Mirrors and Multilayers

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outline

Mirrors

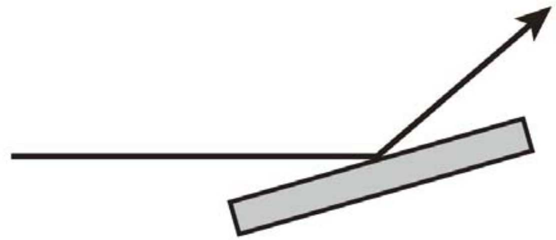
(1) role of mirrors
(2) refractive index and reflection of X-rays
(3) mirror materials and mirror coatings
(4) surface polishing and characterization
(5) curved mirrors
(6) transmission mirror
(7) heat load and cooling

Multilayers

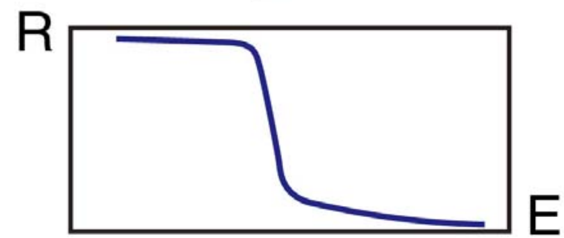
(1) structure and X-ray reflectivity
(2) fabrication
(3) characterization
(4) graded multilayers
(5) applications

Mirrors

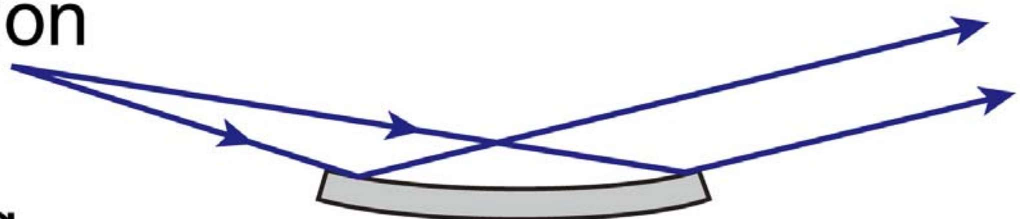
- Deflecting the beam



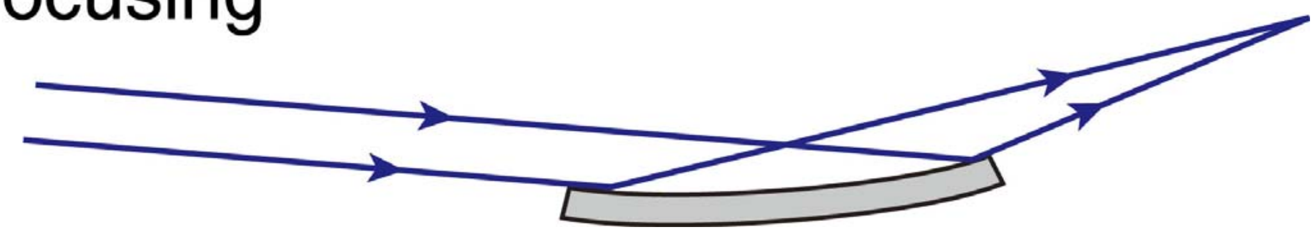
- Low pass filter



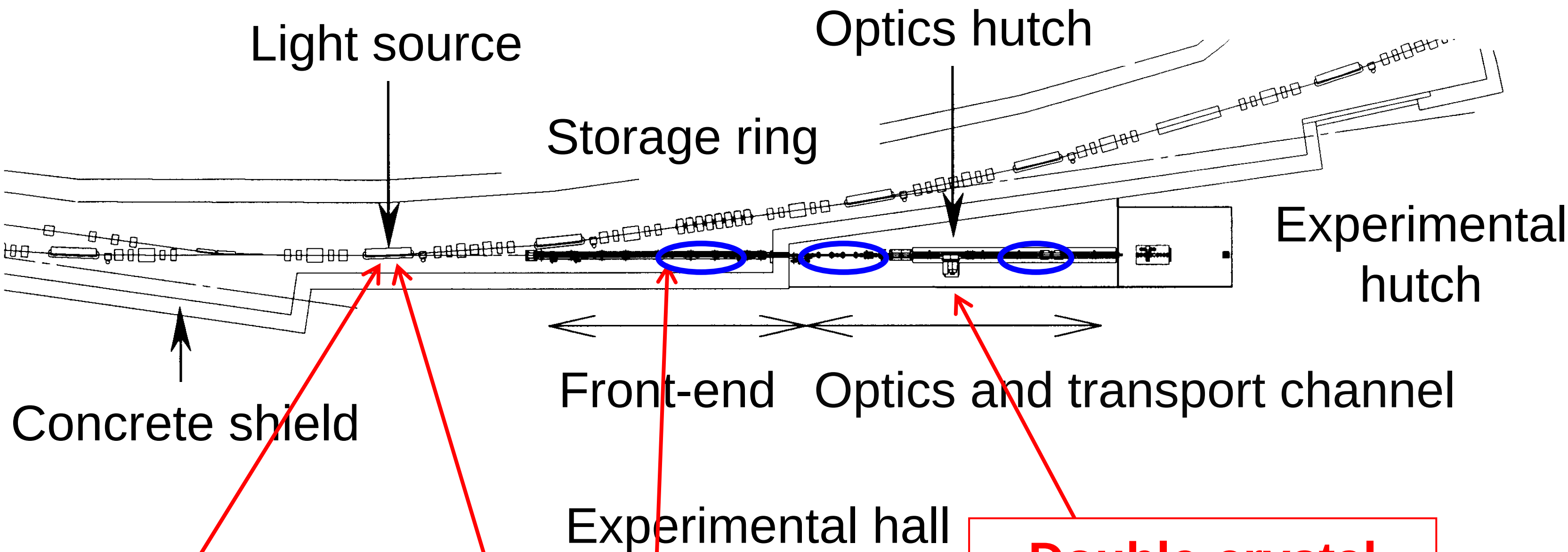
- Collimation



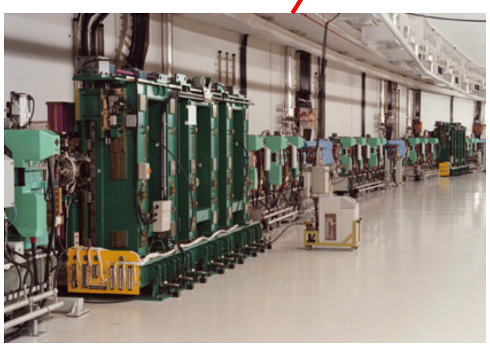
- Focusing



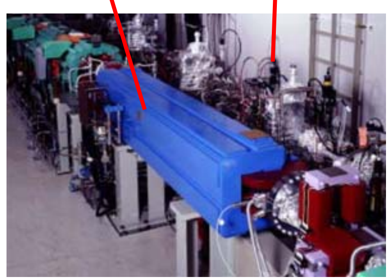
Mirror position on a beamline



Double-crystal monochromator



Undulator



Bending magnet & front-end

Refractive index

For X-rays, refractive index is slightly smaller than unity.

$$n = 1 - \delta + i\beta \quad \text{for} \quad E \propto \exp(+i(\omega t - kr))$$

$$\left(n = 1 - \delta - i\beta \quad \text{for} \quad E \propto \exp(-i(\omega t - kr)) \right)$$

$$\delta = \frac{n_a r_e \lambda^2}{2\pi} f_1 \qquad \beta = \frac{n_a r_e \lambda^2}{2\pi} f_2$$

n_a: atomic density $\quad r_e = \dfrac{e^2}{4\pi\varepsilon_0 mc^2}$: (classical electron radius)

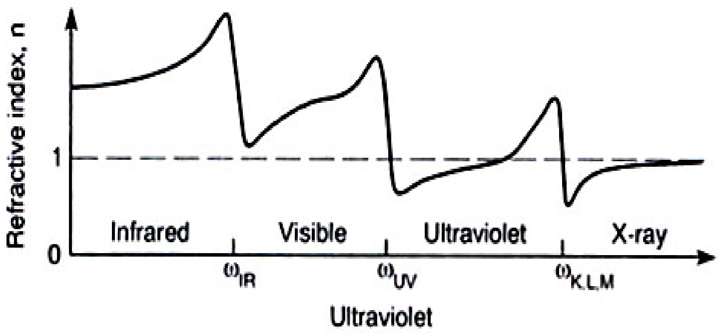
$f_1 = f_0 + f'$ $\quad f_2 = f''$

f_0 : atomic scattering factor

f' and f'': real and imaginary parts of anomalous dispersion terrm

$$f_0 = \int \rho \exp(ikr)$$

For forward scattering, $f_0 = Z$



Refer to Chap. 3 of "soft X-rays and extreme ultraviolet radiation" by D. Attwood, Cambridge Univ. Press, 1999

	$\delta \times 10^5$	$\beta \times 10^7$
Si	0.488	0.744
Ge	0.908	2.33
Quartz	0.555	0.467
Pt	3.26	20.7
Au	2.96	19.5

Interaction of x-rays with matter

Experimental data

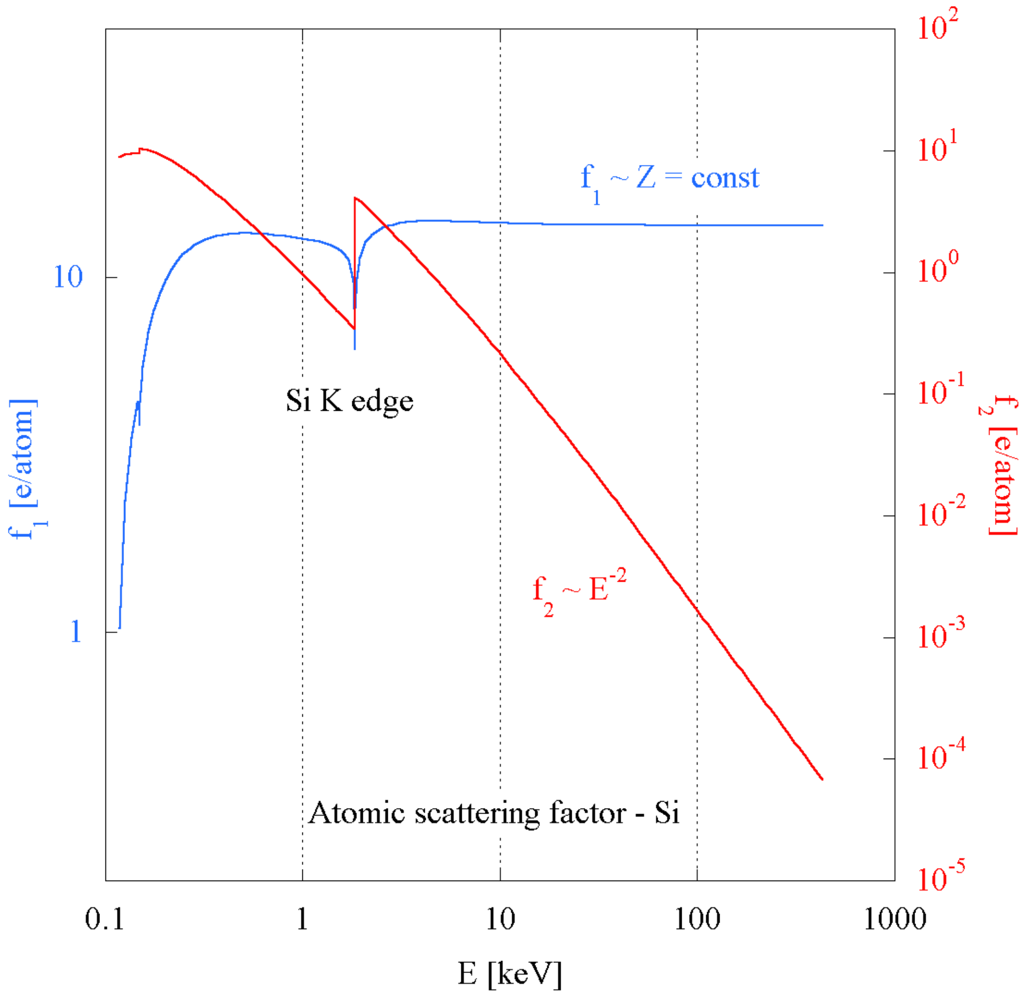
Asymptotic behaviour for E > E(K):

$$f_1 \approx const \qquad f_2 \approx 1/E^2$$

$$\delta \approx 1/E^2 \qquad \beta \approx 1/E^4$$

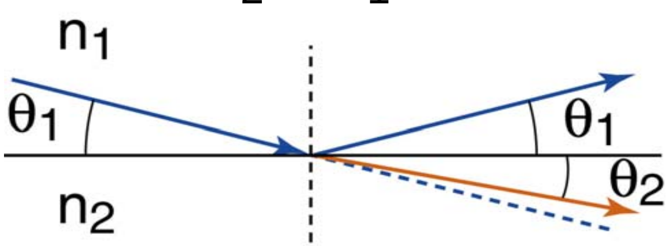
$$\frac{\delta}{\beta} \approx E^2$$

High energy applications !



Snell's law

$n_2 < n_1$



$$\frac{n_1}{n_2} = \frac{\cos(\theta_2)}{\cos(\theta_1)}$$

if $\frac{n_1}{n_2}\cos(\theta_1) = 1 \longrightarrow \cos(\theta_2) = 1 \longrightarrow \theta_2 = 0$

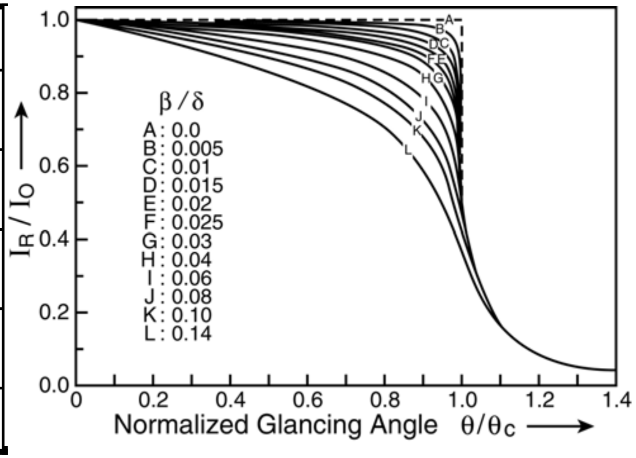
$\longrightarrow$ total external reflection

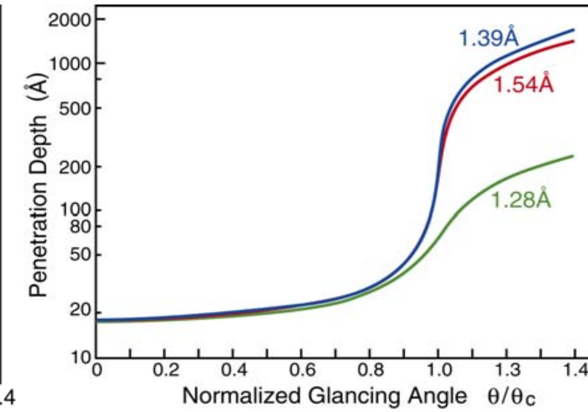
$n_1 = 1$ in vacuum $\longrightarrow \cos(\theta_1) = 1 - \frac{\theta_1^{\,2}}{2} = n_2 = 1 - \delta$

$$\theta_c = \sqrt{2\delta} \approx 1 \sim 20 \text{ mrad}$$

For 10 keV

	$\delta \times 10^5$	θc (degree)
Si	0.488	0.179
Ge	0.908	0.244
Quartz	0.555	0.191
Pt	3.26	0.463
Au	2.96	0.441





L. G. Parratt, Phys. Rev.95 359 (1954)

Reflectivity formula

$$R = \frac{(\theta - A)^2 + B^2}{(\theta + A)^2 + B^2}$$

$$A = \sqrt{\frac{(a^2+b^2)^{1/2}+a}{2}}$$

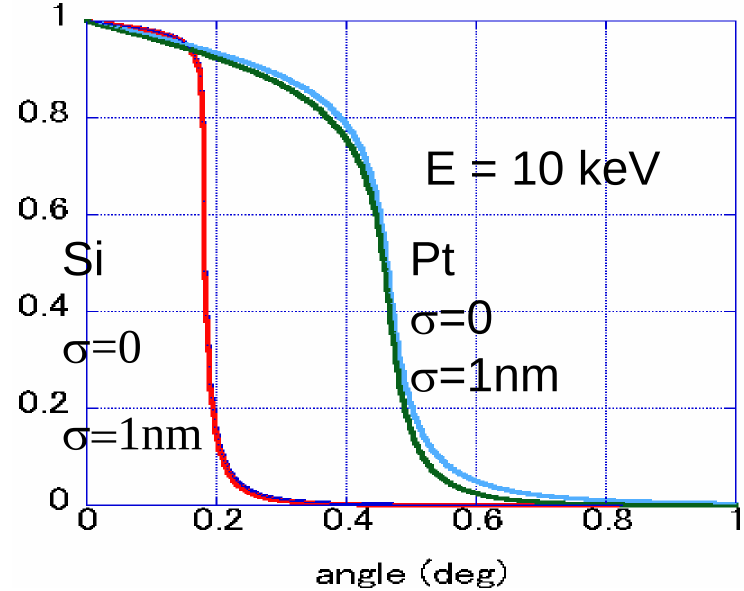
$$B = \sqrt{\frac{(a^2+b^2)^{1/2}-a}{2}}$$

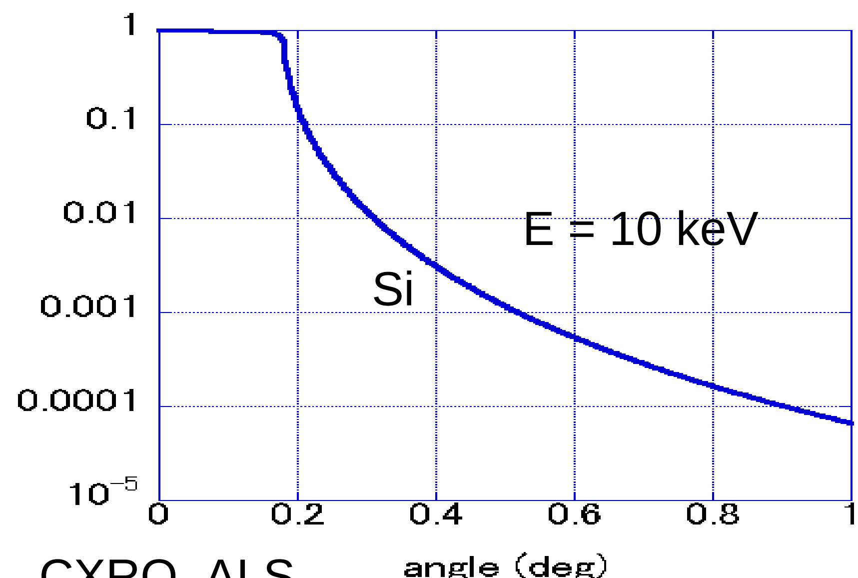
$$a = \theta^2 - \theta_c^2 = \theta^2 - 2\delta \qquad b = 2\beta$$

$$q = 4\pi \frac{\sin(\theta)}{\lambda}$$

For $q \gg q_c$ $\qquad R \propto 1/q^4$

H. ***Compton*** and S. K. ***Allison***, X-Rays in Theory and Experiment (D. Van Nostrand Company, Inc., New York, 1935),





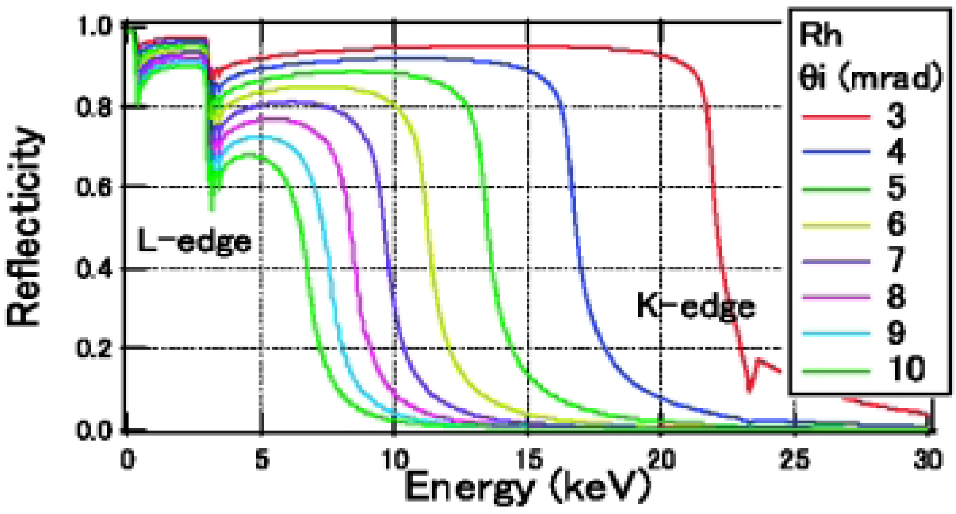
CXRO_ALS

"X-ray Ingteractions with Matter"
http://henke.lbl.gov/optical_constants/

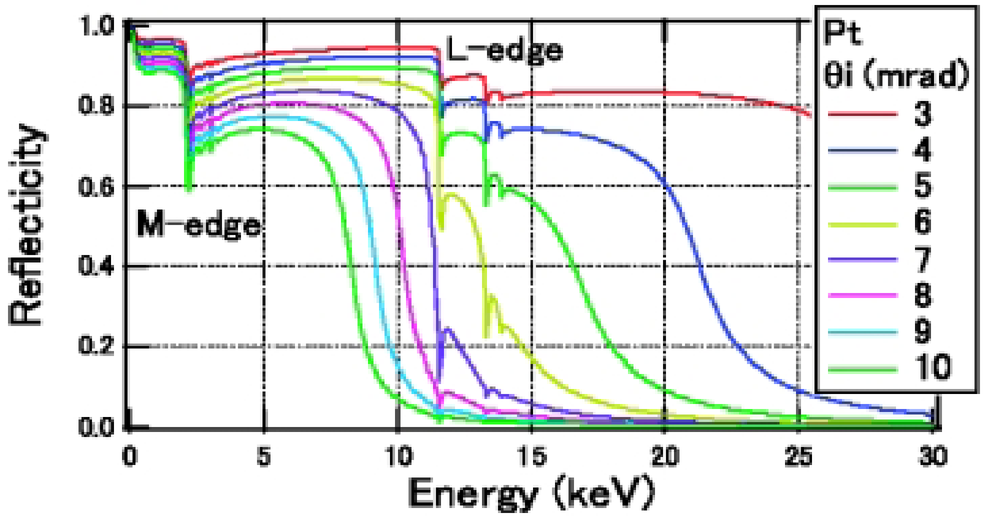
Reflectivity curve

A total reflection mirror works as a low-pass energy filter. The cut-off energy can be controlled by selecting the material and the glancing angle.

Influence of absorption edges sometimes appear in the reflectivity curve.



(a)



(b)

From Uruga & Nomura, Hoshako(放射光) 2006 vol.19 No.4 pp.248-257, Fig.3

Requisites for mirror materials

(1) Can be polished to a smooth and accurate surface figure.
(2) Large material available. (~1.4 m long mirror presently used)
(3) Mechanically hard, Radiation resistive
(4) Good thermal conductivity, low thermal dilation constant
(5) No (low) gas emission under X-ray irradiation
(6) Can be bent.
(7) Reasonable cost (material itself and fabrication)

Important parameters

- Surface figure
  flat, cylindrical, elliptic, parabolic, spherical, toroidal, ellipsoidal, paraboloidal,----
- Roughness
  root-mean-square roughness ~0.1 nm
- Slope error
  root-mean-square slope ~0.5 μrad
- Thermal property
  thermal conductivity
  dilation constant

- Absorption edges : X-ray absorption

Materials for mirrors

Silicon

Zerodur

Fused silica

GlidCop (alumina dispersion strengthened copper alloys)

Cu with electroless Ni layer

Silicon Carbide (CVD)

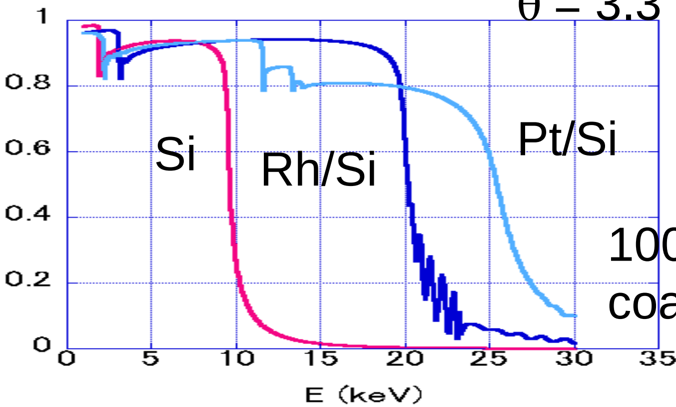
Materials for mirror coating

Desired critical energy with a given glancing angle

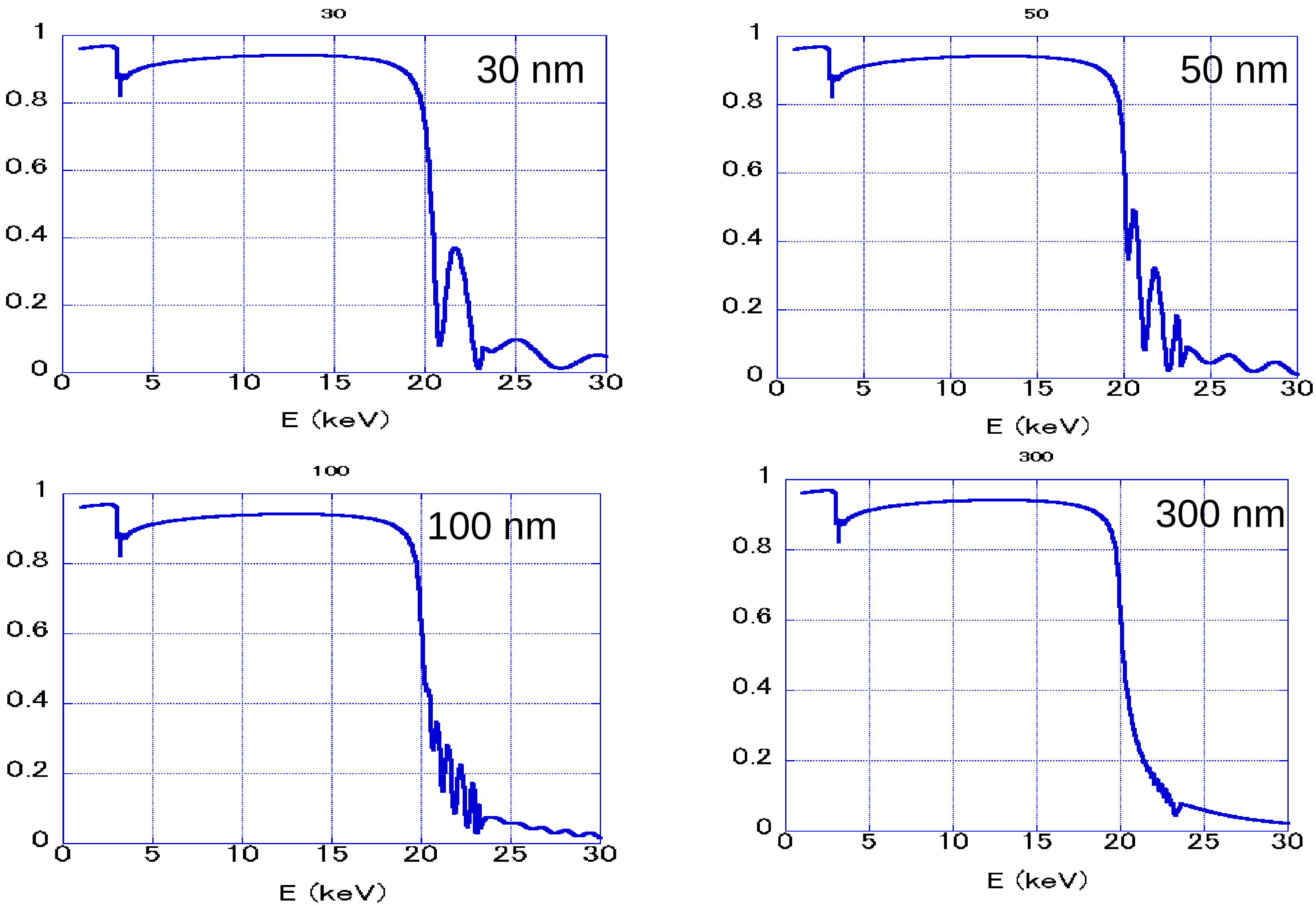
Retain smooth surface

No absorption edges desirable in the required energy range

Au, Pt, Rh, Ni, Pd, Ru



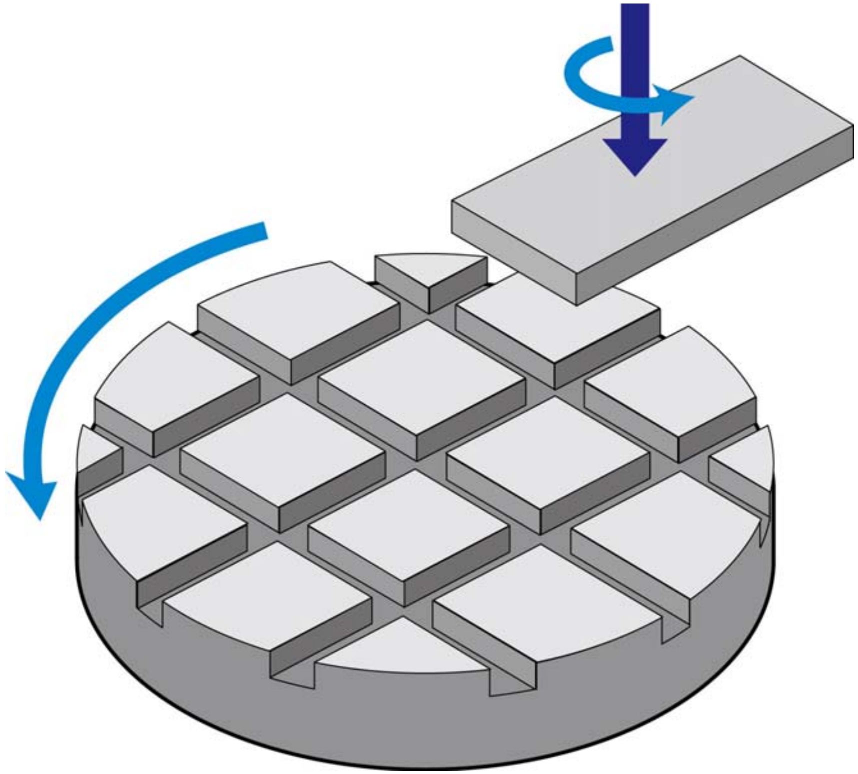
Coating of mirrors
(Rh coating on silicon)



Fabrication of mirrors

- Grinding
- Diamond turning

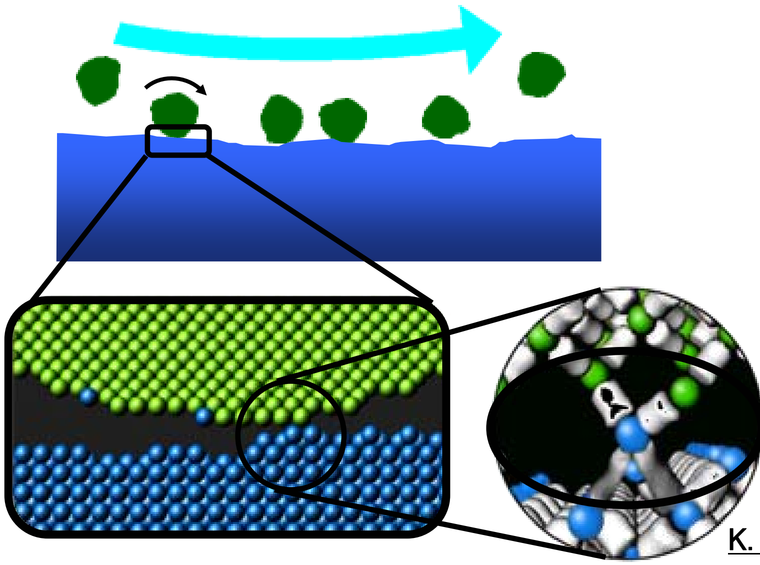
- Lapping
- polishing



Advanced Polishimg Technique (Elastic Emission Machining)

In EEM, chemical reaction between solid surfaces is utilized.

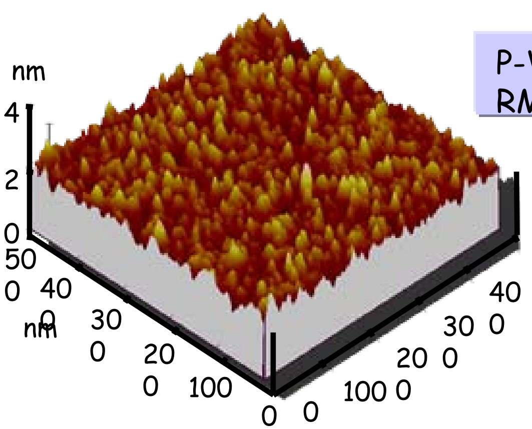
Ultrafine powder particles having reactivity to the work surface are employed.



Ultra-fine particles are supplied to the work surface by ultrapure water flow.

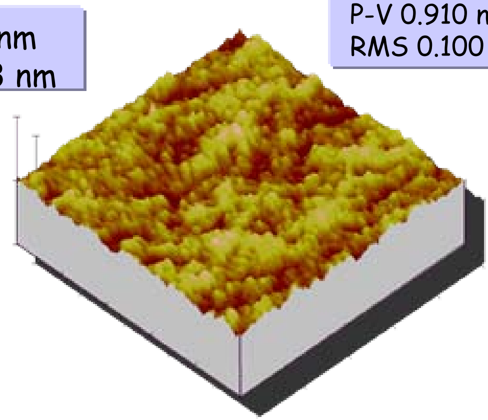
Atom removal occurs selectively at the topmost site of the work surface.

K. Yamauchi, et al.
Rev. Sci. Instrum., 73, (2002), pp. 4028-4033.



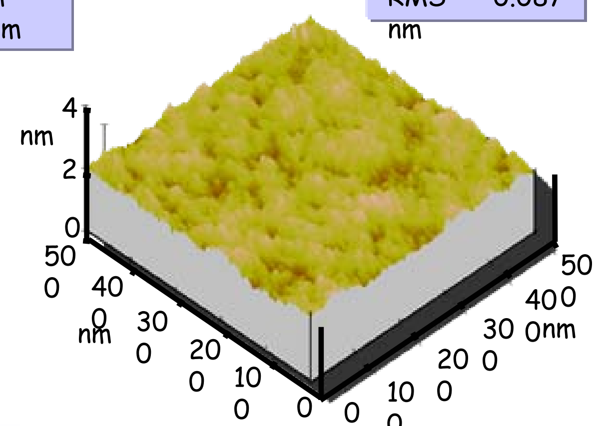
P-V 1.603 nm
RMS 0.183 nm

Pre-machined surface



P-V 0.910 nm
RMS 0.100 nm

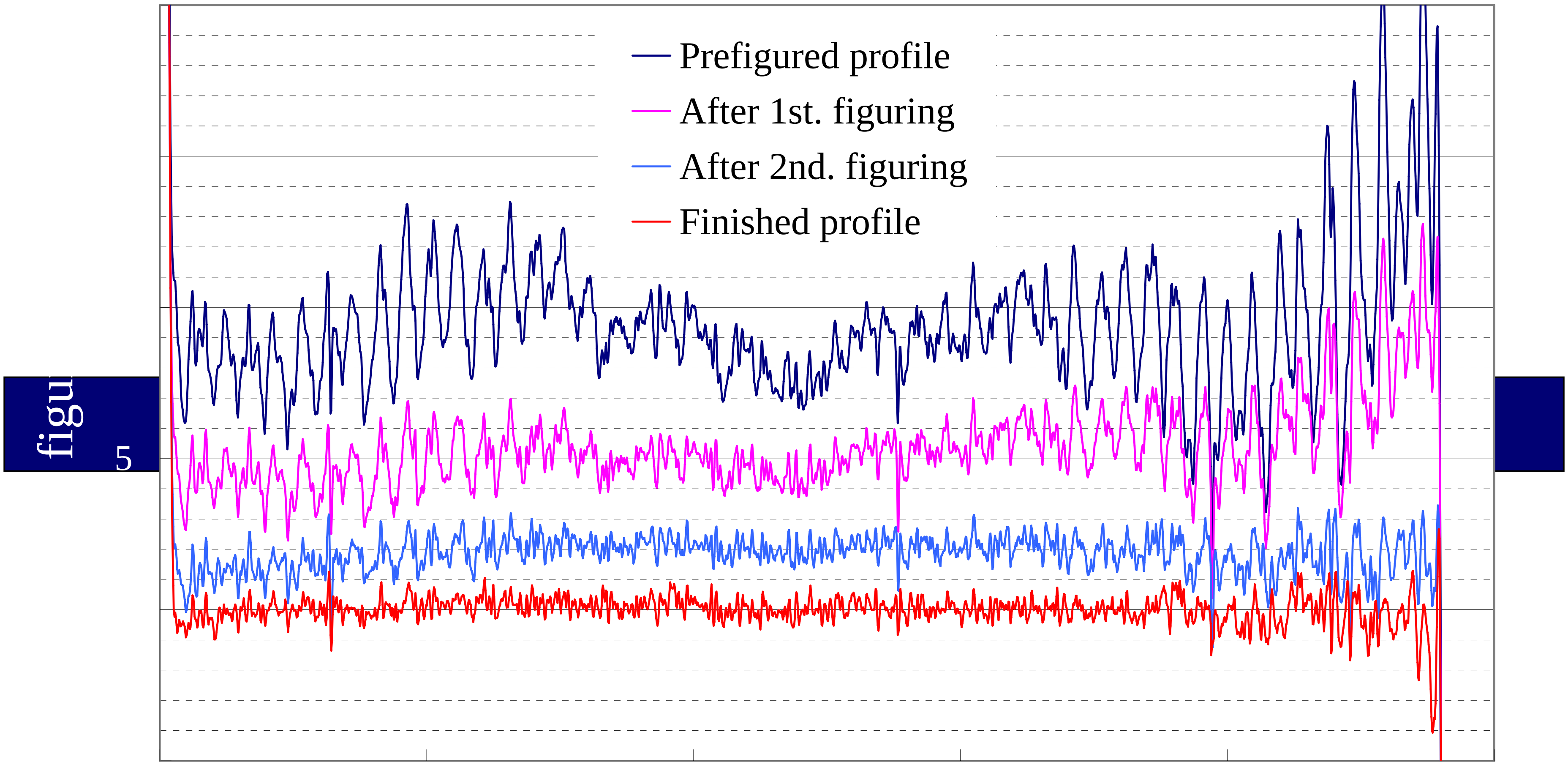
EEM surface (Removal depth:2nm)



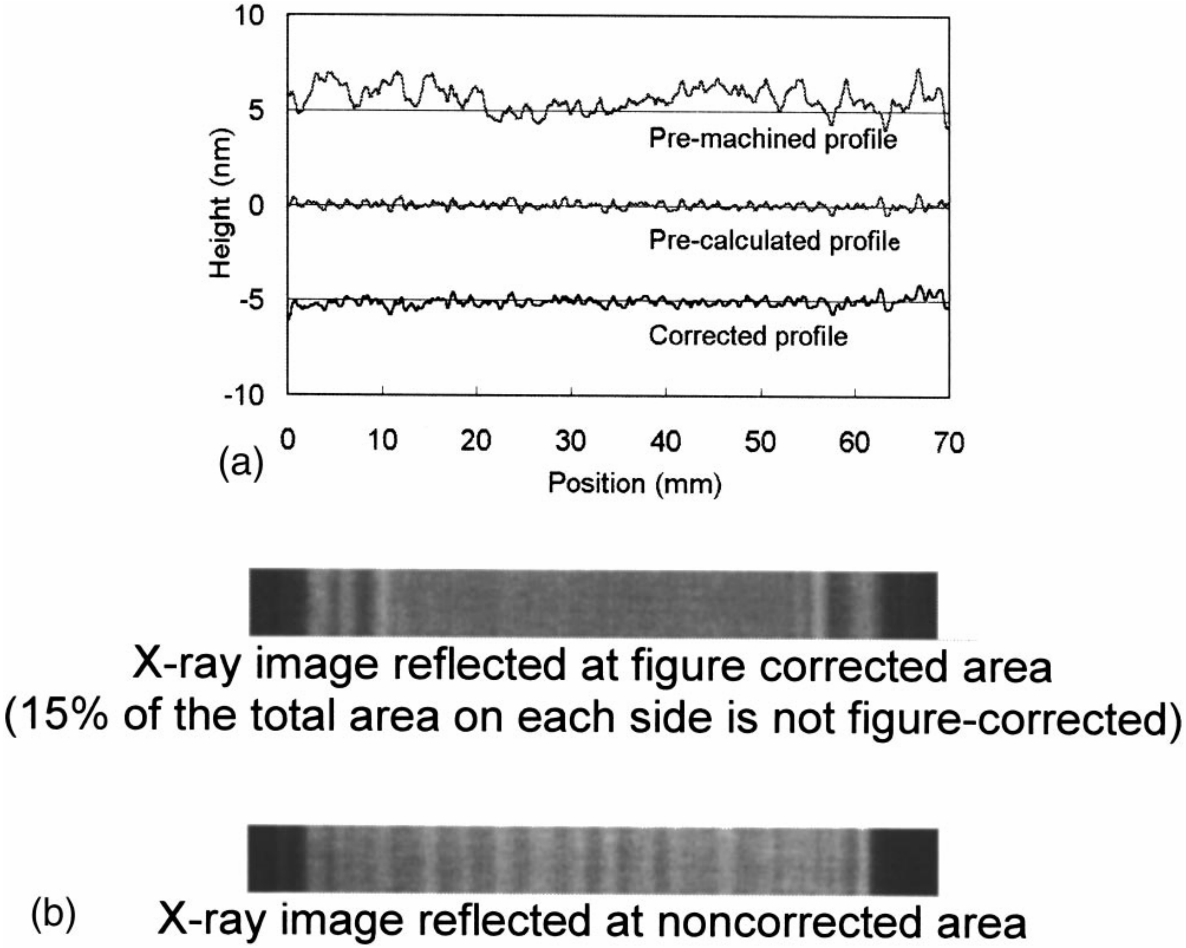
P-V 0.769 nm
RMS 0.087 nm

EEM surface (Removal depth:8nm)

Typical figuring properties using EEM



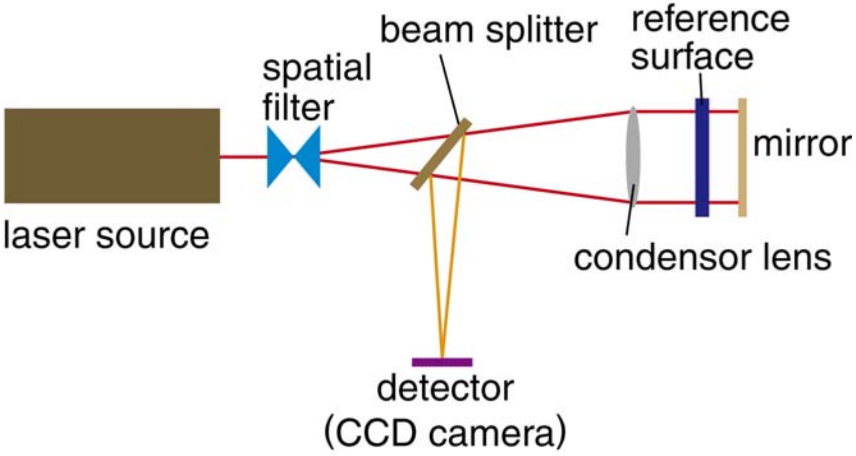
H. Mimura et al.,Rev. Sci. Instrum., 76, 045102 (2005).
K. Yamauchi et al., Rev. Sci. Instrum., 74 (5), 2894-2898 (2003).



K. Yamauchi et al., Rev. Sci. Instrum., 74 (5), 2894-2898 (2003).

Characterization of mirrors
(surface roughness and figure)

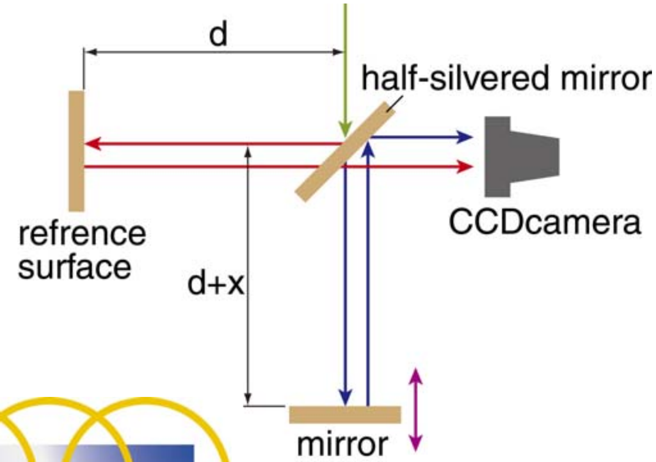
Fizeau interferometer



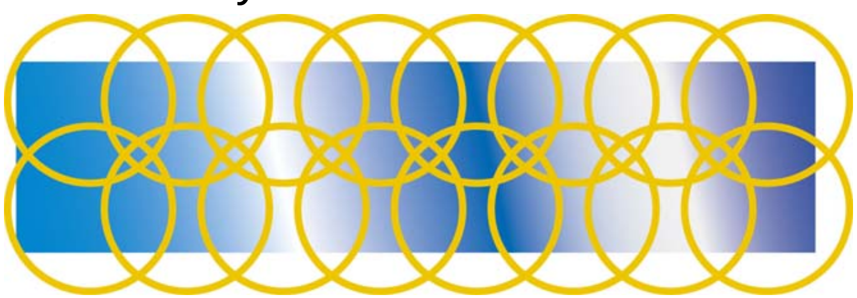
Michelson interferometer

Twyman-Green interferometer

(mirror: rotatable, point source)



Stitching interferometry

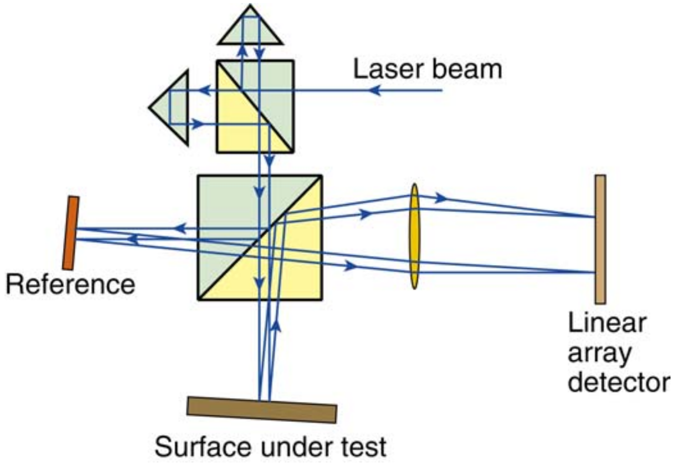


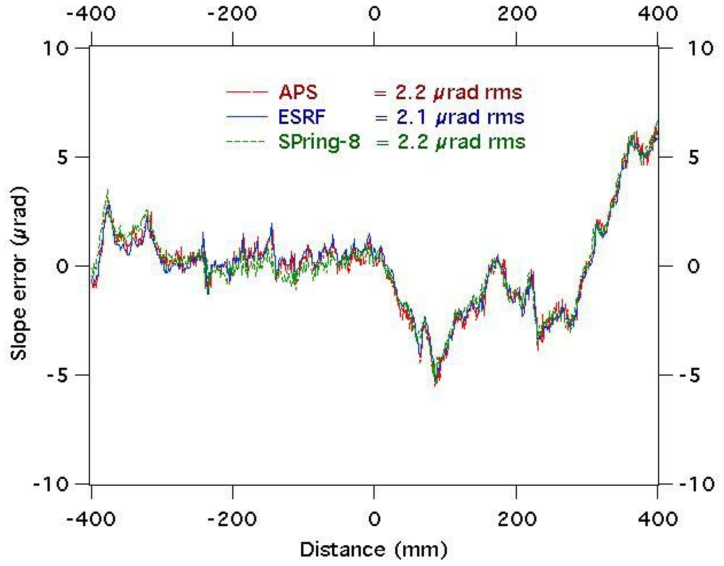
Atomic Force Microscopy

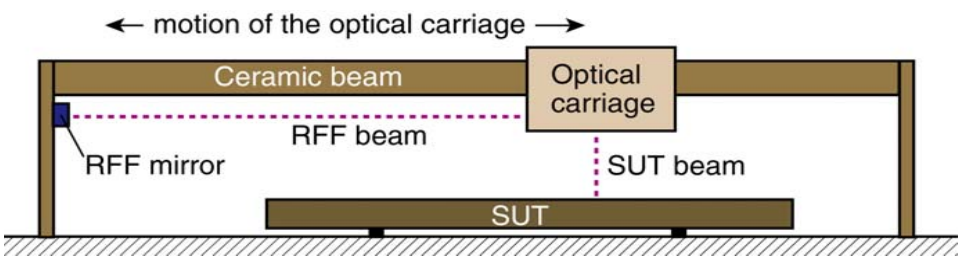
Stylus measurement

Characterization of mirrors (slope errors)

- Long Trace Profiler

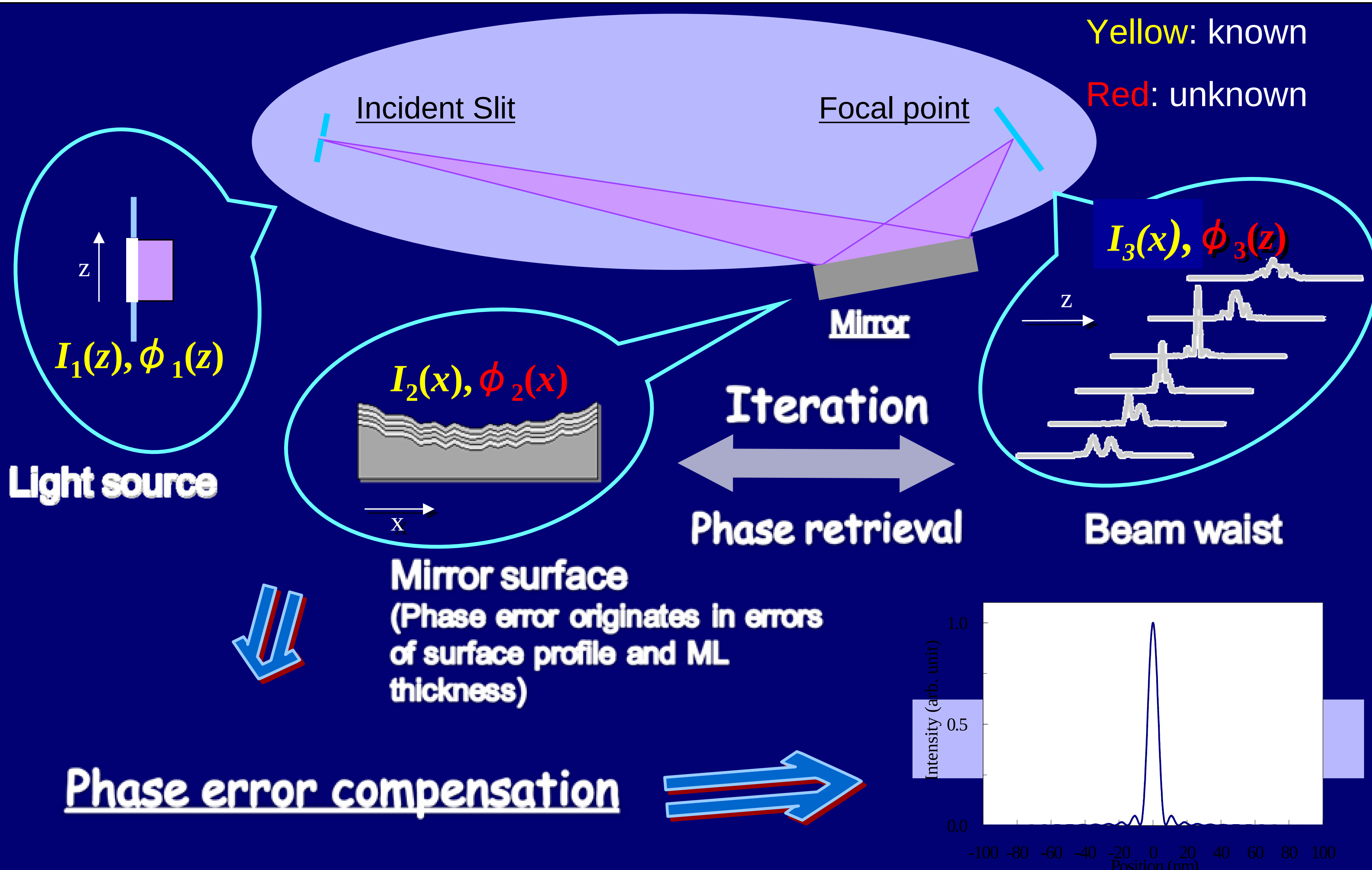






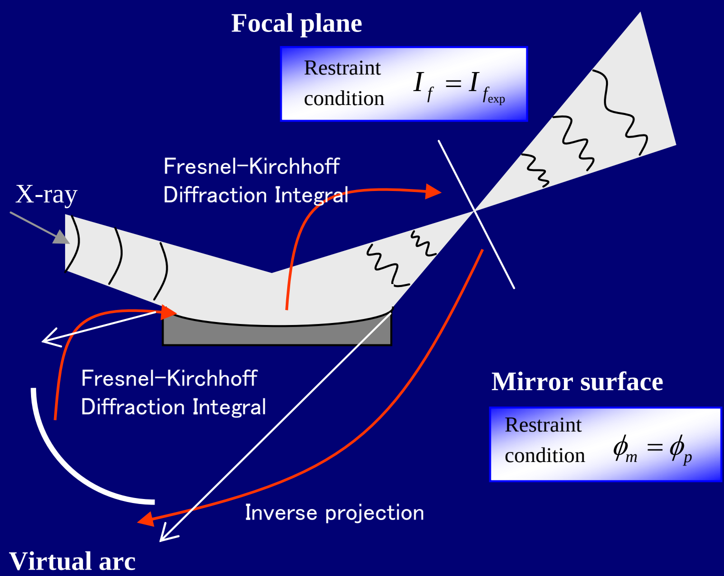
"Results o f x-ray mirror round robin metrology measurements at the APS,
ESRF, and SPring-8 optical metrology laboratories"
L. Assoufid, A. Rommeveaux, H. Ohashi, K. Yamauchi, H. Mimura, J. Qian,O. Hignette, T. Ishikawa, C. Morawe, A. Macrander, A. Khounsary, S. Goto
SPIE Conference on Optics and Photonics, San Diego: SPIE Proc. 5921-21,
129 (2005).

At-wavelength phase-retrieval interferometry



A strategy to fabricate KB mirrors for 10 nm hard X-rays focusing,
K. Yamauchi, X-Ray Optics: A Roadmap for the Next 10 Years
A one-day (August 1, 2005) Workshop at SPIE's Optics & Photonics 2005 San Diego, California USA

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.

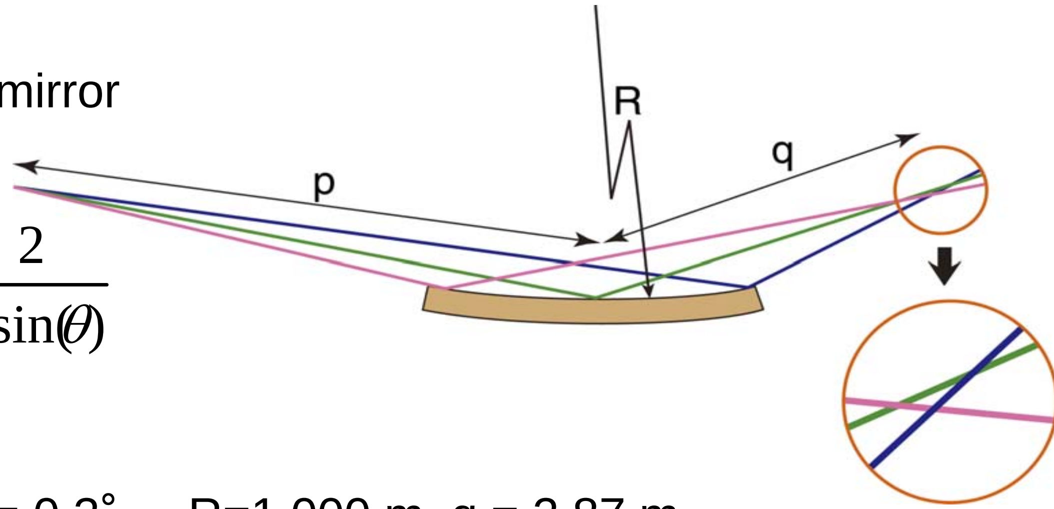
Phase is kept to be recovered value.

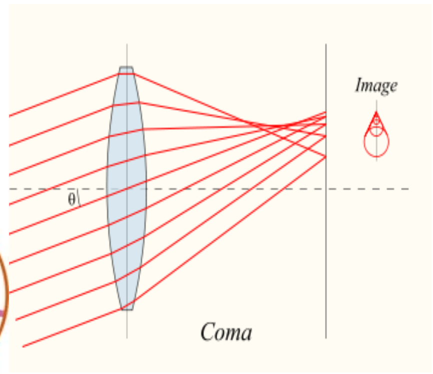
H. Yumoto et al.,
Review of Scientific Instruments 77, 063712 (2006).

curved mirrors (focusing, collimating)

Cylindrical mirror

$$\frac{1}{p}+\frac{1}{q}=\frac{2}{R\sin(\theta)}$$



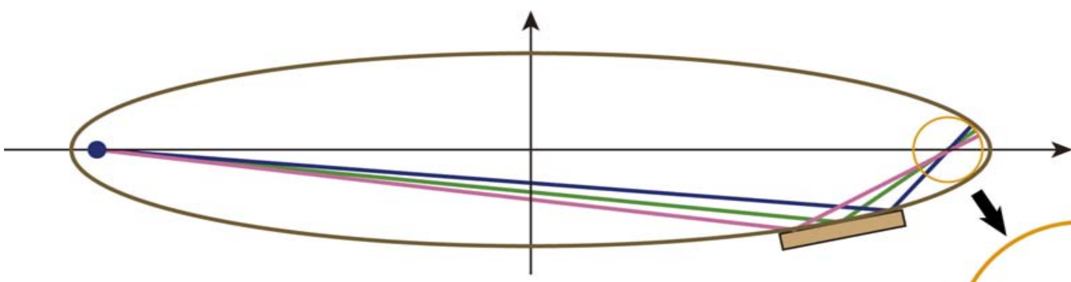


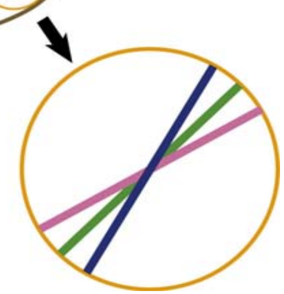
P=30 m, θ = 0.3° , R=1,000 m, q = 2.87 m

R=100 m , q = 0.26 m

Elliptic mirror

$$\frac{x^2}{a^2}+\frac{y^2}{b^2}=1$$

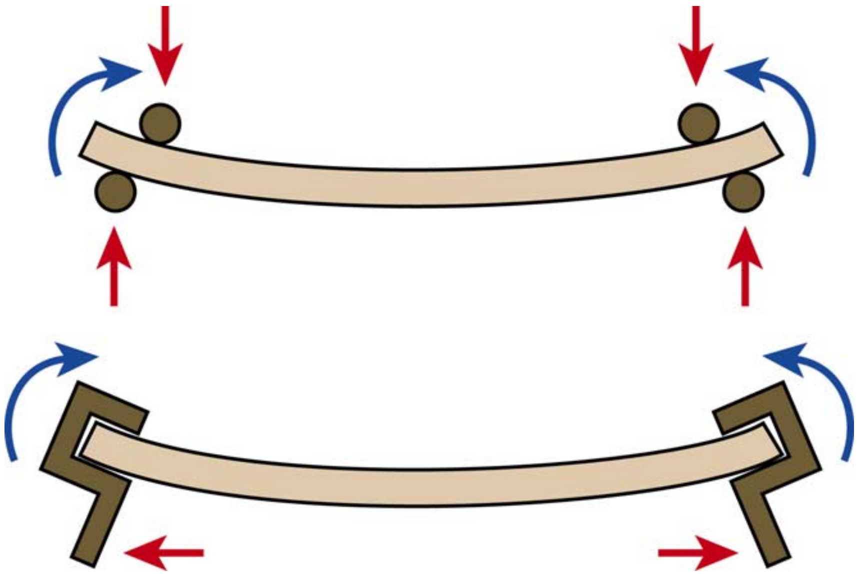


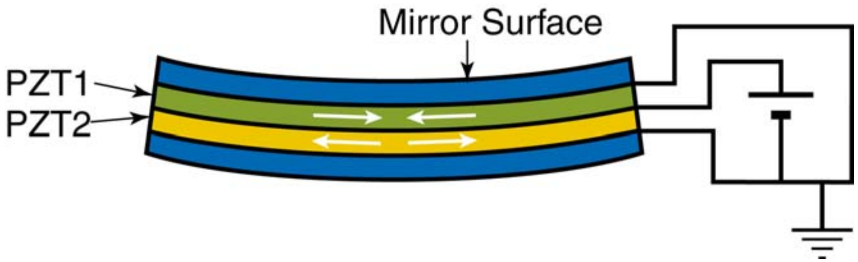


a = 15.5 m, b = 0.03 m, 60 cm long mirror at 30 m from the source,

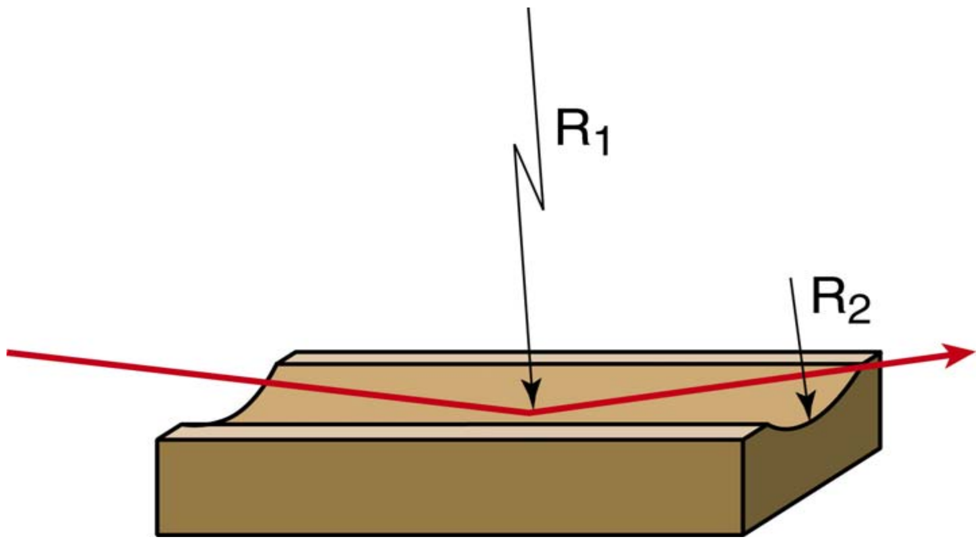
θ ~0.28° - 0.37° , q = 1m

Bending mechanisms



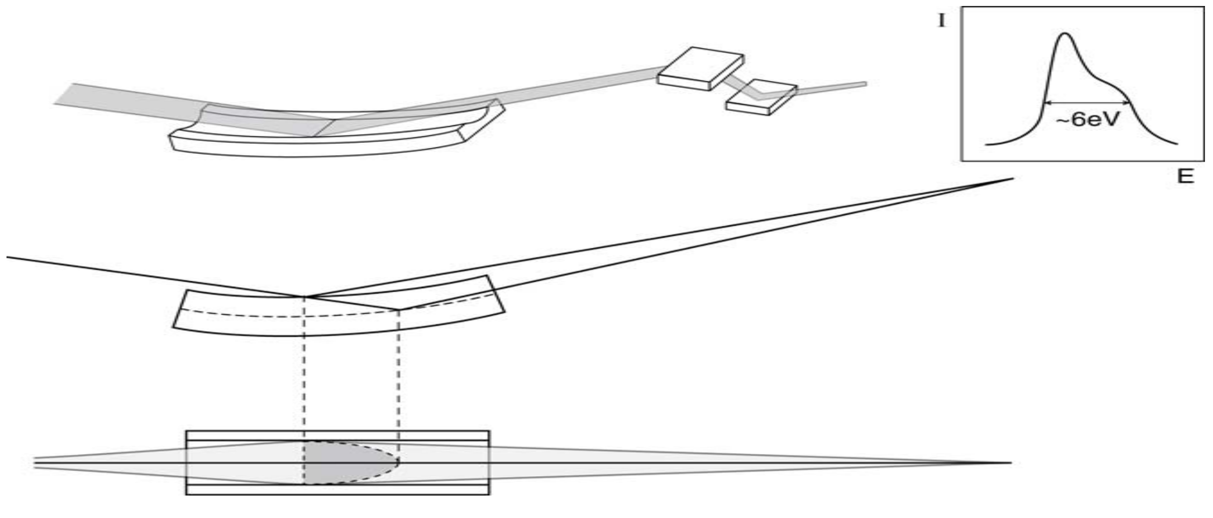


bent-cylinder, toroidal mirror



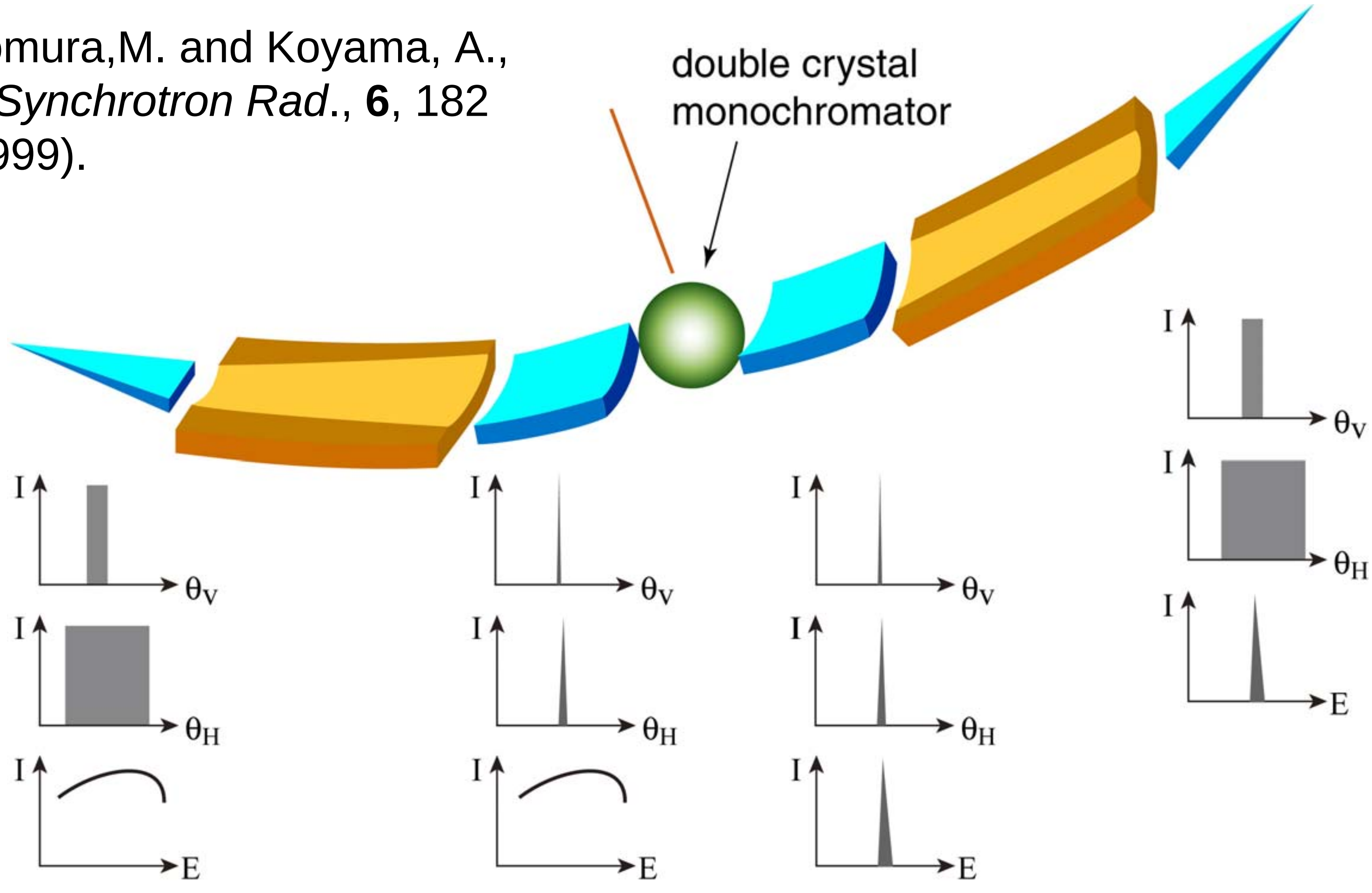
$$R_1 = \frac{2pq}{(p+q)\sin(\theta)}$$

$$R2 = \frac{R_1^{\,2}}{\sin^2\theta}$$



bent conical mirror
– approximation to paraboloidal mirror -

Nomura,M. and Koyama, A., *J. Synchrotron Rad*., **6**, 182 (1999).

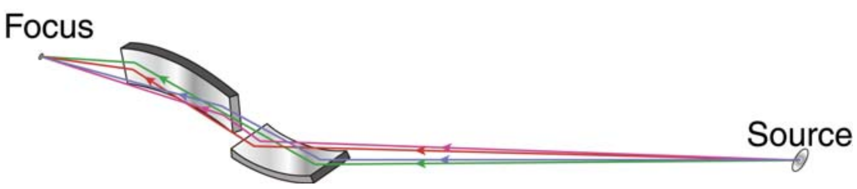


Kirkpatric-Baez (KB) optics for nanometer focusing

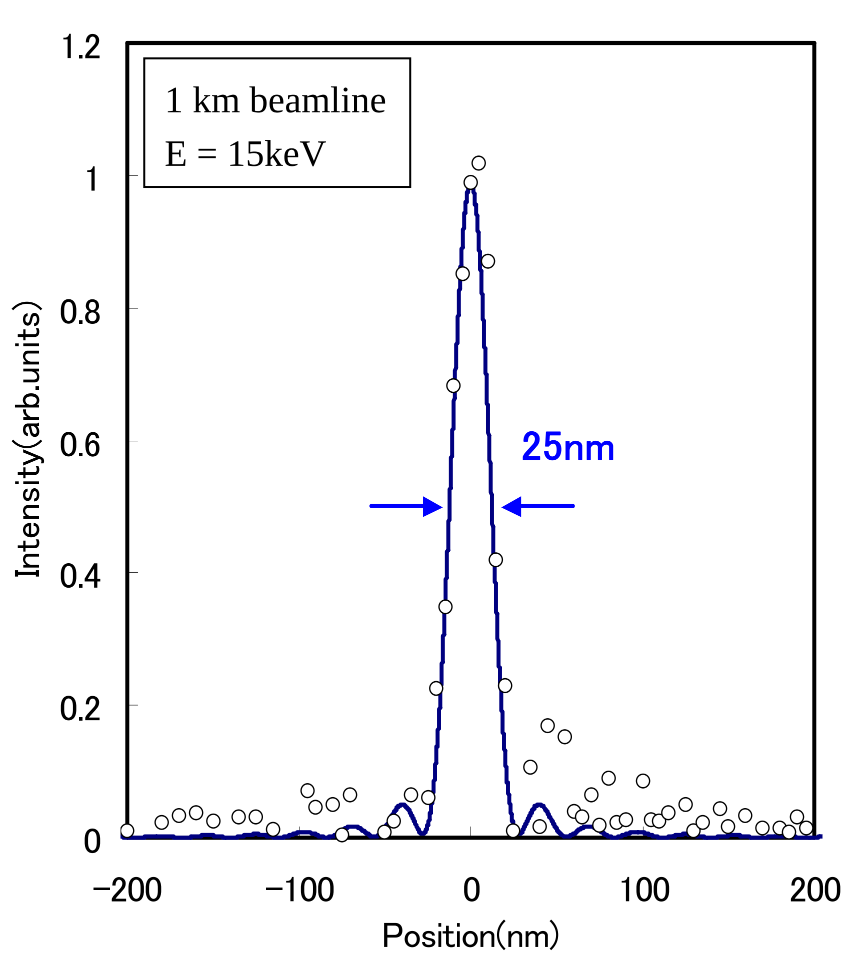
P. Kirkpatrick, A.V. Baez, J. Opt. Soc. Am. 38, 766 (1948)

Spring-8/Osaka KB development

- **Static and figured Si substrates**
- **Plasma Chemical Vapour Machining (PCVM)**
- **Elastic Emission Machining (EEM)**
- **Pt coating + thickness correction**
- **Focal spots below 30 nm**
- **Aiming less than 10 nm using multilayers**



[23] H. Mimura et al,
Jpn. J. Appl. Phys. Part 2 44, 18539 (2005)

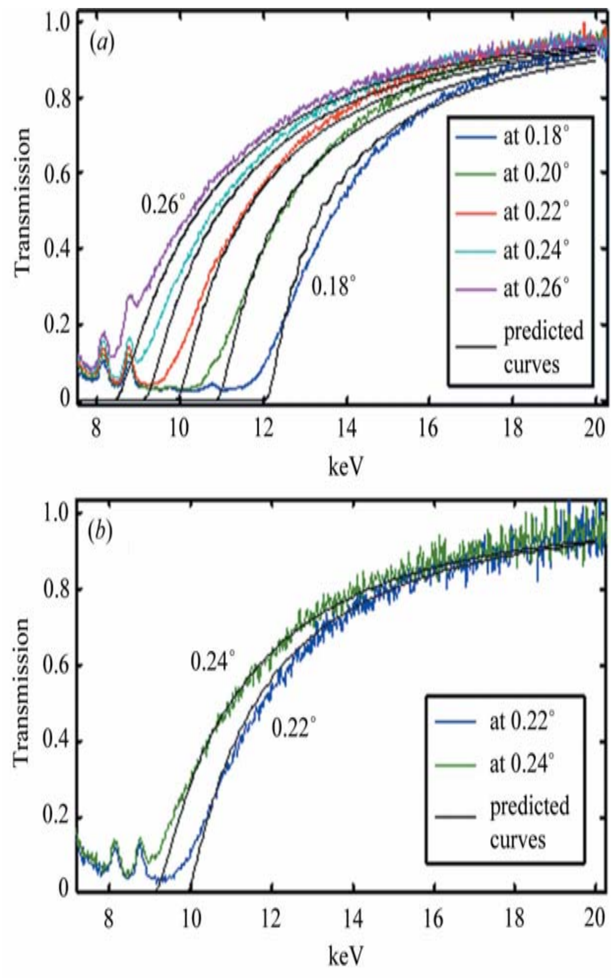


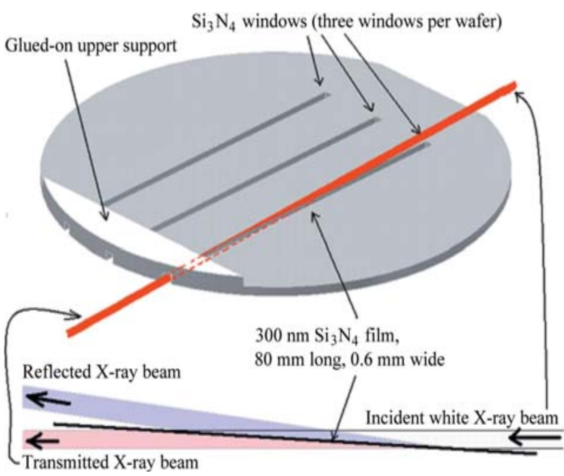
Transmition mirror: a high-pass energy filter

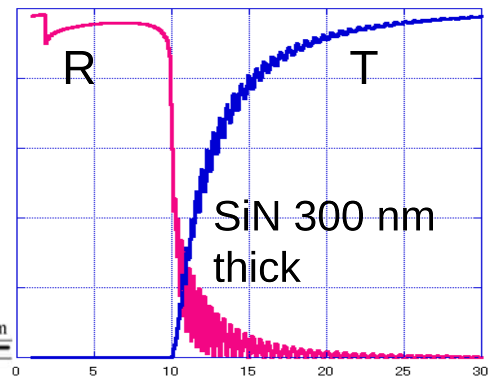
Silicon nitride transmission X-ray mirrors

Cornaby and D. H. Bilderback

J. Synchrotron Rad. 15, 371-373 (2008).





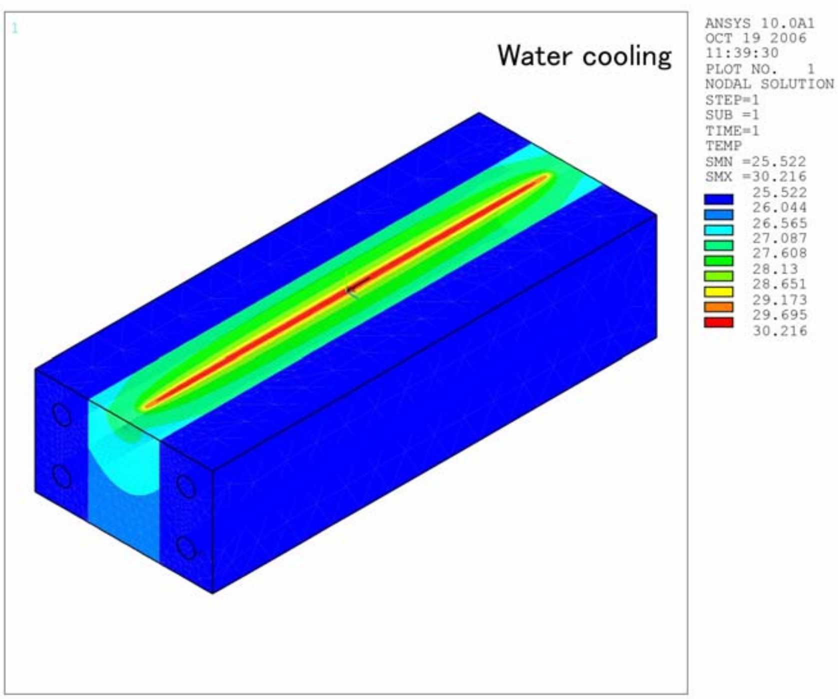


Heat load on and cooling of a mirror

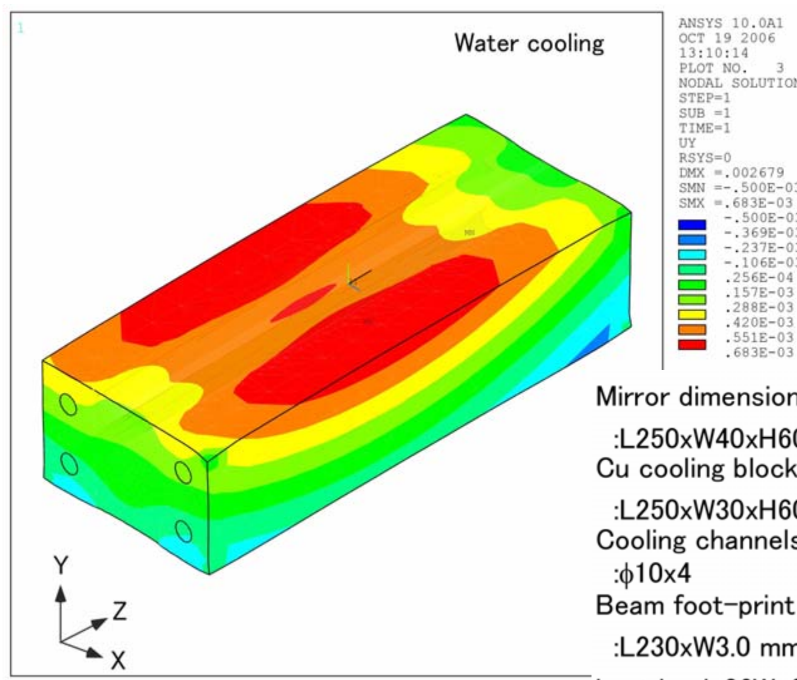
Heat load: 86 W, 0.13W/mm2

Glancing angle: 1.5° T_{water}=25℃

temperature



deformation



Mirror dimension
:L250xW40xH60 mm^3
Cu cooling block size
:L250xW30xH60 mm^3 x2
Cooling channels
:ϕ10x4
Beam foot-print
:L230xW3.0 mm^2
heat load: 86W, 0.13W/mm^2
(Incidence angle=1.5°)
hw=0.005W/mm^2°C
Tw=25°C

Multilayers

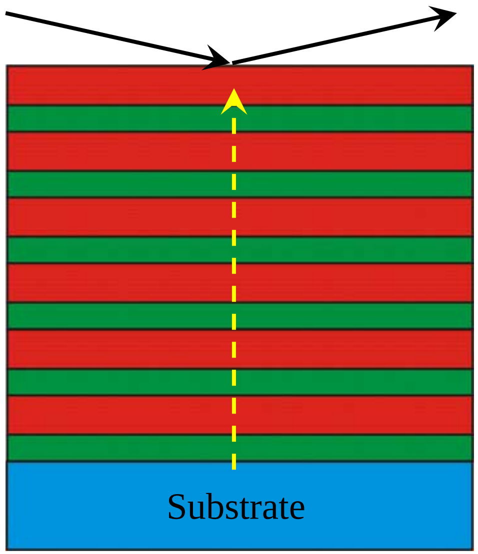
X-ray multilayer reflectivity

Recursive calculation of Fresnel coefficients and propagation

- **Parratt formalism (widely used in x-ray optics) [8]**

Principle

- **Start at semi-infinite substrate surface (no reflection from back side)**
- **Recursive construction of amplitudes and phases from layer to layer**



$$r_{n-1,n} = a_{n-1}^{\;4} \cdot \left[\frac{r_{n,n+1} + f_{n-1,n}}{r_{n,n+1} f_{n-1,n} + 1} \right]$$

$f_{n-1,n}$: Fresnel coefficients

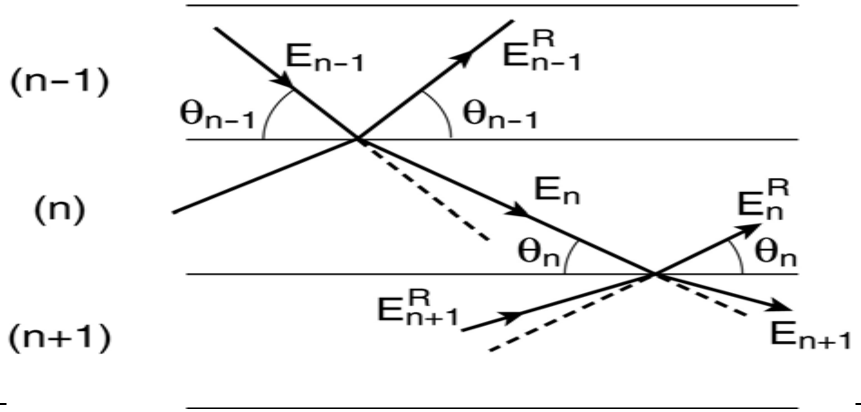
E_n: Electric field at centre of layer n

t_n: Thickness of layer n

$$r_{n,n+1} = a_n^{\;2} \frac{E_n^{\;R}}{E_n}$$

$$a_n = e^{-i\frac{\pi}{\lambda}\cdot t_n \sqrt{n_n^{\;2} - n_0^{\;2}\cos^2\theta}}$$

$\longrightarrow$ $$R = \left| r_{0,1} \right|^2$$



X-ray multilayer reflectivity

Numerical calculations

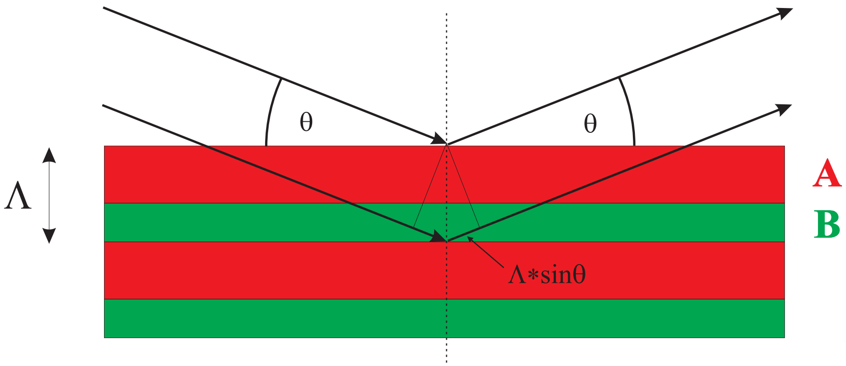
Main features

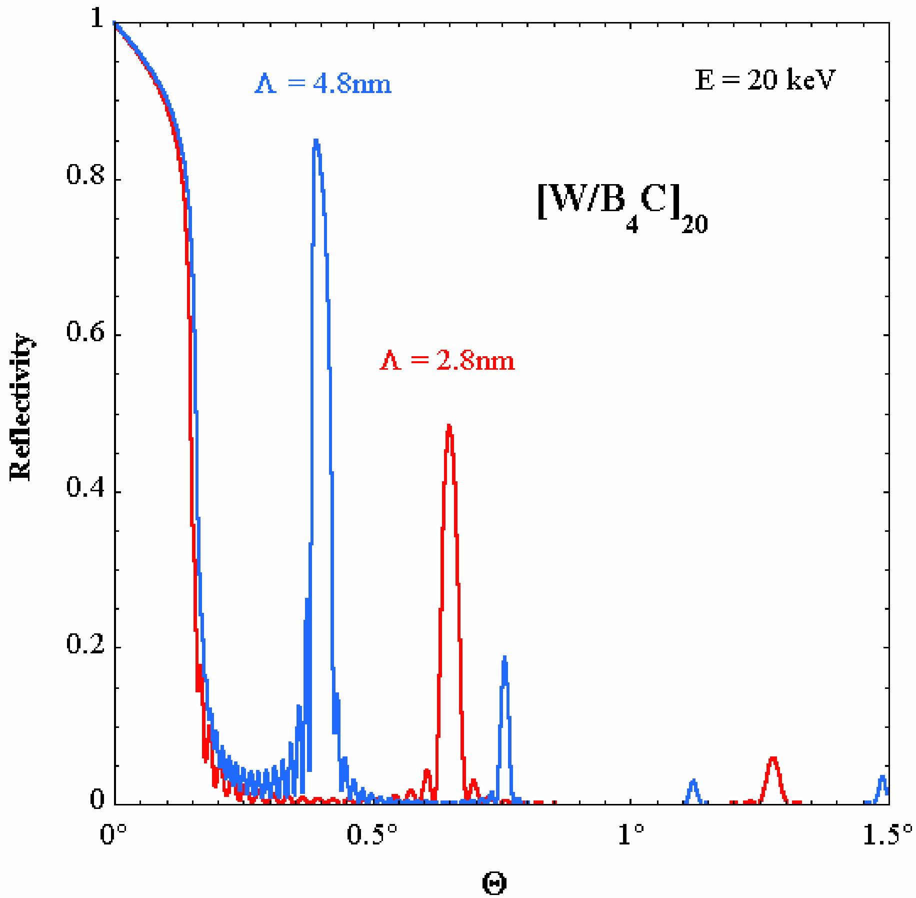
- **Bragg peaks and fringes due to interference**
- **Positions depend on E and Λ**
- **Intensities depend on $\Delta\rho$, N, σ …**

Corrected Bragg equation

$$m \cdot \lambda = 2 \cdot \Lambda \cdot \sqrt{n_2^{\,2} - n_1^{\,2} \cos^2 \theta}$$

For $\theta >> \theta_C \rightarrow m \cdot \lambda \approx 2 \cdot \Lambda \cdot \sin\theta$





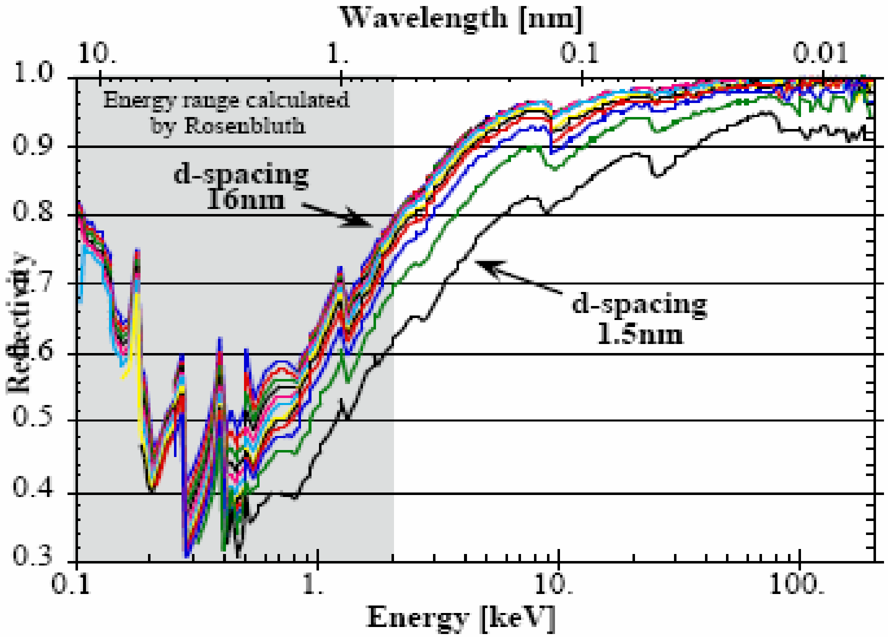
X-ray multilayer design

Materials choice – Basic rules:

1. **Select low-Z spacer material with lowest absorption (β_{spacer})**
2. **Select high-Z absorber material with highest reflectivity with spacer ($\delta_{abs} - \delta_{spacer}$)**
3. **In case of multiple choices select high-Z material with lowest absorption (β_{abs})**
4. **Make sure that both materials can form stable and sharp interfaces (lower d-spacing limit)**

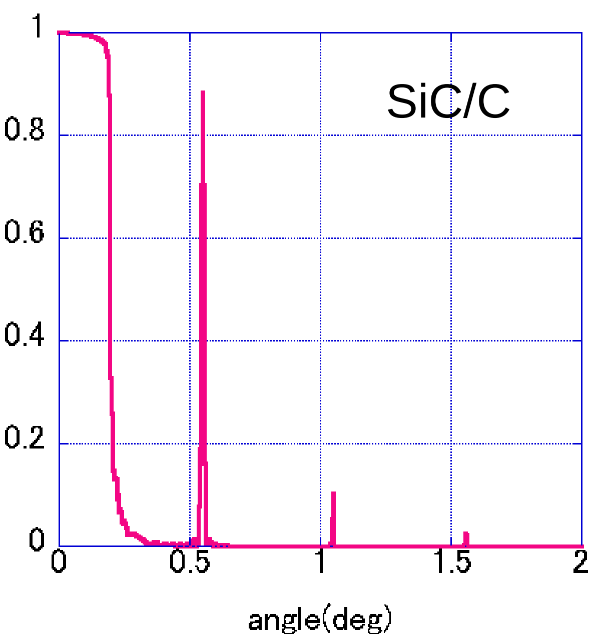
Computational search algorithms

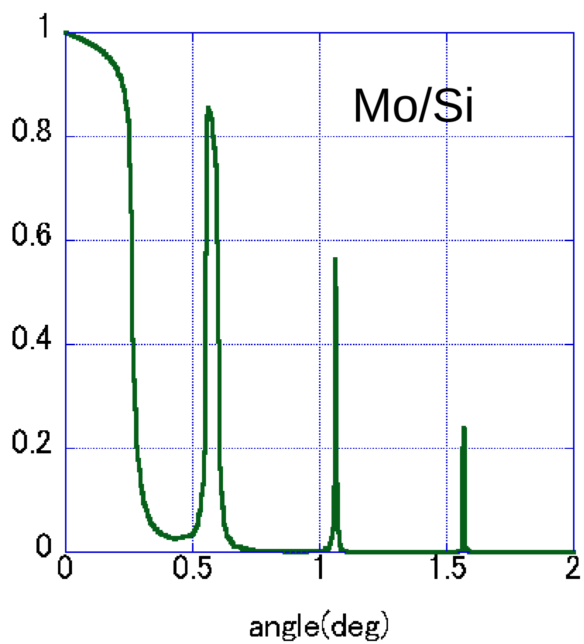
- **Soft X-rays: A.E. Rosenbluth (1988)**
- **Hard X-rays: K. Vestli (1995)**

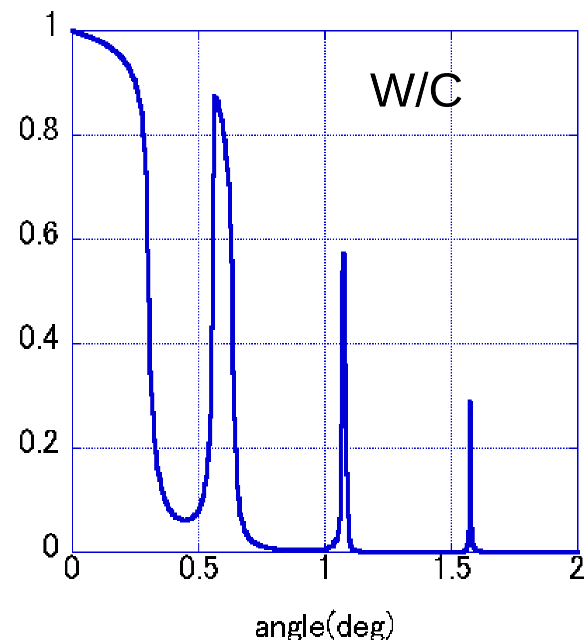


[9] K. Vestli, E. Ziegler, Rev. Sci. Instr. 67, 3356 (1996)

	Atomic number	Density (g/cm3)
C	6	2.26
Si	14	2.33
SiC	-	3.2
Mo	42	10.22
W	74	19.35







X-ray multilayer design

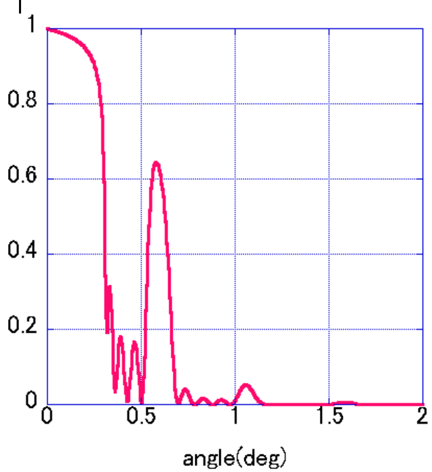
Period number N:

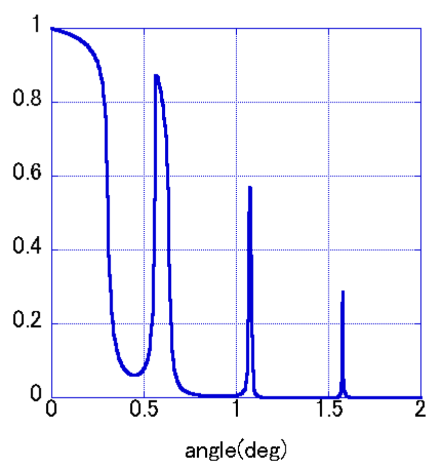
Peak versus integrated reflectivity:

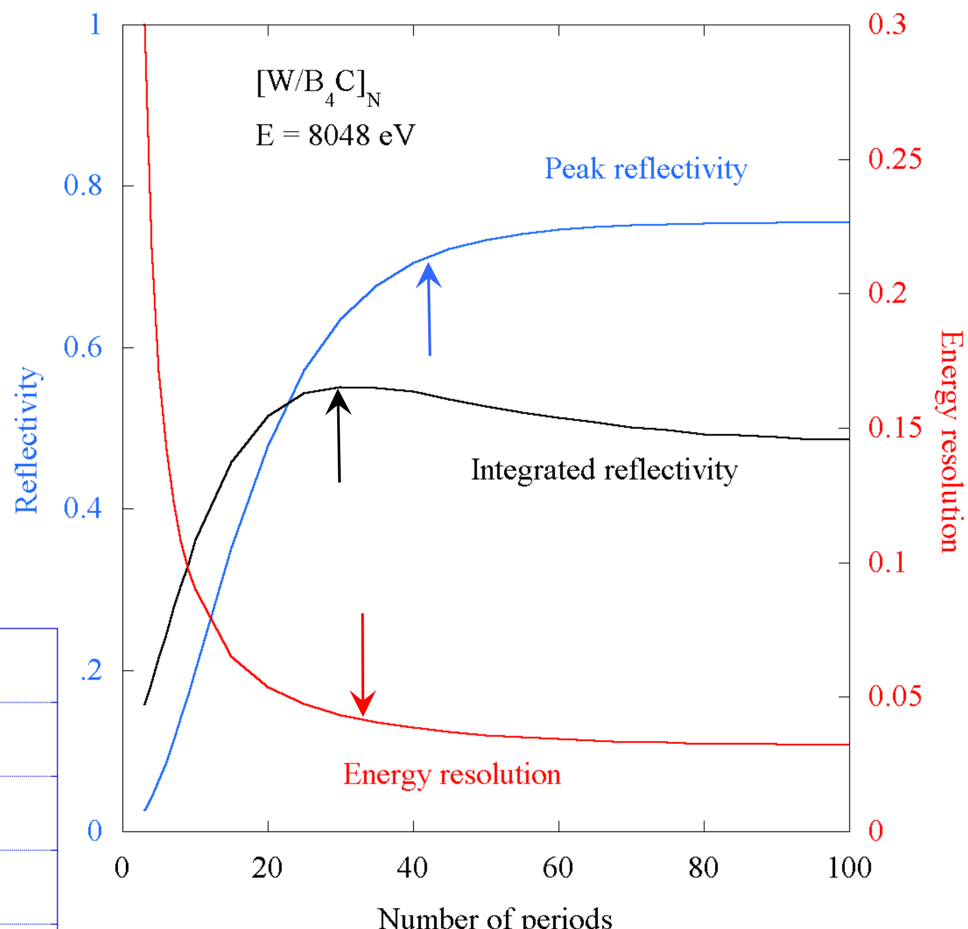
- **R_{peak} increases with N up to extinction**
- **$\Delta E/E$ decreases ~ 1/N in kinematical range**
- **R_{int} is maximum before extinction**

High and low resolution MLs

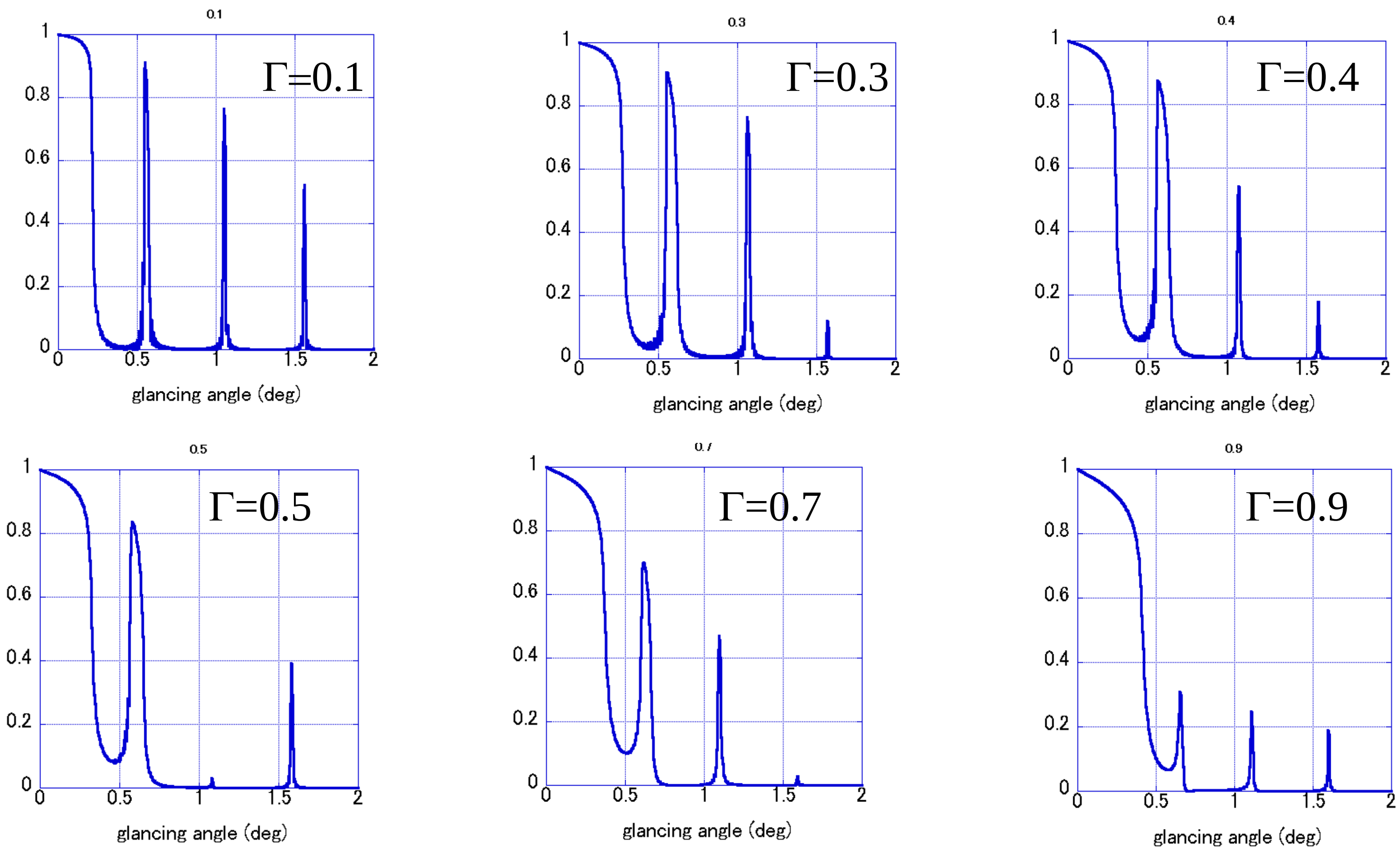
Optimize N according to needs !







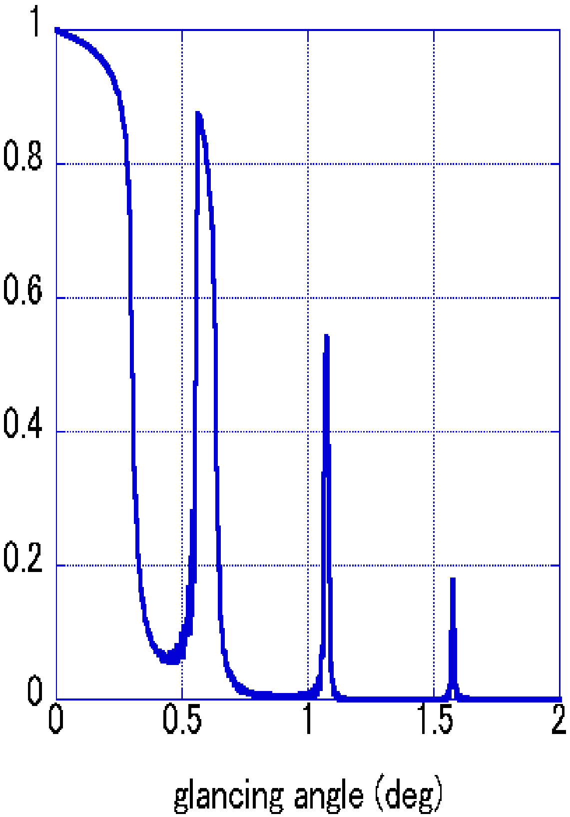
W/C multilayer $\Gamma = t_W / \Lambda$



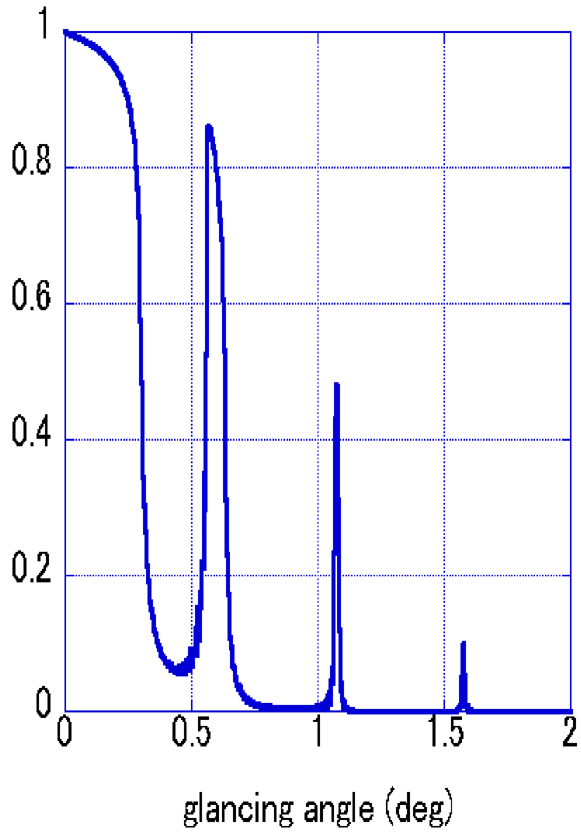
Effect of interlayer diffusion

C/W Λ=6.9 nm Γ=0.4

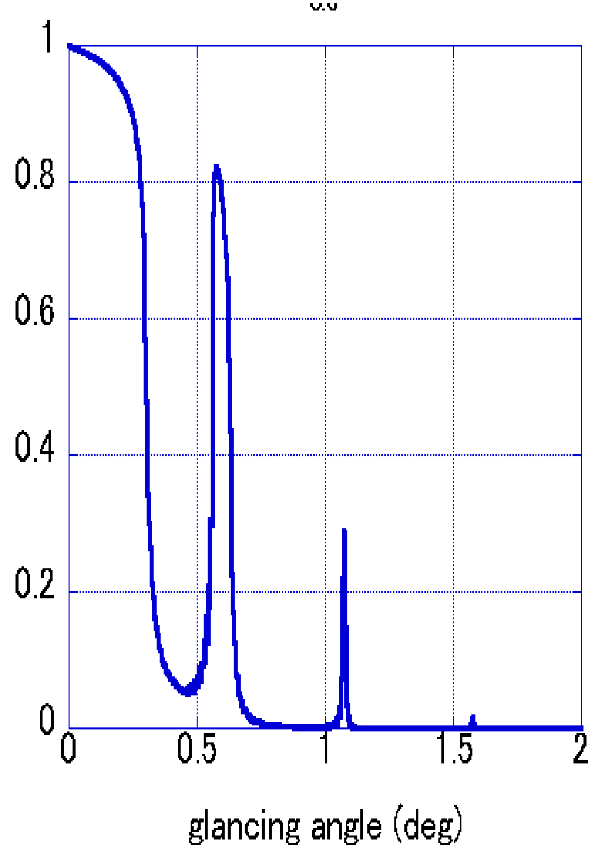
0 nm



0.3 nm



0.6 nm



X-ray multilayer fabrication

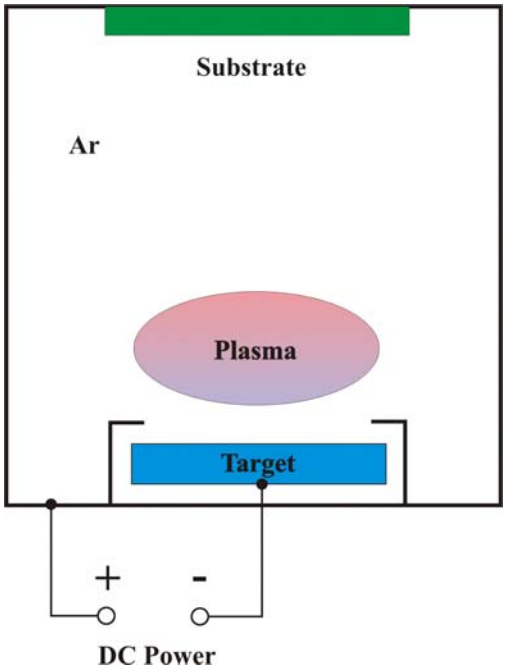
Deposition techniques	Vacuum	Particle energy	Deposition rate	Deposition area
Thermal evaporation	HV (UHV)	Low	Low	Small
E-beam evaporation	UHV	Low	Low	Small
Magnetron sputtering	HV (+Gas)	High	High	Large
DECR sputtering	HV (+Gas)	High	Low	Medium
Ion beam sputtering	UHV (+Gas)	Very high	High	Medium
Pulsed laser deposition	HV	Very high	High	Medium

- **Characteristics may vary depending on equipment and application**
- **Magnetron sputtering most widely used for X-ray multilayer fabrication**
- **Vacuum and purity important for EUV and soft X-rays**
- **High particle energy favors very thin and uniform layers**

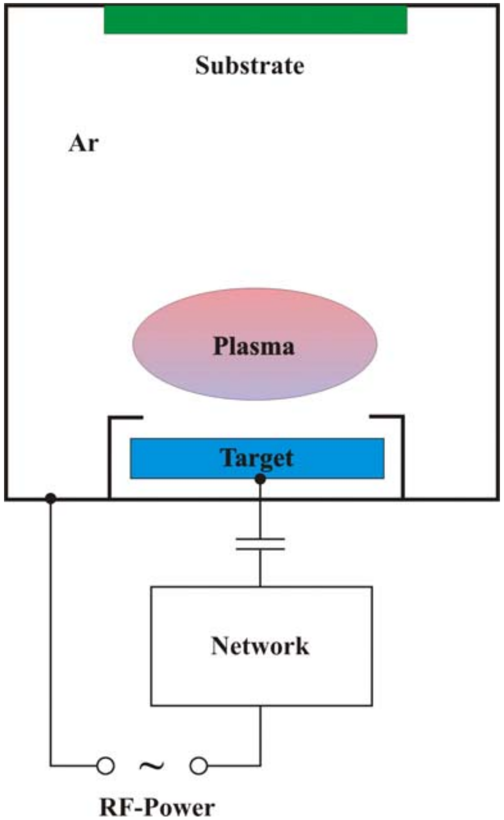
X-ray multilayer fabrication

Sputtering techniques

DC (conductors)



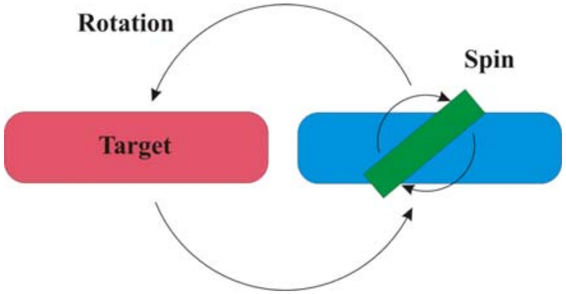
RF (insulators)

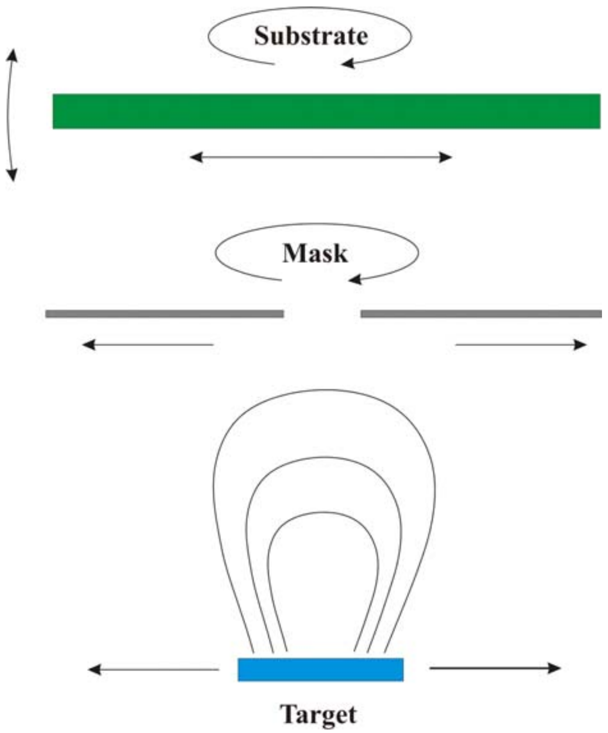


X-ray multilayer fabrication

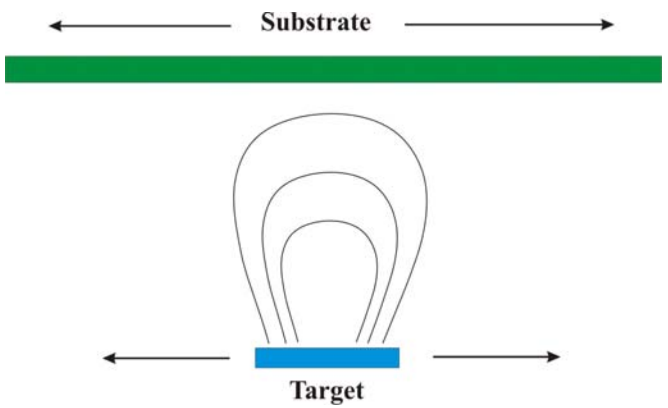
Large area coatings (uniform, gradient)

- **Relative motion source - substrate**
- **Masking techniques**



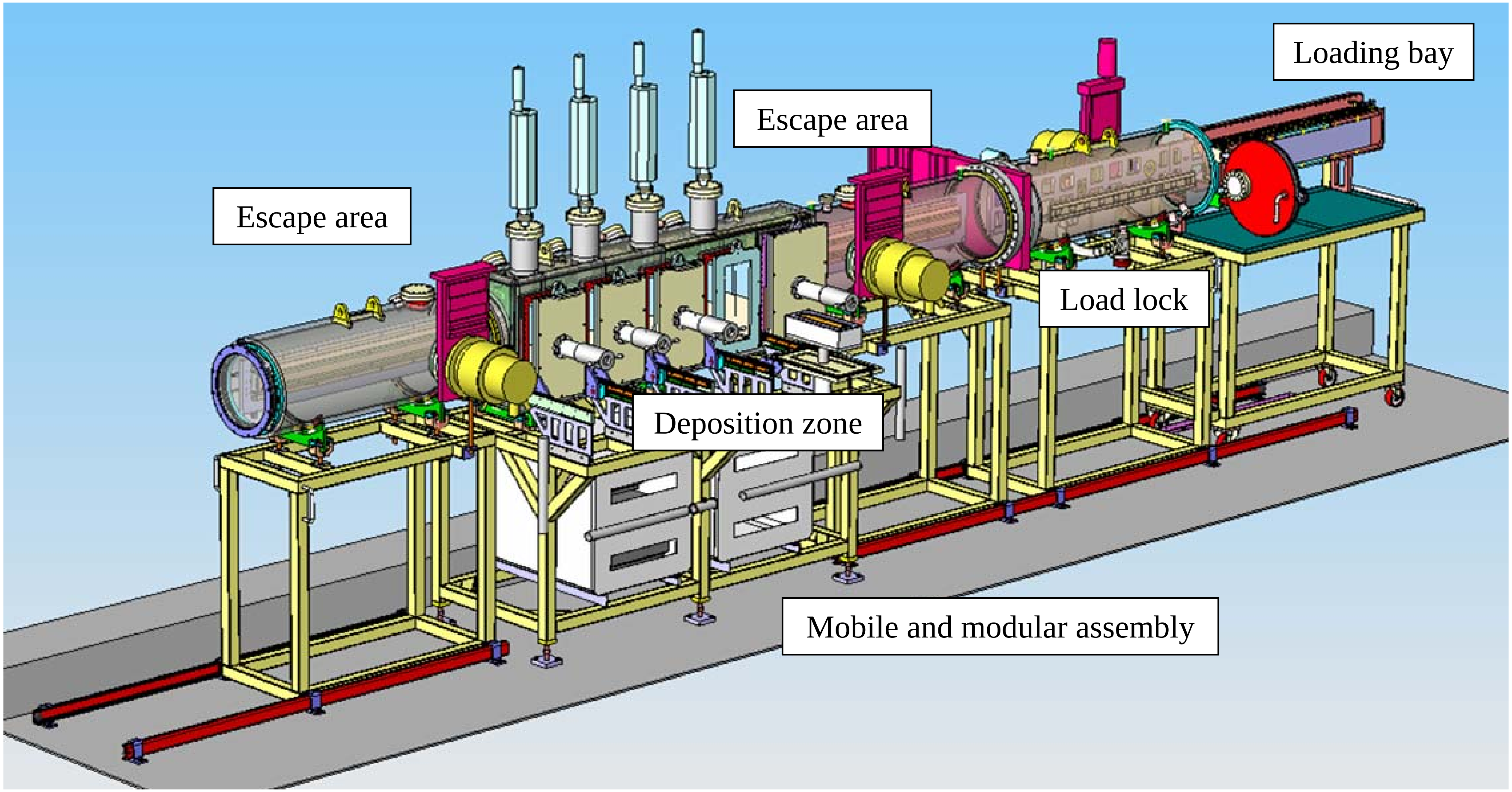


$$t(\vec{r}) = \int \varphi(\vec{r},\vec{r}\,')\frac{d\vec{r}\,'}{v(\vec{r}\,')}$$



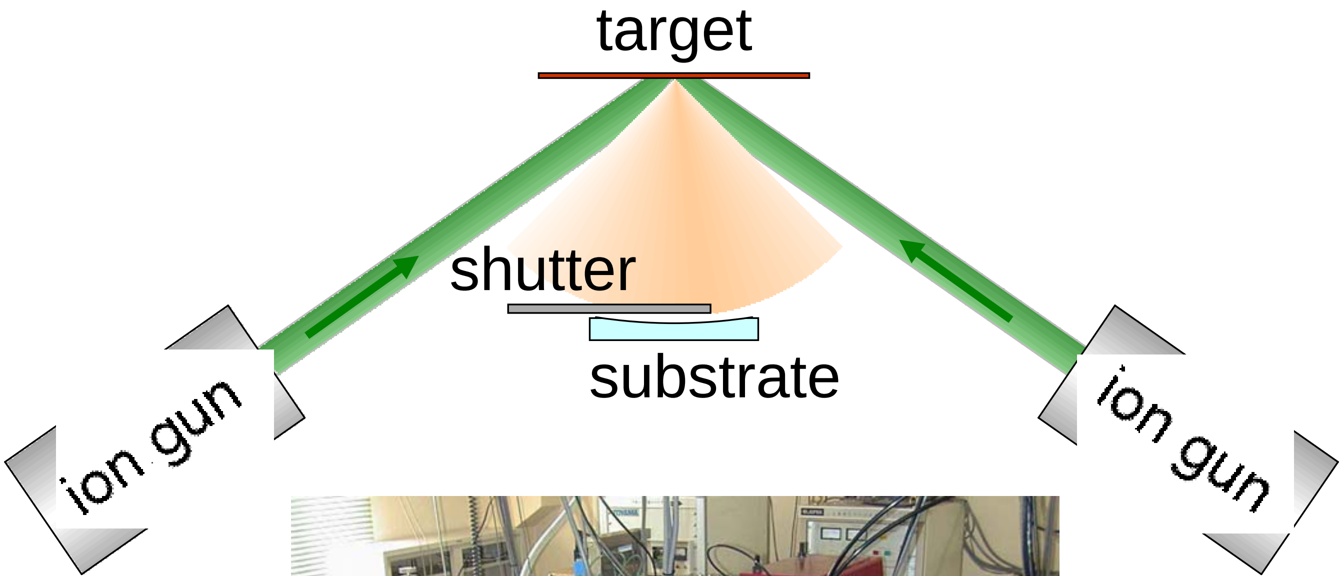
X-ray multilayer fabrication

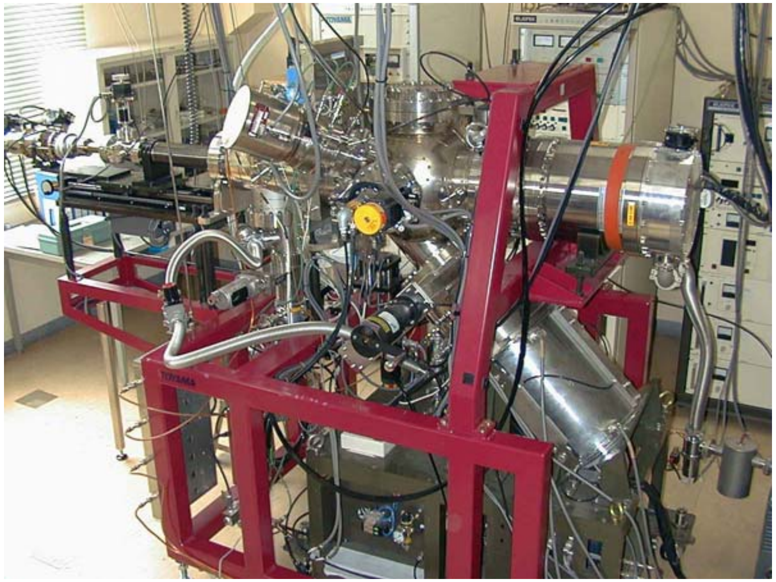
ESRF magnetron sputter deposition system



X-ray multilayer fabrication

<u>Sputtering techniques</u> (Ion beam sputtering at Tohoku Univ.)





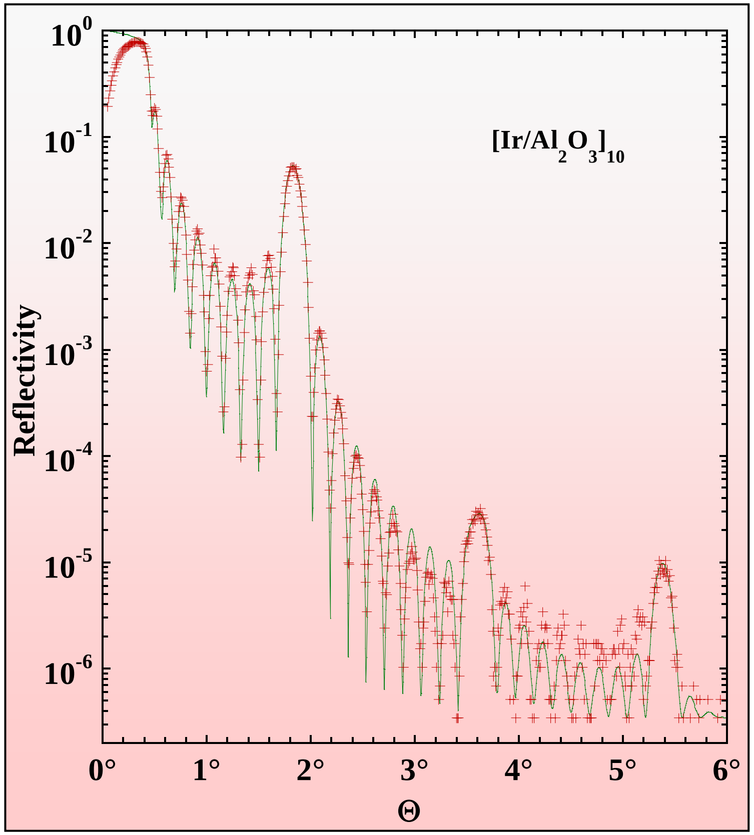
<u>TEM</u>
Mo/Si
D = 6.9 nm
λ = 13.5 nm
ϕ = 5°

<u>TEM</u>
Cr/Sc
D = 1.77 nm
λ = 3.1 nm
ϕ = 27.5°

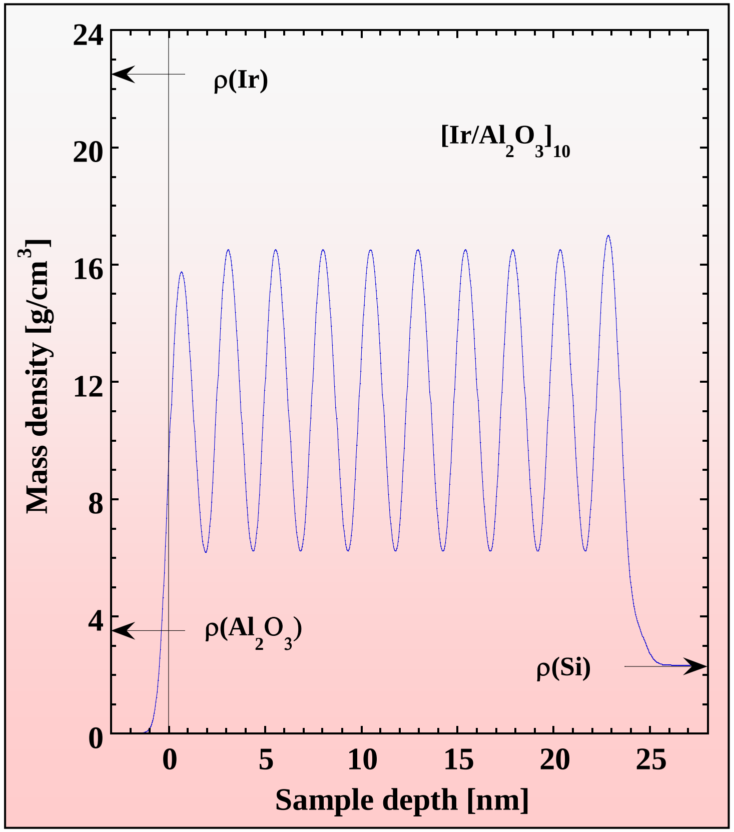
X-ray multilayer characterization

X-ray reflectivity

Simulation of x-ray reflectivity



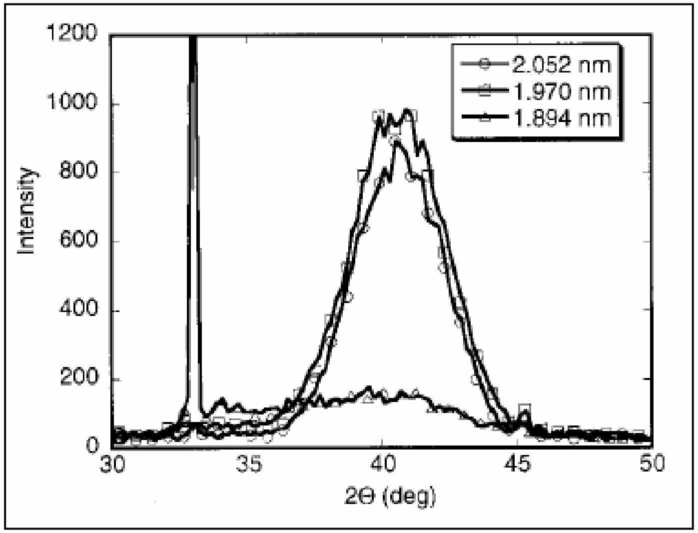
Vertical density profile



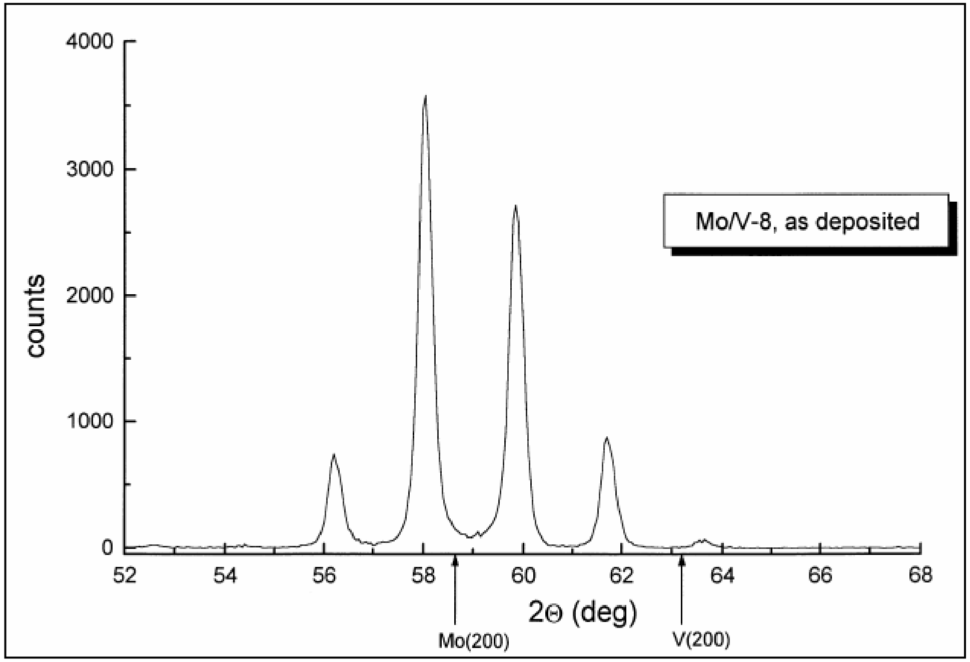
X-ray multilayer characterization

X-ray scattering

- **Access to atomic structure**
- **Phase transitions**
- **Coherence lengths**
- **Superlattice formation**



[16] S. Bajit et al, J. Appl. Phys. 90, 1017 (2001)



[17] D.L Beke et al, Vacuum 50, 373 (1998)

X-ray multilayer characterization

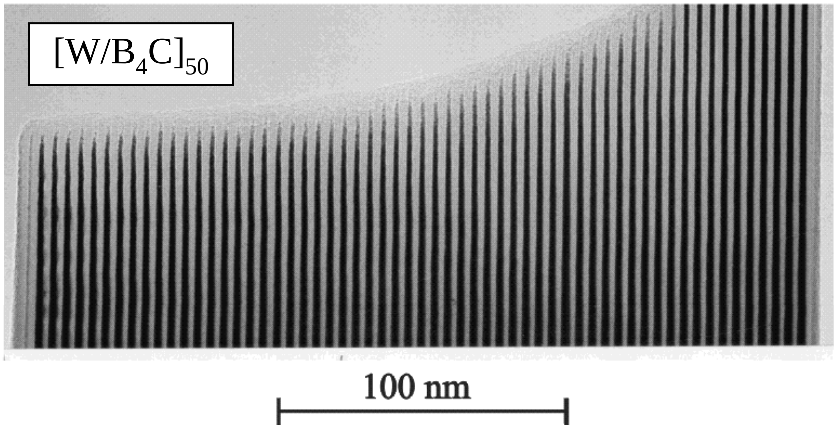
Transmission electron microscopy (TEM)

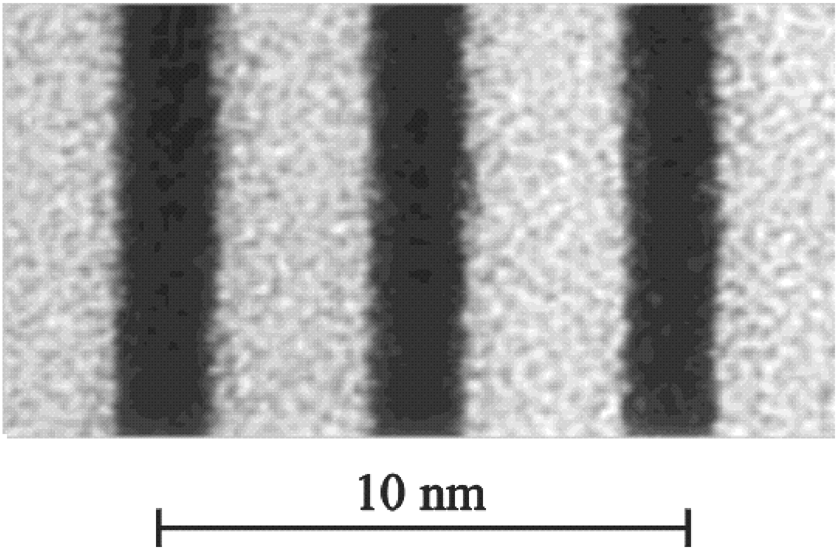
- **Fabrication errors**
- **Roughness evolution**
- **Crystallinity**
- **Interface diffusion**

↓

Complementary to x-ray measurements !

R. Scholz, MPI Halle, Germany





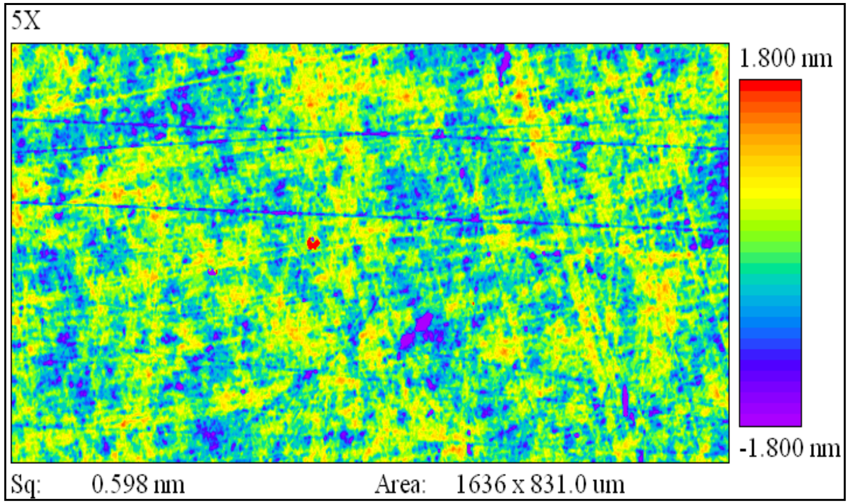
ESRF

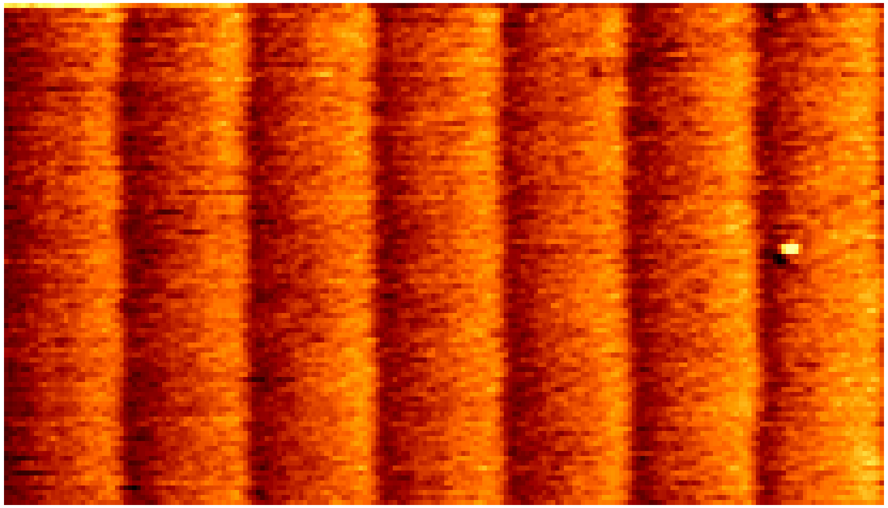
X-ray multilayer/mirror characterization

Surface metrology

- **Atomic Force Microscopy (AFM)**
- **Interferometry**
- **Long Trace Profiler (LTP)**
- **From nm to m scale**
- **No depth information !**

A. Rommeveaux, ESRF Optics Group





ESRF

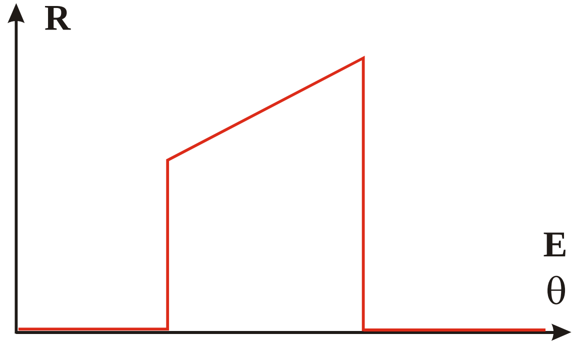
X-ray multilayer design

Aperiodic design:

Goal: ML with particular reflectivity profile

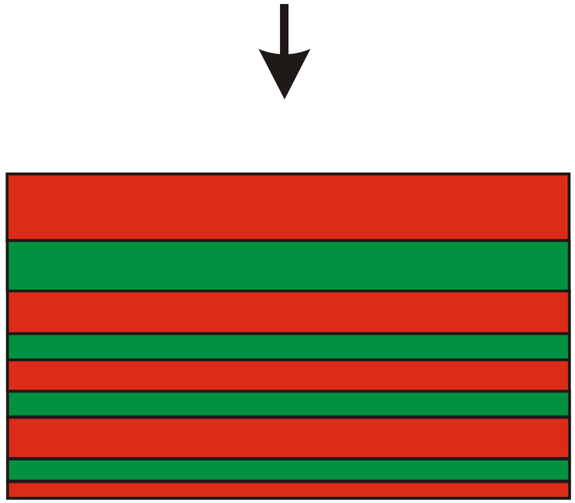
- **R = R(θ) for E = const**
- **R = R(E) for θ = const**

Find vertical composition profile



Method:

- **Derive analytical expression for reflectivity**
- **Do inversion to obtain 1st estimate for layer sequence**
- **Apply fit algorithm to optimize the structure**



[12] I.V. Kozhevnikov et al, Nucl. Instr. And Meth. A 460, 424 (2001)

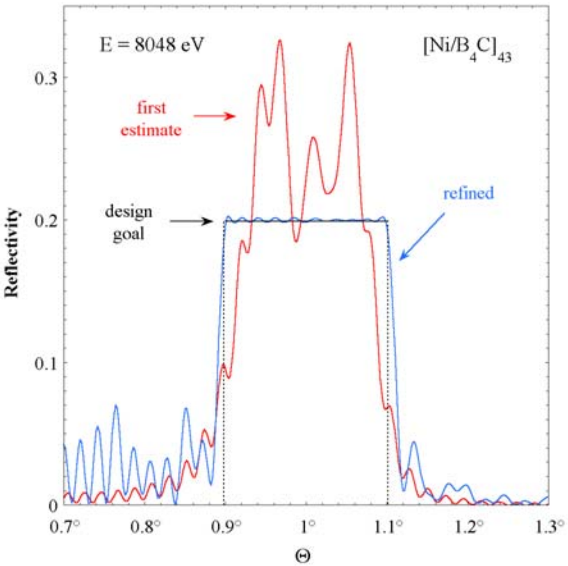
X-ray multilayer design

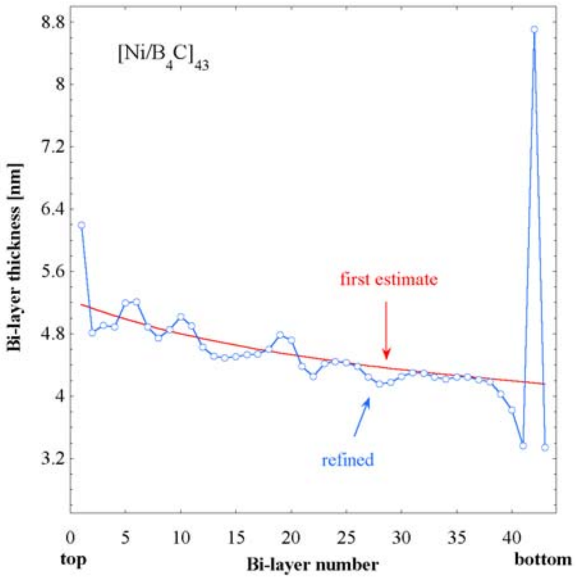
Example:

[13] Ch. Morawe et al, Nucl. Instr. And Meth. A 493, 189 (2002)

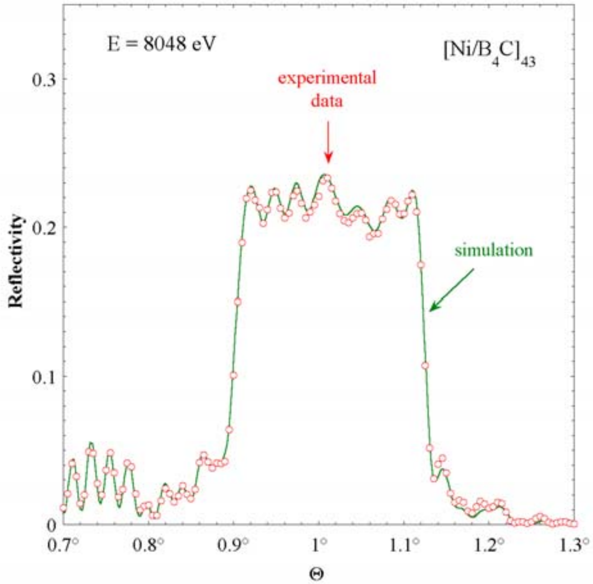
- **Ni/B_4C structure**
- **R(θ) = const over 20% bandwidth**

Theoretical design

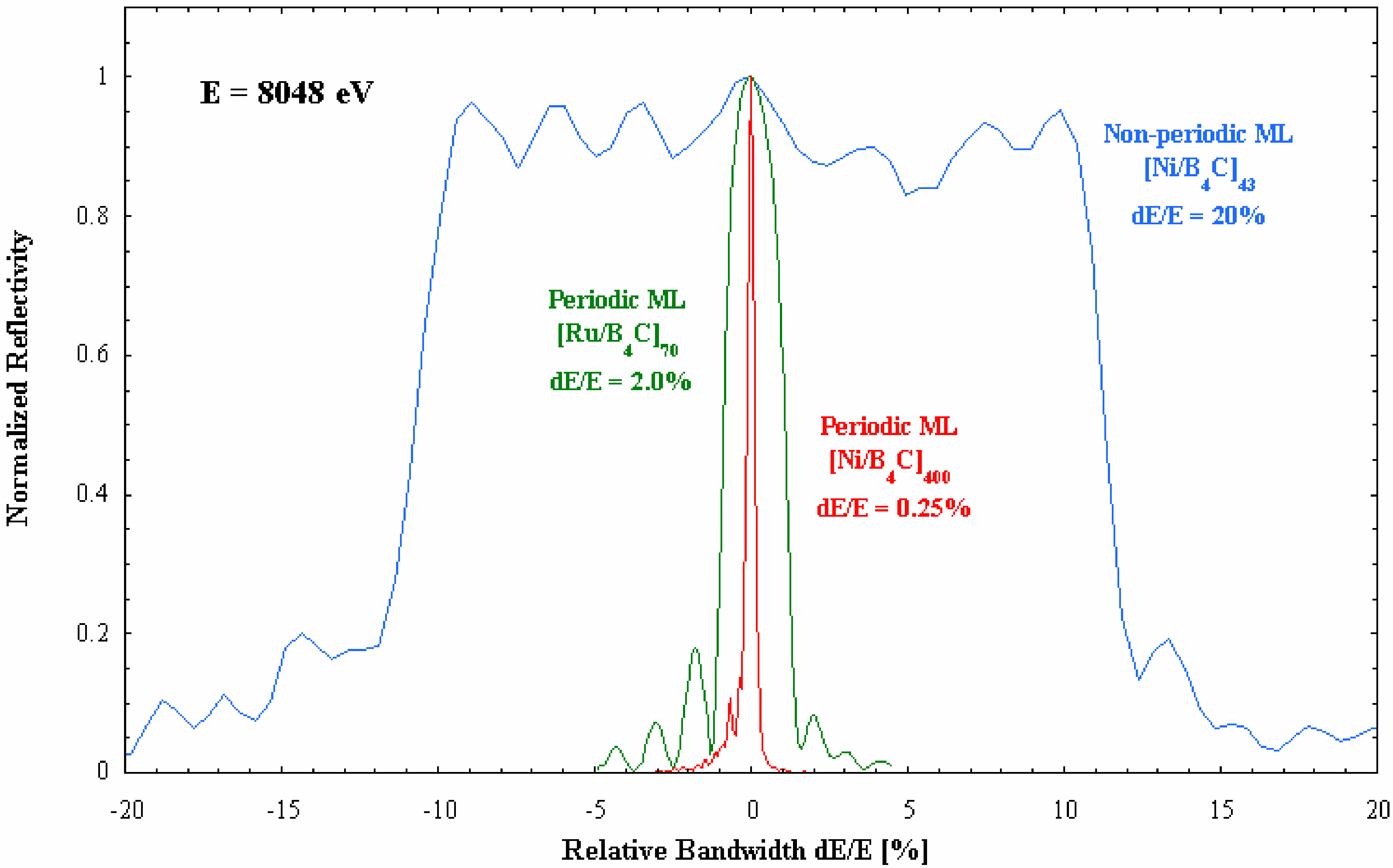




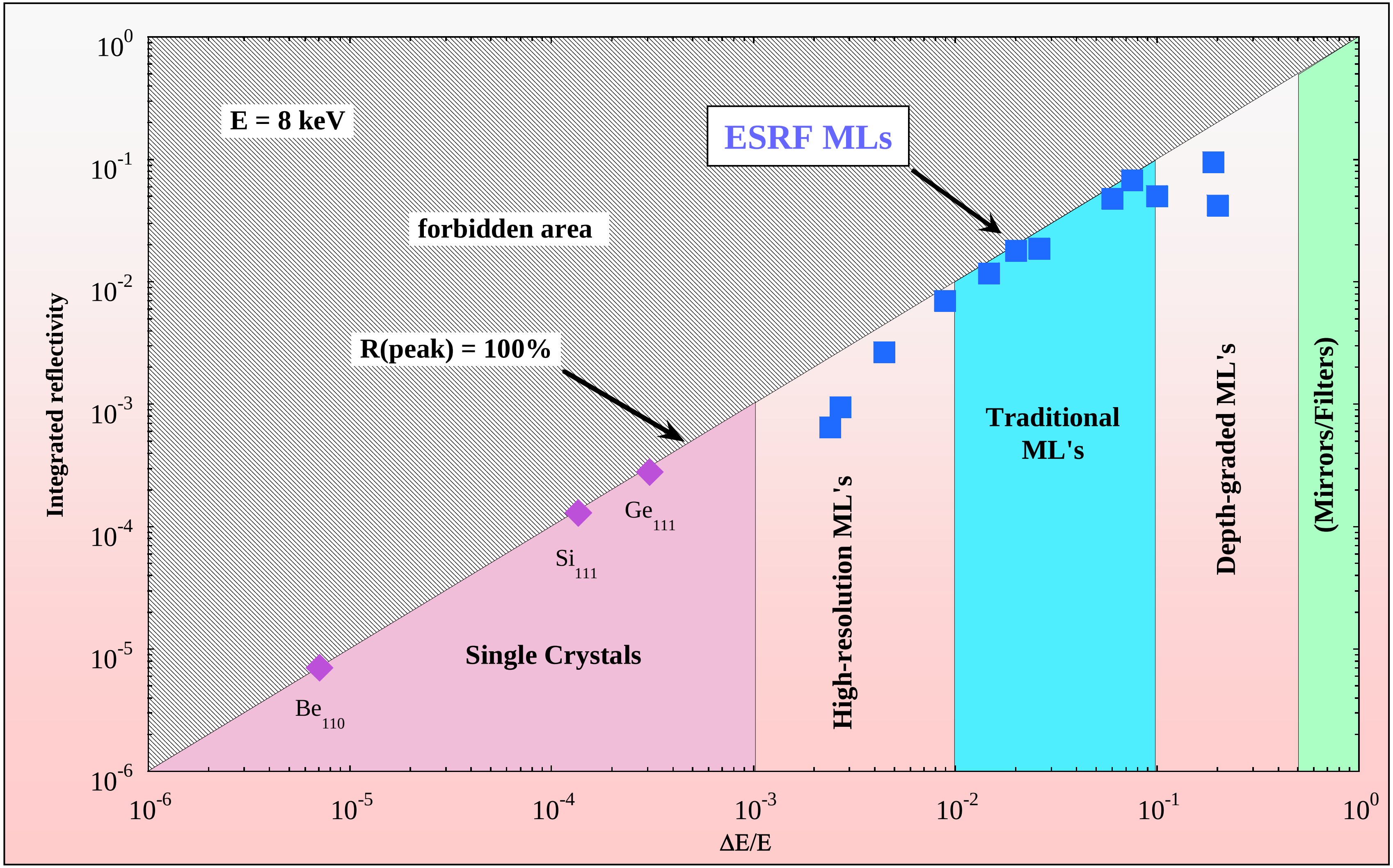
Experimental result



Energy resolution of multilayers

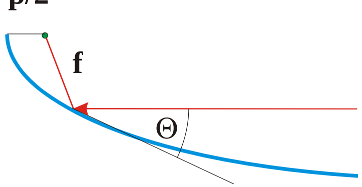
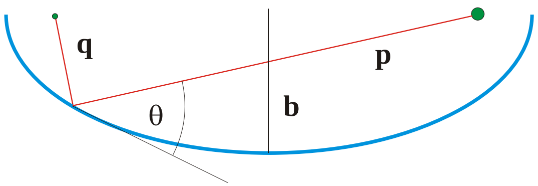
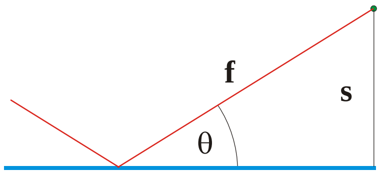


Reflecting X-ray optics – Overview



Laterally graded multilayers

Surface curvature and beam divergence define lateral gradient dΛ /dx

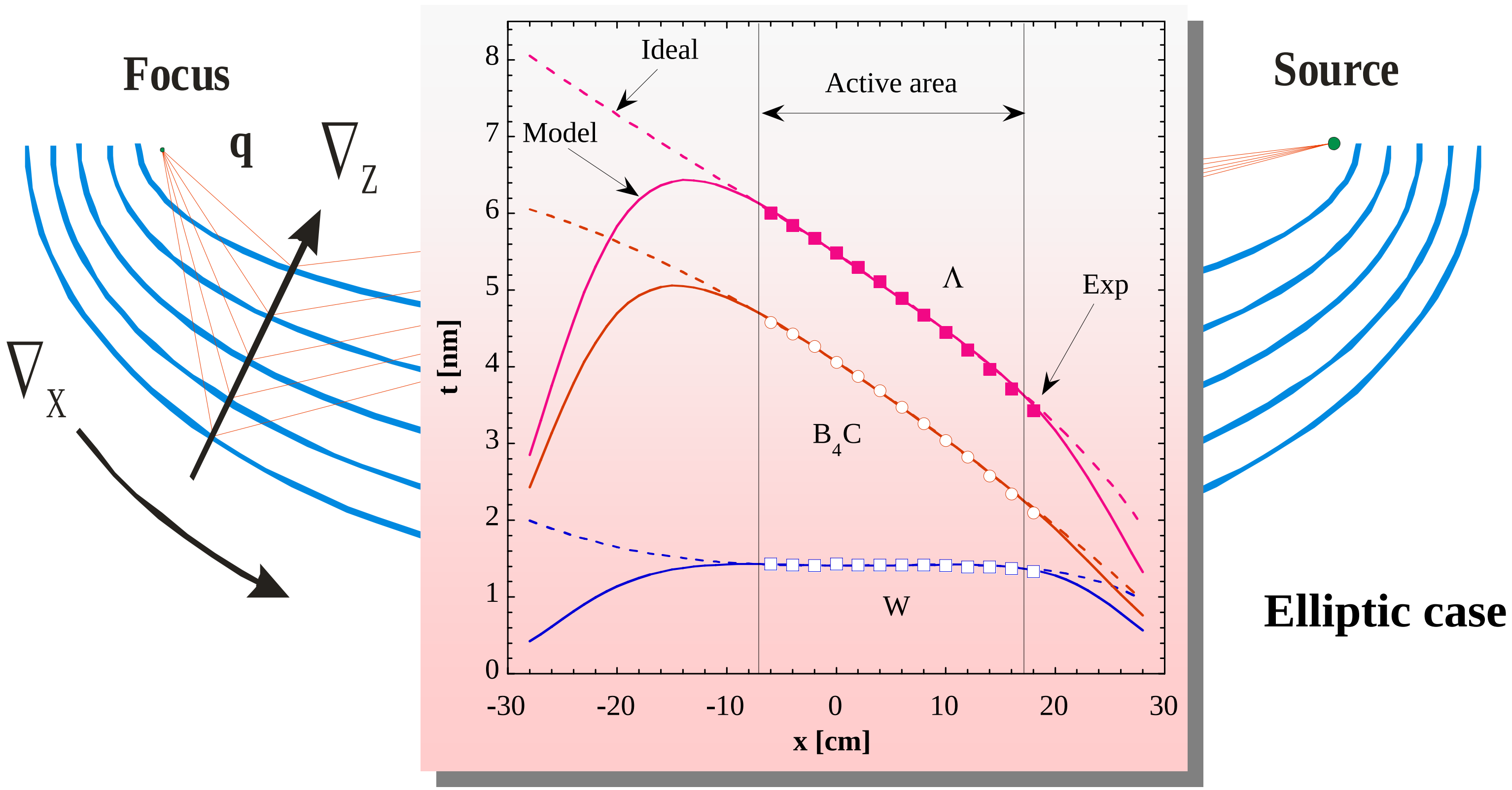
Shape	**Parabolic**	**Elliptic**	**Flat**
Angle θ	$\sin\theta = \sqrt{\dfrac{p}{2f}}$	$\sin\theta = \dfrac{b}{\sqrt{pq}}$	$\sin\theta = \dfrac{s}{f}$
Geometry			

d-spacing including refraction correction
(modified Bragg equation)

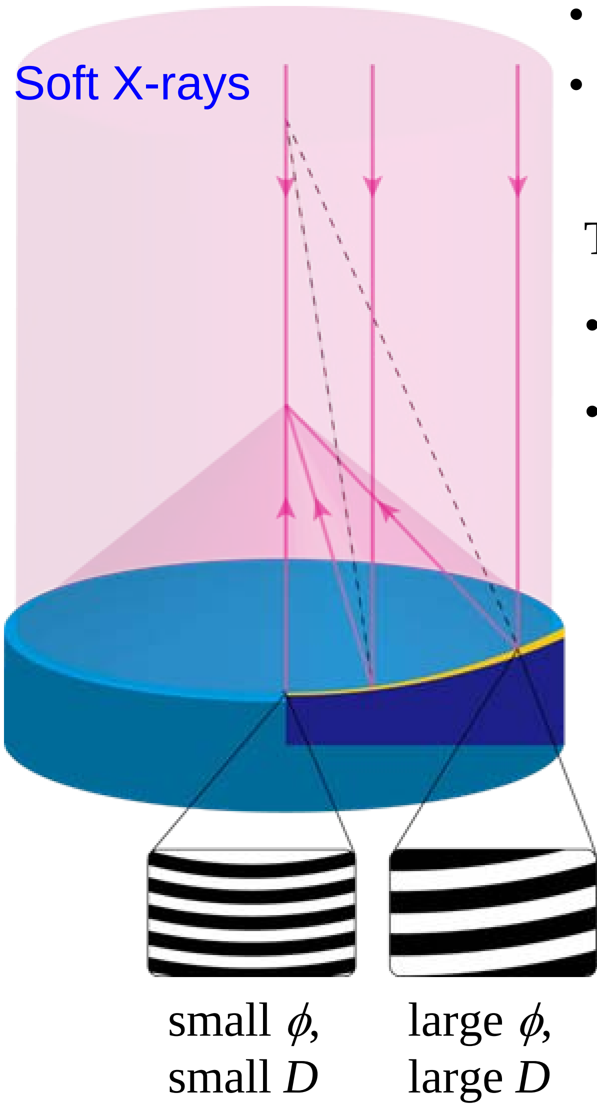
$$\Lambda = \frac{\lambda \cdot m}{2\sqrt{n^2 - \cos^2\theta}}$$

Laterally graded multilayers

- Divergent beam, focusing applications with curved substrates
- Incidence angle variation → laterally graded multilayers
- Additional depth gradient negligible (< 10^{-5})



laterally graded multilayers

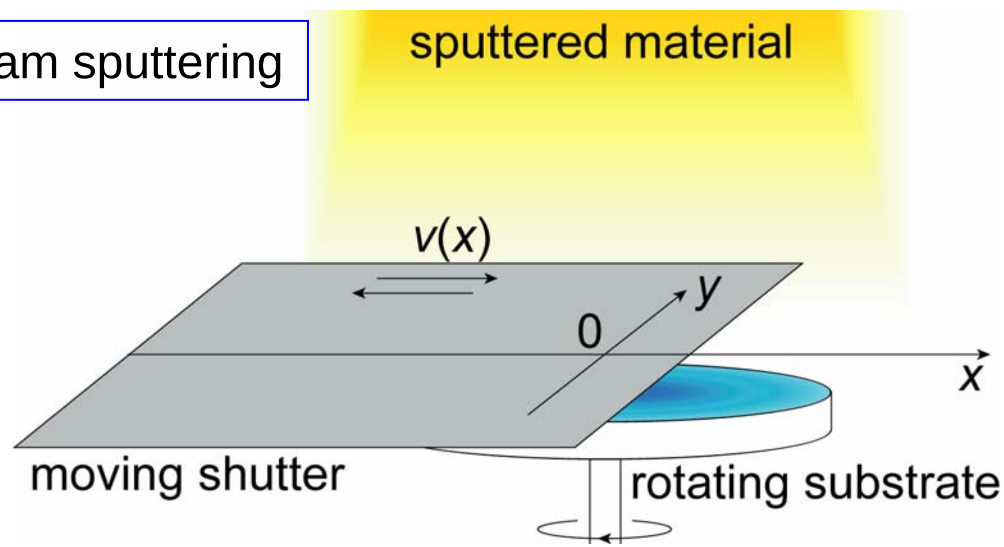


- Divergent beam, focusing applications with curved substrates
- Angle of incidence variation $\rightarrow$ laterally graded multilayers

Thickness control by

- Relative motion of source - substrate (in magnetron sputtering)
- Moving shutter (for a large gradient)

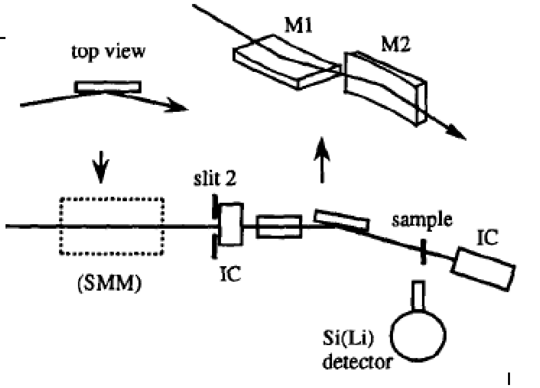
Ion beam sputtering

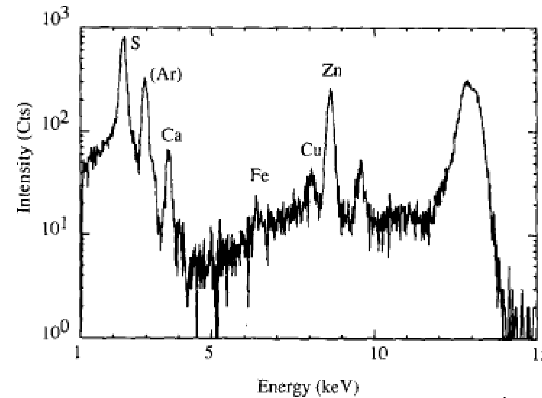


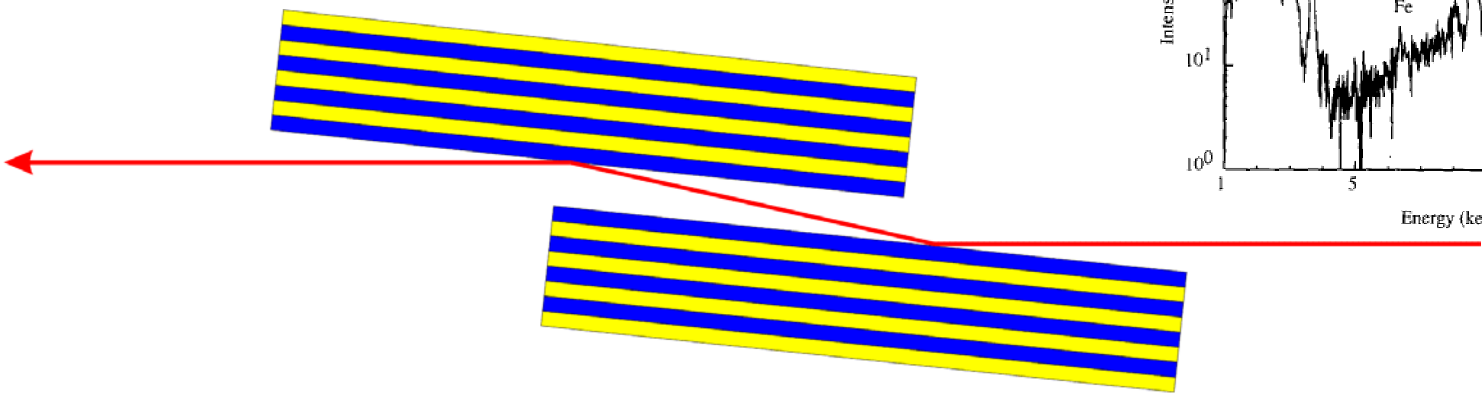
Applications

Synchrotron optics: Multilayer high flux monochromators

- Two bounce optics
- 100x larger bandwidth compared with Si(111)
- Harmonics suppression due to refraction and filling factor
- Radiation and heat load issues !



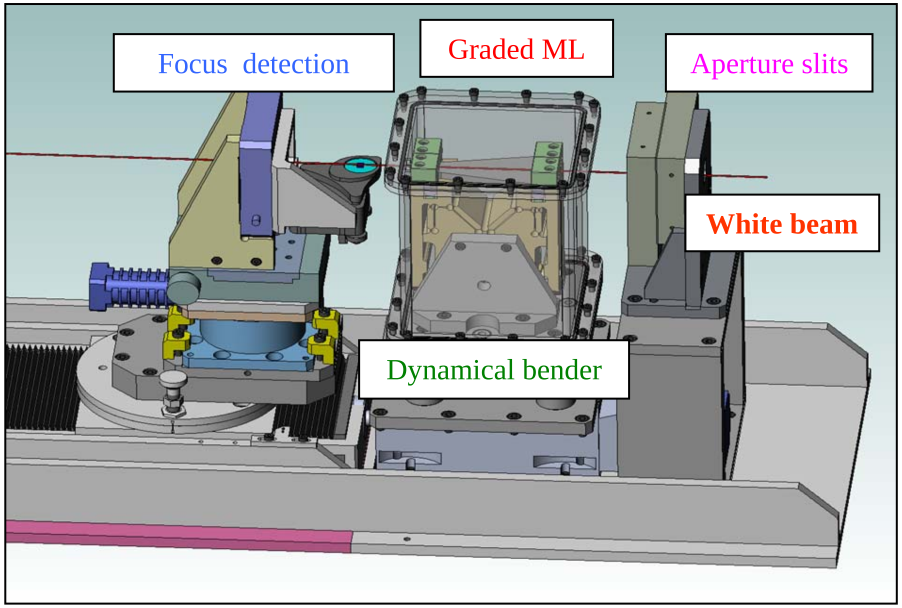


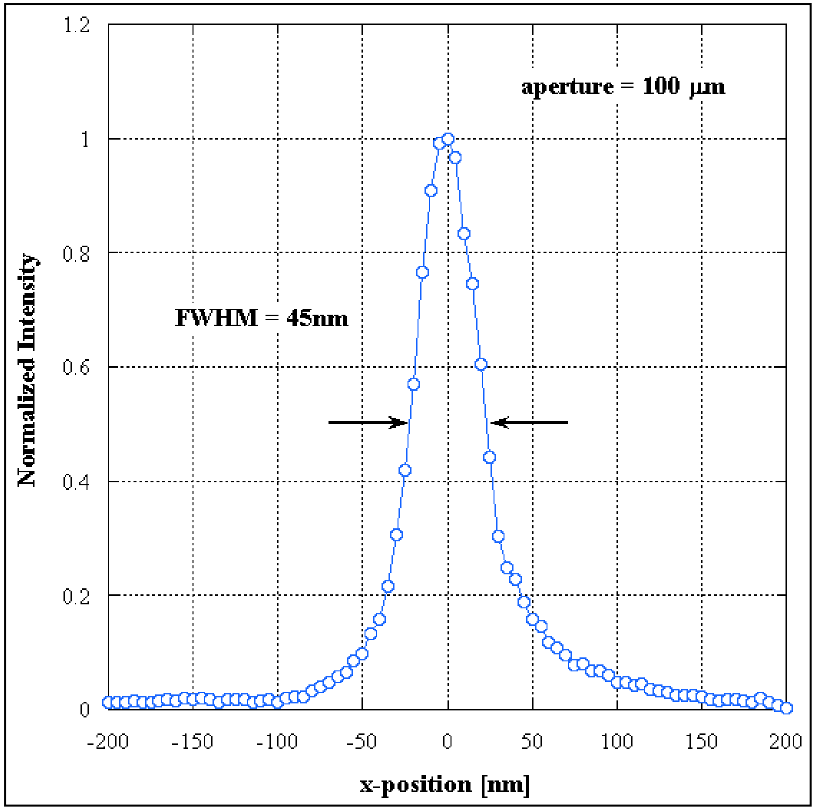


Applications

ESRF focusing experiment

- **Full undulator spectrum**
- **Vertical line focus**
- **Dynamical bender**
- **Raw data 45 nm FWHM @ 100 μm aperture**



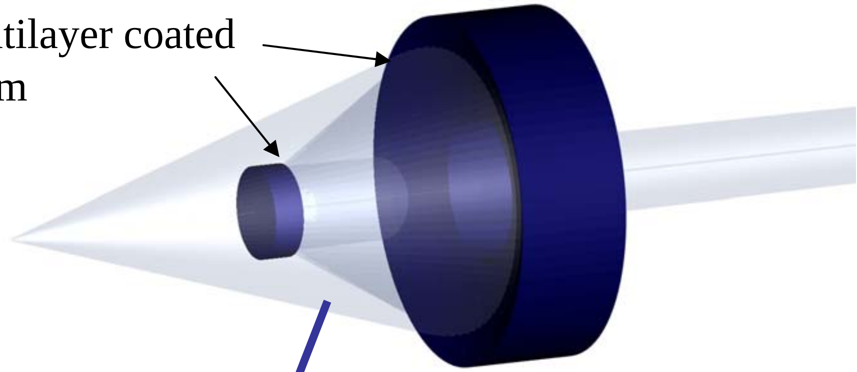


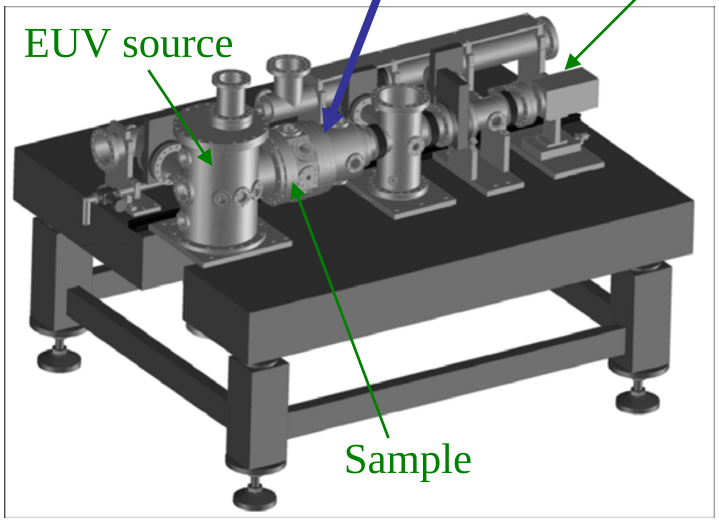
[24] Ch. Morawe et al, Proc. SPIE 6317 (2006)

Multilayer application –Microscope–

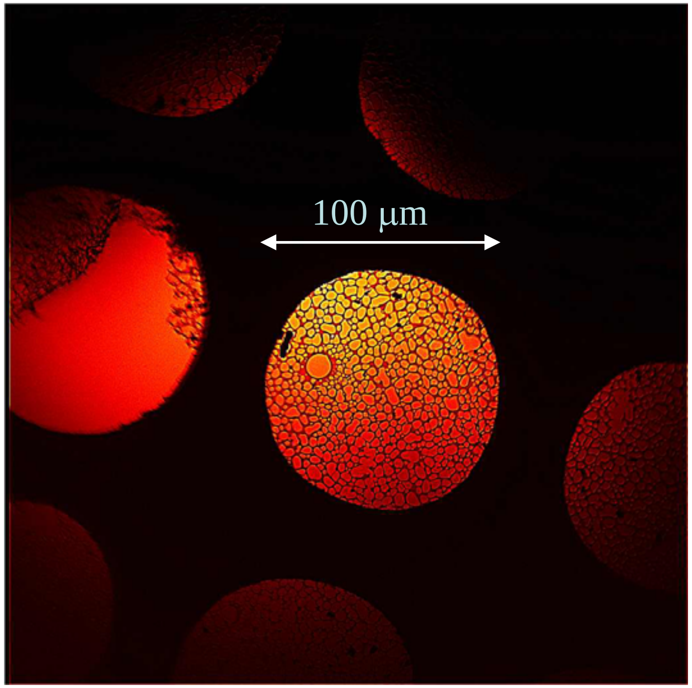
Schwarzschild objective for laboratory microscope

Mo/Si multilayer coated
λ = 13.5 nm



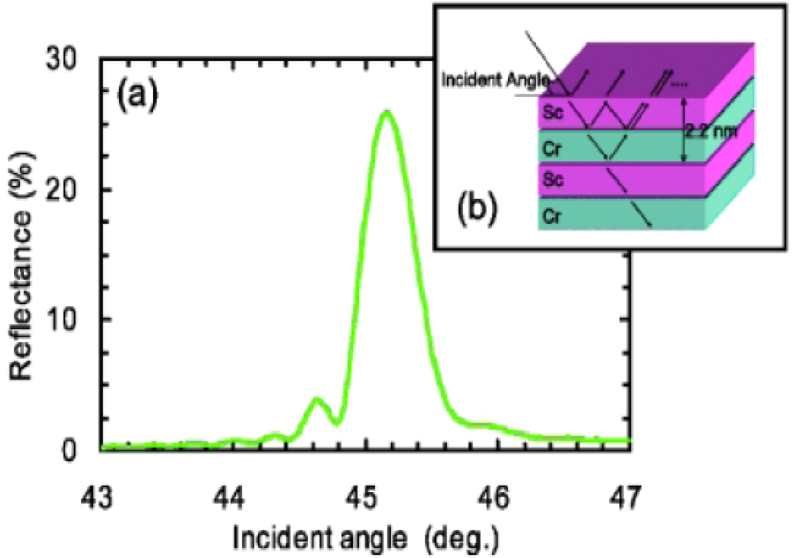


Transmission image of
polymer microgrids (50×)

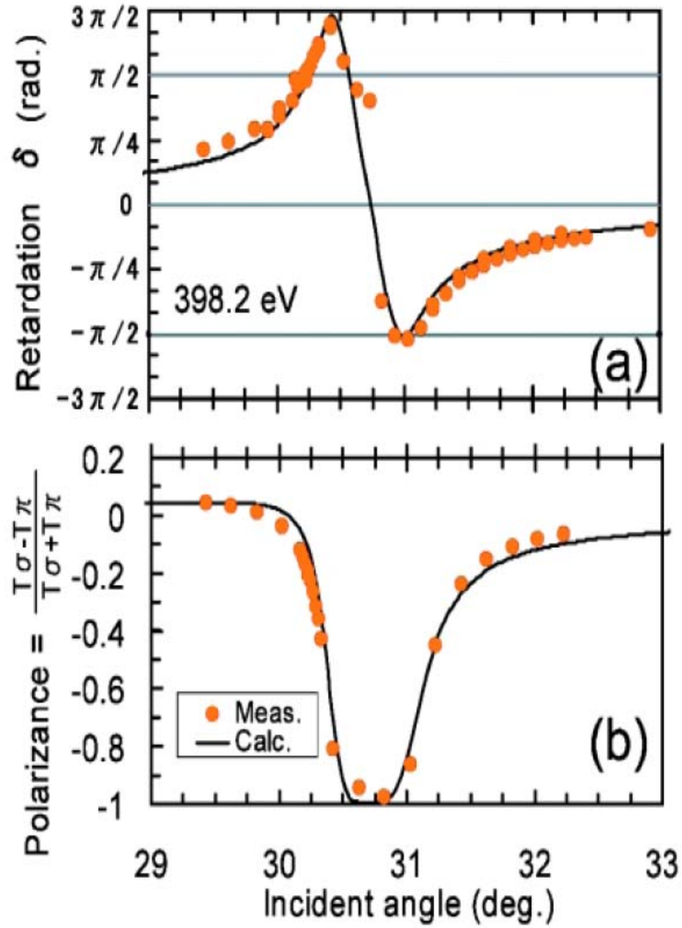


Yamamoto and Yanagihara group, Tohoku Univ.

Multilayer phase plate



Polarization characteristics of a transmitting Sc/Cr phase shifter.



Reflectance of a [Sc(2.2 nm)/Cr(2.2 nm)]$_{200}$ multilayer, which works as a reflecting polarizer.

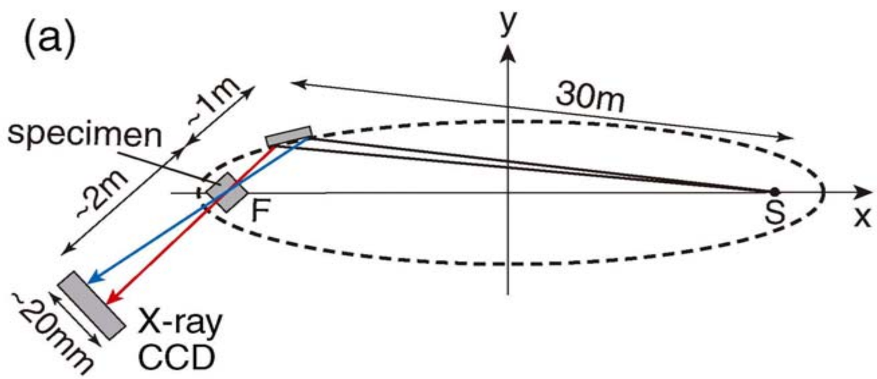
M. Suzuki and Hirono,Hoshako(放射光), Nov. 2006, Vo.19 No6, pp.444-453

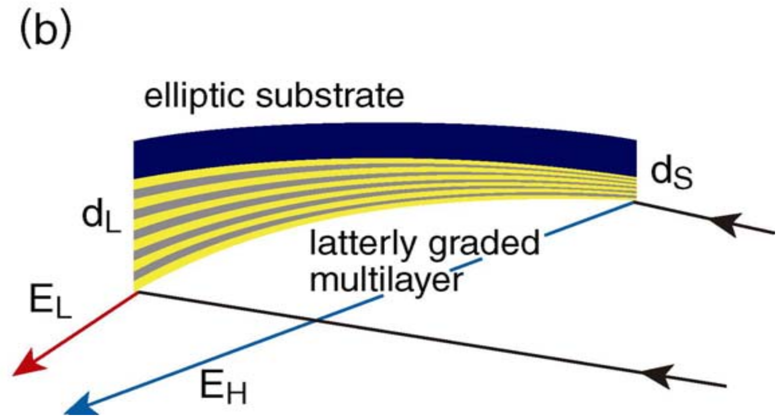
Application

Simultaneous measurement of X-ray reflectivity curve

Laterally d-graded multilayer on an elliptic surface

T. Matsushita et. al., 2008



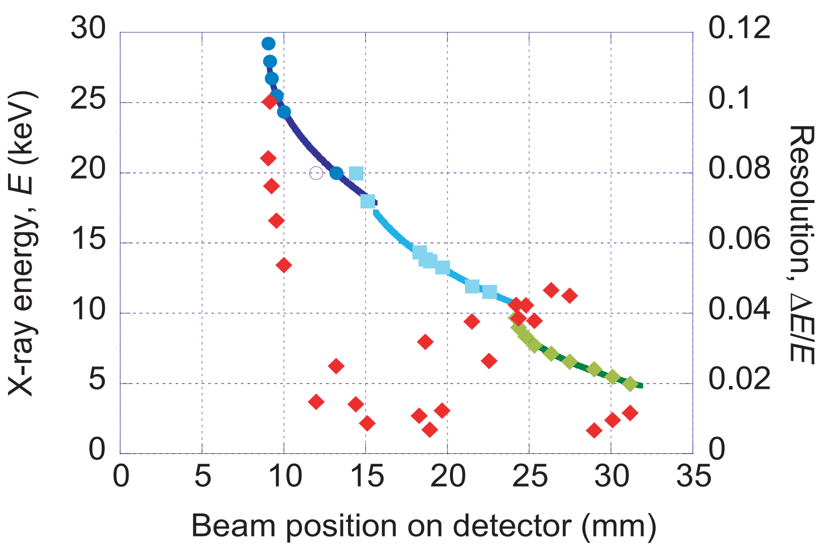


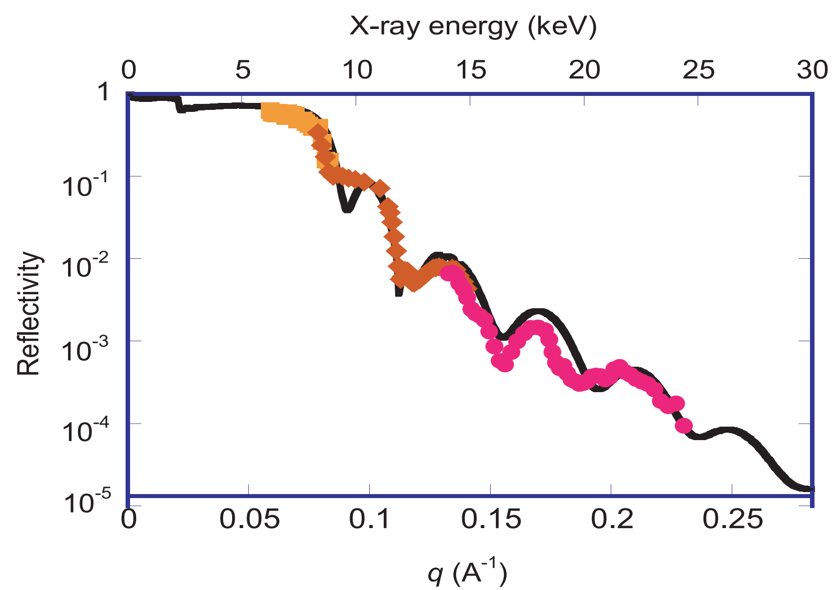
focusing:

elliptic substrate

dispersion:

laterally graded multilayer





Other issues

- Brewster’s angle,
- Other advanced surface polishing techniques
- Influence of imperfections

  lower reflectivity,

  broader multilayer reflectivity curve,

  diffuse scattering
- Radiation damages
- Standing waves

On-going developments

- Mirror and multilayers for handling much more coherent X-ray beam

  slope error < 0.1 μrad, roughness < 0.05 nm

  fabrication and characterization

- Nanometer focusing optics
- Multilayers with shorter period
- Stable mount, tables and support

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CXRO_ALS web site: “X-ray Interactions with Matter” http://henke.lbl.gov/optical_constants