New Ways to See a Smaller World: 
The Hard X-ray Nanoprobe 
at Argonne National Laboratory

Volker Rose
Advanced Photon Source

Robert Winarski, Martin Holt, Jörg Maser
Center for Nanoscale Materials
The Hard X-ray Nanoprobe (HXN) Team

• Jörg Maser
• Robert Winarski
• Martin Holt
• Brian Stephenson
• Peter Fuesz
• Dean Carbaugh
• Volker Rose (vrose@anl.gov)

http://nano.anl.gov/research/nanoprobe.html
Outline

- What is the Hard X-ray Nanoprobe?
- Nanoprobe Beamline design
- Some recent results
Nanoprobe Instrument Overall Specifications

- Hard X-ray microscopy at the highest achievable spatial resolution
  - Initial spot size resolution $d = 30\text{nm}$
  - Energy range 3 - 30keV (nano-spectroscopy excitation of most elements)
  - Large penetration $\rightarrow$ sample environments/fields

- Modes of operation
  - **X-ray fluorescence**: atto-g elemental sensitivity, chemical state sensitivity
  - **Diffraction**: sensitivity to crystallographic phase, strain, orientation
  - **Tomography**: 100 nm$^3$ resolution of transmission absorption / Zernicke phase contrast imaging
  - **Coherent x-ray studies**: disorder, imaging
  - **Magnetic contrast** using polarized x-rays
  - **Dynamic** studies at 100 ps time resolution
  - **Spectroscopy**

---

[Image: Fresnel Zone Plate]
X-ray Characterization at CNM and APS

- The goal of Argonne's Center for Nanoscale Materials (CNM) is to create, characterize, and understand the behavior of new functional materials on the nanoscale.

- Located adjacent to the CNM building at Sector 26 of the Advanced Photon Source (APS)

- Developed in partnership with the APS to build a state-of-the-art beamline that will provide stable, powerful, and coherent x-ray illumination for research at the nanoscale
Hard X-ray Nanoprobe: Combined Analytic and Imaging Mode

Scanning Probe Mode

Area detector: microdiffraction

Rotation for microdiffraction

Energy dispersive detector: X-ray fluorescence

Sample

Area detector: transmission

Crystal monochromator

Double mirror system

Beam defining Aperture (closed)

Two collinear undulators

Argonne National Laboratory

VOLKER ROSE, ROBERT WINARSKI, MARTIN HOLT, JÖRG MASER – HARD X-RAY NANOPROBE
Hard X-ray Nanoprobe Beamline

Scanning Probe Mode

Synchrotron
- Advanced Photon Source: 7 GeV ring
- Two collinear undulators
Enclosure 26-ID-A
- Contains all of our high-heat-load optical components
- Focus divergent beam
- New fully coherent source at BDA
**Hard X-ray Nanoprobe Beamline**

**Scanning Probe Mode**

- Energy
- Polarization
- Dynamics

---

**Enclosure 26-ID-B**

- Crystal monochromator
- Double mirror system
- Shutter Assembly
- Polarizer
- Multilayer Monochromator
- Pink Beam Slit Assembly
- Two collinear undulators

---

**Argonne National Laboratory**
Scanning Probe Mode

Area detector: microdiffraction

Sample

Rotation for microdiffraction

Energy dispersive detector: X-ray fluorescence

Area detector: transmission

Hard X-ray zone plate (focusing)

Crystal monochromator

Beam defining Aperture (closed)

Two collinear

Enclosure 26-ID-C

Nanoprobe Instrument

26-ID-C
Hard X-ray Nanoprobe Instrument

Reference Frame  Laser Optics  Imaging Optics Module
Condenser Module  Focusing Optics Module (Zone Plates)
Specimen Module  Sample

Volker Rose, Robert Winarski, Martin Holt, Jörg Maser – Hard X-ray Nanoprobe
Focusing X-rays With Zone Plates For Synchrotron Use

Resolution limited to $1.22 \cdot \Delta R_n$ (Raleigh criterion):

- Spatial coherence / source demagnification
  (combination of source size and distance)
- Temporal coherence / monochromaticity
  (small wavelength spread)

Constructive interference from adjacent zones
Focal length $f = \text{OD} \cdot \Delta R_n / \lambda$
Examples for Research at the Hard X-ray Nanoprobe

1. Fluorescence: Characterization of heterogeneous Ca and heavy metal distributions in geopolymers

2. Nanodiffraction: Strained Si-on-oxide hetero structures

3. Tomography: Scanning Tunneling Microscopy tip
The Hard X-ray Nanoprobe (HXN) Goes Green

Research may reduce CO₂ Greenhouse gas emissions by at least tens of millions of tones per annum worldwide

Geopolymers are being developed as an environmentally beneficial replacement to Portland cement for concrete production

The exceptional spatial resolution and penetration power of the HXN enables the study of the poorly understood heterogeneity and complex chemistry of geopolymers

John Provis¹, Volker Rose², Susan Bernal¹, Jany van Deventer¹
¹Melbourne University, ²Argonne National Laboratory
Nanodiffraction of strained SOI heterostructures

Conal Murray*, Sean Polvino*, Andrew Ying†, Ozgur Kalenci†, I.C. Noyan†

* IBM, †Columbia University
Hard X-ray Nanoprobe: Combined Analytic and Imaging Mode

Scanning Probe Mode

- Area detector: microdiffraction
- Crystal monochromator
- Double mirror system
- Beam defining Aperture (closed)
- Two collinear undulators

Full-Field Transmission Mode

- Area detector: transmission
- Energy dispersive detector: X-ray fluorescence
- Sample
- Condensor system
- Crystal Monochromator
- Beam defining Aperture (open)
- Rotation for tomography
- Phase ring
- Hard X-ray zone plate (imaging)
Tomography Of Scanning Tunneling Microscopy Tips
Designed to resolve objects smaller than 30 nanometers in size using intense x-rays with energies between 3 and 30 keV

Modes of operation:
- Transmission
- Fluorescence
- Diffraction
- Spectroscopy
- Polarization
- Tomography
- Dynamics

This work was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.
Become a User Today!

Go to www.aps.anl.gov