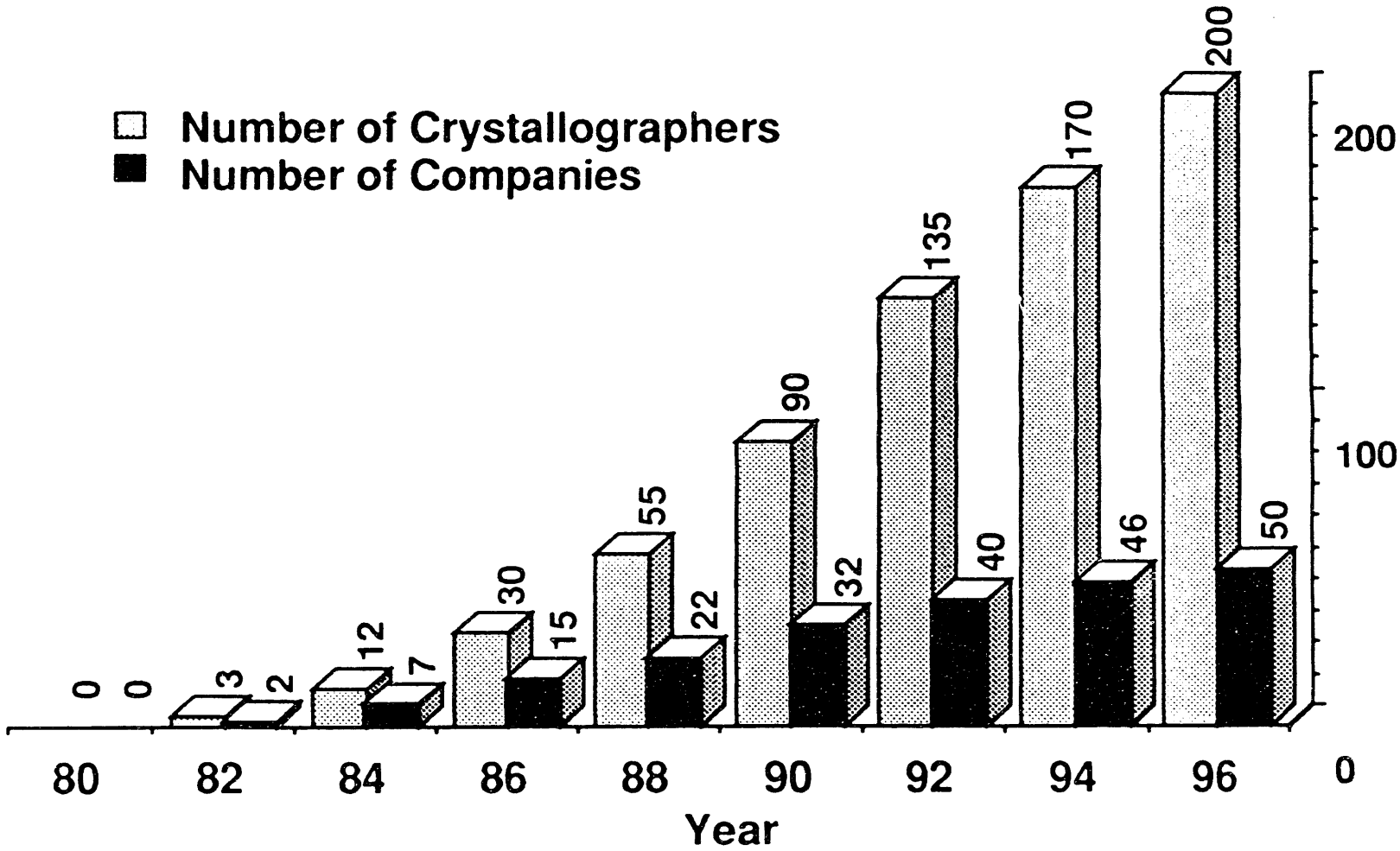

Fig. 1. Growth of macromolecular crystallography in industry worldwide, 1980-92, with projections through 1996.

Legend

A	Beryllium window
B	Beam conditioner
C	Ion pump
D	Lead collimator
E	Horizontal, vertical white beam slits
F	Valve
G	Two crystal monochromator
H	Beam position monitor
J	Tangential bending mirror
K	Photon safety shutter
L	Experimental hutch
M	Bent cylindrical (toroidal) mirror
N	Shielding

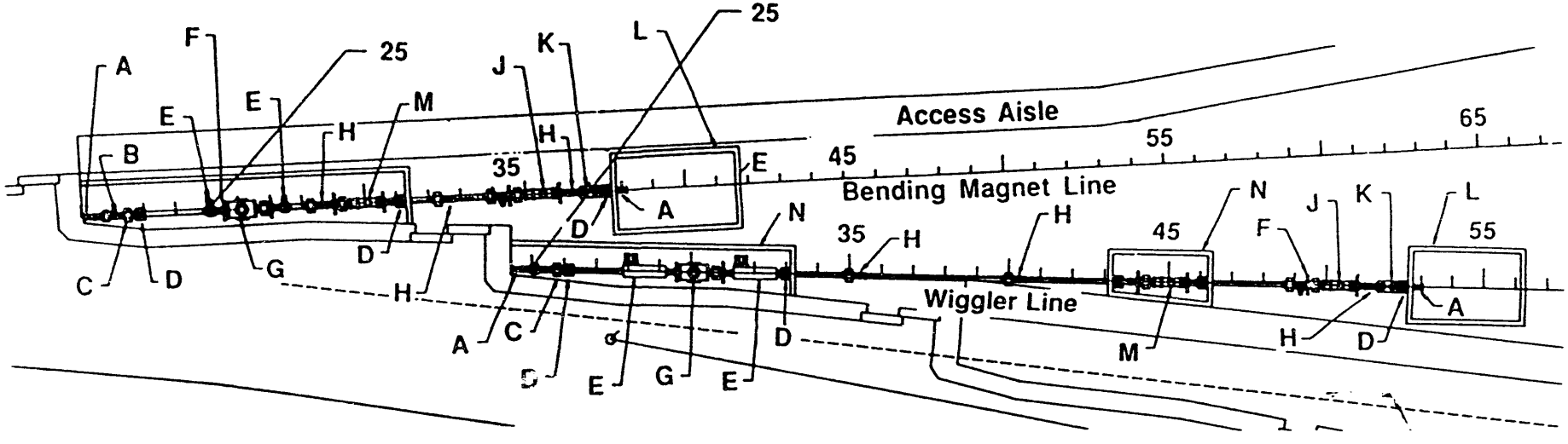

Fig. 2. Plan view of layout of beam lines on experimental floor

Legend

A	Beryllium window
B	Beam conditioner
C	Ion pump
D	Lead collimator
E	Horizontal, vertical white beam slits
F	Valve
G	Two crystal monochromator
H	Beam position monitor
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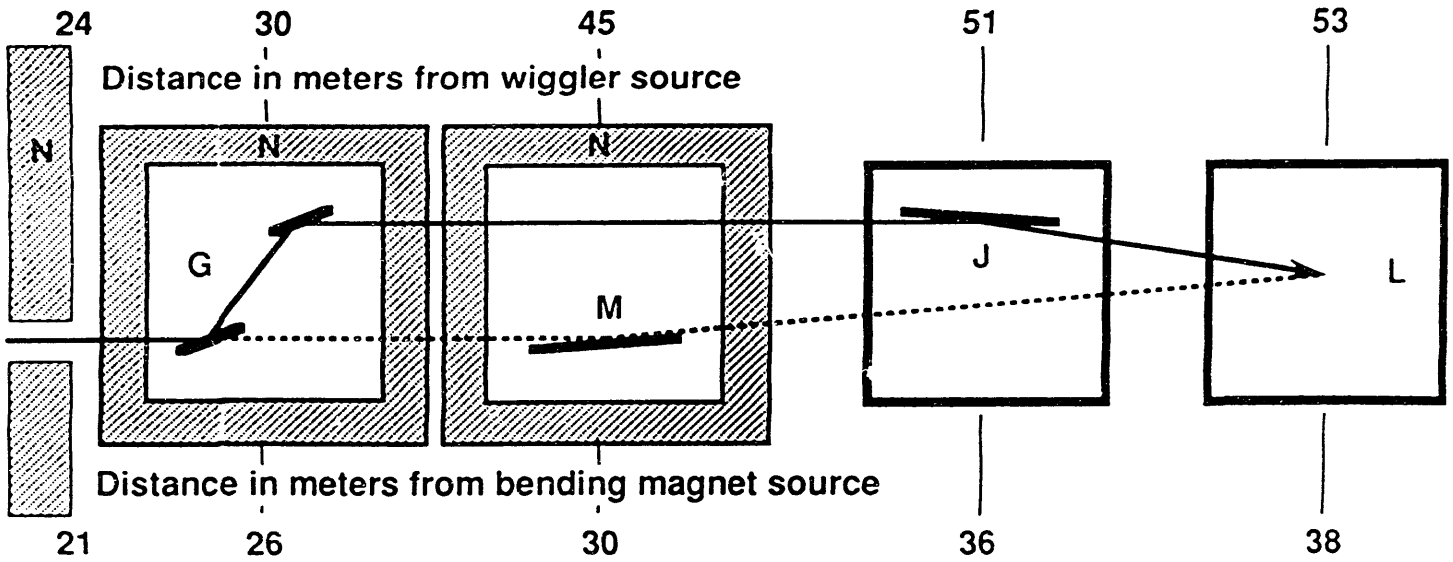

Fig. 3. Schematic of optical components for bending magnet and wiggler beam lines

IMMCAT

IBM-MIT-McGill CAT

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IBM-MIT-McGill Collaborative Access Team

Focus of research: Dynamic phenomena in materials science and condensed matter physics

Institution:	CAT Members:
IBM	Cargill, Held, Jordan-Sweet, Stephenson Gordon, Melroy, Toney
MIT	Birgeneau, Greytak, Litster, Mochrie
McGill	Singh, Sutton

Resources for CAT provided in proportion to size of respective research programs:

Initially

5/12	IBM
5/12	MIT
2/12	McGill

Proposed IMMCAT Research Program

Surfaces and Buried Interfaces: Equilibrium Structure

- Highly reactive metal and semiconductor surfaces
- Low Z surfaces: polymers, He
- Structure of magnetic multilayers and thin films
- Structure of electrochemical double layers

Surfaces and Buried Interfaces: Kinetics

- Millisecond resolution studies of surface diffusion, step and vacancy motion, and reconstruction
- *In situ* studies of MBE, CVD, and electrochemical growth kinetics
- Structure of buried interfaces during deposition
- Kinetics of phase transitions in 2-D overlayers

Bulk Phase Transition Kinetics

- Microsecond- or high-wavenumber-resolution studies in binary alloys, polymer blends and polymer solutions

Intensity Fluctuation Spectroscopy using Coherent X-rays

- Equilibrium fluctuation dynamics
- Fluctuations during growth

Sub-Micron Imaging

- Microtomography of electronic devices
- Diffraction microprobe for strain determination

MATT-CAT

Center for Real-Time X-ray Studies

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Robert MacHarrie
Mathew Marcus
Ron Pindak

University of Michigan

James Allen
Meigan Aronson
Michael Bretz
Roy Clarke (Deputy Director)
Steve Dierker
John L. Gland
James Penner-Hahn

Associate Members

Ben C. Larson — Oak Ridge National Laboratory
Jon Tischler

Areas of interest:

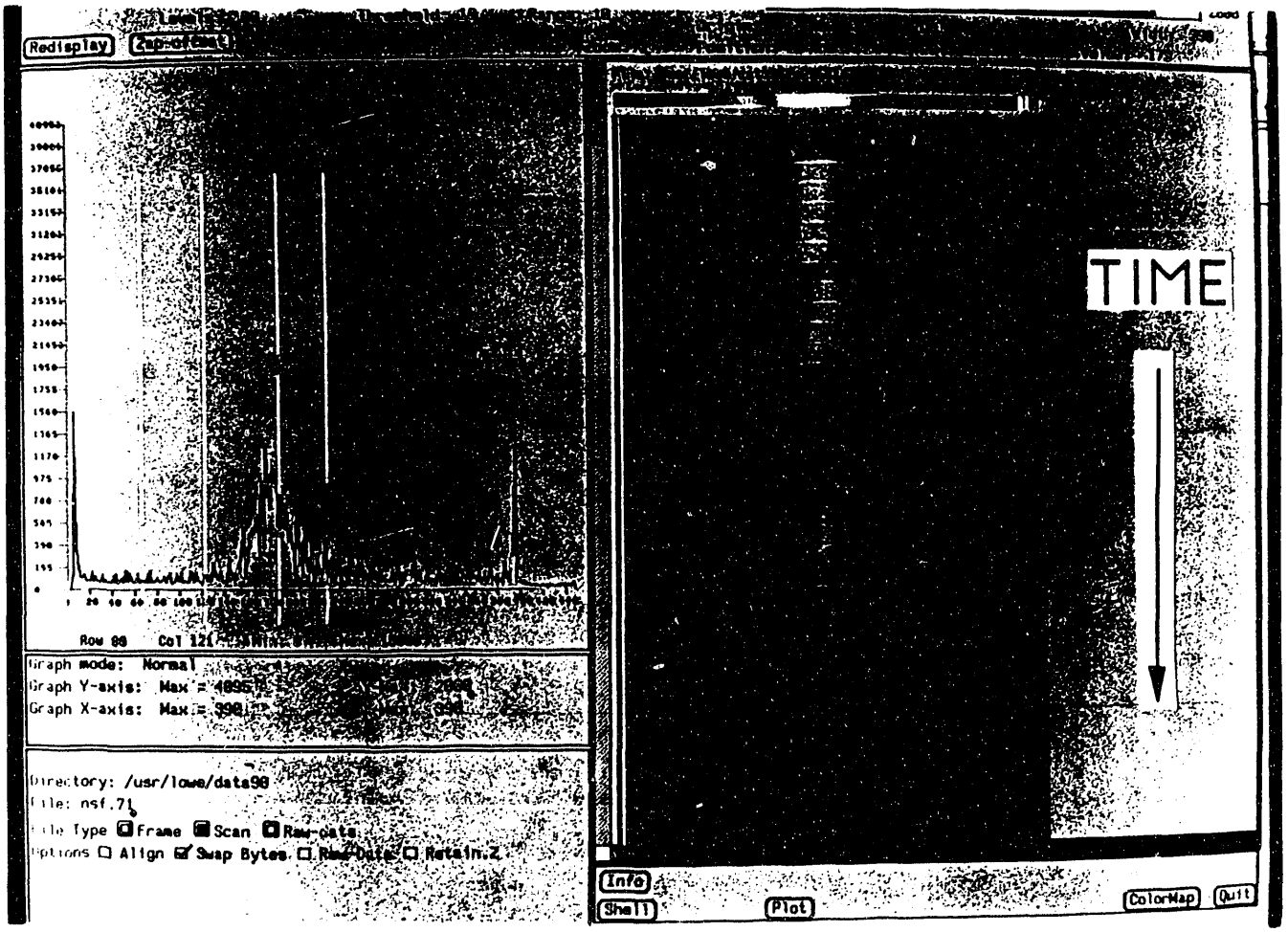
- Time-resolved studies of materials
- Photon correlation spectroscopy
- Chemical sciences
- Biochemical studies

Technical Design

- Type A undulator beamline
- Vertical and horizontal focusing optics
- Preservation of traverse coherence
- Bending magnet beamline with sagittal focusing optics

Adapted from W. Lowe, R. A. MacHarrie, J. C. Bean, L. Peticolas, R. Clarke, W. Dos Passos, C. Brizard, and B. Rodricks, Phys. Rev. Lett. **67** (1991) 2513

TIME RESOLVED X-RAY SCAN DURING RAPID THERMAL ANNEALING

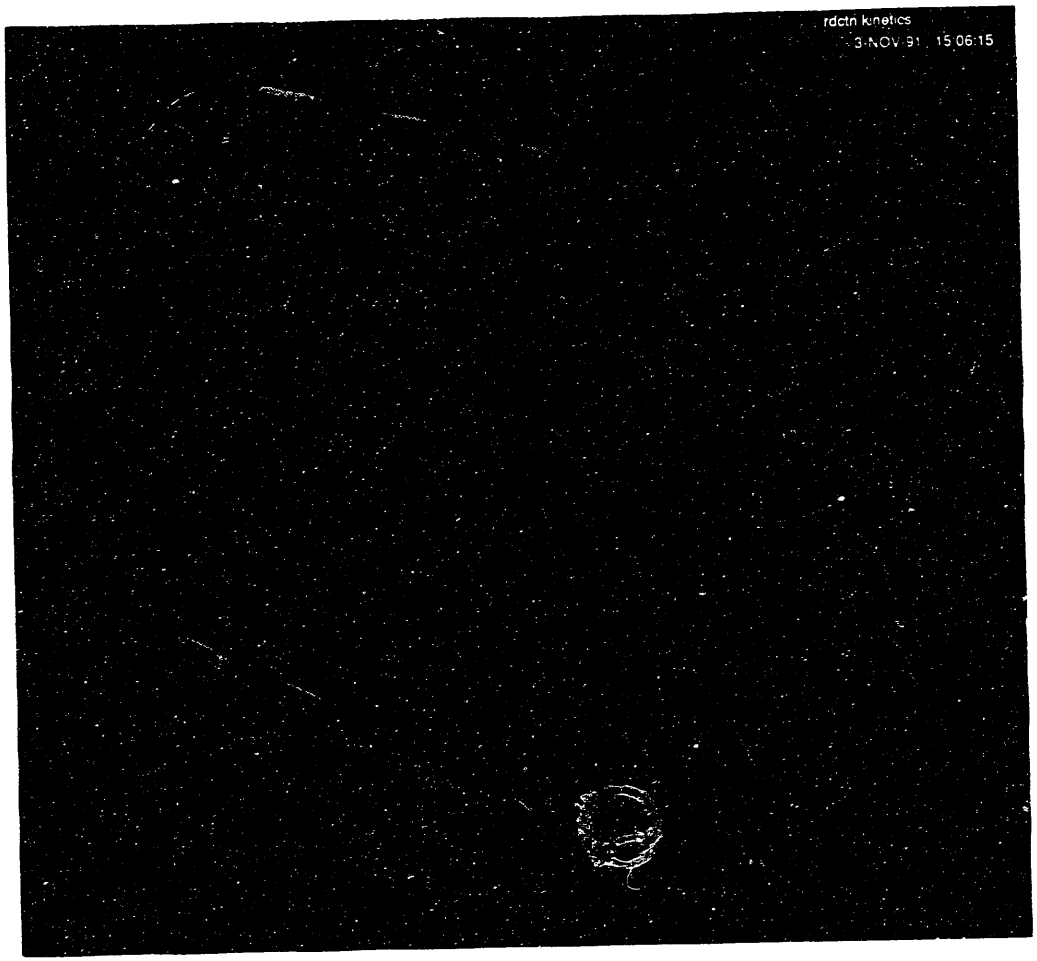


DIFFRACTION ANGLE

Adapted from P. G. Allen, S. D. Conradson, and J. E. Penner-Hahn, Synchrotron Radiation News **5** (1992) 16

Time-Resolved Catalysis Studies

PRINT THIS SIDE



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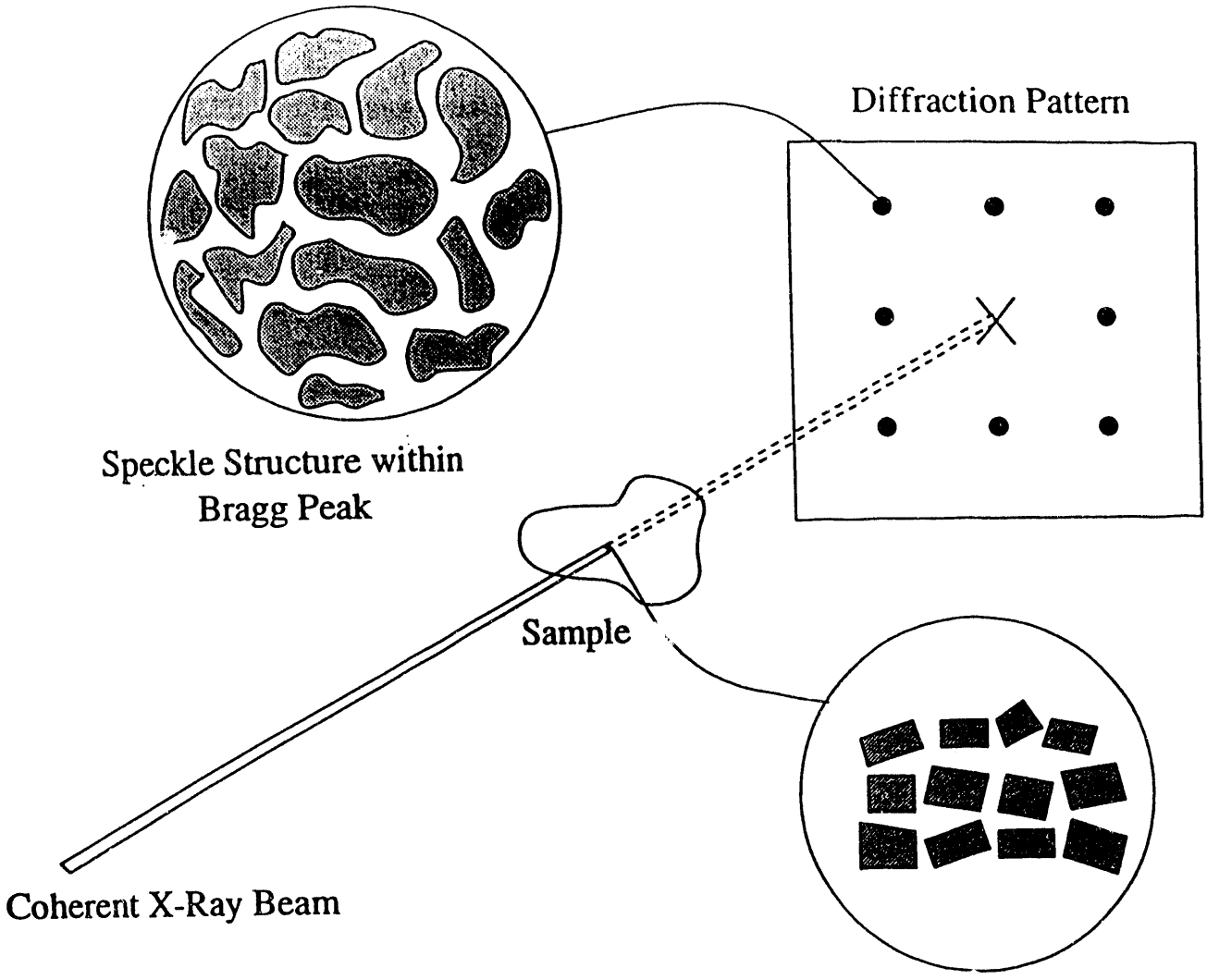
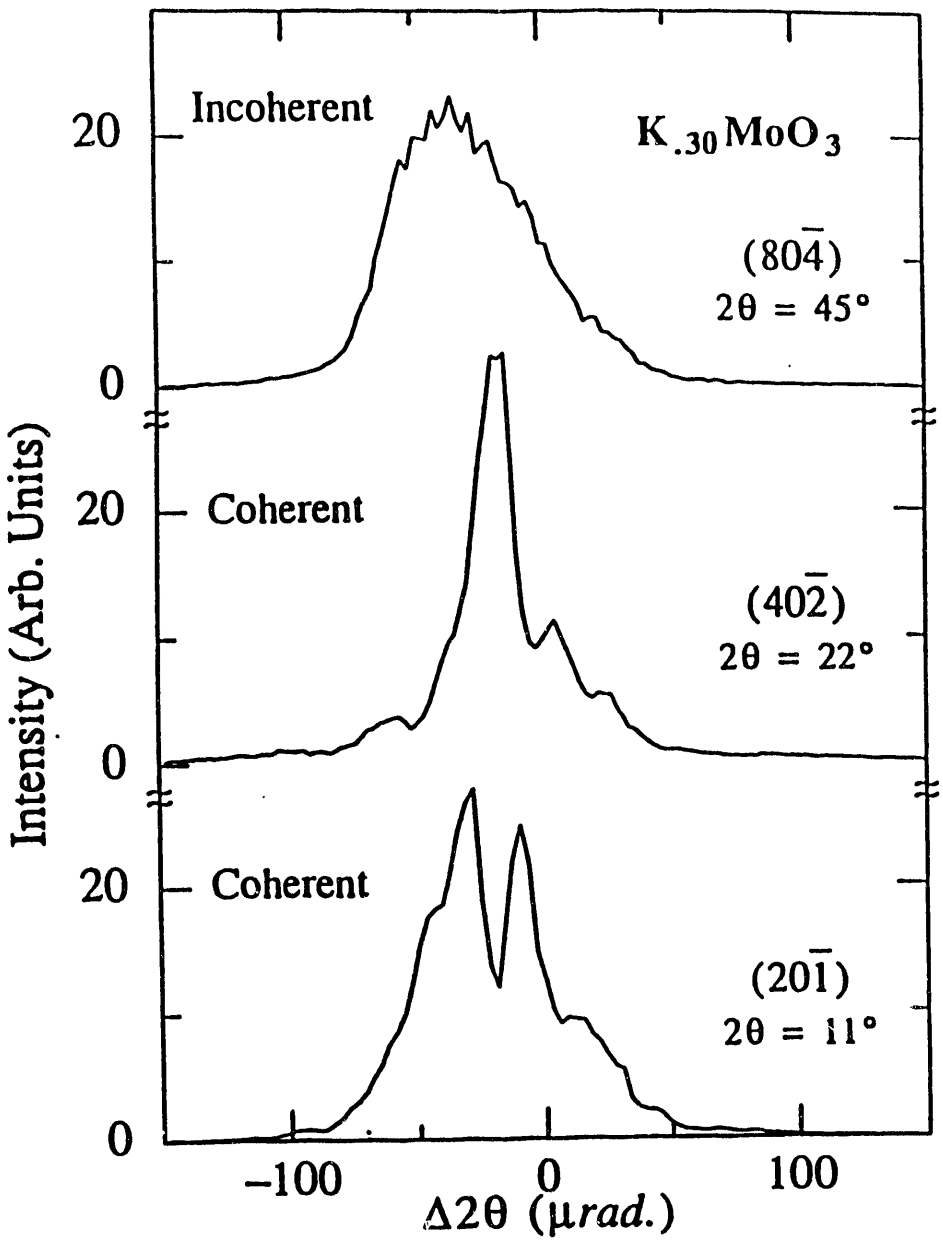
Reduction kinetics of surface PtO_x as measured by PtL_{III} absorption edge vs. time

Coherent X-Ray Diffraction



X-25 NSLS

Study sample domain dynamics by observing time dependent speckle structure.



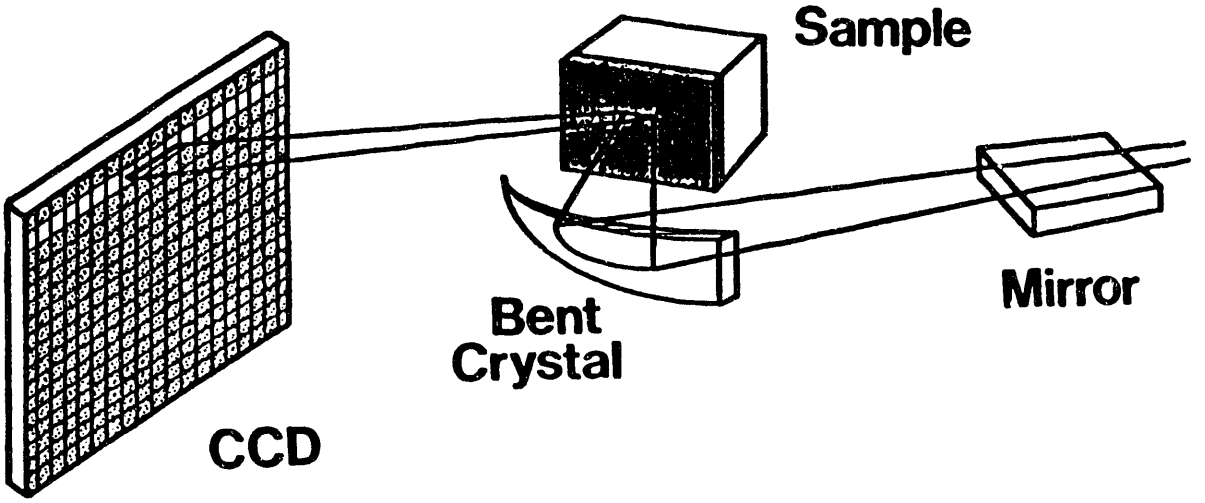
For coherent structure to be observable within Bragg peaks, path length difference introduced by sample must be less than the x-ray beam's longitudinal coherence length. This condition is met in the lower two curves and not met in the upper curve.

(Dierker, Fleming, Pindak, and Robinson, unpublished results)

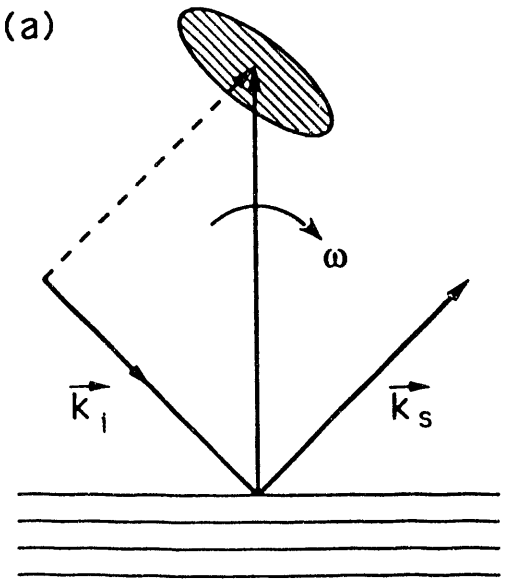
TIME-RESOLVED METHOD

X-16B NSLS

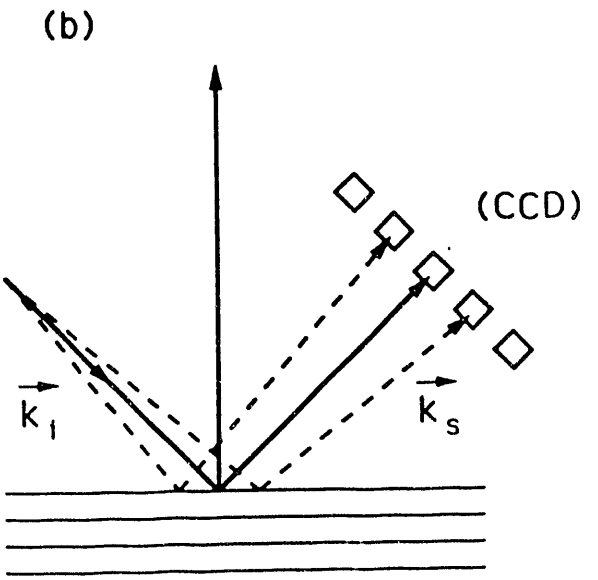
(AT&T BELL LABS SYNCHROTRON BEAMLINE)



STANDARD ROCKING CURVE



ANGULAR DISPERSED
ROCKING CURVE



R&D Program*

- Pixel array detectors
- Dispersive optics
- Fast time-resolved techniques

* in collaboration with APS

MICROCAT

A Micro Investigation of Composition Research Organization CAT

Dr. Gene Ice, Director

Metals and Ceramics Division
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Phone: 615/574-2744
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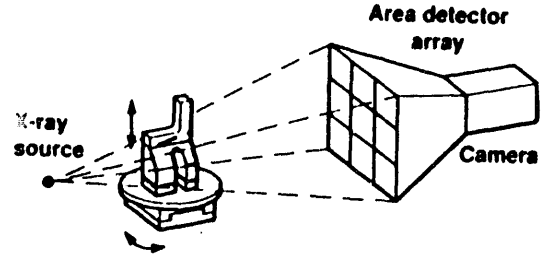
MICROCAT MISSION

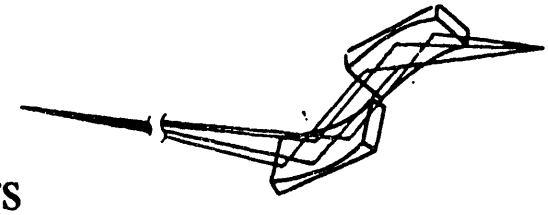
The MICROCAT mission is to provide a state-of-the-art x-ray microprobe. This microprobe will be used for fluorescent spectroscopy, microdiffraction, and micro-tomography.

MICROCAT combines the strengths of three national laboratories to develop a unique scientific instrument.

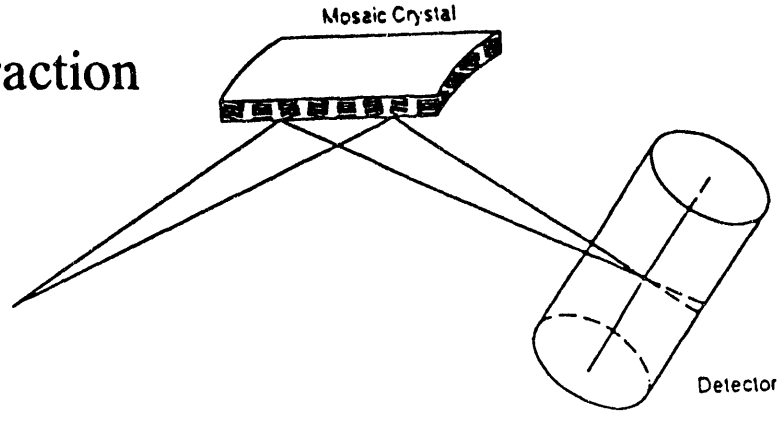
- LLNL
  - -tomography
  - -multilayer fabrication
  - -hard x-ray zone plates



- LBL
  - -Kirkpatrick Baez optics
  - -multilayer/mirror fabrication
  - -advanced solid state detectors



- ORNL
  - -X ray emission optics
  - -high energy x-ray microprobe optics
  - -microdiffraction



AN X-RAY MICROPROBE WILL BE UNIQUE

1. Nondestructive

   - Reduced heat and radiation damage by 10^{-3} to 10^{-5}
   - Intact samples

2. Minimum sample preparation/modification

   - Bulk samples-no thinning
   - Negligible charge collection-uncoated insulators

3. Advanced microanalysis

   - Lower detection limits by 10^{-3} to 10^{-4}; PPB; Fast
   - Strain resolution $\Delta d/d$ to 10^{-5}
   - Penetrate below sample surface
   - Improved spatial resolution in thick samples to 500 Å
   - No vacuum required (in vivo, air, water,gases, encapsulated).

MICROCAT SCIENTISTS

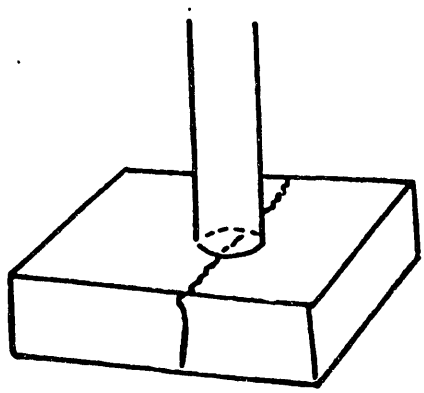
Charles Baes	ORNL
Peter Biltoft	LLNL
Richard Bionta	LLNL
John Budai	ORNL
Thomas Cahill	UC Davis
Charles Coutant	ORNL
Lut DeJounghe	UCB
Tom Gill	UC Davis
Camden Hubbard	ORNL
Gene Ice	ORNL
Bruce Jacobson	ORNL
Mike Kania	ORNL
John Kinney	LLNL
Satish Kulkarni	LLNL
Bruce Kusko	UC Davis
Donald Lesuer	LLNL
John McCarthy	ORNL
Samuel McLaughlin	ORNL
Monte Nichols	Sandia NL
Warren Oliver	ORNL
Dale Perry	UCB
Miroslav Marek	G Tech
Harry Martz	LLNL
Barry Rabin	INEL
Richard Ryon	LLNL
Joachim Schneibel	ORNL
Lee Schugart	ORNL
James Shackelford	UC Davis
Ken Skulina	LLNL
Cullie Sparks	ORNL
Eliot Specht	ORNL
Robert Turner	LLNL
John Whitson	ORNL
Joe Wong	LLNL
Fred Walker	UT Knoxville
Al Thompson	LBL

LBL	-Lawrence Berkeley Laboratory
LLNL	-Lawrence Livermore National Laboratory
ORNL	-Oak Ridge National Laboratory
UCB	-University of California Berkeley
UC Davis	-University of California Davis
G Tech	-Georgia Institute of Technology
INEL	-Idaho National Engineering Laboratory

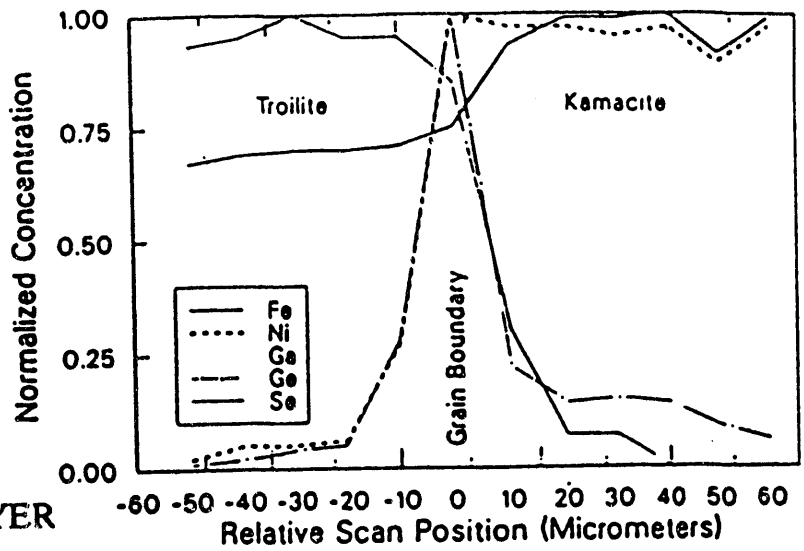
MICROFLUORESCENCE

ELEMENTAL DISTRIBUTION CRUCIAL TO BEHAVIOUR OF MATERIALS

INTERFACES



X RAY MDL ~ 5×10^{-5} MONOLAYER
AES (CLEAVED) MDL ~ 10^{-2}.

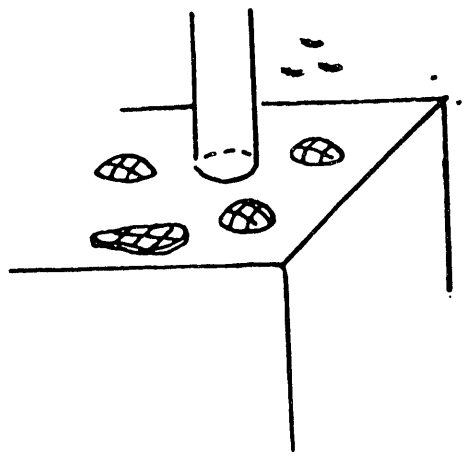


Segregation at a grain boundary
Adapted from M. Rivers, S. Sutton, and B. Gordon, Mater. Res. Soc. Symp. Proc. 143 (1986) 739

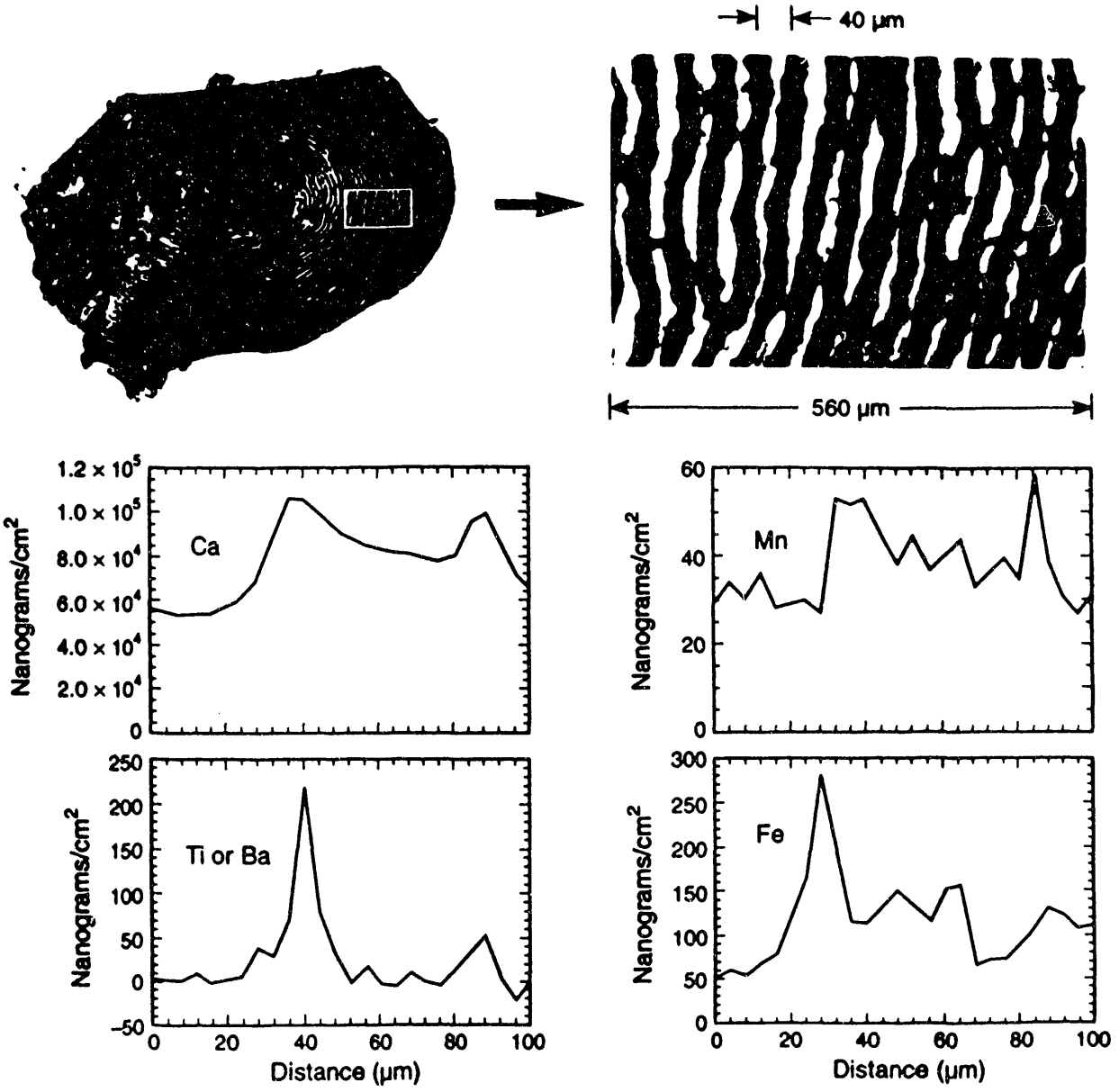
SURFACE IMPURITIES/SECOND PHASES

MDL WITH μm^2 PROBE

- 2.7×10^{-4} Monolayer
- 5×10^{3} Atoms
- 40 Å- diam Particle



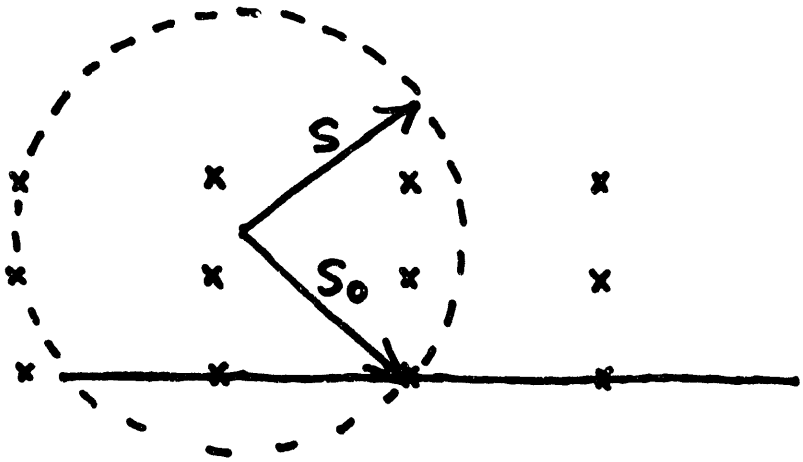
Fluorescent microprobe measurement of elemental distribution in a salmon fish scale. This measurement was made using a Kirkpatrick-Baez multilayer focusing system and undulator radiation from CHESS. [Thompson et al., *Nucl. Inst. & Meth.* **A319** (1992) 320-325]



MICRODIFFRACTION

X-RAY DIFFRACTION IS THE PREMIER TOOL FOR MEASURING ATOMIC STRUCTURE.

- Wavelength well matched to atomic spacing-distinct reflections.



- Samples many planes

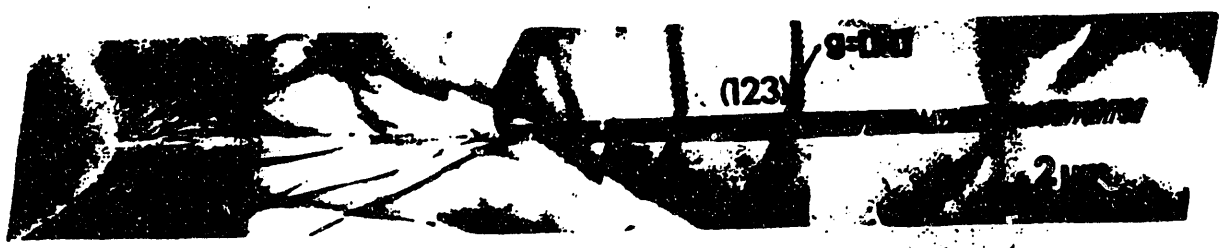
→ Phase identification

→ Strain mapping $\Delta a/a \sim 10^{-7}$-10^{-8}

- Microdiffraction is brilliance limited-APS crucial to achieve submicron resolutions.

UNDERSTANDING CRACK PROPAGATION IS FUNDAMENTAL TO UNDERSTANDING MATERIALS

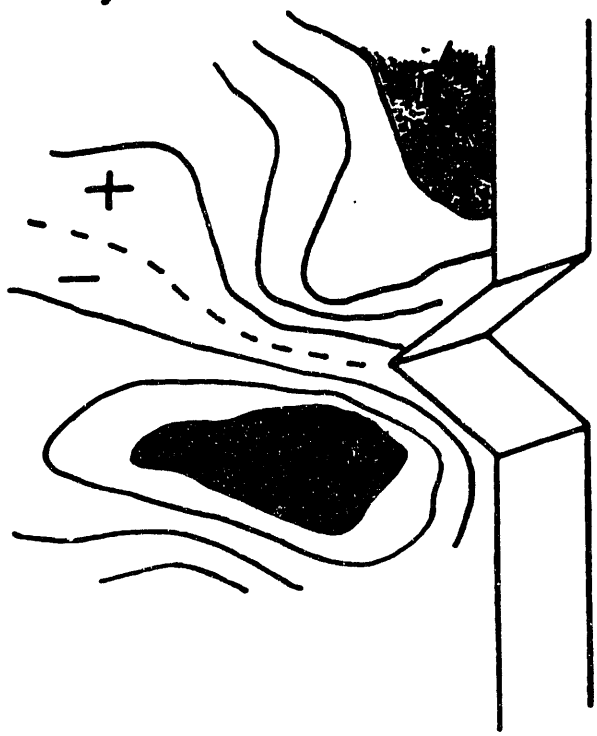
- Strain field near the crack tip crucial to crack growth.
- Existing imaging techniques require thin samples (buckle to relieve stress.)



COPPER

- X-ray microprobe allows study of local strain

1. Thick samples
2. High sensitivity
   - $\Delta d/d$ 10^{-5} x rays
   - $\Delta d/d$ 10^{-2} 100 keV e^-
3. Quantitative

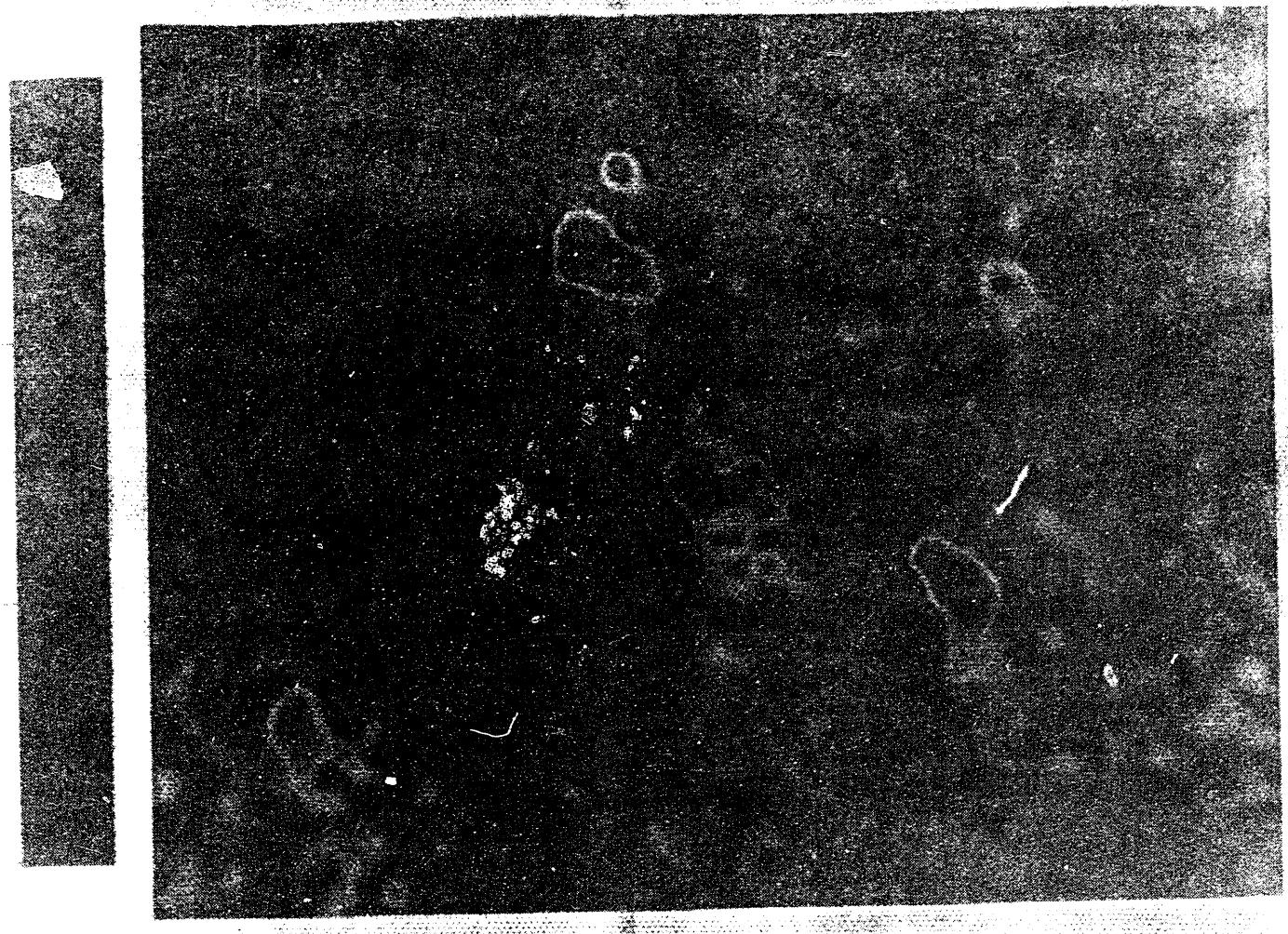


MICROTOMOGRAPHY

X-ray microtomographic reconstruction of an advanced fuel particle taken on the Exxon beamline at the NSLS. The spatial resolution is about 5 μm **(Dunsmuir et al., unpublished)**

Note: An actual particle is shown for scale→

Surface Interaction of a Chromium Salt with Galena, PbS



10 µm

5 µm 2s 240 × 300 µm 10 keV 6 × 7 µm

MRCAT

Materials Research CAT

Dr. Bruce Bunker, Director

Physics Department
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Notre Dame, IN 46556

Phone: 219/239-7219
Fax: 219/239-5952
E-mail: bruce.a.bunker@nd.edu

M R C A T

Materials Research Collaborative Access Team

Four Universities, one major corporation with common research and technical interests

- Principal Members:
  - Bruce Bunker / Notre Dame / Physics
  - Pulak Dutta / Northwestern / Physics
  - John Faber / Amoco Corporation
  - Timothy Morrison / Illinois Institute of Technology / Physics
  - Stephen Nagler / University of Florida / Physics

Formation of MRCAT motivated by

- current "web" of interactions (collaborations involving two or more institutions)
- common facilities interests
- Expertise: extremely useful to study single physical system with several different techniques:

  e.g. study dynamics of ordering in binary alloy using WAXS, SAXS, and XAFS

➢ CAT has specialists in a number of techniques; collaboration on same facility greatly reduces potential barriers for interaction.

As many as 20 other Principal Investigators at each institution will also participate in fund-raising, construction, and utilization

Current Collaborative Research

- Northwestern-Florida collaboration on physisorbed films
- Catalysis research involving Amoco, IIT, Notre Dame
- Joint Northwestern-Amoco studies on Langmuir- Blodgett films
- Research on polymers involving Amoco, IIT, and Northwestern
- Joint Amoco-Notre Dame research on ordering in thin epitaxial films
- Current collaboration on beamlines at NSLS: X11-A and X11-B (IIT, Amoco, and Notre Dame), X6 (IIT and Amoco)

evolving programs:

- Molecular-level structure of liquids in confined geometries (Northwestern, Notre Dame)
- Dynamics of structural phase changes (Florida, Amoco, IIT, Northwestern, Notre Dame)

Proposed Sector

- **Insertion-device beamline for brilliance-limited experiments**
  - tapered undulator A
  - inclined crystal monochromator
  - Two modes of operation:
    - "straight-through" (no mirrors) operation for high-*q* resolution
    - K-B mirrors (and possible zone plates or capillaries) for high spatial resolution
  - Optional multiple-bounce monochromator in hutch for high-*q*-resolution studiesExperimental Capabilities:
    - single-crystal diffraction and scattering
      - time-resolved
      - spatially-resolved
      - DAFS
    - high-resolution powder diffraction
    - high-resolution small-angle scattering
    - surface diffraction

- quick-scan XAFS
- high-magnetic-field capability

- **Bending-magnet line for flux-driven experiments**
  - Two modes for monochromator:
    - two-crystal fixed-exit (for < 1°/sec motion)
    - fixed-exit channel-cut (for > 1°/sec motion) (for quick-scan spectroscopy)
  - sagittal focusing using bent crystal
  - meridianal focusing using elliptically-bent mirror
  - Also:
    - Polychromator for msec-resolution dispersive XAFS
    - Focusing mirror for Laue: cylindrically cut for sagittal focusing, elliptically bent for meridianal focusing

---

- **Future (Phase II): Wiggler on ID line for high-energy diffraction, resonant Raman, other high-energy and / or high-flux experiments**
  - Requires different monochromator and changes in other beamline optics because of beam size

Scientific Program

- kinetics of phase transformations and critical behavior in 2-d and 3-d systems (high-*q* time-resolved scattering and spectroscopy, IFS)
- structure and function in *in-situ* catalytic systems
- static and dynamic structure of surfaces and interfaces
  - dynamics of structure during crystal growth
  - surface and interface reconstruction
- polymers: structure and ordering kinetics; single-fiber studies
- magnetic ordering in 3-d and 2-d systems
- structure of mesoscale particles and domains
- molecular ordering in liquids, particularly near interfaces and impurities
- other projects as driven by constituents in member institutions

μCAT

Midwest Universities CAT

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MIDWEST UNIVERSITIES COLLABORATIVE ACCESS TEAM

μCAT

Iowa State University		
	David W. Lynch	Physics and Astronomy and Ames Lab
	Alan I. Goldman	Physics and Astronomy and Ames Lab
	Michael Tringides	Physics and Astronomy and Ames Lab
	Costa Stassis	Physics and Astronomy and Ames Lab
	H. F. Franzen	Chemistry and Ames Lab
	Clifford G. Olson	Ames Lab
	David Vaknin	Ames Lab
	Douglas Robinson	MRC
Georgia Tech		
	Edward Conrad	Physics
Univ. of Missouri - Columbia		
	Haskell Taub	Physics
	Paul Miceli	Physics
	Michael Greenlief	Chemistry
University of Nebraska		
	Robert de Angelis	Materials Science
Kent State University		
	Satyendra Kumar	Physics
University of Wisconsin - Madison		
	Michael Winokur	Physics
USNY - Stony Brook		
	Peter Stephens	Physics
Washington University - St. Louis		
	Kenneth Kelton	Physics
	Patrick Gibbons	Physics
	Thomas Bernatowitz	Planetary Sciences

μCAT - MIDWEST UNIVERSITIES CAT

BEAMLINES FOR MATERIALS SCIENCE RESEARCH

One undulator line, 3 stations

Two bend magnet lines, one station each

Room for future expansion

Undulator line features:

- 5-20 keV from fundamental of type A undulator
- 1:1 imaging of source "point" onto sample
- Variable polarization via phase plate(s)

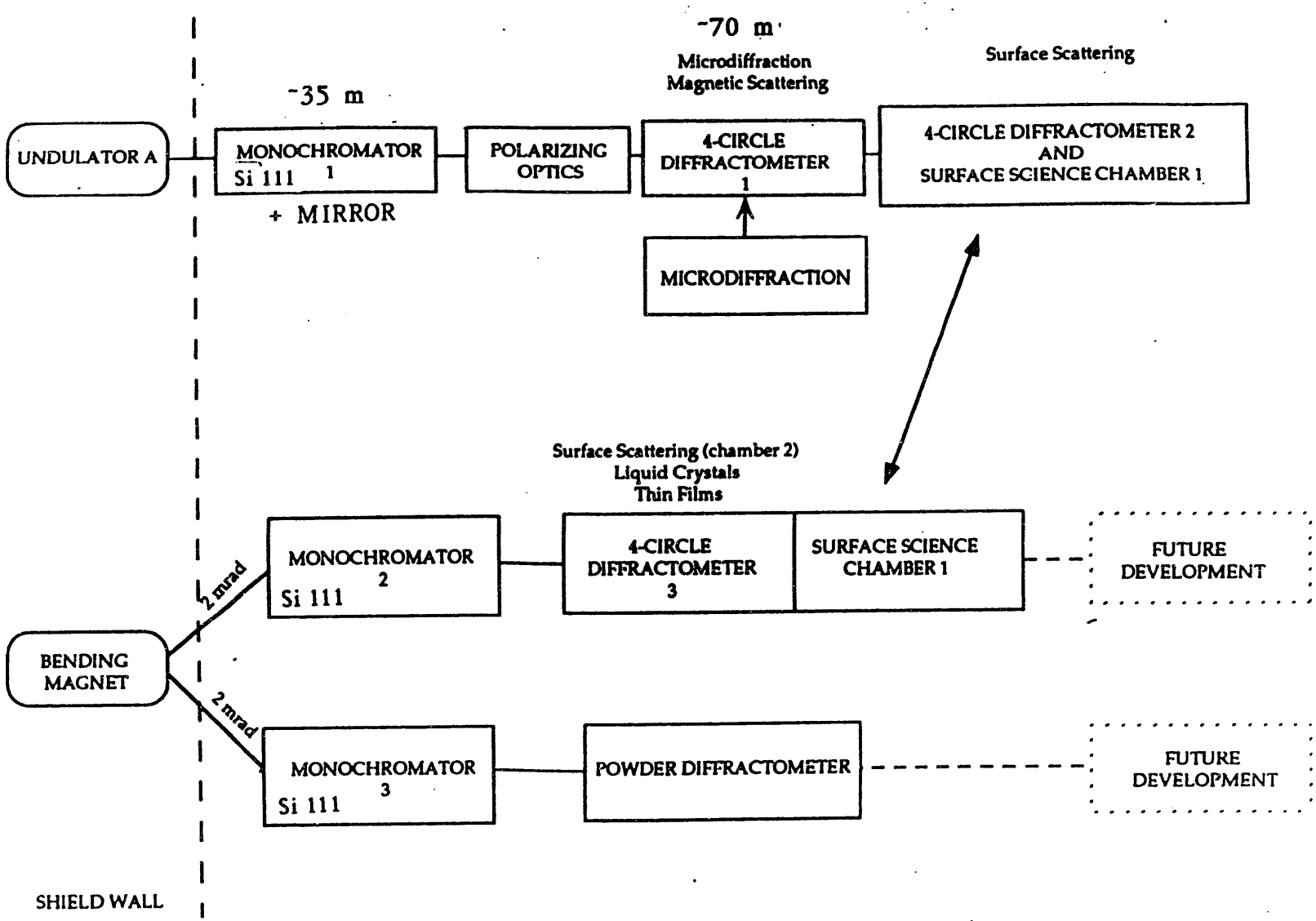


Figure 1: Block diagram of the proposed μCAT Sector

ESTIMATED EFFECT OF FOCUSSING

no correction for optical system efficiencies

N = number per sec of 8 keV photons in 0.02% b.w. on a 1 μm^2 sample

NSLS: 2.5 GeV, 500 mA, sample at 20 m
CHESS: 5.5 GeV, 80 mA, sample at 15 m
APS: 7 GeV, 100 mA, sample at 70 m

SOURCE	BRIGHTNESS*	N
NSLS b.m.	5.7×10^{13}	1.4×10^{5}
CHESS b.m.	6.2×10^{13}	2.8×10^{5}
APS und. A	1.2×10^{17}	2.4×10^{7}
APS und. A, 1:1 focussing	1.2×10^{17}	1.0×10^{9}

"geometrical" gain of ca. 40X from focussing

---

* in photons/sec/$mrad^2$/0.02% b.w.

RESEARCH PROGRAM

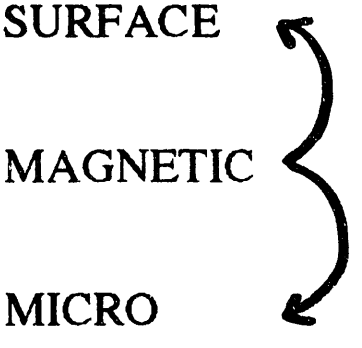
Surface (including liquid) and interface scattering -
structure, dynamics, kinetics

Magnetic scattering and magnetic circular dichroism
including magnetic surface scattering

Microdiffraction
quasicrystals, metastable phases, extraterrestrial
particles

Liquid crystals and membranes

Powder diffraction
rapid data rates, high temperatures



MICRODIFFRACTION

scattering power = $(F_{ooo}/V_u)^2 V_c \lambda^3$ *

$\cong 10^{16} - 10^{17}$ for routine structural measurements

Eisenberger et al. (1984) 800μm^3 of zeolite, SSRL wiggler	3×10^{14}
Bachman et al. (1985) 200 μm^3 of CaF_2, Hasylab	1.3×10^{14}
Rieck et al. (1988) 2.2 μm^3 of CaF_2, CHESS	7.1×10^{12}
Skelton et al. (1991) 0.4 μm^3 of Bi, X-17, NSLS	4.4×10^{10}

APS undulator A, 1:1 focussing, scaling from Eisenberger et al., expect

$10^9 - 10^7$

---

* after Skelton et al., Science 253, 1123 (1991)

MAGNETIC SCATTERING

Consider a $1\mu m^3$ grain of MnF_2, with $\mu = 5.95\ \mu_B$ below T_N of 67.6 K. With 1:1 focussing from undulator A, expect about 100 counts/sec from 100 magnetic peak without using any resonance.

Since count rate varies as $V_{sample} \times \mu^2$ can scale to

1. Structures with ordered moments of $10^{-2}\ \mu_B$ in samples
   100 μm x 100 μm x 10 μm

2. A micron-sized single grain of the moments are of the order of 1 μ_B

EXAMPLES OF MAGNETIC SCATTERING PROBLEMS

1. LnX_3 Ln = lanthanide, X = In, Sn Cu_3Au structure

Many phase transitions at low T, structural and magnetic. Studied by specific heat, transport and magnetic measurements. Small crystals, neutron absorption. Need x-ray scattering to find crystal and magnetic structures.

2. Gd surface orders magnetically 15K above bulk T_c. Use magnetic surface scattering to measure M(z).

EXAMPLES OF SURFACE SCATTERING PROBLEMS

1. 2-d kinetics. domain size in non equilibrium systems grows as $L=A(T)t^{\eta}$, η is universal (1/3, 1/2). Universality leads to $S(q,t) = S_{max}(t)F(q/w(t))$. Tested by LEED (O on W) and x-ray scattering (Pb on Ni). Use x-rays on lower-Z adsorbates, cover larger range of L than LEED can.

2. In-situ growth studies during, e.g., organo-metallic vapor-phase epitaxy, ion-assisted deposition

3. Metastable phase of Eu on W (110) - crystal and magnetic structure. From LEED, structure changes with thickness between 0 and 50 Å. For thicknesses above about 100 Å Eu reverts to normal b.c.c. phase, but still epitaxial.

PNC CAT

Pacific Northwest Consortium CAT

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Conceptual Design

Three beamlines, one insertion device line (ID) and two bending magnet lines (CX and XA), will be implemented. The ID line will be used for radiation in the 4 to 20 keV range from a hybrid wiggler or high field undulator, similar to the APS Wiggler B but with a reduced deflection factor. A highly focused monochromatic beam will be employed in a scanning x-ray microscope. The CX line is for molecular structure analysis, and the XA line is for x-ray absorption spectroscopy.

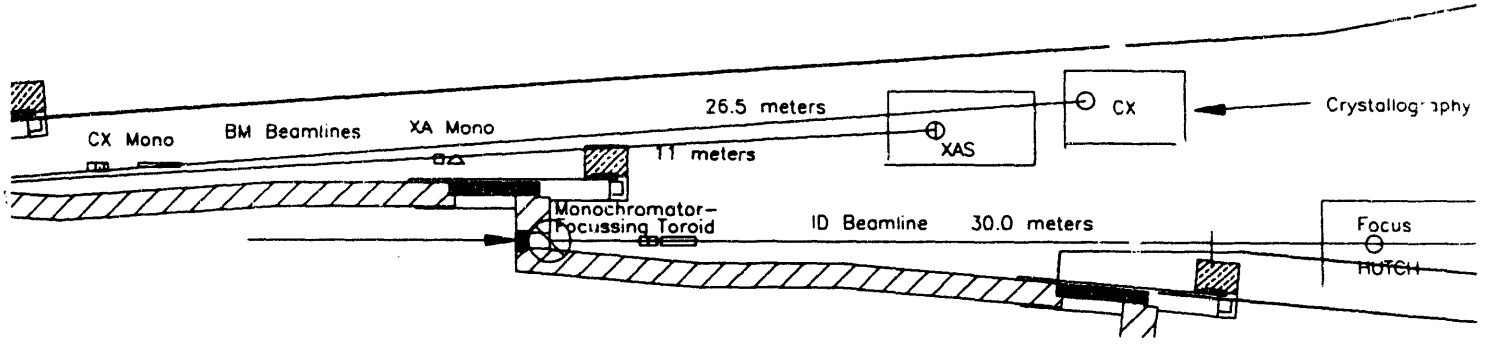


Fig. 1

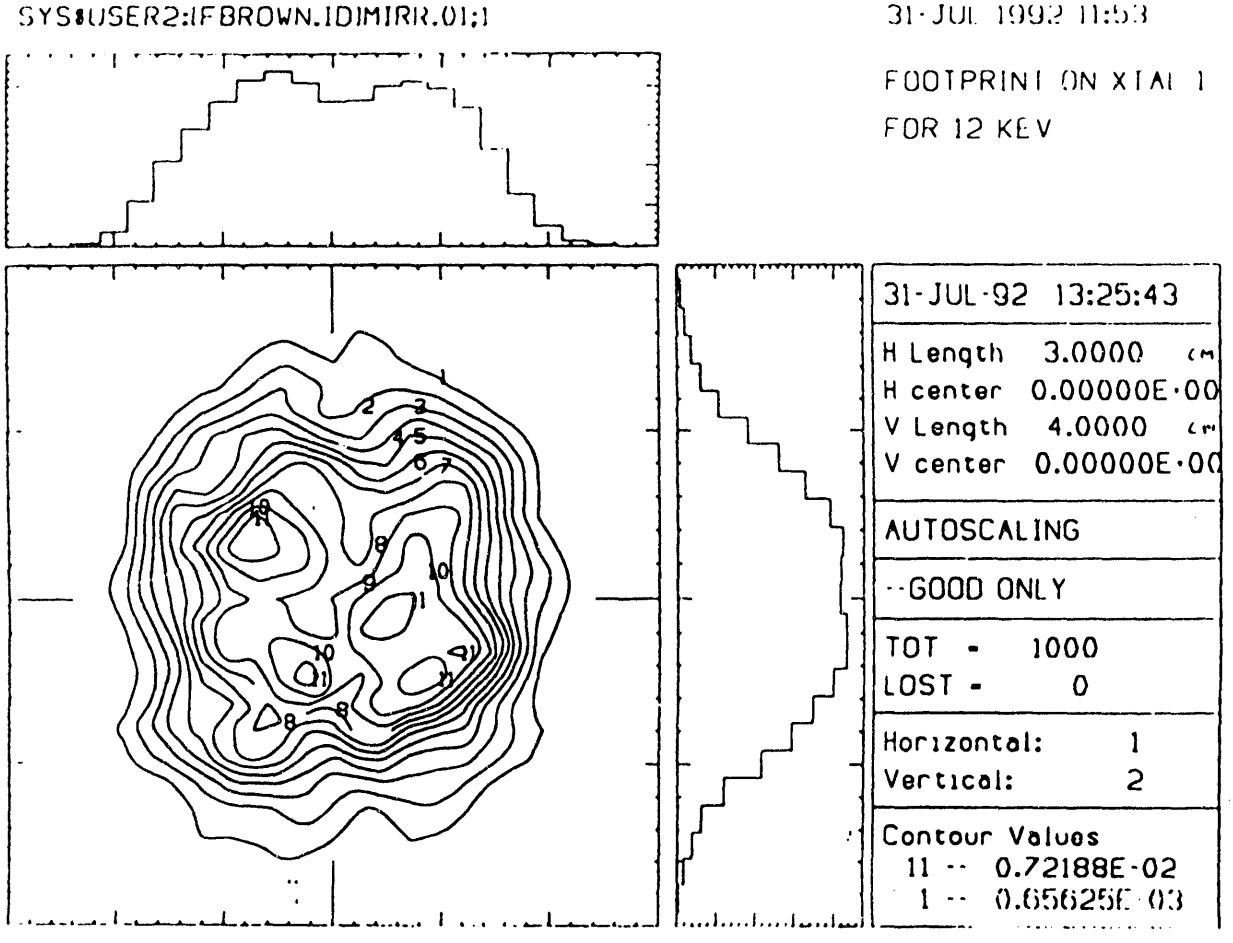
The Scientific Program General Comments

The central focus of the PNC CAT will be the investigation of environmental problems. In addition, research will be carried out on fundamental studies of materials and condensed matter physics, as well as macromolecular structures and their function.

The primary thrust in environmental sciences will be to extend our understanding of the structure and surface chemistry of natural materials such as soils and sediments. In addition, advanced characterization and monitoring of solid wastes such as fly ash will be carried out.

Insertion Device Beamline

The fixed exit beam from the APS high heat load monochromator will be focused by a long toroidal mirror at 6 mrad grazing angle onto the inlet (0.5 mm dia.) of a 2 meter long tapered capillary in the experimental hutch. The focus within this hutch is located 30 meters from the pole of the mirror for 1:1 imaging of the center of the wiggler source with minimum convergence. The footprint of 1000 rays traced by Shadow (realistic ID source) on the first crystal of the monochromator at 12 keV is shown in Fig. 2. The focus 30 meters downstream is shown in Fig. 3. The outlet of the capillary is 1 micron or less in diameter. The capillary is the focusing element of a unique scanning x-ray microprobe with resolution down to 0.1 micron. With the microprobe, spatially resolved diffraction, XAFS, microtomography, and DAFS will be performed.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FOR 12 KEV
31-JUL-92 13:25:43
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H center 0.00000E+00
V Length 4.0000 cm
V center 0.00000E+00
AUTOSCALING
--GOOD ONLY
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LOST - 0
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Vertical: 2
Contour Values
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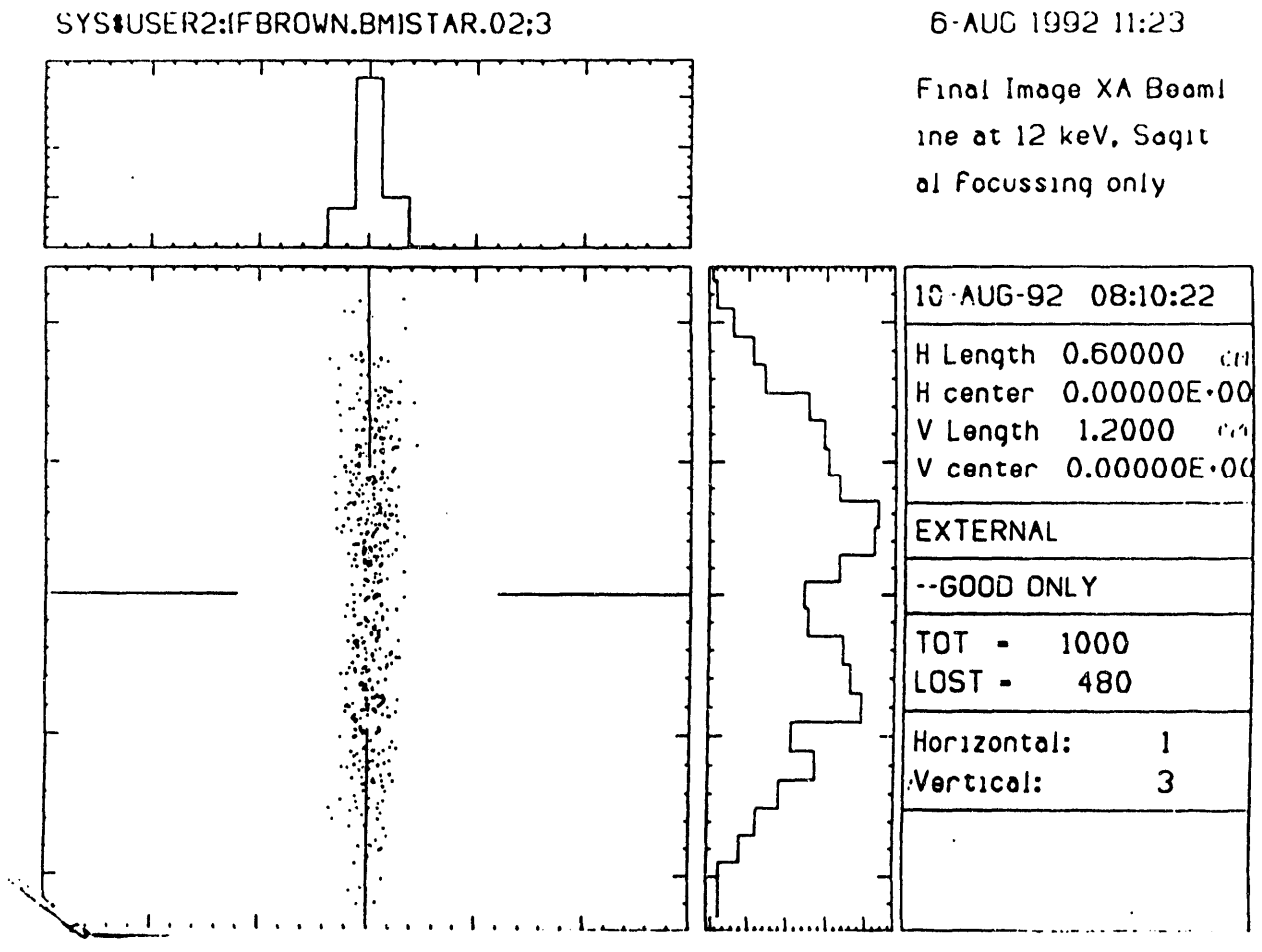
1 -- 0.656625E-03

Fig. 2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Final Image XA Beaml
ine at 12 keV, Sagit
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10-AUG-92 08:10:22
H Length 0.60000
H center 0.00000E+00
V Length 1.2000
V center 0.00000E+00
EXTERNAL
--GOOD ONLY
TOT - 1000
LOST - 480
Horizontal: 1
Vertical: 3

Fig. 3

Some Proposed Experiments

The synchrotron-based techniques for environmental problems include x-ray diffraction and scattering, x-ray absorption fine structure on dilute and small samples, x-ray microtomography, and the x-ray fluorescence microprobe.

Experiments in materials science and condensed matter physics include the study of surfaces and interfaces, time-resolved melting and solidification, high pressure XAFS studies, and photoelectron diffraction on advanced materials.

Crystallography Beamline

A plane double crystal monochromator is used for fixed exit beam in the 4 to 45 keV range. Resolution is 1 in 10^4 and provision is made for rapid scan to fixed address. The monochromator is followed by a long toroidal mirror at 6 mrad grazing angle. This line intercepts about 2.5 mrad horizontal of the bending magnet flux. A footprint on the first crystal at 12 keV is shown in Fig. 2. A star plot (Fig. 3) indicates the focus 26.5 meters downstream.

Macromolecular Structural Analysis of Biological Materials

The relation between structure and function will be pursued. Applications include:

1. The emerging technology of employing biological systems for remediation of toxic waste sites and the degradation of specific contaminants.

2. Characterization of molecular mechanisms involved in adverse effects of contaminants on humans and other organisms.

Measurement Techniques

High brilliance, tunability, or broad band req.

1. Time-resolved XAFS and diffraction including von Laue diffraction

2. Spatially resolved XAFS
   Fluorescence microprobe
   Tomography below and above x-ray edges
   Diffraction

3. Diffraction anomalous fine structure DAFS
   Site and atom specific

4. XAFS of surfaces and interfaces
   Diffraction (anomalous)

5. Photoelectron diffraction PED, especially high-Z atoms

6. X-ray Raman scattering

EXAFS and Anomalous Diffraction Beamline

The XA beamline employs a double crystal monochromator with sagittal focusing, a 4 to 45 keV range with 1 part in 10^4 resolution, and slow scan capability with rapid scan to fixed address. The monochromator is 33 meters from orbit, and the hutch is 11 meters downstream for 3:1 sagittal focusing. A triangular second crystal with adjustable bending force would be used. Meridial focusing in the vertical could be included close to the hutch. A star plot of 500 rays at the final focus with sagittal focusing is shown in Fig. 3.

List of CAT Members

Univ. of Wash.	Pacific NW Lab. Richland	Canadian Universities
F. C. Brown	J. E. Amonette	Daryl Crozier
Wim Hol	D. R. Baer	T. Tiedje
R. Ingalls	M. L. Knotek	L. T. J. Delbaere
Y. Ma	S. V. Mattigod	M. James
E. Merrit	R. Stults	
M. Olmstead		
J. J. Rehr		
L. Sorensen		Wash. State Univ.
R. Stenkamp		
E. A. Stern		Brad Pate
K. Voss		

PNC CAT Insertion Device Beamline

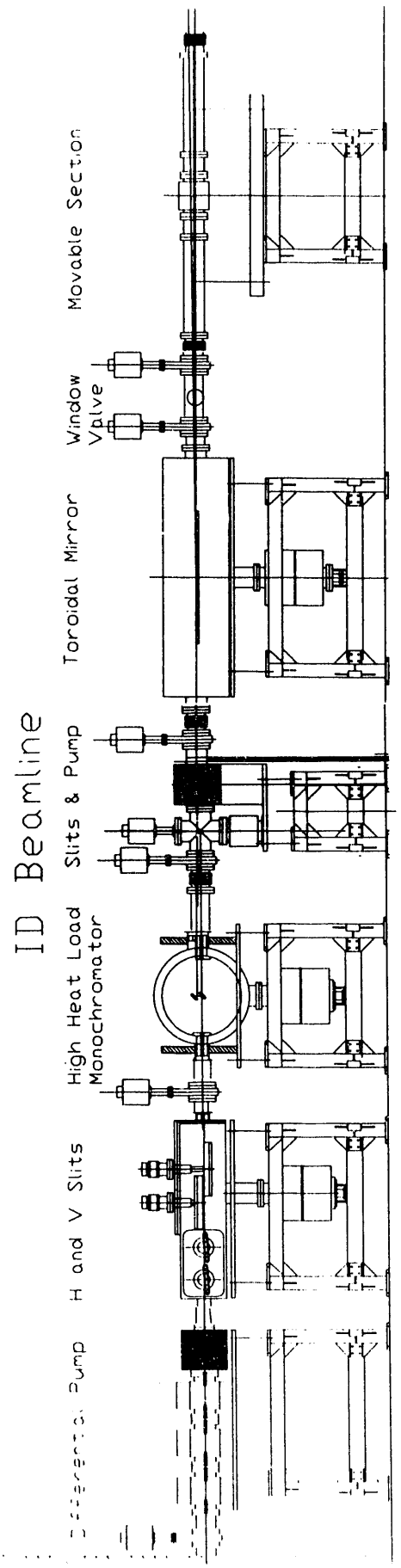
Modular Components 10/4/92

The drawing shows the first components on the user's side of the shielding wall for the PNC CAT Insertion Device Beamline. This line is designed to monochromate and focus a wiggler(or high field undulator) beam onto the 0.5 mm diameter open end of a tapered capillary, 60 meters from the source point, for further reduction to about 1 micron in diameter.

The high heat load monochromator, designed by the APS, should cover the range 4 to 45 keV and have a resolution of the order of one part in 10^4. Operation in both fixed energy and slow scan mode is required. It should be capable of slow continuous scan over about 800 eV, and of rapid scan to a predetermined address.

A pumped region with adjustable slits and collimator separates the monochromator from a long toroidal mirror in ultrahigh vacuum. This mirror can be positioned for focusing the monochromated beam 30 meters downstream(position 1) or it can be slowly moved out of the beam to allow for a straight through beam(position 2). Position 1 should be highly stable and accurately reproducible.

In mirror position 2 the straight-through beam is transmitted into the ambient by a thin window in a closed "window valve". A movable section of closed off beamline can be swung slightly to the side for access to this region for experiments up to 45 keV without the limitations of the total external reflection cutoff of the toroidal mirror. A cross between two valves allows for pump-out connections and a vacuum gauge when re-establishing the deflected beam position 1. A second upstream hutch can be build around the beam in position 2, if required.


ID Beamline
Differential Pump
H and V Slits
High Heat Load Monochromator
Slits & Pump
Toroidal Mirror
Window Valve
Movable Section

SBC CAT

Structural Biology Center CAT

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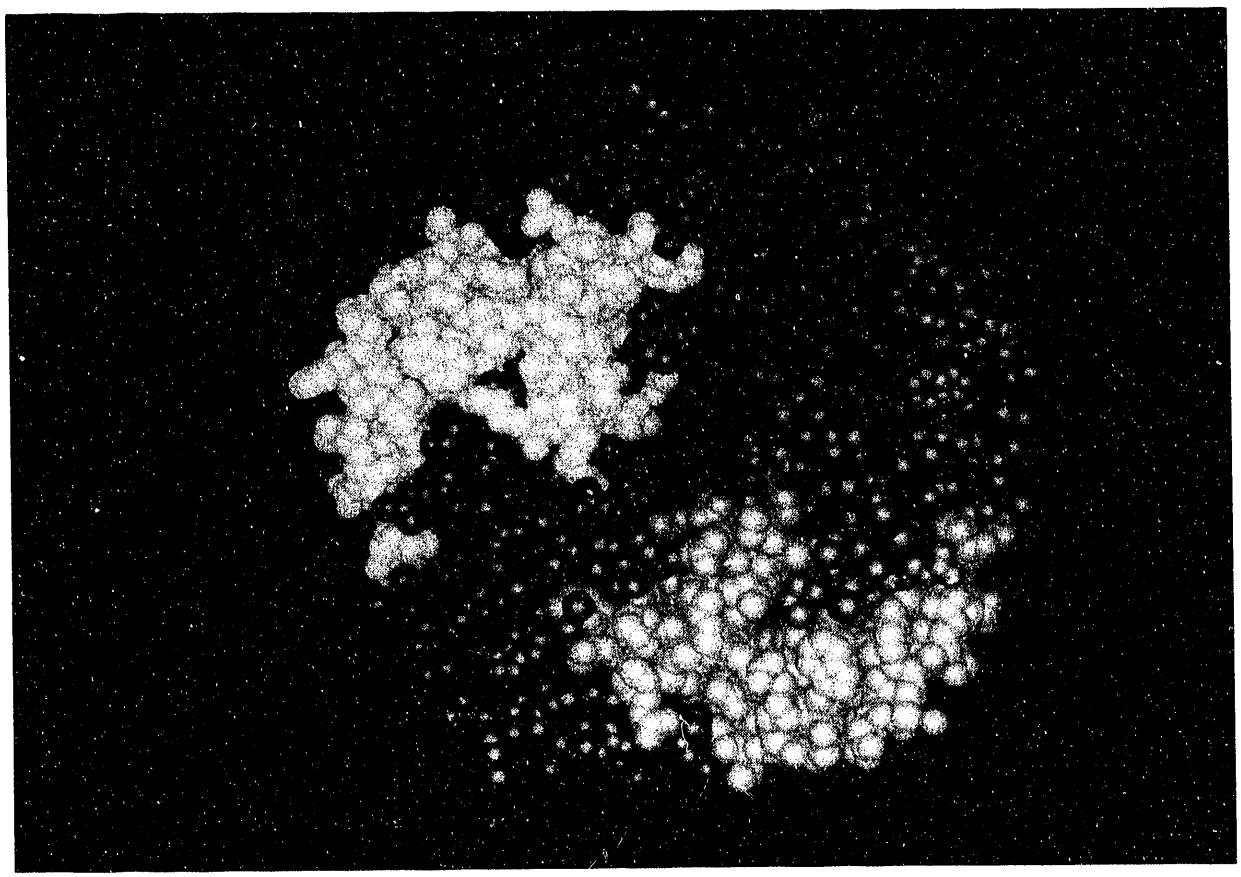
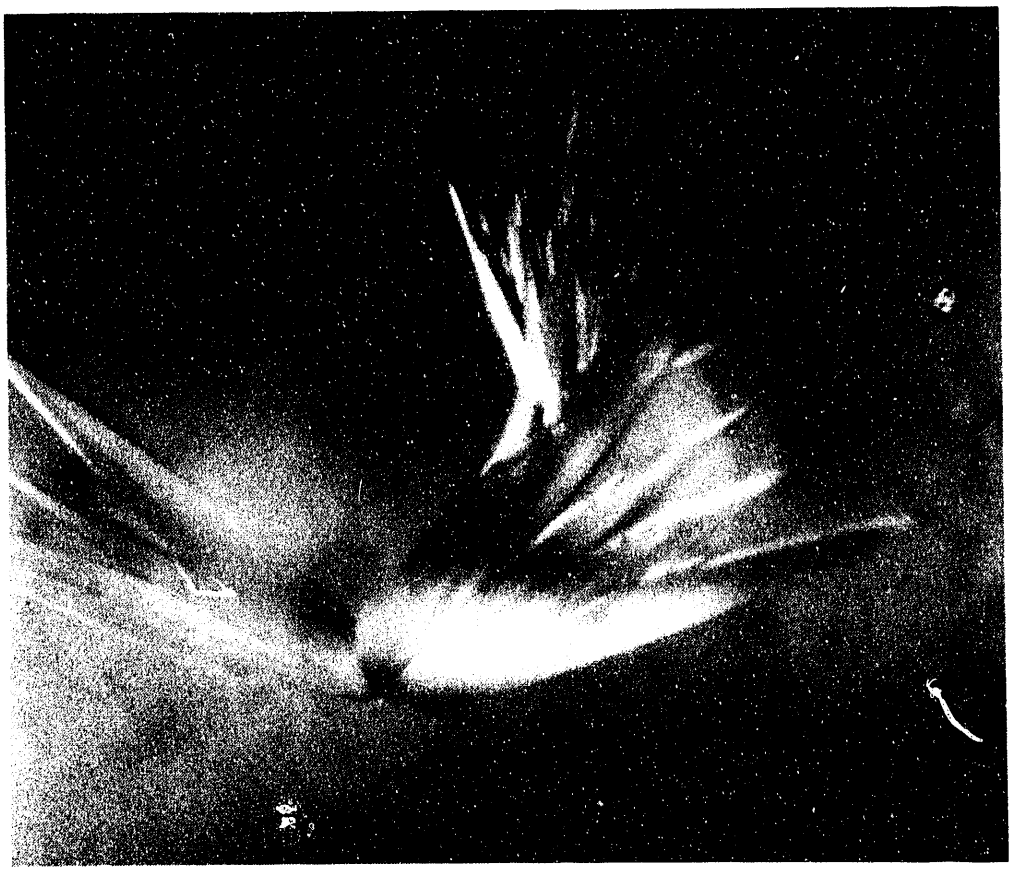
Protein Crystallography — A New Frontier in Biological Research

Research advances in molecular and cellular biology depend increasingly on knowledge of the exact structures of the large, complex biological molecules that carry out and influence critical life processes. Scientists are rapidly gaining insights into precisely how a biological molecule's structure relates to its function, and through medical science and biotechnology those insights can be used to prevent and treat disease. As a result, researchers have a growing need to identify the structures of "macromolecules" such as proteins, nucleic acids, carbohydrates. and their larger aggregates, which include viruses, enzyme complexes, and ribosomes.

Macromolecular crystallographers grow pure crystals of a single type of protein or other large molecule and place them in an x-ray beam. The x-ray patterns diffracted by these crystals are recorded by detectors and computers then analyze the patterns to identify the types and locations of atoms in the crystallized molecule. A single biological macromolecule may contain several thousand atoms.



Computer simulation of cholera toxin molecule



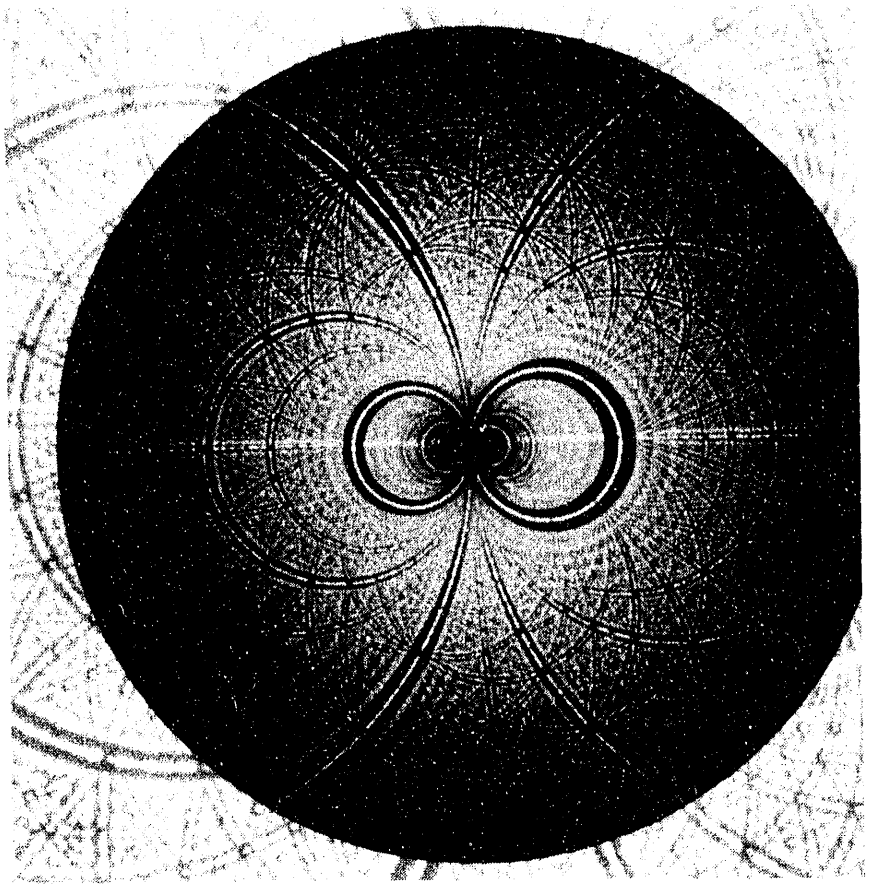
Crystals of an enzyme Δ^5-*3-ketosteroid isomerase*

The Structural Biology Center: Meeting a Need

The Structural Biology Center is a proposed national user facility for macromolecular crystallography at the APS.

The Center, expected to be in full operation in 1996, is being designed to meet protein crystallographer's need for customized, state-of-the-art experimental facilities. It will provide a setting for the next generation of structural biology research, by offering greatly increased speed and quality in recording diffraction data and enhanced control over critical x-ray characteristics. Users will have access to:

- *Synchrotron x-rays of unprecedented brilliance*, optimized for crystallographic research.
- *High-speed area detectors* designed to record diffraction data from APS x-rays.
- A *wide range of x-ray energies* suitable for many diffraction applications, including two recently developed ones: Laue diffraction, which holds promise for studies of the variation of crystal structures with time, and multi-wavelength anomalous diffraction (MAD) phasing, which allows optimized determinations of crystal structures.
- A *full complement of support facilities and staff* to expedite research.

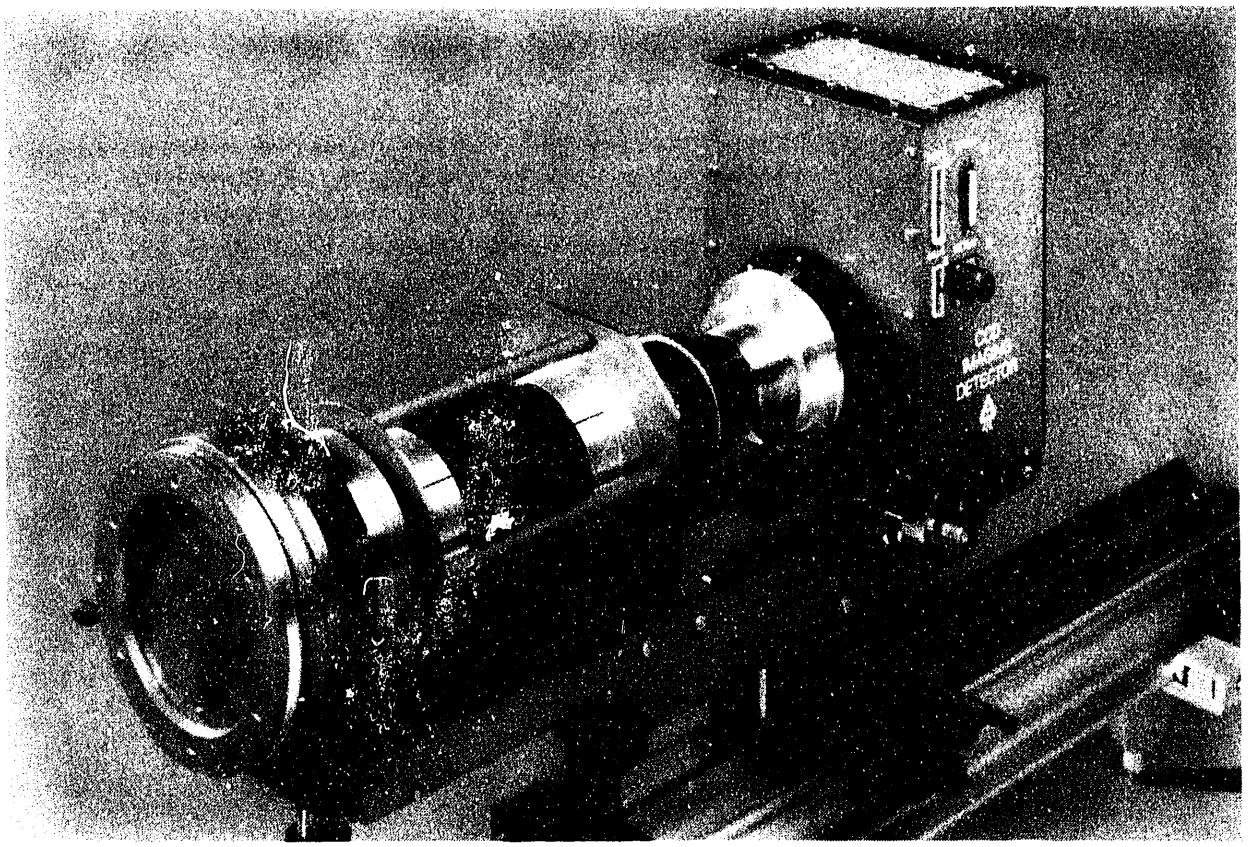


Computer model of Laue diffraction data from glycogen phosphorylase

Brighter X-rays and Faster Detectors

Protein crystallographers have found that as the brilliance of a synchrotron x-ray beam increases, so do the accuracy, completeness, resolution, and speed with which they can collect diffraction data from protein crystals. Since brighter x-rays also cause more rapid deterioration of protein crystals, x-ray exposure times must be kept to a minimum through the use of high performance detectors.

At the Advanced Photon Source now under construction at Argonne National Laboratory in Illinois, the Structural Biology Center will meet these needs for brighter x-rays and faster detectors.



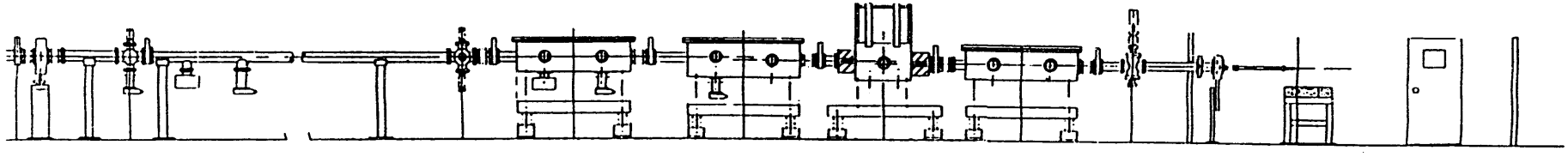
Argonne's high performance area detector for protein crystallography diffraction data from intense synchrotron x-rays.

Principal Users Will Guide the Center

The Department of Energy's Office of Health and Environmental Research has been asked to fund this new biological research facility at Argonne National Laboratory. The Center will be administered through Argonne's Office of Energy, Environmental, and Biological Research.

The scientific program, however, will be defined by advisory groups representing the interests and needs of the entire community of structural biologists that the facility is intended to serve. The Structural Biology Center will be governed by a principal user group responsible for establishing scientific and technical goals and overall performance review. It also will be guided by three other external review bodies: a scientific advisory board, a technical advisory committee to evaluate technological and design issues, and a proposal evaluation committee to ensure appropriate peer review for proposed research.

Elevation layout of APS beamline designed for protein crystallography



SRI CAT

Synchrotron Radiation Instrumentation CAT

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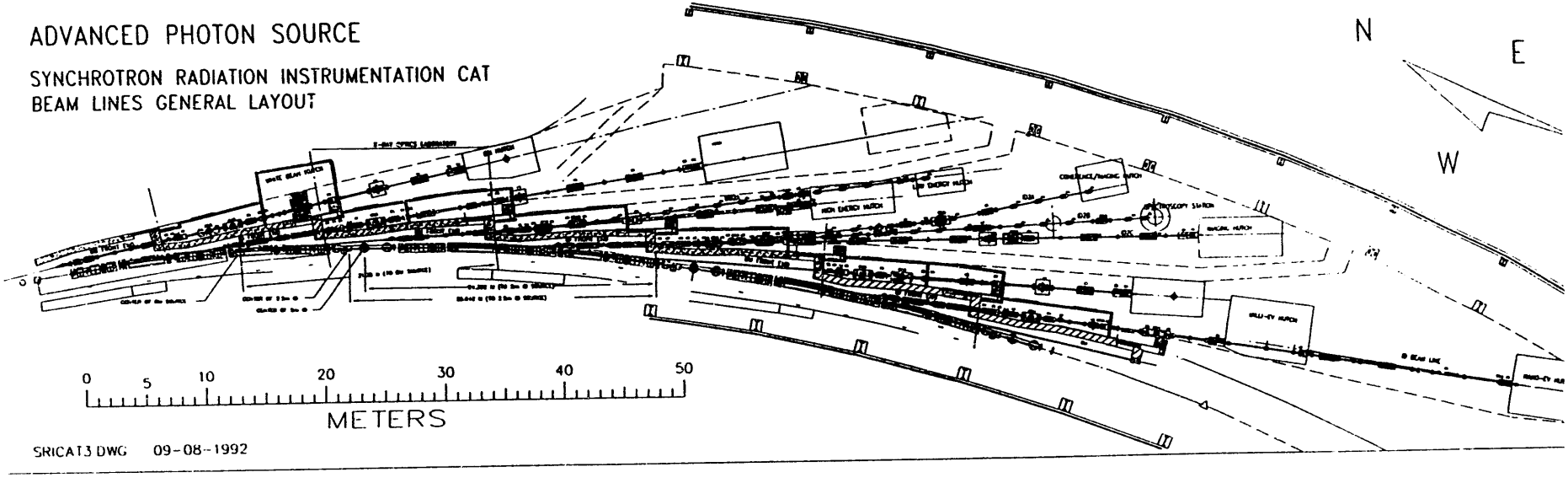
ADVANCED PHOTON SOURCE
SYNCHROTRON RADIATION INSTRUMENTATION CAT
BEAM LINES GENERAL LAYOUT
N
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40
50
METERS
SRICAT3.DWG 09-08-1992

Personnel

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Albert Macrander
Ian McNulty
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Kevin Randall
Brian Rodricks
C. Shawn Rogers
Stan Ruby
Roland Savoy
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Gopal Shenoy
Deming Shu
D. Peter Siddons
Ralph Simmons
Sunhil Sinha
Robert Smither
Cullie Sparks
George Srajer
Bruce Stockmeier
Joachim Störhe
Albert Thompson
Jonathon Tischler
James Tobin
James Trebes
Shenglan Xu
Wenbing Yun

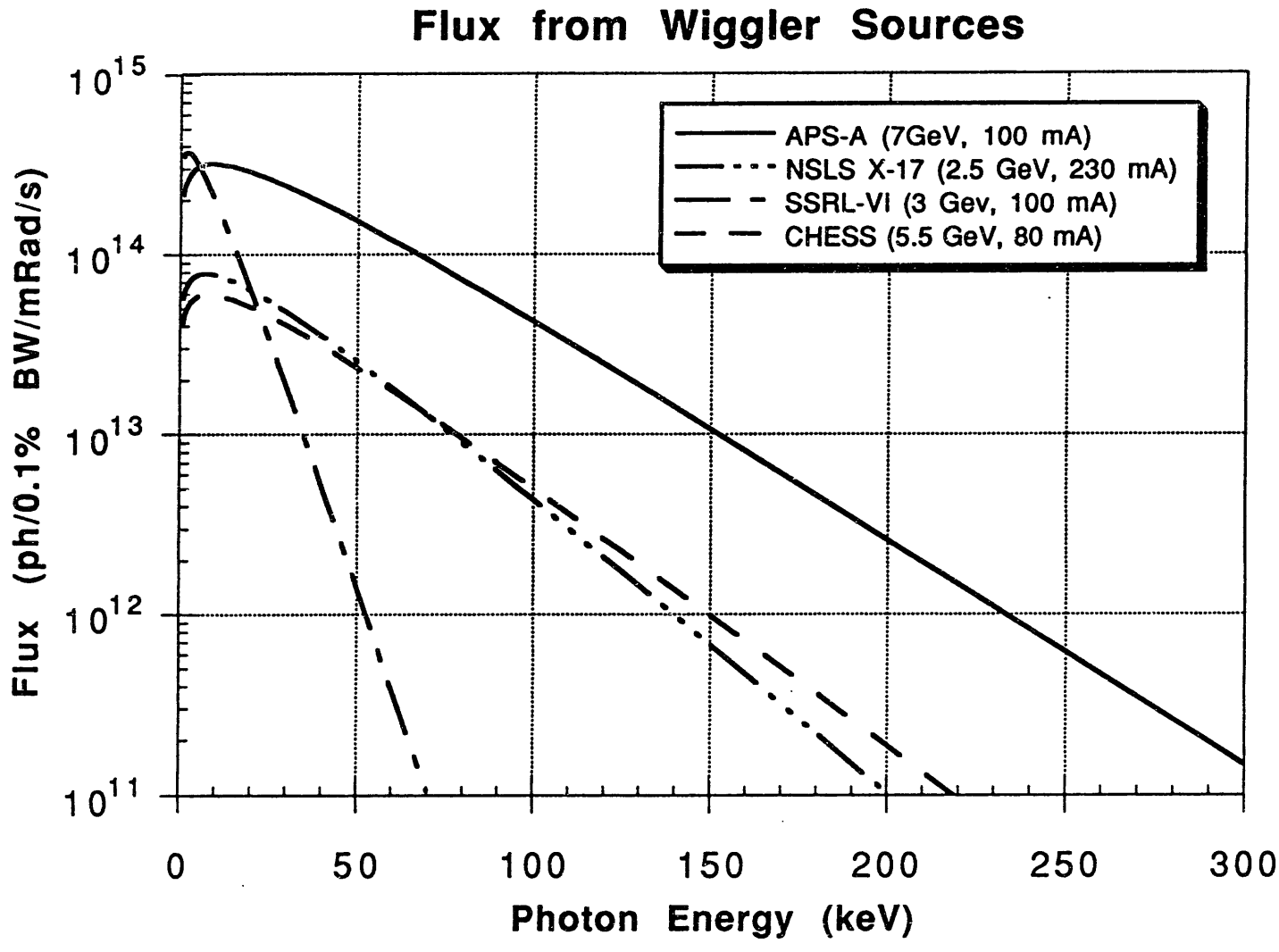
SRI CAT

Sector 1

HIGH ENERGY X-RAY SCATTERING

The APS Wiggler A has a critical energy of 32.6 keV and will be a superb source of photons from 50-200 keV. The flux from this source at 150 keV is approximately equal to the flux from current bending magnet sources at 10 keV. Hence, a program has been established to develop optical elements for x-rays in this largely neglected energy regime. Initially, efforts will center on optics for Compton scattering and triple-axis diffractometry. For high resolution Compton scattering and diffraction measurements monochromators and analyzers with perfect rystals will be employed. For other applications requiring maximal flux, but with relaxed energy resolution, crystals with a range of imperfection will be used. In addition, work will continue on the production of circularly polarized x-rays with phase plates and their use for magnetic scattering measurements.

ADVANCED PHOTON SOURCE

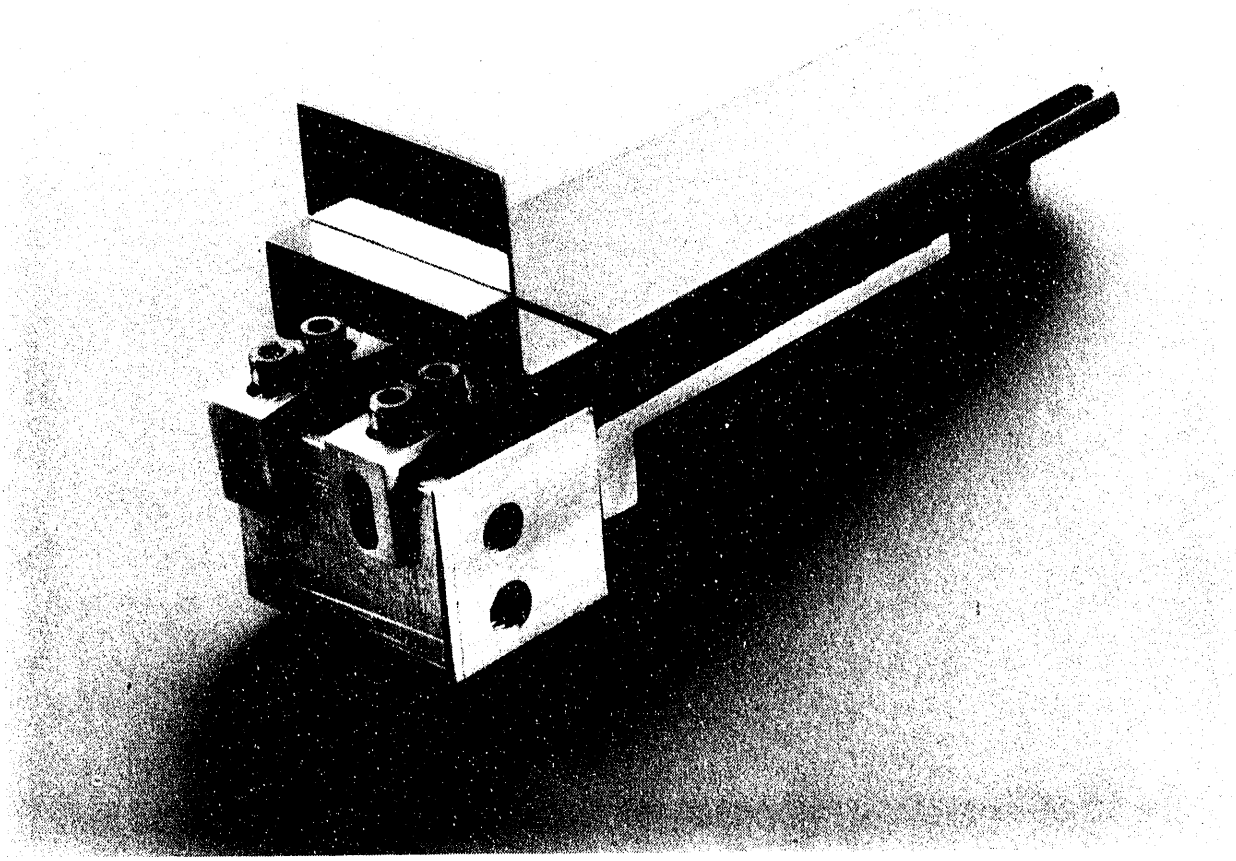


A Germanium X-Ray Phase Plate for the Production and Analysis of Circularly Polarized Radiation

Phase Plates are a novel optical tool whose purpose is to circularly polarize radiation in the x-ray regime. It does this by developing a 90° phase shift between the (σ and π) polarization states of the alpha branch and absorbing both (s and p) polarization states of the beta branch. Previous attempts by D.M. Mills and J.A. Golovchenko have used single crystal silicon in differing geometries to obtain a degree of circular polarization of approximately 75%. In an effort to improve upon this, we have fabricated a similar device using germanium instead of silicon. Not only does the germanium improve the quality of the circular polarization, but it also extends the range of energies over which the device can operate up to 94 Kev.

The performance of these two devices is compared in fig. 1 and fig. 2 below. The difference in the performance of these devices arises from the fact that silicon absorbs significantly less beta branch photons than does germanium. The beta branch photons are, by definition, 180° out of phase with the alpha branch photons and thus can only decrease $|Pc|$.

Some of our experimental results to date include the measurement of the single crystal Darwin curve of the Bragg plate. This was measured using the crystal in it's designed Bragg-Laue geometry with Mo Ka radiation. The results are shown in fig. 3 below.



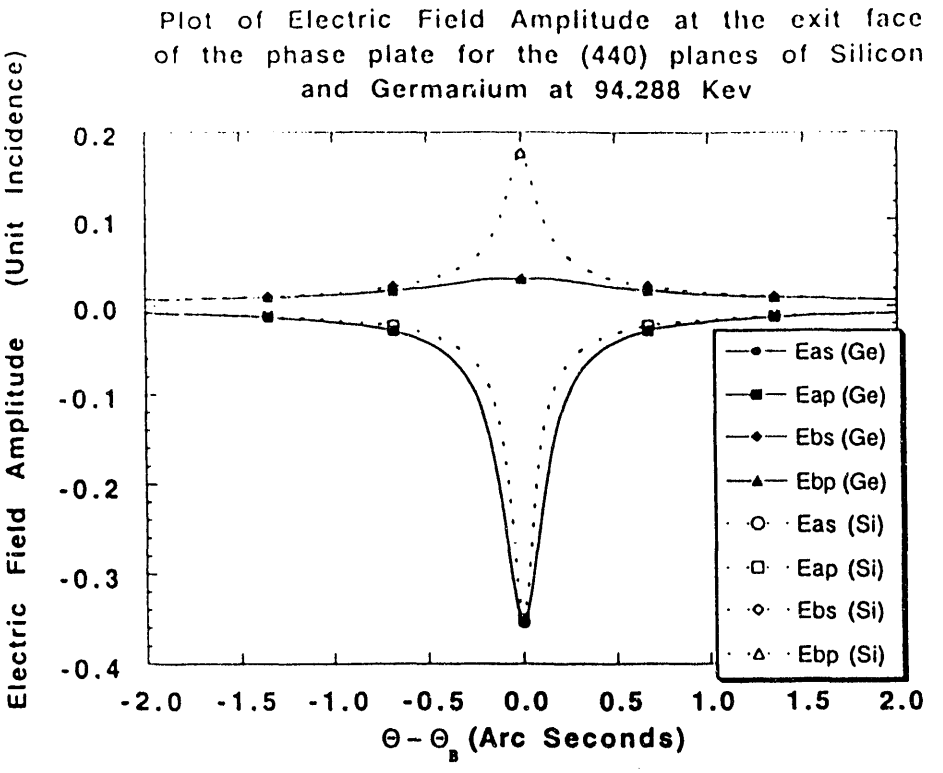


Figure 1

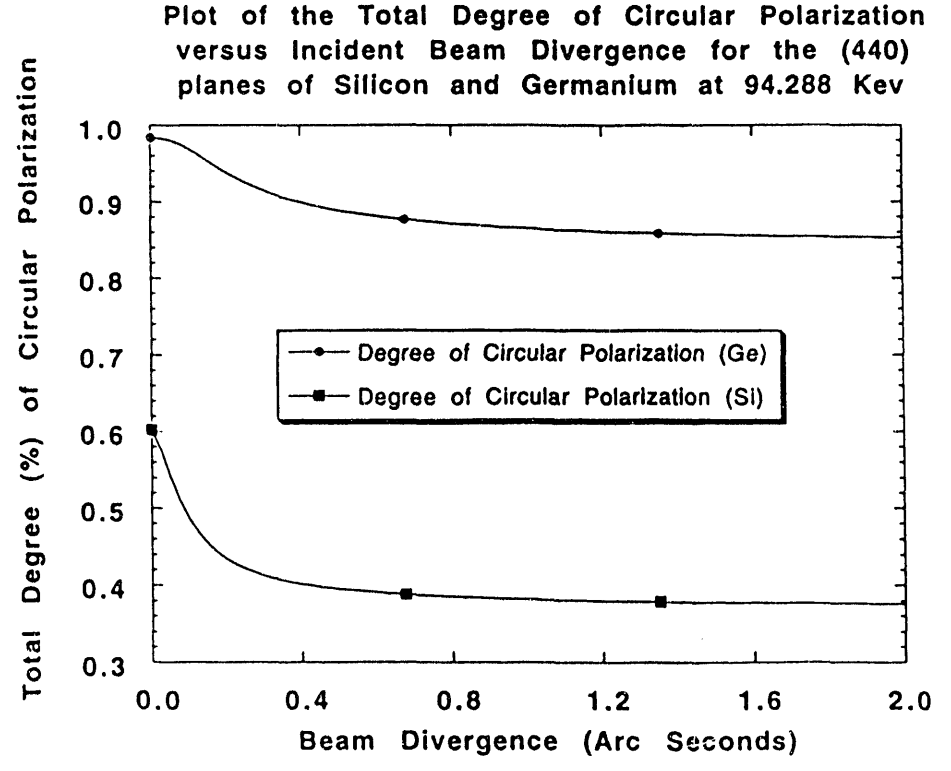


Figure 2

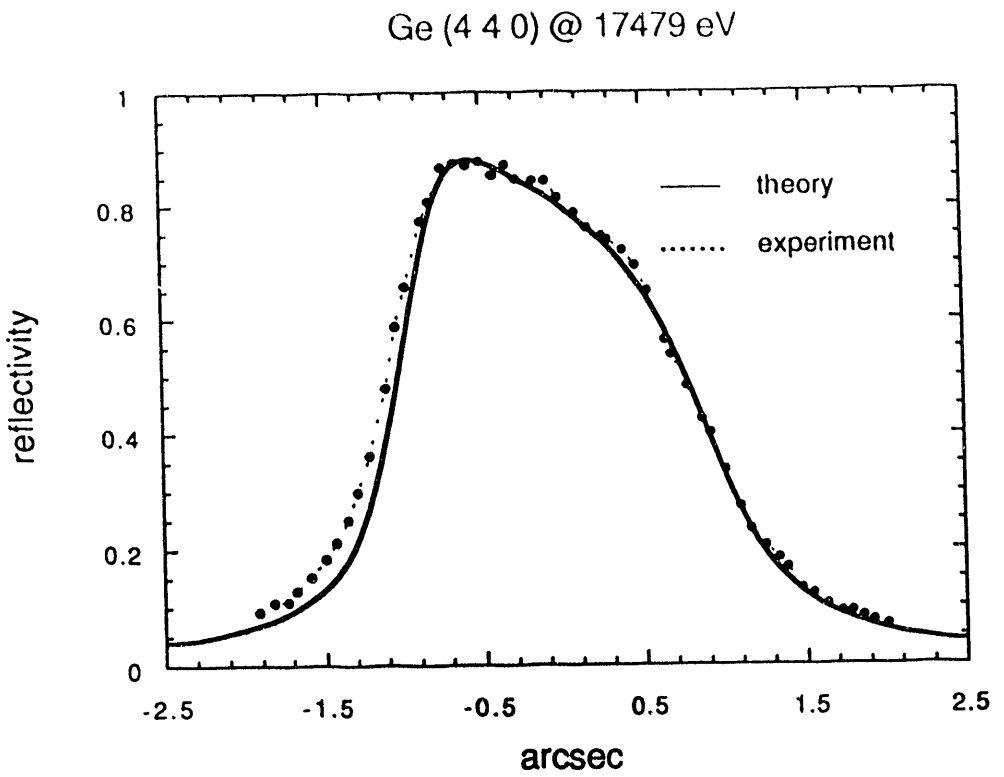
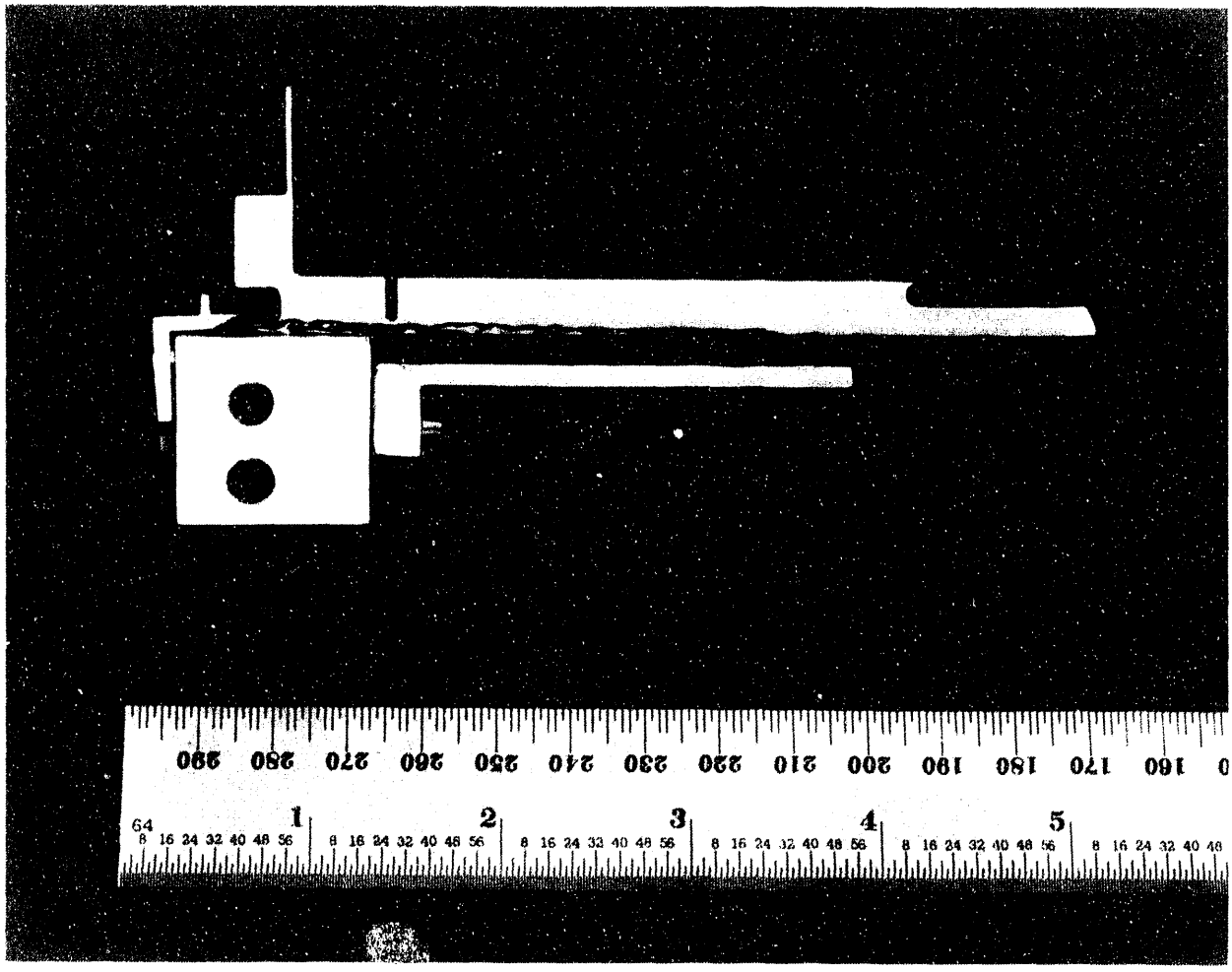


Figure 3 Plot of Reflectivity versus $\Theta - \Theta_B$ for Ge (440) at 17479 eV.



SRI CAT

Sector 2

Major Scientific Programs

- 0.5-4 keV instrumentation for high spectral resolution and high photon flux spectroscopy

- Microfocusing optics for the 0.5-30 keV x-ray energy range and microfocusing-based techniques.

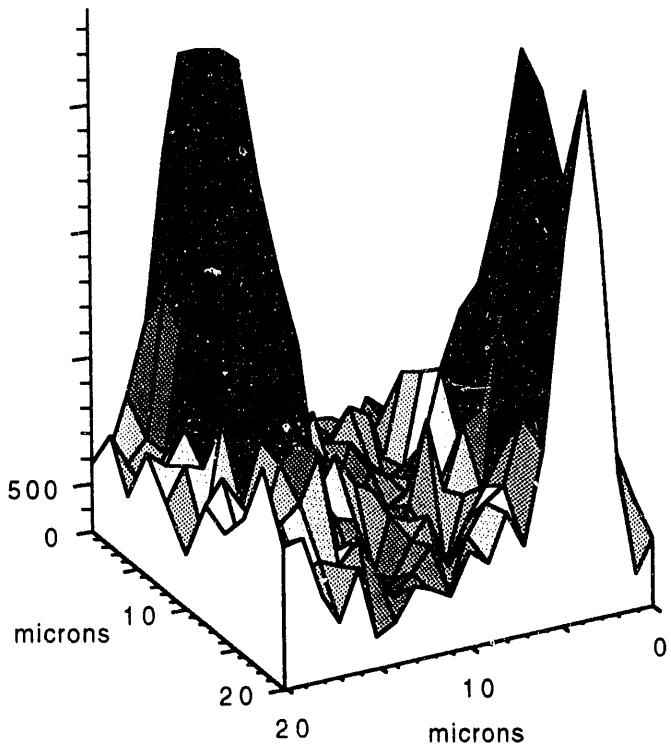
- Coherence-based interferometry and imaging techniques.

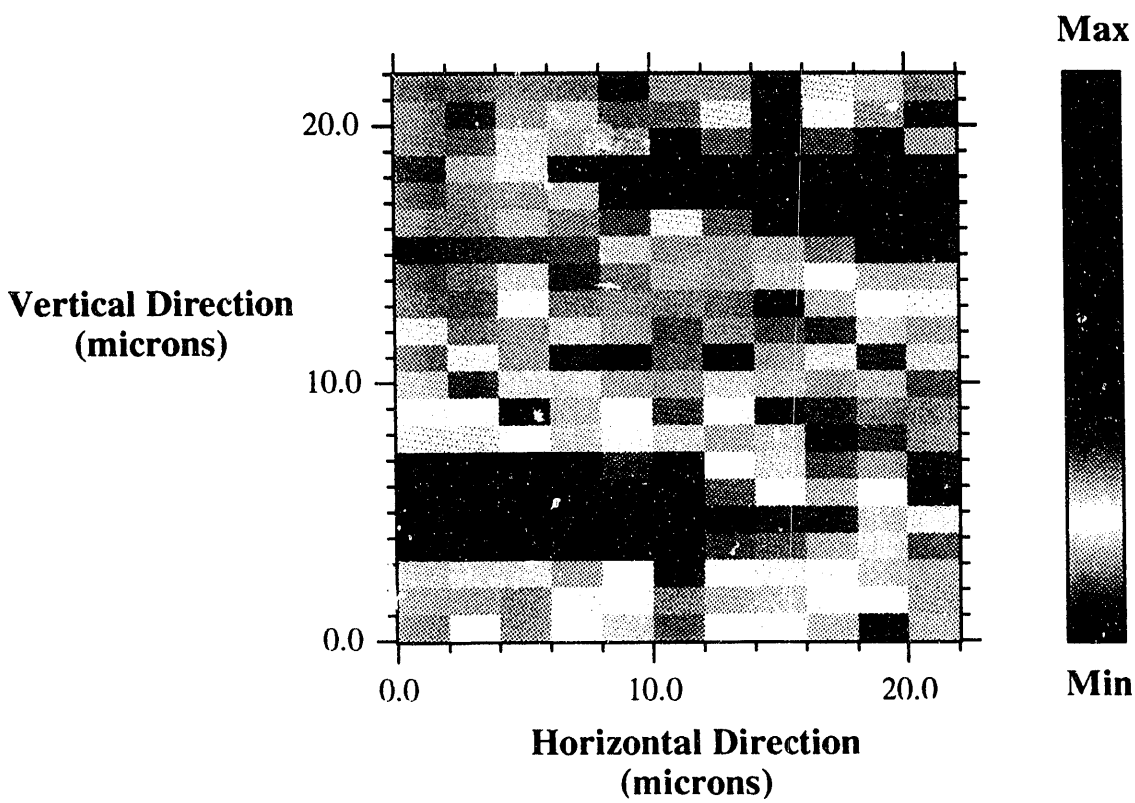
Spectral Specifications

	ID2-A	ID2-B	ID2-C
Spectral range (keV)	0.5-4	0.5-4	2.5-30
Resolving power	10^2-10^3	10^3 - 10^4	10^1-10^4
Beam size (FWHM)@ station (mm)	0.2 - 5	1	0.2 - 3
Flux /0.1% BW@ station	10^{13}	10^{13}	10^{14}-5 x10^{14}
Source coherent flux /0.1% BW	~10^{10}-10^{12}		~10^8-10^{11}

Microfocusing Related Techniques

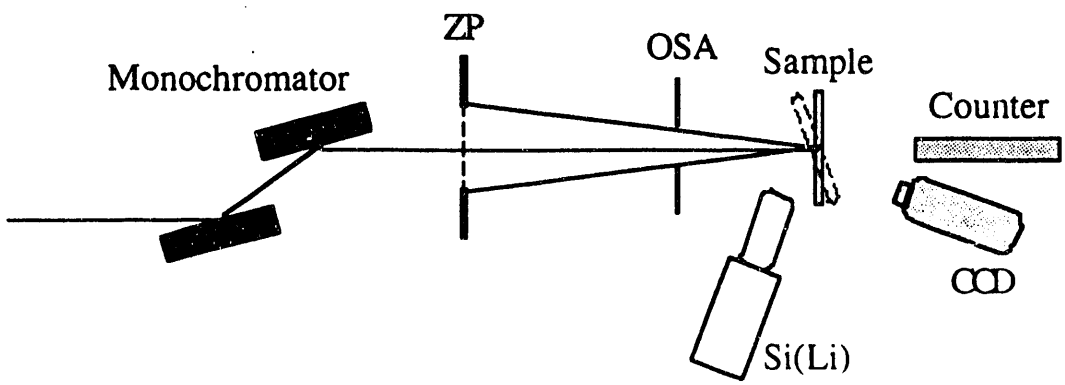
Microdiffraction
from 1μm x 1μm AgBr Crystallites





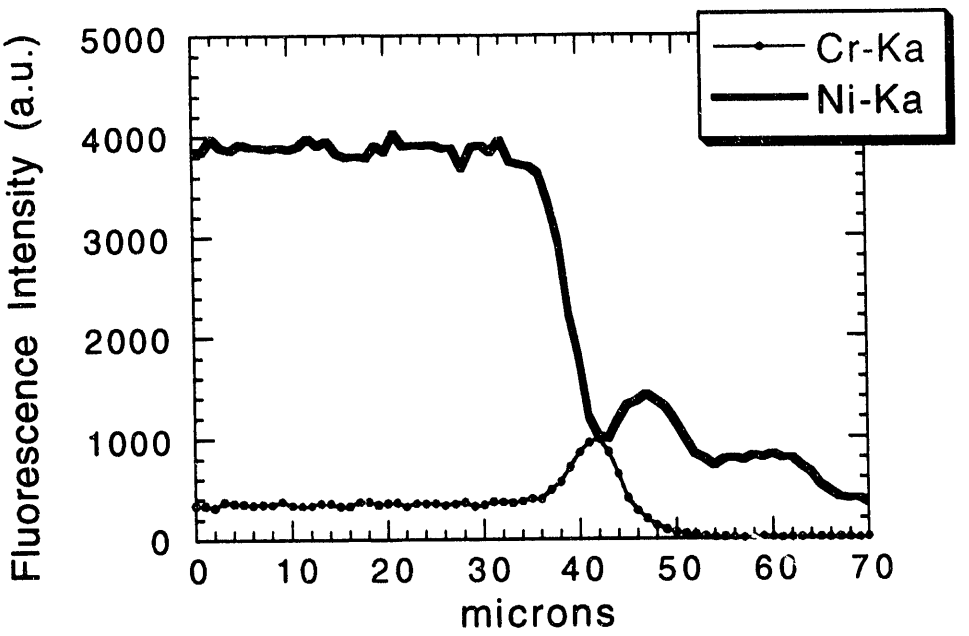
Microfocusing is important for studying the spatial distribution of trace elements, crystallographic structures, chemical states, and electronic properties in many material systems. Techniques to be developed are:

- Microscopy (Absorption and phase contrast)
- Microanalysis (Fluorescence, Auger or photo e^-)
- Microspectroscopy (EXAFS, XANES)
- Microdiffraction (Orientation, rocking curve)
- Microtomography (3D, high spatial resolution)

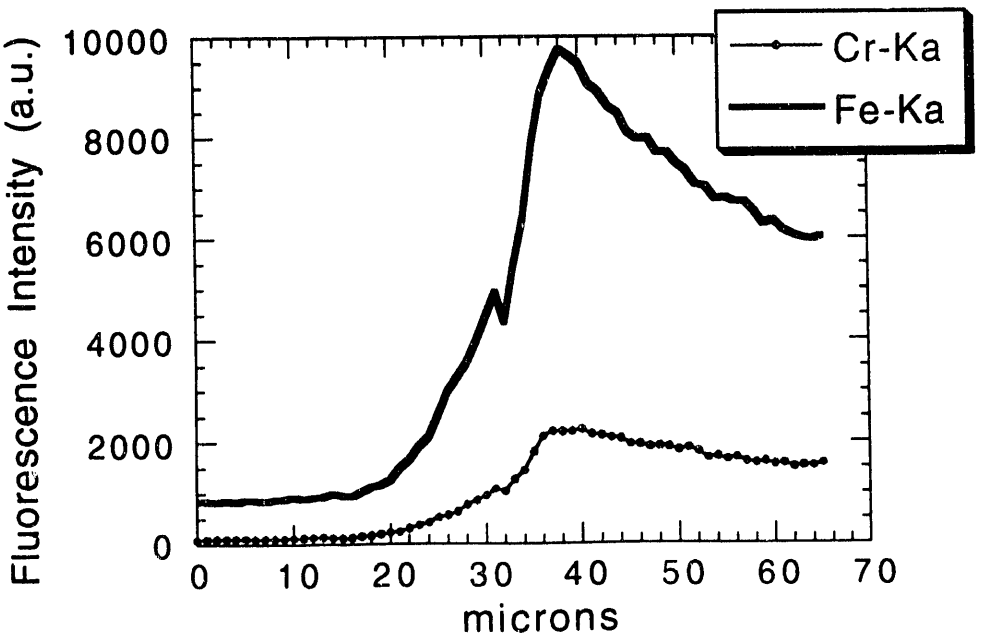


Microanalysis
of Interfacial Distribution

Ni-Cr/U-Zr Interface



U-Zr/Fe-Cr Interface



0.5 - 4 KeV Spectroscopy

Scientific Program

Soft X-ray Magnetic Circular Dichroism

Dilute magnetic systems :
Magnetic impurities & biological samples

Magnetic phase transition and critical phenomena

Magnetic photoelectron microscopy

Science of Nano-Engineered Multilayer Materials

In-situ tin film growth by
EXAFS / NEXAFS and x-ray diffraction

Molecular and Atomic Physics

High resolution spectroscopy

XPS

Auger

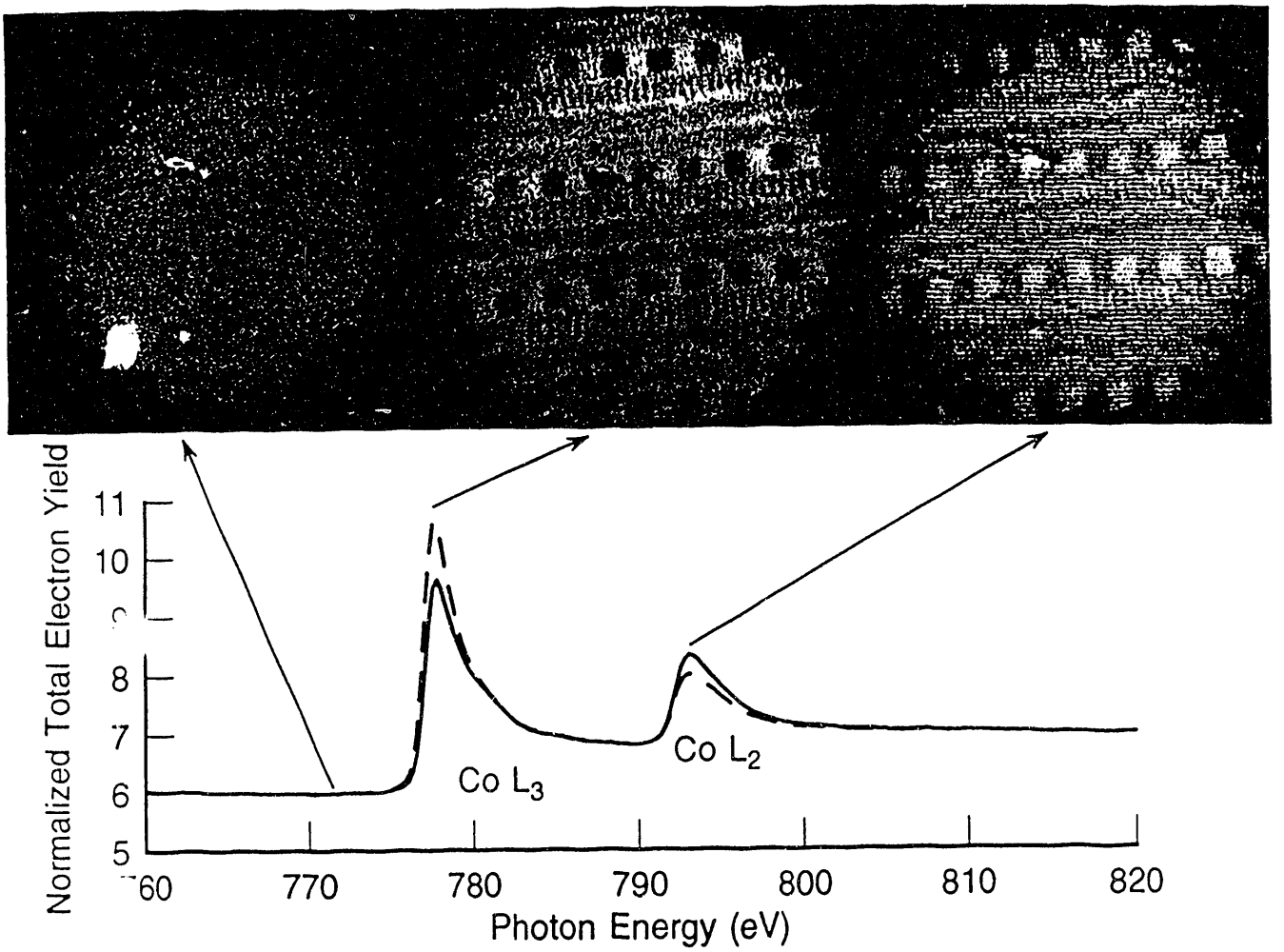
Spin resolved photoemission

SXR Fluorescence

Ion spectrometry

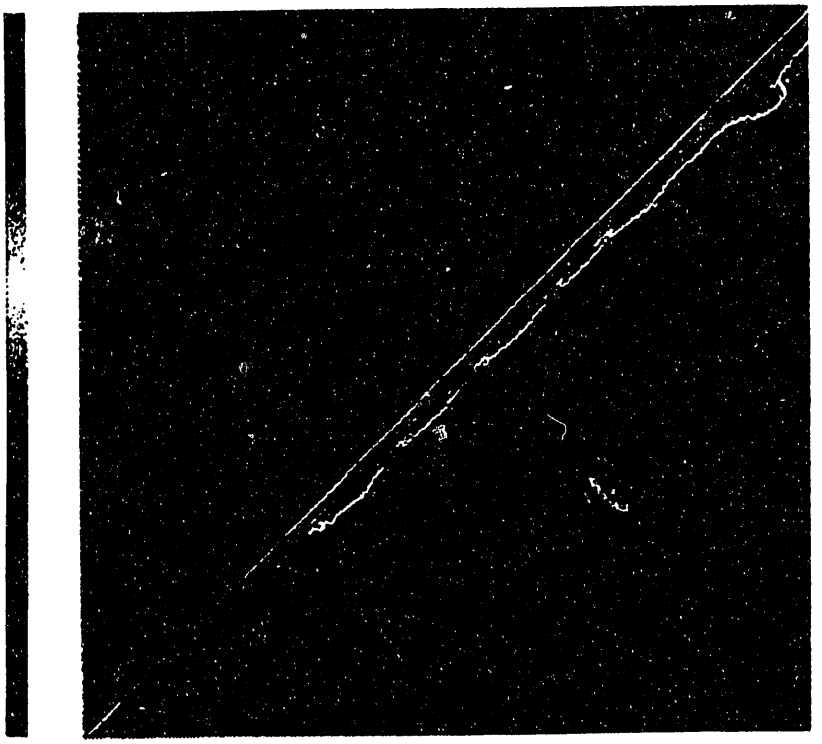
Coincidence techniques

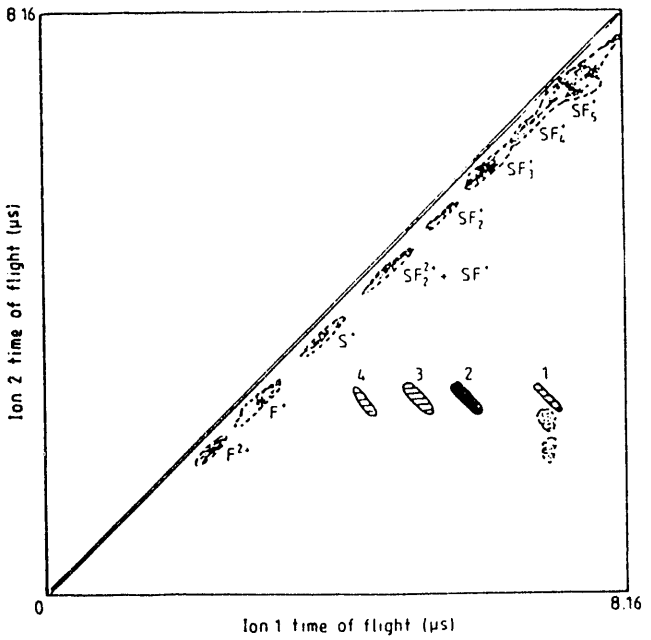
MCXD Microscopy



Magnetic Circular X-ray Dichroism (MCXD) microscopy of a CoPtCr magnetic recording disk (J. Stohr et al, Science-submitted). An alternating pattern of in-plane magnetization direction domains ('bits'). is imaged at the Co L edge. Features of 10x10 μm and 10x2 μm are discerable in this figure.

COINCIDENCE SPECTROSCOPY





Ion-Ion coincidences in the double ionisation of SF6 at 56.4eV. On the intensity scale dark blue represents 1 count, magenta >29 counts. The features are labelled in the figure below.

Coincidence features : 1) $SF_5^+ + F^+$; 2) $SF_3^+ + F^+$; 3) $SF_2^+ + F^+$; 4) $SF^+ + F^+$.

Multiple products (two ions, in the example above) arising from a single photoexcitation can be tagged by temporally correlating their respective signals. Such coincidence techniques examplified in the false color map above give a clear and definitive pictorial insight into the multiple processes available to an atom or molecule at a single photon energy.

Holographic Imaging

High resolution 3D imaging by Fourier transform x-ray holography in the 1-4 KeV energy region

Approach

3D imaging using tomographic (multiple-view) holography.

Elemental, chemical sensitivity with absorption *and* phase contrast.

High 2D and limited 3D resolution in single holographic projections.

Goal is to obtain 100 nm resolution in all three dimensions.

Applications

Imaging and defect analysis of microcircuits

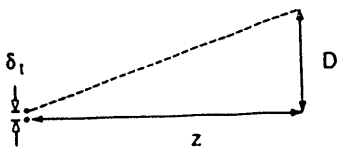
- Non-destructive *in-situ* imaging.
- X-ray penetration depth suited to imaging circuit interconnects through micron-thick substrates (Si, GaAs...).
- Both phase and absorption contrast at K,L-edges of industrially important materials (Al,Cu,Ti,W, Au...).
- Eliminates insulator charging problem in electron microscopy.

Biological microscopy

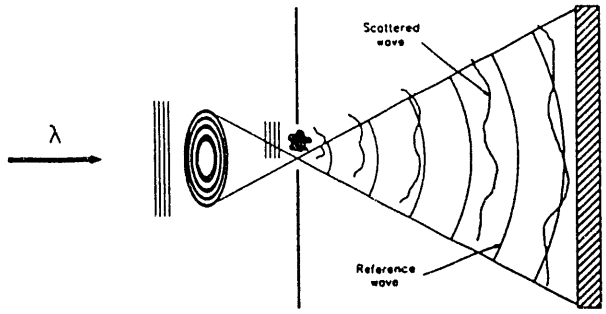
- Specimens can be initially living, thick (< 10 μm), wet, and in air.
- Resolution superior to visible light microscope, less radiation damage than electron microscopes.
- Phase contrast with reduced dose possible for biological objects in aqueous environments.

Hologram Formation

Record interference of wave scattered by specimen with spherical reference wave.



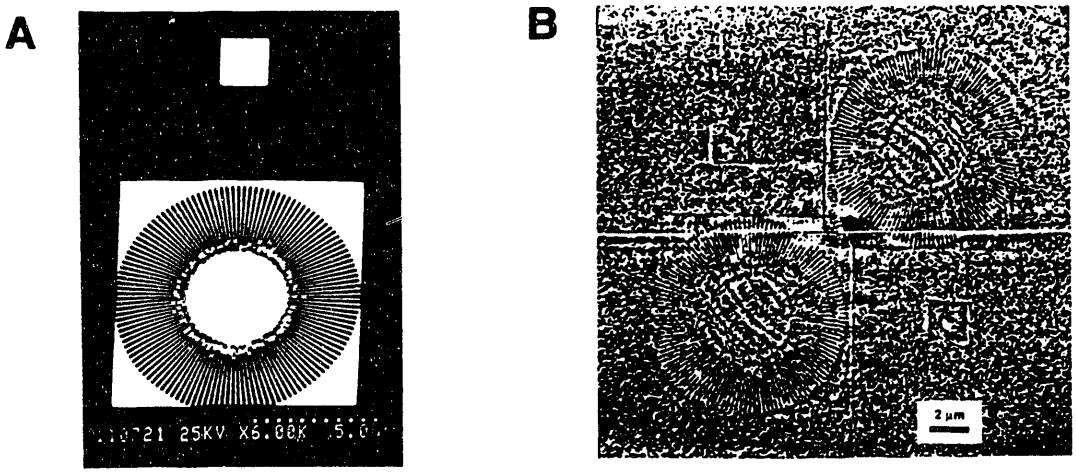
$$\delta_t = 1.22 \frac{\lambda z}{D}$$



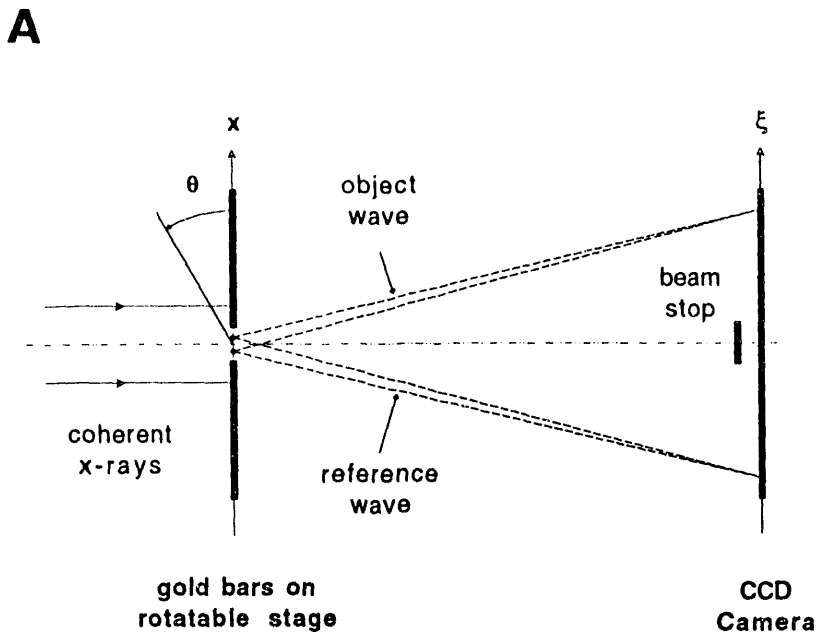
Hologram Reconstruction

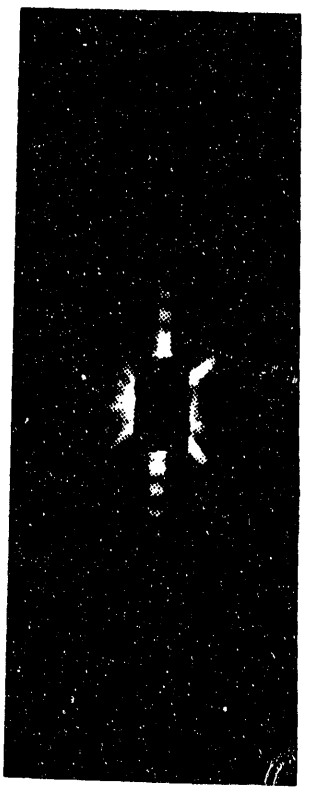
Take Fourier transform of hologram with computer.

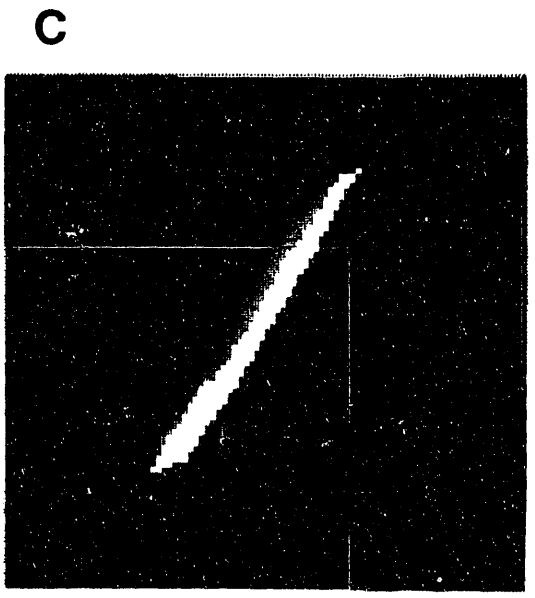
$$I(\xi,\eta) \Rightarrow \iint I(\xi,\eta)\, e^{\frac{-2\pi i}{\lambda z}(x\xi+y\eta)}\, d\xi d\eta = A(x,y)$$

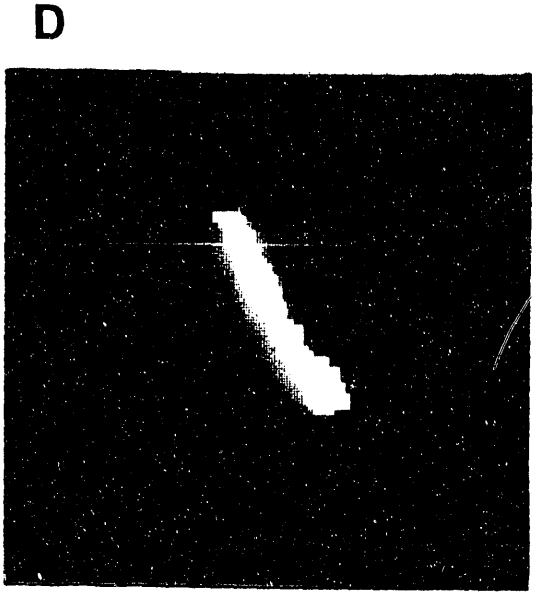


High resolution 2D Fourier transform holography with soft x-rays.
(**A**) SEM image of gold test object on Si_3N_4 membrame with 50-125 nm features.
(**B**) Numerical reconstruction of test object from hologram formed with 3.2 nm x-rays.
See: McNulty, J. Kirz, C. Jacobsen, E.A. Anderson, M.R. Howells, and D. P. Kern *Science* **256**. 1009 (1992).

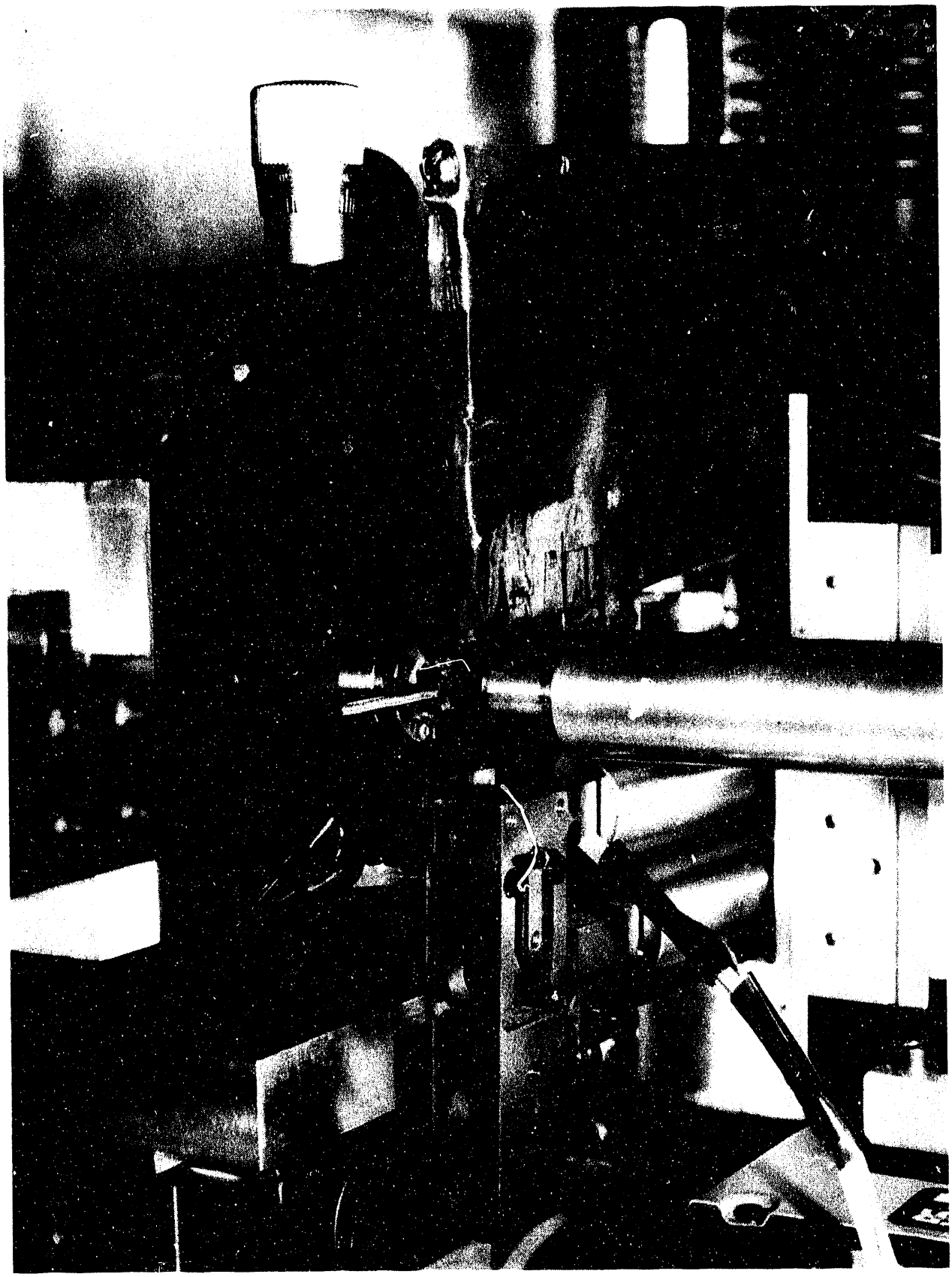


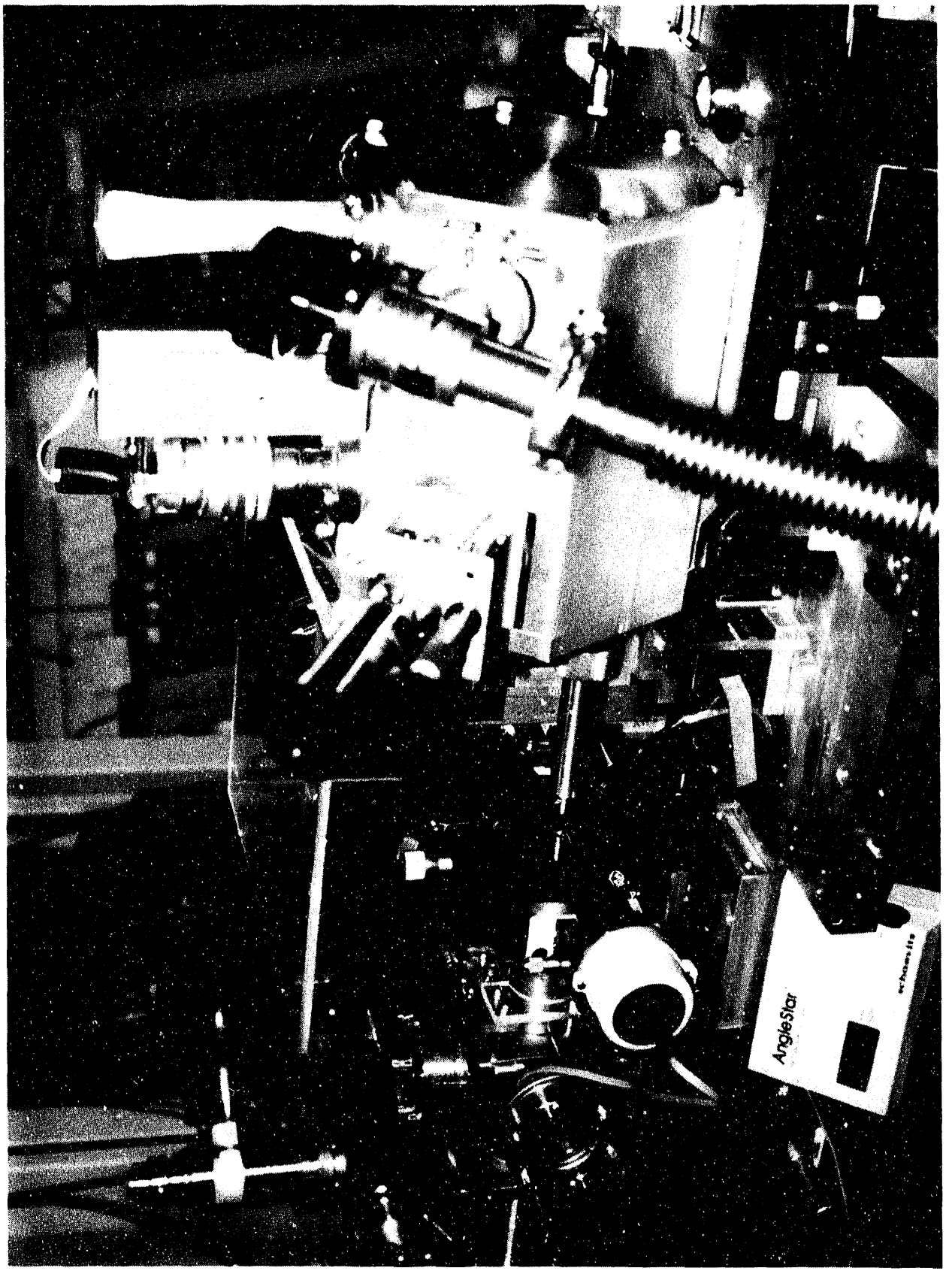






Demonstration of 3D Fourier transform x-ray holography. (**A**) Optical setup. Two gold bars, 130 nm wide, 90 nm thick, and 2.5 μm apart, are coherently illuminated to produce object and reference waves. (**B**) Hologram obtained with 3.2 nm x-rays. (**C,D**) 3D reconstructions of 1-μm-long segment of bar. Object is well resolved and located in space.



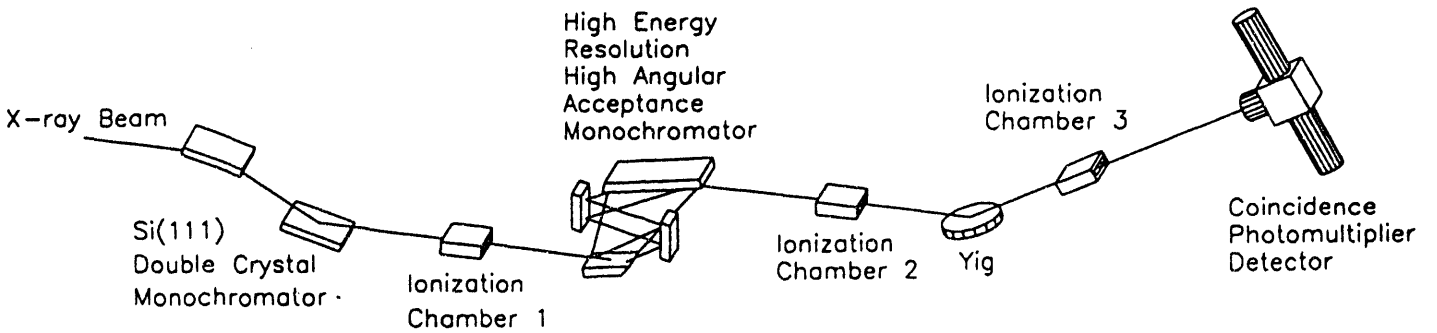

AngleStar

SRI CAT

Sector 3

Nuclear Resonant Scattering

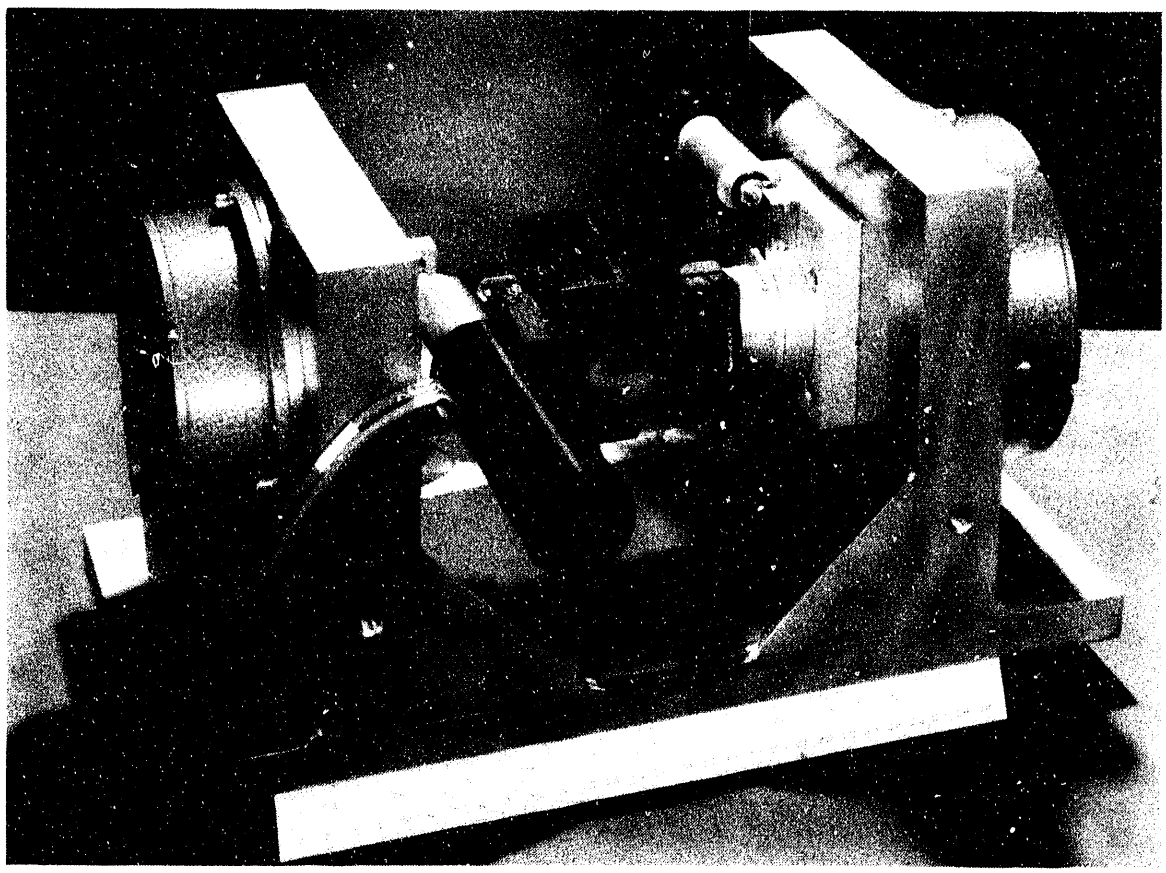
The coherent resonant scattering of synchrotron radiation from nuclei produces highly collimated and monochromatic beam at hard x-ray energies. The brightness of such beams are few orders of magnitude larger than radioactive sources. Typical energy resolution is $\Delta E/E = 10^{-10}$-10^{-13}. Such beams are suitable for studying low energy excitations at the μeV level, in addition to their complementary and at times unique role to traditional Mossbauer sources. At APS, we expect to be able to record a Mossbauer spectrum in few seconds as compared to few days it takes presently. So far this technique has been successfully applied to three isotopes: ^{169}Tm at 8401 eV, ^{57}Fe at 14413 eV and very recently ^{119}Sn at 23870 eV.



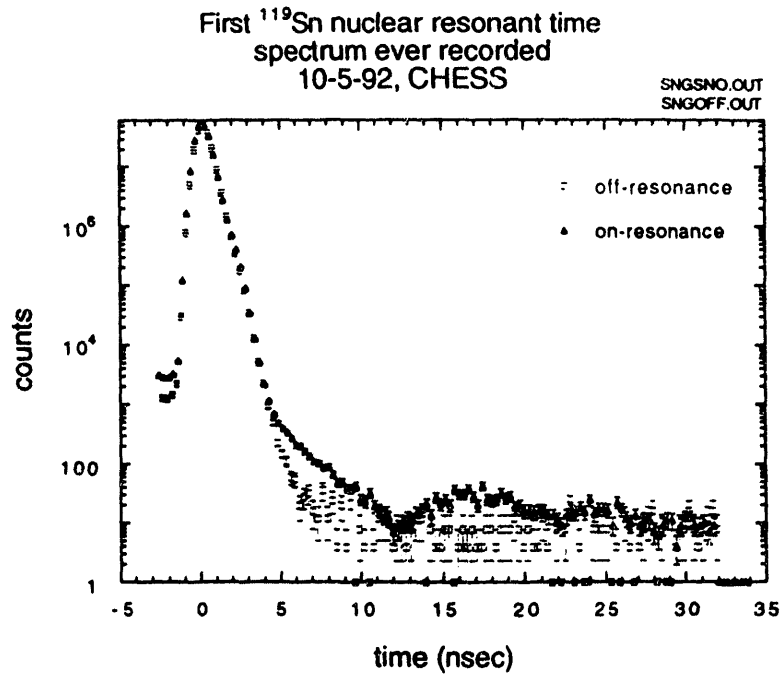
High Energy Resolution, Large Angular Acceptance Crystal Monochromator

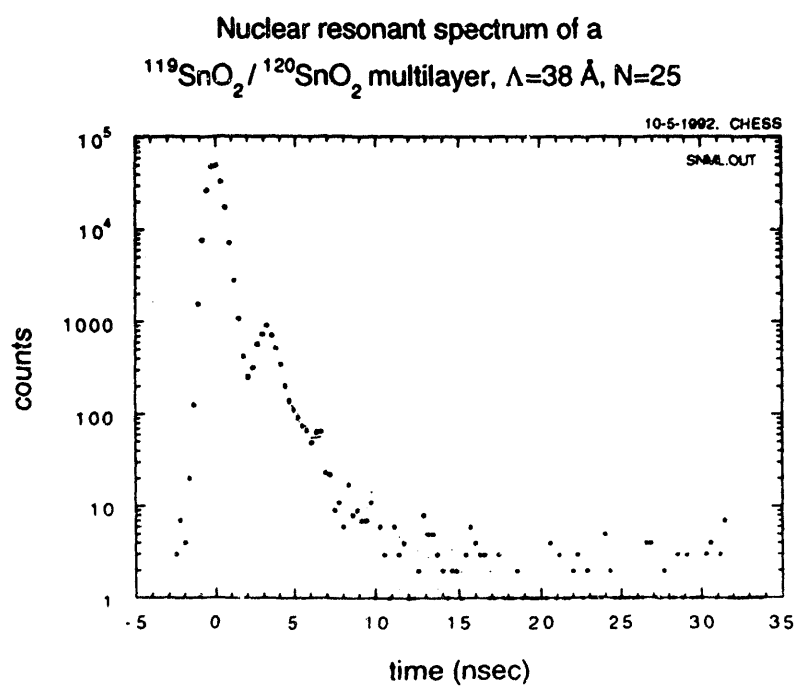
$\Delta E = 10$ meV, $\Delta\Theta = 20$ μrad at 14413 eV

$\Delta E = 50$ meV, $\Delta\Theta = 6$ μrad at 23870 eV



Observation of time resolved nuclear resonant scattering from ^{119}Sn nuclei using SnO_2 / Pd GIAR films





Inelastic X-ray Scattering

INELASTIC SCATTERING INSTRUMENTS

The optical objectives of the beamline are to efficiently deliver focused undulator radiation at the 50 m sample position for inelastic scattering. This will be accomplished in two different but compatible beamline arrangements, one with an overall resolution of 14 meV, and the other with an overall resolution of 0.70 eV.

The first of these is schematically shown in Fig. 1, and it will be referred to as the backscattering arrangement. It is derived from the pioneering INELAX instrument design at HASYLAB. The backscattering arrangement will function with a fixed incident energy of 13.84 keV. It consists of a Si(111) high heat load monochromator (HHLM) followed by a toroidal mirror plus flat steering mirror combination which will be focused at an image point 90 m downstream. The Si(111) Darwin width, 20 μrad, matches well with the vertical divergence of the undulator, 25 μrad. (All width values are FWHM). Ray tracing for the mirror has been performed. The size of the beam at the image point was found to be 0.22 mm vertically by 1.03 mm horizontally, and the corresponding divergences are 20 μrad vertically by 44 μrad horizontally. A bandwidth smaller than 10 meV is calculated for backscattering at a Bragg angle of 89.5 deg from the (777) reflection of a flat Si crystal positioned at 70 m. The scattering plane of the monochromator will be horizontal. The Darwin curve of the Si(777)

reflection at 13.84 keV efficiently matches the horizontal beam divergence exiting from the mirror (42 μrad Darwin width and 44 μrad divergence). Inelastically scattered radiation produced from samples at 50 m will be collected by a Si(777) spherical analyzer. The scattering plane for the inelastically scattered radiation will be vertical, and the analyzer will be positioned 3 m from the sample.

The arrangement in the hutch is schematically shown in Fig. 2. The Bragg angle for the analyzer will be 89.96 deg. This implies a 4 mm offset between the sample and the analyzer output beam. Such a small value is needed so that the entire 1 mm by 1 mm flat area of analyzer elements accepts the inelastically scattered radiation. The analyzer will be grooved for strain relief. It will have flat elements no larger than 1 mm by 1 mm. The size of the focal spot of the mirror must be no larger than this element size in order to attain the desired 10 meV resolution and for the analyzer to operate efficiently. Inelastic spectra will be obtained by varying the temperature difference between the backscattering monochromator and the analyzer. Both of these optical components must be temperature controlled. The thermal expansion coefficient of Si sets the scale for the required temperature control resolution and leads to an energy difference of 1 meV between monochromator and analyzer for a 30 milliKelvin temperature difference. We have set 30 milliKelvin control for the temperature difference as our design goal.

ADVANCED PHOTON SOURCE

BACKSCATTERING ARRANGEMENT

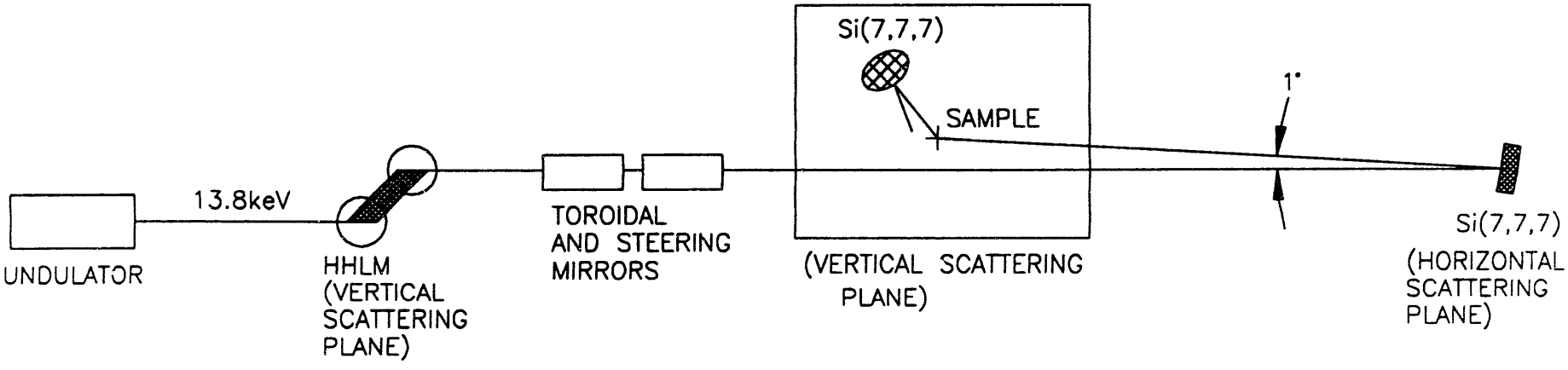


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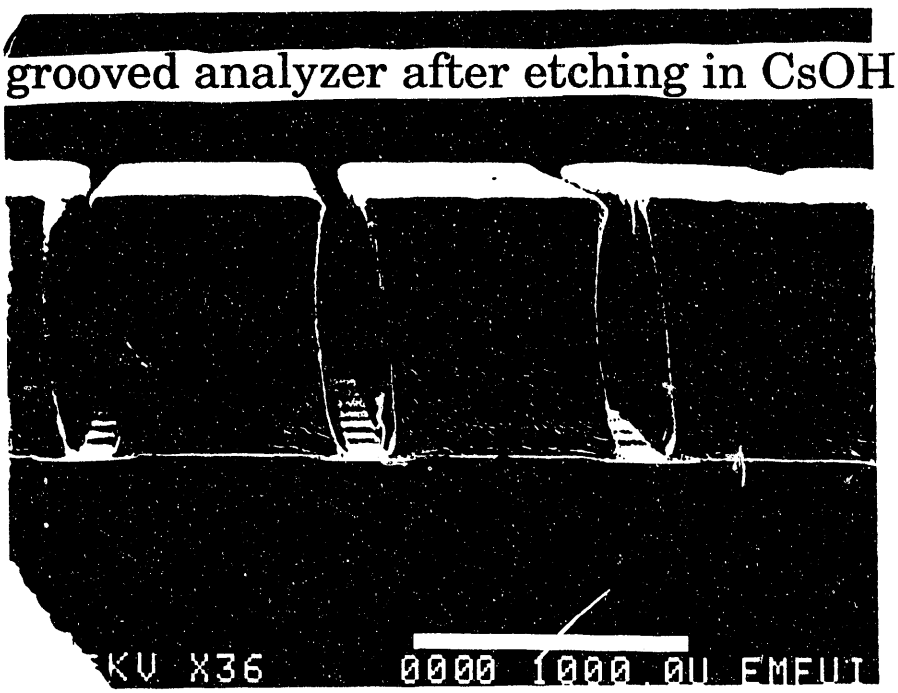
Fig. 1

S05427

grooved analyzer after etching in CsOH



HUTCH PLAN VIEW FOR VERY HIGH RESOLUTION

(SHOWN @ Q=0)

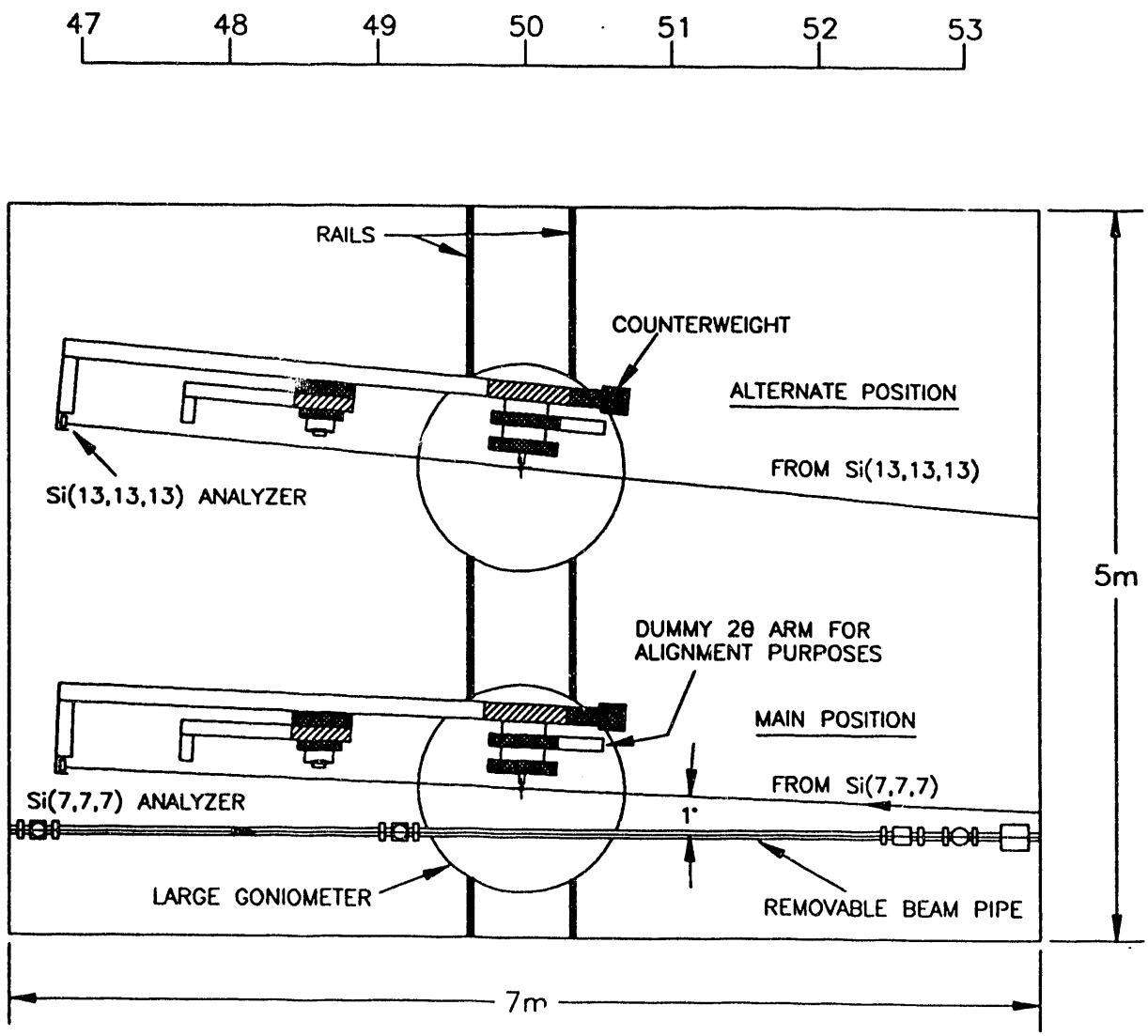


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Fig. 2

X-RAY PHYSICS

John Arthur, SSRL:

- Nuclear resonant diffraction.
- Production of intense resonant beams (several thousands of counts/sec).
- Studies of magnetism in layered structures containing iron.
- Studies of magnetic fluctuations in the time domain.
- Macroscopic x-ray holography.
- Long path interferometry.

Don Bilderback, CHESS

- Microbeam x-ray optics.
- Tapered light pipes, Fresnel lenses, Multilayers.
- Capillary Lenses.
- Applications envisioned in the field of microcrystallography.

Roberto Colella, Purdue University

- Quasicrystallograpy.
- Multibeam experiments in quasicrystals, phasing Bragg reflections.
- Dynamical effects in quasicrystals.
- Compton scattering from small crystals.
- High energy diffraction.

Paul Cowan, APS

- Physical optics.
- Standing waves. Standard and back-reflection geometry.
- Interferometry (Fabry-Pérot, Fourier transform spectroscopy, Moiré fringes).
- Evanescent beams (x-ray depth profiling, creation of microscopic x-ray beams).

Steve Durbin, Purdue University

- Standing waves. Use of undulator radiation.
- Selective area diffraction.

- Imaging of x-ray standing wave yields. Use of area detectors; image intensifier or CCD array.
- Surface studies with very low adsorbate coverages.
- Time resolved studies of adsorbates in excited electronic configurations due to laser excitation.

Terry Jach, NIST

- Standing waves. Surface atoms. Interfaces.
- Grazing incidence. Ultra high vacuum environment.
- Standing wave modulation parallel to surface. Information about atomic registration parallel to surface can be obtained.
- Study of epitaxial overlayers on semiconductor crystals.

Simon Moss, University of Houston

- Diffraction physics studies on microcrystals.
- Huang scattering, defects and their displacement fields.
- Time dependent ordering studies.
- Short range order, phase transitions (grazing incidence).
- "Perfect" quasicrystals.
- Anomalous scattering, order-disorder transition, interdiffusion in semiconductor epitaxial overlayers.

Qun Shen, CHESS

- Use of elliptically and circularly polarized x-rays.
- Magnetic circular dichroism, spin dependent Compton scattering.
- Magnetic x-ray scattering.
- X-ray optical activity in chiral compounds.

- Multiple Bragg scattering as a tool to describe handedness or polarity of non-centrosym crystals.

Al Thompson, Lawrence Berkeley Lab

- X-ray optics.
- Focusing, imaging and spectroscopy with multilayer optical elements.
- Multilayer optical elements on dynamically bent mirrors.
- Special multilayer optical elements.
- Diffraction limited optical elements.

UNI CAT

A University-National Laboratory-Industry CAT

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Member Institutions

University of Illinois at Urbana-Champaign

Allied-Signal Research and Technology

UOP Research and Development

Oak Ridge National Laboratory

Area of Proposed Research

1. Structural Crystallogrphy
2. Macromolecular Crystallography
3. Diffuse Scattering
4. Surface/Interface Diffraction and Scattering
5. Milli/Nanovolt Resolution Scattering Spectroscopy
6. Magnetic X-ray Scattering
7. Time-Resolved Structural Scattering
8. X-ray Absorption Spectroscopy
9. Coherent X-ray Scattering

Overall Thrust

(1) Research at the Cutting Edge

in physics, chemistry, biology, materials science, chemical engineering, polymer science, and geology with emphasis on **close collaboration and joint research/ development** between **universities, industries and national laboratories**

(2) Education of a new Generation of Scientists

with expertise in the use of synchrotron radiation to probe the structure, chemistry and dynamic behavior of materials

Features of the Beam Line

1. Type A Undulator was chosen.

2. Monochromators
   - A. Sagittally Focusing Si(111)
     General Purpose
   - B. Flat Monochromator
     EXAFS and XANES
   - C. Nested Asymmetric Channel-Cut Crystals
     Conditioning to 100 meV Resolution

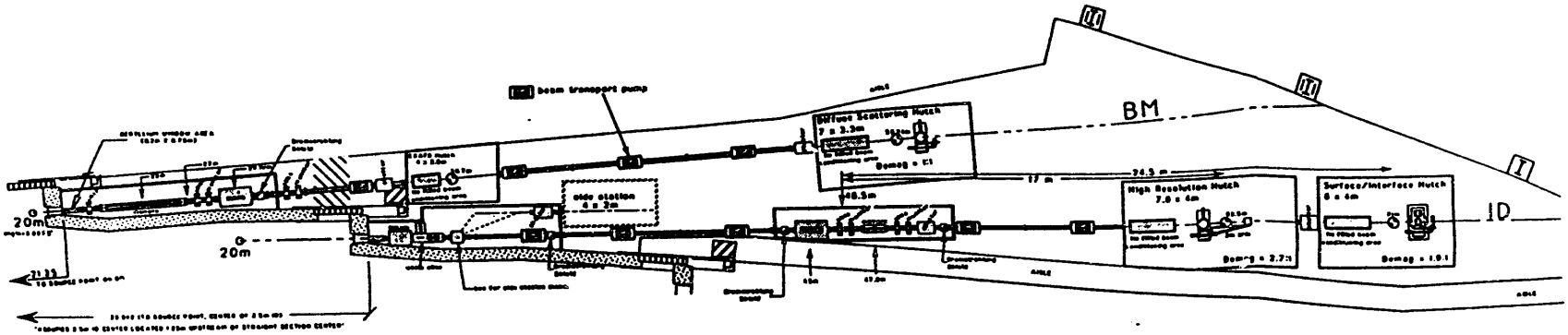
3. Mirrors (Rhodium Coated)
   - A. Vertically Focusing First Mirror
   - B. Secondary Flat Mirror

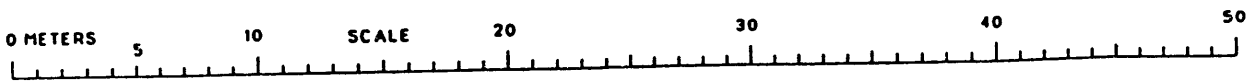
4. Brightness *(conservative estimation)*
   - A. ID line
     Flux: 2×10^{13} ph/s (8 KeV, 1.2 eV B.P.)
     Size: < 0.25 mm^2 (> 0.08 mm^2)
     Divergence: $20 \sim 30$ arcsec (h or v)
   - B. BM line
     Flux: 5×10^{12} ph/s
     Size: 0.25 mm^2
     Divergence: 6 mrad x 15 μrad

Experimental End Stations

A. ID Line**	1. High Energy Resolution Station
	2. Surface/Interface Station
B. BM Line	1. EXAFS Station
	2. Diffuse Scattering Station

**One Side Station is under consideration





UNI-CAT, General Layout

Center for Synchrotron Radiation Research and Instrumentation

Illinois Institute of Technology

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Mission of the CSRRI

The Center for Synchrotron Radiation Research and Instrumentation (CSRRI) at Illinois Institute of Technology is dedicated to promoting the application of the tools and techniques of synchrotron radiation to science and engineering research. The Center serves the synchrotron research community by:

- Acting as a resource for industrial affiliates and university researchers who wish to take advantage of the research opportunities afforded by synchrotron-based research to strengthen their competiveness.
- Participating in the the development of new instrumentation and technologies necessary for the construction of experimental facilities at synchrotron radiation sources.
- Training researchers in the techniques of synchrotron radiation science through short courses for scientists who wish to broaden their research capabilities and through graduate programs of study in synchrotron-related fields of research.
- Disseminating information on synchrotron research opportunities.

In addition, the Center provides a nucleation point for synchrotron-based research at IIT. The strength of synchrotron radiation is in its applicability to a wide range of disciplines, from materials science, chemistry and electronics to biology and medicine. This diversity, and the unique properties of the Advanced Photon Source ensure that synchrotron radiation research will continue to grow in importance throughout the Chicago area.

Relations with CATs

At the present, the CSRRI is working with three different Collaborative Access Teams (CATs) (IMCACAT, BioCAT and MRCAT) in varying degrees. The Center accomodates such joint projects with CATs in three distinct modes:

- CATs may contract with the CSRRI to carry out R&D projects in accordance with CAT requirements.
- CATs may specify presonnel to be hired as members of the CSRRI R&D and engineering teams, to work exclusively on CAT projects, but with daily activities and resource allocation coordinated by CSRRI. CAT management will be responsible for frequent contact with CSRRI personnel working on that CAT project.
- CATs may employ their own staff members within the CSRRI dedicated to specific CAT projects, under direction of CAT magament. CSRRI resource allocation will be handled by coordinators.

Each of these modes offers different kinds of benefits to CATs which choose to collaborate with the CSRRI, from simple contractual work to a more complete control over the project, if desired. In all cases, the environment and resources provided by the CSRRI can help CATs in the design and realization of their synchrotron research facility.

Education and Dissemination

The location of the CSRRI at a university such as Illinois Institute of Technology gives it an opportunity to be more than just an R&D group. The educational mission of IIT is ideally suited to providing the education and training necessary for the research community at large to make full use of the opportunities afforded by the third-generation synchrotron sources such as APS. IIT, under the umbrella of the CSRRI has active programs in synchrotron radiation research which will train graduate students at the Ph.D and M.S. levels. Furthermore, there are short courses and graduate courses specifically designed to introduce scientists and graduate students to the fundamentals of synchrotron radiation and the experimental techniques commonly used in the field.

The formal educational activities are only a portion of a broader outreach program by the CSRRI, which includes a regular newsletter, technical reports and traditional scientific publications. In addition, the Center is developing a pilot EXAFS database, for both software and standard spectra, which will be made available to the scientific community.

Engineering and R&D Activities

The CSRRI has assembled the infrastructure and expertise necessary to carry out much of the work which must go into a successful technical design for a synchrotron beamline at the Advanced Photon Source. These include:

- CAD drawings of beamline and sector layouts using the APS-standard AutoCAD.
- Source calculations for bending magnets, wigglers and undulators with codes developed in-house to give maximum flexibility in determining optimal parameters.
- Optical simulations of ideal and non-ideal cases.
- Finite-element-analysis calculations for identification of potential thermal loading, stress and vibration problems.

Spectral Characteristics of a Variable Taper Undulator

We have developed a general FFT-based method for the calculation of the emission spectra of tapered undulators.† The method allows the inclusion of non-periodic magnetic field errors and does not require a linear approximation of the magnetic field variation with taper. The table summarizes the results for several test cases with the following definitions:

λ_o the magnet period is 3.3 cm,

N the number of periods is 30,

G_{in} is the magnet gap at $z = 0$,

ΔG is the amount of gap taper,

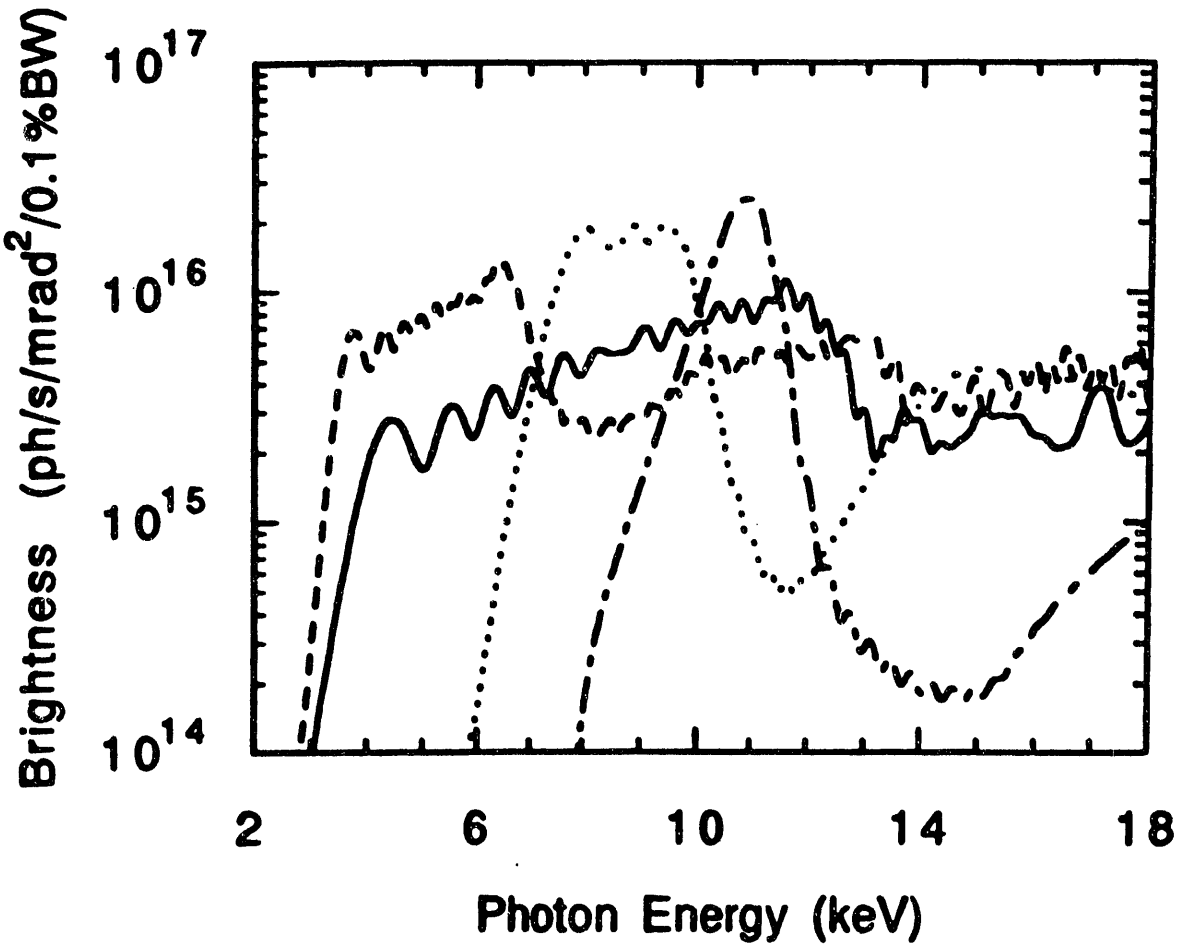
K_{in} is the K-factor of the maximum magnet gap,

ΔK is the maximum variation in K.

Label	G_{in} (cm)	ΔG (cm)	K_{in}	ΔK
U(1.0,1.8)	1.0	1.8	0.410	2.079
U(1.0,0.6)	1.0	0.6	1.232	1.257
U(1.6,0.6)	1.6	0.6	0.675	0.557
U(2.2,0.6)	2.2	0.6	0.410	0.267

The figure shows the spectrum of the on-axis brightness (including emittance) of these undulators with: U(1.0,1.8)–solid line; U(1.0,0.6)–dashed line; U(1.6,0.6)–dotted line; U(2.2,0.6)–dash-dot line.

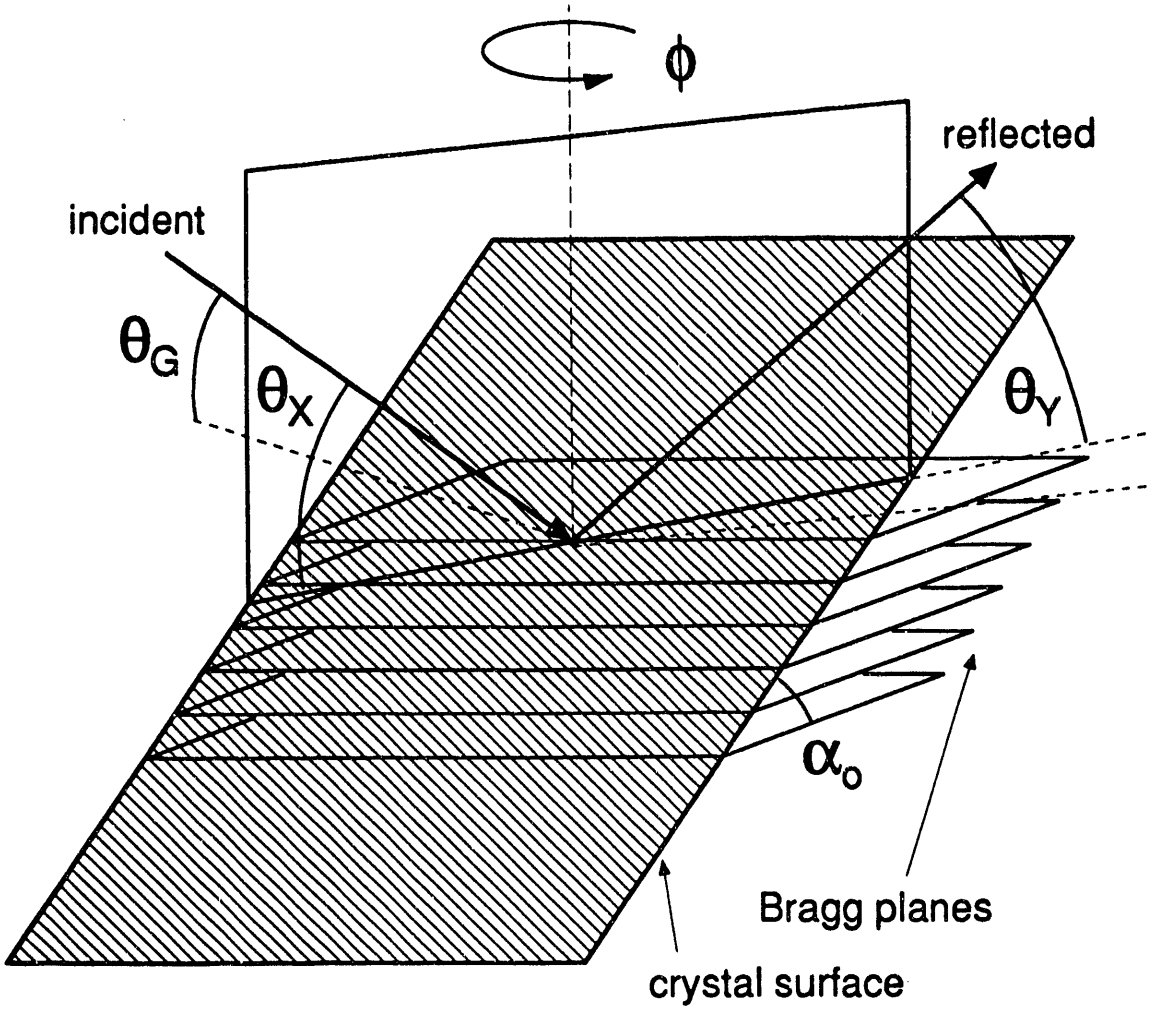
† B. Boyanov et al, *Nucl. Instrum. and Meth.*, submitted.

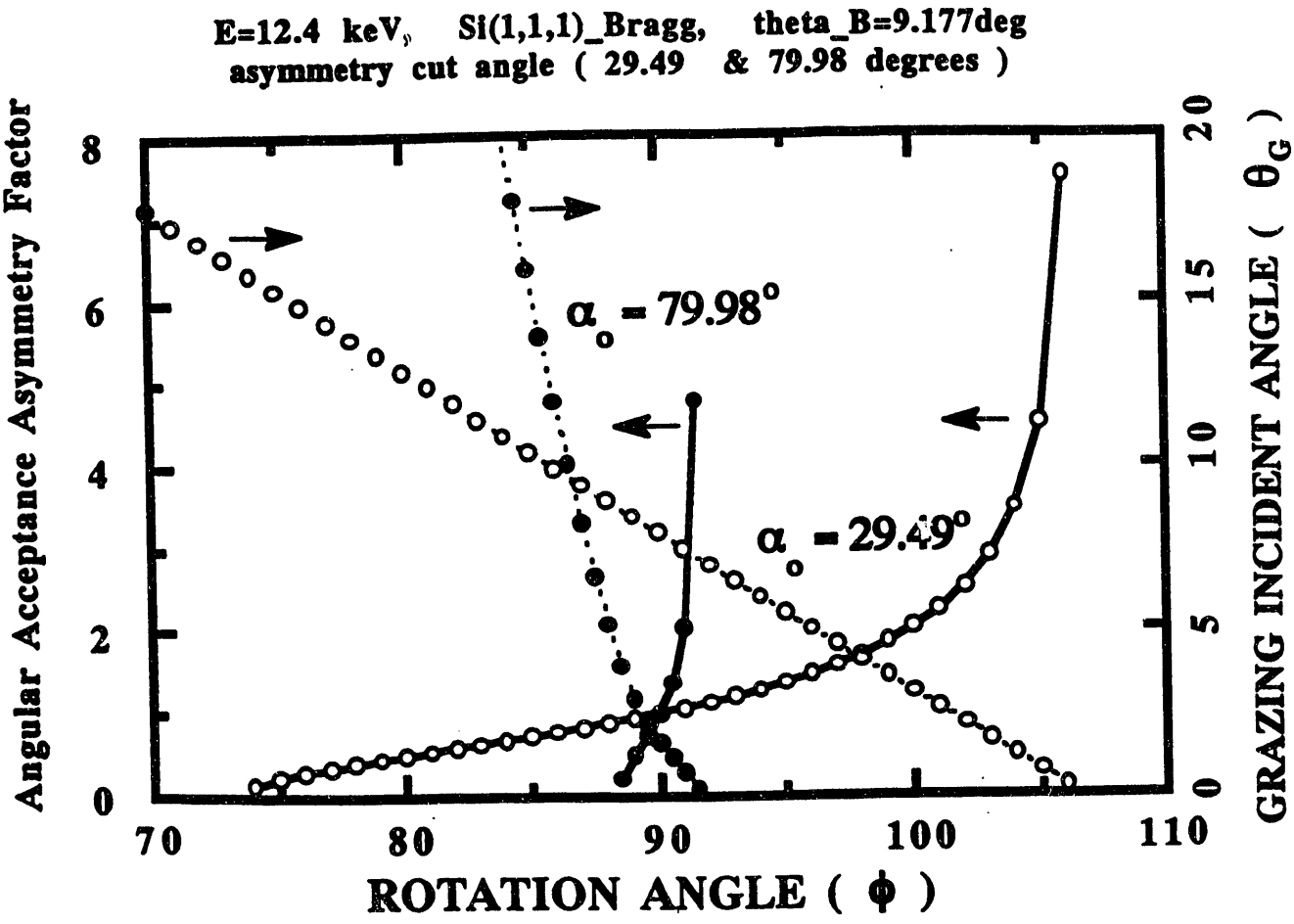

Brightness (ph/s/mrad²/0.1%BW)
10¹⁷
10¹⁶
10¹⁵
10¹⁴
2
6
10
14
18
Photon Energy (keV)

Energy Resolution of a Single Asymmetric–Cut Inclined Crystal

The inclined crystal geometry has been proposed by APS[†§‡] to effectively avoid thermal loading problems expected at a high brilliance third-generation synchrotron source. We have studied the most general case of such a geometry by considering a single monochromator crystal whose surface is cut at an angle α_o to the Bragg planes used for wavelength selection. At the Bragg angle, the angular acceptance of the crystal and the beam footprint depend on the rotation angle ϕ about the normal to the Bragg planes. Referring to the figure, the angles θ_X and θ_Y are defined as the angles, in the plane of reflection, between the incident beam and the crystal surface and the reflected beam and the crystal surface, respectively. We can define the asymmetry factor $b = \sin\theta_X / \sin\theta_Y$. For two different values of the asymmetric cut angle α_o in silicon, we have calculated, as a function of rotation angle ϕ, the grazing incidence angle θ_G, which determines the beam footprint on the crystal surface, and the angular acceptance asymmetry factor, which is proportional to $\sqrt{b}$ and controls the expected bandpass of the crystal. Such a crystal geometry could have uses in reducing thermal effects, producing a polychromatic beam as well as delivering the full divergence of a beam to the experimental station.

† A. M. Khounsary, *SRI 91 Conference, Chester, England.*
§ W. K. Lee and A. T. Macrander, *7th National Conference on SRI, Baton Rouge.*
‡ A. T. Macrander and W. K. Lee, *7th National Conference on SRI, Baton Rouge.*


ϕ
reflected
incident
θG
θX
θY
αo
Bragg planes
crystal surface


E=12.4 keV, Si(1,1,1)_Bragg, theta_B=9.177deg
asymmetry cut angle (29.49 & 79.98 degrees)
Angular Acceptance Asymmetry Factor
GRAZING INCIDENT ANGLE (θG)
ROTATION ANGLE (ϕ)
α0 = 79.98°
α0 = 29.49°
0
2
4
6
8
0
5
10
15
20
70
80
90
100
110

END

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