Hard X-ray Microscopy
-Present Status at SPring-8 & Opportunities in ERL-

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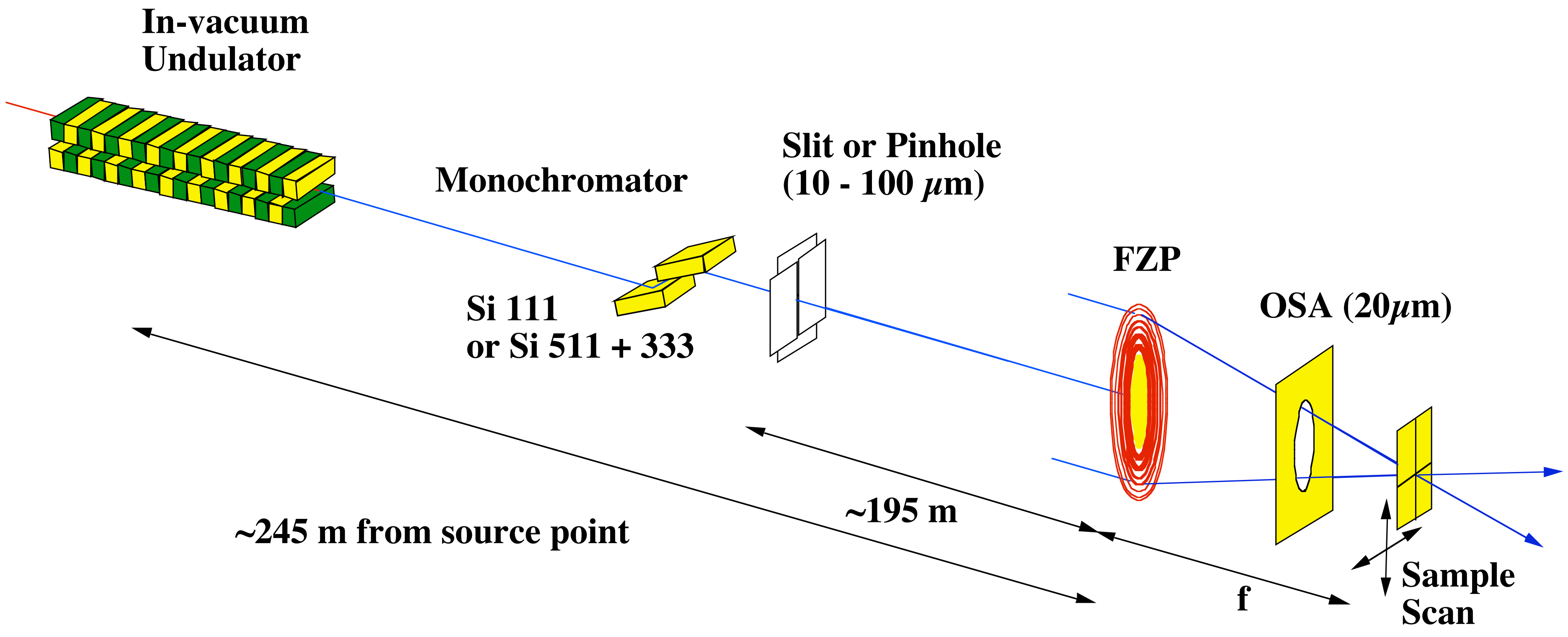
JASRI/SPring-8

1. Optical devices,
FZP, mirror, refractive lens, etc,

2. Optical systems for hard X-ray microscopy,
Imaging microscopy, scanning microscopy, holography, with some applications,

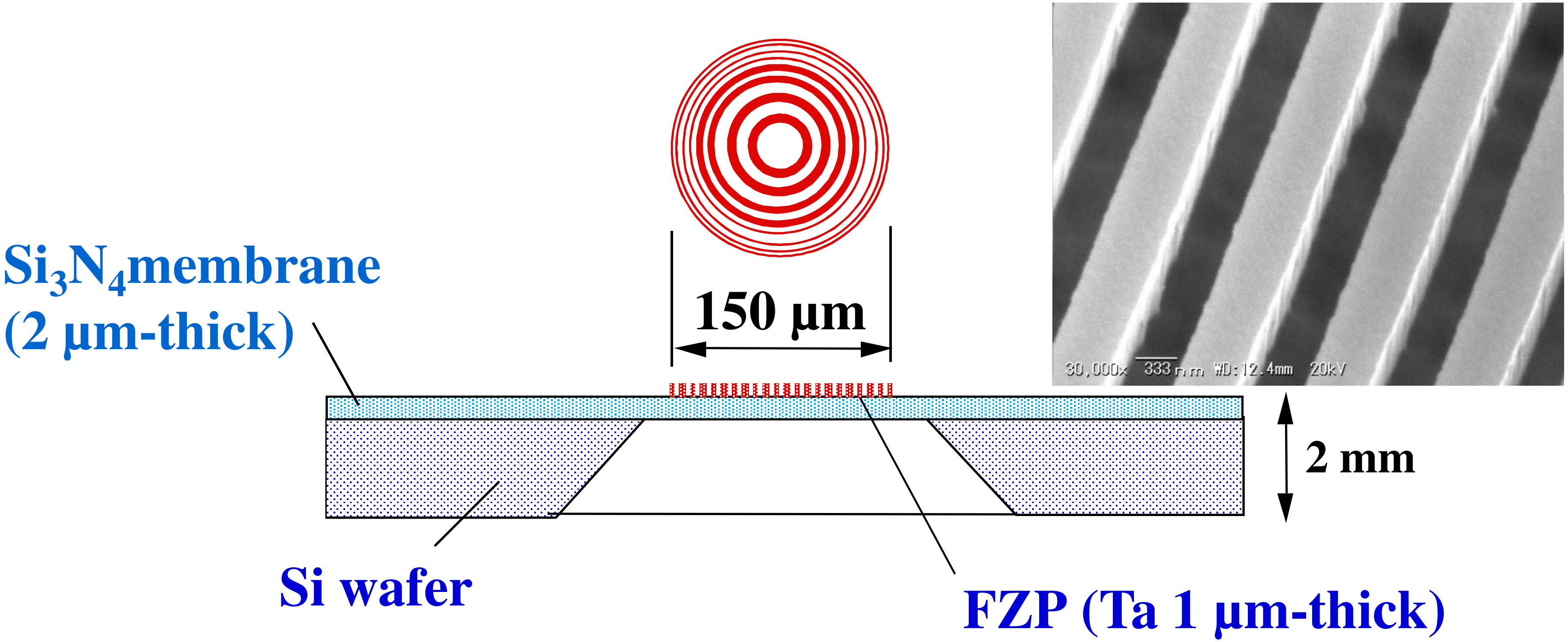
3. Scientific opportunities in ERL
Use of fully coherent beam,
Use of quasi-monochromatic beam,
Speckle noise reduction & spatial coherence preservation.



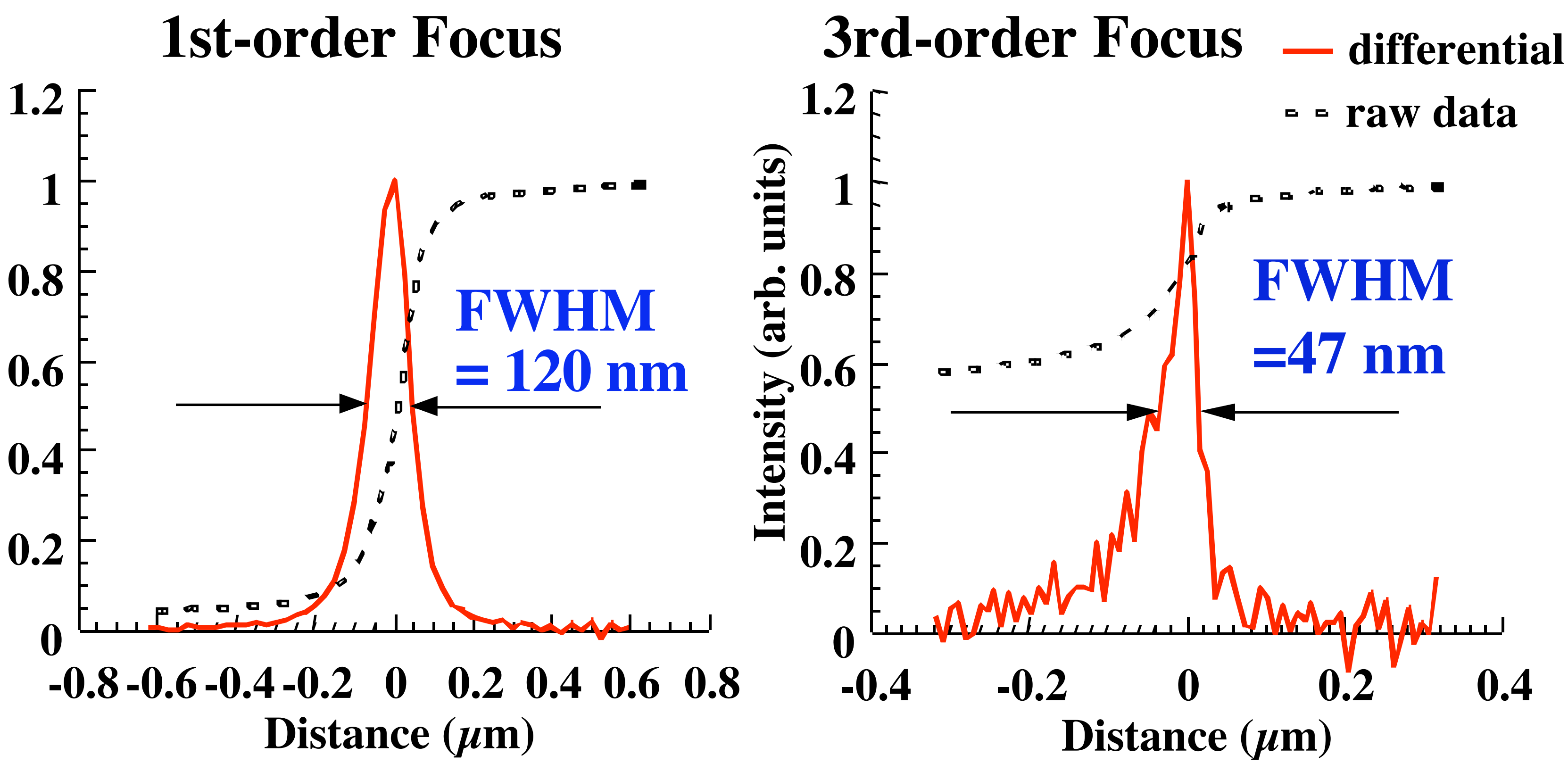
Experimental Setup of X-ray Microbeam/Scanning Microscopy at BL20XU

Specification of Fresnel zone plate

Diameter: 150 μm, Designed focal Length: 100 mm at 8 keV,
Outermost zone width (d_N): 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 , or SiC, 2 μm-thick.
Fabrication method: electron-beam lithography technique at NTT-AT

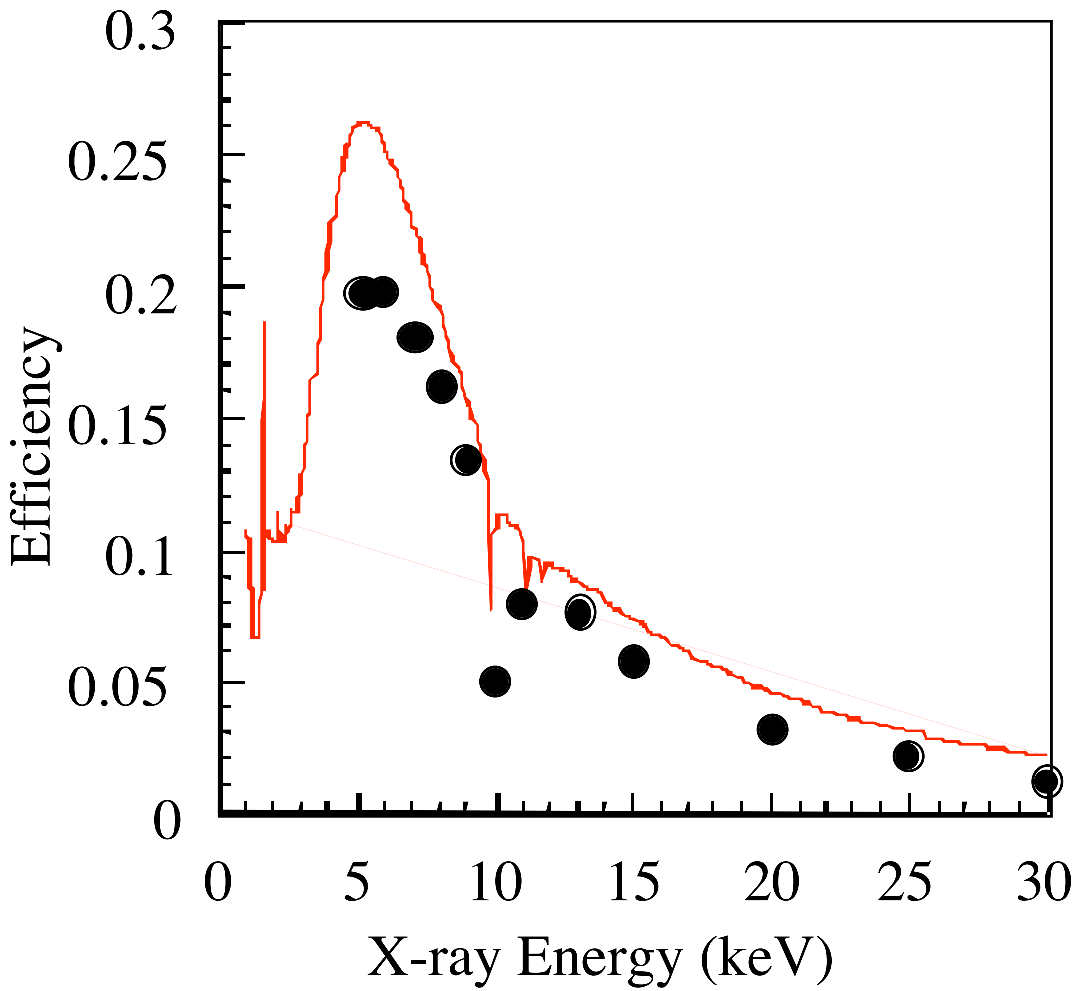


Schematic Drawing of Zone Plate Structure



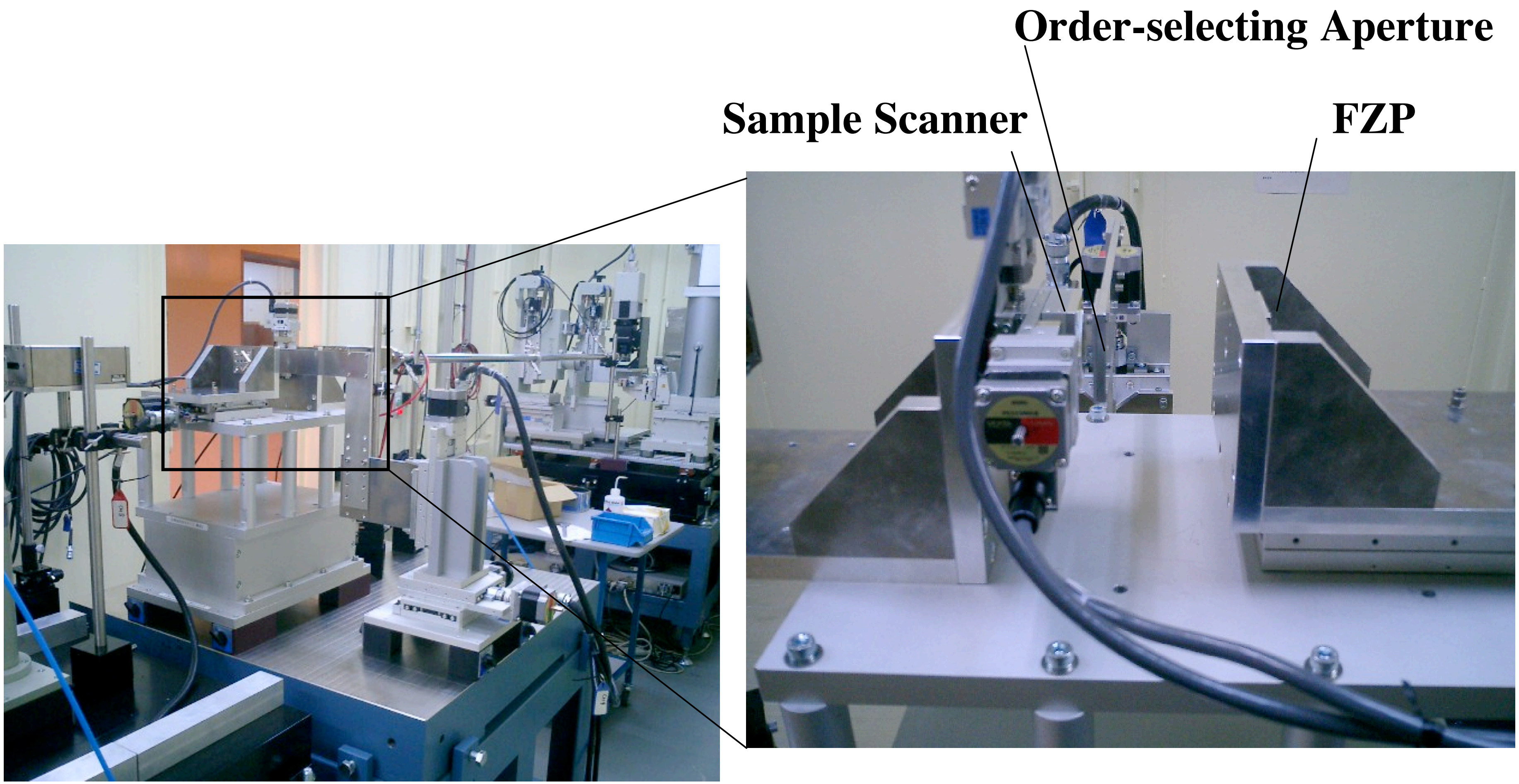
Focused Beam Profile Measured by Knife-edge Scan

FZP: Ta 1 μm-thick,
Outermost Zone Width: 0.1 μm,
Diameter: 155 μm,
EB-lithography at NTT-AT,
Focal Length: 100 mm @8 keV.



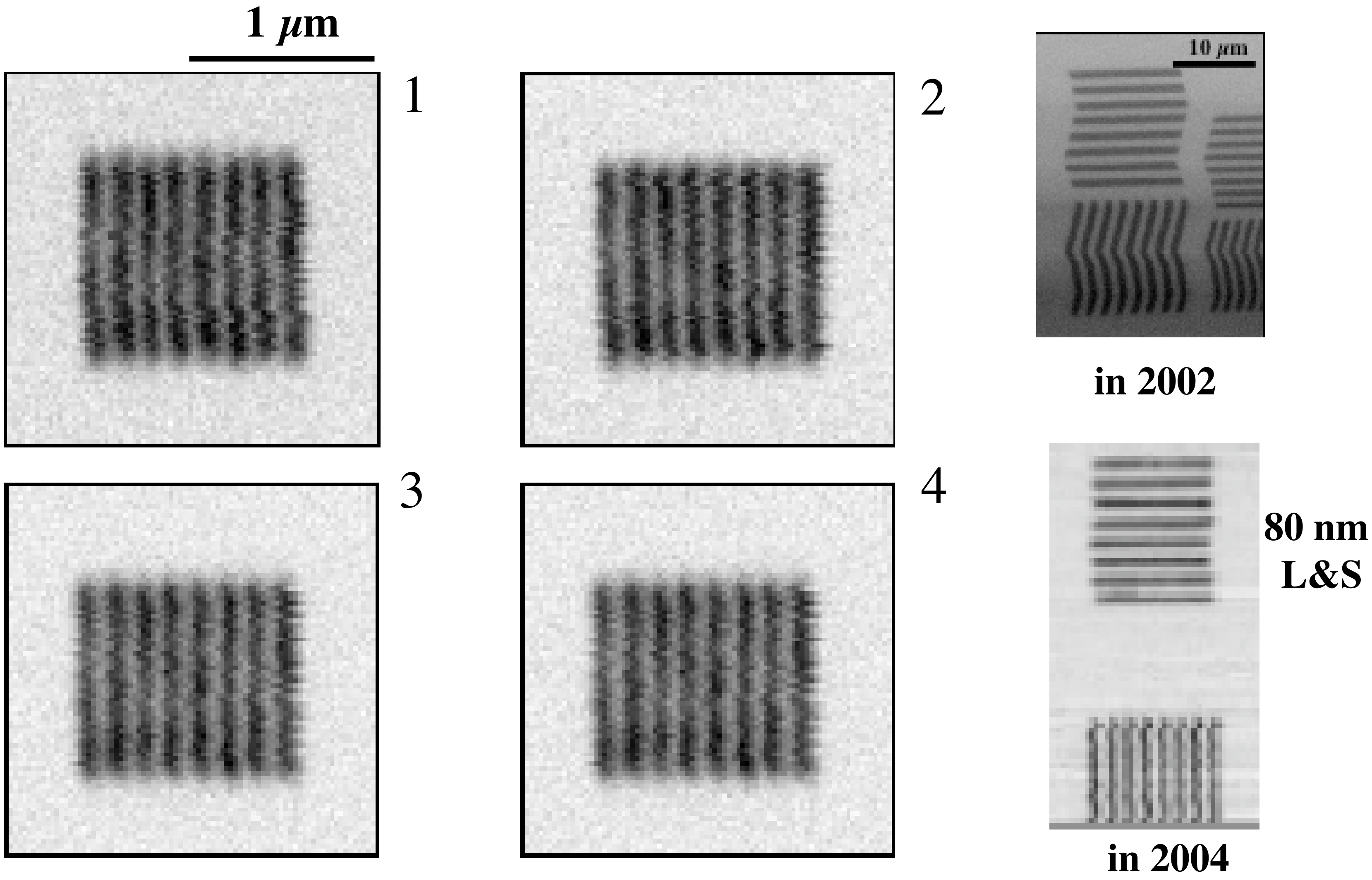
Diffraction Efficiency of Ta-FZP

Closed Circle: Experimental Results,
Solid Line: Calculated Efficiency assuming the Thickness of 1μm.



Stable Nano-probe

Experimental Setup of Scanning Microscopy with Fresnel Zone Plate Optics at BL20XU SPring-8

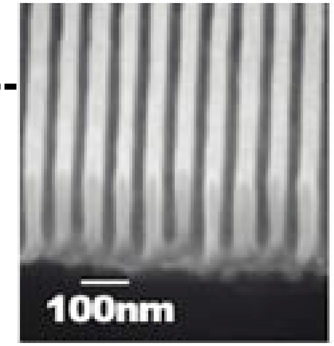


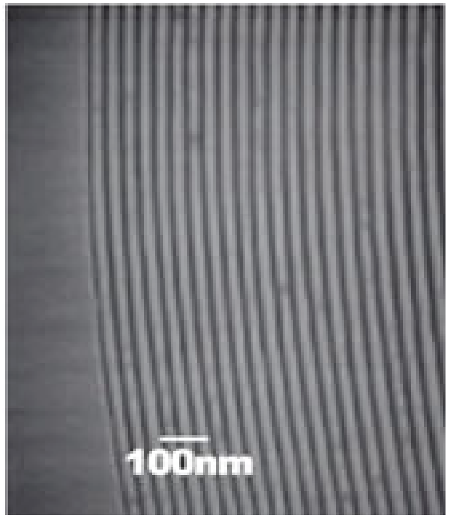
Resolution and Stability of Scanning Microscope

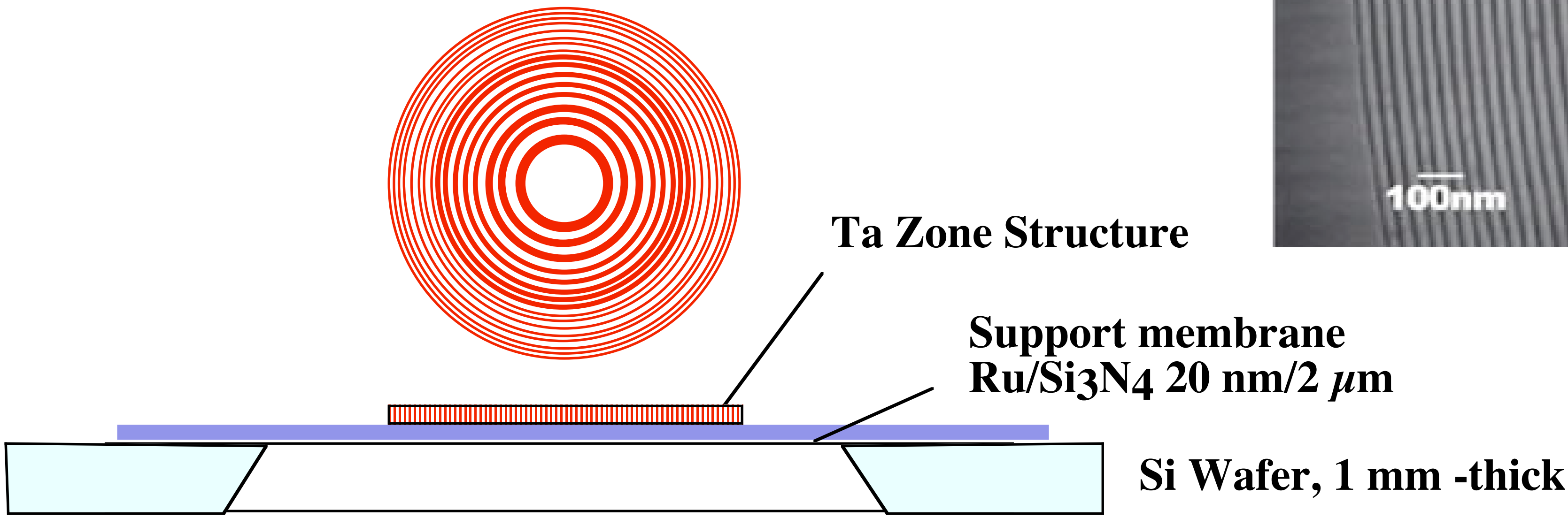
Probe size: 120 nm, 80 nm line/space test patterns,
80 x 80 pixels, 25 nm pixel size, 0.1 s dwell time, 8 keV.

Specification of Fresnel zone plate

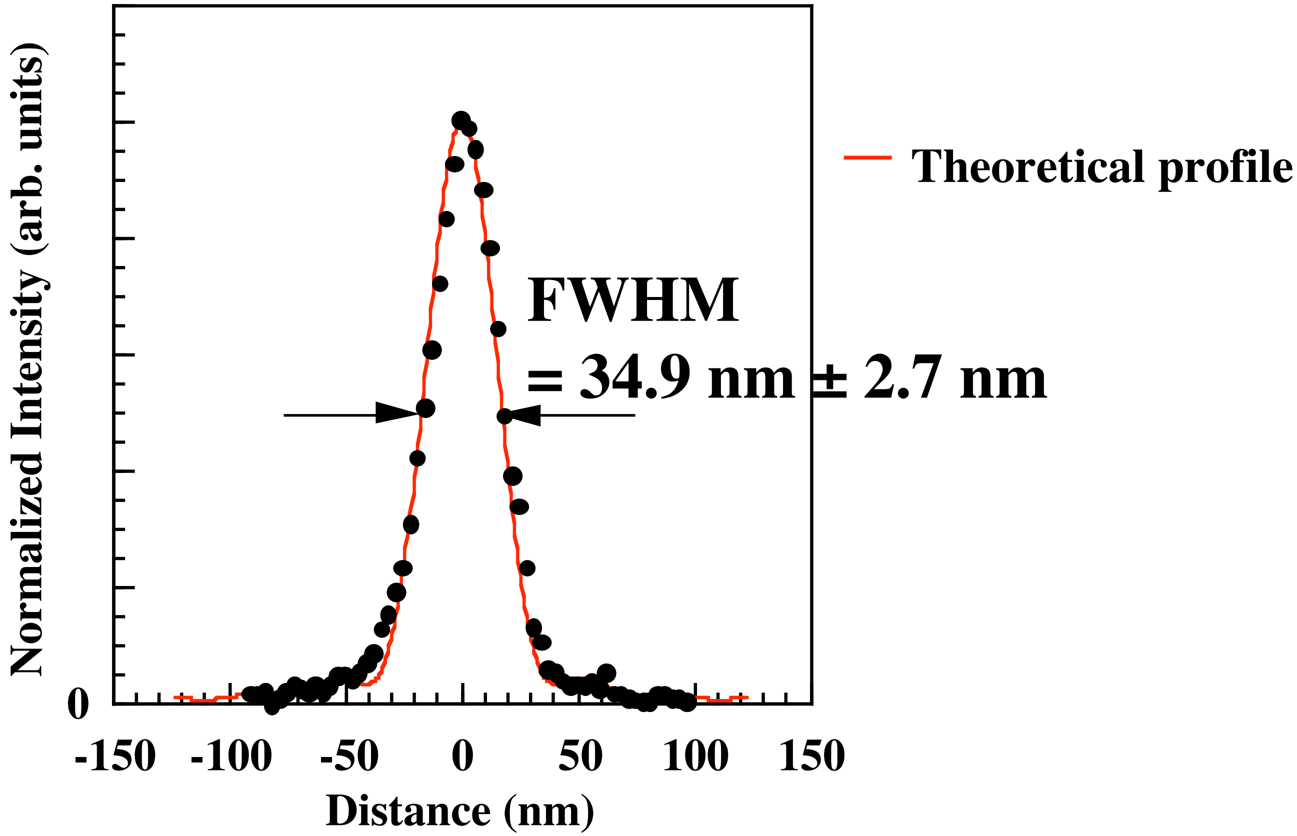
Diameter: 177 μm, Designed focal length: 40 mm at 8 keV,
Outermost zone width (Δr_N): 35 nm.
Diffraction limit ($=1.22\Delta r_N$): 43 nm (Rayleigh's criterion),
Numerical aperture: 2.2×10^{-3} at 8 keV,
Number of total zone: 1265,
Zone material: Ta, 0.2 μm-thick (0.18 μm),
Supporting membrane: Si_3N_4, 2 μm-thick.
Fabrication method: electron-beam lithography at NTT-AT







FZP with 35 nm outermost zone width



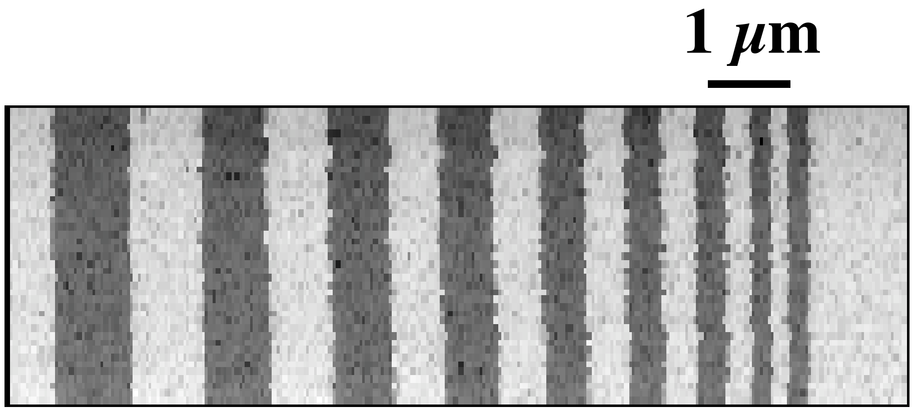
Focused Beam Profile Measured by Dark-field Knife-edge Scan

FZP: 35 nm Outermost Zone Width (Δr_N = 35 nm),
X-ray Energy: 8 keV,
Dark-field Edge-scan Method,
Knife-edge: Ta 0.5 μm-thick.

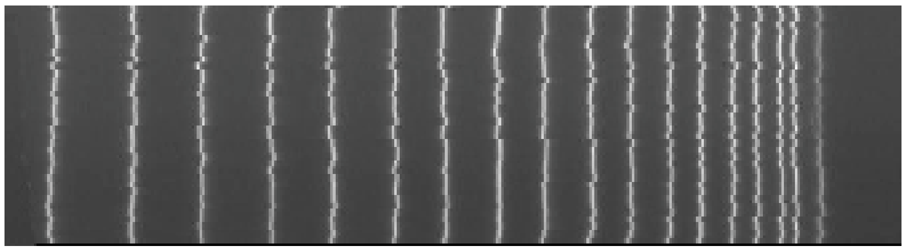
Diffraction Limit = 1.22 Δr_N (Rayleigh's criterion)

Line Spread Function = 1.00 Δr_N (Full-width at Half-maximum)

Y. Suzuki, et al.
X-Ray Optics and Instrumentation (2010) 824387.

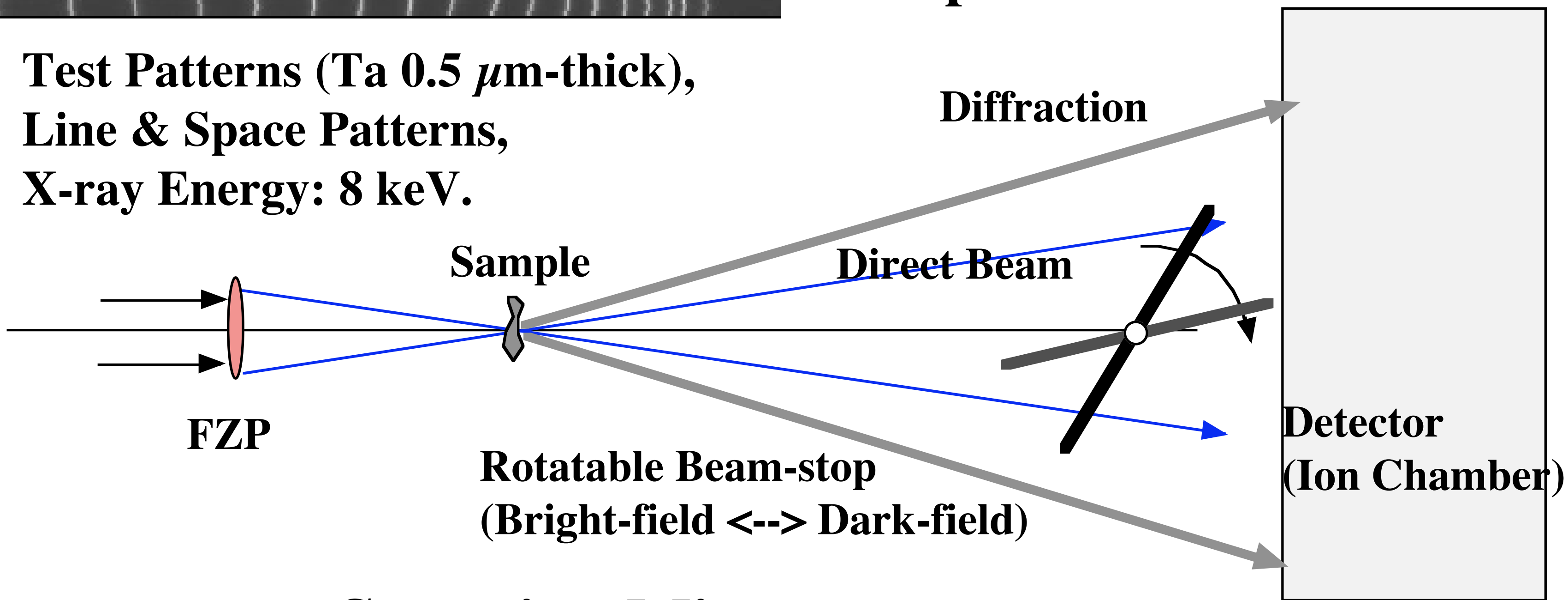


Bright Field
50 nm step,
0.2 s/pixel.

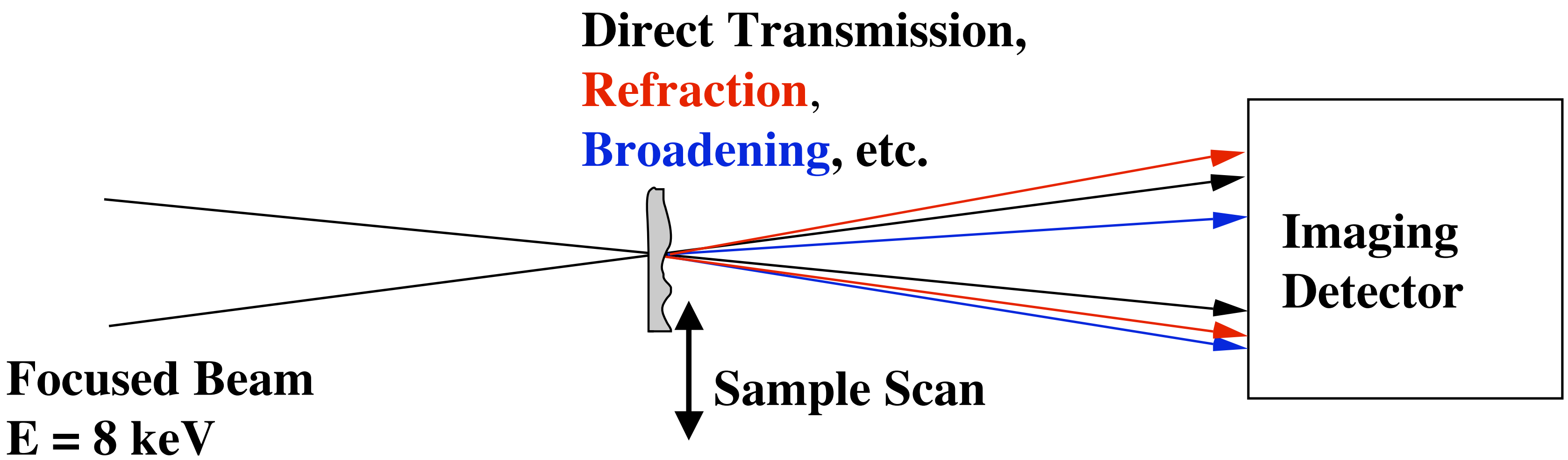


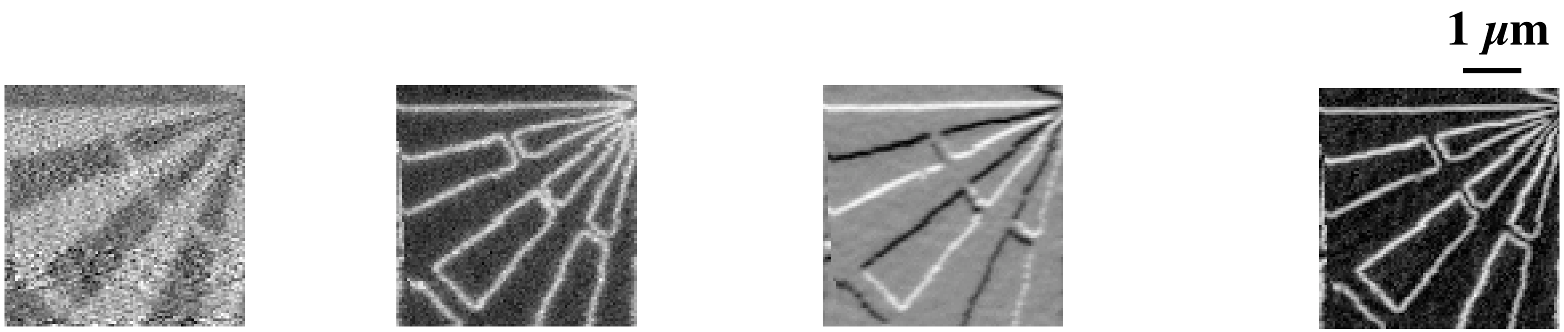
Dark Field
12.5 nm step,
0.2 s/pixel.

Test Patterns (Ta 0.5 μm-thick),
Line & Space Patterns,
X-ray Energy: 8 keV.



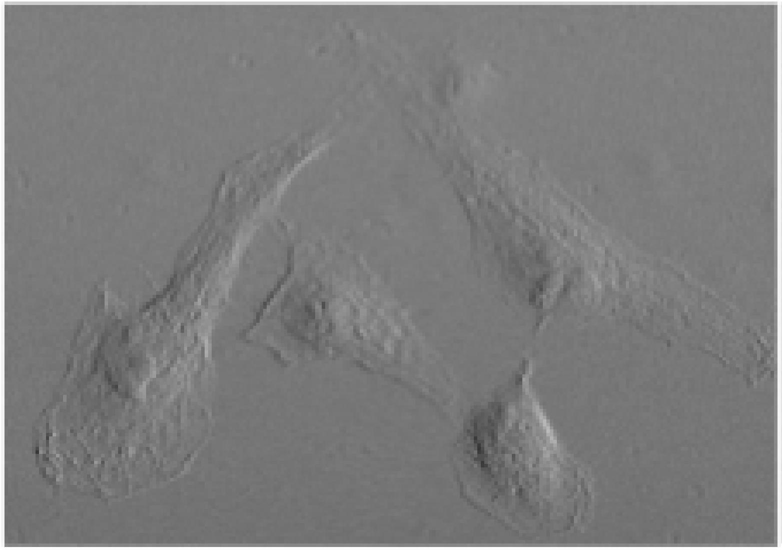
Scanning Microscopy



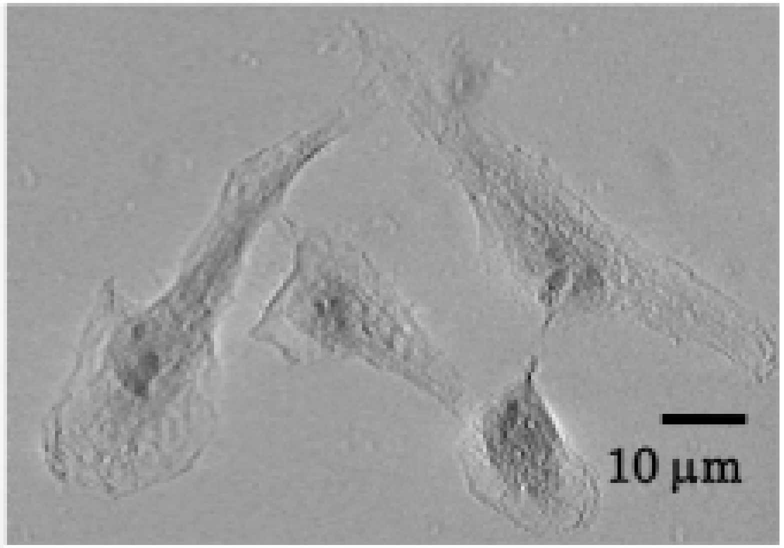


Bright Field | **Dark Field** | **1st Order Moment (Differential-phase)** | **1st Order Moment, (Absolute Value)**

Differential-phase-contrast Imaging in Scanning Microscopy



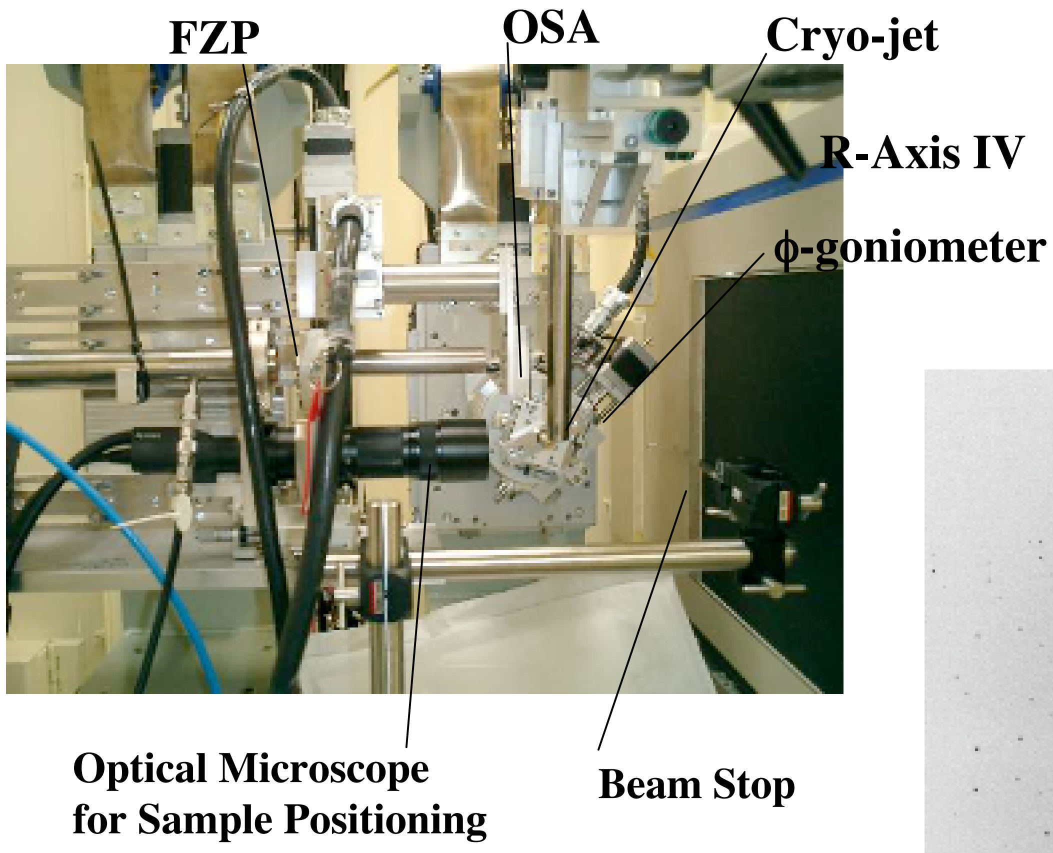
Differential Phase-contrast

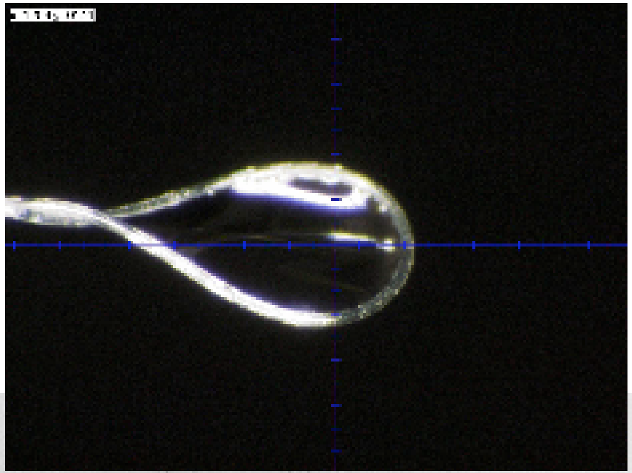


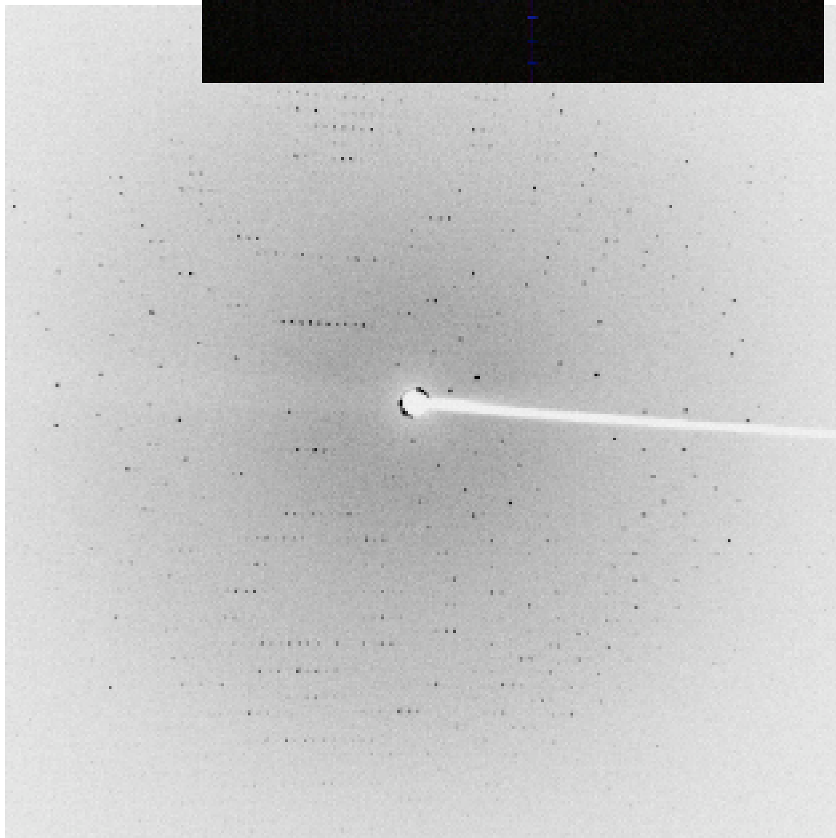
Reconstructed Phase-image

Scanning differential phase-contrast imaging

Sample; HeLa Cell, Dried,
X-ray Energy: 8 keV, Probe size: ~100 nm,
50 nm step, 2000 x 1600 pixel,
Total scan time: ~ 4 h.

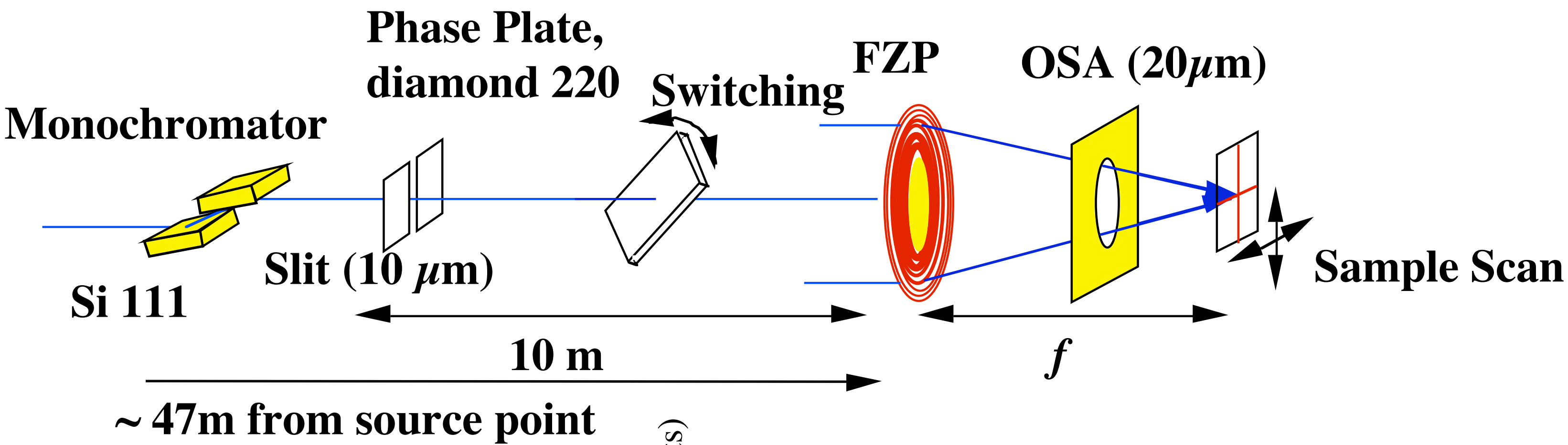


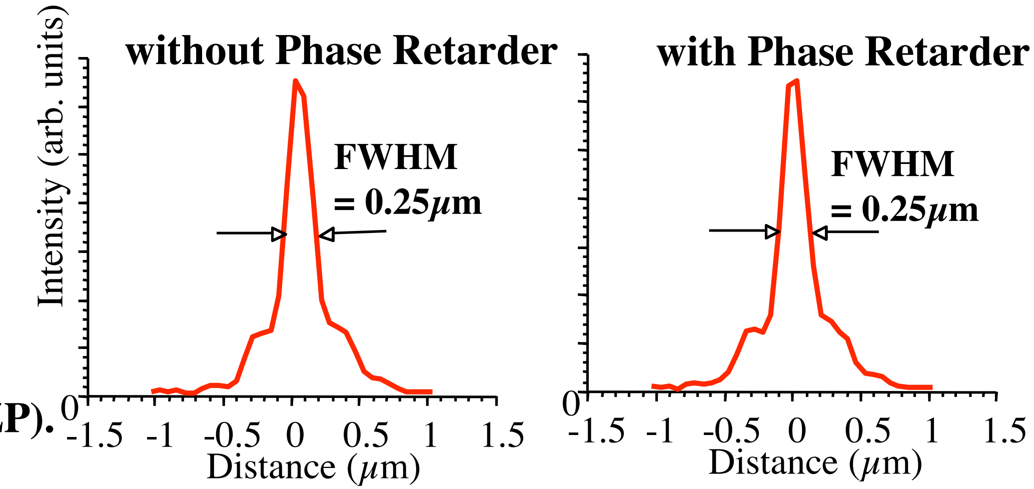




Beamsize: 0.25 μm,
Energy: 9.85 keV,
Oscillation camera method,
Sample: Thermolysin, ~ 10 - 20 μm.

Micro-beam Diffraction with FZP Optics
and Imaging Plate Detector (RIGAKU R-Axis IV) @ BL20XU

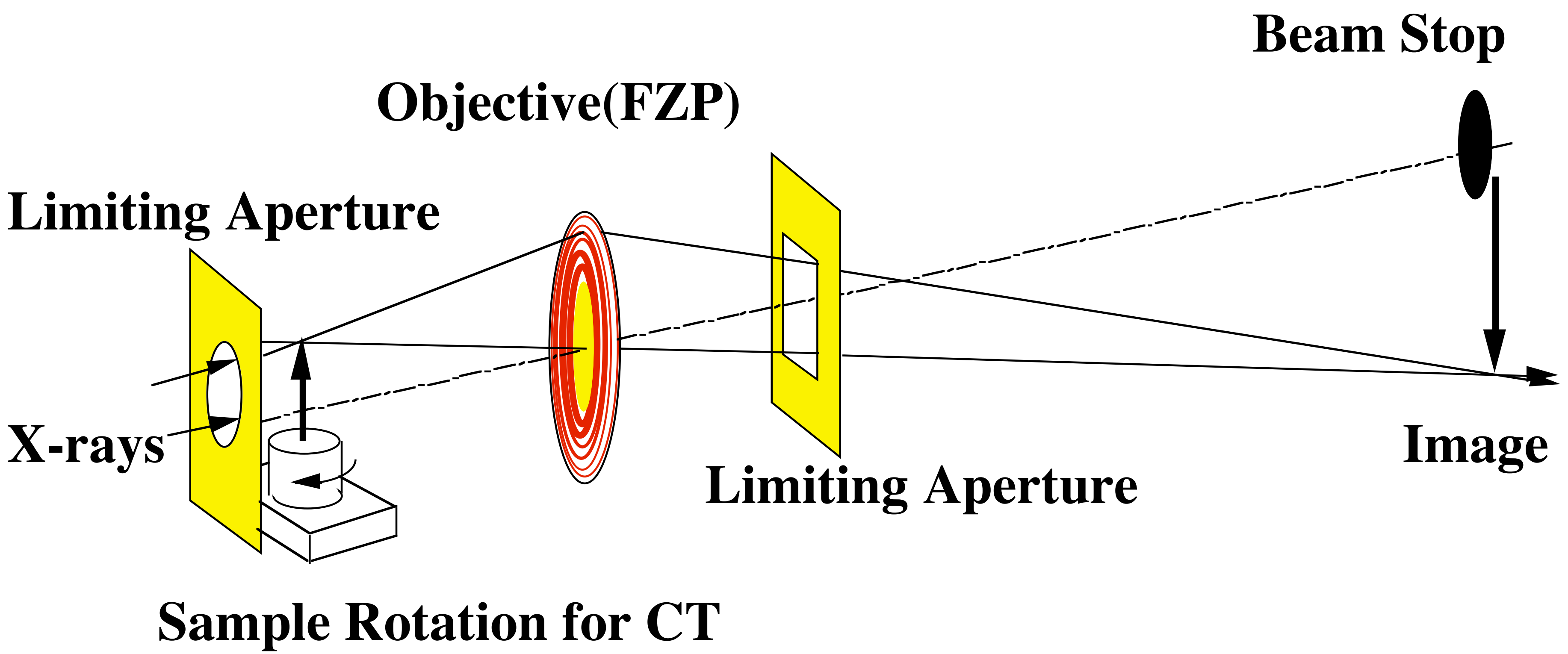




OSA: order selecting aperture,
***f* ~ 142 mm @7.1 keV,**
Source size:
~ 40 µm (vertical, 47 m from FZP),
~ 10 µm Slit (horizontal, 10 m from FZP).
FZP Material: Ta, 1µm-thick,
Outermost zone width: 0.25 µm,
Diameter: 100 µm,
Center Stop: 50 µm-diameter.

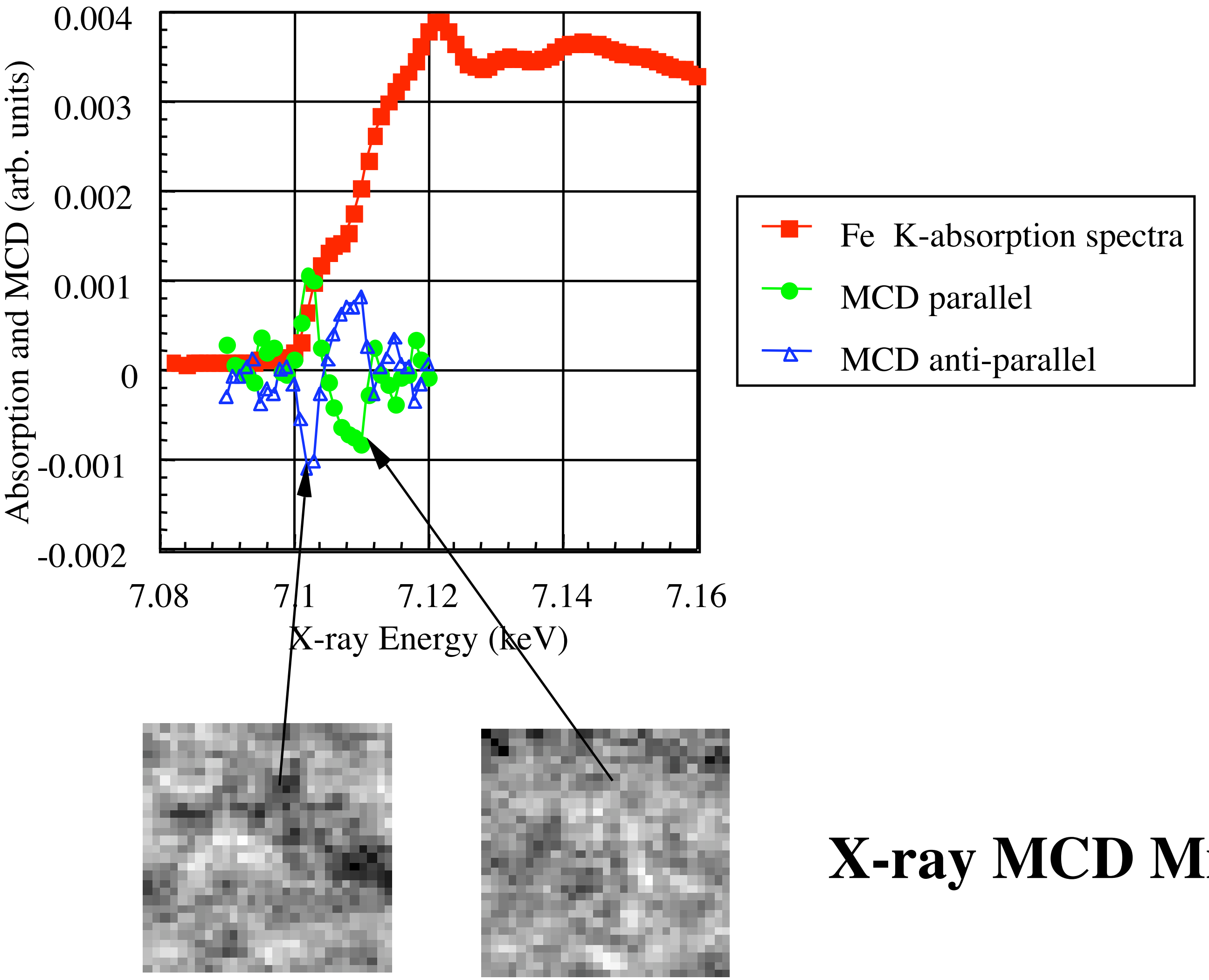
Focused Beam Profile
measured by Knife-edge Scan @7.1 keV

Circularly Polarized X-ray Microbeam with Fresnel Zone Plate at BL47XU



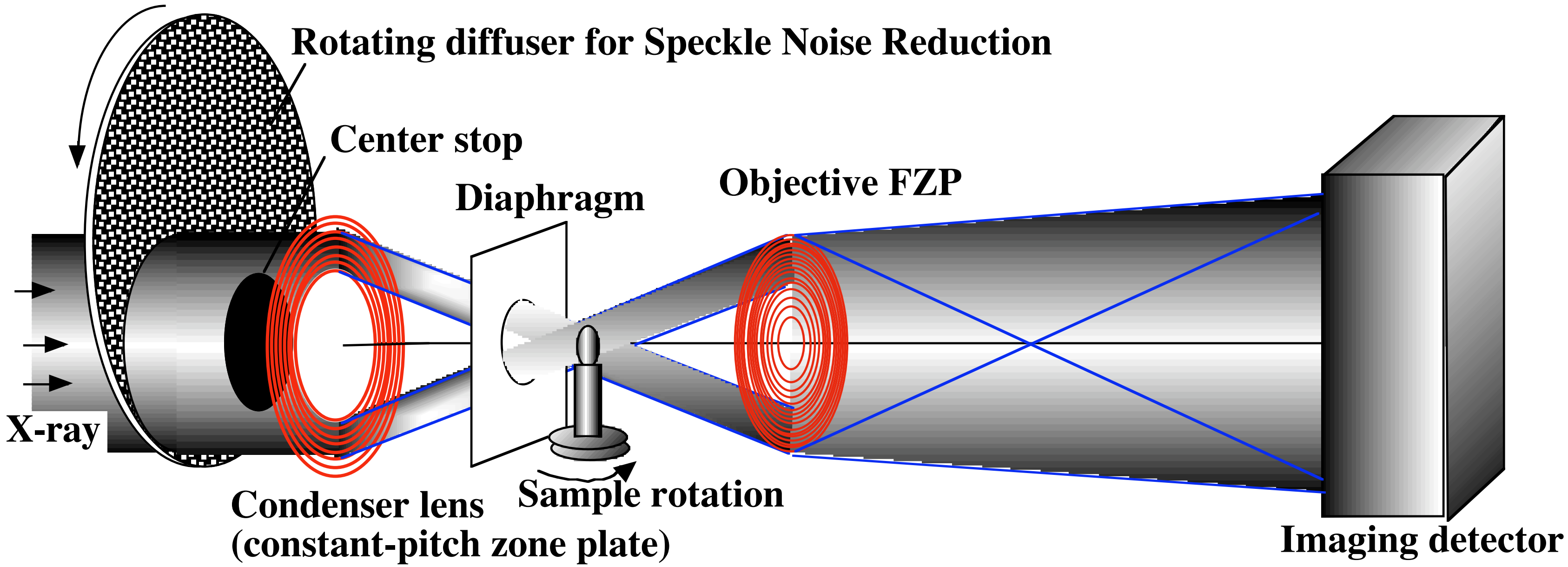
Schematic Diagram Experimental Setup
for Imaging Microscopy and Micro-tomography

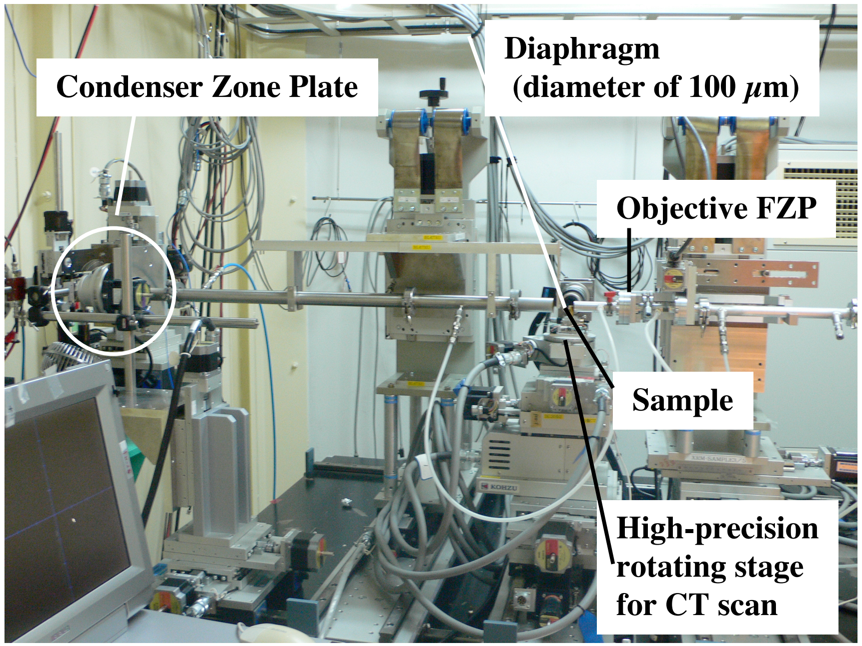
X-ray MCD Microscopy

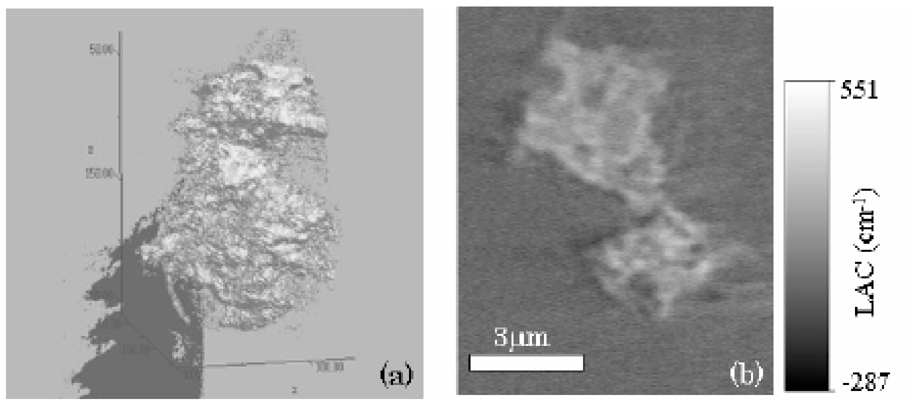


Magnetization Image

Sample: pure Fe foil (5 μm thick),
Image size: 15 μm x 15 μm.

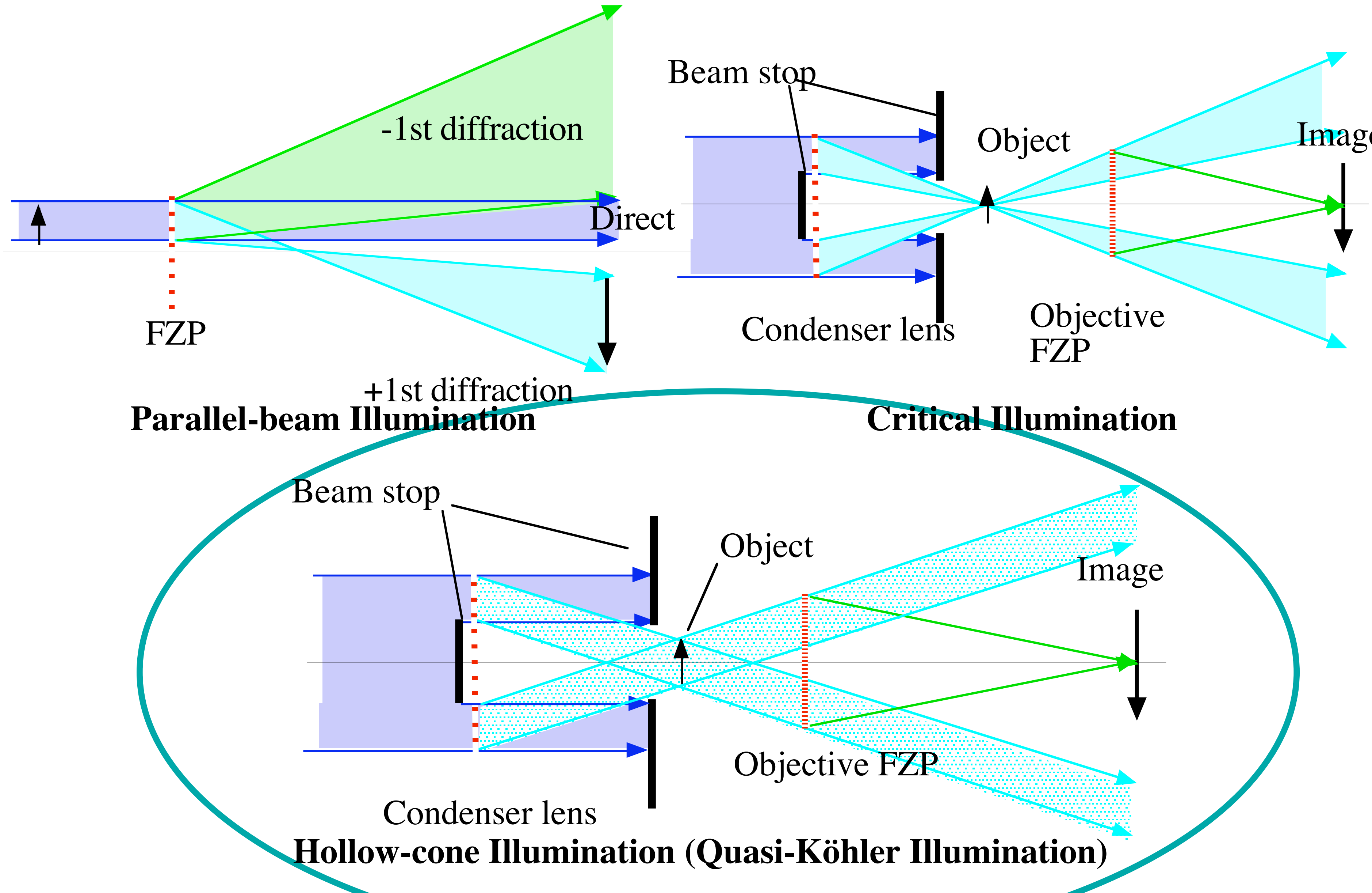






CT image of IDP (L2008D3 #17). (a) three dimensional reconstruction from CT images. (b) sagital slice derived from three dimensional reconstructed image.

Imaging Microscopy/tomography at BL47XU



Various Illumination Optics for X-ray Imaging Microscopy

Illumination Optics for Imaging Microscopy with Low-emittance Synchrotron Radiation Sources

Parallel beam illumination.

--> Edge-enhancement artifact,
Strong speckle noise,
Diffraction artifacts for crystalline samples,
(c.f. diffraction is sometimes signal)

↓

Partial coherent illumination by diffuser.

--> Less artifacts, and less speckles,
Weak edge-enhancement,
Nonuniform imaging properties in the field of view,
Asymmetric feature of imaging properties.
(off-axial illumination)

↓

Need of condenser optics for imaging microscopy.

Low-emittance SR source is not suitable for imaging microscopy, because of

high spatial coherence:

Small source size (~ 10 μm vertical x 100 μm horizontal),
Small divergent angle (~ 10 μrad).

Critical illumination with simple condenser lens:

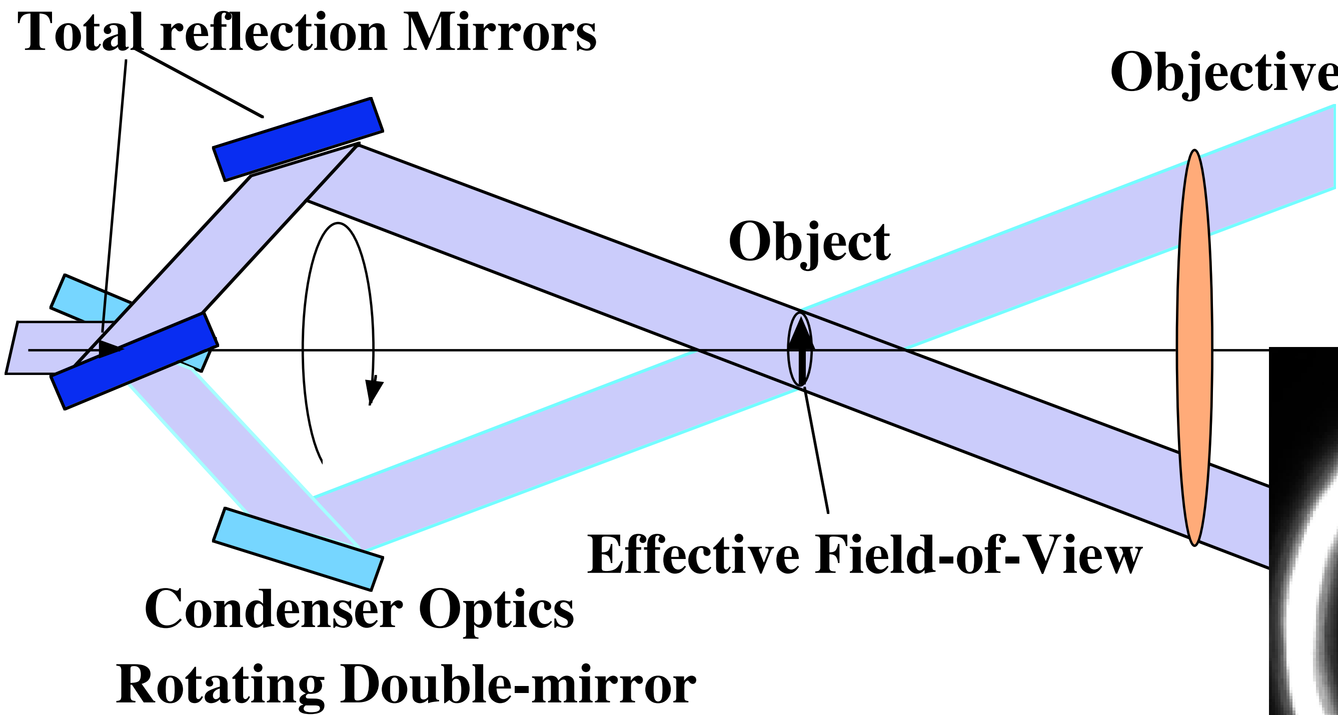
F-number matching --> small field of view (< a few μm),
Coherent illumination --> Speckle noise.

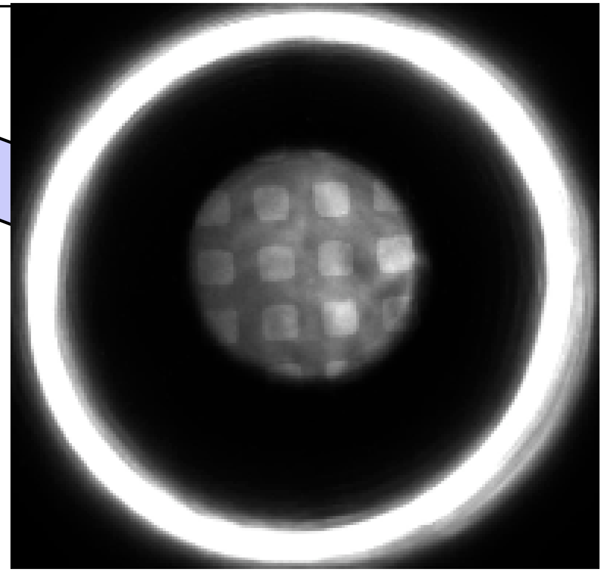
Critical illumination:

Demagnified image of source at the object plane.,
Each point of source corresponds to each point of field of view,
Not suitable for 3rd generation SR source.

Köhler illumination:

Infinite focus,
Each points at source to each angle of illuminating beam.





Test Image of Köhler Illumination
Rotating Double-mirror Optics
X-ray Energy: 12.4 keV,
Objective FZP: 100 nm outermost zone width,
Sample: Cu grid mesh, 12.7 μm pitch.

Effective Hollow-cone Illumination (Quasi-Köhler Illumination)

Synthesized Aperture by Rotating Double Mirror

Soft X-ray Imaging Microscope at BESSY-II
B. Niemann, et al., AIP CP507 (2000) 440.

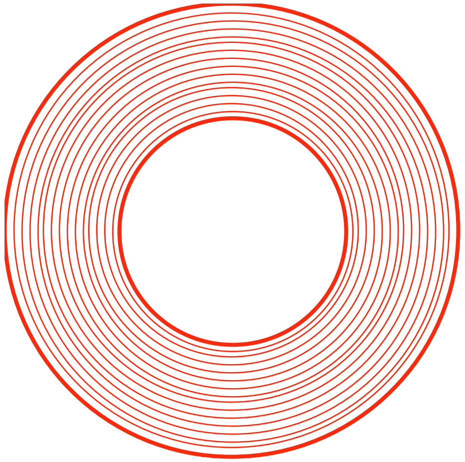
Advantages of Köhler illumination by rotating mirror optics:

1. **F-number matching, adjustable *NA*,**
2. **High efficiency, (cf. periodic condenser zone plate),**
3. **Achromatic,**
4. **Reduction of speckle noise,**
5. **Large and uniform field of view.**

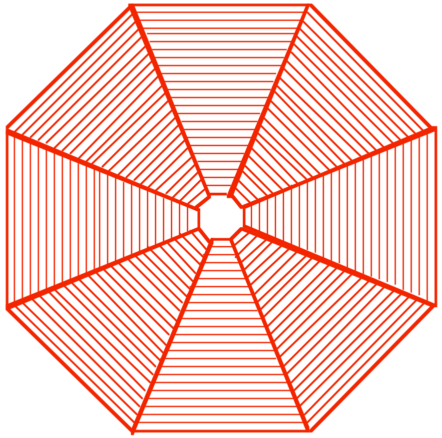
Disadvantages:

1. **Difficult to align,**
2. **Measuring time: rotation speed and synchronization of long mirrors for hard X-ray total reflection.**

Annular Condenser

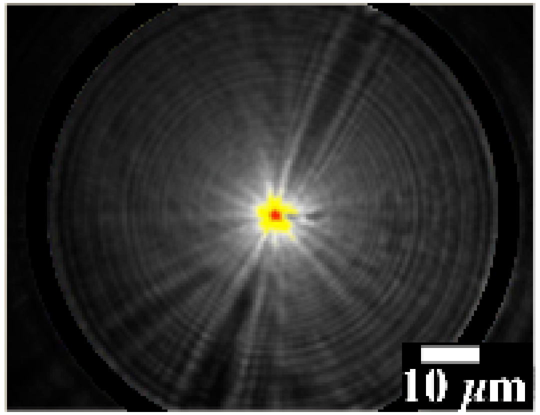


Sector Condenser (Octagon)



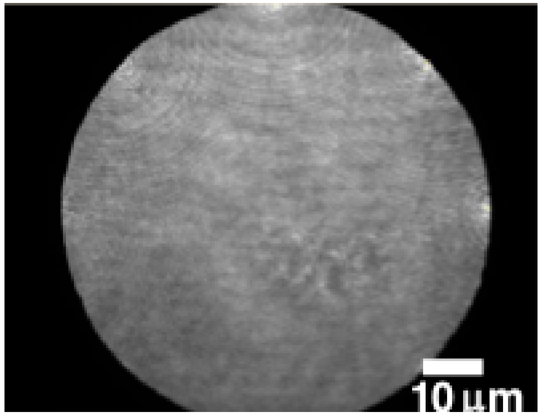
Condenser zone plate fabricated by NTT-AT, Material: tantalum, 1.65 μm-thick, Diameter: 1 mm, Grating pitch: 400 nm.

Constant-pitch Circular Grating



Intensity ~ 1/*R*

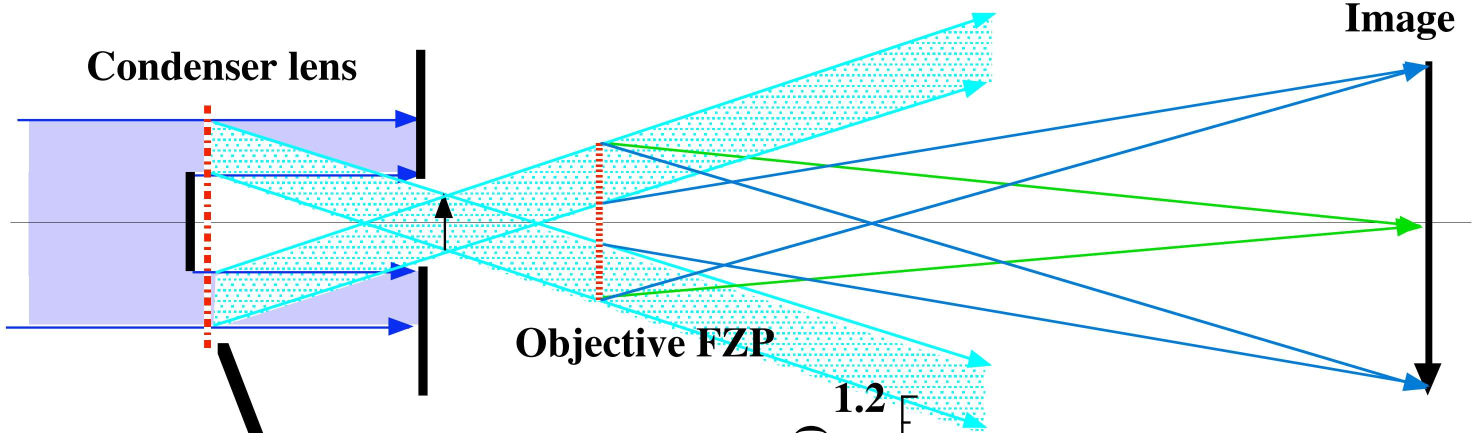
Combination of Constant-pitch Linear Gratings

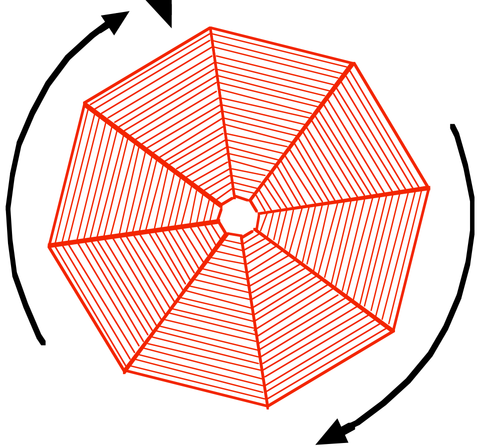


Flat Field,

Speckle noise, Interference fringes between gratings.

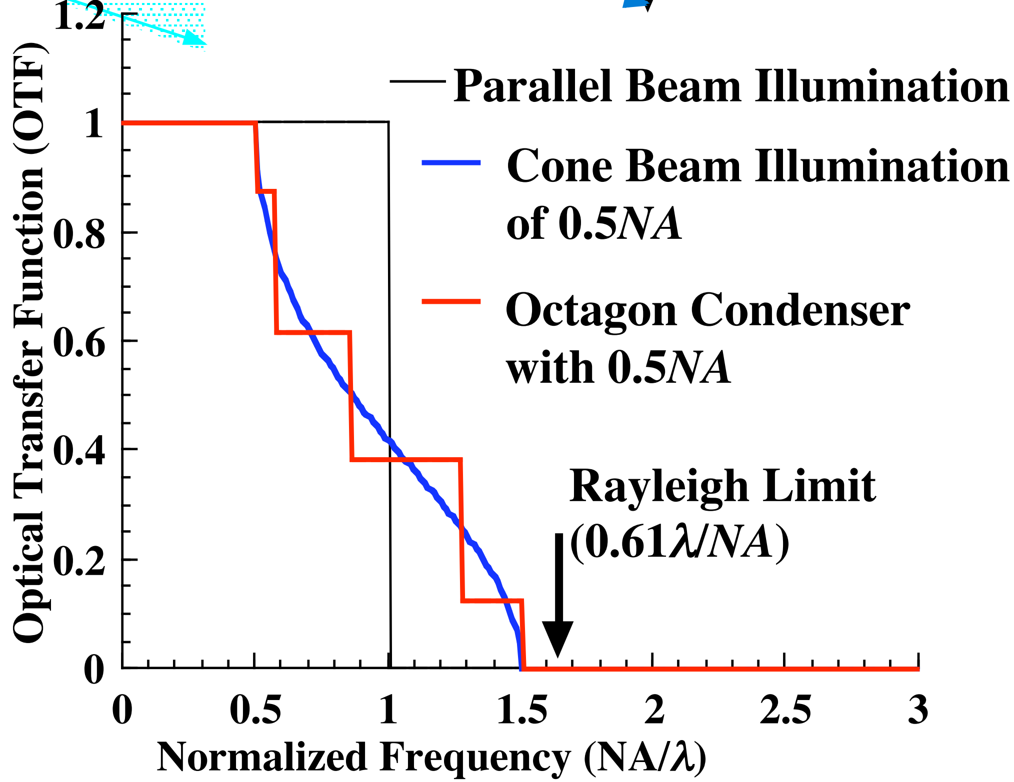
Illuminating Optics for Imaging Microscopy



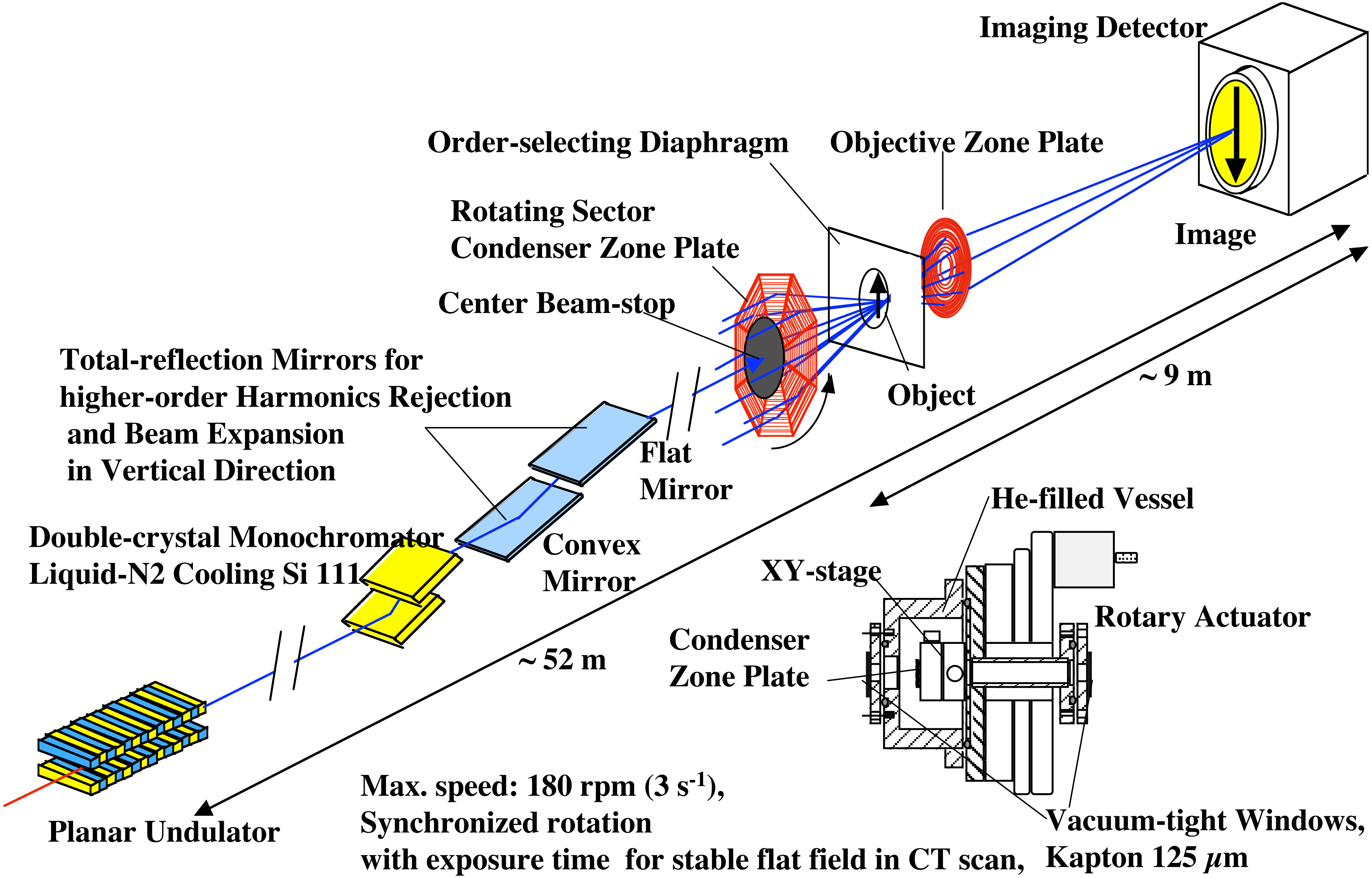


Rotating Sector Condenser Zone Plate (Octagon)

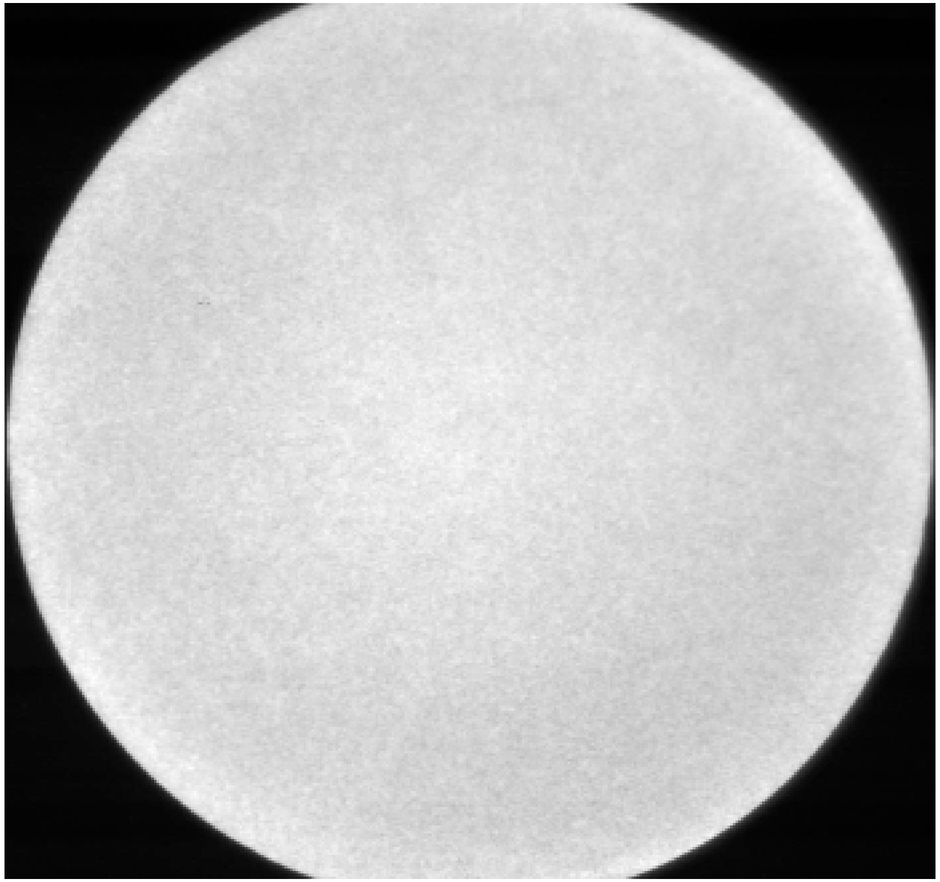
1. Uniform & flat field,
2. Speckle-free,
3. Less stray light & low background.



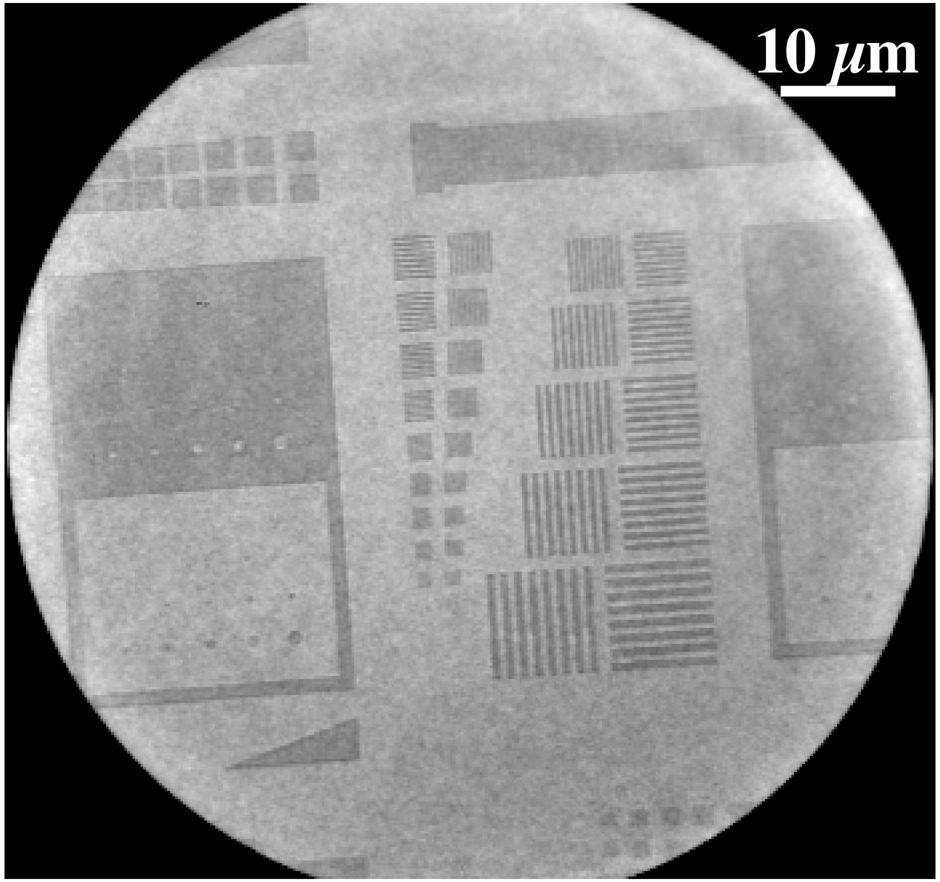
Theoretical Resolution of Imaging Microscope under Various Illumination Conditions



Experimental Setup of Imaging Microscopy/microtomography at BL47XU



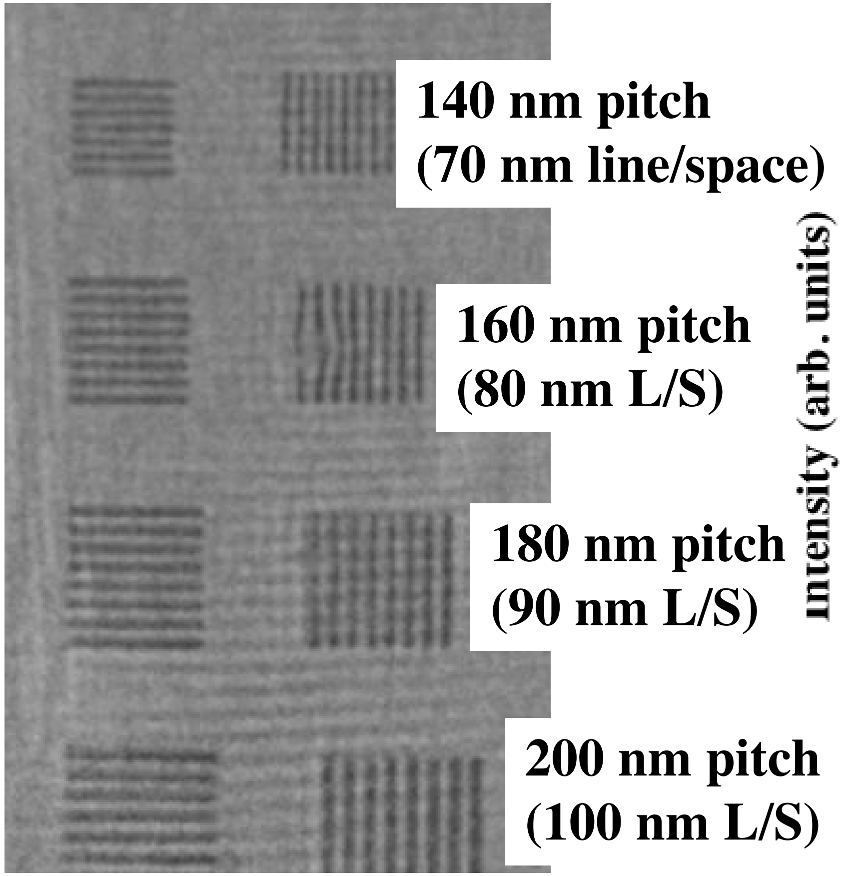
Blank Image

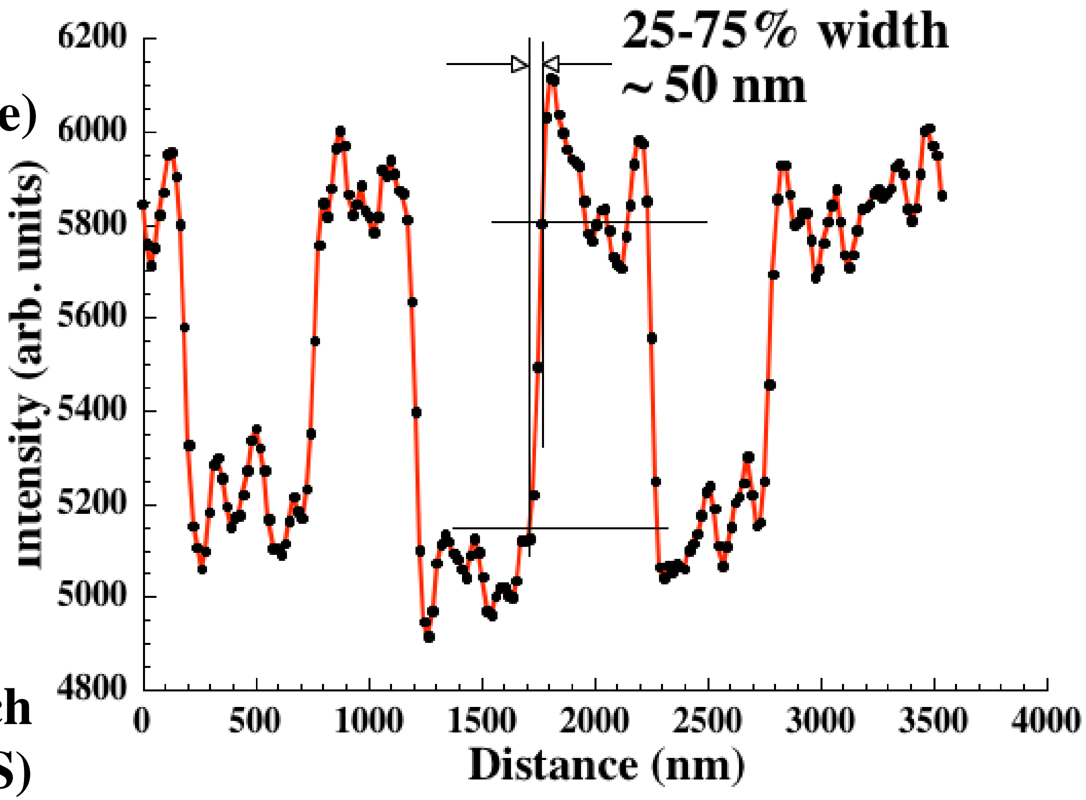


Resolution Test Patterns

X-ray Microscopy Images
with FZP Objective and Rotating-grating Condenser Illumination

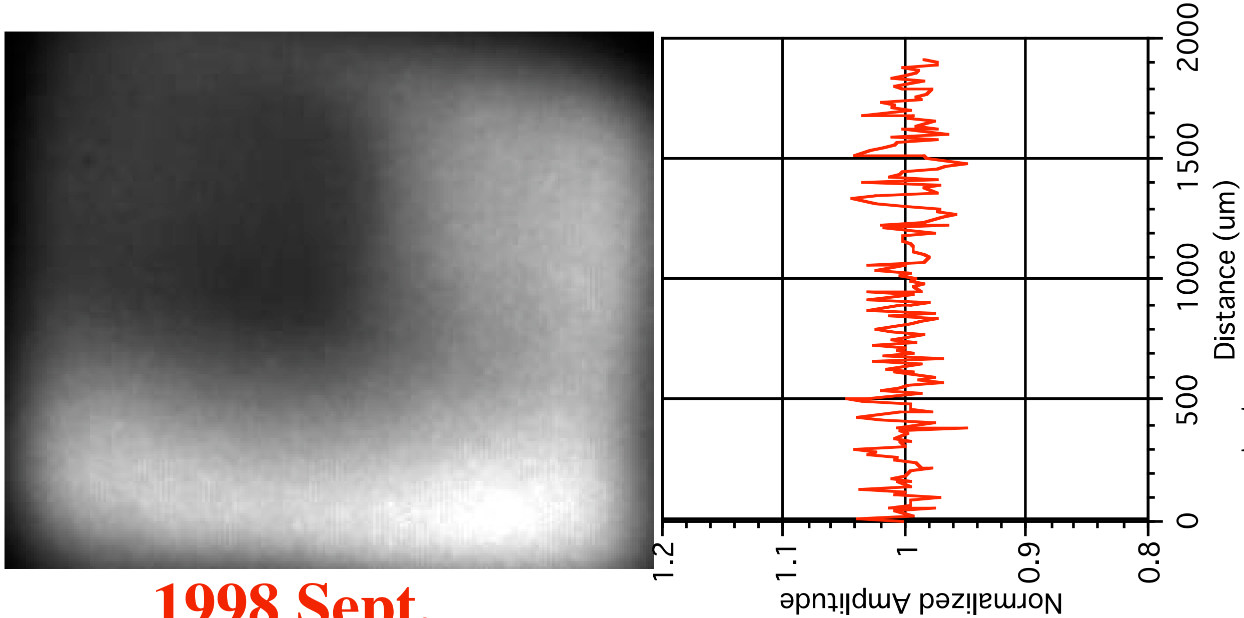
X-ray Energy: 8 keV,
Exposure time: 1 s,
Rotation speed of CZP: 1 rotation/s.





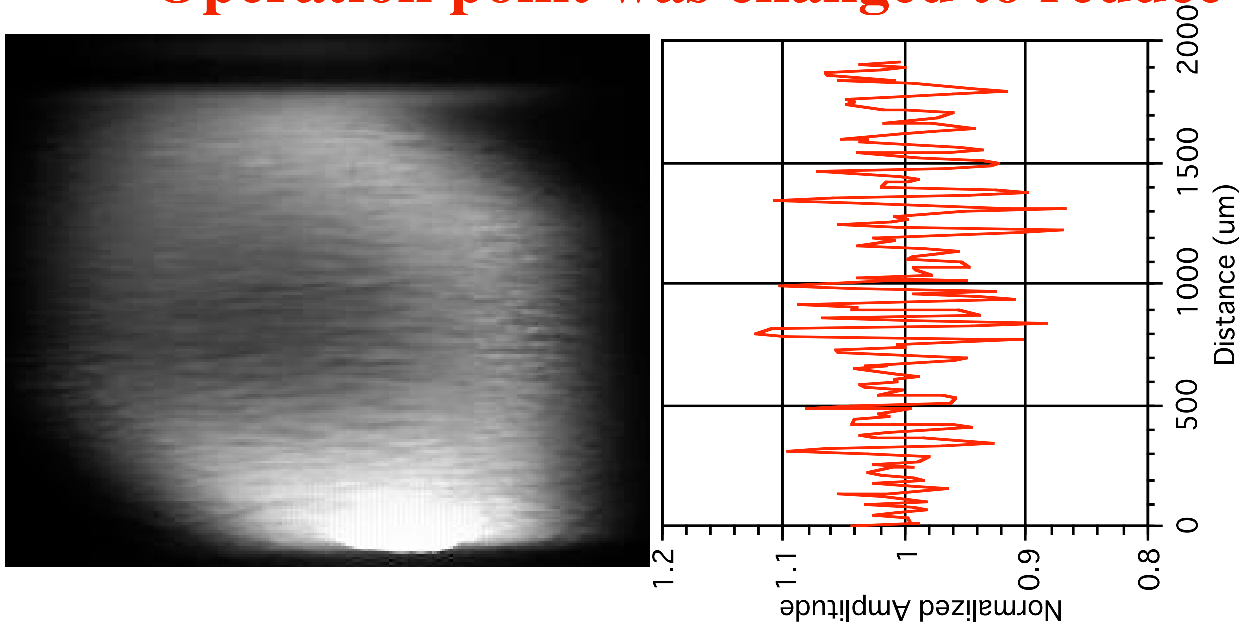
Imaging Microscopy

Resolution test patterns: 140 nm - 200 nm pitch, Ta 0.5 μm-thick patterns
Objective FZP: 50 nm outermost zone width, 155 μm Diameter
X-ray energy: 8 keV.



Before Improvement (March 1998)

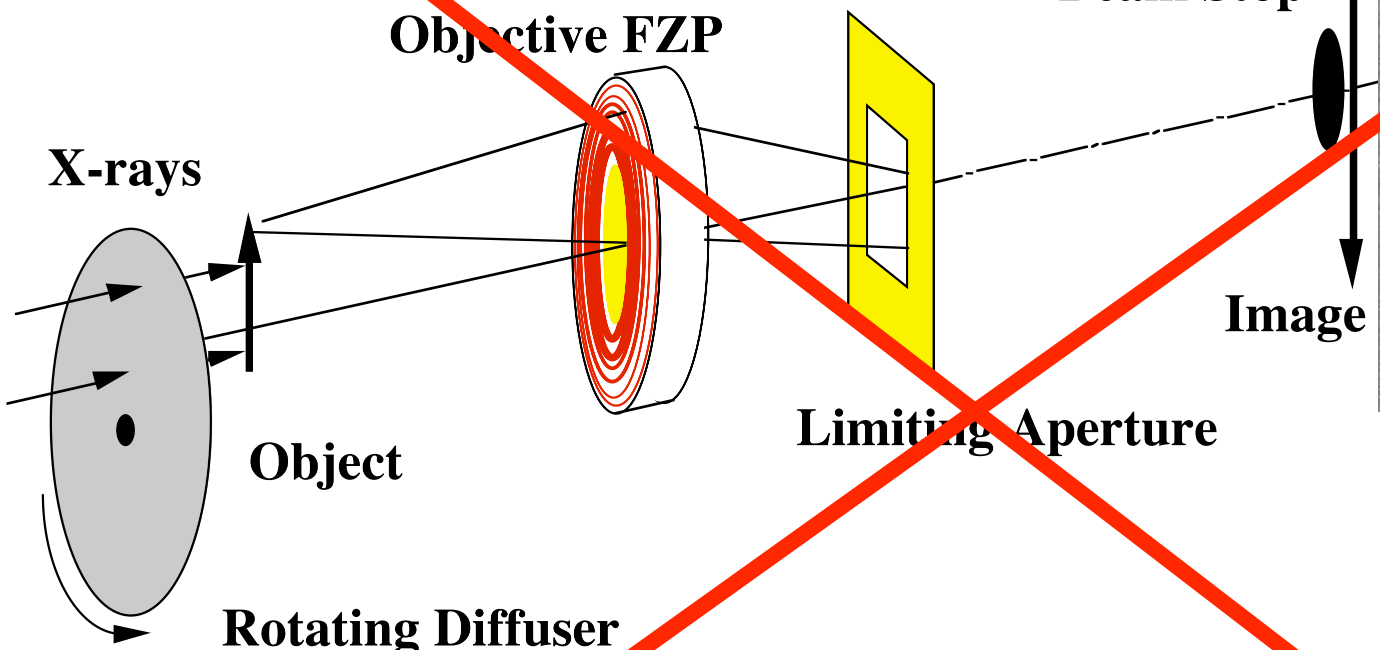
1998 Sept.
Operation point was changed to reduce vertical emittance.

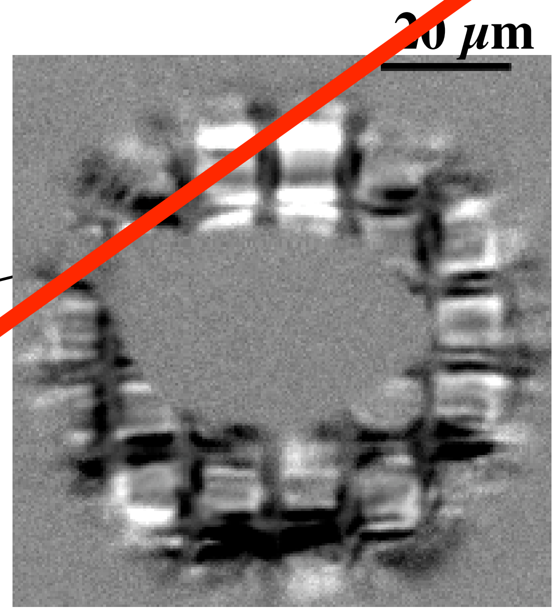


After Improvement (Oct. 1998)

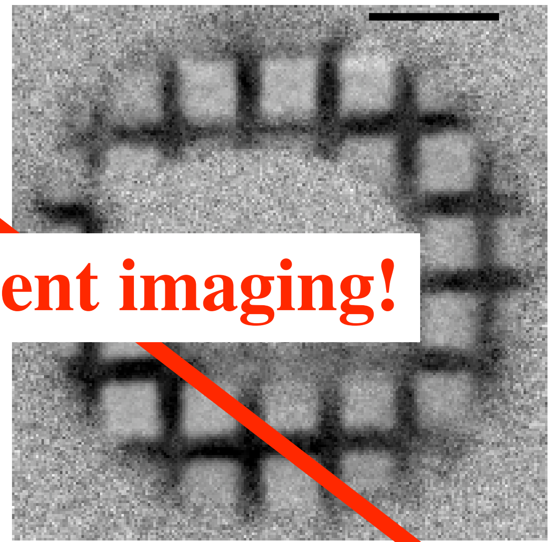
Speckle Pattern from Front-end Beryllium Windows

A Way of Speckle Reduction -Use of Diffuser-





Without Diffuser

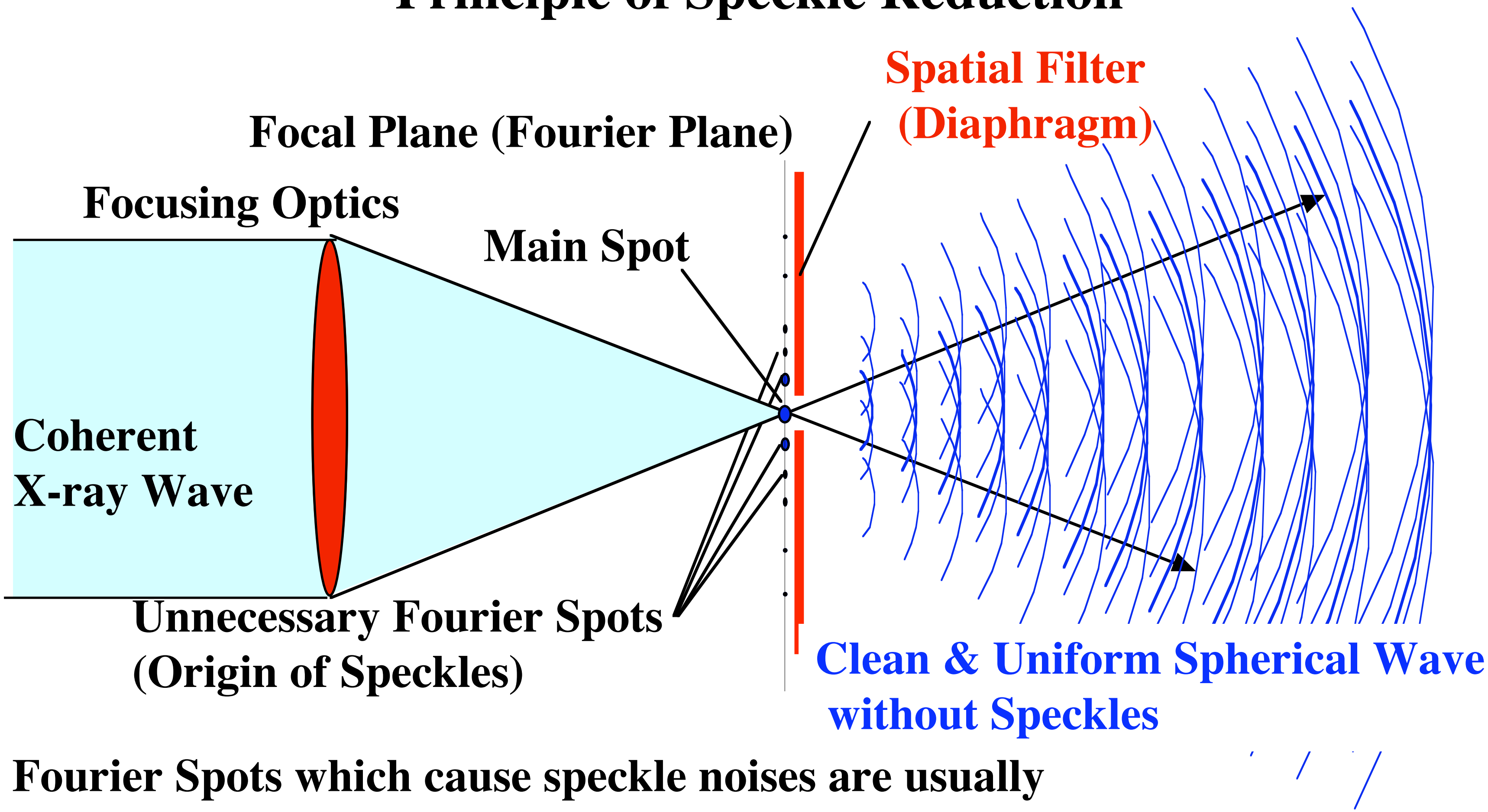

With Diffuser

This method cannot be applied to coherent imaging!

X-ray Imaging Microscopy with FZP Objective

Sputtered-slice Fresnel Zone Plate, 25 keV

Principle of Speckle Reduction

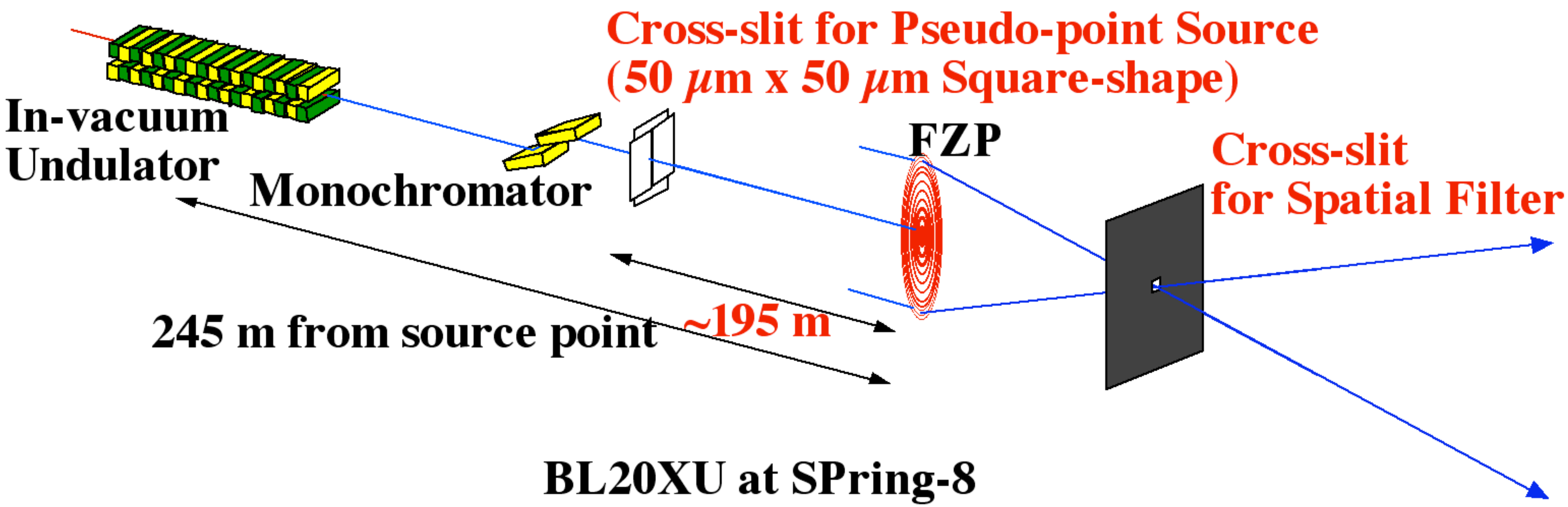


1. Fourier Spots which cause speckle noises are usually far from main focal point on the focal plane,

2. Speckles can be removed by blocking the unnecessary Fourier components using a spatial filter on the focal plane,

3. Spatial and temporal coherence is perfectly preserved.

Techniques for Practical Optics #1
Coherent X-ray Beam, a Long Beamline with Undulator Source



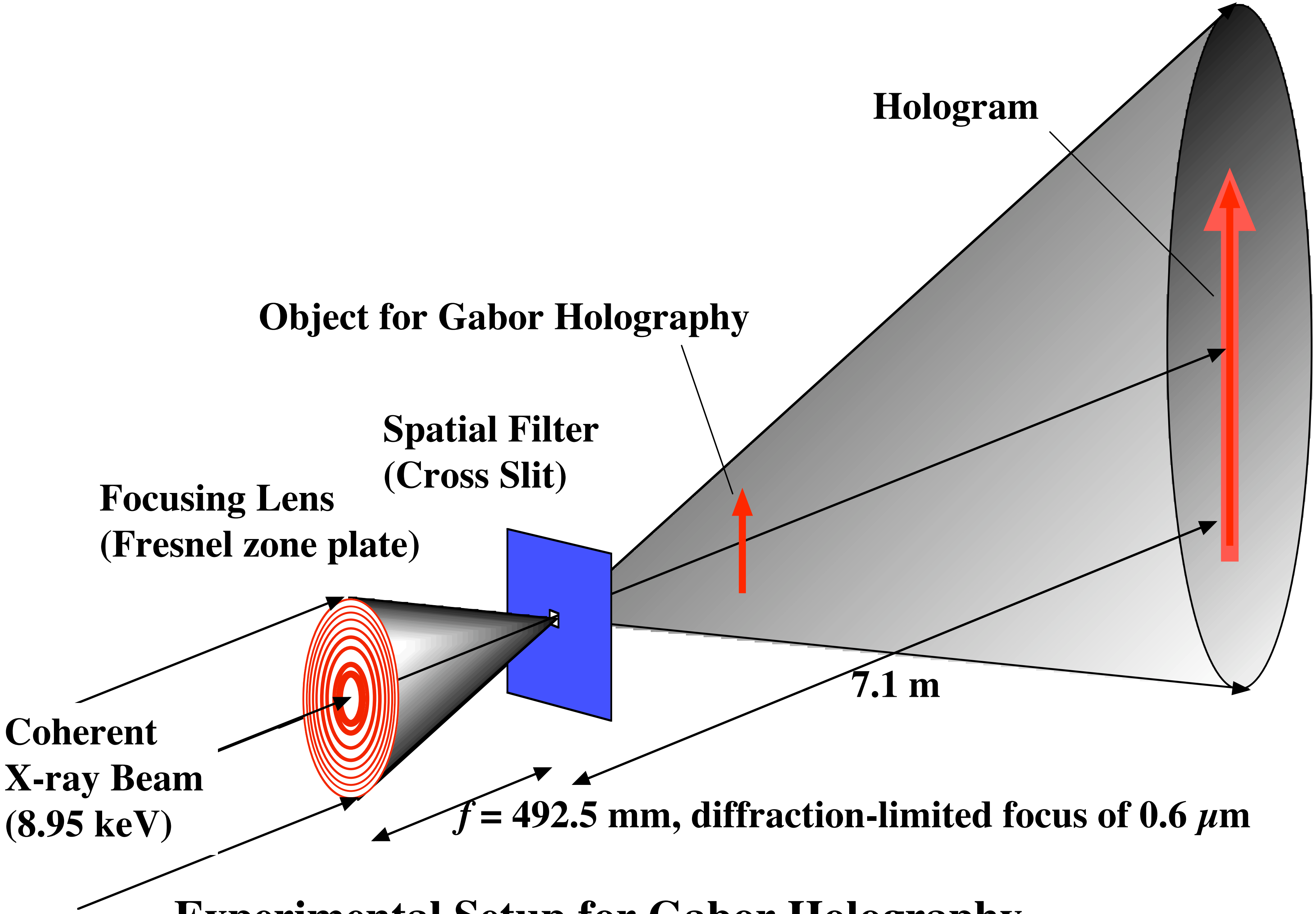
BL20XU at SPring-8

$\gamma = \sin(\pi x \Delta\theta / \lambda) / (\pi x \Delta\theta / \lambda)$

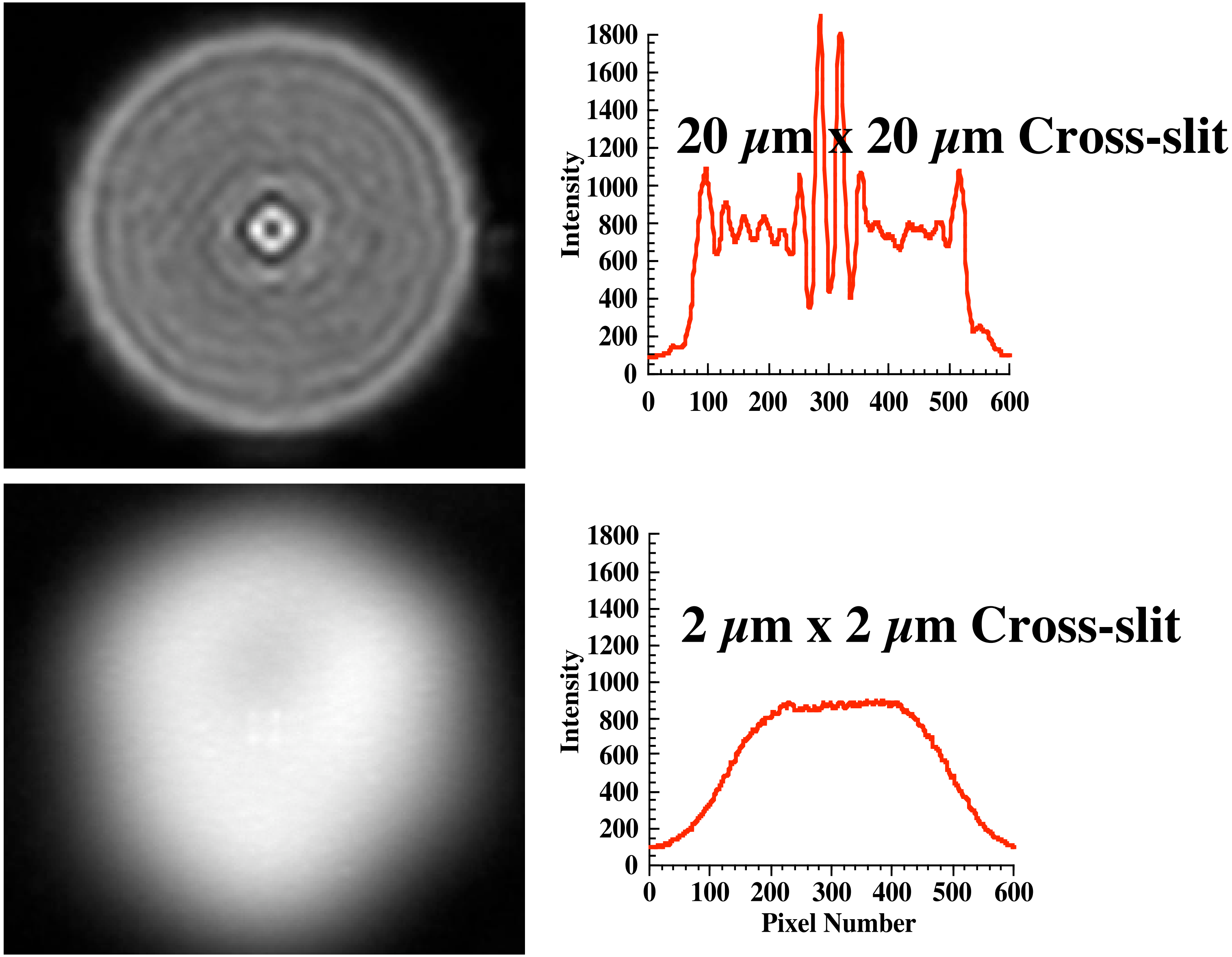
λ: X-ray wavelength,
$\Delta\theta$: angular size of light source,
x: distance at observation plane.

$\gamma = \sin(\pi x \Delta\theta / \lambda) / (\pi x \Delta\theta / \lambda) = 0.927$

for $\lambda = 1.2589$ Å (9.85 keV),
$\Delta\theta = 50\ \mu m / 195\ m = 2.56 \times 10^{-7}$ rad,
$x = 104.46\ \mu m$ (Diameter of FZP).

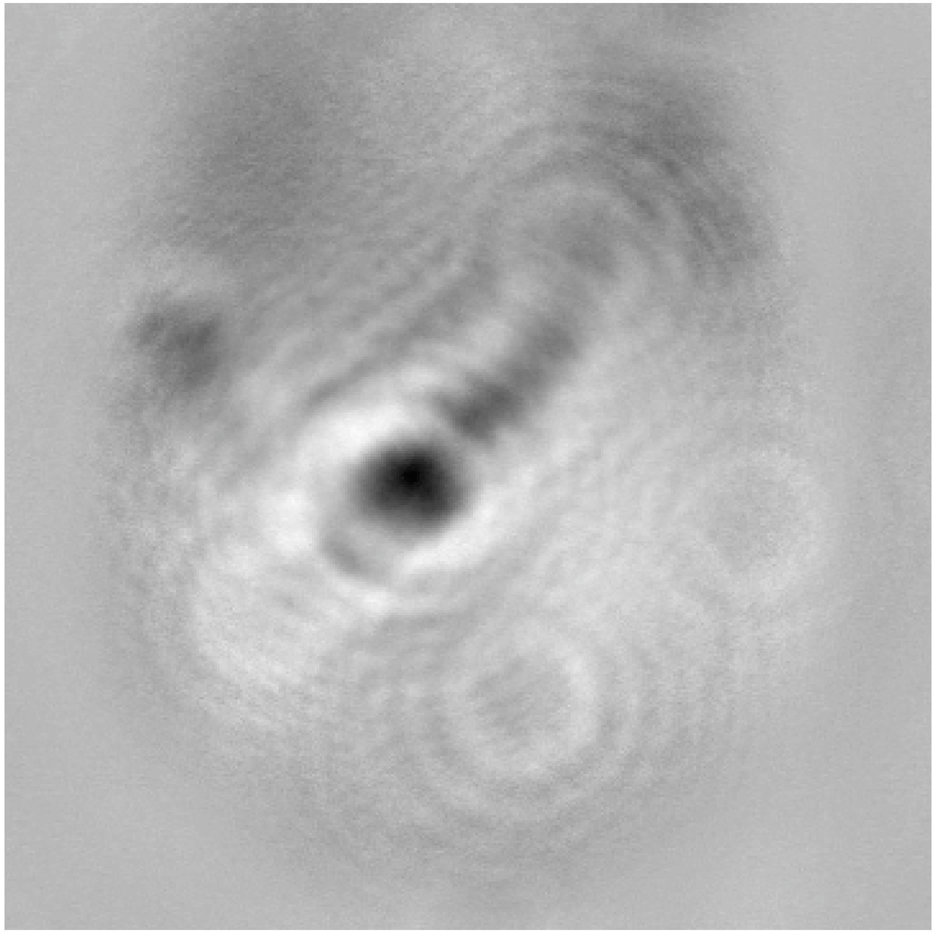


Experimental Setup for Gabor Holography

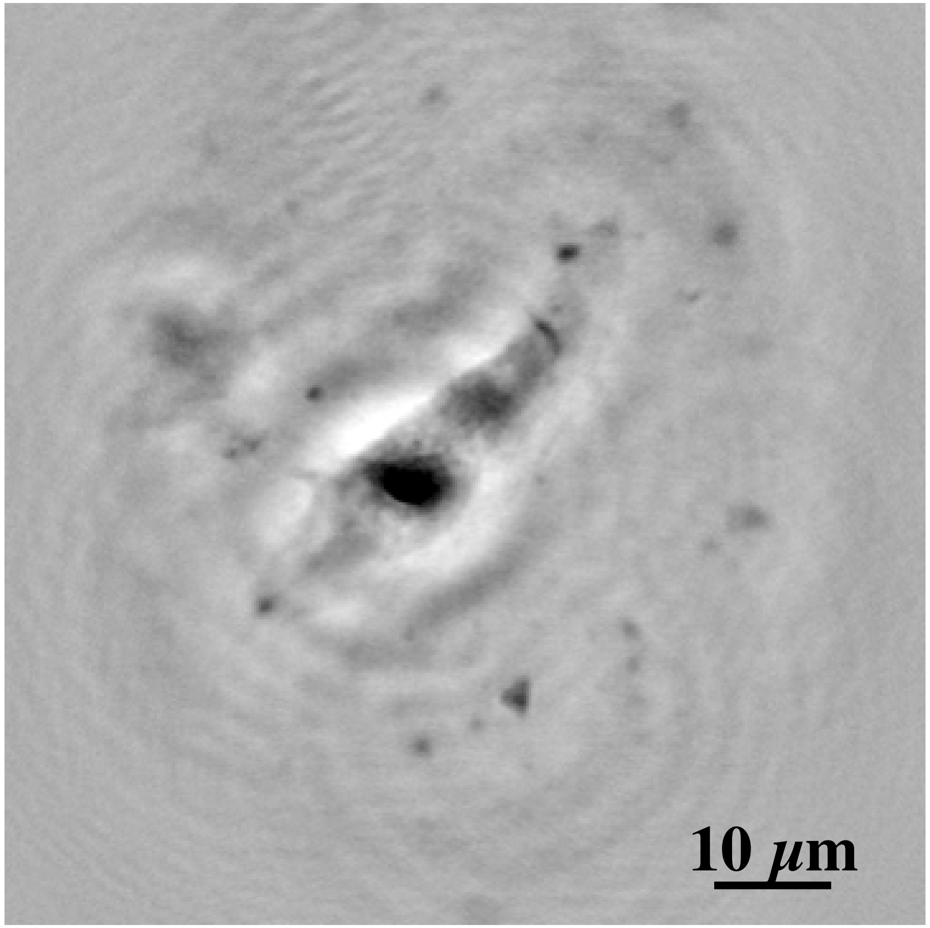


Reduction of Speckle Noises, Far-field Images

X-ray Energy: 9.85 keV, exposure time: 10 s, focus spot size of 0.6 μm,
Imaging Detector: P43 Phosphor Screen + Relay Lens + CCD Camera,
Hamamatsu Photonics, C4742-98-24A, 1344 x 1024 pixel, cooled CCD.



Hologram

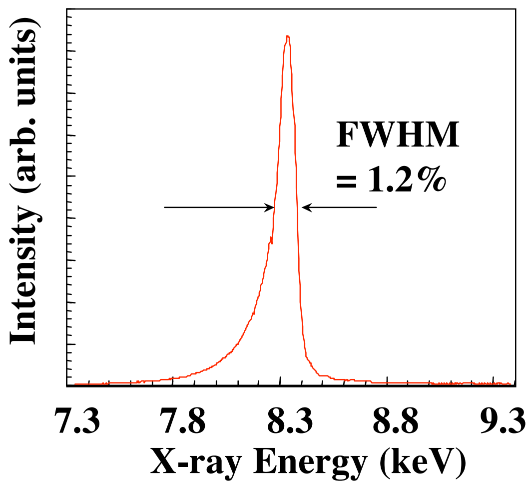


Reconstructed Image
(Phase-image)

Gabor Holography of Test Object

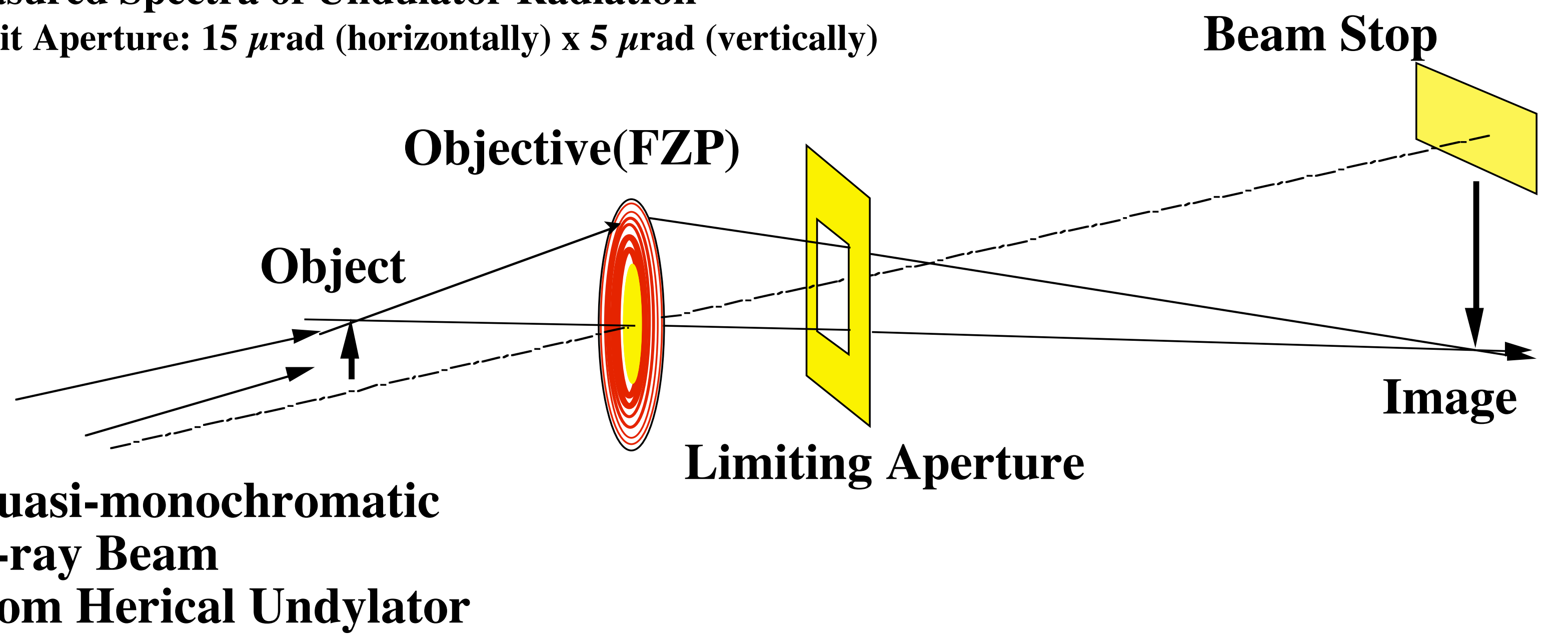
Sample: HeLa Cell (Dry), X-ray Energy: 9.85 keV, exposure time: 10 s.

Y. Suzuki and A. Takeuchi, AIP Conference Proceedings 1234 (2010) 453.



Hard X-ray Imaging Microscopy with Fresnel Zone Plate Objective & Quasi-monochromatic Undulator Radiation

Measured Spectra of Undulator Radiation
Front-end Slit Aperture: 15 μrad (horizontally) x 5 μrad (vertically)



Experimental Setup at BL40XU SPring-8

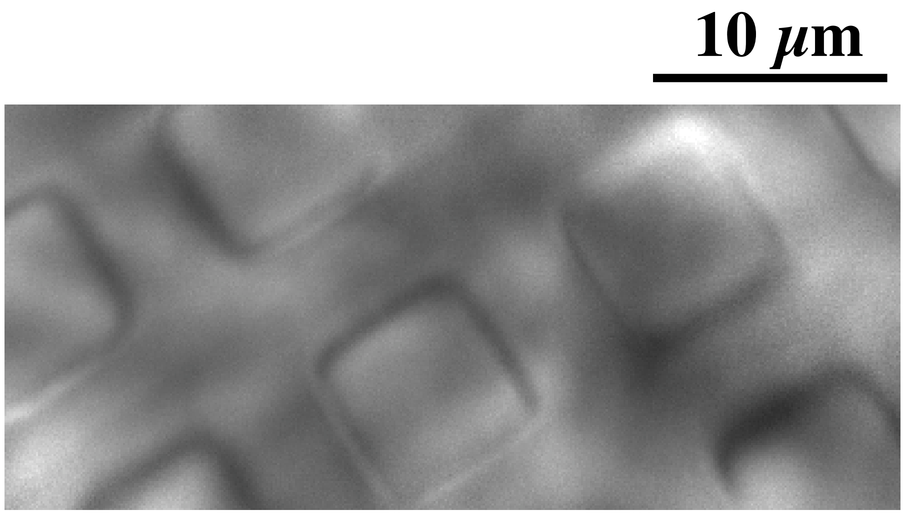
BL40XU of SPring-8 (High Flux Beamline)

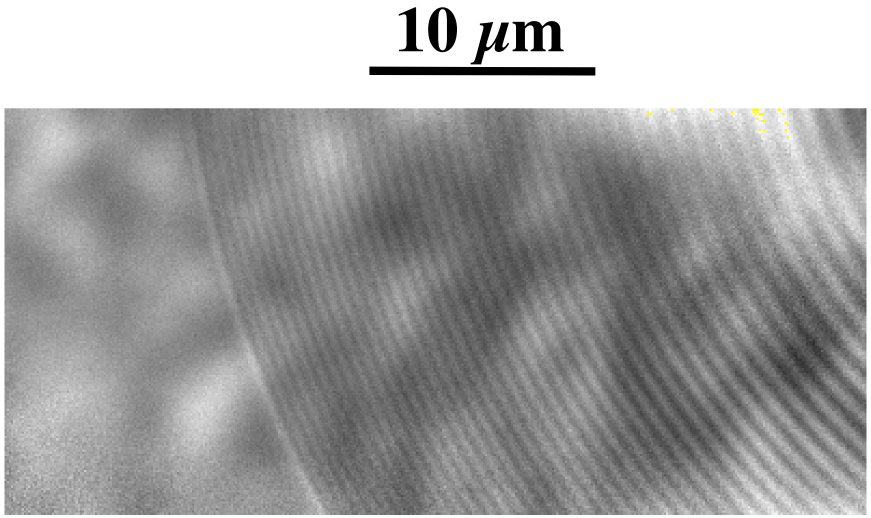
1. Undulator radiation without monochromator, $\Delta\lambda/\lambda \sim 1\%$.
2. Helical Undulator --> Suppression of higher order,
3. Condenser Optics: K-B mirror

Available flux ~ 1000 times conventional beamlines (undulator beamlines with crystal monochromator).

Requirement on monochromaticity for Fresnel zone plate ~ Number of Fresnel zone.
--->$\Delta\lambda/\lambda \sim 1/N$ (number of Fresnel zone)

Use of direct undulator radiation, $\Delta\lambda/\lambda \sim 100$.
X-ray imaging microscopy with sub-μm resolution and milli-second exposure time!





**Object: Cu mesh,
2000 lines/inch**

**Object: Fresnel zone plate,
0.25 μm outermost zone width**

Image of test object

**Objective: FZP, 0.25 μm outermost zone width, 100 zones,
Magnification: 11.3,
X-ray energy: 8.34 keV,
Exposure time: 1.5 ms (Single Shot)**

Hard X-ray Imaging Microscopy with Fresnel Zone Plate Objective & Quasi-monochromatic Undulator Radiation at BL40XU

Y. Suzuki, et al., Rev. Sci. Instrum. 75 (2004) 1155.

Hard X-ray Imaging Opportunities at ERL

1. **Hard X-ray Microbeam (Probe size of ~ 25 nm by FZP),**
   Scanning microscopy,
   ***μ*-XAFS, *μ*-Diffraction, *μ*-XRF,**

2. **Imaging Microscopy (~ 50 nm Spatial Resolution),**
   3D imaging by CT/tomo-synthesis,
   Zernike's phase contrast, Dark-field method,

3. **Projection Imaging (Gabor Holography),**
   Absorption/phase contrast,
   Zooming capability (magnification from 1 to ∞),
   High speed imaging (> kHz),

4. **Quasi-monochromatic beam w/o Crystal Monochromator,**

Problems: radiation damage, deformation, transition.