

Beamline design Optics and Instrumentation

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Outline

- Beamline Definition
- Beamline Components
	- Vacuum system
	- Safety systems
	- Optics
		- Mirrors
		- Focusing Optics
		- Monochromators

Beamlines

Beamlines by technique

Photoelectron

emission

Imaging

 7.1

 7.2

 $8.1L$

10.2R

11.1R

11.2C

11.2R

Lithography

MCX

ALOISA

BaDEIPh

XAFS

XRD2

Xpress

Diffraction

Absorption

BEAR

❑ X-ray diffraction - MCX ❑ X-ray absorption - XAFS ❑ X-ray Fluorescence - XRF

A beamline is the "equipment" required to **transport** SR from the source (BM/ID) to the sample and to **condition** the radiation for the experiment.

Figure 1: A Schematic of 28 operational beamlines of Elettra Sincrotrone Trieste

Scattering

Reflection

Emission

Beamline Components

What does SR Transport ?

- Steer beam from the source to the experimental end station
- Steer beam in an efficient manner preserving flux
- Steer beam in a safe manner both for equipment and personnel

Figure 2: Layout of the XRF beamline of Elettra

Proper beam transport and beam conditioning are guaranteed by the proper choice of beamline components.

Figure 3: A schematic optical layout of the BL-16 beamline showing different components of the XRF beamline of INDUS-2.

What does condition mean?

•Control energy (E) and bandwidth (ΔE) of the beam

- Monochromatic beam ($\Delta E = 1-2$ eV @ 10KeV; $\Delta E / E = 10^{-4}$)
- Polychromatic beam ($\Delta E = 1-2$ KeV @ 10KeV; $\Delta E / E = 10^{-1}$)
- **■** High resolution beams (ΔE a few meV @ 10KeV; $\Delta E / E = 10^{-7}$)
- Control size/divergence of the beam
	- Micro or nano beams
	- Highly collimated beams
- •Control polarization of the beam
	- Linear
	- **Circular**

Beamline control System

Figure 4: Layout of the beamline control software of XRF, Elettra

Vacuum system

Ring pipe is maintained in ULTRA HIGH VACUUM (μ**Pa/10- ⁹ mbar)**

LOW VACUUM

Roughing pumps - $1x10^{-3}$ mbar Mechanically noisy, contain lubricants

HIGH VACUMM (HV)

Turbo pumps - 1x10-8 mbar Mechanically quiet, contain lubricants

High and Ultra High vacuum No moving parts, no lubricants CONFLAT flange with cupper gasket Low vacuum

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ULTRA HIGH VACUUM (UHV)

Ion pumps - lx10-11 mbar

Safety systems

Radiation protection at Synchrotron Facilities

A radiation is defined as ionizing if it carries enough energy for ionizing atoms and/or molecules

DIRECT IONIZATION

Massive charged particles traveling at relativistic speed: α -particles (He nucleus at relativistic speed), β-particles (electrons or positrons at relativistic speed)

Electrons

INDIRECT IONIZATION

- High energy photons that exert ionization through photoelectron, Compton effect or pair production (γ-rays, X-rays and High Energy UV)
- Uncharged fast neutrons can dislodge a proton upon collision the "recoil proton" may induce secondary ionization effects

http://hyperphysics.phy-astr.gsu.edu/hbase/nuclear/

alpha emitters

radium, radon, uranium, thorium

beta emitters

strontium-90, carbon-14, tritium, and sulfur-35

gamma emitters

iodine-131, cesium-137, cobalt-60,

radium-226, and technetium-99

Penetration of the Different Radiations

Radiation sources at Synchrotron Facility

- ❖ **X-ray** and **UV** synchrotron radiation produced by Bending Magnets and Insertion Devices
- ❖ **Bremsstrahlung** produced by electromagnetic cascade or shower due to e-beam loss

Prompt radiation sources at electron accelerators are generated by e-beam loss

❖ All the electron injected into the ring pipe are lost **naturally**:

during injection ➔ due to the electron collision with injector components

in the storage ring \rightarrow due to scattering with residual gas particles

Abnormal beam losses are most likely due to beam miss-steering (mismatch between the beam trajectory and the ring lattice for inaccurate orbit adjustment) and interlock-safety system failure

- •An electron traveling within a material can lose energy primarily by two phenomena:
	- COLLISION LOSS (through excitation or ionization of atoms)

• RADIATION LOSS: BREMSSTRAHLUNG

Radiation protection at high energy electron accelerators:

- \checkmark The critical energy Ec for one material defines the boundary where electron collision losses equal radiation losses
- ✓ Beam energies at high-energy electron accelerators are well above Ec for common materials used for the electron ring manufacture (stainless steel)

Vylet V, Liu JC., Radiat Prot Dosimetry 2001;96(4):333-43

High-energy electrons hitting a target material will loose energy almost exclusively by generating bremsstrahlung photons

It should be mentioned that, even when the electron beam is perfectly steered, it still interacts with atoms of the residual gas in the evacuated beam pipe, generating the so called **gas bremsstrahlung**. The yield of gas bremsstrahlung is more forwardpeaked than bremsstrahlung in solid targets. The yield is then proportional to the length of the straight section (more problematic for long-insertion devices) and to the vacuum level

In general, the average power losses at most places around the booster-ring pipe is low (meV), and thin concrete walls and roofs are sufficient to guarantee radiation protection.

At specific locations, where the power losses <u>can be higher,</u> thicker concrete walls and/or additional local shielding **are present**

• During electron injection into the storage ring, thick heavy metal injection **stoppers** are inserted into the beamline (**frontend**) to block bremsstrahlung radiation in case of beam accidentally directed into the beamline Injection stoppers are usually located inside the ring wall. Stoppers are cooled in order to efficiently dissipate the SR power.

- Photon stoppers and gamma stoppers (**shutters**) are positioned also in the frontend of each beamline
- The beamline front also contains a **fast valve** (time of response, ms) and an acoustic delay line for slowing the pressure wave coming accidentally from the beamline

- ➢ Shutter screen SR radiation :
- Glidcop (copper that is dispersed-strengthened with ultra-fine particles of alumium oxide)
- ➢ Stopper screens bremsstrahlung radiation: Havier metals (alloy of tungsten)

Ionizing radiations, as a result of the electron beam loss, are present only when the beam is injected or stored

SR is substantially less penetrating than gas bremsstrahlung, but it its higher energy components can be a source of indirect ionizing particles. Depending on the beamline type (energy employed, monochromaticity, sample positioning) the beamline design will be done accordingly for protect workers and people.

If all the aforementioned safety rules are properly accomplished, the dose rate in the experimental area of a Synchrotron Facility will be acceptable for all credible beam loss scenarios

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Radiation monitoring at SR facilities is done by using gamma-monitors placed at specificsensitive locations in the beamlines and by using personal gamma-monitors when required

should not exceed some tens of mSv per year (total body)

Beamline Optics X-ray Optics

Beamline Optics acts in order to transform the SR photon beam characteristics for obtaining the best match to experimental requirements

They act:

- Shaping the beam upon BM or ID emission
- Selecting the photon energy/bandwidth
- Modulating beam divergence
- Selecting polarization

oSlits, pinholes oFilters, Windows oMirrors oBeam splitters oMonochromators oLenses oPhase plates

X-ray optics

Schematic diagram of the frequency dependence of refractive index and absorption of hypothetical solid near IR, UV, and x-ray resonances, and the general tendency towards unity for very short wavelengths where the frequencies are higher than all atomic resonances. Only the real part of the refractive index is shown here.

Reflection and refraction at an interface

Interface geometry for incident, reflected, and refracted waves

Gold $(Z=79)$:

 600 eV $\rightarrow \theta c \approx 7.4^{\circ}$

 $1200 \text{ eV} \rightarrow \theta c \approx 3.7^{\circ}$

 5 keV $\rightarrow \theta c \approx 0.9^{\circ}$

Total external reflection

If radiation impinges at a grazing angle θ < θ c, it is totally external reflected.

Nickel $(Z=28)$: 6 keV $\rightarrow \theta c \approx 10$ mrad (0.57°)

Carbon $(Z=6)$: $100 \text{ eV} \rightarrow \theta c \approx 250 \text{ mrad} (14^{\circ})$

It is the counterpart of total internal reflection of visible light. Visible light is totally reflected at the glass/air boundary if $\theta < \theta_c = 48.2^{\circ}$

> n^* cos θ c=1 $\rightarrow \theta$ c = arccos (1/n) = 48.2° $n = 1.5$ refraction index of glass

Reflectivity of single layer

Reflectivity from a thin surface film on top of an arbitrary substrate, where multiple reflections can take place in the top layer.

X-ray reflectivity spectra of Cr film of thickness 500 Å and 100Å on silicon substrate (λ=1.54 Å). Figure shows the dependence of the oscillation frequency on the film thickness.

Reflectivity of multilayer structure

Reflectivity of multilayer structure

Reflectivity (reflectance) is the fraction of the incident electromagnetic power that it is reflected at an interface I^R/I

 I^R /I = $|E^R/E|^2$ from Fresnel equations

• From Fresenl equations it is possible to show that below 0 c there is unit reflectivity when the absorption coefficient, p, equals 0

The only optical elements which can work in the VUV, EUV and soft x-rays regions are mirrors and diffraction gratings, used in total external reflection at grazing incidence angles

Exceptions: multilayer coated mirrors, zone plates

L. G. Parratt

Surface Studies of Solids by Total Reflection of X-Rays Phys. Rev. 95, 359 -1954

1- Beam deflection

The first action of a FLAT mirror is to steer photon beam

- **2- Energy cut-off for a Fixed Angle of incidence**
- All the above considerations have been developed assuming a monochromatic incident beam and a variable angle of incidence
- Often mirrors are used as first elements of a beamline, that is that they are stroked by a polychromatic beam at a fixed angle
- Reconsidering the relation of critical angle, we can define the cut-off energy *E^c* of a mirror for a fixed angle θ

$$
\theta_c = (2\delta)^{\frac{1}{2}} = \lambda (n_e r_e / \pi)^{\frac{1}{2}} \qquad E_c = \frac{hc}{\lambda_c} = \frac{(hc}{\theta_c}) (n_e r_e / \pi)^{\frac{1}{2}}
$$

• *E^c* is the maximum energy (or minimum wavelength) that will be totally reflected by the mirror; energies above E_c will be reduced because of the fall off of reflectivity

2- Energy cut-off for a Fixed Angle of incidence

- Since there is an energy cut-off above which radiation is reflected with low-efficiency, mirrors can be used to effectivelly suppress high energy component of the synchrotron emission. Therefore, they act also as low-pass filters
- Depending on the critical energy of the source, mirrors can effectivelly remove a considerable amount of heat and reduce the thermal loading on downstream optics
- Since in many cases the angle of incidence of the SR beam on the mirror is fixed due to the various beamline compenents downstream, the energy cut-off can be varied changing the coating deposited on the mirror substrate

Reflectivity curves as a function of the X-ray energy for a fixed incidence angle (3 milliradians) for Gold, Cupper and Silicon

- 2- Energy cut-off for a Fixed Angle of incidence
- This means that higher is the elctron density, higher is the reflectivity of the

- In the c ases when significant power is carried by X-rays with energies above the intended range of operation, it is usual to absorb such Xrays in the first mirror at grazing incidence.
- The fraction of synchrotron emission power absorbed by the mirrors can be very huge (power densities on mirror up to hundreds of W/cm²), so efficient cooling systems are needed for preserving both material properties and mirror shape

Rh: 12.4g/cm³ Pt: 21.4 g/cm³

Thermal deformation of mirrors

Total power emitted from sources is in the order of 2-4 KW (but it can reach up to 10-14 KW with new superconducting or in-vacuum undulator). The power density on the first beamline mirror (optics) illuminated on-axis can be higher than $400W/mm²$

Ox: Local variation in thickness (thermal bump)

$$
\Delta_{\text{bump}} = \frac{\partial z}{\partial x} \propto \frac{\alpha}{\kappa} GP_s
$$
 Usually a mirror is over illuminated for minimizing Φx

Oz: Differential expansion hot-cold slides (thermal bending)

$$
\Delta_{bending} = \frac{\partial x}{\partial z} \propto \frac{\alpha}{\kappa} GP_t
$$

 α : thermal expansion κ : thermal conductivity G: cooling geometry facor $(-0.02 - 1)$ Ps: Power density Pt : Total power

Thermal load simulation on the first collecting mirrir of IR beamline SISSI

Fig. 2. Photograph of the intact gold film, carbon coated gold film, and the area on the sample where the carbon film is removed and underneath gold film is visible.

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Cleaning of carbon layer from the gold films using a pulsed Nd:YAG laser

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3- Long Mirrors to capture full beam

• As a consequence of beam divergency and of the small critical angles in the X-ray, mirrors have to be manufactured very long in order to capture the full SR beam

Carbon-like deposits on mirror surface

Typical length for X-ray mirrors is 1 m, with radius of curvature of the order of kilometers

2- Other energy filtration beamline components

• Windows and filters

Low absorbing windows are placed along a beamline to separate different vacuum sections. In hard X-ray regime they are typically made of beryllium, polymeic materials or pyrolitic graphite, while diamond is often chosen for visible-IR SR emission.

Very often a window is installed just after the front-end, in order to separate the baemline vacuum for the ring vacuum. The ultimate function of these windows is to save ring vacuum in case of vacuum break at the beamline.

This windows act also absorbing most of the low-energy part of SR emission, therefore reducing the overall power delivered to downstream optics.

Beryllium windows

Both windows types are mounted on flanges

CVD diamond window

Focusing properties of mirrors

X-rays mirrors can have different geometrical shapes, their optical surface can be a plane, a sphere, a paraboloid, an ellipsoid and a toroid. KEK-PF, BL-18B

The meridional or tangential plane contains the central incident ray and the normal to the surface. The sagittal plane is

the plane perpendicular to the tangential plane and containing the normal to the surface.

Focusing properties of x-ray lenses

 $1.00/m$ 8863

compound refractive lenses (CRLs)

Be lenses Refractive x-ray lenses on a holder

Fresnel zone plates. (a) The path difference between adjacent transparent rings, $P_{n+2} - P_n$, in a zone plate should be equal to the wavelength of the x-rays being focused. (b) An electron microscopy image of a zone plate manufactured using electron-beam microlithography.

nanofocusing lens array fabricated by lithographic and ion-etching techniques

Dispersive optical elements

Monochromators

$$
2dS\,in\theta = n\lambda \quad d_{hkl} = \frac{1}{\sqrt{\left(\frac{h}{a}\right)^2 + \left(\frac{k}{b}\right)^2 + \left(\frac{l}{c}\right)^2}}
$$

Diamond Structure

h,k,ℓ all odd h+k+ℓ 4 n $(n$ is an integer) Si(111), d=3.13 Å, Emin ~ 2 KeV Si(220), d=1.92 Å, Emin ~ 3.2 KeV Si(311), d=1.64 Å, Emin ~ 3.8 KeV InSb(111), d=3.74 Å, Emin ~ 1.7 KeV Ge(111), d=3.27 Å, Emin ~ 1.9 KeV W/Si Multilayer Mo/Si Multilayer W/B4C Multilayer $Ru/B₄C$ Multilayer Rh/C Multilayer Ni/C Multilayer

Structural factor F(hkl) : it defines how atomic arrangement influence the intensity of the scattered beam as well as which reflection-peaks to be expected in a diffraction pattern

Diffraction gratings:

- ❑ Ruled gratings
- ❑ Holographic gratings
- ❑ Transmission gratings
- ❑ Reflection gratings

Grating profile:

❑ Lamellar grating ❑ Sinusoidal grating ❑ Blaze grating

Grating surface profile: ❑ Spherical grating ❑ Elliptical grating ❑ Toroidal grating

Grating Monochromators

The diffraction grating is an artificial periodic structure with a well defined period d. The diffraction conditions are given by the well-known grating equation:

 $\frac{d_{\text{sin}\alpha}}{d_{\text{cos}\alpha}}$

$$
\sin \alpha + \sin \beta = Nk\lambda
$$

 α and β are of opposite sign if on opposite sides of the surface normal $N=1/d$ is the groove density, k is the order of diffraction $(\pm 1, \pm 2,...)$

Bendable diffraction grating can be used for both monochromatize and focus a beam of light. The arrangement of a monochromator followed by a focusing mirror is however the most common.

Hard X-rays dispersive optical elements

- \triangleright The most used optical components for X-rays (especially hard X-rays) are crystal satisfying the Bragg's law
- \triangleright Radiation of wavelength λ is reflected by the lattice planes. The outgoing waves interfere. The interference is constructive when the optical pat difference is a multiple of λ
- \triangleright The role of a monochromator is to deliver an X-ray beam with a high monochromaticity, with an energy resolution $\Delta E/E \sim 10^{-4}$ for the most common applications.
- ➢ In hard X-rays monochromators are usually constructed using perfect crystals (typically silicon, germanium or diamond)

$$
\frac{\Delta E}{E} = \frac{\Delta \lambda}{\lambda} = \cot(\theta_B) \Delta(\theta)
$$

 \triangleright The energy resolution of a crystal monochromator is determined by the angular spread $\Delta\theta$ of the diffracted beam **Beam Divergence** and by the Bragg angle θ_R

 \triangleright $\Delta\theta$ contribute:

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angular divergence of the incident beam $(\Delta \Phi)$ intrinsic width of Bragg reflection or Darwin width

A collimating mirror in front of the crystal reduces the ΔΦ beam of incident beam, improving energy resolution

Darwin width

- ❖ There is an angular width over which the diffraction occurs (the incidence angle does not need to exactly meet the Bragg angle due to the small lattice vibrations). This is usually called the Darwin width (better known as rocking curve width), ω_{D} .
- ❖ The Darwin width is the full width at half-maximum (FWHM) of the total reflective profile of the monochromator crystal and is given by

$$
\varpi_D = 2.12 r_e \left(\frac{\lambda}{n+1}\right)^2 \frac{NF(hkl)}{\pi 2 \sin(\theta_B)}
$$

For single Bragg reflection, where r_e is the electron radius, λ is the incident photon wavelength, N is the atomic density, F(hkl) is the structural, θ_B is the Bragg angle and n is the order of harmonic present in the beam.

> **The energy resolution is determined by the monochromator**

Silicon and germanium are readily available and are grown in large "boules" that relatively inexpensive. They are often used as single crystal monochromators. The value of ω_D for Si(111) at 8KeV (1.5Å) is about 40 microradians. For an ondulator, the typical opening angle is about 10-15 microradians

or

used in Sec. 3-7.) The condition for zero diffracted intensity is therefore

 $2Na\Delta\theta$ sin $\theta_R = (N + 1)\lambda$. $\Delta\theta = \frac{(N+1)\lambda}{2Na\sin\theta_B}$

This equation gives the maximum angular range of crystal rotation over which appreciable energy will be diffracted in the direction $2\theta_B$. Since I_{max} depends on this range, we can conclude that I_{max} is proportional to $1/\sin \theta_B$. Other things being equal, I_{max} is therefore large at low scattering angles and small in the back-reflection region.

Double crystal monochromator (DCM)

- ❖ The double crystal monochromator is made by two parallel crystals (usually with same *hkl* exposed face) and produces a monochromatic beam running parallel to the incident X-ray beam
- ❖ The whole system is mechanically designed in order to rotate the pair of crystals to change the incident angle corresponding to a given energy
- ❖ In order to make h fix (that is important for properly illuminating the downstream optics), g should be changed accordingly to the selected energy
- ❖ Maintaining the perfect parallelism is crucial for maximizing the throughput of the system For minimizing the dependence of g by θ , g needs to be very small (\blacktriangleright 1/cos(θ))

Deformation of the crystal due to the thermal load can severely affect the performances of the monochromator

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References

- D.Attwood, "Soft x-rays and extreme ultraviolet radiation", Cambridge University Press, 1999
- B.W.Batterman and D.H.Bilderback, "X-Ray Monocromators and Mirrors" in "Handbook on Synchrotron Radiation", Vol.3, G.S.Brown and D.E.Moncton, Editors, North Holland, 1991, chapter 4
- "Selected Papers on VUV Synchrotron Radiation Instrumentation: Beam Line and Instrument Development", D.L. Ederer Editor, SPIE vol. MS 152, 1998
- W.Gudat and C.Kunz, "Instrumentation for Spectroscopy and Other Applications", in "Syncrotron Radiation", "Topics in Current Physics", Vol.10, C.Kunz, Editor, Springer-Verlag, 1979, chapter 3
- M. Howells, "Gratings and monochromators", Section 4.3 in "X-Ray Data Booklet", Lawrence Berkeley National Laboratory, Berkeley, 2001
- · M.C. Hutley, "Diffraction Gratings", Academic Press, 1982

References

- R.L. Johnson, "Grating Monochromators and Optics for the VUV and Soft-X-Ray Region" in "Handbook on Synchrotron Radiation", Vol.1, E.E.Koch, Editor, North Holland, 1983, chapter 3
- G. Margaritondo, "Introduction to Synchrotron Radiation", Oxford University Press, 1988
- T.Matsushita, H.Hashizume, "X-ray Monochromators", in "Handbook on Synchrotron Radiation", Vol.1b, E.-E. Koch, Editor, North Holland, 1983, chapter 4
- W.B.Peatman, "Gratings, mirrors and slits", Gordon and Breach Science Publishers, 1997
- J.Samson and D.Ederer, "Vacuum Ultraviolet Spectroscopy I and II", Academic Press, San Diego, 1998
- J.B. West and H.A. Padmore, "Optical Engineering" in "Handbook on Synchrotron Radiation", Vol.2, G.V.Marr, Editor, North Holland, 1987, chapter 2
- G.P. Williams, "Monocromator Systems", in "Synchrotron Radiation Research: Advances in Surface and Interface Science", Vol.2, R.Z.Bachrach, Editor, Plenum Press, 1992, chapter 9

Typical components found at an x-ray synchrotron beamline.

