X線ナノ集光技術の展望

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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
Elliptical mirror \times 2

Diffraction-limited focusing

Waves are in constructive interference state.

Waves are in constructive interference state.

Phase error = \frac{2d \sin \theta}{\lambda} wave

Error height: 2nm

Error height: 2nm

Error height: 4nm

Error height: 4nm

Error height: 6nm

Error height: 6nm

Designed profile (ellipse)

Beam profile

\( \lambda \)

\( \theta \)

\( \sin \theta \)

\( d \)

\( \frac{2d \sin \theta}{\lambda} \) wave

40mm<\(L_s\)<50mm

100mm<\(L_s\)
Fabrication and figure testing technologies of Osaka University

◎ Plasma CVM (chemical vaporization machining)
   → Rough figuring (Rapid figuring with 10nm (P-V) level accuracy)

◎ EEM (elastic emission machining)
   → Final figuring and smoothing (Fine figuring with atomically smoothing)

◎ MSI (microstitching interferometry)
   → Figure tester with spatial resolution close to 0.01mm

◎ RADS1 (relative-angle determinable stitching interferometry)
   → Figure tester for steeply curved ellipse of large NA mirror

JTEC  URL http://www.j-tec.co.jp
Removal mechanism of EEM (Elastic Emission Machining)

In EEM, chemical reaction between solid surfaces is utilized.

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

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

The bump site is selectively removed.
Typical deterministic figuring properties using EEM

Position (mm) vs. Residual figure error (nm)

- Prefigured profile
- After 1st. figuring
- After 2nd. figuring
- Finished profile
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

100 µm

Focal point
“Hard-X-ray sub-10nm focusing and realization of high-resolution X-ray microscopy”
To realize Sub-10nm focusing K-B mirrors

- Slit (10μm)
- Focal point
- X-ray energy: 20keV
- Focal length: 150mm
- Acceptance width: 1.1mm
- Incidence angle: 11.1mrad

MSI with RADS1 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

Not only figure error but also thickness deviation of the multilayer induce wavefront phase error.
At-wavelength phase-retrieval interferometry

Light source

Incident Slit

Focal point

Mirror

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

Beam waist

Phase retrieval

Iteration

I_1(z), \varphi_1(z)

I_2(x), \varphi_2(x)

I_3(z), \varphi_3(z)

Blue : known
Red : unknown

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.

Yumoto et al.,
New knife-edge method

Conventional knife-edge method

New method

Mimura et al.

Spherically propagating X-ray

Beam waist

Mimura et al.
Details of the new knife-edge method

Scanner is made of Pt with the thickness of 2.5 μm.

X-ray detector

Spherically propagating X-ray

Beam waist

Scanner is acting as a phase object

Modified wavefront

Phase is shifted with λ/2.

Microroughness at the bridge

A-A' profile

A'
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

Converged profile in the phase retrieval processing

 Recovered profile

Mimura et al.
Recovered wavefield is local-minimum solution or not

Calculated and measured intensity profiles on A-A' line. (50μm 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.

![Diagram showing the components of the deposition system including Mirror Scanner, Sputtering Gun, and a diagram of the deposition setup with labeled parts such as Sample, Slit, Gate valve, Load lock chamber, Main chamber, Shutter, Pump, and a Magnetron sputtering cathode target: Pt & C.]
Figuring by differential deposition

Additionally deposited film thickness using differential deposition method
Focused beam profiles before and after DD

Comparison between the wave fields before and after phase compensation

Handa et al.  SIA(2008)
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
Phase error = \(2kd \sin \theta\)

- \(d\): Shape error
- \(\theta\): Glancing angle
- \(k=2\pi/\lambda\): Wave number

Design concept

🌟 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

Objective shape
- Elementary beam theory
- Controlled shape using optical interferometer

Feedback system

Optical interferometer
- Measurement control
  - PC for Optical interferometer
  - Mirror profile
  - PC for active mirror control

Active mirror
- Voltage supply
- Voltage control

Kimura et al.,
Sub-10nm focusing mirror

Source Focus

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

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

\(\Lambda\) : d-space
\(\lambda\) : X-ray wavelength
\(n\) : Index
\(\theta\) : Glancing angle

EEM Machine
Micro- and RAD- Stitching Optical Interferometry
Laterally-Graded Multilayer Coater
Optical configuration for active phase compensation

- **Fizeau interferometer**
- **Knife edge scanner**
- **Focusing mirror (Mirror B)**
- **CCD camera (for alignment)**
- **Zooming tube + X-ray CCD**
- **Deformable mirror**
- **Optical interferometer (ZYGO GPI)**
- **Adaptive mirror**
- **Focal point**
- **Zooming tube + CCD**
- **APD placed at dark field**

X-ray

- **Ion chamber Adaptive mirror**

Dimensions:
- 150mm
- 250mm
Sub-10nm focusing by using phase compensator

Profile at focal point

Compensated phase profile (shape of compensation mirror)

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

Beam waist structure

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

Source

Focus

22 mm

18 mm

~1 km

Intensity (arb. units)

Position (nm)

8 nm

Ideal
Experimental
**Achromatic imaging device (AKB Mirrors)**

- **KB mirrors**
- **Total reflection axial-symmetric optics**
  - Focusing
  - Imaging
    - It fills Abbe’s sine condition

**Advanced Kirkpatrick–Baez mirrors**


- **Ellipse**
- **Hyperbola**

**One-dimensional Wolter mirror**

**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

Magnified image

Magnified image

Elliptical mirrors

Hyperbolic mirrors

Designed one-dimensional Wolter mirror

43 nm

PSF

@11.5keV
Spatial resolution test

Demagnified imaging system

- X-ray: 11.5 keV
- Slit (10 μm)
- Focal point
- PIN
- Gold wire (φ 200 μm)
- 45 m

43 nm

- 10 nm
- 11 nm
- 13 nm
- Fit
- Calculation
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 $\mu rad$ was obtained.

It is equivalent to the FOV of 12 $\mu m$. 
Field curvature aberration

![Diagram showing field curvature aberration](image-url)

- FOV = 12 μm
- Relative distance from beam axis (μm)
- Angular distance from the field center (μrad)
- FWHM (nm)

**Graphs:**
- Calculated vs. Experimental
- Angular distance from the field center (μrad)
- Relative distance along beam axis (mm)

**Data Points:**
- FOV = 12 μm
- Angular distance from the field center
- Relative distance from the focus (mm)
- FWHM (nm)
Summary of AKB development

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

- 10nm
- 11nm
- 13nm

Fit
Calculation

FOV = 12μm

Matsuyama et al., Optics Lett (2010)

AKB optics will be useful in coming XFEL experiment.
We are now trying to realize 2-dimensional sub-50nm focusing of XFEL.

We have already developed 1μm-level focusing unit for day-1 system of Japanese XFEL, and also fabricated and tested sub-100nm focusing mirror.

XFEL focusing

WD: 350mm
1\mu m-level focusing

From undulator

- $M_A$: f=2000mm
- $M_B$: f=1550mm
- WD: 1340mm

Photon density per pulse (Photon/pulse/mm²)

- 1.0 x 10^{17}
- 0.8 x 10^{17}
- 0.6 x 10^{17}
- 0.4 x 10^{17}
- 0.2 x 10^{17}
- 0.0

Vertical direction

- 1.6\mu m

Horizontal direction

- 1.2\mu m
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

- Spot size: \( \sim 1 \mu m \)
- Photon density: \( \sim 1.0 \times 10^4 \) photons/nm\(^2\)/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: 20μm
1300 × 1340 pixels

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.

Ag nanocube
~100 nm
Large wavenumber range

\( |q| = \frac{2\sin(\Theta/2)}{\lambda} \)

\( \lambda \): wavelength

\( \Theta \): Scattering angle

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

\( \wedge \): 5 photons are counted at 145 \( \mu \)m\(^{-1} \) during 800 sec irradiation

PRTF(1/e) resolution : 3 nm

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

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.