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ESRF: SPECIFICITY AND GRAZING TECHNIQUES

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Résumé
L'ESRF (Laboratoire Européen de Rayonnement Synchrotron) est actuellement en phase de construction. Pour l'utilisateur, il s'agit d'une multisource de rayonnement couvrant avec une très haute brilliance toute la gamme des rayons X (de 0,3 à 300 KeV). Chaque source est modulable suivant les besoins expérimentaux, qui peuvent être une haute résolution spectrale (ex. meV), temporelle (ex. msec), spatiale (ex. µm), ou une polarisation donnée.

Dans le contexte de la conférence, l'ESRF permettra un essor important pour les applications - en spectroscopie et en diffraction - qui exigent une haute définition angulaire; quelques progrès possibles sont décrits et une estimation du gain en flux est fournie dans un cas particulier.

Abstract
The ESRF (European Synchrotron Radiation Facility) is at present under construction phase. For the user, it consists in a multisource of radiation spanning with very high brilliance the whole X-ray range (from 0.3 to 300 KeV). Each source can be tailored for the experimental requirements, that could be a high spectral (e.g. meV), temporal (e.g. msec) or spatial (e.g. µm) resolution, or a given polarization.

In the context of the conference, the ESRF will allow important developments for the applications - in spectroscopy and diffraction - that demand a high angular definition; some possible advances are described, and an estimated gain in flux is obtained in a specific case.
1. Introduction

The European Synchrotron Radiation Facility (ESRF) in Grenoble, already officially gathering eleven countries, is now in the construction phase. As a service-oriented facility, it will deliver, in 1994-95, optimized X-rays on eighteen specialized beamlines. Following a "General User Meeting" held in March this year, the nature of these beamlines will soon be decided. In addition to these so-called "public" lines, other beamlines may also develop in joint responsibility between the ESRF staff and external groups (collaborative research groups, or "CRG's").

The purpose of this paper is to give some information on the facility, and its relevance to the present meeting for "grazing" techniques. The discussion will be limited to the "sources" themselves, and not to the conditioning optics\(^1\) that is downstream, although it is clear that each experiment should be optimized as a whole from the source to the detection system. After a general background on ESRF, and the specificity of the various X-ray sources, possible grazing techniques developments will be addressed. Finally some quantitative data using a simple optics will be presented.

2. Generalities on ESRF

The ESRF is located near the confluent of the Isere and Drac rivers on the so-called "Scientific Polygon", two kms away from the railway and road stations in Grenoble. The local laboratories are mainly sited within the Polygon, including CEN-G, CNRS, national (SNCI) and international Institutes (ILL, EMBL). Therefore many experimental techniques for condensed matter studies are available at a short walking distance from the ESRF.

A general layout of the facility is given on fig.1. The main part consists of an annular building of around one km circumference, in the inner part of which the ring tunnel is installed. Around it, the experimental hall (20,000 m\(^2\)) allows beamlines up to 70 meters to be implemented, with a first optical element around 30 meters from the X-ray sources; finally, various laboratories and offices (mainly user-oriented) are located on the periphery. The other buildings are intended for the personnel and additional laboratories, the technical support and the accelerator system. The present staff - which amounts to around one hundred - uses two temporary buildings, one for general activities and the second for the recently created ESRF laboratories: accelerator labs (magnets measurements, levelling systems, insertion devices), and experimental ones (X-rays, optics, surface science). A general plan of the buildings, with construction times indicated, is given in fig.2.

\(^1\) X-ray Optics for synchrotron radiation, Proceedings of Brookhaven Biology Symposium, May 1988, A.K. Freund
3. Specificity of ESRF

Only the main or newer aspects will be sketched here, since more information can be found in previous reviews\(^2,3\).
The ESRF is a multisource of synchrotron radiation, whose essential technical characteristic relative to present rings is a high brilliance in a large range of X-ray photons. There are two categories of sources: the bending magnets and the insertion devices; the energy and the magnetic structure has been defined in view of the last ones, which are more flexible and performant. General parameters for the ring are gathered on table 1.

Energy range

The XSR ranges traditionally considered for ESRF are the low: 0.3 - 3 KeV (*), medium: 3 - 30 KeV (**), and high: 30 - 300 KeV (***) energy ranges, where the number of stars in brackets corresponds to the source performance. The excellency of ESRF, for most applications, is indeed the medium range of classical X-rays (Angstrom region). A variety of insertion devices can be used to cover these wavelengths: e.g. a possible alternative for tunability, to the variable-gap undulators are - at least in the 6 -12 KeV range - the "tapered" undulators\(^4\), based on a variable period and/or gap along their length that enlarges the energy spectrum seen through a given pinhole (fig.3).

In contrast, no optimized undulator is available in the high energy range, for the nominal case (20 mm minimum magnetic gap). In the long term, smaller gaps could be tested in view of "micro-undulators" that could shift the high energy side of the excellency region.

Finally, the softer part - i.e. below the beryllium window - is considered as best served by lower energy machines around 2 GeV (the cross-over point is around 1-2 KeV). However, for special purposes, ESRF is well suited in that range:

- some experiments using classical X-rays need to be complimented at lower photon energies;
- it has special polarization properties: it may be anticipated that circular polarization, and more generally flexible polarization parameters will be increasingly

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\(^2\) ESRF: an optimized X-ray source, ESRF report EXP/MB /01/88
\(^3\) ESRF à Grenoble : une source de rayons X optimisée, M. Belakhovsky in Bulletin National SFP (janvier 1989)
\(^4\) "RedBook": ESRF Foundation phase report (01/87)

Concerning the use of tapered undulators at ESRF, P. Elleaume, ESRF-SRDD-88-24
used, particularly at low and medium energies (absorption-based experiments, grazing techniques, resonant magnetic diffraction etc.). In that case a 6 GeV ring is optimized at smaller photon energies when insertion devices are considered: although single bump or asymmetric wigglers can give (off-axis) circular polarization at high energies, undulators give the best performance. A versatile "planar/helical" undulator concept has recently been addressed\(^5\); several such systems with various periods could cover the 0.5 - 10 KeV range on a 6 GeV ring (and 50 eV - 1 KeV at 2 GeV), the lower range with high tunability (fig. 4); it consists of two independent jaws of permanent magnets, one giving a vertical magnetic field, and the other an horizontal field. Nearly complete polarization could be obtained on axis, with high brilliance especially in the circular mode. By translating one jaw parallel to the other, one could quickly change between various circular and linear polarizations; a prototype will be built at ESRF in the near future.

- it includes special temporal sequences that would be specific of the ESRF ring.

Since the photoelectric cross-section decreases rapidly with the XSR energy, the ESRF will offer better potential than present rings for in situ studies. Also, the large range of tunability will, for example, allow one:
- to access to all electronic thresholds (the actinide K-shell binding energies are around 100 KeV), as well as low-lying nuclear resonances;
- to play with the different variations with XSR of photoelectric and Compton cross-sections (e.g. contrast in calcium microtomography).

\textit{Emittance and Brilliance}

The emittances of the particles in the ring are related to the 6D-phase space of transverse and longitudinal conjugated variables.

The length of the RF-buckets is at a minimum around one cm, which results in an ultimate time resolution of a few tens of picoseconds for stroboscopic experiments. The high harmonic number allows various temporal sequences, with high filled-to-empty bucket ratios.

Let us now turn to the plane perpendicular to the orbit trajectory. In these transverse directions, a distinction is made below between the "emittance" (occupied volume \(x,x',y,y')\) and the "brilliance" (density in that volume) of the particles (or of the emitted photons). The main parameters for the electrons are given in table 2, assuming a Gaussian distribution. Whereas the minimum horizontal emittance is given once the ring is defined, the ratio of the vertical to horizontal electron emittances could in practice span a range from 1 to 0.01; it is at present fixed to 10 percent (thus 0.7 nm.rad in vertical). A very crucial point, the beam \textit{stability} in position and direction, has been addressed intensively by the accelerator physicists, in order to guarantee a transverse

\(^5\) A flexible planar/helical undulator design for synchrotron sources, P. Elleaume ESRF- SR-ID-88-23
phase space stability at a 10 percent level. The resulting XSR, obtained after convolution with SR, also has small emittances since the divergences of SR (natural or pinhole-collimated in the undulator case) are limited. More precisely, the angular spread of XSR and particles are comparable for the undulator regime, whereas the vertical XSR emission is larger by an order of magnitude for bending magnets and wigglers. Therefore these last two sources have a better purity, i.e. their properties (spectrum, geometry, polarization) are in closer agreement with the theoretical result of a filament with zero cross-section.

A first consequence of the small transverse emittances is an improved optics, as well for the incident XSR (better match to the optical acceptance downstream, smaller aberrations) as for the analysis devices (X, e−). Moreover - and this is reinforced by the chromatic dispersion of undulators - it is possible to use small beam conditioners (e.g. Bragg-Fresnel optical microstructures that can be fabricated only in mm size).

Some demanding applications require the full use of the small emittances, i.e. small dimensions and divergences in both dimensions at the sample (or detector). In the other cases, there is an obvious trade-off (in 1-D or 2-D):
- a lateral resolution improved by XSR refocusing onto a small (e.g. single crystals, high pressure) or inhomogeneous sample. The spot sizes range from dimensions comparable to the source (~ 0.1 mm) down to the submicrometer level. The ultimate limit for the studied sample area depends on several factors: the progress in microfocusing at XSR wavelengths, the detection scheme (parallel with magnifying optics, secondary processes) etc. In a general way, one could state that:
  * breaking-off the micrometer barrier is ESRF-specific. Many XSR techniques should benefit of this "microprobe" or "imaging" capability;
  * "microstructures" are at the same time a means (optical, detection systems) and a goal (the object of analysis) for ESRF.
- a very parallel beam, of interest for various experiments (e.g. total reflection, techniques exploiting stationary waves, ultra small angle scattering, lithography); we shall come back to this point in grazing applications.

Another consequence of the small emittances is the partial transverse coherence of beam; however, the diffraction limit is not reached for ESRF at X-rays wavelengths.

The high phase space density of XSR within the (more or less Gaussian) phase volume - or in other words the high brilliance - especially in the undulator mode, has the additional obvious consequence of giving potential access to:
- ultra high spectral resolution, through electronic (meV range) or nuclear (micro-to-nano - eV range) monochromators; this opens new fields for elementary excitations,
Mössbauer effect, γ - optics ; the extension of the longitudinal coherence relies -
beyond the nanometer value as given in the undulator regime - on the downstream
monochromatization and can reach tens of meters ;
- weaker effects, that necessitate otherwise prohibitive or unacceptable data acquisition
times (e.g. in surface science);
- kinetics of irreversible processes, with smaller time scales (typically the millisecond
range or less).

4. Grazing incidence techniques

One of the trade-offs of a highly brilliant source, as already discussed, is relevant for
grazing incidence beams : the optics can increase the parallelism of the XSR, which is
detrimental to the beam size.
In the context of this conference, where standing waves are also invoked ( since it
compliments on perfect crystals the in - plane information provided by surface
diffraction with perpendicular structural data), the "grazing" concept will be enlarged
below into high collimation of the XSR. Also, due to the reversibility theorem, one
should also include the grazing emergence conditions.

Grazing (or highly collimated) experiments can be classified as follows :
- the energy can be fixed, and the incidence angle varied between below the critical
angle to one order of magnitude higher. In addition, this fixed energy may have to be
chosen more or less precisely in the XSR range (signal - to - noise, anomalous etc.).
One could first perform scattering experiments with perpendicular momentum
transfer (specular beam, and the diffuse scattering around it), or parallel (grazing
detection of XSR diffracted by lattice planes normal to the surface of single crystal
samples); or in intermediate case ( off-surface diffraction on polycrystalline samples
8). One can also perform secondary process detection (with possible energy angle,
polarization or spin analysis), of namely fluorescences or electrons. This includes
techniques such as standing waves, X-ray fluorescence, photoelectron diffraction etc.)
- its energy can be varied, i.e. essentially spectroscopic experiments are performed
such as EXAFS-type measurements (Refl - or Fluo - EXAFS etc.). In that case, an
additional difficulty occurs : since the critical angle varies with energy (as square root of
5), the angle of incidence may have to vary as a function of the scanning energy ; the
measured signal depends thus on two linked variables.
In some cases, scattering experiments could also be performed at variable incident
energies ( anomalous scattering, Bragg diffraction in λ-scan, XSW-type
experiments9 ).

9 A simple X-ray standing-wave technique for structure determination-theory and application, D.P.
New applications could develop at ESRF, some of which may be traced from advanced work; a few directions are given below:

- more systematic use of both grazing incident and emergent angles, with higher accuracy for improved depth analysis; some advances have already been made in diffraction\textsuperscript{10}, but not much in absorption-based experiments;
- use of higher values of the grazing angle with respect to the critical angle;
- small sample areas: mm range in length, or even smaller by relying only on grazing emergence;
- use of diffuse scattering data around the Bragg peaks for partly disordered surfaces and interfaces;
- study of dynamic processes by snapshot experiments, on a time scale of e.g. one second (epitaxial growth using MOCVD\textsuperscript{11}); relaxation after a perturbation such as laser pulse using the ESRF time structure;
- study with variable polarization in surface magnetic scattering, thus at fixed but sometimes well defined (edge resonance) XSR energy; similar work in magnetic XAFS;
- use of softer XSR implying large absorption losses (hence high incident flux) for low Z elements or increased critical angles;
- study of buried interfaces: the favourable case is when the electronic density increases with depth, in such a way that the corresponding external angle is sufficiently separated from the external critical angle\textsuperscript{12}. It may also be appropriate to use harder XSR (e.g. in electrochemistry), since, off resonances, the critical angle is linear with the wavelength whereas the absorption is proportional to its third power.
- nothing has been done up to now with surface SAS nor with inelastic scattering, and ESRF could provide such an opportunity; the same is true for nuclear resonance applications;
- combination of techniques will occur more frequently at ESRF: a first example is the energy dispersive approach in reflection geometry; both Refl - EXAFS and grazing incidence scattering data can be recorded using 2D-detection, as recently done on a multilayer system\textsuperscript{13}. Apart from fast data acquisition, an advantage of this approach is a small illuminated width (demagnification of an already small horizontal source size). A second example is the creation of a stationary wave in perfect crystals (or periodic

\textsuperscript{11} Application of X-ray scattering to the in situ study of organometallic vapor phase epitaxy, Paul Fuoss et al, Bell Labs activity report 1989
\textsuperscript{12} - Standing-wave-assisted EXAFS study of a multilayer, S.M. Heald and J.M. Tranquada, J. Appl. Phys. 65 (1989) 290
- M. Belakhovsky, R. Cortés and A. Chamberod, project at LURE (1989)
\textsuperscript{13} Dispersive X-ray synchrotron studies of Pt-C multilayers, B. Rodricks et al, Rev. Sci. Instrum. (in press)
multilayers), to perform, on this prepared photon state, EXAFS-type experiments (e.g. ref. 9) or grazing diffraction\textsuperscript{14}. It has also been recently shown that a large and variable period SW can be created by total reflection onto a mirror\textsuperscript{15}.

The geometries generally in use for grazing interface studies are with the sample vertical for solids and horizontal for liquids. In the first case, the use of a vertical sample is to avoid the effect of the horizontal linear polarization on a diffracted beam that is parallel to the surface; however, this is detrimental to the compromise between flux versus angular precision on existing rings. ESRF can help in this respect in some ways. First, it provides, per definition of the ring (high energy and brilliance), good undulators for XSR; a low-K undulator gives a pinholed beam of nearly as high precision in horizontal as in vertical, therefore suppressing the above compromise. Second, the horizontal spread of a multipole wiggler, given by $2K/\gamma$, varies rapidly with the ring energy. At ESRF, a typical multipole wiggler (e.g. $E_c = 12$ keV, $p = 20$ cm, $B = 0.5$ T, $2N_p = 10$) emits horizontally within less than 2 mrad - which is already smaller at $E_c$ than most critical angles for total reflection. Finally, one should consider - below 10 keV for ESRF - the potential merit of the planar / helical concept recalled before, especially the nearly-complete vertical polarization by removal of the upper jaw. When tuneability is required over some moderate energy range (spectroscopic measurements etc.), the tapered undulator (cf §3) can be of interest.

5. Practical improvements

A new beamline is under study at LURE / DCI on the wiggler central branch (W21). It will be partly reserved to experiments requiring a high vertical collimation. Therefore, it is of some relevance to compare here such a beamline with e.g. a directly transposed one at ESRF, that will merely be installed on a standard bending magnet. The optics proposed for W21 consists of a cylindrical mirror (1 : 1 vertical refocusing), followed by a double crystal monochromator (horizontal 3 : 1 refocusing by the second crystal). The parameters for the source, geometry and optics on both rings are gathered in table 3. The comparison of these two analogous beamlines have been performed using the Shadow program. In particular, it is shown - also on table 3 - that a gain in flux of two orders of magnitude is obtained for a given vertical acceptance of the experiment.

Acknowledgments : I thank A. Boeuf for the ray tracing calculations.

\textsuperscript{14} Observation of the diffraction of evanescent X-rays at a crystal surface, P.L. Cowan et al, Phys. Rev. Letters 57 (1986) 2399
Table 1: Main parameters of the ESRF storage ring

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy</td>
<td>6 GeV</td>
</tr>
<tr>
<td>Current (in multibunch mode)</td>
<td>≥ 100 mA</td>
</tr>
<tr>
<td>Filling time (for e- and multibunch mode)</td>
<td>6 ms</td>
</tr>
<tr>
<td>Circumference</td>
<td>844 m</td>
</tr>
<tr>
<td>Radiofrequency</td>
<td>552 MHz</td>
</tr>
<tr>
<td>Maximum number of insertion devices</td>
<td>29</td>
</tr>
<tr>
<td>Free length for straight sections</td>
<td>6 m</td>
</tr>
<tr>
<td>Horizontal emittance of the e-beam</td>
<td>6.8 nm*rad</td>
</tr>
<tr>
<td>Number of bending magnet ports</td>
<td>26 (20 &amp;10 KeV)</td>
</tr>
</tbody>
</table>

Table 2: Beta and 1σ values of the electrons at the three generic source points *

<table>
<thead>
<tr>
<th>Source Type</th>
<th>Bending magnet</th>
<th>High beta (undulator)</th>
<th>Low beta (wiggler)</th>
</tr>
</thead>
<tbody>
<tr>
<td>β_H (m)</td>
<td>2.2</td>
<td>26.6</td>
<td>0.8</td>
</tr>
<tr>
<td>β_V (m)</td>
<td>26.8</td>
<td>11.3</td>
<td>3.5</td>
</tr>
<tr>
<td>σ_H (mm)</td>
<td>0.16</td>
<td>0.41</td>
<td>0.069</td>
</tr>
<tr>
<td>σ_V (mm)</td>
<td>0.129</td>
<td>0.084</td>
<td>0.047</td>
</tr>
<tr>
<td>σ'_H (mrad)</td>
<td>0.137</td>
<td>0.015</td>
<td>0.089</td>
</tr>
<tr>
<td>σ'_V (mrad)</td>
<td>0.005</td>
<td>0.007</td>
<td>0.013</td>
</tr>
</tbody>
</table>

* for the bending magnet sources, the source point is 5 mrad downstream from the entrance, where the vertical ellipse is at a waist (which is not the case for the horizontal one).
Table 3: Comparison of two analogous beamlines at LURE and ESRF*

<table>
<thead>
<tr>
<th></th>
<th>DCI / W21</th>
<th>ESRF / BM</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Distances (m)</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>source - mirror</td>
<td>18.5</td>
<td>30</td>
</tr>
<tr>
<td>mirror - monochromator</td>
<td>11.5</td>
<td>15</td>
</tr>
<tr>
<td>monochromator - sample</td>
<td>10</td>
<td>15</td>
</tr>
<tr>
<td><strong>XSR sources</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>critical energy (keV)</td>
<td>~ 10</td>
<td>19.2</td>
</tr>
<tr>
<td>divergences (mrad)</td>
<td>3.3 * 0.55</td>
<td>6 * 0.11</td>
</tr>
<tr>
<td>sizes (mm)</td>
<td>1.85 * 6.6</td>
<td>0.3 * 0.3</td>
</tr>
<tr>
<td>Flux (mrad(H) / 0.1% rbw)</td>
<td>~ 10 13</td>
<td>~ 10 13</td>
</tr>
<tr>
<td>Power (kW)</td>
<td>0.3</td>
<td>0.44</td>
</tr>
<tr>
<td><strong>Optics</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(1:1) mirror; size (cm)</td>
<td>8 * 120</td>
<td>16 * 120</td>
</tr>
<tr>
<td>angle (Pt, mrad) and cutoff (keV)</td>
<td>3.8 ; 21</td>
<td>2.5 ; 30</td>
</tr>
<tr>
<td>311 crystals; sizes (cm)</td>
<td>9 * 4.5</td>
<td>16 * 8</td>
</tr>
<tr>
<td><strong>Ray Tracing results (at sample)</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>spot size A (mm)</td>
<td>2.2 * 2.2</td>
<td>0.25 * 0.52</td>
</tr>
<tr>
<td>energy resolution ∆E (eV)</td>
<td>25</td>
<td>9</td>
</tr>
<tr>
<td>relative flux / (∆E * A)</td>
<td>1</td>
<td>90</td>
</tr>
<tr>
<td>relative flux / (∆E * Ωv)</td>
<td>1</td>
<td>30</td>
</tr>
</tbody>
</table>

* Note:
- when two quantities are given, the first one is in the horizontal plane and the second one in the vertical plane; the FWHM is given.
- whenever the quoted values are energy-dependent, E = 20 keV; the relative gain in flux is very similar at 10 keV;
- Ωv is the vertical phase space volume; A is the spot size normal to the beam.