

## HARD X-RAY OPTICS FOR SYNCHROTRON BEAMLINES



Synchrotron radiation

- Principles of X-ray optics
- Mirrors
- Diffractive optics
- X-ray micro-/nano-focusing
- Summary

Ray Barrett X-ray Optics Group Leader European Synchrotron Radiation Facility Grenoble, France



barrett@esrf.fr

#### A STORAGE RING BASED SYNCHROTRON SOURCE





#### SCHEMATIC OF A SYNCHROTRON RADIATION (SR) LIGHT SOURCE





#### THE X-RAY SOURCES OF A SYNCHROTRON LIGHT FACILITY





#### X-RAY BEAMS AT 3<sup>RD</sup> GENERATION HARD X-RAY SR SOURCES

- Beam size
  - Unfocused: few mm to few cm (source is weakly divergent)
  - Focused beam: < 100 nm to ~10's μm</li>
- Energy range/tunability
  - 0.1eV < E < 0.5 MeV but mostly 2-100 keV</li>
- Energy bandwidth (ΔE/E):
  - $10^{-2}$  to  $10^{-8}$  at sample, typically  $\Delta E \sim \text{few eV} @ 20 \text{keV}$
- Polarized radiation
  - 100% linear or circular or elliptical
- Pulsed radiation
  - 50 ps pulses every ns
- Power
  - several kW total power, several 100 W/mm<sup>2</sup> power density (white beam)
- High degree of coherence
- Photon Flux
  - Brilliance:  $10^{22}$  ph/sec/mrad<sup>2</sup>/mm<sup>2</sup>/0.1%bw ( $10^{11}$  higher than conventional sources)  $\Rightarrow$  photon flux (@  $\Delta E/E = 10^{-4}$ ):  $10^{9}$ - $10^{14}$  ph/s
  - Extremely variable photon rates on detectors (< 1 ph/s to full beam flux)</li>

ESRF





- Synchrotron radiation
- Principles of X-ray optics
- Mirrors

•

- Diffractive optics
- X-ray micro-/nano-focusing
  - Summary



#### **VISIBLE LIGHT OPTICS**



Page 10 Hard X-ray Optics for Synchrotron Beamlines|R.Barrett|TOGIS 2016 Autrans

#### **X-RAY OPTICS**



The European Synchrotron

ESRF



 $\varphi(z)=2\pi\delta z/\lambda$ 

e.g. Kirz et al., *Quart. Rev. Biophys* 28, [1] (1995): 33–130. doi:10.1017/S0033583500003139.



ESRF

The European Synchrotron

#### **X-RAY OPTICS: MANY APPROACHES** \* The refractive index.... cannot be more than 1.05 at most.... W.C. Röntgen Über eine neue art von Strahlen. ....X-rays cannot be concentrated by lenses..." Phys.-Med. Ges., Würzburg, 137, p. 41, (1895)n=1- $\delta$ -i $\beta$ with $\delta$ , $\beta$ <<< 1 English translation in Nature 53, p. 274 $\delta$ (phase-shift), $\beta$ (absorption), materials **REFLECTION** DIFFRACTION (and energy) dependent optical constants $2dsin\theta = n\lambda$ Reflectivity Reflectivity Very weak refraction E<sub>c</sub> ΔE Quite high absorption E Energy Energy Ε, λ REFRACTION < E<sub>c</sub> d Crystals & multilayers X-ray mirrors X-ray gratings •Refractive lenses • Fresnel zone plates Capillaries **Bragg-Fresnel lens** Waveguides

ESRF



- Synchrotron radiation
- Principles of X-ray optics
- Mirrors

•

- Diffractive optics
- X-ray micro-/nano-focusing
  - Summary



#### **TOTAL EXTERNAL REFLECTION: X-RAY MIRRORS**



$$n_o \cos \theta_i = n_1 \cos \theta_r$$
  
for  $\delta \ll 1$  and  $\beta \ll \delta$ 

$$\theta_c \approx \sqrt{2\delta} \propto \lambda \sqrt{Z}$$

The critical angle for total external reflection.

See also: http://www.coe.berkeley.edu/AST/sxreuv/ 15 Hard X-ray Optics for Synchrotron Beamlines|R.Barrett|TOGIS 2016 Autrans

$$\theta_{c_{[mrad]}} E_{c_{[keV]}} = 19.83 \sqrt{\rho_{[g/cm^3]}}$$

E=10keV	Gold		9 mrad
	•	Nickel	6 mrad
	•	Silicon	3 mrad



## Deflection

beam steering (different experiments, Bremsstrahlung)

## •Power filter

lower incident power on sensitive optical components

## Spectral shaper

energy low-pass filter (harmonic rejection)

mirror+filter = spectral window

## Focusing

wiggler & bending magnet : spherical, cylindrical, and toroidal mirrors

microscopy & microprobe : source demagnification (ellipsoidal mirror, KB .....)

## Collimation

parabolic mirror : matching the monochromator angular acceptance with the beam divergence

J. Susini, Optical Engineering, <u>34(</u>2), (1995



The European Synchrotron

ESRF

#### **REFLECTIVE X-RAY OPTICS**



Topography of surface typically described by:

- Slope error (orientation of local surface compared with ideal surface). Distinguish between meridional/tangential (along mirror) and sagittal slope error. Usual units for X-ray mirrors: µradian or arcsecs (1" ≈ 5 µrad)
- 2) Figure error (height of local surface compared with ideal surface). Usual units for X-ray mirrors: nm.



## Typical Requirements

*micro-roughness* < 3Å rms and *slope error* < 1 µrad rms for blur 10% source size

Ultra-precise shaping, figuring and super-polishing

- Very accurate and stable mechanical mounting, bending mechanisms, UHV environment Efficient cooling scheme
- Technically limiting parameters

vibration

$$\Delta_g \propto \frac{5g}{32} \frac{L^3}{t^2} \frac{\rho}{E}$$

thermal deformation

thermal bending

$$\Delta_t \propto \frac{\alpha}{\kappa} P_s$$

$$F_b \propto E \frac{\alpha}{\kappa} P_t w t^3$$



#### **X-RAY MIRRORS FOR SYNCHROTRON RADIATION**

**Grazing incidence => rectangular optical aperture** 

Long mirrors (up to 1.5m): gravity sag

Typically Si with surface coatings (Rh, Pt, ...) to tailor X-ray reflectivity, E<sub>c</sub>

Intense X-ray beams: thermal deformation, cooling

Use of curved surfaces: focusing, collimation: flat, sagittal/meridional cylinders, Focus toroïdal, spheres, ellipsoids, composite (e.g. Wolter) ...



Page 19 Hard X-ray Optics for Synchrotron Beamlines|R.Barrett|TOGIS 2016 Autrans

ESRF

#### TOROIDAL MIRROR (ID09 ESRF)



Material-coating: Silicon-Pt Supplier: SESO (France) <u>Roughness</u>  $\leq$  2Å rms Radii of curvature:

- Sagittal: 71.60 mm
- Meridional: 25 km Slope error (RMS)
  - 0.7 µrad over 450 mm
  - 1.0 µrad over 900 mm





- Synchrotron radiation
- Principles of X-ray optics
- Mirrors

•

- Diffractive optics
- X-ray micro-/nano-focusing
  - Summary



#### X-RAY REFLECTION GRATINGS: SOFT X-RAYS

#### surface reflection and diffraction to disperse X-rays according to energy:





$$d(\sin\alpha + \sin\beta) = m\lambda$$

- surface grating structures mechanical ruling or masked etching
- monochromators or energy analysers
- Grating line densities (lines/mm) either constant or variable (VLS variable line spacing) latter have a focusing effect
- Substrates flat, spherical, (toroïdal)
- Groove profile important for efficiency



Duty ratio : a/d

Courtesy:Shimadzu



Blazed: saw-tooth grooves

 Laminar: rectangular grooves
 Cou

 Page 22
 Hard X-ray Optics for Synchrotron Beamlines|R.Barrett|TOGIS 2016 Autrans

#### **X-RAY DIFFRACTION: HARD X-RAYS**

X-ray diffraction results from elastic scattering of X-rays from structures with longrange order. For X-ray optics generally concerned with highly perfect single crystals (typically Silicon) cf neutron mosaic crystals



- Incident X-rays are "reflected" at atomic planes in the crystal lattice (bulk effect)
- Path difference of the rays  $2d_{hkl} \sin \theta_{B}$
- Constructive interference if the path difference amounts to  $\lambda$  (n  $\lambda$ ?)
- Use of curved, elastically deformed crystals allows focusing

#### **CRYSTAL MONOCHROMATORS**

Energy, E, determined by incidence angle,  $\theta_B$ , of X-ray beam onto crystal planes according to Bragg equation: 1<sup>st</sup> crystal support

$$E = \frac{hc}{\lambda} = \frac{hc}{2d_{hkl}\sin\theta_B}$$

*c* = light velocity *h* = Plancks constant

Energy resolution depends upon type of crystal and reflecting planes used (described by angular Darwin width  $\omega_s$ ) & divergence of incident beam,

$$\frac{\Delta E}{E} = \frac{\Delta \lambda}{\lambda} = \sqrt{\omega_s^2 + \psi_0^2} \cot \theta_B$$

e.g. Si 111 reflexion,  $d_{hkl}$ = 3.1355Å  $\omega_s$  = 10.7 µrad (@ 8keV):  $\theta_B$  = 14° with a parallel incident beam:

Monochromators typically need to be able to position the crystal planes with <µrad resolution over ~90° particularly stringent demands on stability over many hours:

2<sup>nd</sup> crystal support



#### **X-RAY MULTILAYERS**





Crystal (3-dimensional)

Multilayer (Synthetic crystal) (1-dimensional)

X- region : inside the multilayer, as a result of refraction, modified Bragg's law needed

$$n\lambda = 2(d_1 + d_2)\sqrt{1 - \frac{\overline{\delta}}{\sin^2 \theta_b}} \sin \theta_b \begin{cases} \gamma = \frac{d_1}{d_1 + d_2} \\ \overline{\delta} = \gamma \delta_1 + (1 - \gamma) \delta_2 \\ \overline{\delta} = \gamma \beta_1 + (1 - \gamma) \beta_2 \\ \overline{\beta} = \gamma \beta_1 + (1 - \gamma) \beta_2 \\ P(\theta_b) = 1(s) \quad or \quad \cos(2\theta_b)(p) \end{cases}$$



#### X-RAY MULTILAYERS

## high reflectivity x-ray mirrors... or 'synthetic crystals'



single boundary

For  $\theta_{i} > \theta_{c}$ 

 $E_r = rE_0$  where  $E_r$ ,  $E_0$  are reflected and incident wave amplitudes, r is the amplitude reflectivity

 $r < 10^{-2}$  and  $R = |r|^2 < 10^{-4}$ 

$$R \propto \frac{1}{\sin^4 \theta_i}$$



multiple boundaries

ideally  $|r| \cdot N_{layer} \Rightarrow R \rightarrow 1$ 

 $n_1=1-\delta_1+i\beta_1$  and  $n_2=1-\delta_2+i\beta_2$ 

$$R \propto \frac{\Delta \delta^2 + \Delta \beta^2}{4} \frac{N_{layer}^2}{\sin^4 \theta_b}$$



**X-RAY MULTILAYERS : MAIN FEATURES** 

1 - <u>Materials</u> : *highest contrast* 

$$R \propto \frac{\Delta \delta^2 + \Delta \beta^2}{4} \frac{1}{\sin^4 \theta_b}$$

- 2 <u>Gamma parameter</u> : *order suppression* 
  - $\gamma = \frac{d_1}{(d_1 + d_2)}$

$$\gamma = \frac{1}{n}$$
  $\Longrightarrow$  order n



3 - Number of layers : energy resolution

$$FWHM = \frac{0.888 \,\lambda}{N_{layer} \, d\cos\theta_b} \quad \Longrightarrow \quad \frac{\Delta E}{E} = \frac{1.776}{N_{layer}}$$

N<sub>layer</sub> limited by :
$$N_{\text{max}} = \frac{\sin^2 \theta_b}{2 \pi \beta} = \frac{2 \sin^2 \theta_b}{\lambda \mu}$$

Typically N<sub>layer/max</sub>~ 100

$$\Rightarrow \quad \frac{\Delta E}{E} \sim 10^{-2}$$



#### **X-RAY MULTILAYER CHARACTERIZATION**



Hard X-ray Optics for Synchrotron Beamlines|R.Barrett|TOGIS 2016 Autrans 28

The European Synchrotron

## Power filter

- >  $\theta_{b}$  multilayer <<  $\theta_{b}$  crystal  $\rightarrow$  crystal length << multilayer length
- $\rightarrow$  lower power density
- Wide band-pass monochromator analyser
  - > 5 10<sup>-3</sup> < ∆E/E < 5 10<sup>-1</sup>
- Harmonic rejection
  - $\succ \gamma = 1/n$
- Monochromator for soft(er) X-rays
  - d-spacing >2nm cf 3-4Å for radiation hard crystals
- Focussing

- >  $\theta_{b}$  multilayer >>  $\theta_{c}$  mirror  $\rightarrow$  multilayer length << mirror length
- $\rightarrow$  lower spherical aberrations (~L<sup>2</sup>), increased numerical aperture
- Super-mirror (depth graded ML) : extending total reflection
  - R > 40% at 60keV : not possible with a mirror





- Synchrotron radiation
- Principles of X-ray optics
- Mirrors

- Diffractive optics
  - X-ray micro-/nano-focusing
  - Summary



#### **COMPOUND REFRACTIVE LENS**



Gaussian lens equation  $\frac{1}{f} = \frac{2(n_i - 1)}{R}$ 

Thin lens equation : 
$$\frac{1}{f} = \frac{1}{p} + \frac{1}{q}$$



#### PARABOLOIDAL & PARABOLIC CYLINDER X-RAY LENSES

- Parabol-ic/oidal profile  $\Rightarrow$  no spherical aberration
- Be ~2-40 keV  $\Rightarrow$  absorption  $\downarrow$
- AI ~40-80 keV
- Ni ~80-150 keV

Typical parameters : R = 50 to 1500 µm  $2R_0 = 0.45$  to 2.5 mm d < 30µm



ESRF

#### PARABOLIC REFRACTIVE LENSES



C. Schroer et al, Applied Physics Letters, <u>82(9)</u>, 2003 RWTH, Aachen, Germany

The European Synchrotron

ESRF

#### 2D FOCUSING WITH PLANAR REFRACTIVE LENSES





47 x 55 nm<sup>2</sup> beam focus @21keV Schroer et al.,Appl. Phys. Lett. 87, 124103 (2005) Aperture limited by absorption





#### **FRESNEL ZONE PLATES**



#### **FRESNEL ZONE PLATES**

## **Diffractive X-ray Lenses: Circular transmissive diffraction** gratings with radially decreasing line width giving focusing effect





Alternate 'zones' modify phase/amplitude of incident wavefront: for material of thickness, t, wavelength,  $\lambda$ , refractive index 1- $\delta$ -i $\beta$ , phase shift,  $\Delta \phi$ , is:

 $\Delta \phi = \frac{2\pi \delta t}{\lambda}$ 



#### FOCUSING BEHAVIOUR OF FRESNEL ZONE PLATE LENSES

## Rejection of unwanted diffraction orders requires central stop & OSA



The European Synchrotron

#### FOCUSING WITH ELLIPTICAL MIRROR SURFACES



38 Hard X-ray Optics for Synchrotron Beamlines R.Barrett TOGIS 2016 Autrans

The European Synchrotron ESRF

#### **DYNAMICALLY BENT MIRRORS**

## 2 major classes:

- Piezoelectric bimorph systems
- Mechanically actuated systems

Extension of these technologies – increase number of actuators to correct local figure errors – active optics (several projects)



Principle of Bimorph Mirror (from FMB-Oxford)

ESRF mirror bender based on monolithic flexure hinge technology



#### ESRF NANOFOCUSING KB SYSTEM: ID16B

### Smallest ESRF dynamic bending system

- HF Mirror focal distance ~83 mm
- Close to the current limits of bending technology:
  - Local bending radius down to 10 m Mirror stress up to 30 MPa

Optimised figure at 17keV: 8 mrad					
	slope error	figure error			
	[µrad rms]	[nm rms]			
HF (40 mm)	0.15 (0.11)	0.38 (0.16)			
VF (70 mm)	0.09 (0.06)	0.32 (0.16)			



## **ID16B END STATION**





#### FOCUSING OPTICS PERFORMANCE ON ID16B



sampy

The European Synchrotron

ESRF

#### **ID16A "NI" OPTICAL SCHEME**



#### Nano-imaging applications:

- 17 and 34 keV
- High throughput
- Pink beam (full undulator peak)
- Target fwhm spot size ~20-25 nm

Horizontal: Secondary source by focusing ML
Three ML stripes for E = 11.2 / 17.0 / 33.6 keV

- •2 sets of static figured KBs with graded ML coatings
- •KB1: 17.0 keV, KB2: 33.6 keV



#### STATICALLY FIGURED NANOFOCUSING KB MIRROR SYSTEM

Radius of curvature and figure specifications too severe for bending ⇒ Fixed curvature KB mirrors (JTEC, Japan) ML coating at ESRF and integrated into in-house designed mechanics 2 mirror pairs KB1:17 keV, KB2: 33.6 keV **Extreme asphericity:** VFM: 22 to 7 m over 70 mm HFM: 10 to 2.7 m over 36 mm (for KB1)

Target figure errors < 1 nm p.v. Surface roughness < 1 Å rms





#### **ID16A-NI: KB OPTICS 17KEV**

## Focus size: 75 nm (July 2014) $\rightarrow$ 50 nm (August) $\rightarrow$ 30 x 40 nm (September) $\rightarrow$ 23 x 37 nm (October)

Acceptance:550µm x 300µm





Hard X-ray Optics for Synchrotron Beamlines R.Barrett A. Pacureanu, Y. Yang, F. Fus, P. Cloetens

ID16A / UPBL04: KB OPTICS 34 KEV



#### **DYNAMIC VS STATIC KB SYSTEM**

# **XRF: Dynamic bending**







50 nm/step 100ms exposure



Study of As metal uptake within bacteria populations.

S.Kirchen, IFG, Karlsruhe Institute of Technology

The European Synchrotron



Hard X-ray Optics for Synchrotron Bramines R Barrett TOGIS 2076 Autrans, F. Fus, S. Bohic, P. Cloetens

Ш

ശ

#### **REQUIRED QUALITY OF X-RAY OPTICS (DIFFRACTION LIMITED FOCUSING)**

**Strehl ratio:** > 80% (i.e. <20% of intensity outside spot)

**Maréchal Criterion**: rms wavefront error  $\lambda/13$  (but  $\lambda \sim 1$  Å!)

**Reflective Optics:** Any deviation *h* from the ideal surface introduces a phase distortion  $\varphi$ . At grazing angle  $\theta$ ,  $\varphi = (4\pi/\lambda) \cdot h \cdot \sin \theta$ 

X-ray energy (keV)	Coating material	Incidence angle θ (mrad)	Figure specification $\sigma$ (nm, rms)
8	Rhodium	6.0	1.0
20	Platinum	3.0	0.8
50	Multilayer (W/B <sub>4</sub> C)	5.9	0.15

e.g. O. Hignette et al., Proc. SPIE 4501:43–53. San Diego 2001

# **Refractive Optics:** Cumulated thickness errors, t, of lenses introduce phase distortion $\varphi$ . For material with n=1- $\delta$ -i $\beta$ , $\varphi = 2\pi\delta t/\lambda$

X-ray energy	Lens	delta	Figure specification $\sigma$	Figure specification $\sigma$
(keV)	material		(nm, rms) (full stack)	(nm, rms) (per lens *)
8	Be	5.3E-06	2200	980
20	Be	8.5E-07	5600	1000
50	Al	2.2E-07	8700	810

\* Assumes focal length of 1m with lenses R=50µm

Fresnel Zone Plates: Zone placement accuracy ~ 1/3 zone width (3-4 nm!)

e.g. A.G. Michette, Optical Systems for Soft X Rays. Plenum Press, 1986



#### **DEFECTS ON COHERENTLY ILLUMINATED MIRRORS**



H. Mimura et al, Rev. Sci Inst 76, [4] (2005) 045102-6 doi:10.1063/1.1868472.



Current state of the art of X-ray mirror quality is for slope errors ~0.1urad rms, figure errors ~1nm p.v. - both still limit optimal source exploitation



#### **CURRENT BEST FOCUSING PERFORMANCE**

## **Deterministic Polishing**



#### ntensity (a.u.) 4 um **Coherent illumination** -40 -30 20 0 10 20 40 50 -50 -10 30 Position (nm) SPring-8 1km BL Hard X-rav Deformable mirror Focal point F 2ε Focusing mirror Deformable mirror Hard X-ray Focal point **Graded multilayer** deposition Active wavefront Focusing mirror correction

Advanced Metrology

20keV: 7 nm measured focal spot

7 nm

Techniques

H. Mimura et al., "Breaking the 10 nm barrier in hard-X-ray focusing," Nat Phys 6, (2010): 122-125

Hard X-ray Optics for Synchrotron Beamlines|R.Barrett|TOGIS 2016 Autrans Page 50

## Wave-optical Modeling



The European Synchrotron



## Moore's law adapted to the X-ray world:

ESRF Red Book (1987): very few beamline projects aiming even for 10 micron sized beams

Now optics exist for 10nm beams

Routine application of submicron beams still complicated

Also many engineering issues in implementing stable, reliable X-ray nanofocusing systems



Historical evolution of the measured spot size for different hard x-ray focusing elements *(courtesy C. Morawe)* 

- H. Mimura et al. Nature Physics, 6, 122-125 (2010).
- J. Vila-Comamala et al., Ultramicroscopy, **109**, 1360–1364 (2009)
- H. Kang et al., Physical Review Letters, 96:127401 (2006)
- C. Schroer et al., Physical Review Letters, 94:054802 (2005)



#### COMPARISON OF DIFFERENT MICRO/NANOFOCUSING OPTICS





•

The advent of 3<sup>rd</sup> generation synchrotron X-ray sources has encouraged the development of new hard X-ray optics

 Modern beamline design can draw on a toolbox of optical components based on diffraction, reflection and refraction of X-rays

- Interpretended and a second second
  - Iow roughness, high-accuracy figuring, perfect crystals (Ge, Si), diamond, ...
- improved power management strategies
- focusing optics (spot size ~ 50- 0.01µm)
  - zone-plate and refractive lenses, elliptically figured mirrors
- wide range of experimental requirements no one ideal optic

R&D programs continuously in progress: current hot-topics
 preservation of the wave-front quality – especially important for anticipated use of fully coherent XFEL sources

routine sub-10nm focusing

