

Hard X-ray Microscopy Activities at **SPring-8**



-from sub- μm to nm size probing -

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Introduction and Overview

Optics:

**Fresnel Zone Plate,
Sputtered-sliced FZP,
Total-reflection mirror,
Refractive lens., etc.**

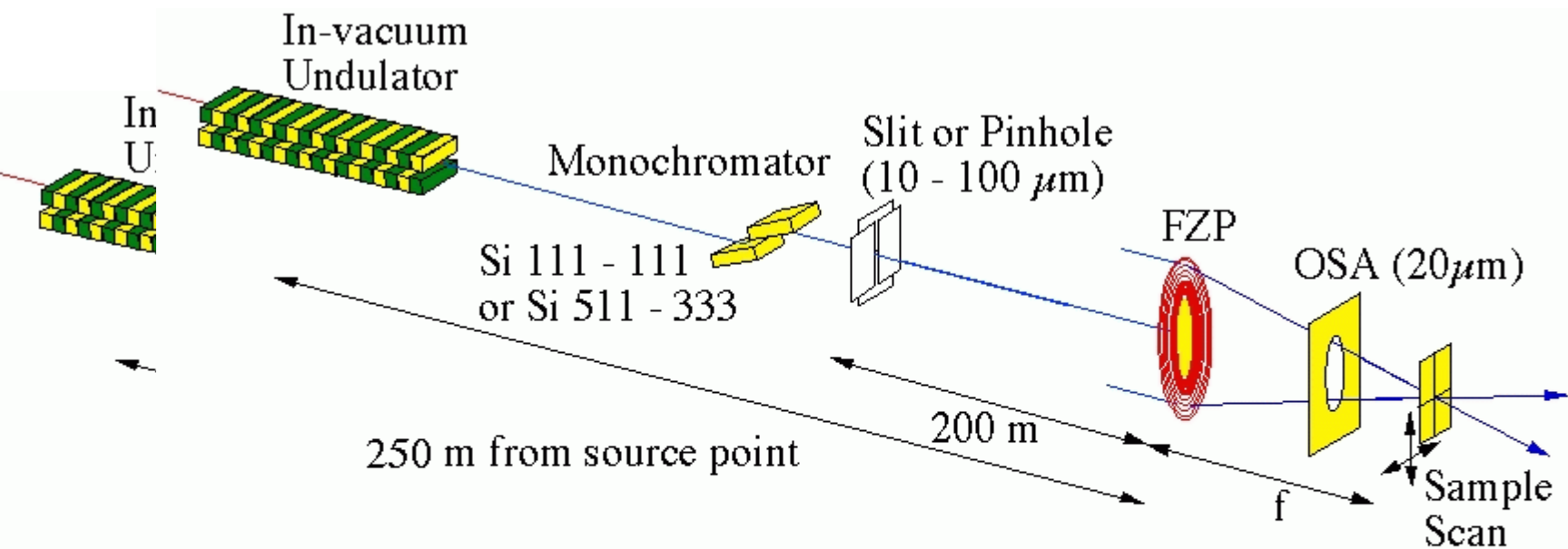
Beamlines:

**BL47XU (Microbeam, Imaging Microscopy, Computer Tomography)
BL20XU (Microbeam, Holography, Interferometer, etc.)
BL20B2 (CT, Topography, Medical Imaging, etc.)
BL37XU (Fluorescent X-ray Micro-analysis)**

Energy range:

6-113 keV,

**"Spatial resolution of $< 0.5 \mu\text{m}$ has been achieved with all the optics,
and the best resolution is $< 100 \text{ nm}$."**



Experimental Setup of X-ray Microbeam/Scanning Microscopy

Ex

at BL20XU

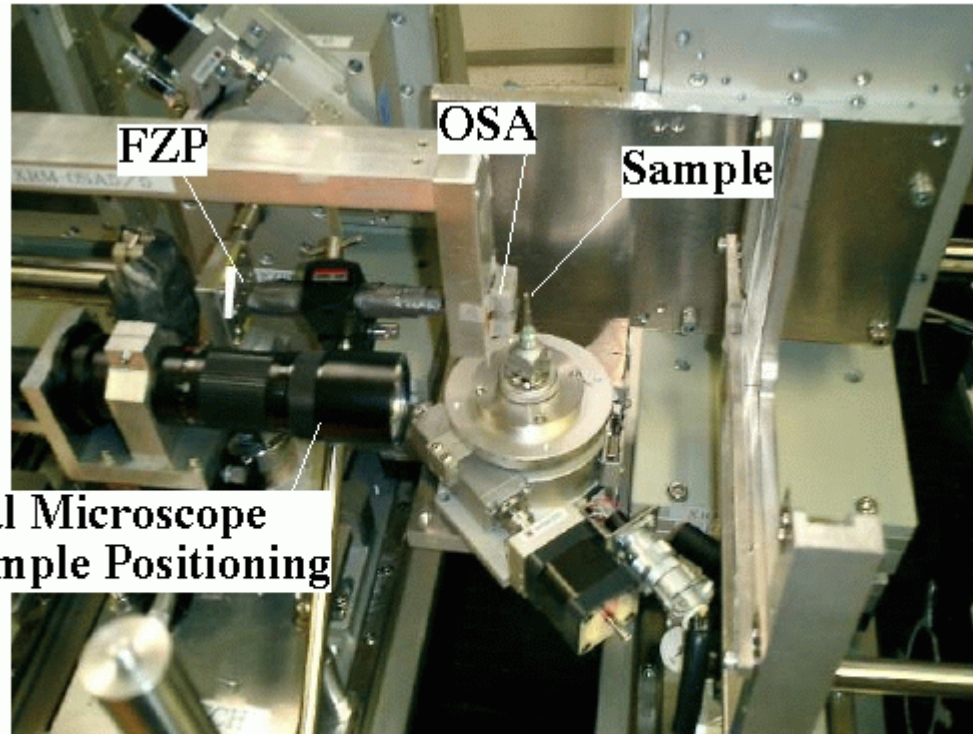
at BL20XU



Storage Ring Building

BL20B2

**BL20XU
250 m-long BL,
Undulator Source.**



**Optical Microscope
for Sample Positioning**

**Typical Setup for Microbeam Experiment
with Fresnel Zone Plate Optics**

Specification of Fresnel zone plate

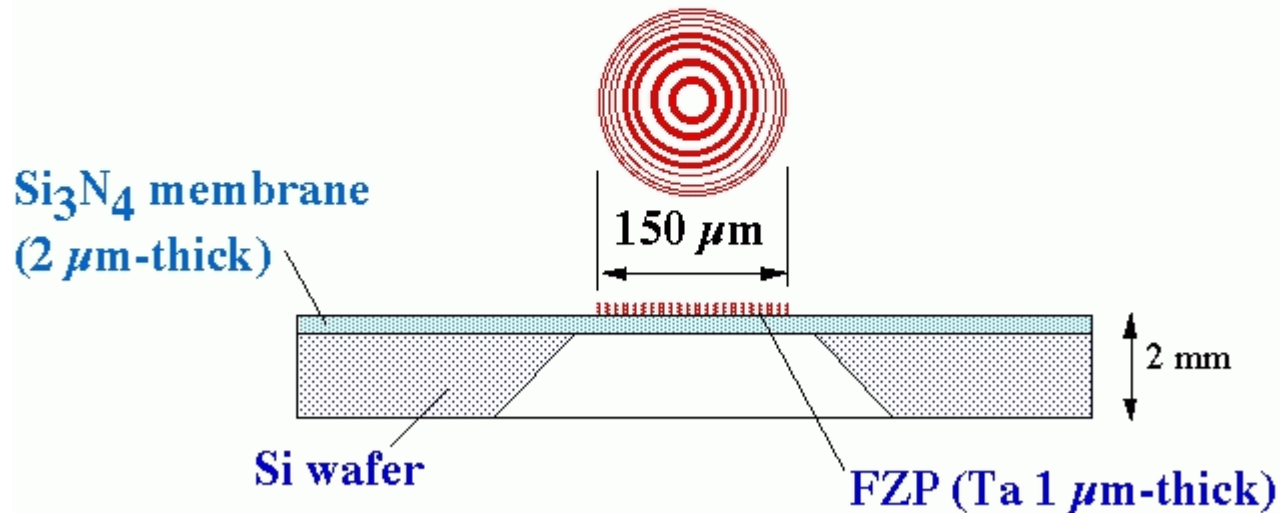
Diameter: 150 μm , Designed focal Length: 100 mm at 8 keV,
Outermost zone width (dN): 0.1 μm .

Diffraction limit ($=1.22d_N$): 0.12 μm , numerical aperture: 7.5×10^{-4} at 8 keV,

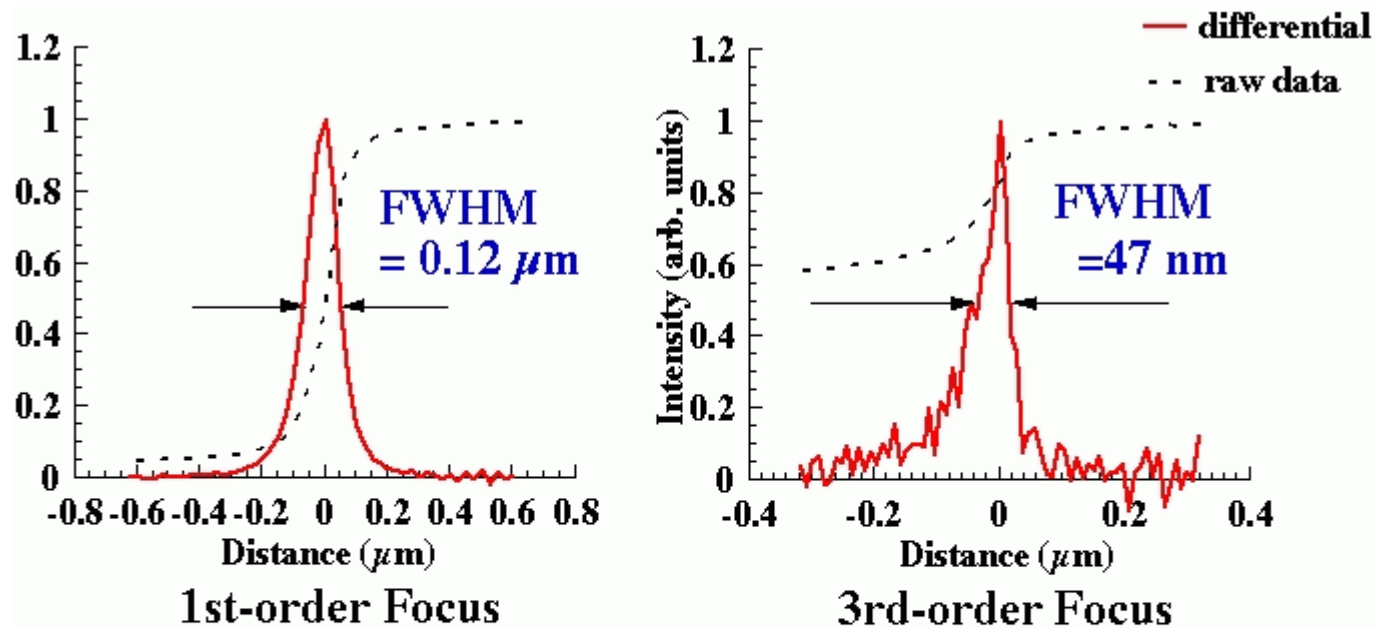
Zone material: Ta, 1 μm -thick,

Supporting membrane: Si_3N_4 , 2 μm -thick.

Fabrication method: electron-beam lithography technique at NTT-AT

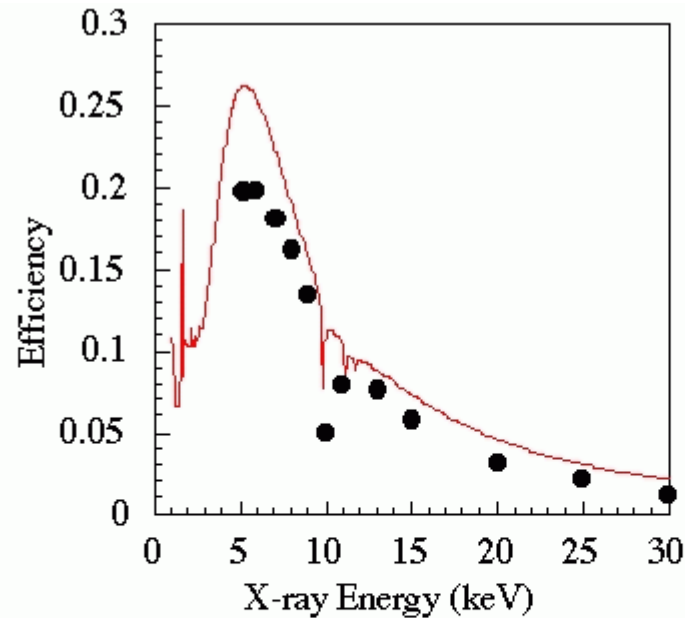


Schematic Drawing of Zone Plate Structure



Focused Beam Profile Measured by Knife-edge Scan

FZP: Ta $1 \mu\text{m}$ -thick,
Outermost Zone Width: $0.1 \mu\text{m}$,
EB-lithography at NTT-AT,
Focal Length: 100 mm @ **8 keV**.



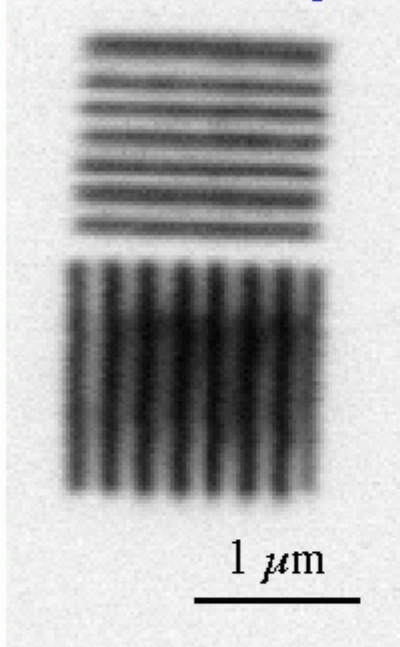
Diffraction Efficiency of Ta-FZP

Closed Circle: Experimental Results,

Solid Line: Calculated Efficiency assuming the Thickness of $1\mu\text{m}$.

Total flux of microbeam: 10^9 photons/s, Focused beam size: $0.12\mu\text{m}$.

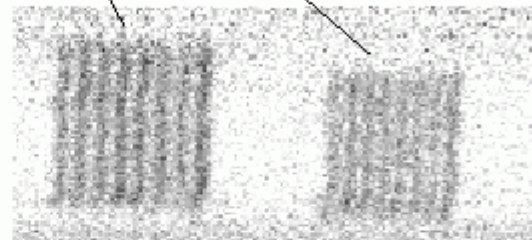
100 nm line&space



Test Patterns (Ta 500 nm-thick)
X-ray Energy: 8 keV,,
100 x 176 pixel,
25 nm/pixel, Dwell Time: 0.1 s.

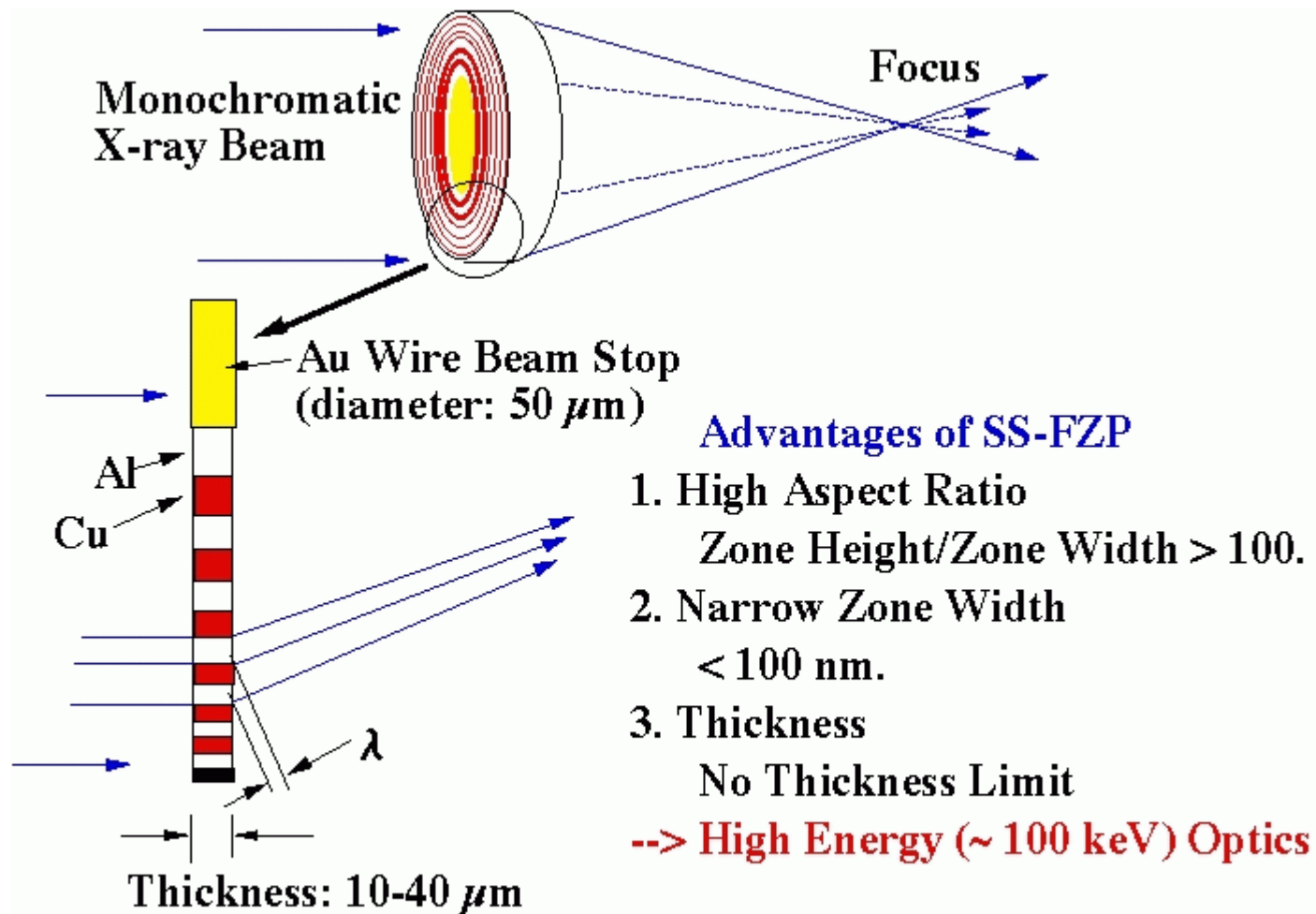
80 nm line & Space

70 nm line & Space

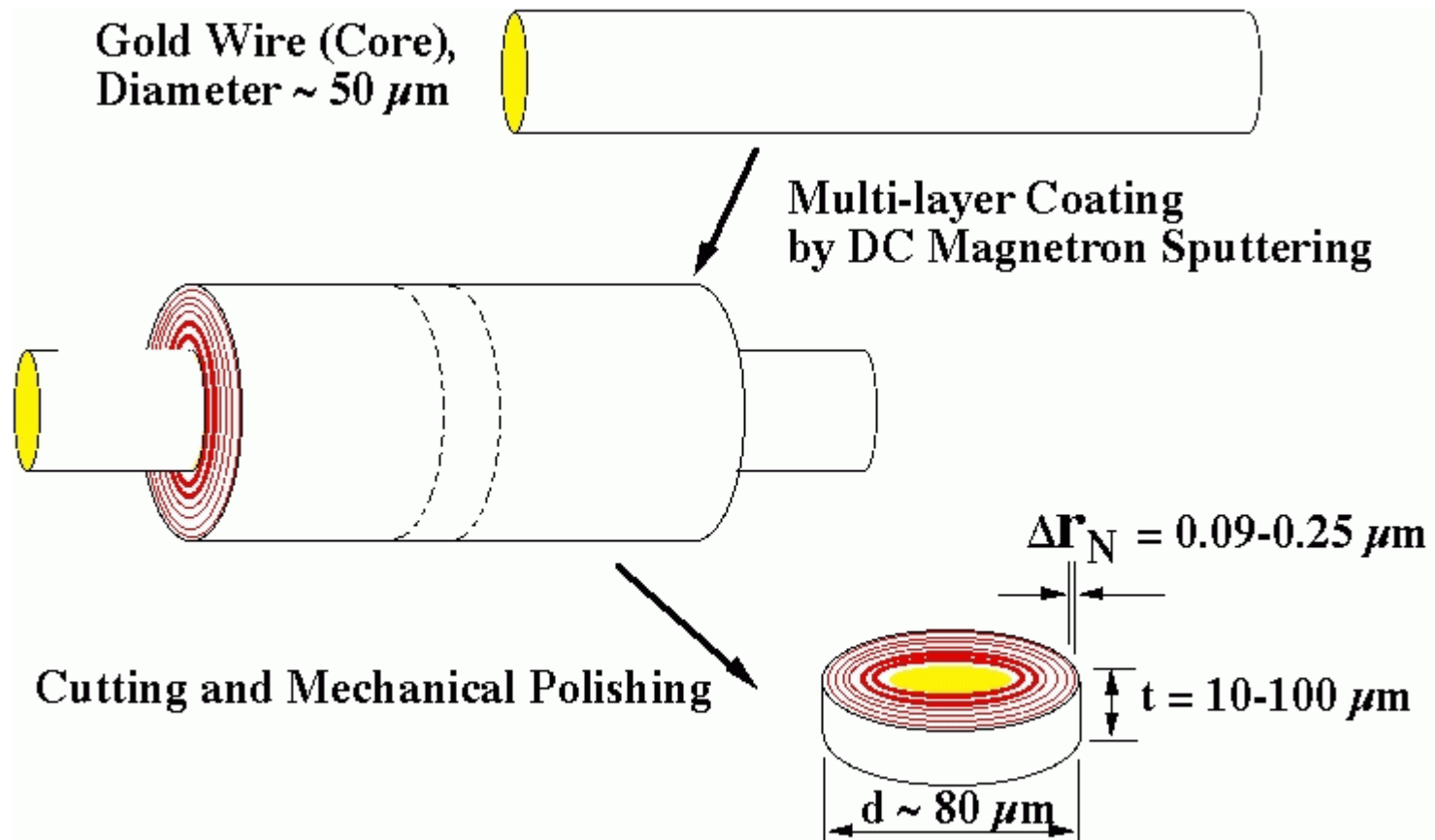


Test Patterns (Ta 100 nm-thick)
X-ray Energy: 10 keV,
160 x 80 pixels,
25 nm/pixel, Dwell Time: 0.2 s.

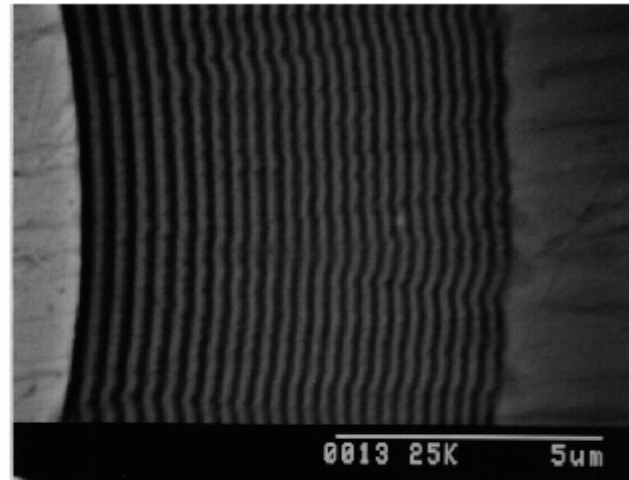
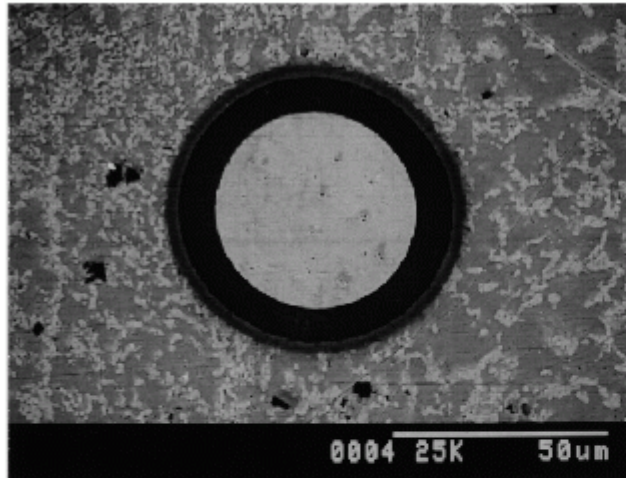
Scanning Microscopic Image of Resolution Test Pattern



Schematic View of Sputtered-sliced Zone Plate

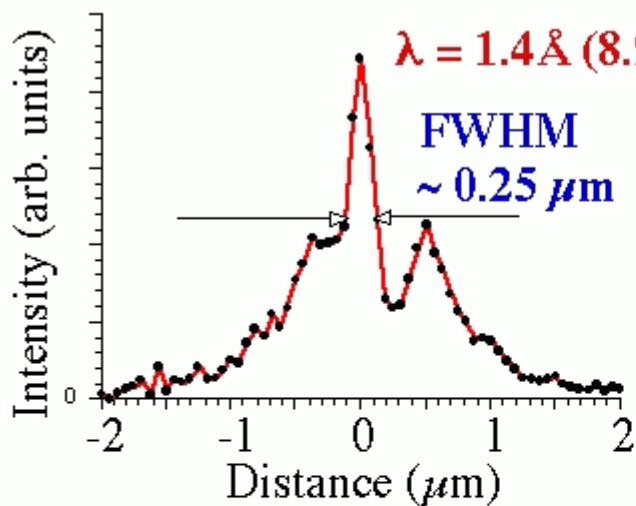


Fabrication Process of Sputtered-sliced Fresnel Zone Plates

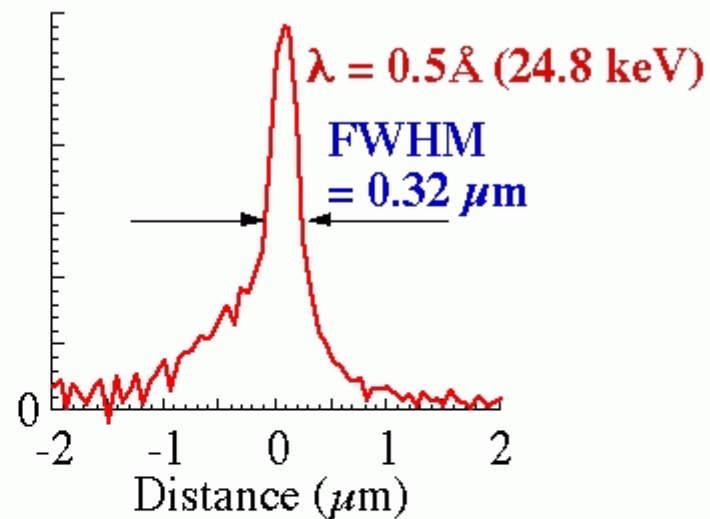


**SEM Image of
Sputtered-sliced Fresnel Zone Plate**

**Au Core (50 μm in diameter), Cu/Al 50 Layers,
Outermost zone width of 0.15 μm .**

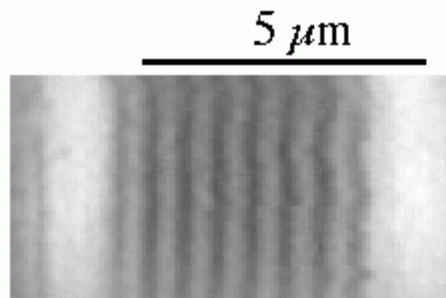


X-ray wavelength: 1.4 Å (8.9 keV), $f \sim 158 \text{ mm}$,
Cu/Al sputtered-sliced FZP (50 layers),
Core (beam stop): Au 50 μm in diameter,
Outermost zone width: 0.25 μm ,
Thickness: $\sim 20 \mu\text{m}$.
Diffraction efficiency: 25% @ 1.4 Å

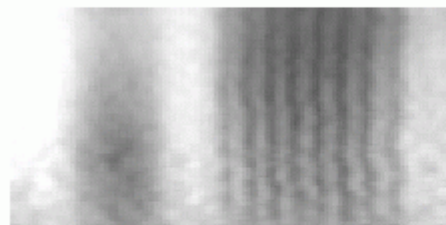


X-ray wavelength: 0.5 Å (24.8 keV), $f \sim 220 \text{ mm}$,
Cu/Al sputtered-sliced FZP (70 layers),
Core (beam stop): Au 100 μm in diameter,
Outermost zone width: 0.09 μm ,
Thickness: $\sim 60 \mu\text{m}$.
Sagittal Focus (1/4 of annular aperture)

Focused Beam Profile Measured by Edge-scan @BL20XU

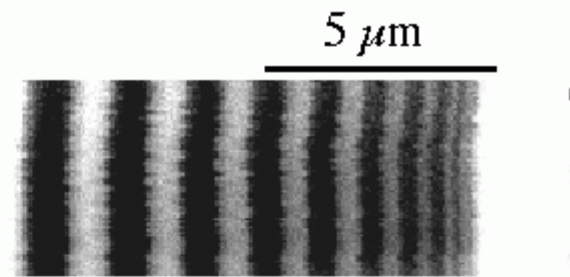


0.3 μm line & space



0.2 μm line & space

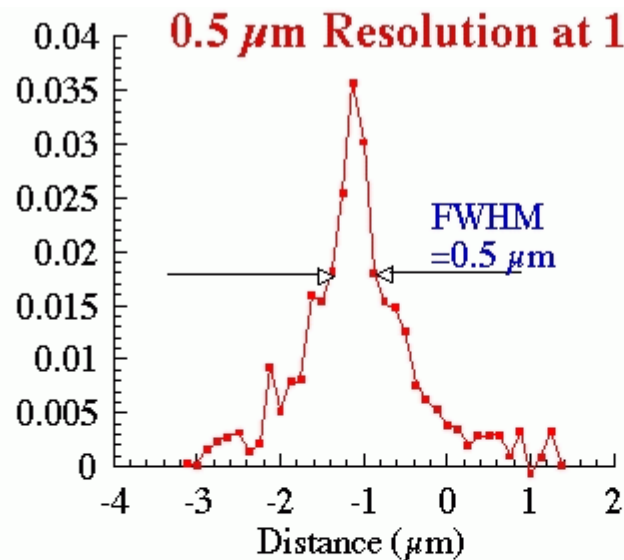
X-ray wavelength: 1.4 Å,
128 x 64 pixel,
0.0625 $\mu\text{m}/\text{pixel}$,
Dwell time: 0.4s/pixel.



0.1 μm line & space

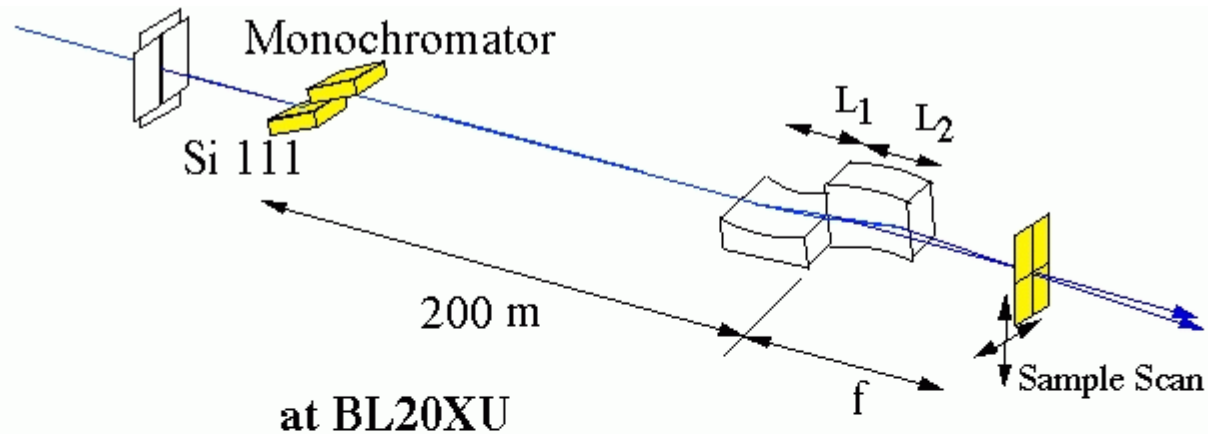
X-ray wavelength: 1.0 Å,
256 x 70 pixel,
0.0625 $\mu\text{m}/\text{pixel}$,
Dwell time: 0.4s/pixel.

Scanning Microscopic Image of Resolution Test Pattern



X-ray wavelength: 0.124 \AA (100 keV), $f \sim 900$ mm,
Cu/Al sputtered-sliced FZP (70 layers),
Core (beam stop): Au 50 μm in diameter,
Outermost zone width: 0.16 μm ,
Thickness: ~ 180 μm .

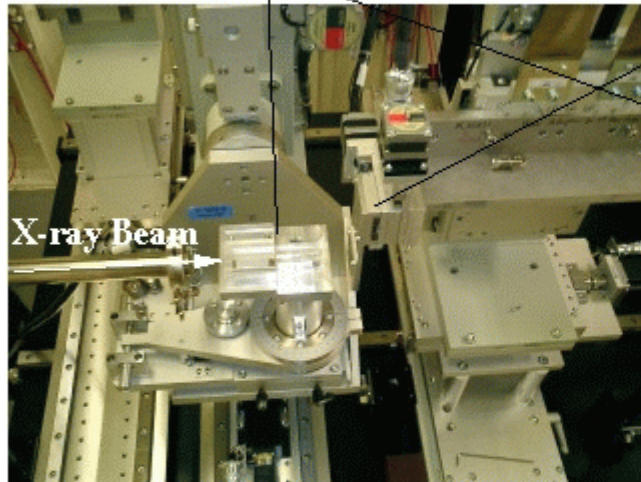
Focused Beam Profile Measured by Edge-scan @BL20XU



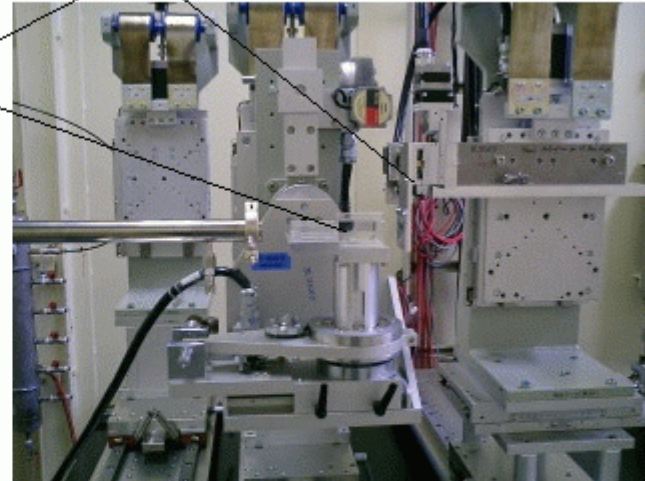
Experimental Setup of X-ray Microbeam/Scanning Microscopy with Total-reflection Mirror Optics (Kirkpatrick-Baez Configuration)

**Kirkpatrick-Baez Optics with Aspherical (Plane Parabola) Mirrors,
 L_1 : 45 mm, L_2 : 45 mm, f : 75 mm.
Glancing angle: 2.8 mrad,
Mirror material: SiO_2 (Pt-coat)**

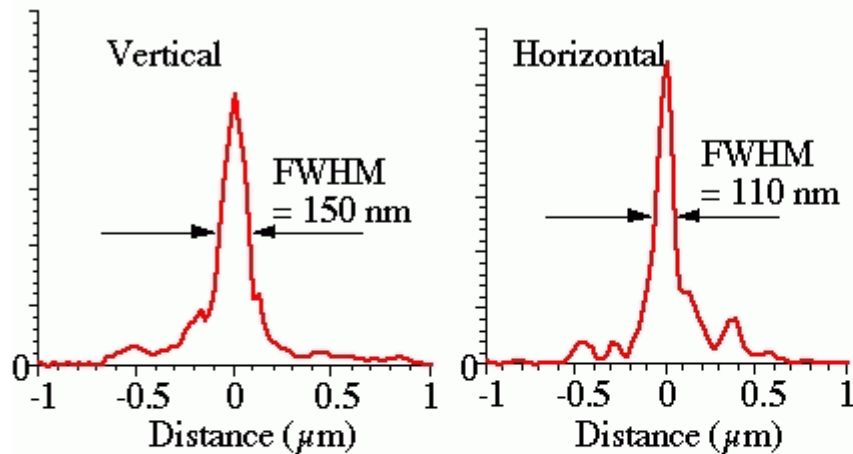
Mirrors



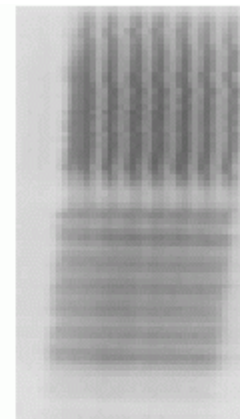
Sample Scanner



**Experimental Setup of Kirkpatrick-Baez Optics
at BL20XU**



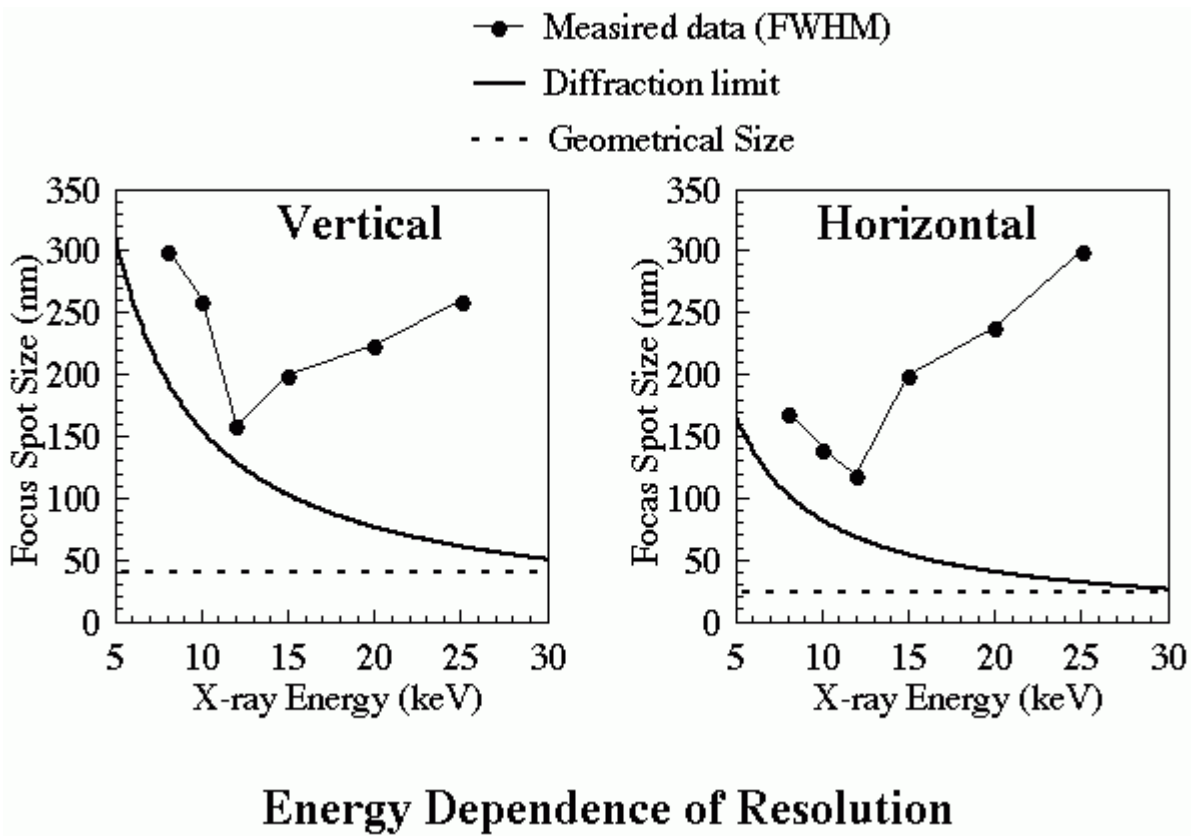
Focused Beam Profiles
measured by Knife-edge Scan
X-ray Energy: 12 keV

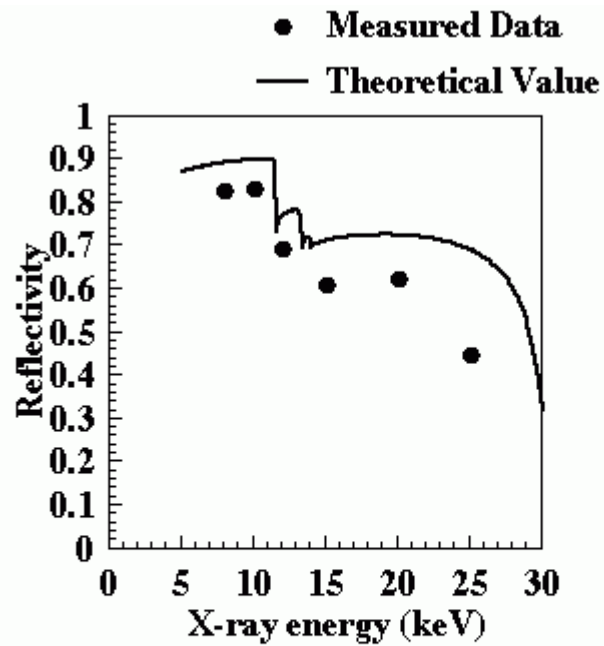


Scanning Microscopy Image
of Test Patterns

0.1 μm line & space

**Microbeam and Scanning Microscopy
with Total-reflection Mirror Optics**





Reflectivity of Total Reflection Mirros

Pt surface,
Glancing angle: 2.8 mrad .

A (possible) Way to Nonometer Probe at Hard X-ray Region

Present Status:

- ~ 50 nm with FZP,**
- ~ 100 nm with SS-FZP,**
- ~ 100 nm with total-reflection mirror.**

Applications:

- Semiconductor (quantum) devices,**
- Micro-crystal analysis,**
- Biology, Medicine, Material Science, etc.**

FZP: manufacturing limit of EB lithography ~ 30 nm?

**SS-FZP: no-limit for zone width and aspect ratio,
but control of zone structure may be problem.**

Mirror: fabrication and stability may be problem.

Theoretical limit ~ 10 nm (for simple optics)

Other problems for nanometer probe

- 1. Stabilities:
temperature, vibration, air turburance, etc.**
- 2. Radiation damage:
cryo-microscopy, phase-contrast.**
- 3. Evaluation of probe size (in higher energy region).**
- 4. No standard specimen for resolution test.**
- 5. Sample handling.**