

X線ナノ集光技術の展望

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Acknowledgement

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Background and today's contents

3rd generation SR facilities widely contribute to many S&T fields.
4th generation facility (XFEL) has already been here.



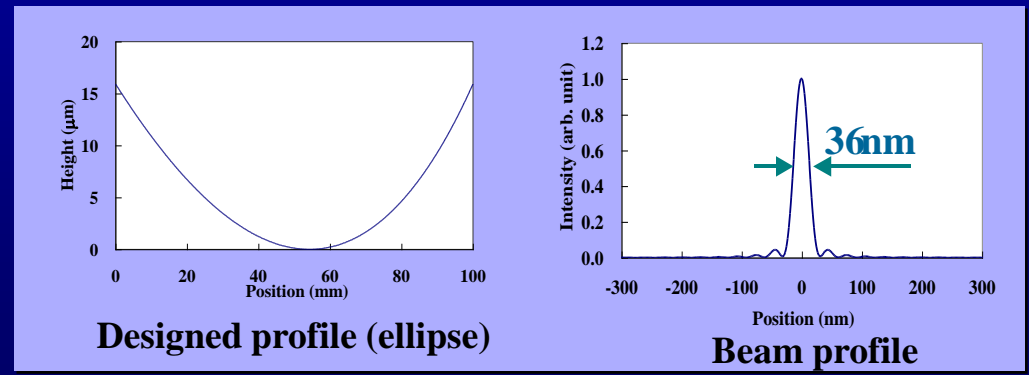
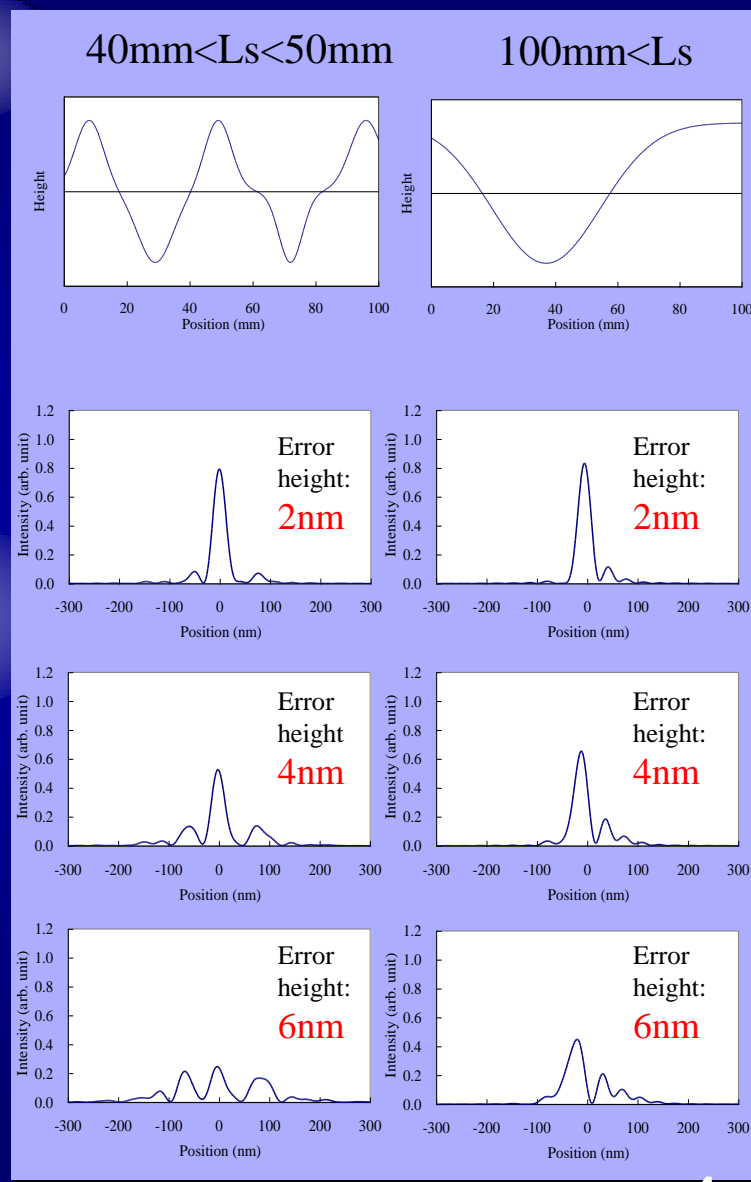
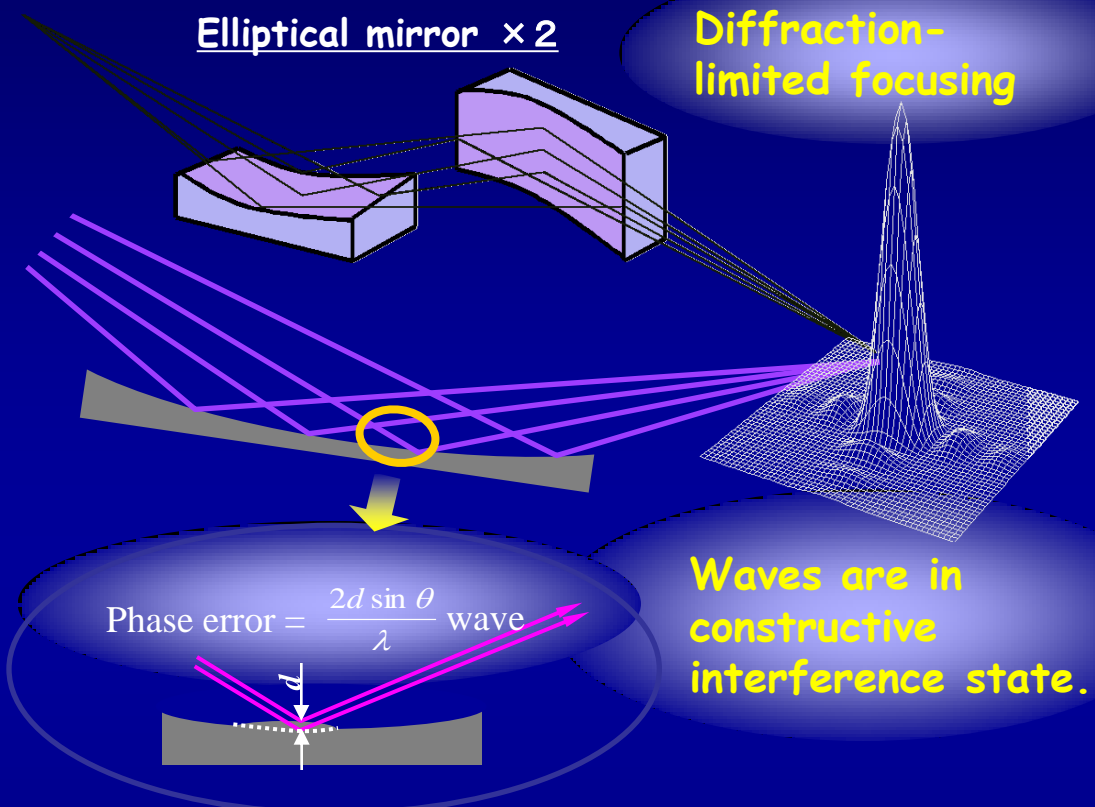
How should mirror optic contribute to advanced X-ray optical system?

Current research targets

1. Focusing down to sub-10nm (including brief introduction of sub-50nm focusing)
2. Full-field, achromatic and high-resolution imaging of incoherent X-rays
3. XFEL focusing (400mm-long mirror development)
4. Focused beam diffraction microscopy

Required accuracy for nano-focusing under D-limited condition

Kirkpatrick-Baez mirrors



© Plasma CVM (chemical vaporization machining)

→ Rough figuring (Rapid figuring with 10nm (P-V) level accuracy)

K. Yamamura et al., Rev. Sci. Instrum. 71 (2000), 4627

© EEM (elastic emission machining)

→ Final figuring and smoothing (Fine figuring with atomically smoothing)

K. Yamauchi et al., Rev. Sci. Instrum. 73 (2002), 4028

© MSI (microstitching interferometry)

→ Figure tester with spatial resolution close to 0.01mm

K. Yamauchi et al., Rev. Sci. Instrum. 74 (2003), 2894

© RADSI (relative-angle determinable stitching interferometry)

→ Figure tester for steeply curved ellipse of large NA mirror

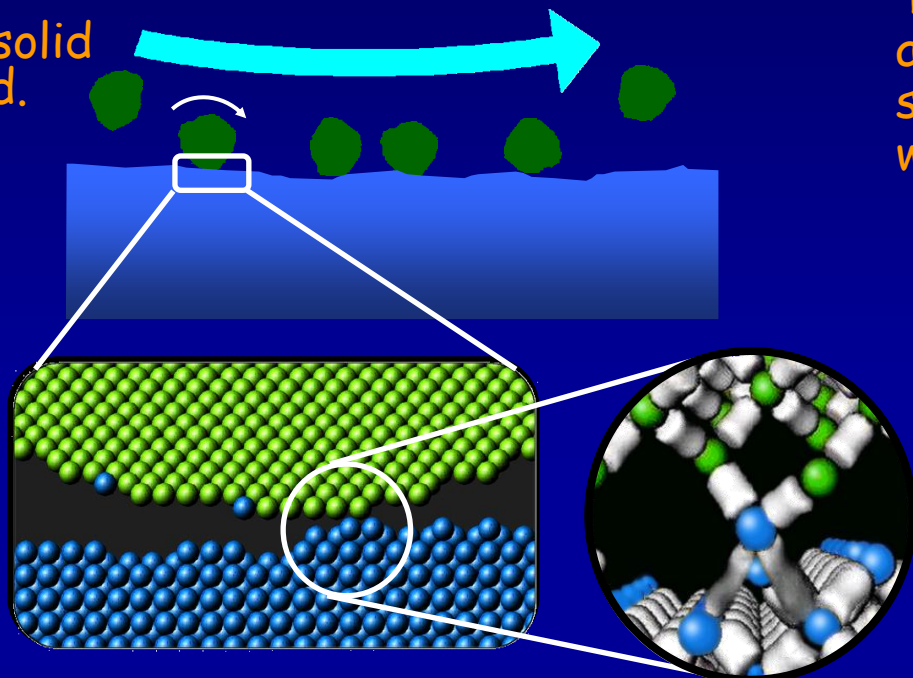
H. Mimura et al., Rev. Sci. Instrum. 76 (2005), 045102

JTEC URL <http://www.j-tec.co.jp>

Removal mechanism of EEM (Elastic Emission Machining)

In EEM, chemical reaction between solid surfaces is utilized.

Ultrapure water flow



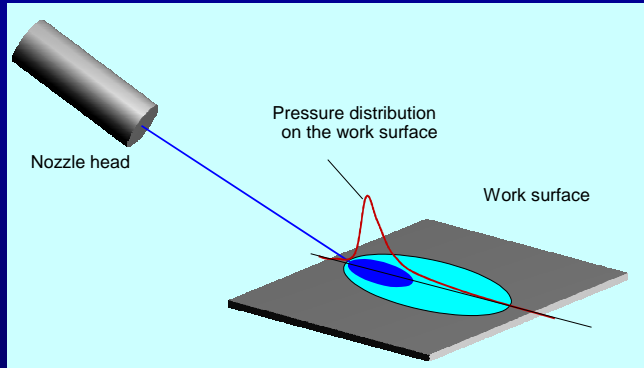
The ultra-fine particles are supplied to the work surface by ultrapure water flow

Atom-by-atom removal

Bump site is preferentially removed

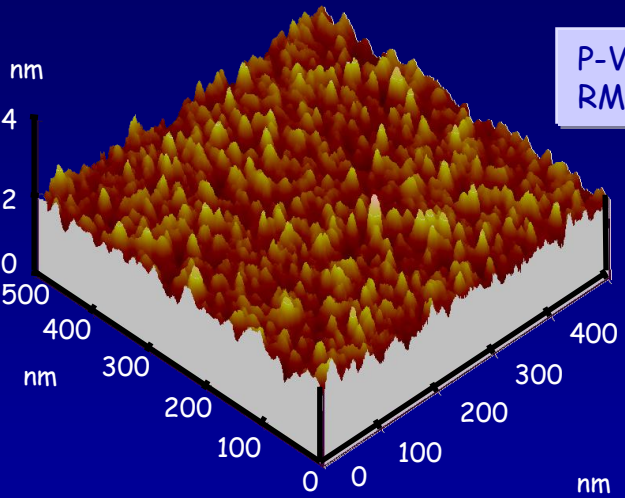


Atomically flat surface can be obtained



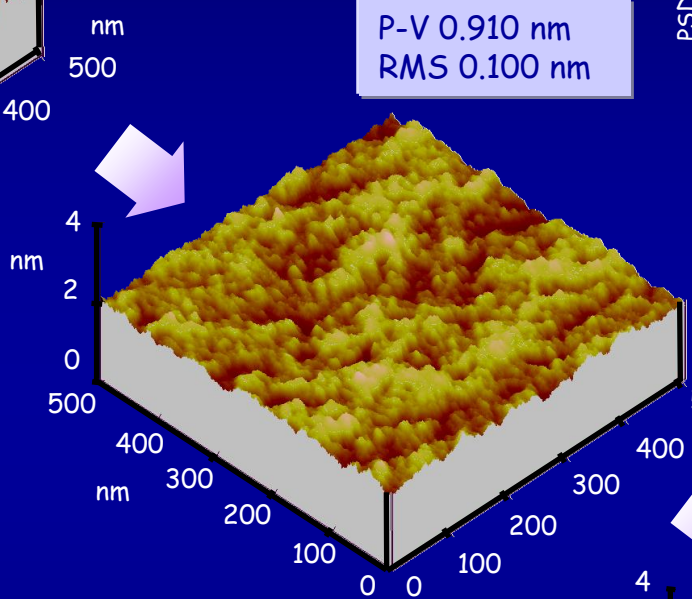
Deterministic figuring

Surfaces smoothing properties in EEM



P-V 1.603 nm
RMS 0.183 nm

Pre-machined surface

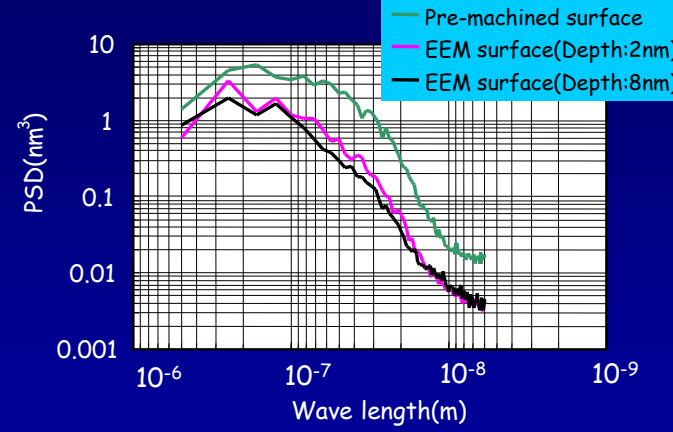


P-V 0.910 nm
RMS 0.100 nm

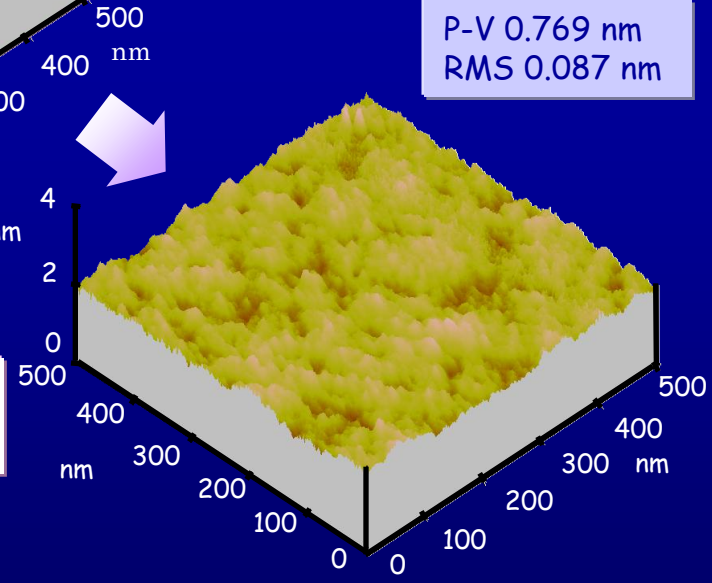
EEM surface (Removal depth:2nm)

Surface with the roughness of about 2nm (P-V) can be smoothed within the removal depth of 2nm.

The bump site is selectively removed.



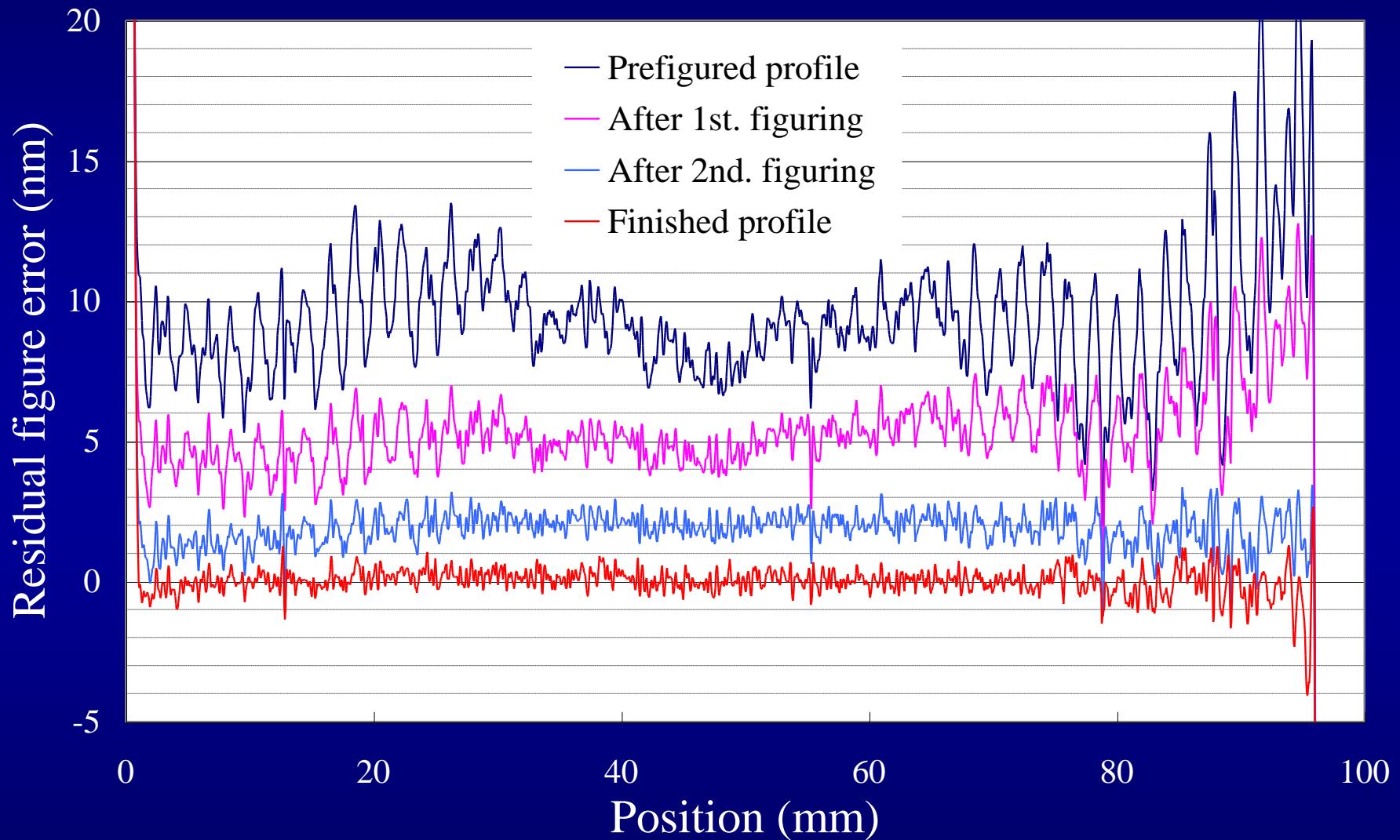
PSD analysis



P-V 0.769 nm
RMS 0.087 nm

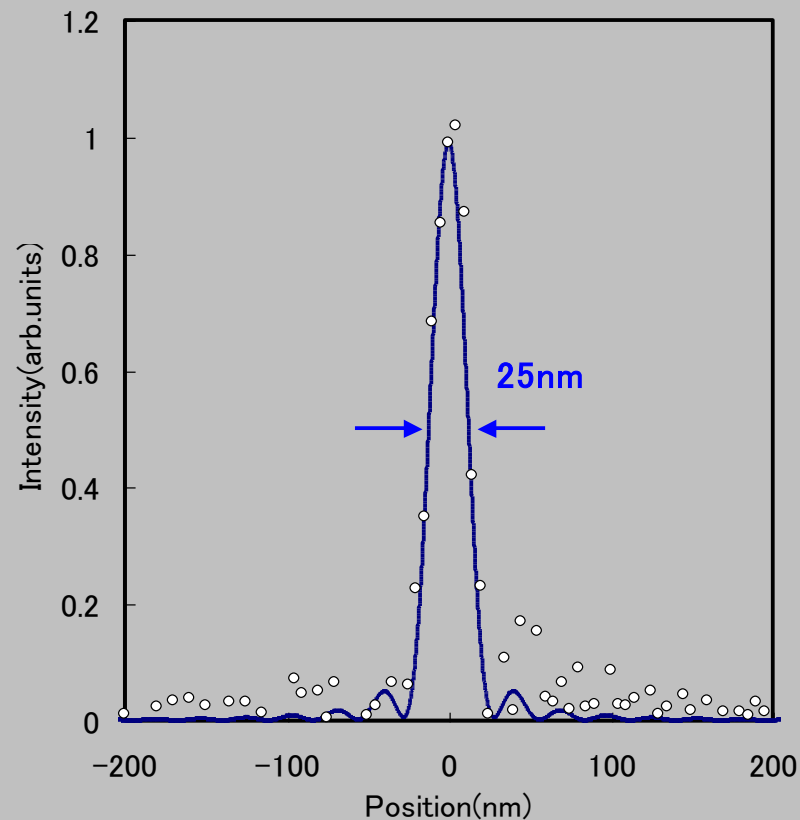
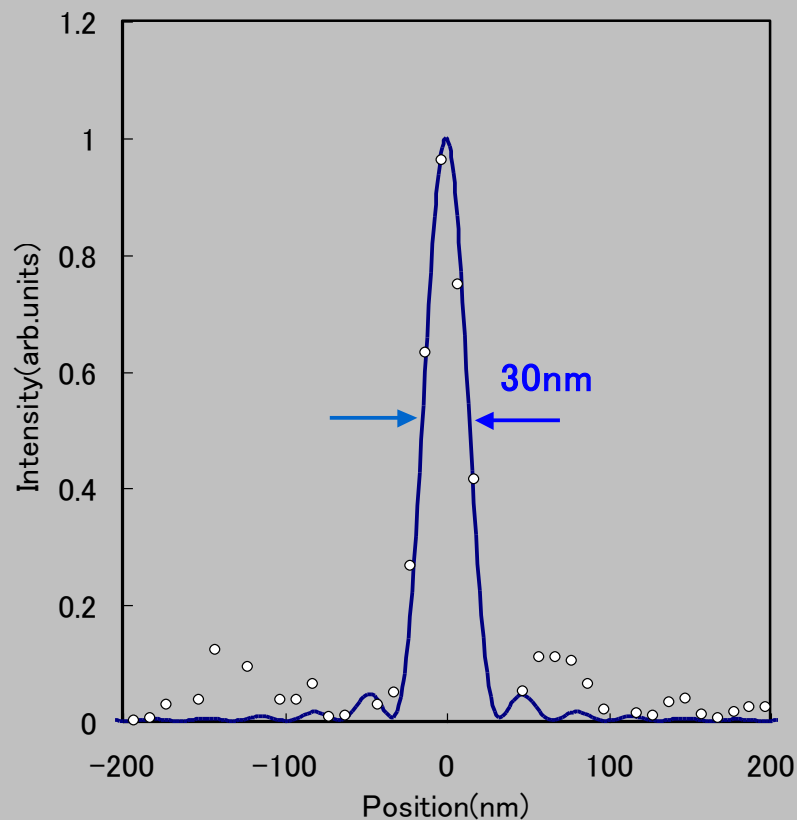
EEM surface (Removal depth:8nm)

Typical deterministic figuring properties using EEM



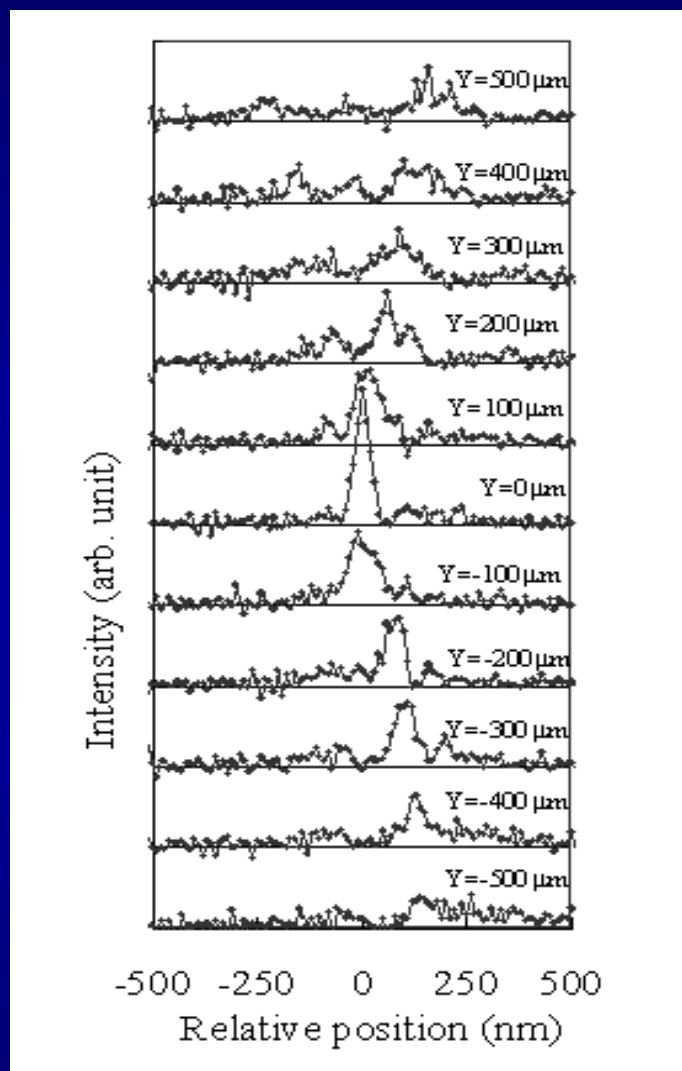
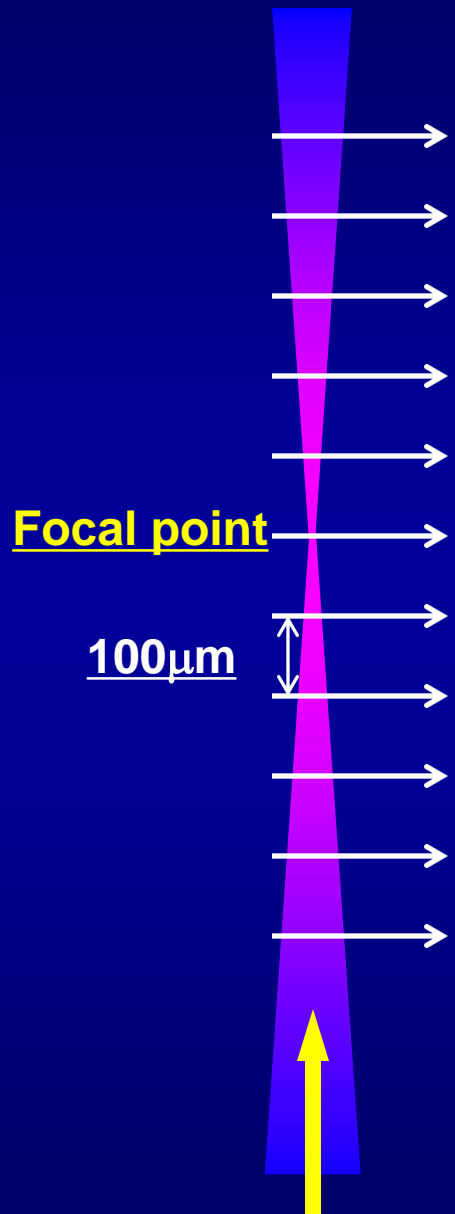
Sub-30nm focusing (2006)

Smallest size in hard-X-ray realized by total reflection achromatic mirror optics (focusing under diffraction limited condition)

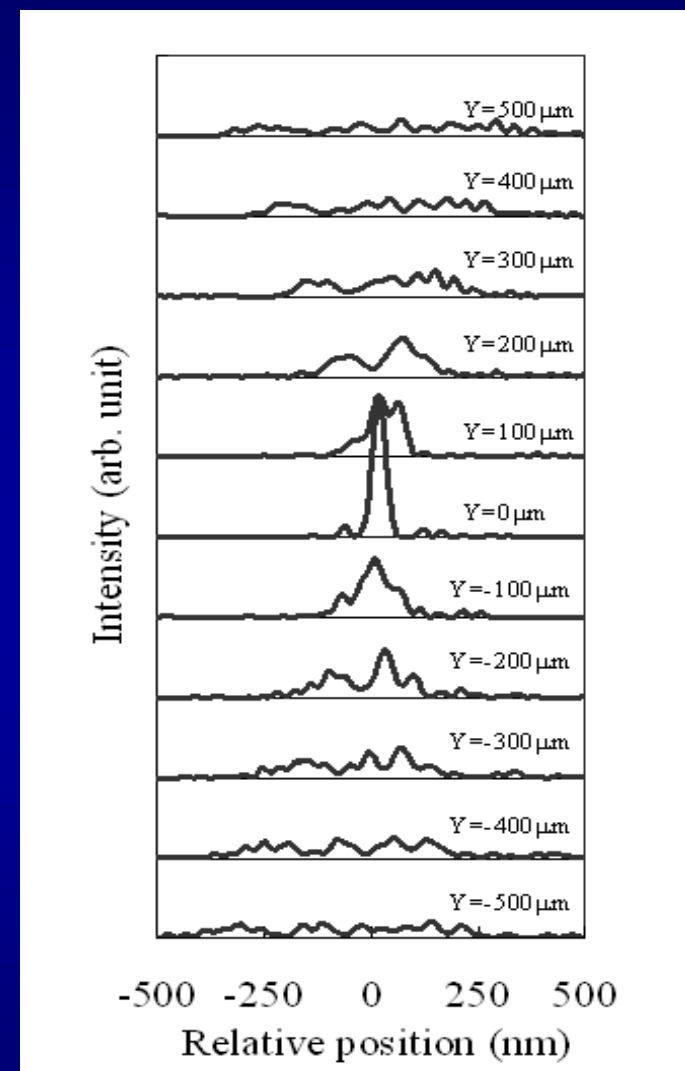


Mimura et al., APL (2007)

Beam waist structures

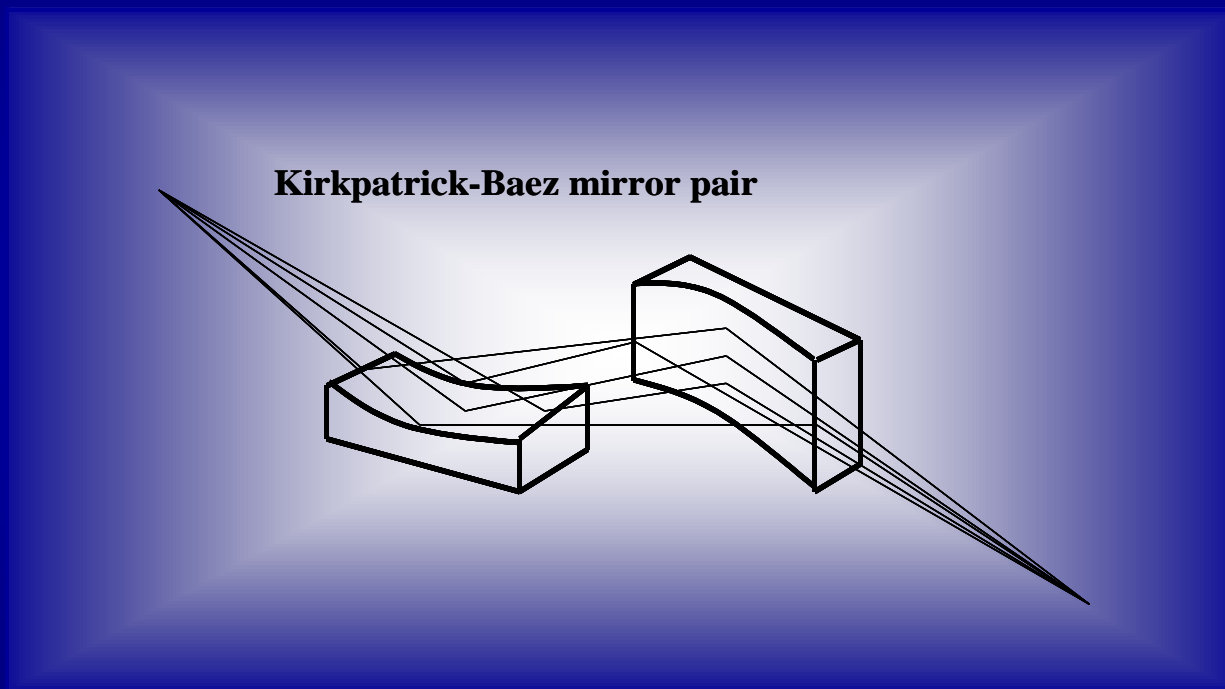


Measured profile

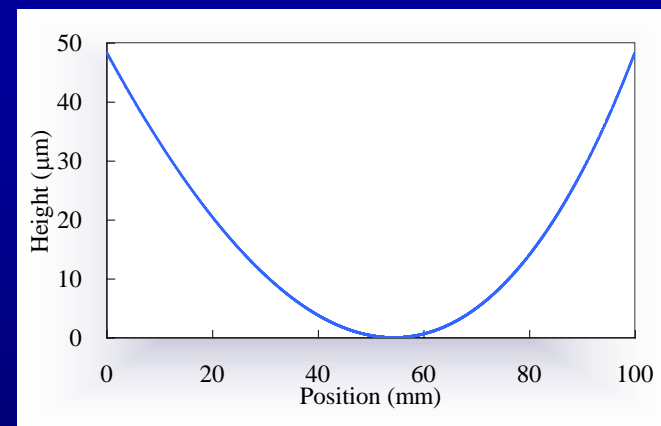
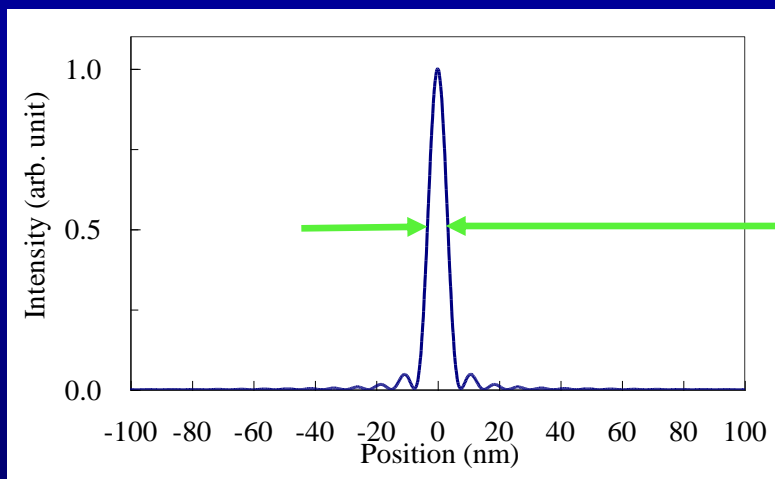
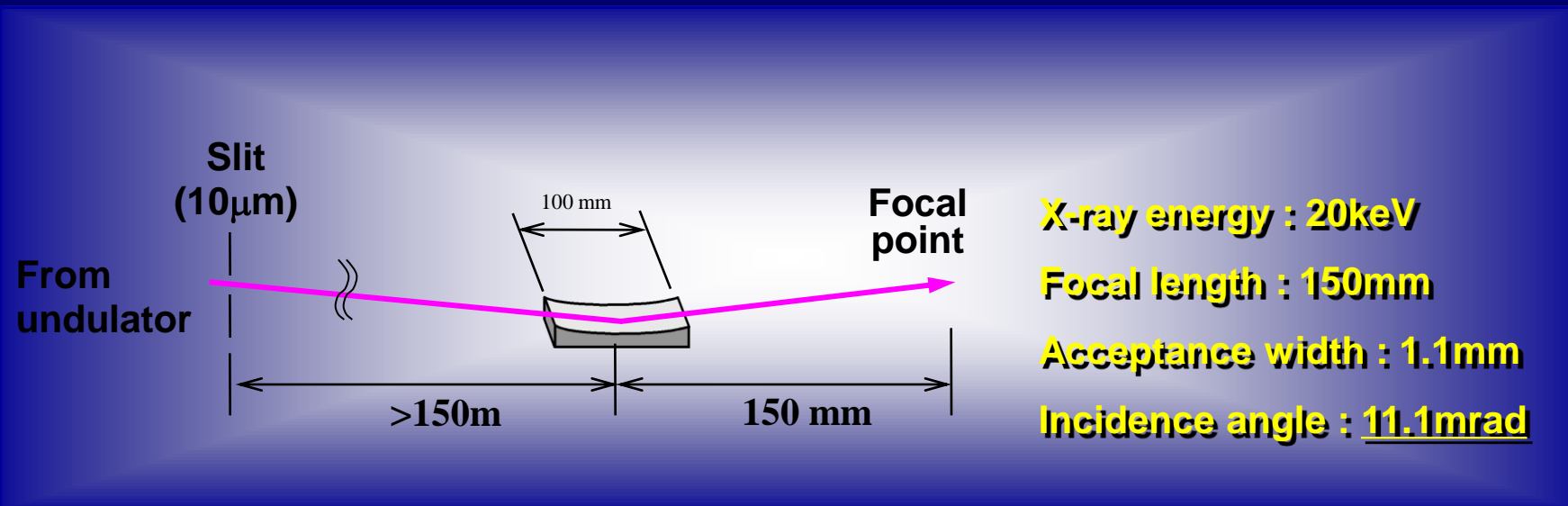


Expected profile

“Hard-X-ray sub-10nm focusing and realization of high-resolution X-ray microscopy”



To realize Sub-10nm focusing K-B mirrors



MSI with RADSI and EEM can prepare the surface figure with 1nm (P-V) accuracy.

Estimation of required accuracy

@20keV Mirror length: 100mm, Focal length: 150mm

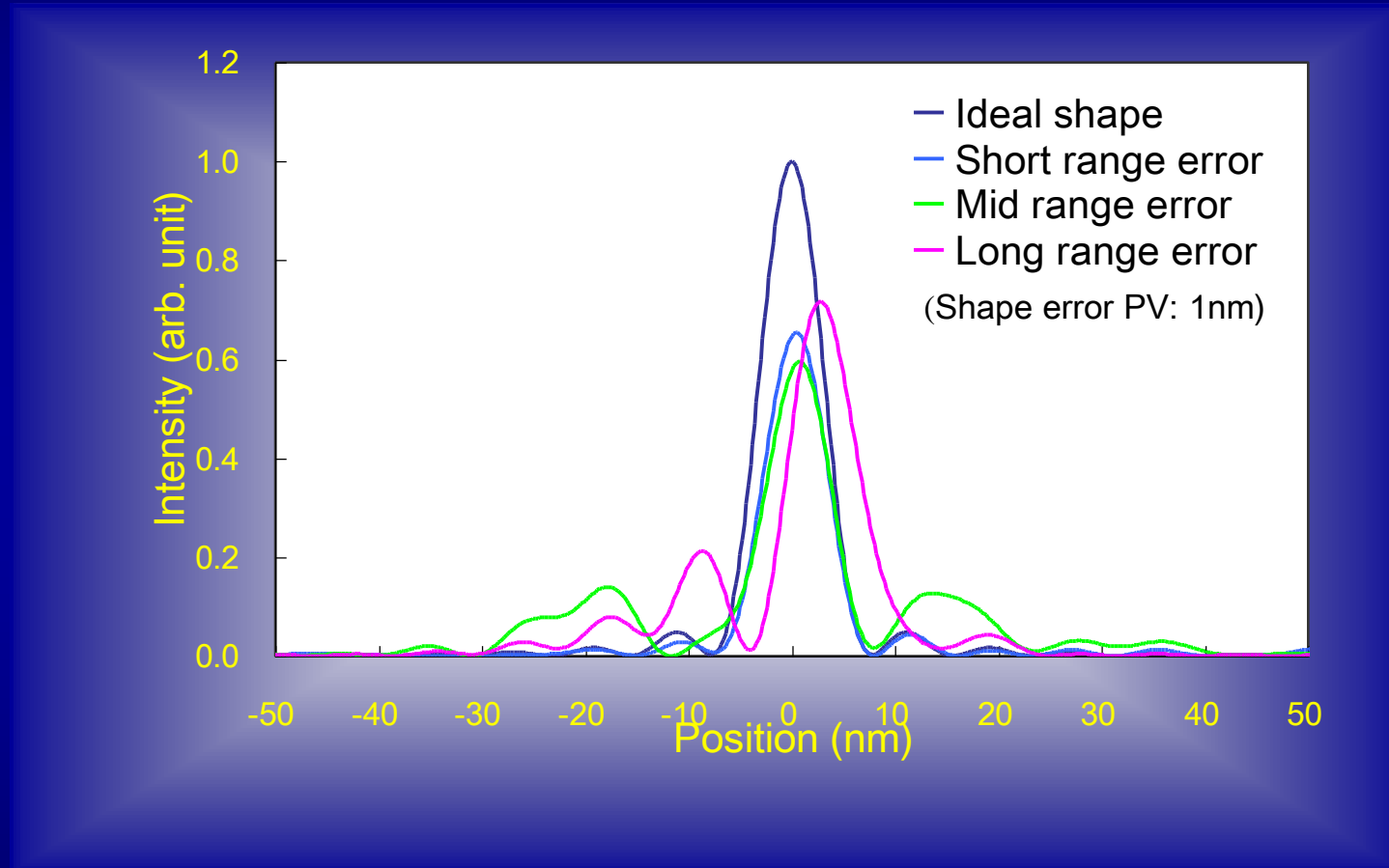
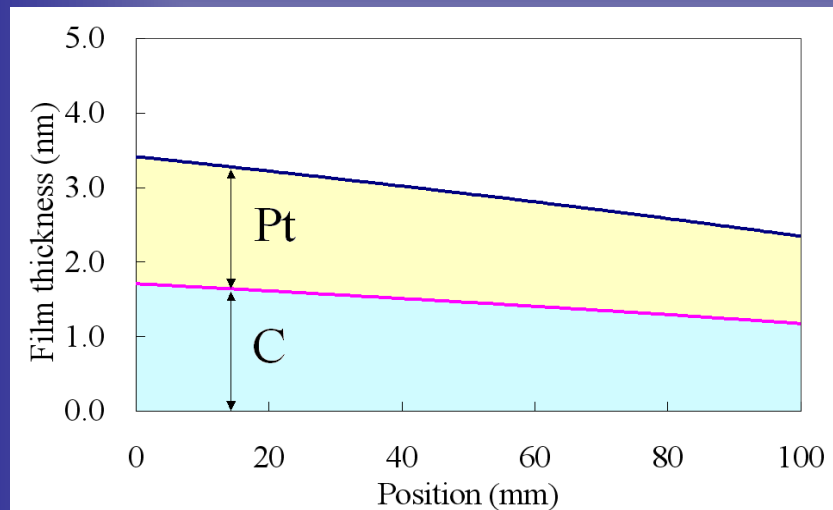
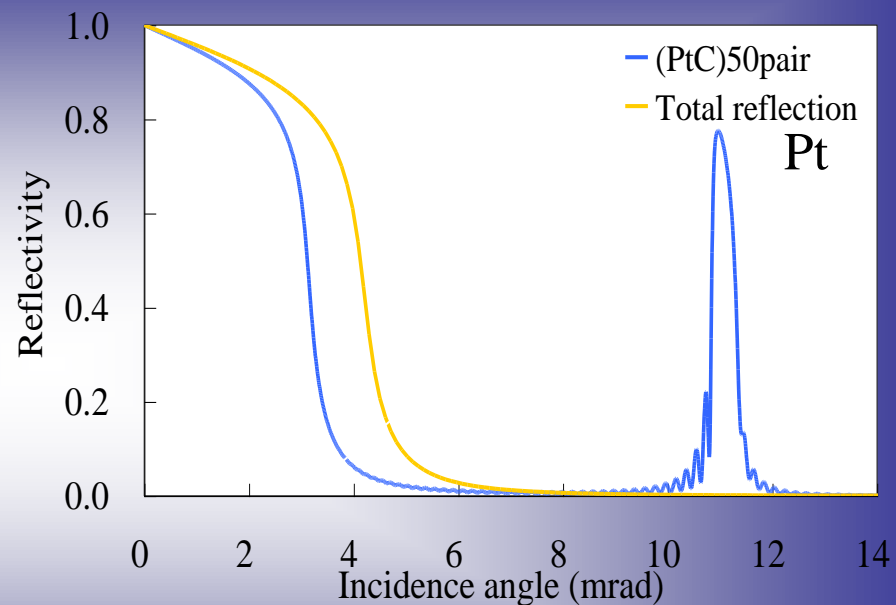


Figure error of 1nm is not allowable

Multi-layer technology is needed to realize large NA



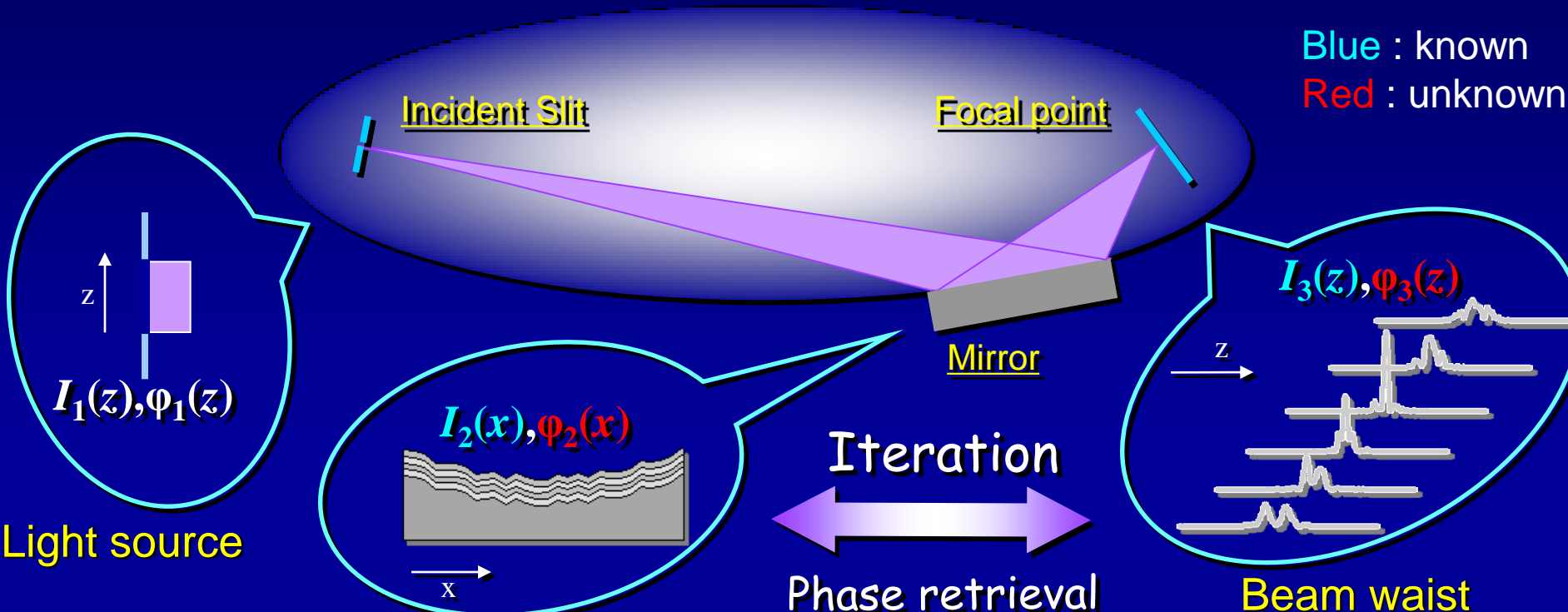
Graded multi-layer



Reflectivity

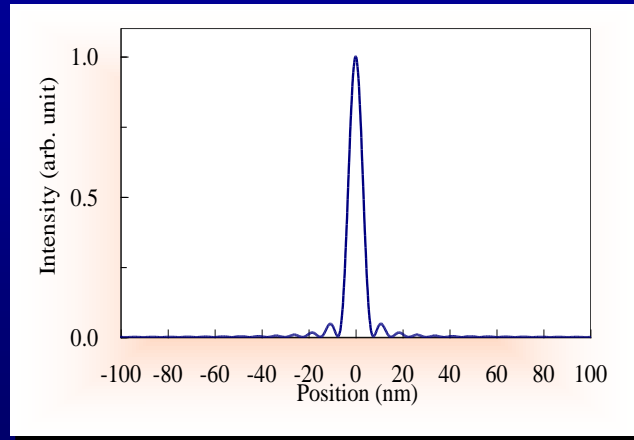
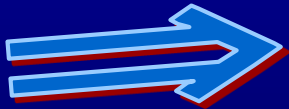
Not only figure error but also thickness deviation of the multilayer induce wavefront phase error.

At-wavelength phase-retrieval interferometry



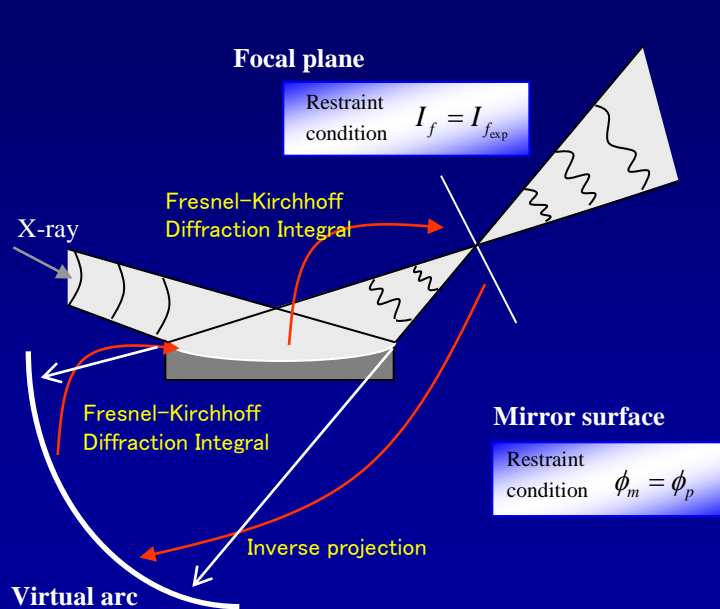
Mirror surface
(Phase error includes surface figure and ML thickness errors)

Phase error compensation



K. Yamauchi,
SPIE's Optics and Photonics 2005

Phase retrieval properties



On focal plane

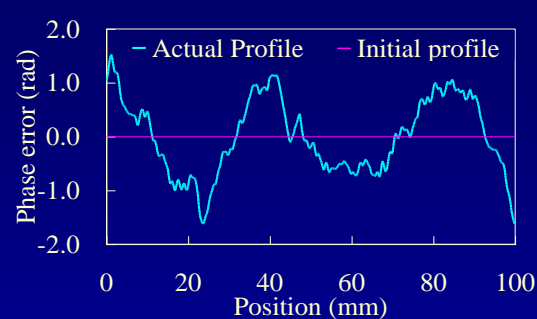
Intensity is changed to experimental value.

Phase is kept to be recovered value.

On mirror surface

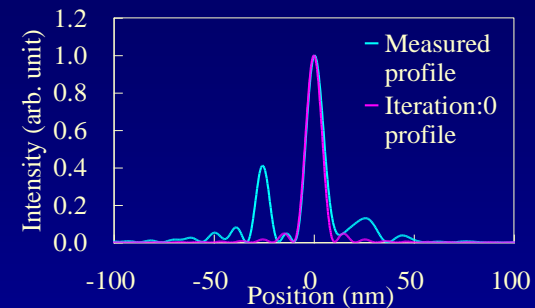
Intensity is changed to theoretical value.

Phase is kept to be recovered value.

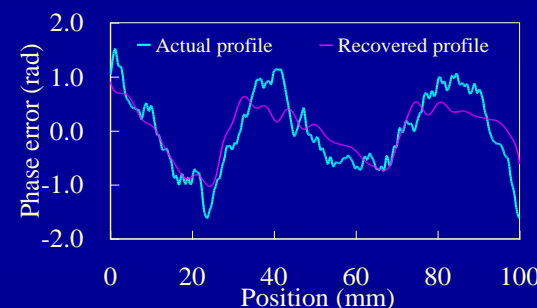


Wavefront errors

Iteration 10

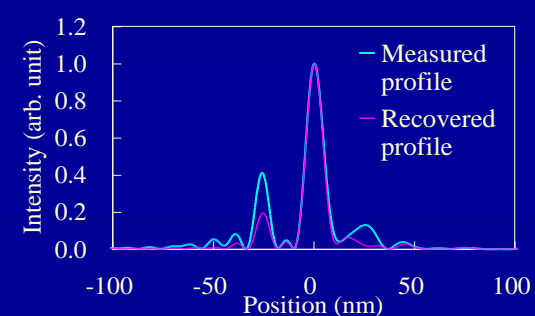


Intensity profiles

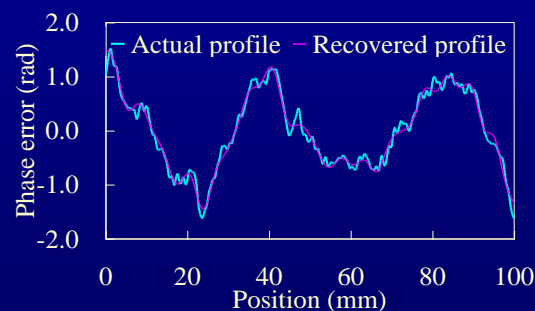


Wavefront errors

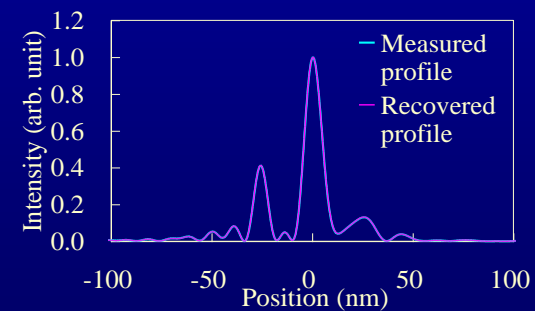
Iteration 1000



Intensity profiles



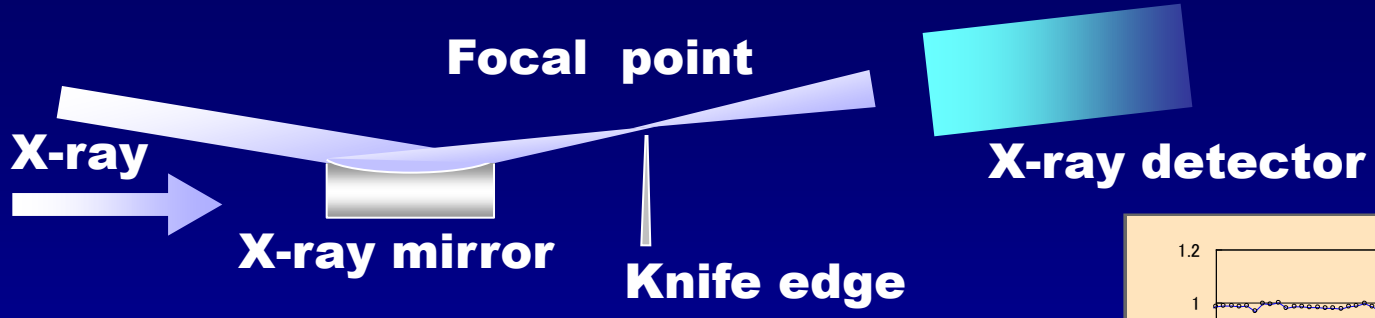
Wavefront errors



Intensity profiles

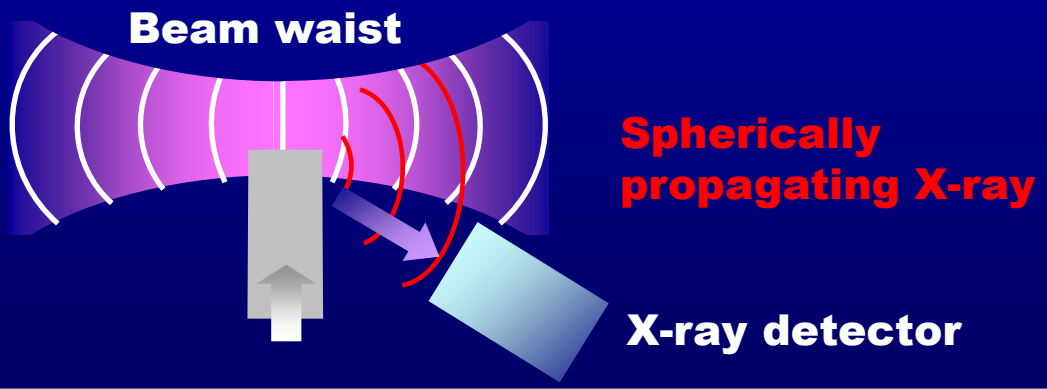
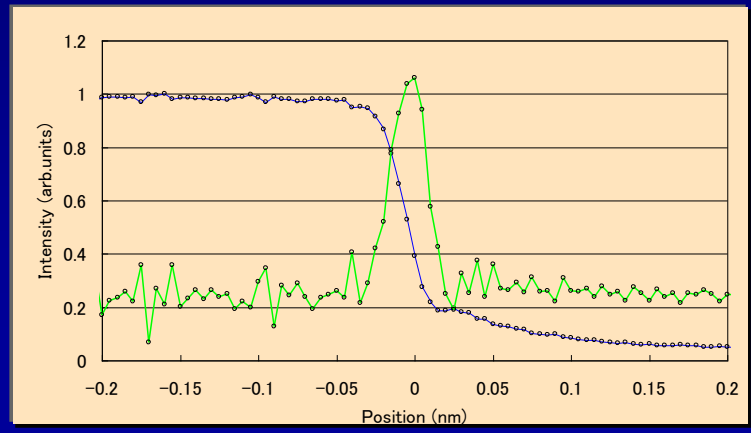
Yumoto et al.,
Rev. Sci. Instrum. (2006)

New knife-edge method



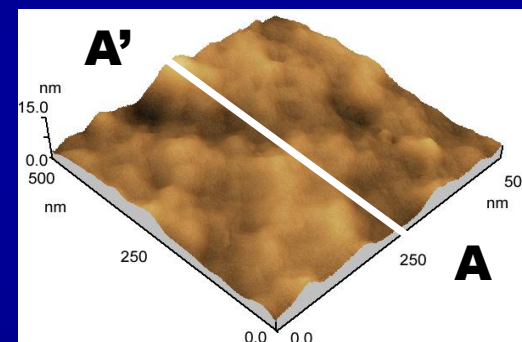
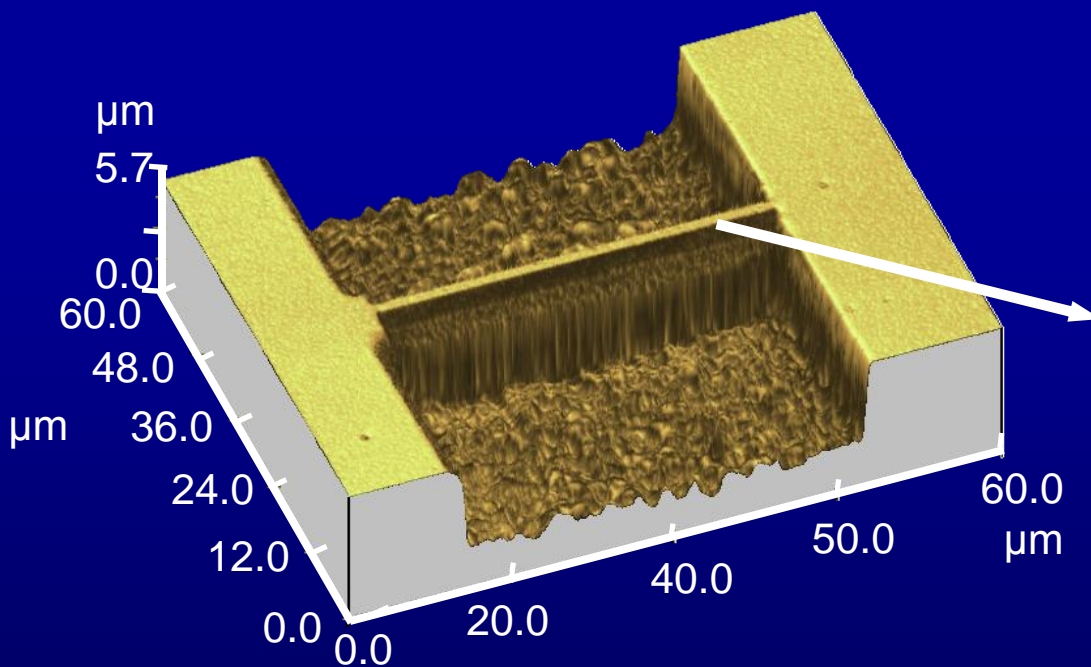
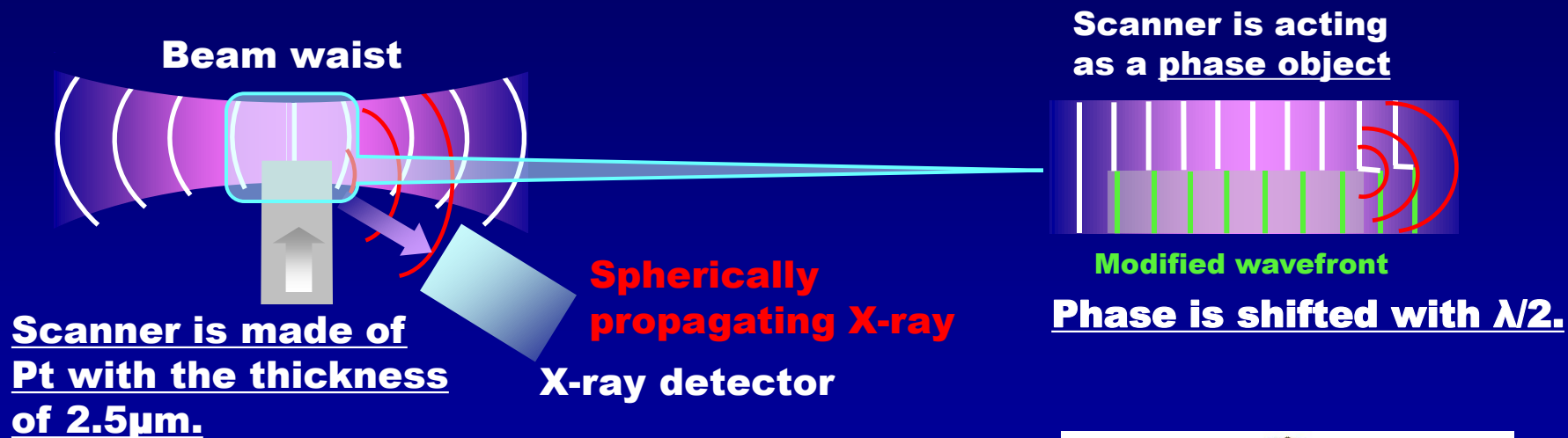
Conventional knife-edge method

New method

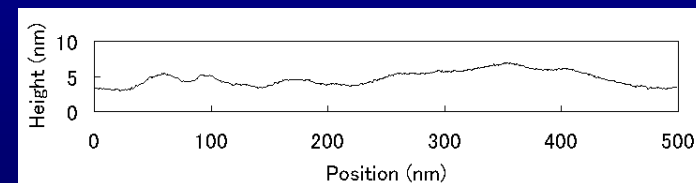


Mimura et al.
Phys. Rev. A (2008)

Details of the new knife-edge method

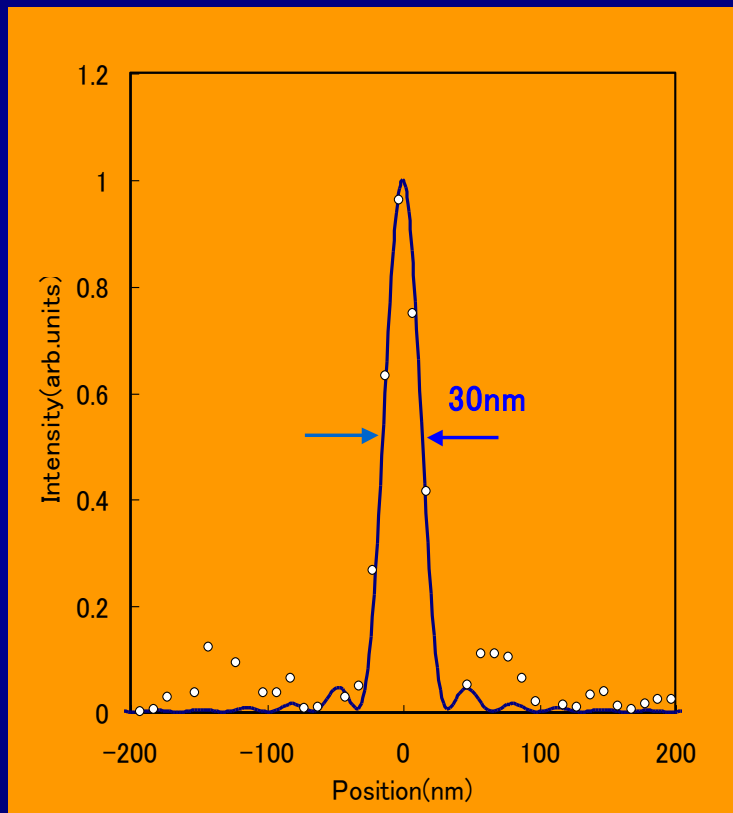


Microroughness at the bridge



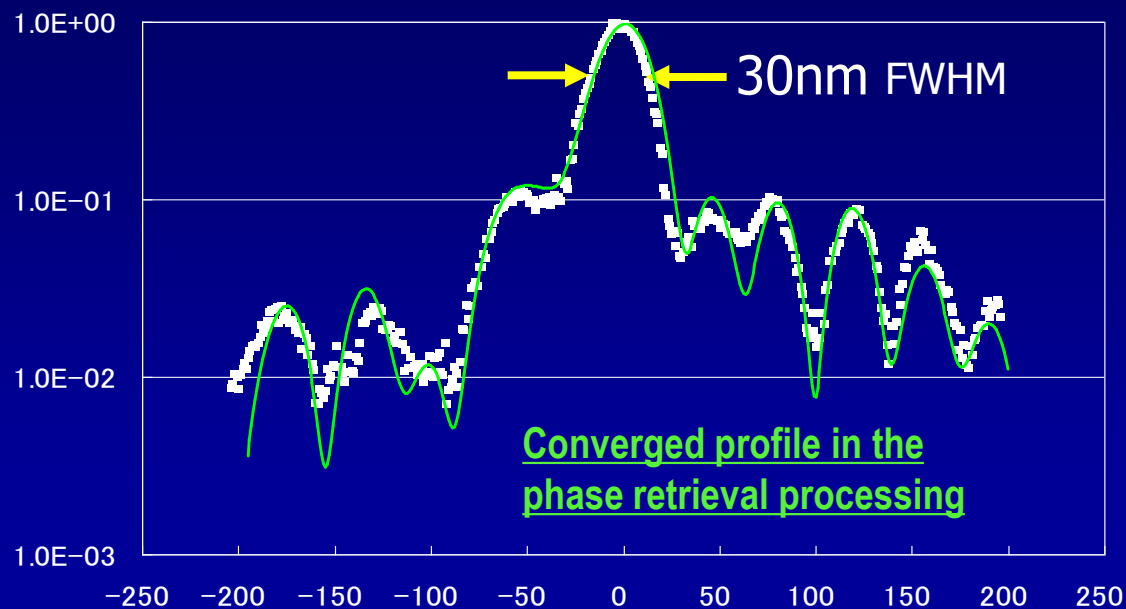
A-A' profile

30nm-focusing mirror was employed for a demonstration of the proposed at-wavelength measurement

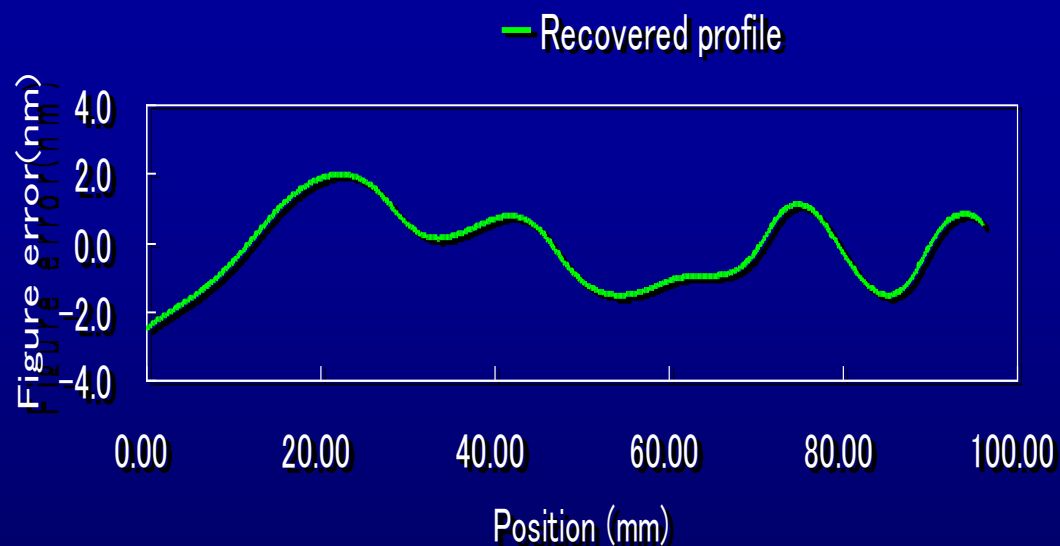


- Not a multilayer optic.
- Surface is coated by Pt.

Performance of phase retrieval

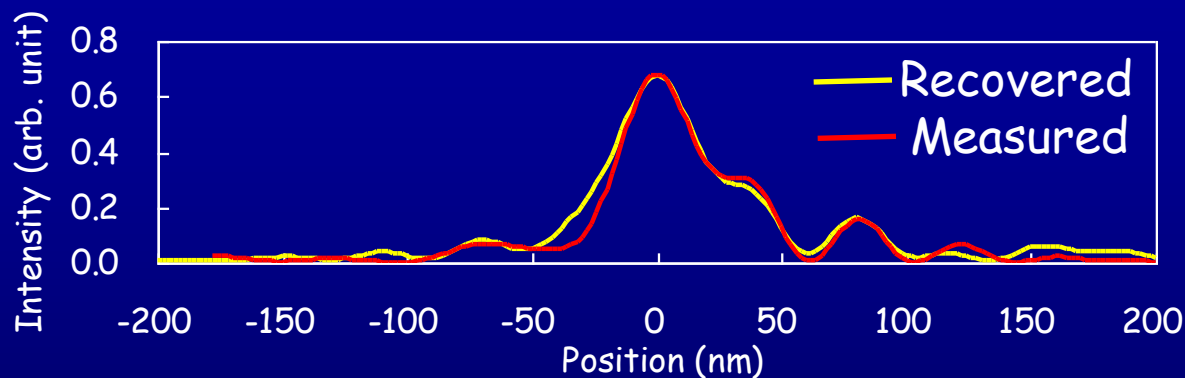
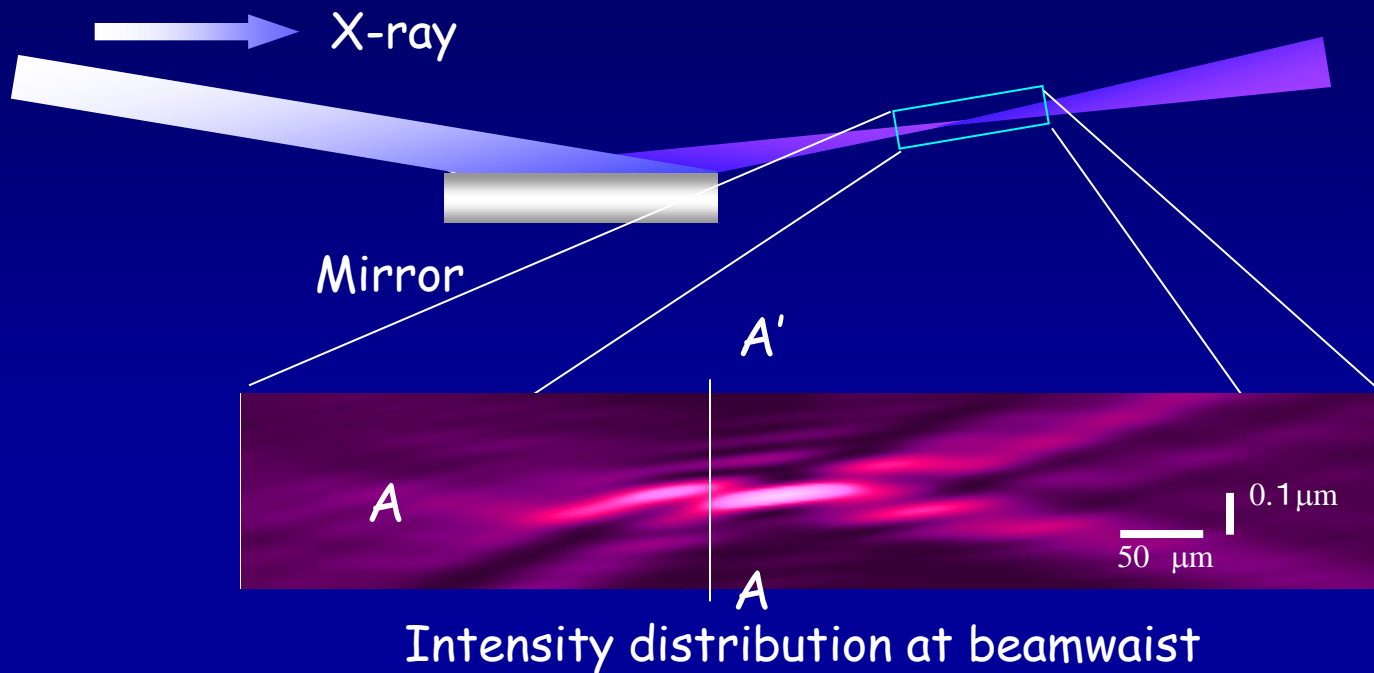


Measured beam profile



Mimura et al.
Phys. Rev. A (2008)

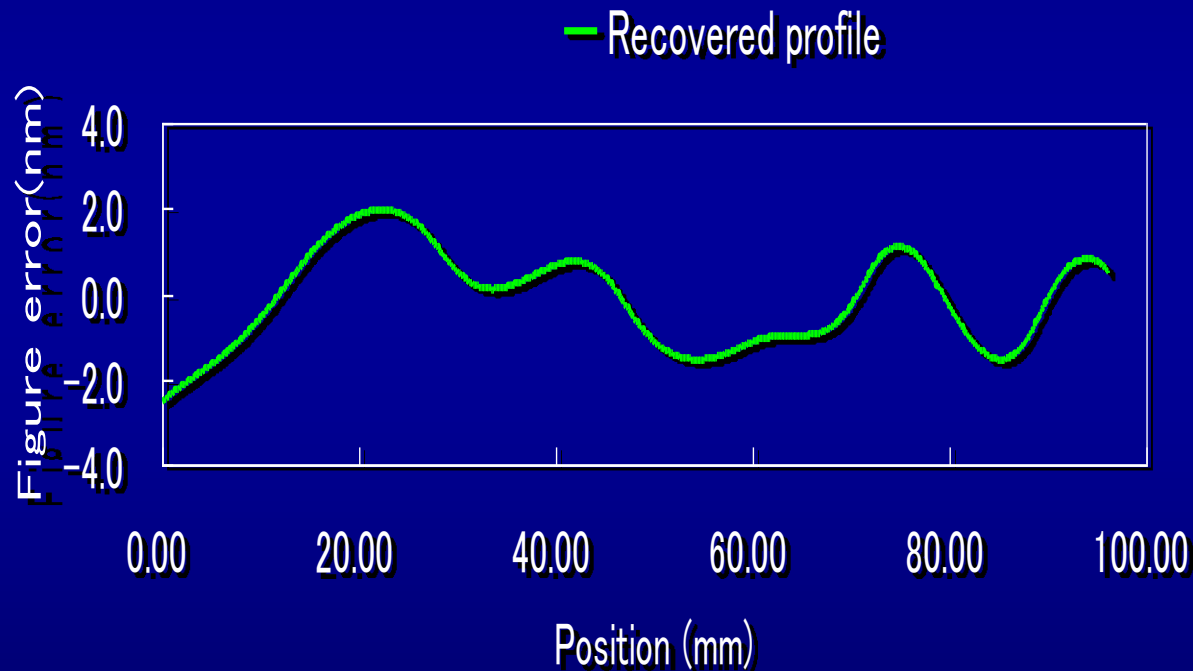
Recovered wavefield is local-minimum solution or not



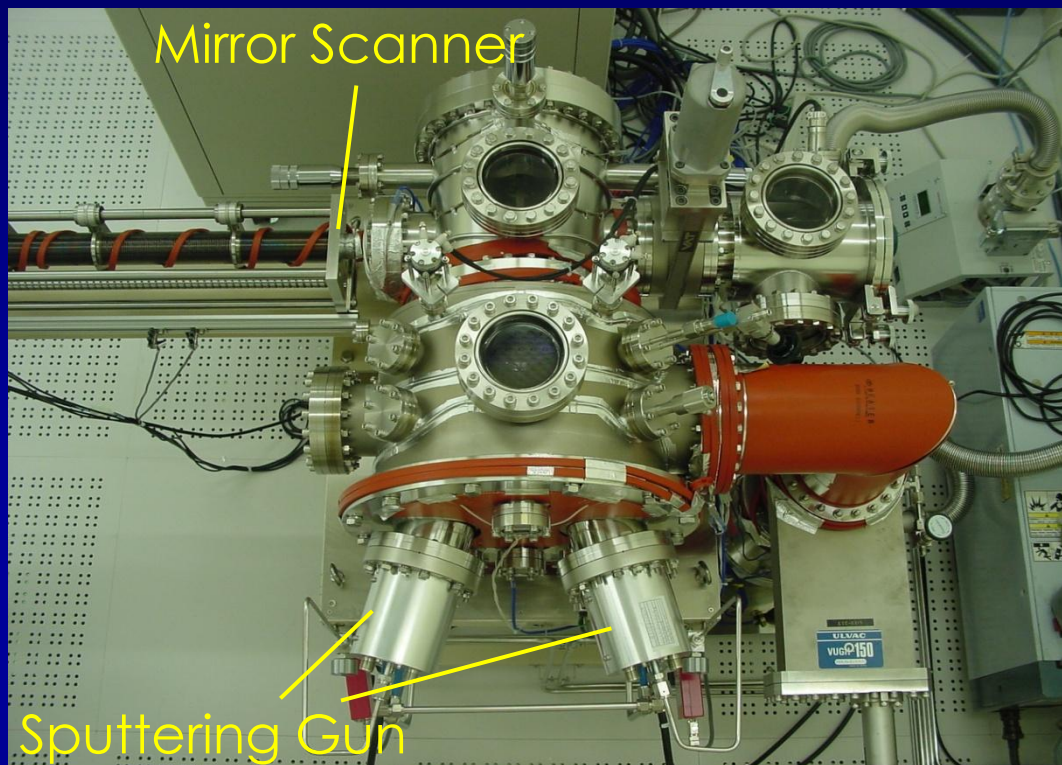
Calculated and measured intensity profiles on A-A' line.
(50 μm upstream from the focal point)

Verification

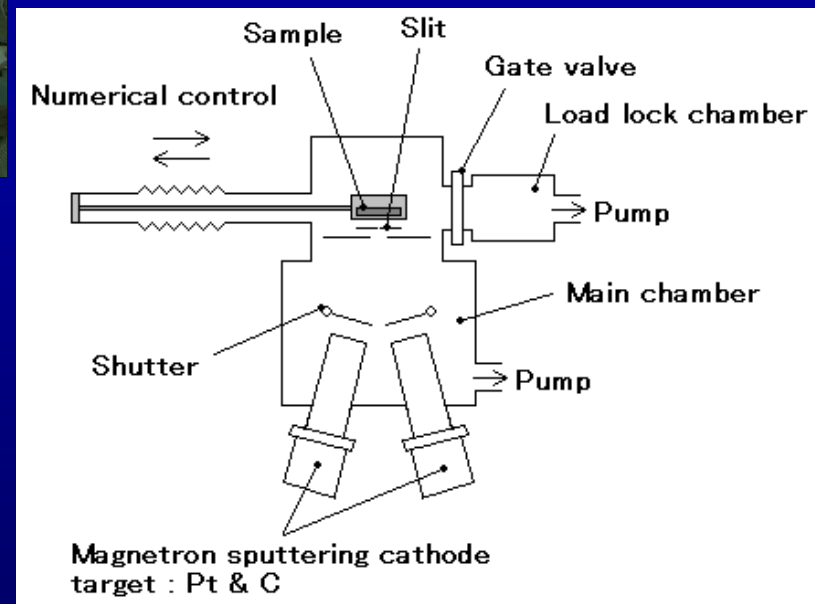
To verify the reliability of the recovered phase error profile, we actually refigured the mirror by differential deposition method (G. Ice) using the recovered profile.



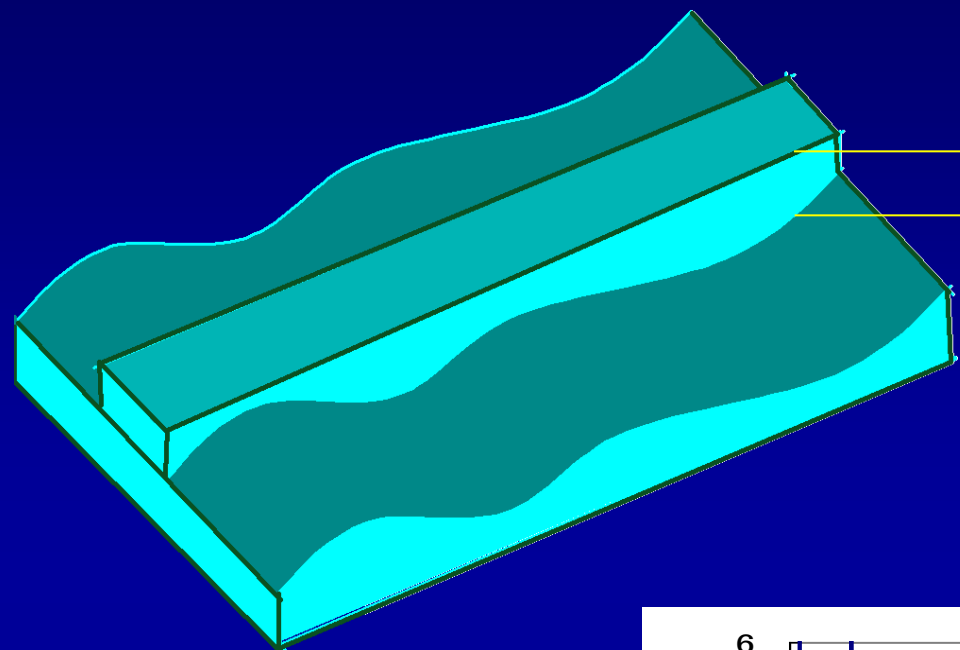
Computer-controlled deposition system



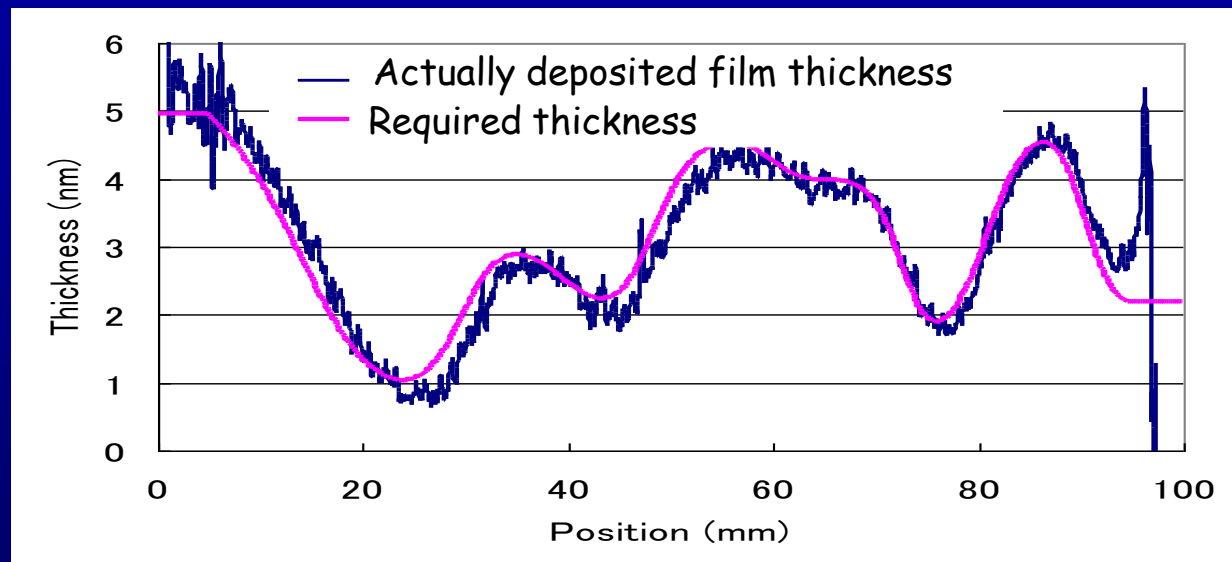
Mirror is placed on the computer-controlled stage, and local deposition can be done through the slit.



Figuring by differential deposition

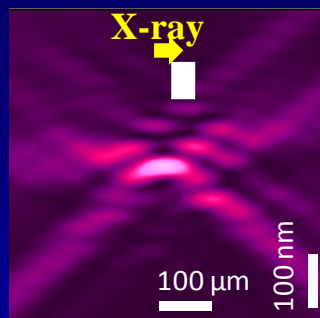


Additionally deposited film thickness using differential deposition method

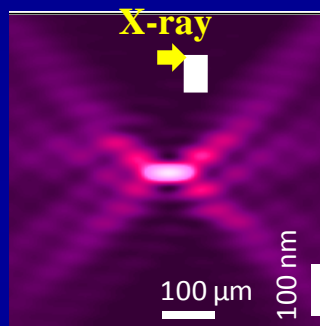


Focused beam profiles before and after DD

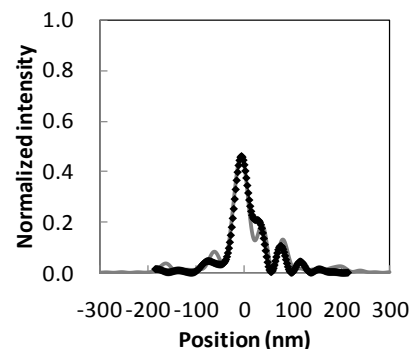
Before DD



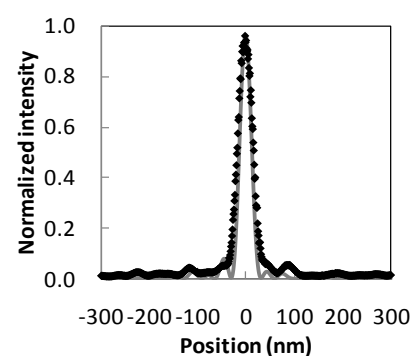
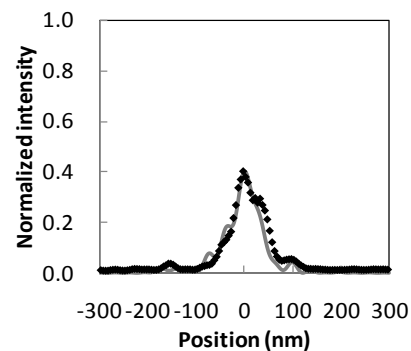
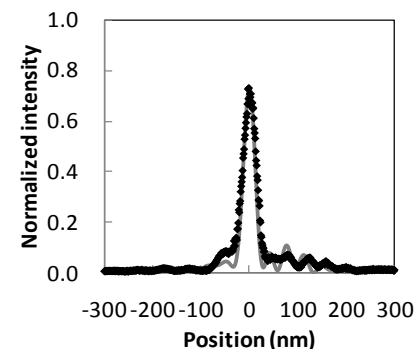
After DD



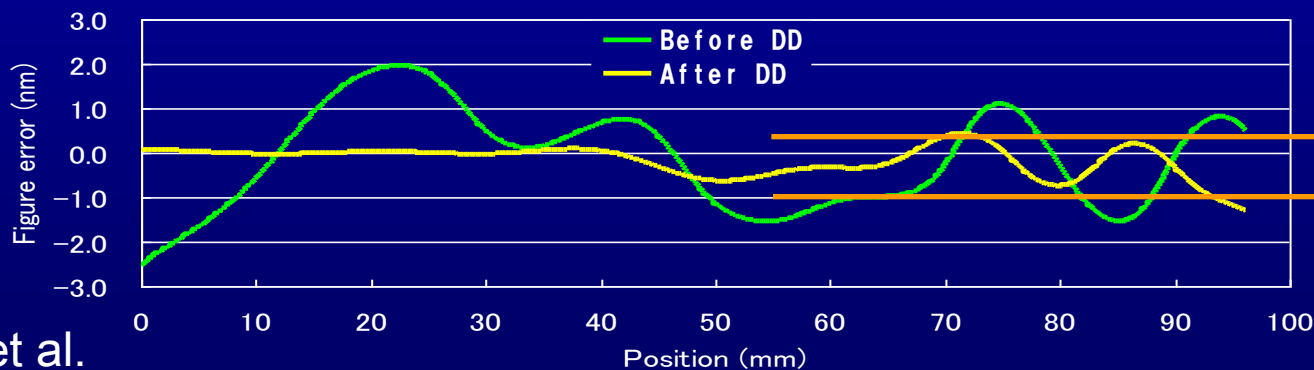
@50 μm upstream



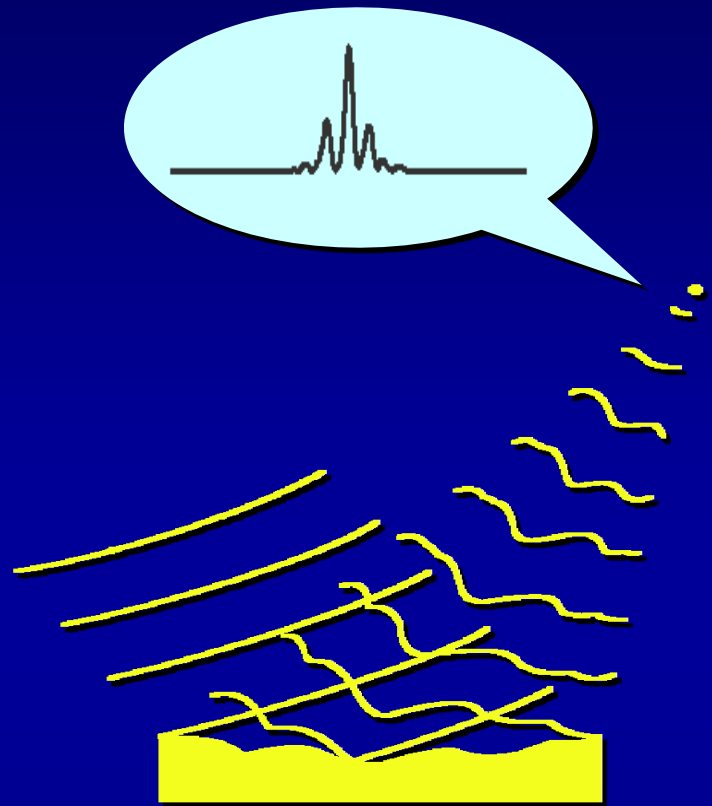
@focal plane



Comparison between the wave fields before and after phase compensation



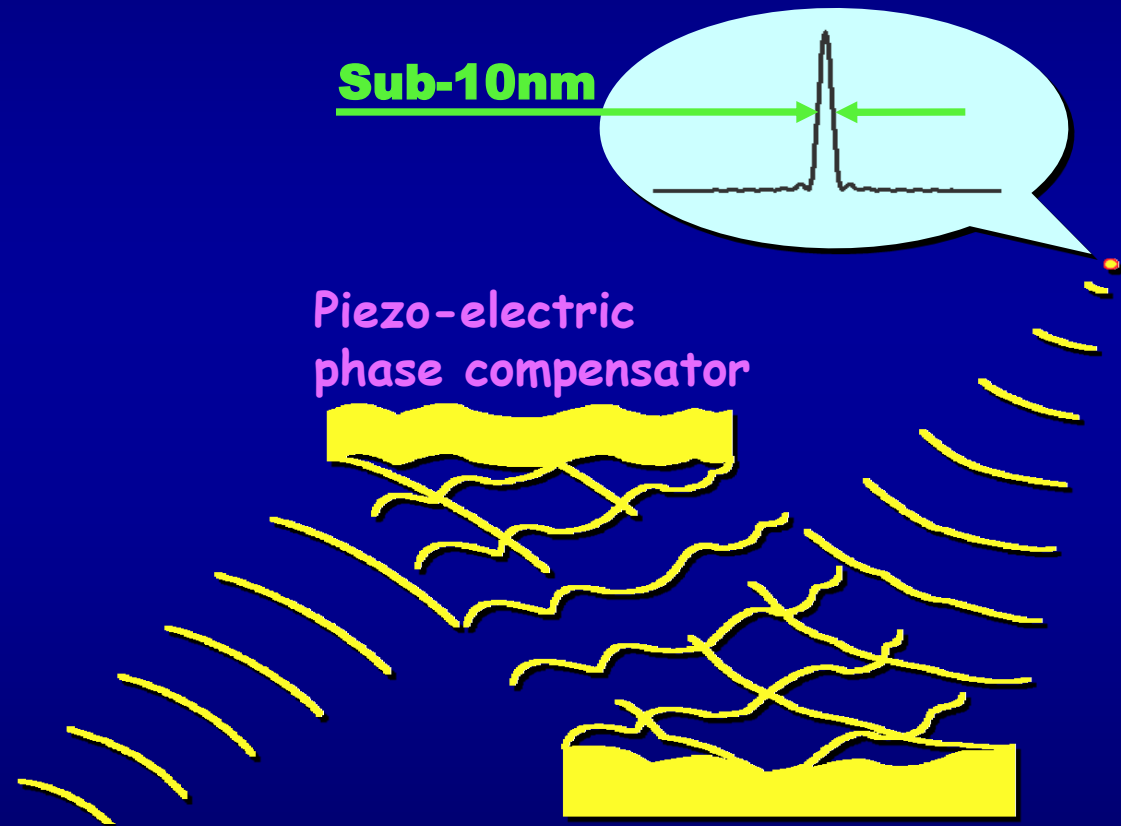
On-line compensation of wavefront



Focusing mirror with phase error

In-situ phase compensation

Sub-10nm

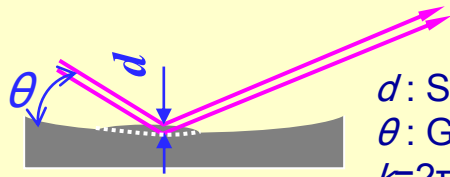


Piezo-electric
phase compensator

Focusing mirror with phase error

Design concept

$$\text{Phase error} = 2kd \sin \theta$$



d : Shape error
 θ : Glancing angle
 $k=2\pi/\lambda$: Wave number

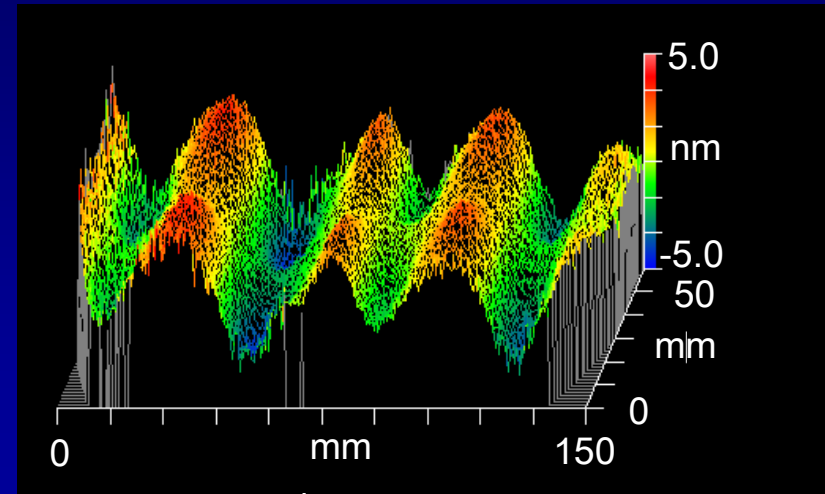
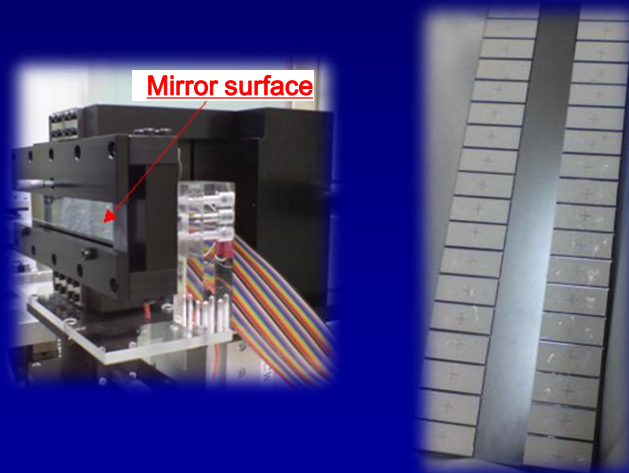


- ★ Glancing angle of compensator mirror is N times smaller.
 (However, Consequently the length of the compensator becomes longer)

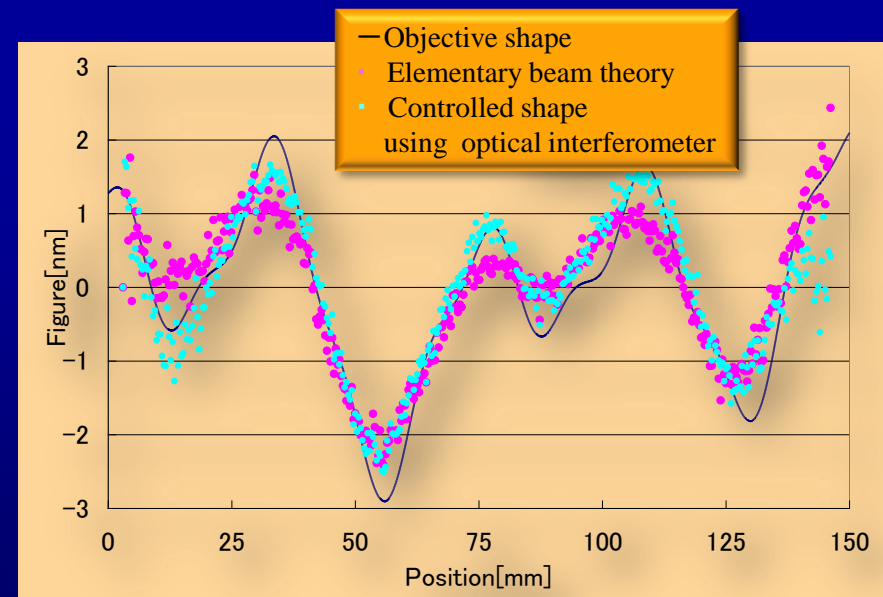
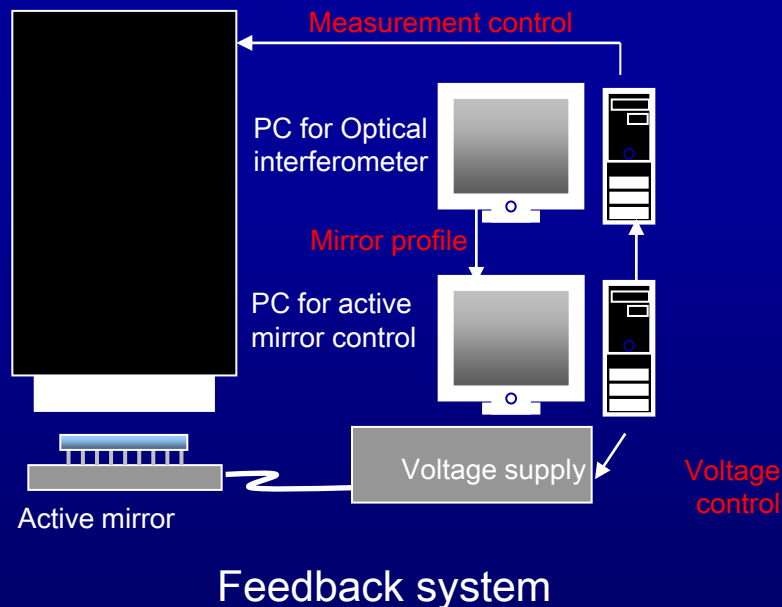


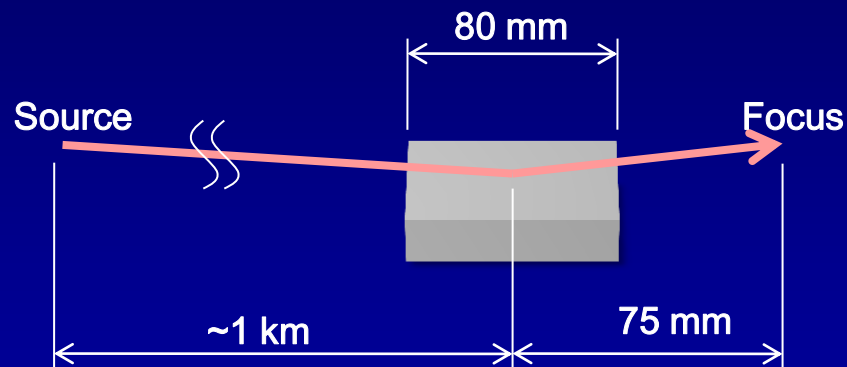
Required figure accuracy of the compensator mirror becomes N times lower.

Phase compensator



Optical interferometer





X-ray energy : 20 keV
Mirror length : 80 mm
Focal distance : 75 mm
Glancing angle : 7.0 mrad
Multilayer material : [Pt/C]₂₀
Substrate material : quartz glass

$$\Lambda = \frac{\lambda}{2\sqrt{n^2 - \cos^2 \theta}}$$

Λ : d-space

λ : X-ray wavelength

n : Index

θ : Glancing angle



EEM Machine

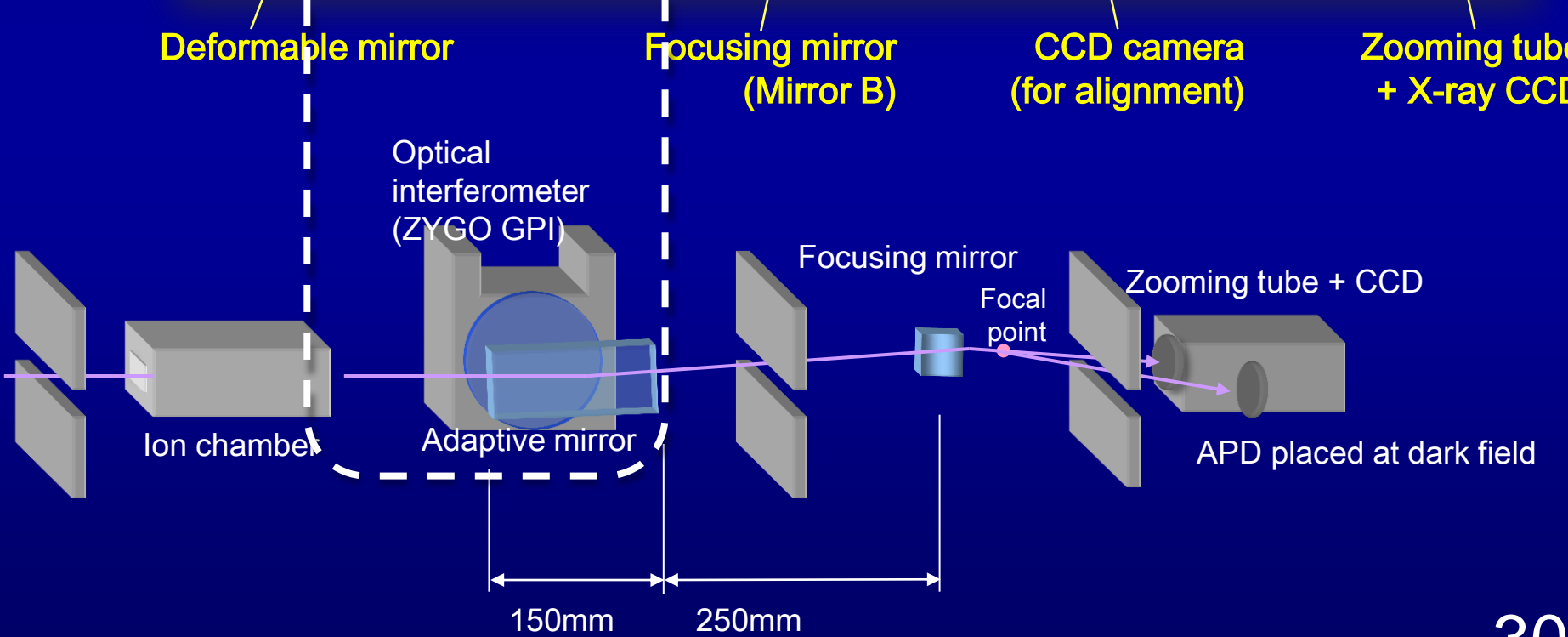
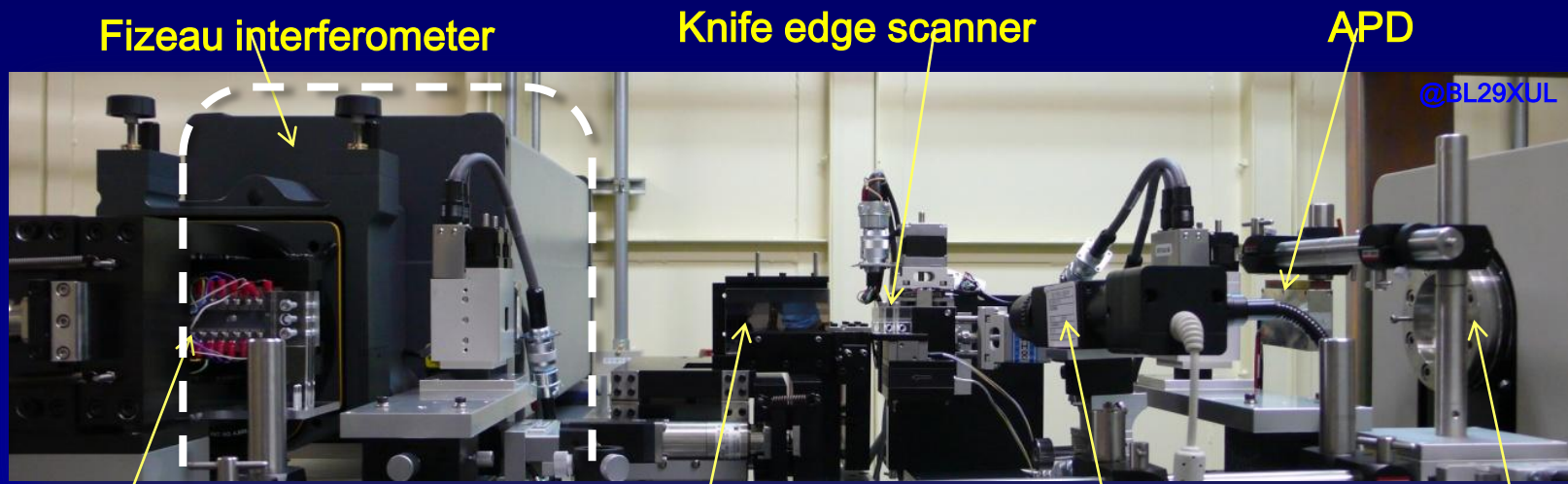


Micro- and RAD- Stitching
Optical Interferometry

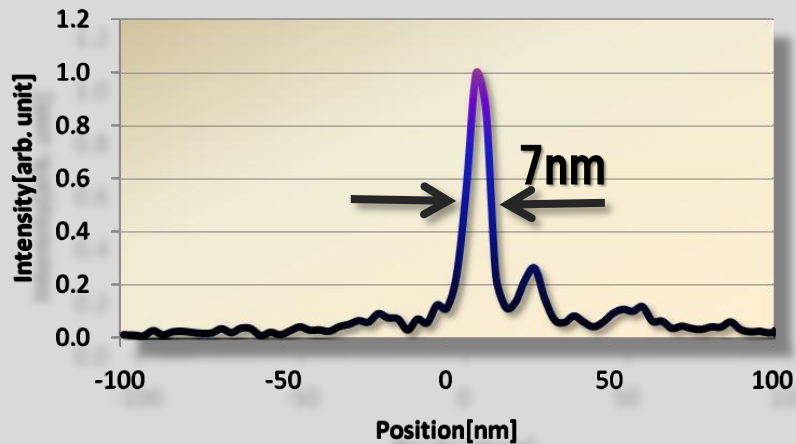


Laterally-Graded
Multilayer Coater

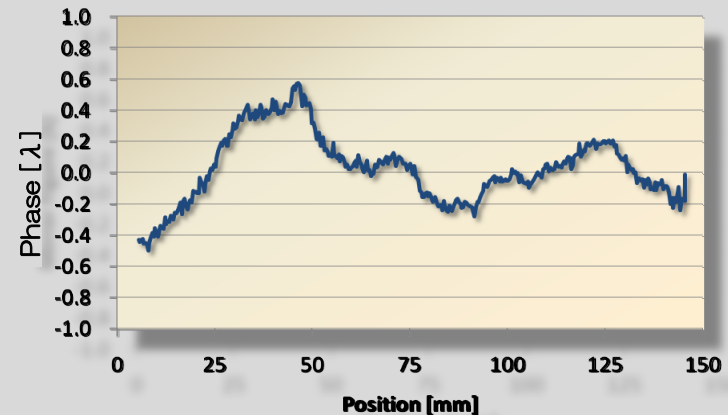
Optical configuration for active phase compensation



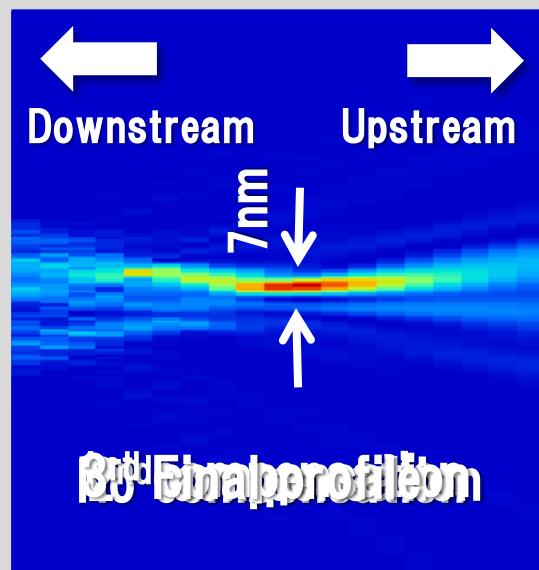
Sub-10nm focusing by using phase compensator



Profile at focal point



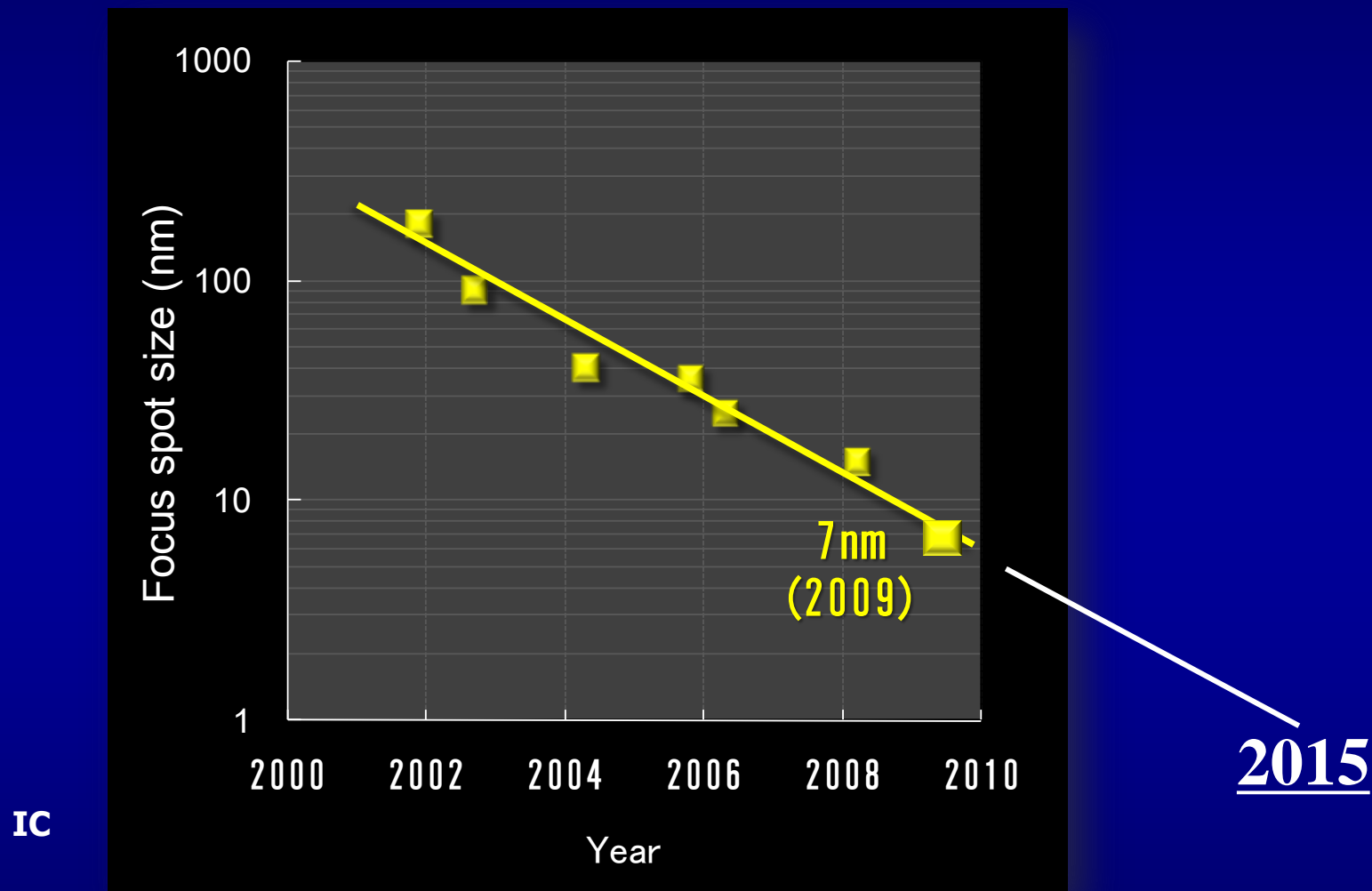
Compensated phase profile
(shape of compensation mirror)



Maximum phase compensated here was $\lambda/2$.
 λ was 0.06 nm.

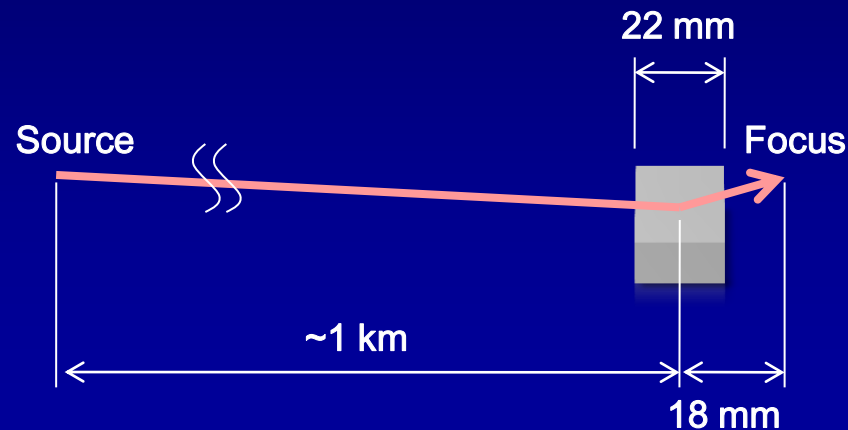
Beam waist structure

Nature Phys. (2010)

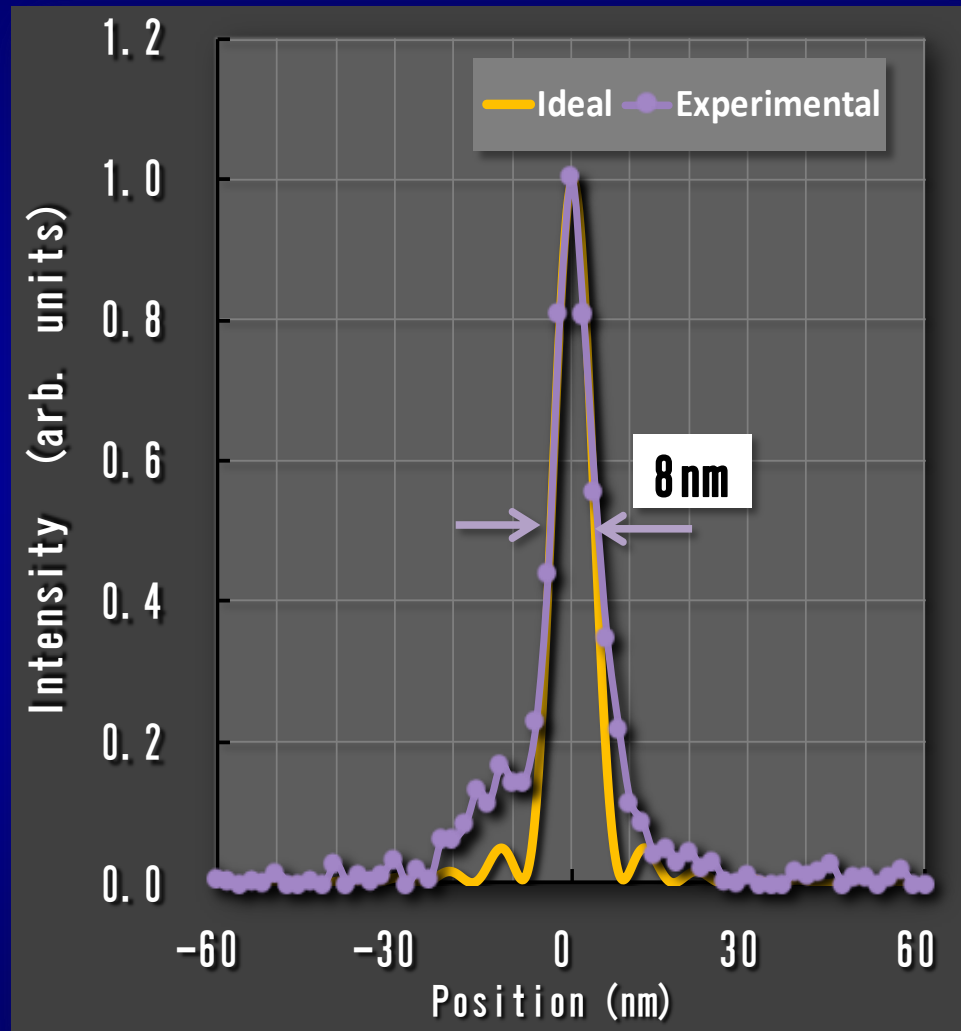


This is the smallest light beam human-made.

Another mirror



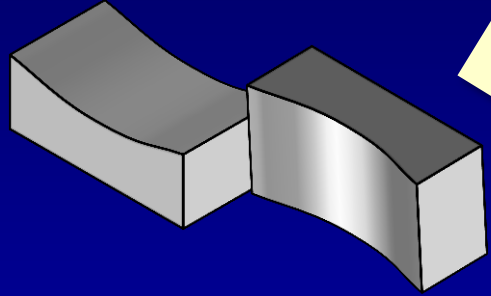
X-ray energy : 20 keV
 Mirror length : 22 mm
 Focal distance : 18 mm
 Glancing angle : 7.0 mrad
 Multilayer material : [Pt/C]₂₀
 Substrate material : quartz glass



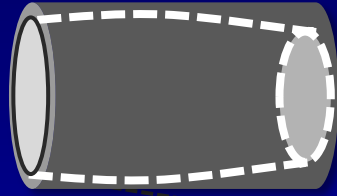
Achromatic imaging device (AKB Mirrors)

Total reflection axial-symmetric optics

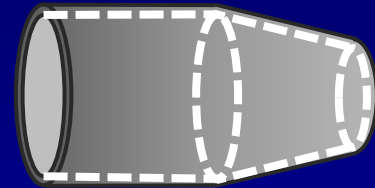
KB mirrors



easy to fabricate



Focusing



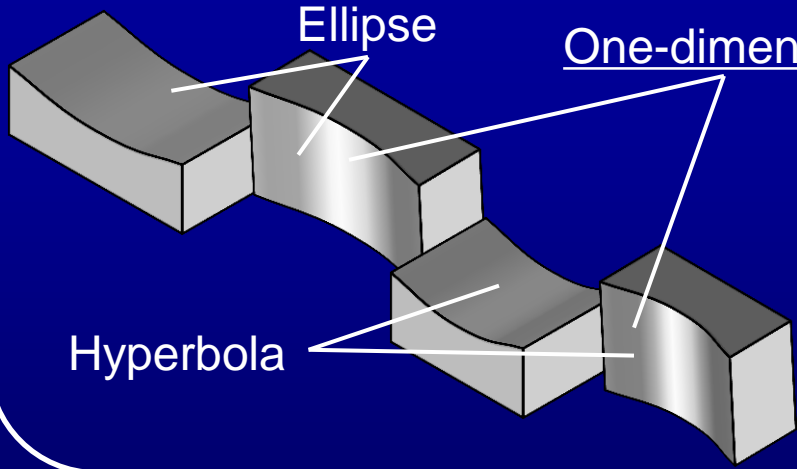
Imaging

Wolter mirror

It fills Abbe's sine condition

Advanced Kirkpatrick-Baez mirrors

R. Kodama et al., *Optics Letters* 21 (17), 1321-1323 (1996).



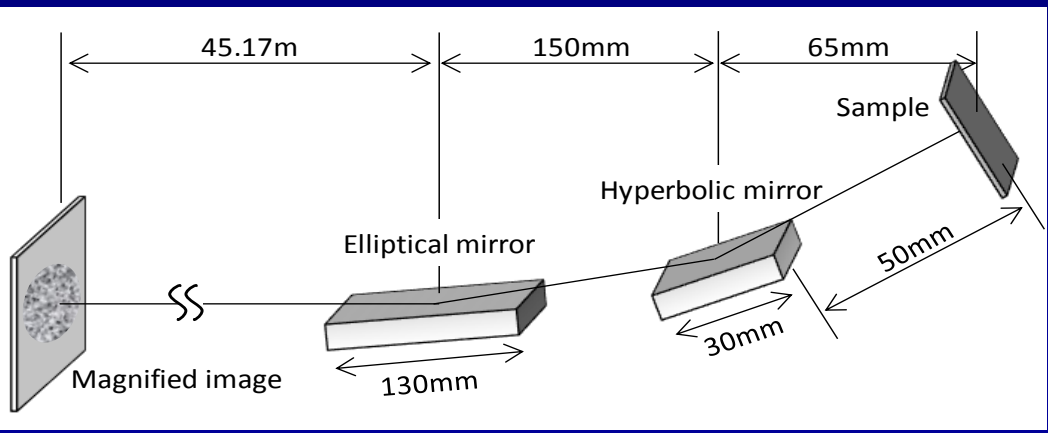
★ Advantage

- Wide field of view
- Easy fabrication

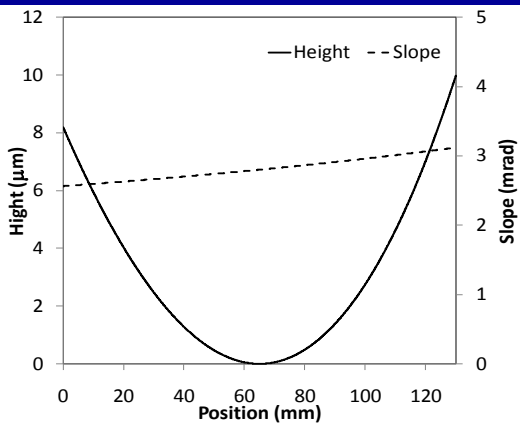
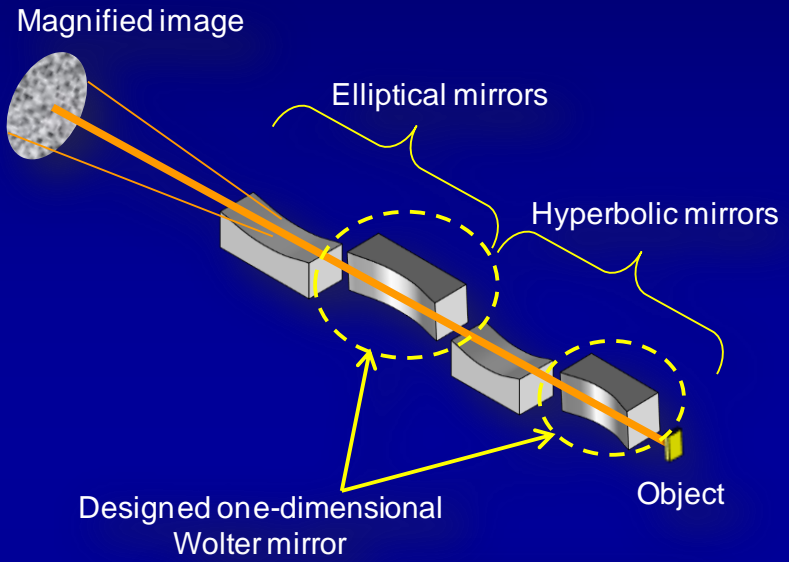
We tried to realize AKB mirrors having diffraction-limited performance

1-dimensional Wolter mirror system

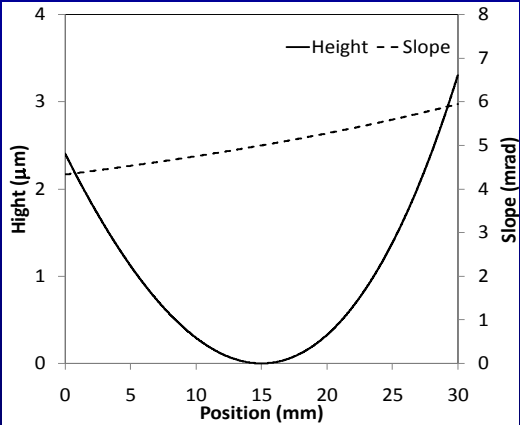
Magnification: 385x , Size of the point spread function: 43nm



Optical system of a one-dimensional Wolter optics

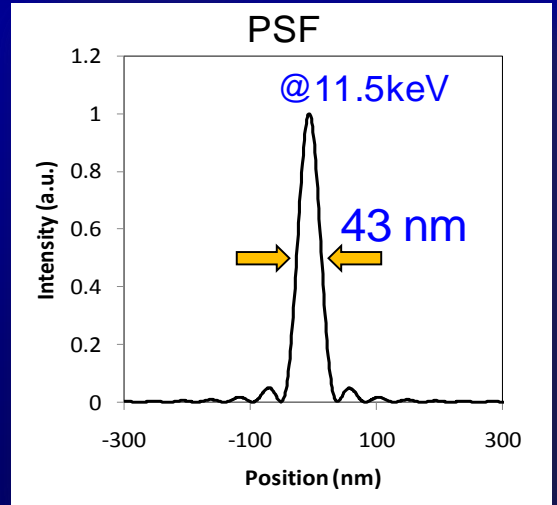


Elliptical mirror



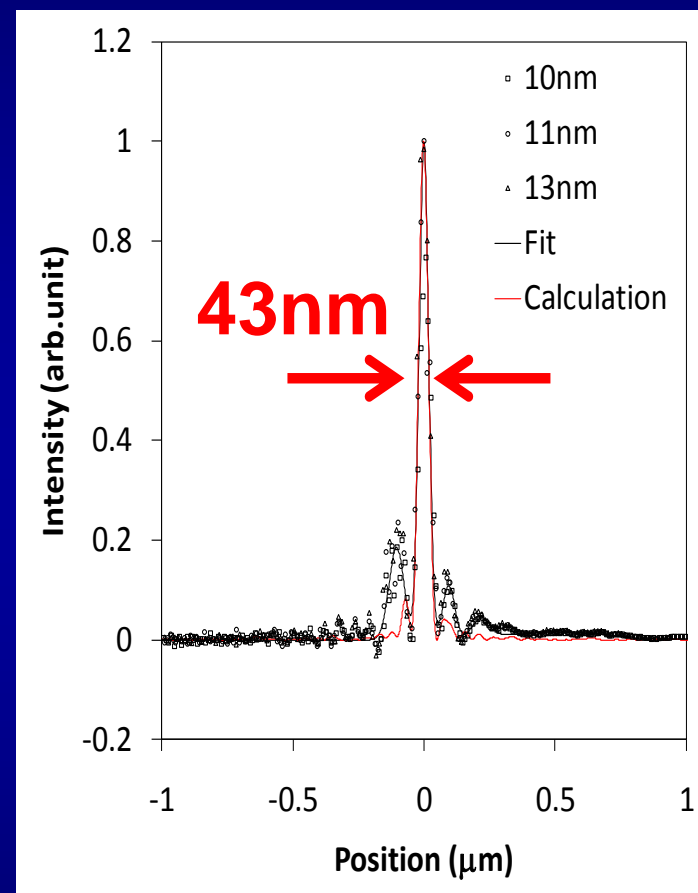
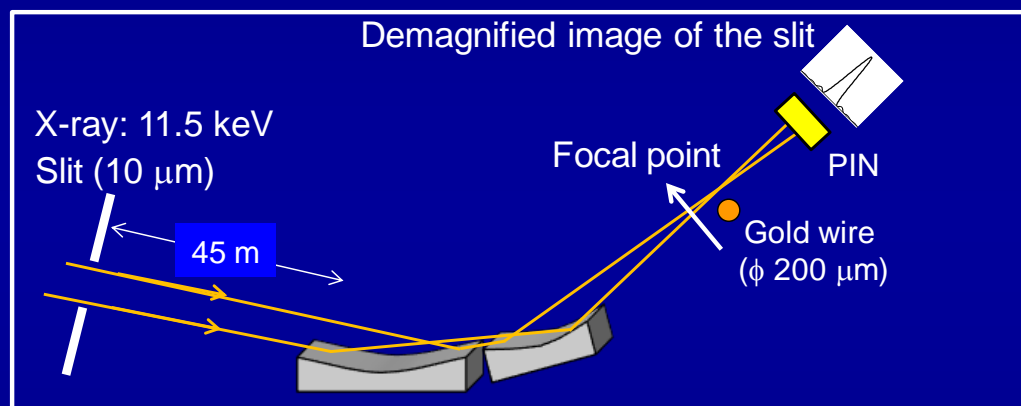
Hyperbolic mirror

Mirror figures

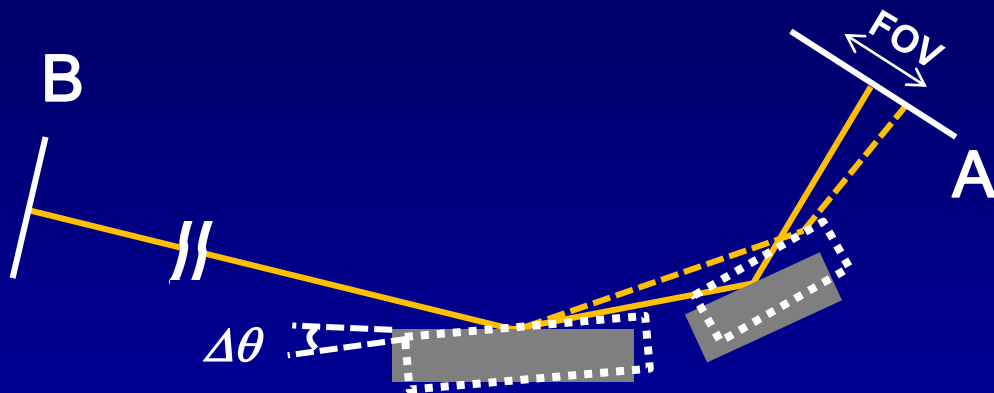


Spatial resolution test

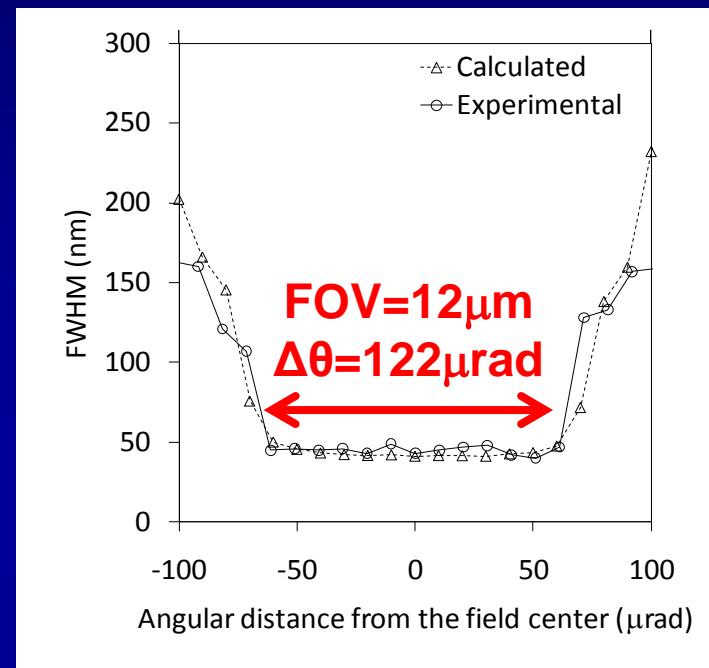
Demagnified imaging system



Evaluation of FOV

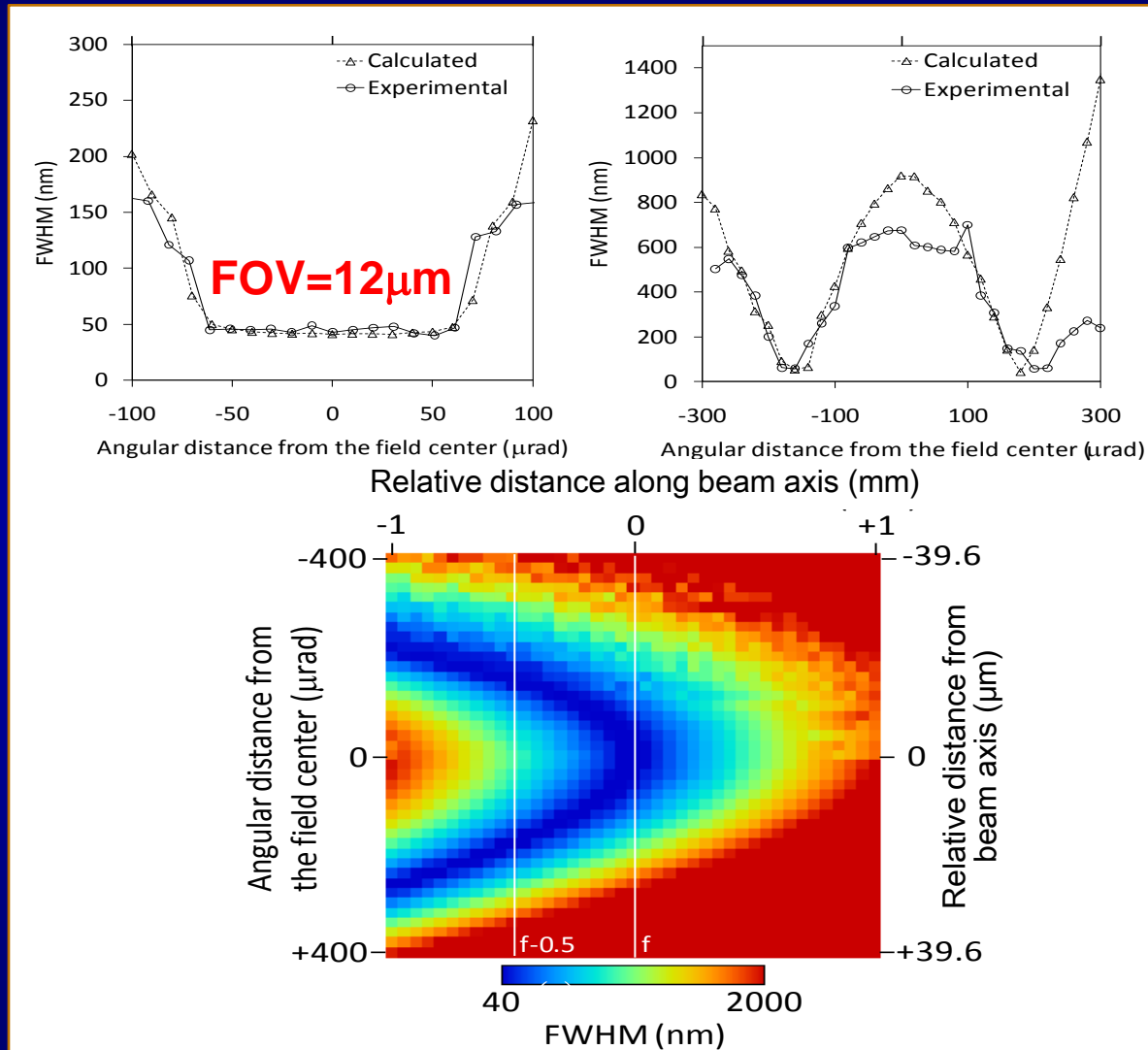


- To evaluate a field of view (FOV), we measured beam size on plane A by changing the glancing angle ($\Delta\theta$).
- This procedure is equivalent to shifting relevant points on the planes A and B.

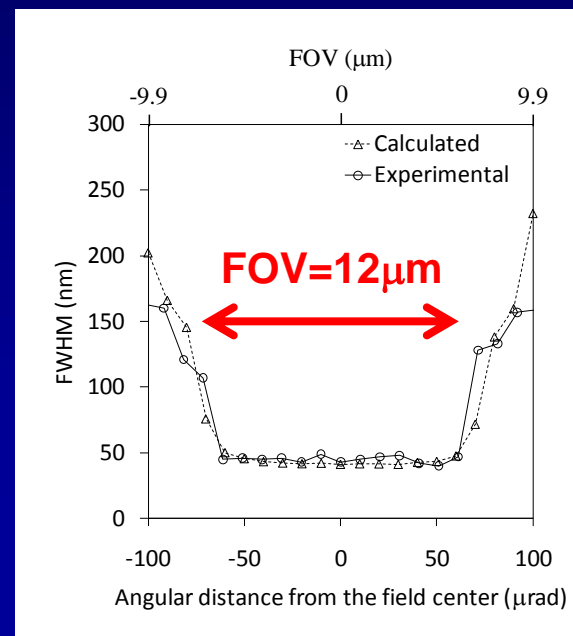
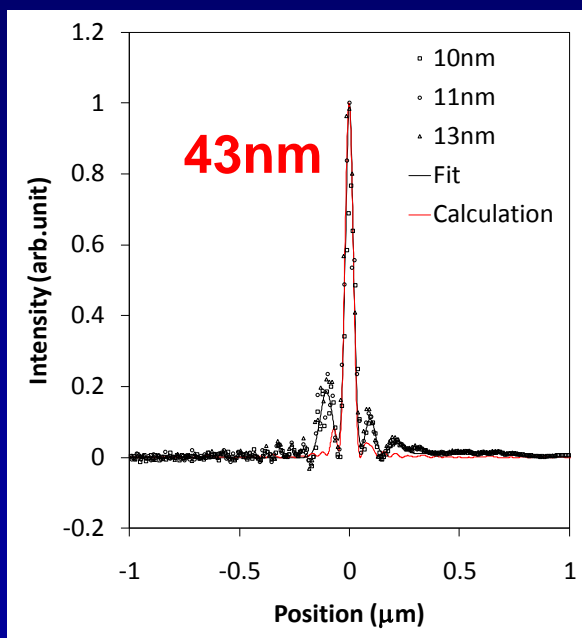


- ◆ Very wide angular width ($\Delta\theta$) of 122 μrad was obtained.
- ◆ It is equivalent to the FOV of 12 μm .

Field curvature aberration



Summary of AKB development

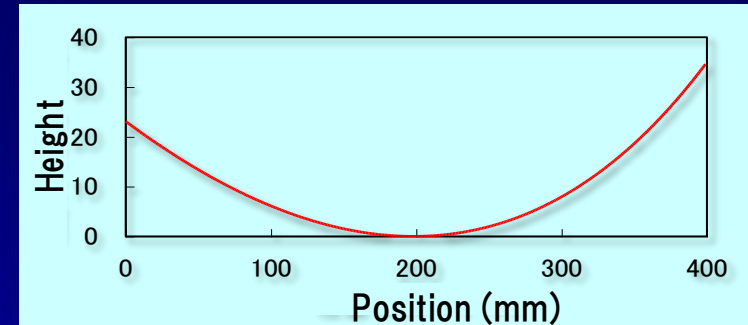
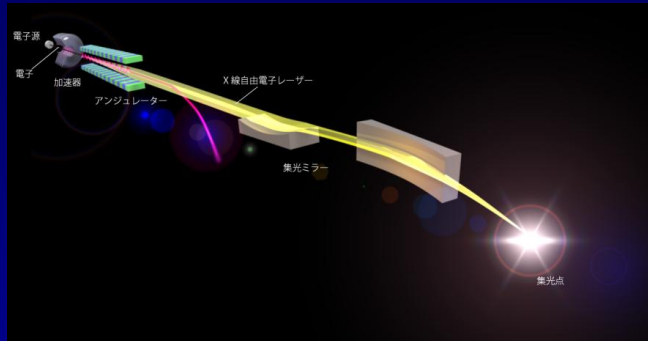


1-dimensional Wolter mirror demonstrated theoretically expected performances both in the resolution and FOV!

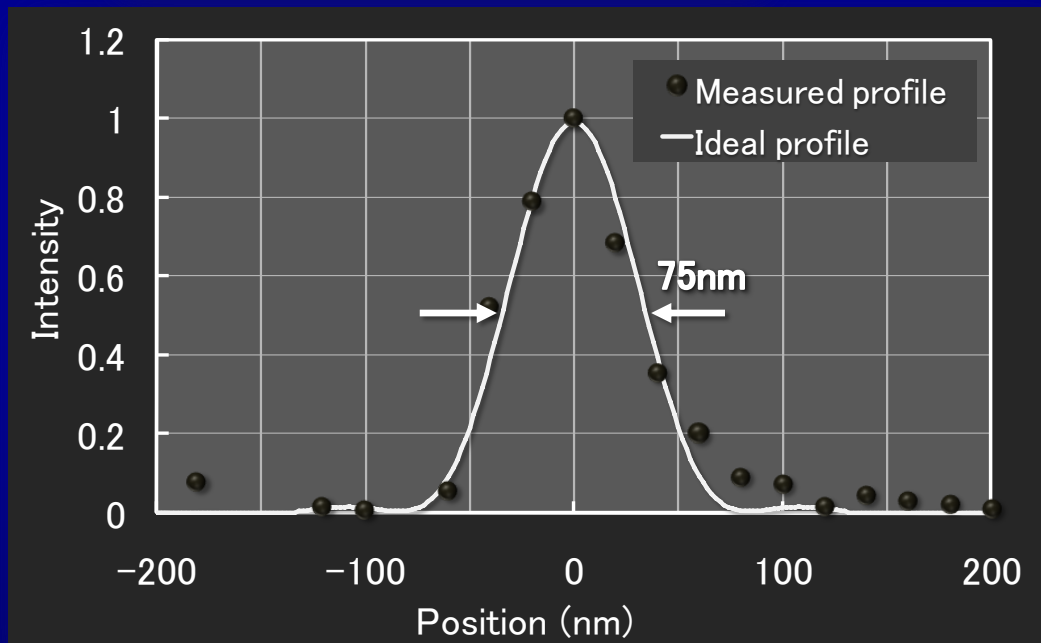
Matsuyama et al., Optics Lett (2010)

AKB optics will be useful in coming XFEL experiment.

XFEL focusing



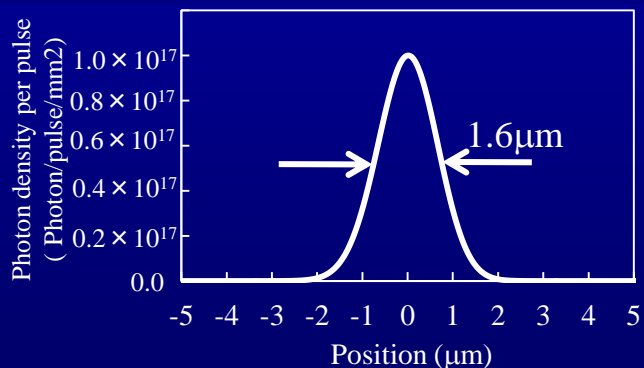
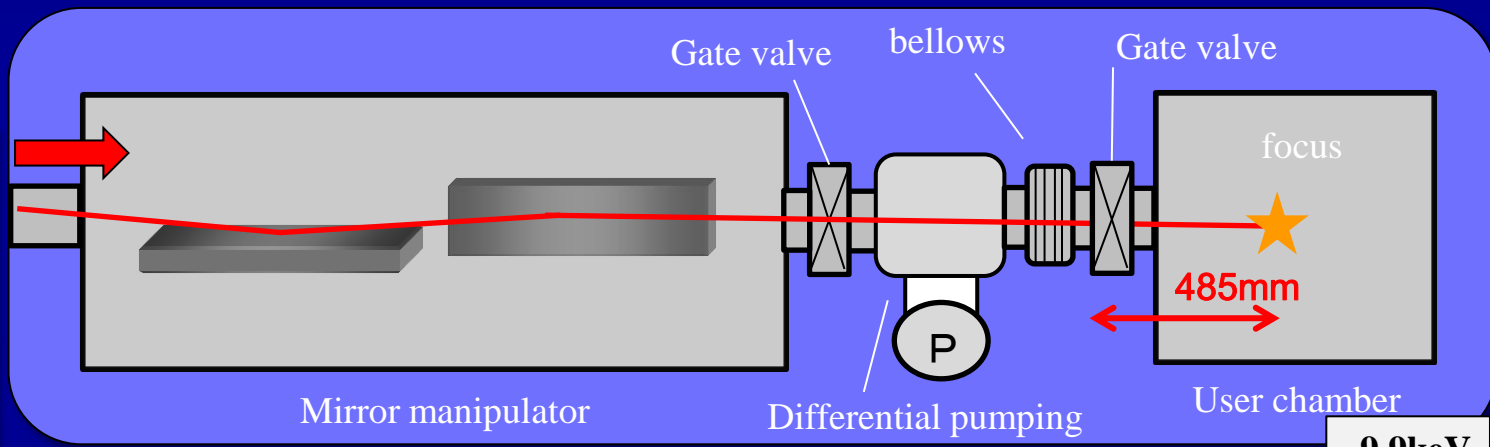
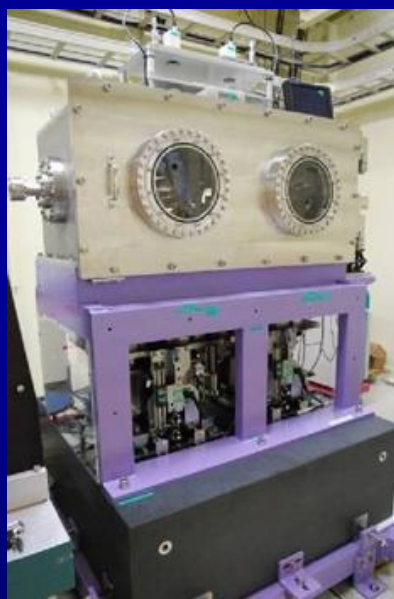
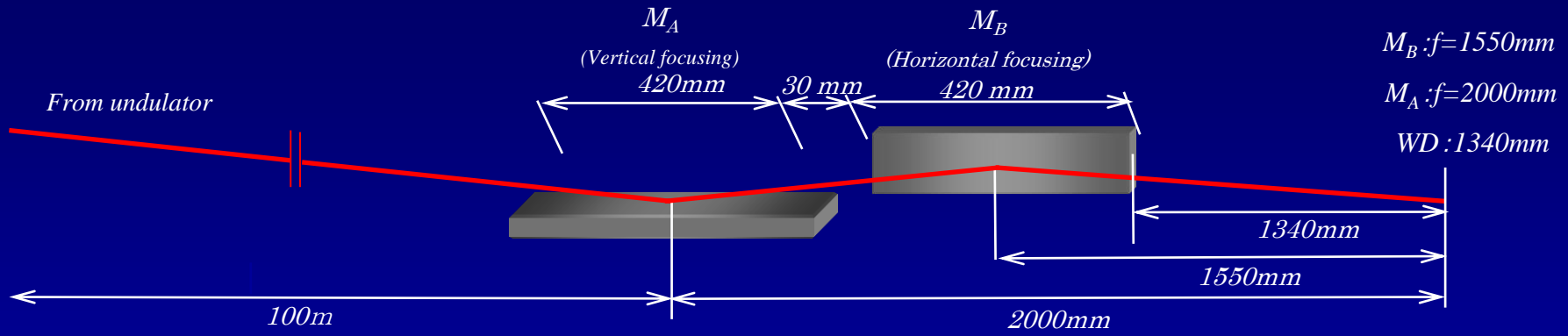
WD: 350mm



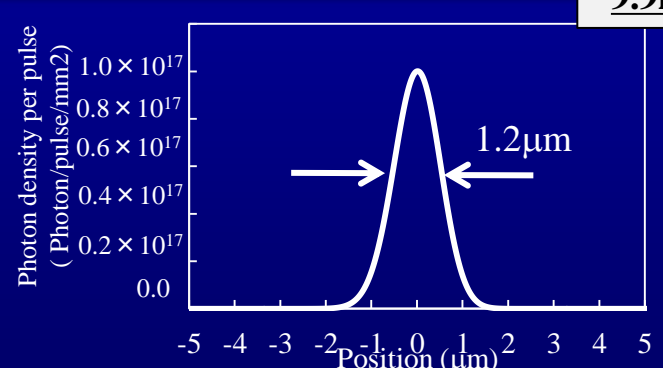
We have already developed $1\mu\text{m}$ -level focusing unit for day-1 system of Japanese XFEL, and also fabricated and tested sub-100nm focusing mirror.

We are now trying to realize 2-dimensional sub-50nm focusing of XFEL.

1 μ m-level focusing



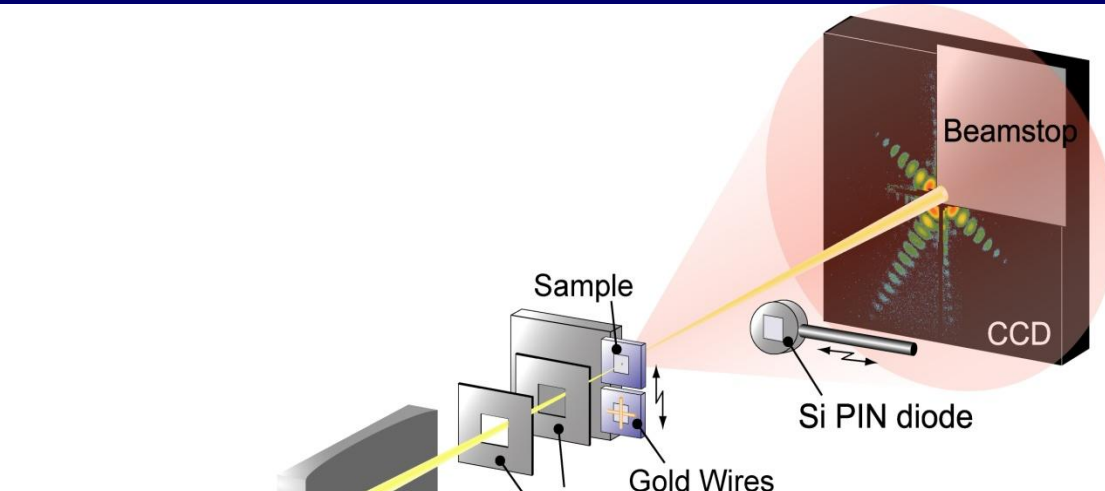
Vertical direction



Horizontal direction

9.9keV

Focused x-ray illumination for diffraction microscopy



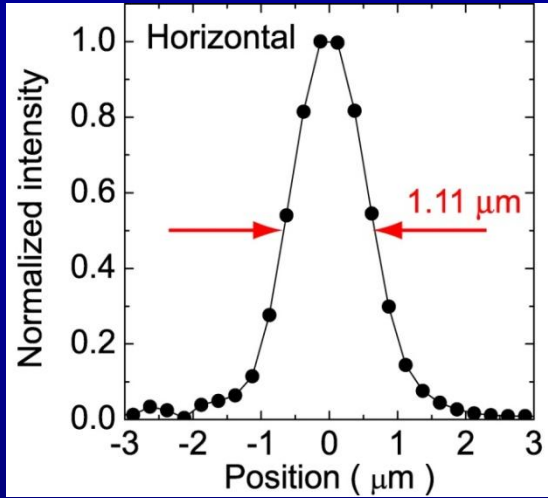
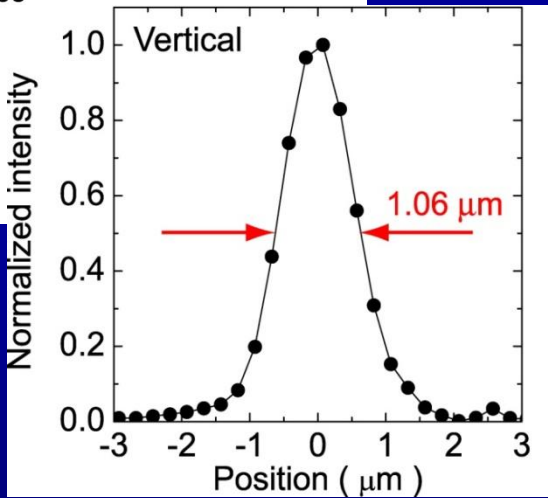
How wide area in k-space can we observe?



We must heighten the photon density at sample.

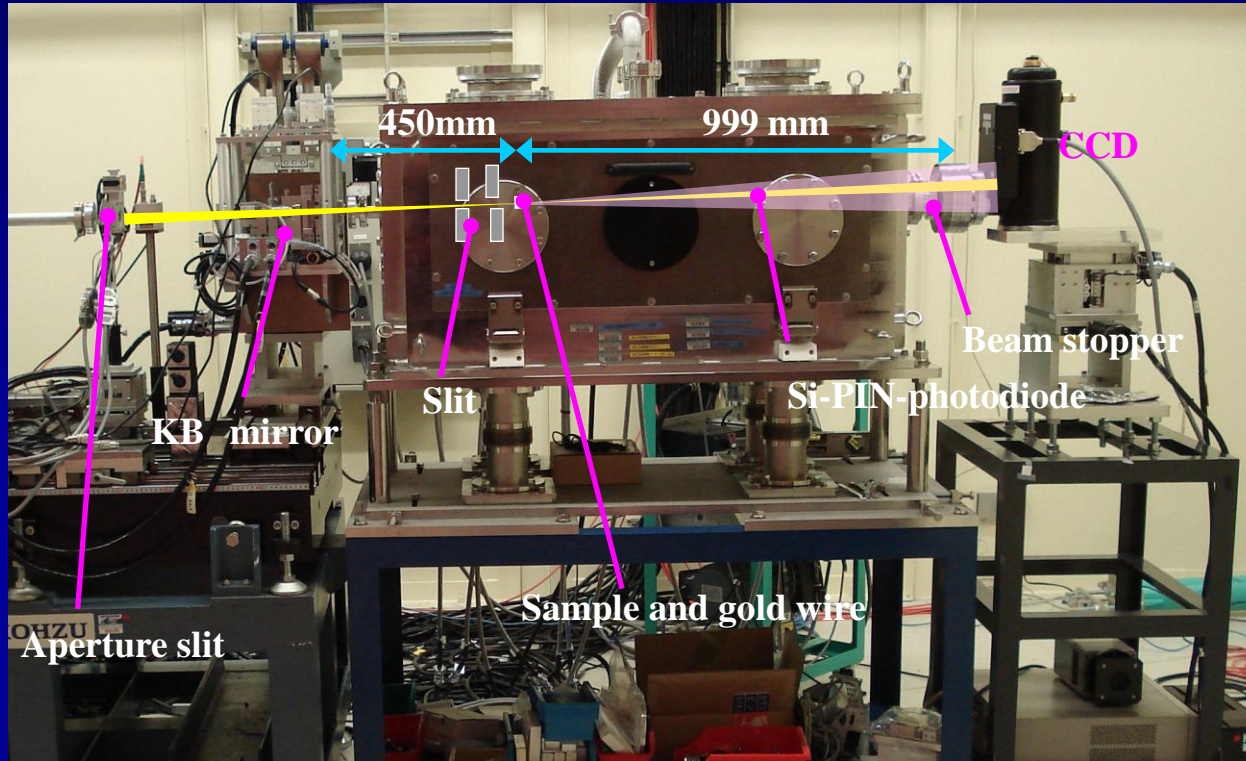
Beam profiles

Synchrotron X ray
@SPring-8 BL29XUL



- Spot size: $\sim 1 \mu\text{m}$
 - Photon density: $\sim 1.0 \times 10^4 \text{ photons/nm}^2/\text{s}$
- ➡ More than 100 times larger**

Set-up and samples



- ◆ X-ray energy: **12keV**
- ◆ Working distance: **450mm**
- ◆ Camera length: **999mm**
- ◆ CCD (Princeton Instruments PI-LCX:1300)

Pixel size: 20 μ m
1300 \times 1340 pixels

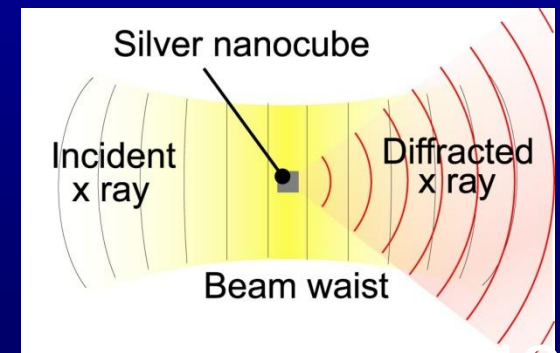
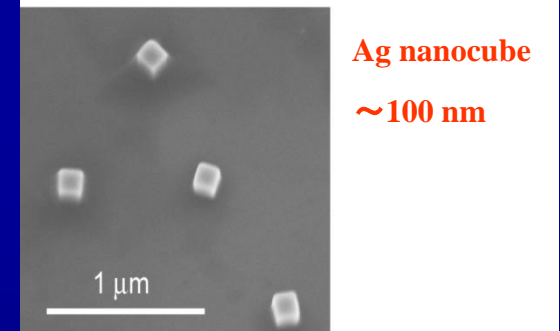
Science 298, 2176 (2002)

Shape-Controlled Synthesis of Gold and Silver Nanoparticles

Yugang Sun and Younan Xia*

Monodisperse samples of silver nanocubes were synthesized in large quantities by reducing silver nitrate with ethylene glycol in the presence of poly(vinyl pyrrolidone) (PVP). These cubes were single crystals and were characterized by a slightly truncated shape bounded by {100}, {110}, and {111} facets. The presence of PVP and its molar ratio (in terms of repeating unit) relative to silver nitrate both played important roles in determining the geometric shape and size of the product. The silver cubes could serve as sacrificial templates to generate single-crystalline nanoboxes of gold: hollow polyhedra bounded by six {100} and eight {111} facets. Controlling the size, shape, and structure of metal nanoparticles is technologically important because of the strong correlation between these parameters and optical, electrical, and catalytic properties.

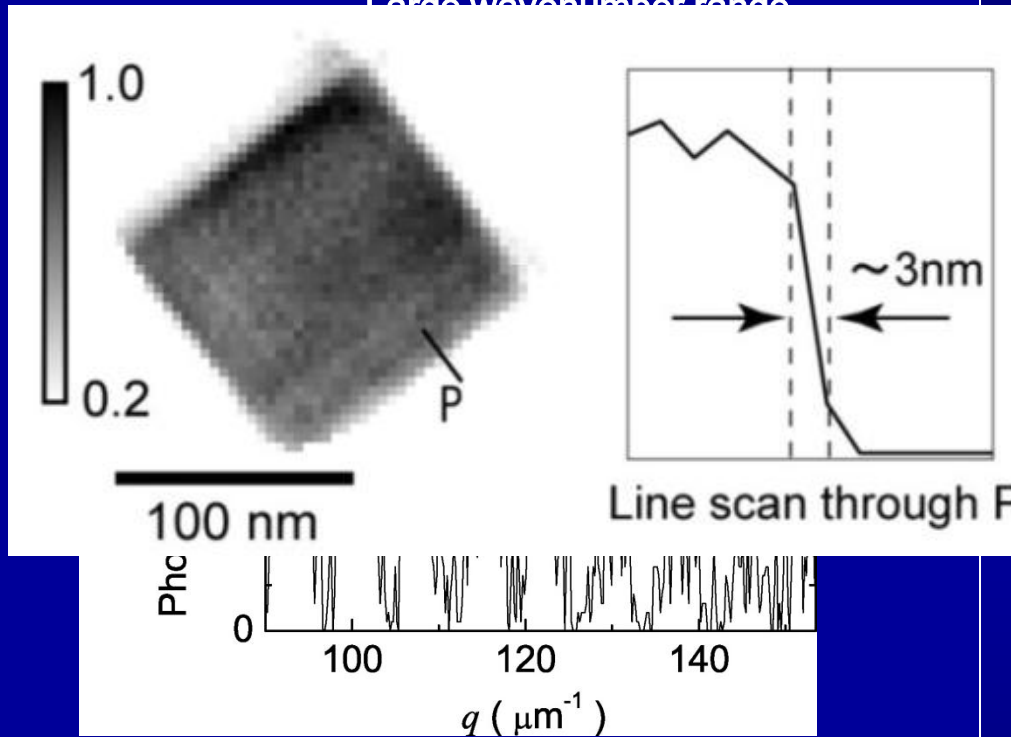
SEM image



Coherent Diffraction Intensity (a.u.)

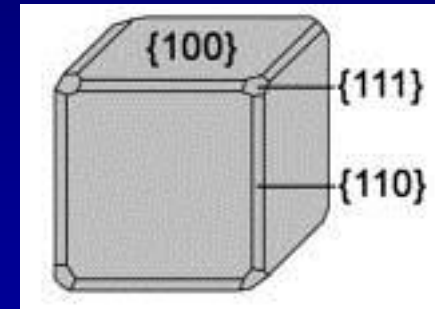
10^0 10^1 10^2 10^3 10^4

Large wavenumber range



◆ 5 photons are counted at $145 \mu\text{m}^{-1}$ during 800 sec irradiation **PRTF(1/e) resolution : 3nm**

$q_x (\mu\text{m}^{-1})$



$$|q| = 2\sin(\Theta/2)/\lambda$$

λ : wavelength

Θ : Scattering angle

Sinc function:
$$\frac{\sin(\alpha q)}{\alpha q}$$

Exposure time : 800sec, 1.5×10^{11} Photons to the cube

Y.Takahashi et al., Phys. Rev. B 80, 054103 (2009).

Summary

- Achromatic total-reflection mirrors realized sub-30nm focusing of hard X-rays.
- In-site wavefront correction are promising techniques to construct highly accurate optical system of hard X-rays.
- KB mirrors could reach sub-10nm focusing.
- AKB mirrors enable achromatic imaging of incoherent x-rays with sub-50nm-resolution.
- Focused X-ray by KB mirrors could heighten the spatial resolution in diffraction microscopy.

Thank you for your kind attention.