

*The Metrology beamlines*  
*at*  
**SOLEIL**

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## Summary

*The objectives of this project is to design and install at the SOLEIL synchrotron radiation source a calibration and metrology test facility for the R&D of optical components and detectors. We propose to build, on a bending magnet, three branches dedicated to VUV, soft x-rays and hard x-rays energy ranges to cover 10 eV to 15 keV. This installation will first address the needs of the SOLEIL experimental groups (Optics and Detectors) and will be used by a large community. **This beamline will also be valuable as a general-purpose beamline to prepare, test and set up a wide range of experiments.***

*A complementary important aspect of this installation is the realization of primary standard : the metrology beamline of SOLEIL could become the national primary standard source in collaboration with the **Bureau National de Métrologie (BNM)**.*

## 1) Introduction

We propose to build at SOLEIL a facility for the accurate measurement of the properties of optical elements and detectors from the extreme ultraviolet (EUV), soft x-ray spectral regions up to the hard x-rays. The beamline will serve as a standard for such measurements and hence we call it a calibration and standards beamline, or alternatively a *metrology* beamline. **This beamline will also be valuable as a general-purpose beamline to prepare, test and set up a wide range of experiments.**

With the construction of many synchrotron radiation (SR) facilities around the world in recent years, there has been an explosion of interest and activity in the soft x-ray and extreme ultraviolet (which we shall call the XUV region) and also on the hard X-rays regions of the spectrum. However, the interest in this spectral region is not restricted to SR based science and technological applications. There has been a comparable extension in the field of x-ray astronomy, atomic physics, x-ray fluorescence, soft x-ray lasers of various types, High Harmonic Generation and in the new technological area of EUV lithography. This growth has led to a need for XUV optical components such as mirrors, telescopes, gratings, zone plates and other diffractive optics, multilayer coatings, filters and of course XUV detectors. These objects must be built with very high precision and reliability to give high performances (high spatial or spectral resolution, no overlapping orders, low diffuse scattering, resistance to degradation, etc) to the instruments for which they are designed so that the qualities of the light beam emitted by the source are conserved to be fully exploited. The development of such high performance optical components and systems for the XUV region requires a method to qualify them at the wavelength(s) of interest, first during the design and next to confirm the qualities before installation on the beamlines or in an experiment.

Development of advanced optical components must progress with a parallel development of metrology because as one often says :

***“If you can measure it, you can make it”.***

While this may not be universally true, it is certain that if one cannot measure the performance of whatever “it” may be at the operational wavelength, one will never know if “it” has been made correctly. In particular, one needs to get optical components having defects below the scale of the used wavelength, and so to reach almost the atomic scale precision. This aspect is crucial for all the mirrors which need to be manufactured with a few  $\mu$ rad slope errors and the angstrom level in roughness in order to preserve the good wavefront quality of the incident beam and to obtain very small focal spot.

## Metrology for Synchrotron radiation R&D

In the past, the x-ray optical designer, like the x-ray physicist, had been constrained by the low brightness and limited tunability of conventional x-ray sources. Nowadays, with synchrotron radiation sources, particularly the third generation synchrotron sources and the development of instruments which reach high spatial and spectral resolution, the situation has improved significantly. To preserve the high quality of the beam delivered by the new synchrotron source, one needs to improve the qualities of the optical elements. Checking this improvement obviously requires a source with equivalent qualities.

A perfect example is given by the 6 GeV European Synchrotron Radiation Facility (ESRF), where a high-energy test beamline has been in operation since 1991. For the past ten years, extraordinary results have been obtained. These results range from the first observation of

speckles produced by beryllium windows (which drew attention to the coherent aspects of synchrotron-produced x-rays), to the evaluation of traditional optics and the development of novel ones, such as multilayers, Bragg-Fresnel Optics, modulated optics, refractive x-ray optics, microfocusing systems including capillary, Kirkpatrick-Baez optics, etc. **Flexible instrumentation and quick access to a DEDICATED beamline opened the way to very fast realization and test for new ideas in the field of x-ray optics and x-ray detectors.** The fast progress of ESRF's scientific and technological capabilities was to a large extent due to the investigations at the x-ray optical tests beamline. **This facility was one of the first ESRF beamlines.**

The construction of a new third generation synchrotron radiation facility in France (SOLEIL) brings with it the problem of metrology and calibration of the various components involved. Throughout all of the wide range of applications covered by SOLEIL, **there is a need for a beamline where all the optics and the detectors could be calibrated and characterized.**

### **Metrology for external users R&D**

Another example illustrating the need for a metrology beamline, from the soft x-ray/EUV spectral region, is given by the Calibration and Standards beamline 6.3.2 at the Advanced Light Source (ALS) in Berkeley, California. Since this beamline gave its first results in 1994 it has become *the standard* for the measurement of the optical properties of optical elements and materials in the XUV and one of the most heavily utilized beamlines at the ALS. In the quest to find the most reflective, low scatter and stable multilayer coatings for Extreme Ultraviolet Lithography (EUVL), the US program has relied heavily on this beamline. Measurements from the beamline are fed back to the coating laboratory iteratively, to improve the deposition process. In fact it is no exaggeration to state that the rapid progress made in the US EUVL program could not have been made without the capabilities provided by beamline 6.3.2.

In Germany, the Physikalische-Technische Bundesanstalt (PTB) has taken an important place with a specific laboratory having 8 dedicated beamlines on BESSY I and an equivalent complex on BESSY II.

The experience gained at these beamlines has clearly had a deep impact on the progress of specific instrumentation in their respective spectral ranges. The expectation that similar positive results that could be obtained using a dedicated beamline for VUV radiation and soft X-rays at SOLEIL is very high.

A group of reflection on X and XUV Optics called PRaXO (Pôle Rayons X d'Orsay<sup>1</sup>) has been set up at the Université d'Orsay since 1999, on the initiative of three laboratories directly involved: the LSAI, the LURE and the IOTA. One of the objectives of this group is to define common strategies for the X and XUV optics to solve some technological problems which hamper, or may hamper, the scientific community working with XUV sources. In this framework, a close collaboration between these three laboratories was set up for the conception, fabrication and metrology of optical systems. Since 2000, several other laboratories, mainly from CEA, are associated to this effort. This x-ray/EUV beamline at SOLEIL will complement the ESRF's hard X-ray test facilities and, with the support of the Bureau National de Métrologie (BNM) those of the PTB at the European scale

Thus, concurrently with the construction of SOLEIL, an instrumental effort must be established. This effort has two aspects:

- to set up, test and calibrate the optics and the detectors to be installed on this new source.

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<sup>1</sup> <http://lure.u-psud.fr/optique/PRaXO/xxuv.html>

- to assure the continuity of the already existing calibration activities at LURE performed by various laboratories (LURE, CEA-DAM<sup>2</sup>, IAS<sup>3</sup>, LULI<sup>4</sup>, LOA<sup>5</sup>, LNHB<sup>6</sup>, ...), and to upgrade them.

## 2) Interest of the synchrotron radiation (SR) for metrology

Synchrotron radiation is not only a source for fundamental research, it also has unique properties of brilliance, polarization, coherence, and temporal structure that makes it the reference source for metrology over a wide range of energy.

### 2.1 Source of continuous radiation

Unlike most laboratory sources (discharge lamps, X-ray tubes, lasers, radionuclides...) whose emission is made of discrete spectral lines, synchrotron radiation is « white », meaning it delivers a continuous spectrum from infrared to X-rays. It allows the extension of radiometry, traditionally bracketed by the infrared and the ultraviolet domain, toward higher energies, up to the hard X-ray range.

### 2.2 Calculable photons flux

The spectral photon flux of radiation originating from relativistic electrons deflected in the magnetic field of a **bending magnet** can be calculated from fundamental electrodynamics relations (Schwinger theory<sup>7</sup>), thus **making these sources primary radiation standards**. A very favorable feature of storage ring sources is the fact that the radiant power can be varied in a controlled manner over about 12 orders of magnitude by appropriately adjusting the electron beam current. Even the spectral shape can be altered by selection of the electron energy to best fit a certain calibration task.

To calculate synchrotron radiation intensity, some beam parameters (electron beam energy, electron beam current, magnetic flux density in the bending magnets, beam size, beam acceptance) need to be evaluated with a high degree of accuracy<sup>8</sup>.

### 2.3 Brilliant source

The brilliance of synchrotron radiation, when used in conjunction with well-designed monochromators matched to the source characteristics, allows the production of a beam with high spatial and spectral quality.

The very small beam emittance (small divergence coupled with small source size) will allow focusing to spot sizes of micron and sub-micron level throughout practically the whole energy range thus upgrading the performances of the imaging and microanalysis techniques.

Many beamlines of SOLEIL will require to focus a micro spot of X-rays on the sample. The refocusing stages could be made with bendable mirrors and should allow full control of incidence angles, radius of curvature and may be asphericity of the individual surfaces. Alignment tools will be permanently associated with this kind of optics to supplement or

<sup>2</sup> Commissariat à l'Energie Atomique – Direction des Applications Militaires

<sup>3</sup> Institut d'Astrophysique Spatiale

<sup>4</sup> Laboratoire pour l'Utilisation des Lasers Intenses

<sup>5</sup> Laboratoire d'Optique Appliquée

<sup>6</sup> Laboratoire National Henri Becquerel

<sup>7</sup> Schwinger J. (1949) *Phys Rev.* 75, 1912-1925

<sup>8</sup> Hollandt J. et al *Rev. Sci. Instrum.* 63 (1) 1278-1281 (1992)

Klein R. et al (1997) *Nucl. Instrum. Methods*, A384, 293-298

Lei F et al (1996) *Metrologia* 22,75-85

Tegeler E., ulm G. (1988) *Nucl. Instrum. Methods*, A282, 706-713

assist the user in the focusing task. Determination of the feedback parameters of the system and its commissioning will require beam time before the destination beamline is ready. Such small spots with both a high spectral purity and a high radiant power are essential to perform absolute calibration of the monochromatized beam with an absolute detector (such as small electrical substitution cryogenic radiometer). In a second step, calibration of reference detectors with a high accuracy (secondary standards) will be possible.

## 2.4 Temporal structure

Synchrotron radiation has a pulsed temporal structure. On SOLEIL, it will be possible to produce light pulses of 50 ps every 140 ns. The use of this temporal structure will allow the characterization of fast detectors used in the study of systems with fast evolution like laser produced plasmas.

## 2.5 Polarization - Coherence

Finally, the polarization (adjustable, linear or circular) and the coherence of the beam are intrinsic characteristics of the synchrotron radiation that will be exploited.

The range of application of coherent experiments is quite wide. In principle, any object or phenomenon resulting in an optical phase variation (either vs space or vs time) can be accessed through interferometry. This includes the calibration of phase plates (CALCORR ESPRIT project<sup>9</sup>, within the EUV lithography context), but also the determination of optical constants and dispersion, the study of the “phase roughness” issuing from multilayered reflectors (possibly by interferential microscopy), and even the test of aspherical wavefronts.

On a slightly different principle, coherent microscopies such as dark field, Zernike phase contrast are also experiment which benefits from an increased brightness.

These experiment have already been demonstrated on Super ACO (LURE/Orsay) so it is obvious that the same metrology principles could be performed on any beamline dedicated to metrology. As a matter of fact, an important gain in performance can be expected from the transfer on SOLEIL due to the increase of brightness (see contributions).

## 3) Calibration and Metrology in X and X-UV

### *Examples of metrology and R&D activities using synchrotron facilities.*

Different institutes have been using specialized beamlines on synchrotron facilities to develop metrological studies :

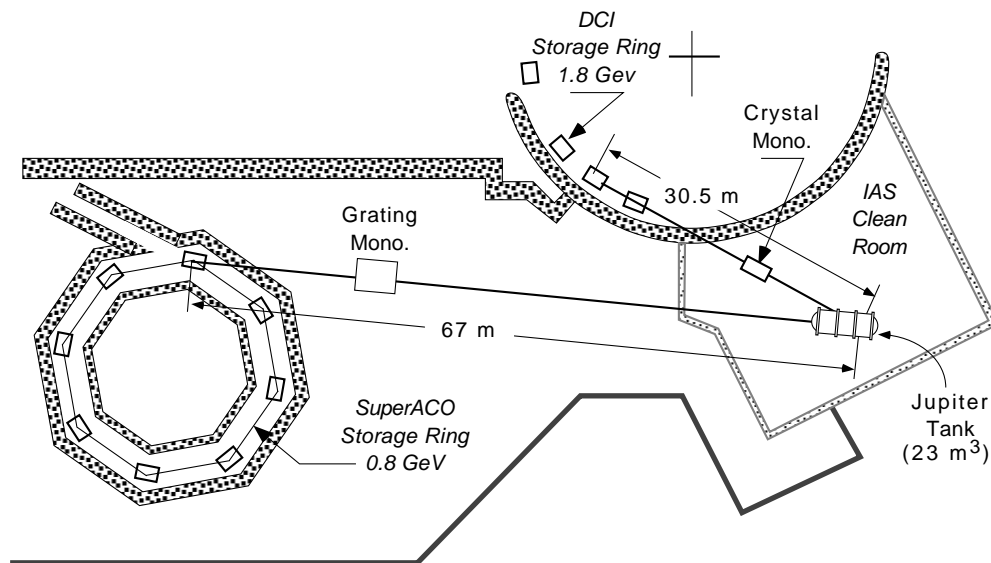
- **In France**, calibration, strictly speaking, was an activity undertaken on the first beamlines of ACO. A team of the Bureau National of Metrology (BNM) wanted to develop a secondary standard source in the UV domain, and attempted to characterize an electric arc in comparison to ACO. From 1995, DAMRI, a CEA laboratory linked to the BNM, uses Super ACO SB3 beamline to measure the quantum efficiency of Si(Li) detectors and scintillators.

Practically from the start of LURE, the utilization of synchrotron radiation by CEA-DAM has remained a constant instrumental activity. The needs of this laboratory consist principally of the qualification of X-UV optics and detectors designed for the study of hot and dense plasmas. Similar study are done by LULI at the École Polytechnique of Palaiseau. These activities are for the main part achieved on a dedicated beamline (SB3), functioning between a few hundreds of eV and a few keV.

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<sup>9</sup> EU-funded ESPRIT-4 project #28146 « CALCORR » (*Interferometric CALibration of a CORRective phase layer for diffraction-limited EUV mirror optics*)

In the Astrophysics field, synchrotron radiation was used for the calibration of a multilayer mirror telescope and for the CCD camera of the EIT experiment on the SOHO mission. For the experiment EPIC in 1993, the Institut d'Astrophysique Spatiale was in charge of the calibration of the 3 CCD cameras functioning between 0,1 and 15 keV. The EPIC cameras were installed on the XMM satellite, launched in 2001. This effort involved the construction of a dedicated laboratory in a clean room environment to use the synchrotron beam from Super ACO for the soft x-rays, and DCI for the hard x-rays To achieve this calibration, two specially adapted monochromators were built: a grating monochromator on SACO and a double crystal monochromator on DCI. For this wide energy range, these two beamlines possess characteristics that provide most of the specific needs for detectors calibration. A schematic drawing of these beamlines is presented in figure 1.



**Figure 1 : Calibration beamlines at LURE**  
**Energy range 100 eV – 15 keV**

Since 2000, these beamlines are used both by LURE and IAS for detectors and optics characterization.

- **In the U.S.A.**, the National Institute of Standards and Technology (NIST) operates the 405 MeV synchrotron SURF III in Gaithersburg<sup>10</sup> : it is used as a calculable photon source for the far UV (50-260 nm) with an overall measurement uncertainty of about 5 % , and a cryogenic radiometer is used for detector calibrations in the UV range (125-320 nm). This facility also includes functions for UV material characterization. Moreover, several synchrotron facility (APS, ALS and BNL) have dedicated beamlines for R&D on X-ray optics and detectors.
- **In Germany**, the Physikalische-Technische Bundesanstalt (PTB)) has access to an important dedicated metrology facility at BESSY II in Berlin<sup>11</sup>. Various experimental stations allow for the use of undispersed, calculable radiation up to an energy of about 50 keV and of monochromatized synchrotron radiation in the 3-15000 eV spectral

<sup>10</sup> P-S Shaw, T.C. Larason, R. Gupta, S.W. Brown, R.E. Vest and K.R. Lykke  
 Rev. Sci. Instrum. Vol 72 N°5 (2001), 2242.

<sup>11</sup> G. Ulm, B. Beckhoff, R. Klein, M. Krumrey, H. Rabus, R. Thornagle SPIE Vol.3444 (1998), 610.

range. Major activities include the calibration of radiation detectors and radiation sources, as well as the characterization of optical components in the VUV and soft X-ray spectral range with low uncertainty. In addition, the BESSY Center has built its own optical beam line to develop new optical concepts and devices. The PTB is also deeply involved in all optics calibration for the European project on EUV lithography.

- **In Russia**<sup>12</sup> (Budker Institute of Nuclear Physics – VEPP-2M and VEPP-3 – Novosibirsk), the work on radiometry has been done since the 1980's on the VEPP-2M synchrotron for the wavelength range : 50 – 550 nm (2.3 to 25 eV) and on spectrometry in the 120-400 nm range (3 to 9.7 eV)<sup>13</sup>. The standardization beamline in particular includes a permanent normal incidence monochromator. The uncertainty of the standardization of the transfer detectors is close to 5 % for energies below 6 eV and 10 % for higher energies. The qualification of the transfer sources (hydrogen or deuterium discharge lamps–hollow cathode lamps) is also achieved on this beamline.
- **In China** : the national institute of metrology uses two beamlines reserved for metrology on a synchrotron shared with the Chinese universities.
- **In Japan**, at the Electrotechnical Laboratory (ETL)<sup>14</sup> storage ring, (TERAS – Tsukuba), the synchrotron radiation is used for radiometric standards and as a light source with calculable radiant power in the soft X-rays region.

The determination with a very high precision of the absolute photon flux emitted by a bending magnet to make it a primary radiation standard is a task that presently only the PTB at BESSY and NIST at Washington have made. Even with all the constraints involved in these kinds of experiment, this activity is ensured on BESSY II, for the needs of the PTB and because the uniqueness of this facility attracts laboratories from around the World such as the Lawrence Livermore Laboratory and many laboratories of Space Astrophysics.

### *Calibration and Metrology on the new French machine.*

It is clear that in the beginning of SOLEIL, an important effort in terms of optics and detectors metrology will be required. **The objectives of this metrology test facility is to first perform the test and the R&D of x-ray optical components and detectors.**

The Metrology Beamline will be operated through the Optics Group. It should be located close to the Crystal Preparation Laboratory and the Optics Metrology Laboratory. An horizontal opening angle of around 8 mrad of available bending magnet radiation will be divided into three Branches. The main purpose of the beamline is to serve as a test and development station for x-ray optical elements and for beam characterization (ex.: coherence) and as an opportunity to perform feasibility experiments related to optics instrumentation.

The absence in our country of a complete X-UV metrology facility such as the one developed by the PTB at BESSY is an element which must be taken into account. **However, needs are clearly expressed (see annexes and contributions).** To satisfy these needs, we suggest to couple them with the instrumental activities aim at developing SOLEIL beamlines.

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<sup>12</sup> E. S. Gluskin et al, *Nucl. Instrum. Methods A* 246, 397, (1986)

<sup>13</sup> A.N. Subboton, V.V. Gaganov, A.V. Kalutsky, V.F. Pindyurin, V.P. Nazmov, A.D. Nikolenko and A.K. Krasnov *Metrologia* Vol.37 (2000), 497

<sup>14</sup> I.H. Suzuki and N. Saito *Japanese Journal of Applied Physics* V. 25 N°1 (1986) 130.  
T. Zama et al *J. Synchrotron Radiation* (1998) 5, 759-761

One can regard this complementary instrumental effort as a trial to extend the use of SOLEIL to a kind of applied physics and instrumentation facility accessible to several French industries and the Bureau National de Metrology. Evidently, the coupling and the calendar of the development of both aspects must be discussed but integrated early in the design of the calibration beam line.

#### 4) Characteristics of the calibration beamline at SOLEIL

Most synchrotron radiation users require high flux density and high spectral resolution on their samples. Such characteristics are not really necessary for the metrology of optics and detectors. For metrology one requires mostly parallel or quasi-parallel beams, together with very high spectral purity (no harmonics) and the smallest possible amount of scattered light. The energy range must be as large as possible, typically from 10 eV to 20 keV. To cover this wide energy range, the use of two kinds of monochromators is required :

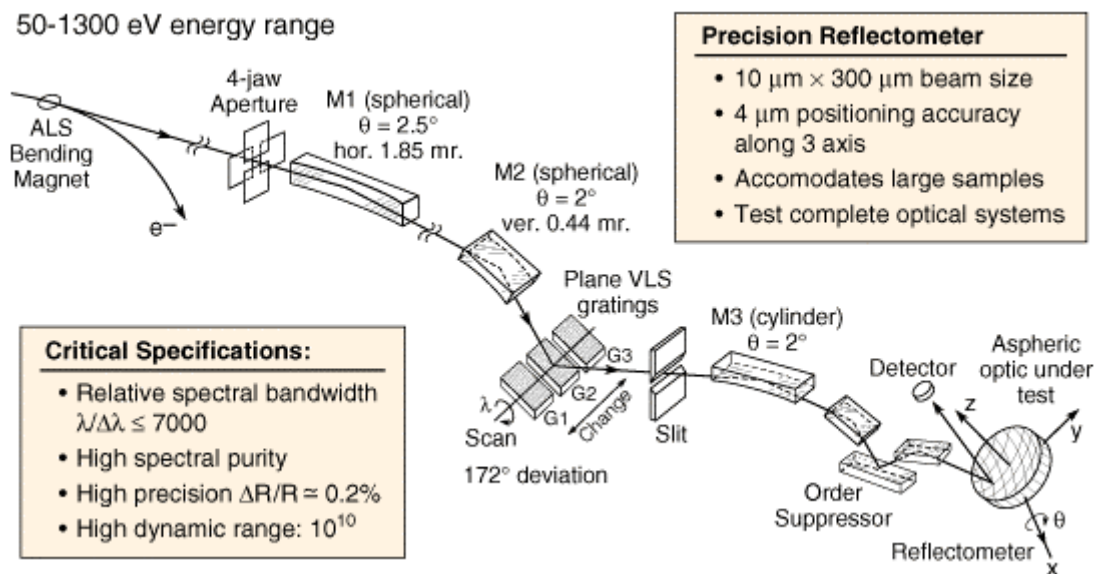
- gratings monochromators to cover the EUV and the soft x-ray range (10 eV – 1900 eV)
- a double crystal monochromator for the hard x-ray part of the spectrum (1.5 – 20 keV).

We need to be able to make absolute measurements of the incident photon flux for detectors quantum efficiency measurement. To achieve this, we need to use an order suppressor based on total reflection of mirrors in grazing incidence.

The flux required to calibrate detectors is usually very low and so difficult to measure, thus we need a neutral and reproducible densities of various given ratios such as fast mechanical choppers. In addition, we need to be able to insert various filters to avoid UV and visible light.

**To provide a basis for discussion of the needed beamlines, we hereafter give two examples of metrology beamlines recently constructed on 3<sup>rd</sup> generation synchrotron facilities.** These two calibration beamlines are sketched in figures 2 and 3. They both use a bending magnet.

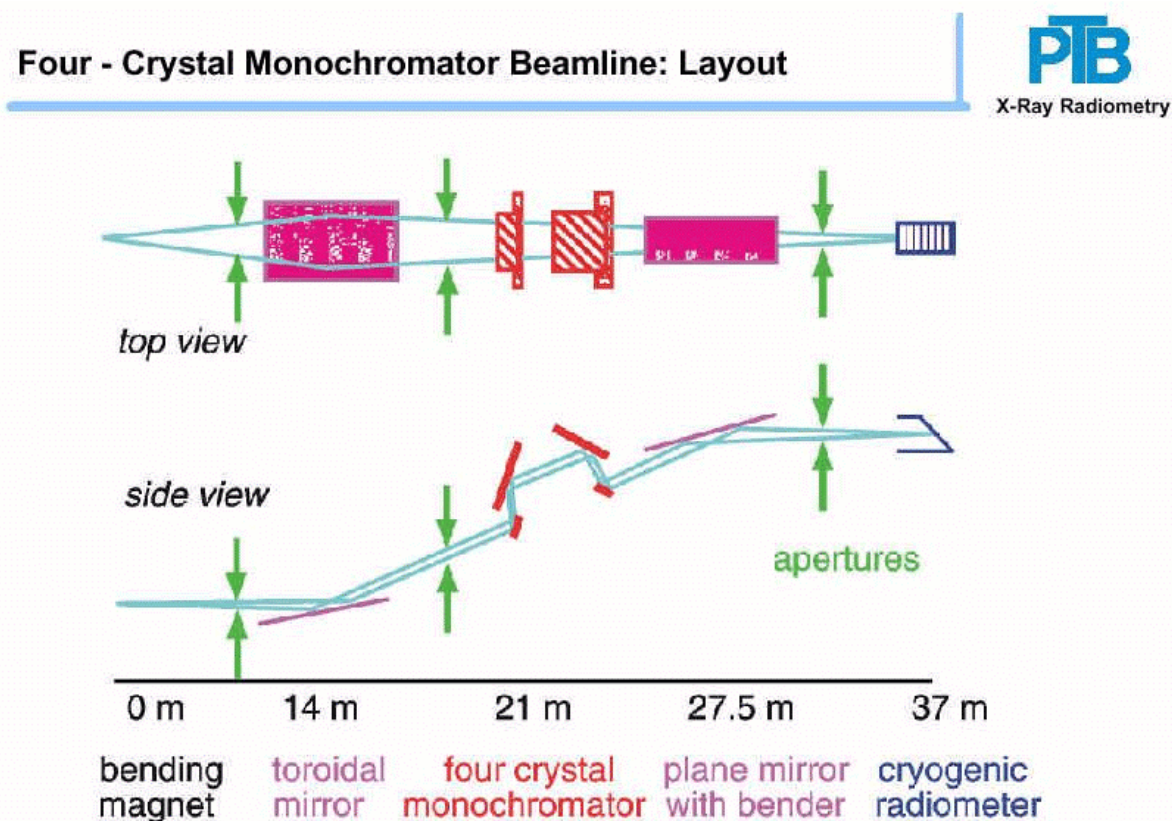
Figure 2 shows a schematic drawing of the 6.3.2 calibration beamline at the Advanced Light Source (ALS).



**Figure 2: Schematic view of the calibration beamline 6.3.2 of the Advanced Light Source.**

This beamline was built for the characterization of optics and detectors for the XUV radiation (multilayers, mirrors, gratings, diodes..) in the energy range 50 eV to 1500 eV<sup>15</sup>. This beamline was mainly developed to make precise measurements of multilayer reflectivity for the project of lithography by projection. The beamline consists of a horizontal focusing mirror M1 (making an image of the source at the sample location), a variable line spaced grating monochromator operating in the converging light produced by a concave spherical mirror (M2 in figure1). There is no physical entrance slit in this beamline; the small size and the stability of the ALS source allows it to be used instead of a classical entrance slit. A vertical refocusing mirror (M3 in figure 1) images the exit slit of the monochromator at unit magnification at the sample location. An unequal end couple mirror bender allows the mean radius of curvature of M3 to be varied. For greater spectral purity, a 3 mirrors order suppressor can be inserted into the beam after the M3 mirror. The beam size on the sample is 50 x 300  $\mu\text{m}$  approximately. Due to the simplicity of the mechanical design an additional advantage is the high stability and reproducibility for the energy calibration.

Figure 3 represents the “hard x-ray” calibration beamline of the PTB on BESSY II<sup>16</sup>.



**Figure 3 : the “hard x-ray” calibration beamline of the PTB on BESSY II.**

In this beamline, in order to achieve a very high reproducibility in photon energy scans, monochromatization and focusing are completely decoupled. A toroidal mirror focuses the

<sup>15</sup> “Calibration and Standards beamline 6.3.2 at the Advanced Light Source”

J. H. Underwood et al. Rev. Sci. Instrum. 9 (Sept. 1996).

<sup>16</sup> “The PTB radiometry laboratory at the BESSY II electron storage ring”

G. Ulm et al. Proc. SPIE 3444 (1998)

beam in the horizontal plane and collimates it in the vertical one (figure 3). A flat bendable mirror behind the monochromator can be bent to focus the horizontal and the vertical dimension at the sample location. A 4 crystals monochromator provides a very high stability of the monochromatic beam for detectors and optics calibration without crystals translations. The photon energy is scanned by two precisely controllable rotations. To cover the spectral range from 1.7 to 10 keV, a set of 4 InSb (111) and Si (111) crystals are used

It is possible to get the white beam by removing the double crystal monochromator and the mirrors from the beam or to use only the monochromator without focusing optics. With this last option, we can get a monochromatic beam up to 30 keV or more.

On these two beamlines, the end station is a  $\theta$ - $2\theta$  reflectometer with several degrees of freedom allowing reflectivity measurements on X and XUV optics and detectors quantum efficiency measurements.

**The proposed idea is to construct two “similar” beamlines on a single bending magnet with the addition of a Large Glancing Angle Grating Monochromator. With this configuration, we can simultaneously use the three monochromators to cover a very broad energy range**

### **5) Schematic view of the possible implantation of the Metrology beamlines at SOLEIL**

The table below summarizes the needs for the realization of a metrology beamline at SOLEIL.

<b>Source Characteristics</b>	<b>Bending Magnet</b>
<b>Beamline Characteristics (monochromator)</b>	<ul style="list-style-type: none"> <li>• <b>Grating Mono. (Large glancing angle monochromator) (10 eV – 100 eV)</b></li> <li>• <b>Grating Mono. (grazing incidence) (50 eV – 1800 eV)</b></li> <li>• <b>Crystal Mono. (1750 eV – 20 keV)</b></li> <li>• <b><u>Access to the white radiation</u></b></li> </ul>
Temporal structure	50 ps (bunch width)
Description of the various elements under consideration on the beamline.	<ul style="list-style-type: none"> <li>• Mirrors low-pass filter (high order suppressor)</li> <li>• <math>\theta</math>-<math>2\theta</math> Reflectometer</li> <li>• Adjustable collimation slits (Horizontal and vertical)</li> <li>• Different size of pinholes</li> <li>• Filters Wheels</li> <li>• Provision of a signal for synchronization</li> </ul>
Requirement in additional rooms	"Clean" room for : --assembly of thin filters. --intervention on optical parts, detectors, photographic films and photostimulables screens. -- storage of hardware, tools, data processing, electronics, optics, filters and detectors.
Planning of the experiments	Semi-annual planning with some restricted beamtime to provide short time access for urgent work.

On SOLEIL, a bending magnet source will deliver a continuous spectrum of photons with a critical energy  $E_c = 6.5$  keV. The main characteristics of the SOLEIL bending magnet source are summarized in Table 1.

	$\sigma_x$ ( $\mu\text{m}$ )	$\sigma_z$ ( $\mu\text{m}$ )	$\sigma'_x$ ( $\mu\text{rad}$ )	$\sigma'_z$ ( $\mu\text{rad}$ )
$1^\circ$	60.1	24.9	134.8	2.1
$4^\circ$	42.1	24.5	107	2.1
Energy 2.75 GeV		Radius 5.817 m		

**Table 1 : Characteristics of SOLEIL**

**From the SOLEIL bending magnet, we would like to extract around 8 mrad in the horizontal direction.** From this 8 mrad, we can set up three different beamlines equipped with the necessary three different monochromators. One similar to beamline 6.3.2 (cf. figure 2) with a Variable Line Space (VLS) grating monochromator for the 0.05–1.5 keV energy range, another with a double crystal monochromator for the 0.8–15 keV energy range and the last one with a near normal incidence monochromator for the 0.01–0.1 keV energy range. Each monochromator will accept around 2 mrad in the horizontal plane from the bending magnet. From the hard x-ray monochromator, we will get access to the white radiation. A schematic view of the implantation of these three monochromators is illustrated in figure 4.

**We request that the metrology beamlines lie close to the annex that houses the metrology support laboratory (Crystal preparation room, Long trace profilometer, AFM, interferometers,...).**

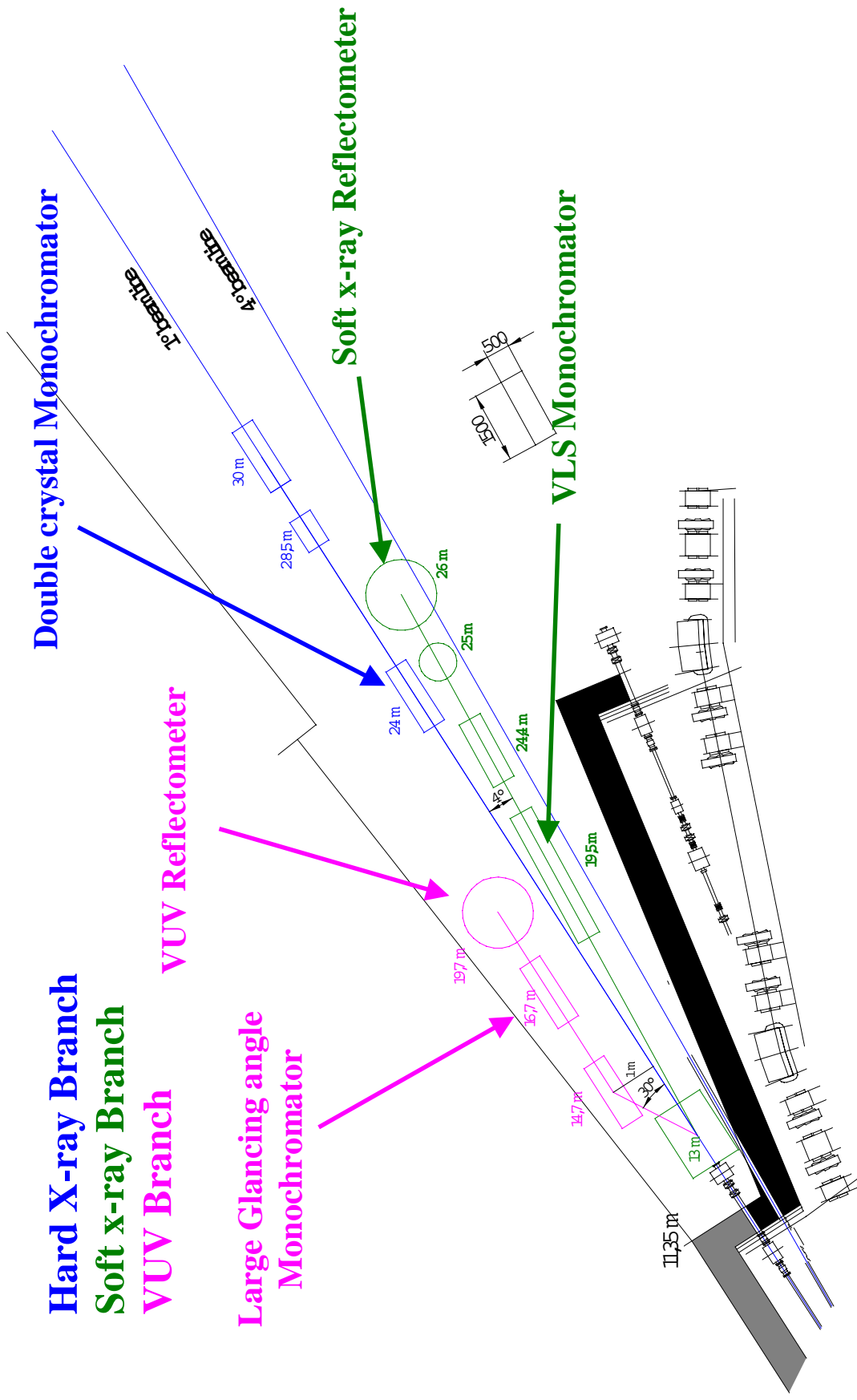


Figure 4 : Schematic implantation of the metrology beamlines on SOLEIL

## 5.1) Calculated performances of the beamlines

### a) Hard x-ray Branch Energy range 1.75-15 keV

#### Beamline Layout

Optical component	Distance from the source (m)	Orientation (deg.)	size(mm)	Optic geometry
collimator (slit)	12		24 x 7.2	
Mirror 1 M1 Image the source (vertical + horizontal focusing)	13	0	50 x 1100	Toroidal $p=13\text{ m}$ $q=17\text{ m}$ $\theta=0.4^\circ$ Pt 500 Å
Double crystal monochromator Si111/Si311 InSb 111	24	0-180	40x40	Plane $\theta$ variable $\theta\sim 5-75^\circ$
Order Sorter	28.5	0-180	50x150	Plane Si/C $\theta\sim 0.3-0.6^\circ$
Sample	30			

Table 2: Position and geometry of the optical components.

A layout of the beamline is illustrated on figure 5.

Vertical and horizontal focusing will be provided by a toroidal mirror M1 located at 13 m from the source. This mirror is made of silicon with a Pt coated layer of about 50 nm.

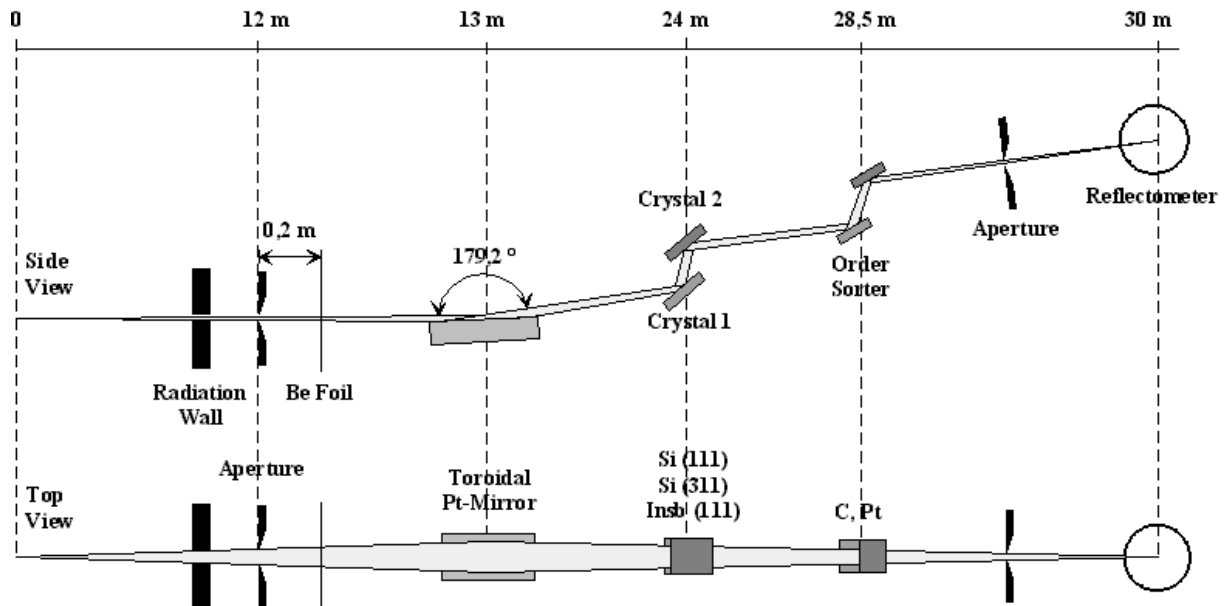
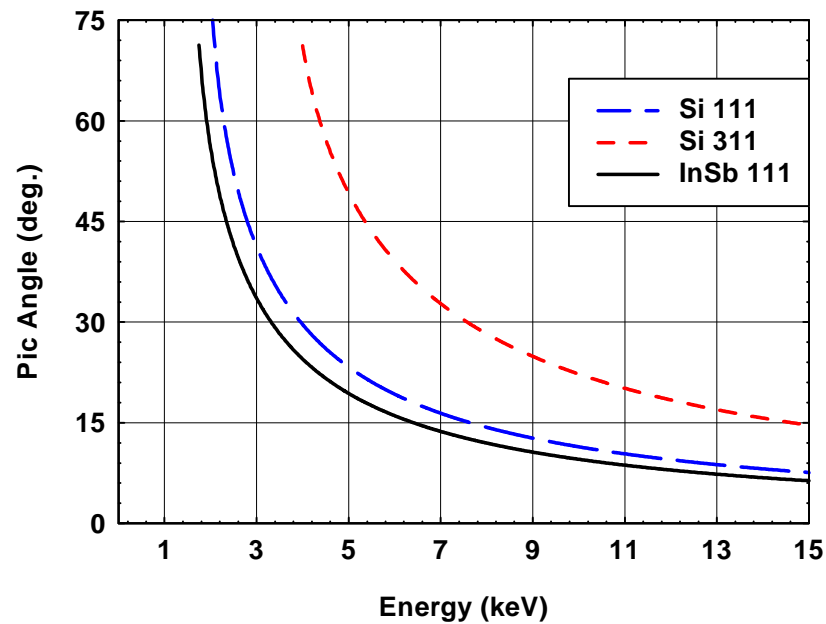


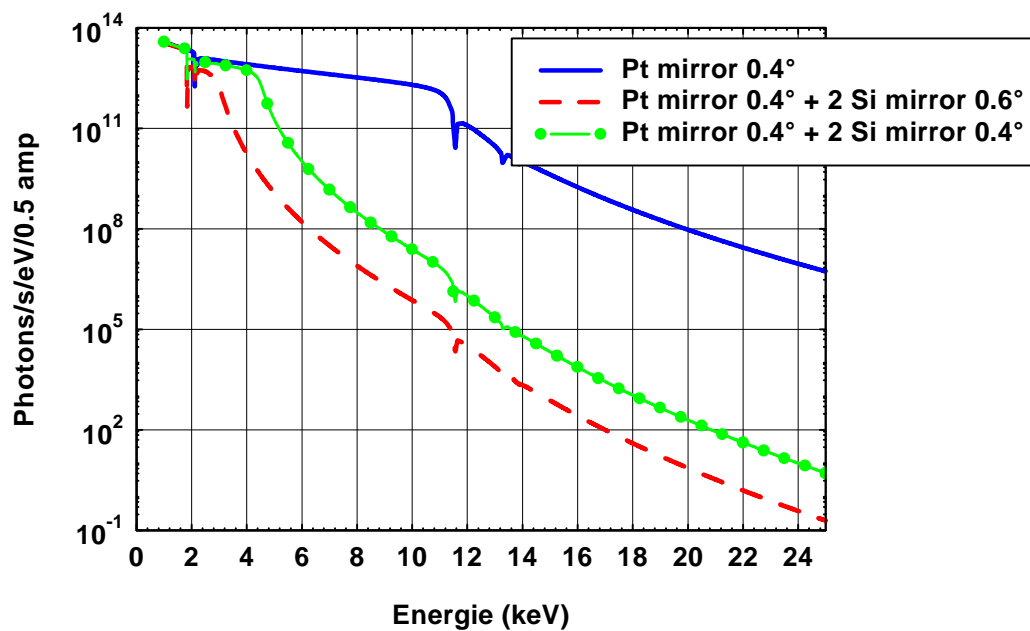
Figure 5 : Hard x-ray Branch

From this hard x-ray branch we will cover 1.75 to 20 keV energy range. To be able to do this with a double crystal monochromator (DCM), we need three different crystals. Figure 6 represent the angular range of Si 111, Si 311 and InSb 111 crystal.



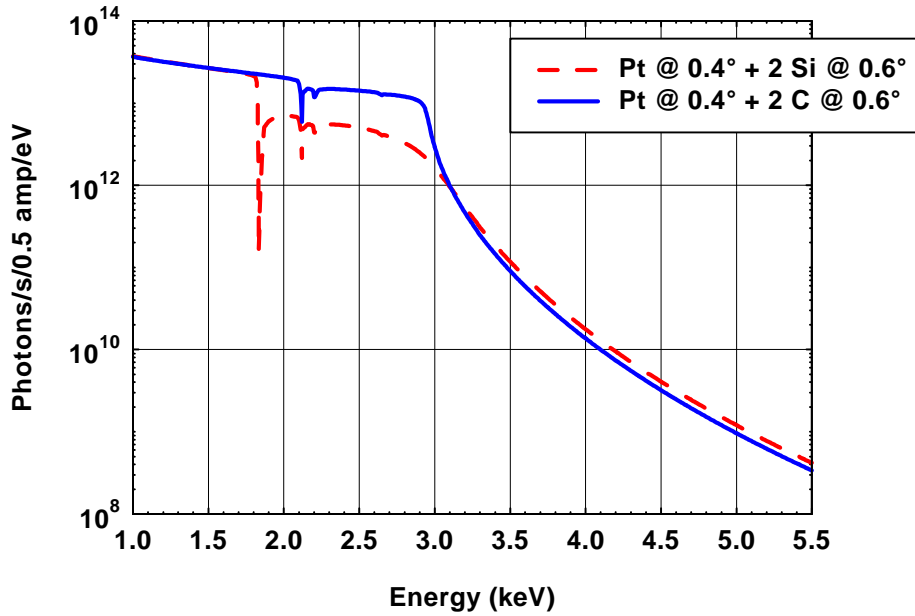
**Figure 6 : Angular range of the double crystal monochromator to cover 1.75 to 15 keV**

Figure 7 represents the theoretical photons flux for a single Pt mirror at  $0.4^\circ$  grazing angle and two order sorter silicon mirror at  $0.4^\circ$  and  $0.6^\circ$  mirror. From this curve, it is clear that an order sorter mirror coated with Si can be used to remove high order that may be reflected by the DCM.



**Figure 7 : Mirrors throughput**

A double coating Si and C on the same mirrors will be useful for experiment close to the silicon edge (1.74 keV).



Ray tracing calculation have been done to estimate the flux, the spectral and spatial resolution of the beamline. The results are summarized in the following Table.

Energy keV	N (ph/s/0.5 amp)	$\Delta E$ (eV)	Geo. Image ( $\mu m$ ) (80% energy)	Harmonic (ph/s/0.5 amp)	Mono Angle
1.75	$3.7 \times 10^{12}$	0.52	210x215	$1 \times 10^7$ (Si @0.6°) 2 <sup>nd</sup> order $1 \times 10^7$ (Si @0.6°) 3 <sup>rd</sup> order	InSb 111 71.33°
2	$4 \times 10^{12}$	0.7	210x215x	$1.5 \times 10^7$ (C @0.6°) 2 <sup>nd</sup> order $4 \times 10^6$ (C @0.6°) 3 <sup>rd</sup> order	InSb 111 56°
5	$1.5 \times 10^{12}$	3	200x220	<i>Negligible</i>	Si 111 23.3°
10	$3 \times 10^{12}$	10	186x210	<i>No mirror OS</i>	Si 111 11.4°
10	$9 \times 10^{11}$ (Slit 1mm)	3.5	186x210	<i>No mirror OS</i>	Si 111 11.4°
10	$2 \times 10^{11}$ (Slit .2mm)	1.9	186x210	<i>No mirror OS</i>	Si 111 11.4°
15	$1 \times 10^{11}$	17	190x210	<i>No mirror OS</i>	Si 111 7.58°
15	$10^{10}$ (Slit .2mm)	3.5	190x210	<i>No mirror OS</i>	Si 111 7.58°
25 <i>No mirror</i>	$9 \times 10^{10}$ (Slit 1 mm)	13	50 x2.6 mm	<i>No mirror</i>	Si 311 8.71°
35 <i>No mirror</i>	$3.2 \times 10^{10}$ (Slit 1 mm)	25	50 x2.6 mm	<i>No mirror</i>	Si 311 6.21°
35 <i>No mirror</i>	$7.2 \times 10^9$ (Slit 1 mm)	5	50 x0.5 mm	<i>No mirror</i>	Si 311 6.21°

Table of estimated flux, size and spectral resolution of the hard x-ray branch

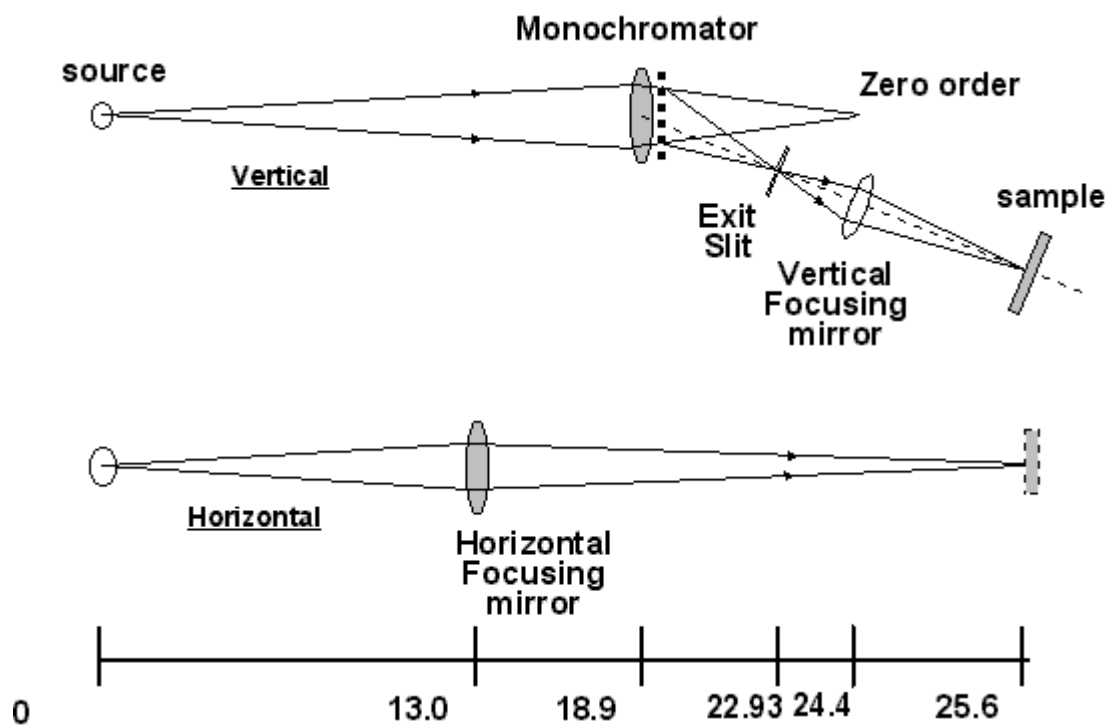
**Of course, it is always possible to get higher spectral resolution by reducing the slit size but with a reduction of flux.**

b) **Soft x-ray branch**

**Energy range 0.05-2 keV**

A layout of the beamline is illustrated on figure 8.

An horizontal focusing mirror S1 (figure 8) located 13 m from the source will make a 1:1 image of the source at the sample location. The monochromator is a variable line spaced grating operating in the converging light produced by a concave spherical mirror (S2 in figure 8). There is no physical entrance slit in this beamline; the small vertical size of SOLEIL source allows it to be used instead of a classical entrance slit. A vertical refocusing mirror (S3 in figure 8) images the exit slit of the monochromator at unit magnification at the sample location. An unequal end couple mirror bender allows the mean radius of curvature of S3 to be varied and so to focus the beam at different location. For greater spectral purity, a 3 mirror order suppressor can be inserted into the beam after the S3 mirror. The beam size on the sample will be 20 X 150  $\mu\text{m}$  approximately.



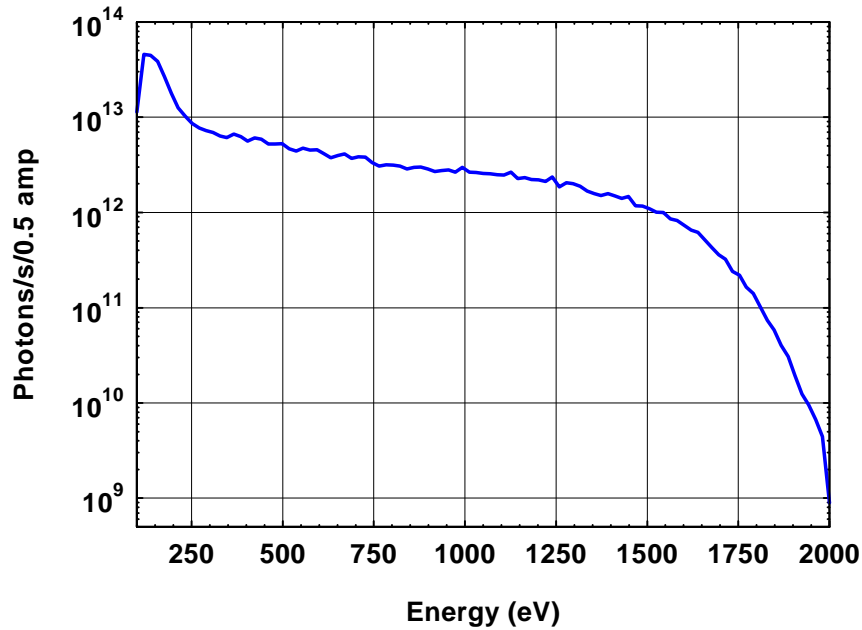
*Figure 8 : Soft x-ray Branch*

### Beamline Layout

Optical component	Distance from the source (m)	Orientation (deg.)	size(mm)	Optic geometry
collimator (slit)	10		20 x 2.2	
Mirror 1 S1 Image the source (horizontal focusing)	13 <i>p=13m</i> <i>q=12.93 m</i> <i>θ=2°</i>	90	60 x 750	Spherical Radius=371.5 m Au 500 Å @ 2°
Mirror S2	18.9 <i>p=18.9 m</i>	0	50x400	Spherical Au 500 Å @ 2° Radius~190 m
VLS Grating monochromator 200 lines/mm 600 lines/mm 1200 lines/mm 2400 lines/mm Exit slit	19.4 <i>p=19.4 m</i>  <i>θ variable</i>  22.93	180	30x100	Plan VLS Au @ 2.5°
Mirror 2 S3 Image the exit slit Bendable (vertical focusing)	24.4	180	50x500	Bendable R = ∞ to  Au 500 Å @ 2°
Order Sorter 3 reflections mirrors	25	0 180 0		Plan Si,C,Ni @ 0-3°
Sample	25.9			

**Table: Position of the optical components.**

Figure 9 represents the theoretical photon flux with three gold coated mirrors (S1, S2 and S3) at 2° and assuming that the grating is a gold mirror at 2.5°.



**Figure 9 : Mirrors Reflectivity throughput**

c) EUV branch Energy range 0.01-0.5 keV

A layout of the beamline is illustrated on figure 10.

An horizontal Carbon mirror V1 (figure 10) deflects the beam with a grazing angle of 15°. With another 15° grazing angle Carbon mirror (V2), this branch is parallel to the hard x-ray beamline. The monochromator is a variable line spaced grating operating in the converging light produced by a concave spherical mirror (V3 in figure 10). The general design of this beamline is similar to the soft x-ray branch.

**Beamline Layout**

<b>Optical component</b>	<b>Distance from the source (m)</b>	<b>Orientation (deg.)</b>	<b>Size (mm)</b>	<b>Optic Geometry</b>
<b>Aperture</b>	<b>12</b>		<b>19x22</b>	
<b>1<sup>st</sup> mirror V1 Deflects the beam in the horizontal plane</b>	<b>14.7</b>	<b>90</b>	<b>30x110</b>	<b>Spherical Radius = 54.7 m C @ 15°</b>
<b>2<sup>nd</sup> mirror V2 Deflects the beam in the horizontal plane</b>	<b>16.7</b>	<b>90</b>	<b>30x150</b>	<b>Plan C @ 15°</b>
<b>Spherical mirror  +</b>	<b>19.7</b>	<b>180</b>	<b>15x40</b>	<b>Spherical Radius = 9.5 m</b>
<b>Large Glancing angle monochromator</b>		<b>0</b>	<b>100x30</b>	<b>VLS 30°</b>
<b>Refocusing mirror</b>	<b>25</b>	<b>180</b>	<b>30x150</b>	<b>Bendable mirror</b>
<b>Sample</b>	<b>30</b>			

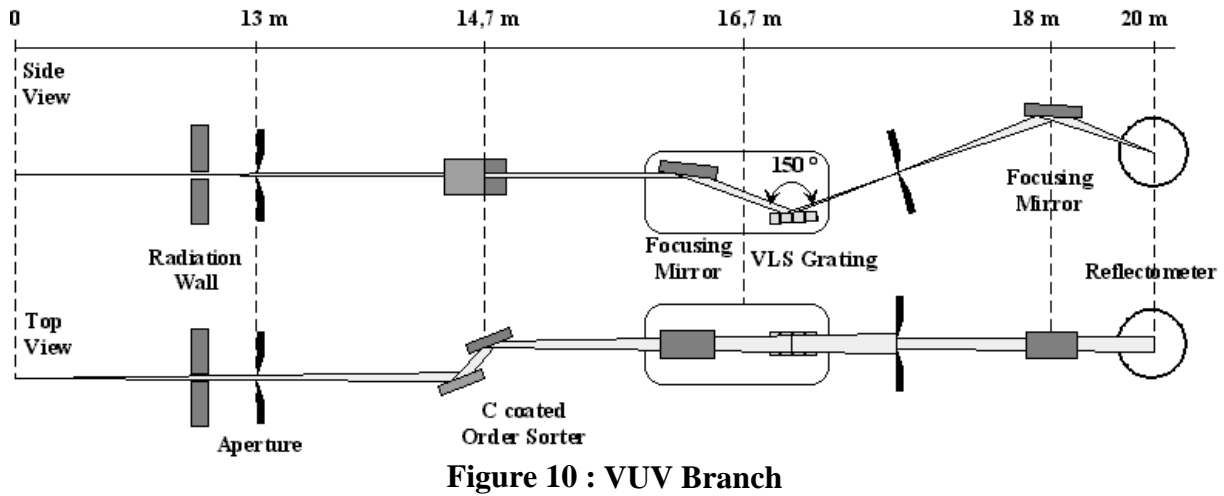
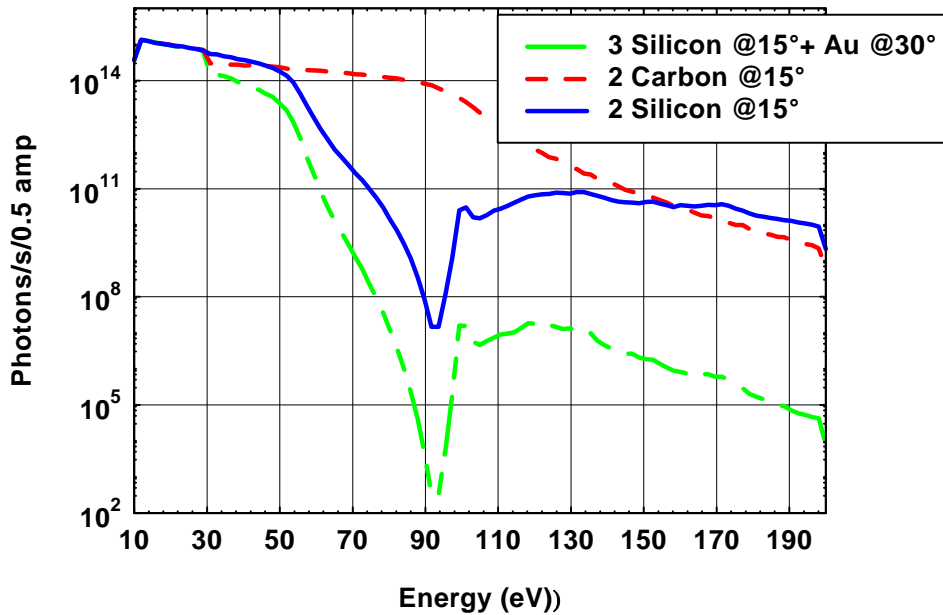


Figure 11 represents the theoretical photon flux with two Carbon or two Silicon coated mirrors (S1 and S2) at 15° grazing angle and assuming that the grating is also a Silicon mirror at 30°.



**Figure 11 : 2 mrad Bending Magnet + Mirrors Reflectivity throughput**

**Of course this design is the first step and needs to be more precisely defined but this shows that three beamlines with acceptable characteristics in terms of accessible energy range, flux, spectral and spatial resolution and harmonic rejection can be designed.**

## **7) Conclusion.**

The example of the USA and Germany, who dedicate a significant part of the available devices on their radiation synchrotron sources to the metrology and calibration of optics and detectors, shows the need for supporting a metrology beamline in the future French synchrotron radiation facility SOLEIL.

The Groups POMADE (Pole of Metrology Applied to DEtecteurs) and PRaXO (Pole of optics X-rays of Orsay) work already to take part in the creation of this project devoted to calibration. Several laboratories of the Bureau National de Métrologie (BNM) are concerned with such a project.

Given the characteristics of the desired beamlines, it does not seem too constraining to develop a beamline adapted to both instrumental studies and metrology. The latter should at least ensure, and extend if possible, the possibilities currently assured on the beamlines of LURE. As demonstrated, the experience gained at the optical test beamline of the ESRF had evidently a strong impact on the progress of specific instrumentation and the qualified technical and scientific support to synchrotron radiation research. Similar to the success obtained in the high energy X-ray range from the optics beamline at ESRF, one can expect correspondingly positive results of the activities that could be pursued at a dedicated beamline for VUV radiation and soft X-rays.

## **Possible Referees**

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## **Acknowledgments**

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# ANNEXES

## **Constraints on the operating mode of the ring (see BNM contribution in annex)**

The characterization of the RS as "standard source" requires an operation with very few electrons, even a single one. This is in opposition with the normal operating mode of the synchrotron. Therefore, it will be necessary to grant the BNM with special low current beamtime for absolute calibration experiment. This constraint is accepted at BESSY II for the PTB.

### **a) Electrons energy measurement**

This information is of general interest for the users and the designers of the machine. For this measurement, two techniques are used by the PTB, the Compton back scattering and the resonant spin depolarization. The comparison of the results obtained by these two methods shows an agreement within  $2 \times 10^{-4}$  taking into account the combined relative uncertainties.

#### **a.1 Compton Back Scattering**

The Compton Back Scattering photons produced by the collision of a CO<sub>2</sub> laser beam with the electron beam are measured with a GeHP detector calibrated in energy. (This method is in addition used for the production of intense gamma radiation). Compton back scattering of laser photons is used as a diagnostic tool for:

- electron energy,
- beam polarization,
- beam émittance.

#### **a.2 Resonant spin depolarization**

This alternative method presents the disadvantage of requiring electrons with polarized spin. The assembly of polarization in the stored electron beam takes a considerable time, strongly depending on the energy of the electrons and the radius of curvature (for example, the duration of polarization at BESSY I at 800 MeV was 3 hours, and much more when the ring was working at 340 MeV for the needs of the PTB for calibration).

**The machine Group has planned anyway to set up this experiment to measure the electron beam energy of SOLEIL for his own research on the machine and these dedicated machine shifts could be shared wit the BNM users.**

## Contributions

- **LURE/SOLEIL Optics Group** 25
- **The Bureau National de Métrologie (BNM)** 28
- **Laboratoire de Chimie Physique Université Pierre et Marie CURIE** 31
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- **CESR (Centre d'Etudes Spatiales des Rayonnements)** 48
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Groupe Optique

## Metrology beamline for SOLEIL

SOLEIL Optics group : F.Polack, M.Idir, B. Lagarde, M. Thomasset

### **Introduction**

From the start of the SOLEIL project during the APD phase, a very strong commitment has been made that, in order to set up high performances and reliable beamlines, optical metrology should be developed **as the first SOLEIL beamline**. Because synchrotron optical elements are enclosed in vacuum vessels and become inaccessible when they are set into operation, a general agreement is made that **no element should be mounted on a beamline before complete characterization**. Characterization means are of two kinds:

- Laboratory controls that do not require VUV or X-ray beams.
- At wavelength characterizations, such as reflectivity and diffraction efficiency or diffuse scattering measurements that are best done on a synchrotron radiation beamline.

Both kinds of metrology must be easily available in the SOLEIL facility, because one cannot only rely on the manufacturers' qualification tests and, of course, because the desired characterization cannot easily be subcontracted.

Therefore means for both at wavelength and laboratory optical metrology have been included in the SOLEIL APD report of June 99. The volume of tests to be carried on this beamline should decrease with time when most of the beamlines will be installed, and it was clearly stated that the beamline would evolve as a general purpose metrology beamline.

### **At wavelength control of synchrotron optics**

Three large classes of problems need to be studied at VUV or X-ray wavelengths: reflectivity measurements or gratings diffraction efficiency, scattering measurements and performance test of optical components. The use of X-ray beams for the characterization of optical surface shapes is also considered.

### **Reflectometry**

Optical coatings for X-ray optics are getting often more sophisticated than a single layer of gold or platinum. Besides multilayer interferential mirrors which are often too selective for beamline optics, graded multilayers (also called super-mirrors) and other multi-element coatings can be used to extend the angle domain of total external reflection. The agreement of the real performances with theoretical calculations must be controlled before setting these mirrors into operation.

More difficult even to predict is the diffraction efficiency of the gratings of the VUV and soft X-ray optics. Diffraction efficiencies depend on the shape of the groove profiles, which can be locally measured with an AFM. However, it is difficult to accurately measure absolute relief depth of several tens of Angstroms and to reliably predict the efficiency with computer code from such data. Reflectometry on the contrary gives directly optically meaningful data. However, even measurements made at a different wavelength from the working one can yield valuable information on the grating profile.

A reflectometer is the basic instrument of optical characterization. A simple 2-circle

instrument is enough to perform most of the required characterization tasks. The chamber must be large enough to accommodate real synchrotron optics – 300 mm long at least. This basic instrument is already available from LURE Super-ACO. Extra movements could be added to the sample and detector stages to perform more specialized measurements.

High energy resolution is not required for reflectivity measurements. However, it is of primary importance that the beamline should be equipped with a tunable multiple mirrors low-pass filter to reduce high order harmonics. **Let us also recall that the full range of wavelengths available at SOLEIL should be obviously also available on the optics and metrology beamline.**

### **Scattering measurements**

Due to the very short wavelength and the grazing incidence of X-ray optics, even a very small roughness of the surface of optical component can produce an extended halo of scattered light around the image of a source. This is especially important in the monochromators because it limits the bandpass of gratings toward high energies to wavelength which are diffracted far enough from the tail of scattered light of the zero order. It should therefore be checked before mounting the optical elements on a beamline, and, despite that the fact geometrical roughness can be determined by other instruments, at wavelength measurement are more than useful.

Scattering measurements need some special properties of the beam. Very clean beam must be delivered to the experiment. The beam needs to be precisely focused on the detector, for detection of very small angle scattering and moreover variable focusing is required to allow the test of curved optics. Let us remark that at lower energy such a beam will be almost completely coherent, at least in one direction.

### **Test of optical components**

Many beamlines of SOLEIL will require to focus a micro spot of X-rays on the sample. The refocusing stages of the beamline optics will therefore have very tight fabrication and alignment tolerances. It is foreseen that these optical components will be made with bendable mirrors and will allow a full control of incidence angles, radius of curvature and perhaps asphericity of the individual surfaces. Such a complicated system cannot be delivered to the users without a thorough characterization. Moreover alignment tools will be permanently associated with this kind of optics to supplement or assist the user for the focusing task. These alignment tools will be based on Shack-Hartman devices. Determination of the feedback parameters of the system and its commissioning will require beam time before the destination beamline is ready.

More generally, the optics and metrology beamline is expected to provide an easy beam access to develop optical subsystems, mirrors, zone-plates and detectors, prior to their installation on a beamline

### **Surface shape measurements**

Though the characterization will be mainly done with off-beam instrumentation (LTP or interferometers), one may think to use a collimated hard x-ray beam for surface shape profiling, as successfully done at ESRF. The idea is close to the Hartmann test : a narrow pencil of X-Rays is not widened by diffraction along the propagation; hence, the centroid of this beam can be accurately measured. In practice, a sub-aperture is scanned in front of the mirror. The position of the centroid of the reflected beam is measured for each aperture position and the surface shape deduced from this measurement.

With respect to the conventional optical profiler (LTP) the advantage is that the measurement can be done in vacuum, for instance in the chamber of an ion milling or sputtering machine, which will realize a local shape correction of the surface.

### **Summary**

The construction of a high performance beamline for synchrotron radiation requires that a beamline with modest performances in terms of resolution and flux is easily available to carry out a variety of optical characterizations. A reflectometer workstation is the minimum required equipment.

In soft X-rays, the beam will be coherent in one direction. This will open interesting possibilities for new optical experiments.



## **Bureau National de Métrologie (BNM)**

### **The Bureau National de Métrologie and the metrology beamline at SOLEIL**

The Bureau National de Métrologie (BNM) is a National Metrology Institute (NMI), *i.e.* the organization designated by the French government as responsible for the development and maintenance of national and primary standards, and transfer methods necessary to give users access to the metrological references they need.

BNM also assures the traceability of industries and users to the SI system by the realization of specific instrumentation and calibration facilities.

BNM represents France in the international metrological authorities such as :

- the Conférence Générale des Poids et Mesures from which depends the Bureau International des Poids et Mesures (BIPM) in the frame of the Metre Convention,
- or EUROMET (Western Europe organization) which objective is to promote the co-ordination of metrological activities and services with the purpose of achieving higher efficiency.

BNM and thirty-seven other NMIs signed a Mutual Recognition Arrangement (MRA) for national measurement standards and for calibration and measurement certificates issued by NMIs. This MRA is a response to an increasing need for an open, transparent comprehensive scheme to give users reliable quantitative information on the comparability of national metrology services: it also provides the technical basis for wider agreements negotiated for international trade, commerce and regulations. International comparisons are the foundations of this MRA in which all the NMI signers are participating.

BNM is a “Groupe d’Intérêts Publics” and consists of a core of laboratories split in two categories

- National Metrology Laboratories (NMLs)
- Associated laboratories (ALs).

There are four NMLs (BNM-INM, BNM-LNE, BNM-LNHB, BNM-LPTF) and eight ALs. For BNM, the fields involved in this range are X-rays and UV radiometry and the first three NMLs are working in these fields.

### **Interest of a metrology beamline at SOLEIL**

In the frame of the synchrotron radiation and of the relevant metrological areas (X-Rays-UV radiometry), different elements argue for a metrology beamline at SOLEIL :

- First of all, there is no such structure in France in the energy range of interest, and more generally, there are no national standards in the VUV-soft X rays energy range (10 eV – 1 keV) ;

- Second, at the European level, only Germany has an integrated facility (synchrotron radiation – metrology beamlines) on BESSY II storage ring. The goal is not to copy or rebuild what is done elsewhere, but to apply one of the basic principles of metrology that is to have redundant means to try to realize the SI fundamental units ;
- Then, this approach could facilitate collaborations and international comparisons between SOLEIL and BESSY in the common energy range of both facilities ;
- Last, in the very last years, the utilization of both UV and X-rays dramatically increased due to the need of such type of radiation in different applications such as industry, medicine and environment. In these spectral ranges, measurements are delicate and difficult and the radiometrical scales maintained by the national laboratories are no longer adapted to the users requirements and must be developed according to new methods.

The traditional activity of the BNM in the radiation’s measurement field includes the spectral range 200-2500 nm for the visible, UV and IR radiation and 1-10 keV for the X-ray range. With a metrology beamline at SOLEIL, we could develop more specifically the spectral ranges 150-400 nm for the UV and a few tens of eV – 10 keV for the soft X-rays, thus including the actually maintained spectral ranges and developing new metrological means for the intermediate energy range.

### **Use of the synchrotron radiation for fundamental metrology**

Radiation measurements in the metrological field includes two major tasks : realization of primary standard sources and detectors, and transfer to secondary sources and detectors.

#### **Primary standard source**

The synchrotron radiation emitted by storage rings from the magnets is a primary standard source, because the photon flux is calculable from its physical parameters such as magnetic field, energy and intensity of the electron beam : these quantities can be linked to other primary standards in the electrical or dimensional ranges. National laboratories such as PTB (Germany), NIST(USA) already use the synchrotron radiation for calibration of sources and detectors. Thus the metrology beamline of SOLEIL could become the **national primary standard source**. This necessitates accurate measurement of the beam parameters and a special running mode with few electrons in the storage ring to complete the calibration procedure.

#### ***Transfer standard sources***

In a second stage, the calibration of a transfer source (deuterium lamp, X-ray tube, etc.) relative to the synchrotron beam needs to send successively on a “detector” the calculable radiation directly emitted (without any optics) by the synchrotron and the radiation emitted by the source to be calibrated absolutely in the same conditions. The “detector” generally consists of a monochromator linked to a photodetector adapted to the specific energy range. The detector global transfer response must be the same between the synchrotron beam and the source to be calibrated ; thus linearity and stability of the photodetector are essential.

### *Calibration of detectors*

“A measurement is a comparison”, thus as it is easier to compare two objects of the same nature, it is easier to calibrate a transfer detector to a reference detector. The detector calibration is thus obtained by comparing their responses when they receive the same monochromatic radiation for each wavelength. Generally, the cryogenic radiometer is used as the primary reference detector for metrology : the detector heating due to the photon flux is compared to an electrical induced heating. For such calibration, the geometrical spread is an important parameter of the source, as the same beam must be “seen” by both detectors ; moreover, a continuous spectrum with great brilliance is of interest to accurately select a monochromatic energy : this suppose to use monochromators to obtain high spectral purity. As the comparison is carried out after the dispersive elements, the different optics used to prepare the beam will not affect the measurement results.

For these experiments a total of 10-15 shifts per year are necessary.

<i>Parameter</i>	<i>1 eV</i>	<i>5 keV</i>
<i>Electron Energy</i> $W = (850.0 \pm 0.1) \text{ MeV}$	<i>0.0008 %</i>	<i>0,16 %</i>
<i>Magnetic field</i> $B = (1.05926 \pm 0.0002) \text{ T}$	<i>0.0008 %</i>	<i>0.08 %</i>
<i>Beam Current</i> $I = (10.000 \pm 0.005) \text{ mA}$	<i>0.05 %</i>	<i>0.05 %</i>
<i>Source size</i> $\Sigma = (33.5 \pm 7.5) \mu\text{rad}$	<i>0.002 %</i>	<i>0.25 %</i>
<i>Distance</i> $D = (6\,468 \pm 5) \text{ mm}$	<i>0.15 %</i>	<i>0.09 %</i>
<i>Collimators - Slits</i> $R = (2.501 \pm 0.001) \text{ mm}$	<i>0.08 %</i>	<i>0.05 %</i>
<i>Angle</i> $\Psi = (0 \pm 15) \mu\text{rad}$	<i>0.001 %</i>	<i>0.00 %</i>
<i>Total Uncertainty</i> <i>(quadratic sum)</i>	<i>0.18 %</i>	<i>0.35 %</i>

***Uncertainty Contribution on the total spectral photons flux  
of different useful parameters versus photon energy.***

The « Groupe Optique et Source X : GOSX » (in the Laboratoire de Chimie Physique – Matière et Rayonnement of the Université Pierre et Marie Curie) works in the field of conception, modeling and characterization of optical devices and sources for the X-UV and soft-x-ray domain. The group has fabricated several reflectometers [1] designed for the metrology of x-ray optical components (multilayer mirrors, multilayer gratings, multilayer monochromators, Fresnel lenses, ...). GOSX collaborates closely with the staff SDE of the CEA-DAM Bruyères-le-Châtel for metrology, using the facilities of the beamlines SA23 and SB3 of Super-ACO (LURE). During the past decade, the main subject of interest was the fabrication of multilayer monochromators with improved performances, based on etched multilayer mirrors [2, 3]. The use of the synchrotron radiation has played a crucial role in the development of these improved multilayer monochromators [4, 5]. GOSX has also extensively used the synchrotron radiation for measurements of optical constants in the x-ray range [6] and the calibration of new kinds of monochromators and spectrometers, for instance monochromators using multilayer transmission plate, multilayer mirrors with graded period [7, 8]. The activity of GOSX requires a synchrotron beamline dedicated to the metrology, covering the spectral domain 50 eV – 10 keV with a moderate resolution ( $\Delta E/E$  around  $10^{-3}$ ) but a high spectral purity (not less than 99 %). A reflectometer is also needed for the metrology of optical devices.

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**Laboratoire Charles Fabry de l'Institut d'Optique  
"Thin films for X-Ray optics"**

**1- Fields of research on XUV optics at IOTA**

For more than fifteen years, the Institut d'Optique Théorique et Appliquée (IOTA) has acquired an experience in the design and manufacturing of optics for the 10nm-40nm wavelength range. The steps needed to make such optics include polishing, ion etching of mirrors followed by the deposition of thin films and multilayer coatings onto so obtained flat, spherical or aspherized surfaces. In the past one of the most noticeable realizations of IOTA was the Extreme UV Imager Telescope of the SOHO mission (NASA-launched in 1995). This telescope consists of a primary mirror and a secondary mirror coated with multilayers on each of four selection quadrants. Images of the sun for the four wavelengths 17.1 nm, 19.5 nm, 28.4 nm and 30.4 nm delivered by the telescope have been largely shown among the scientific community as well in the public field. At that time, the calibration of the tests, witness samples and telescope were achieved on the IAS/LURE beam line at SuperACO. Today, the Laboratoire Charles Fabry de l'Institut d'Optique (LCFIO) is involved in other international programs in astrophysics. The current one is the STEREO mission (NASA) that requires two identical Extreme UV Imagers for the 3D observation of the solar corona (launch due in 2004).

*In another area of interest, LCFIO develops new kinds of optics to be used with the new X-ray sources like synchrotron, X-ray laser, plasma laser, harmonics generation. So, X-ray laser cavities, mirrors, monochromators, are being currently fabricated in the frame of cooperation with the research teams that develop and use these sources. X-UV interferometry experiments are particularly demanding dedicated optics.*

For all these applications in astrophysics and physics, the fundamental issue of the LCFIO lies in the design of original combinations of materials adequate for the low energy X-UV radiation. More specially, the study of light materials systems like B/Si, B<sub>4</sub>C/Si, Mg<sub>2</sub>Si/B, Mg<sub>2</sub>Si/B<sub>4</sub>C for which the absorption is lower in the energy region <50 eV allowed to obtain narrower band pass than for the classical system Mo/Si, with comparable reflectivities.

More specific optics have been also successfully made at LCFIO during the past two years: a Kirkpatrick-Baez optics which involves depositing an aperiodic sequence of multilayer, narrow band pass monochromators obtained by etching linear gratings into the multilayer stacks and also beamsplitters for the interferometry at 13.9 nm consisting of depositing multilayer on both sides of a transparent membrane.

Basically, these fundamental achievements require to get a feedback coming from the measurements of the multilayers reflectivities in order to improve the deposition parameters and fabrication conditions.

So far, the measurements of the reflectometry could be performed at IOTA on a home built reflectometer working with hard X-rays delivered by a classical Cu K $\alpha$  source. The layer thickness and interfacial roughness optical constants can be deduced from these experiments. However, measurements of the reflectivity at the used wavelength of the optics are absolutely

mandatory in order to get the optical indices of the materials at these photon energies and also to predict the performance of the optics in the conditions of their use. For that, measurements could be done every year on SA23, SB3 and SA62 beam lines on the SuperACO storage ring. Reflectometers implemented by CEA/DAM, IAS and LURE respectively could be used in the frame of scientific cooperation. However a main drawback comes from the fact that the access to an adequate synchrotron beam line is submitted to schedules which are not always tuned to the manufacturing schedules of a given kind of multilayer optics.

## **2- Prospects and needs for a Metrology beam line at SOLEIL**

In the future, the research program on the X-UV optics at IOTA will be enhanced. The reasons for this come from the fact that the French community working on X-ray sources and X-UV optics has structured his network around the SAXO GDR on a national area and also around the PRaXO group on the Campus of Orsay. The needs in X-UV optics in France and also on a European area have been evaluated and in that context, the LCFIO has been considered as a main provider for this community. In that aim, the PRaXO group has decided to create a “*Center for deposition and calibration of multilayers for X-ray optics*” located at IOTA. This project has been evaluated successfully enabling the PRaXO group to win funding from national and regional agencies to start to acquire deposition and calibration equipment. So, in 2002, a new deposition computer controlled sputtering machine will be implemented at IOTA which will be dedicated to deposition of multilayers on a routine mode.

In addition, the PRaXO group has been recently involved in the huge European program on the EUV Lithography. The concerns of the LCFIO in that European network lies in the deposition of new combinations of materials, multicomponents stacks, study of buffer and capper layers in the aim to obtain optics with a high level of thermal and time stability.

On another hand, an increasing interest of astronomy has recently appeared for the spectral bandwidth ranging between visible and extreme ultra-violet (EUV), named vacuum ultra-violet (VUV) or far ultra-violet (FUV), between 80 and 120 nm. The goal is to make optics in order to study the radiation emitted by hot plasmas in interstellar medium. This domain was not so much studied because most of materials are absorbing for these energies, more than for EUV or XUV. Only aluminum can give a noticeable reflectivity under normal incidence at 90 nm. Nevertheless, it needs to be protected against oxidization by one or more capper materials that do not reduce drastically the reflectivity of the stack. So a first research consists to reach optical indices of materials in the literature, or to measure them. After deposition, multilayers should be tested by reflectometry at these wavelengths. Presently, it is possible to make reflectivity measurements between 20 nm and 160 nm at LURE, in the IAS station. The incident beam comes from a platinum etched monochromator (1200 lines/mm), and the reflectometer works as the XUV one. This spectral bandwidth between 20 and 160 nm (7 eV-65 eV), not so much studied until now has to be reached in the future metrology beamline, with the possibility of goniometric reflectivity measurements ( $\theta$ - $2\theta$ ) ( $\lambda$  fixed), as well as for reflectivity versus wavelength measurement ( $\theta$  fixed).

**In conclusion**, for all the issues described above, the need of **an access to a Metrology beam line on the SOLEIL machine will be crucial for LCFIO in the future.**

The main requirements for this beam line regarding the LCFIO fields of interest are:

- Energy range: 10 eV to 10 keV
- A low level of harmonics in the aim of absolute measurements of the reflectivity
- A high precision in the definition of the energy
- The ability to scan energies for glancing angles ranging from 0 to 90°, and also to scan glancing angles for well-defined energies.

- A clean environment because X-UV optics are very sensitive to the surface contaminants like water or hydrocarbons, dust, etc...
- A high flexibility in the access to the beam line in order to achieve the calibration in parallel with the development of the coating processes.

**X-UV and X-ray sources at SPAM laboratory**  
**(CEA Saclay)**

H. Merdji, B. Carré, P. Salières

Most of the programs carried out at the Service des Photons Atomes et Molécules (SPAM) at Saclay involve radiation-matter interaction and consequently rely on photon sources. The development of these sources bring a strong need for accurate calibration and standards facilities, in particular for measuring reflectivity of mirrors and multilayer coatings, transmission of thin films, bandpass of multilayers, efficiency of gratings or detectors, etc. Home calibration has already been accomplished however these measurements are spectrally limited and require more stability.

1. **Production of coherent XUV light by harmonic generation and applications.**

The team maintains expertise in both theoretical and experimental investigation of far UV to soft X-ray (XUV) production through harmonic generation. With high order harmonic pulses intensities in the  $10^{10} - 10^{12}$  W/cm<sup>2</sup> range have been obtained below 20 nm. The high order harmonic pulses are also highly coherent and ultrashort: pulses of a few tens of femtosecond are now typical and are extending now to the attosecond range. Applications of this ultra-short coherent XUV light are developed in plasma physics (X-UV interferometry) and solid state physics (photoelectron spectroscopy) [1-4]. Each type of study needs efficient XUV optics capable of reflecting, selecting and focusing the light without degrading its properties. However calibration facilities covering low harmonic orders (typically between 20 nm and 100 nm) do not exist. Therefore, we have also used harmonics to qualify XUV devices such as filters, gratings, multilayer mirror or Bragg-Fresnel lens [5-7]. However, these measurements are spectrally limited and require more stability. There is also a strong need of a standard X-UV calibration facility for the measurement of the absolute quantum efficiency of our detectors (X-UV photodiodes and CCD cameras).

2. **Clusters in strong laser field.**

In the case of large size clusters, production of XUV light, ejection of highly charged energetic ions are studied with both fundamental and applied purposes [8-10].

The production of incoherent ultra-short X-rays in the keV range has shown to be efficient. We are currently studying the fundamental aspects in order to optimize the photon flux. This source will be used in 2002 for spectroscopic applications. In such spectroscopic applications, one needs to focus on a tiny spot a narrow spectral band. Schwarzschild systems and focusing spectrograph are envisaged. These devices need to be calibrated both spatially and spectrally before being used.

SPAM has been part of a national R&D project called PREUVE that aims at developing the future technology for nanolithography processing. SPAM efforts concentrate on developing an intense Extreme UV light source at 13nm based on laser-clusters interaction. It will be inserted in a EUV lithography test bench that is developed in parallel by other partners. This bench will allow for experimental studies on EUV lithography and photo-resist processes. During the last two years, the characterization of optical elements such as mirrors, thin filters and photodiodes has been essentially made at the PTB (BESSY2). Concerning the MoSi mirrors, the PTB has realized absolute scan as a function of the wavelength, the incident angle, and spatial scan at a fixed wavelength and angle in order to characterize the surface. The absolute spectral response of our photodiode has been also measured. In the near future

we plan to ask for the characterization of transmission gratings. The development of a beamline adapted to metrology at 13.5 nm is essential in the framework of EUV lithography MEDEA+ program.

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GDR 1851 du CNRS  
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et leurs Applications**

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**SAXO support the “metrology” beamlines at SOLEIL**

The french research pool (GDR<sup>1</sup>: groupement de recherche) SAXO « Nouvelles sources de rayons X et leurs applications » (New X-rays sources and their applications) regroups seventeen laboratories<sup>2</sup> from the CNRS, Universities, CEA, ONERA and Industrials partners, THALES and INEL. This GDR is dedicated to the study and development of new systems, including sources, optics and diagnostics, for production or applications of photons with energy ranging in a wide domain extending from tens of eV to tens or hundreds of keV, XUV to hard X-rays.

Research is done along three main directions:

- \* Soft and hard X-ray systems and their applications
- \* XUV systems and their applications
- \* Theoretical and numerical developments for the optimization of X-ray systems

The two first topics are greatly concerned by the development of a specific SOLEIL beamline dedicated to XUV-X ray metrology. The needs from that community are important and will continue to grow in the coming years. Beside the synchrotron sources, coherent or incoherent laboratory or industrial sources of energetic photons are more and more used in research and industry. Everything in research or new technologies tends to push to the use or development of very small scale devices. Diagnostics and characterizations using low wavelength photons are expending rapidly. The best example today is the race all around the world for the development of the next generation lithography machines including new sources around 13.5 nm and associated optics, based on Mo/Si multilayers mirrors, for the illumination of the mask and the wafer for production of patterns with size below 50 nm.

The needs of SAXO group concern

- calibration of detectors
- characterization and metrology of optics and optic systems
- development, characterization and calibration of new optics and detectors

The energy range proposed for the “metrology” beamline at SOLEIL, from 5 eV to 10 keV (with possible extension to 20 keV ) clearly covers most of these needs.

### ***Photons sources***

It is interesting to present the variety of photons sources studied or developed by the SAXO teams. In the XUV domain, photons are mainly produced by laser plasma on gas or solid targets, interaction of laser with clusters beam or liquid jet, high order harmonic generation, surface discharge, capillary discharges and Z-pinch discharges. In the X-ray domain, we found again laser plasma on gas or solid targets, interaction of laser with clusters beam. Discharges are quite different; they concern ECR (Electron Cyclotron Resonance) discharge and High and very high voltage direct discharges.

### ***Detectors calibration***

It is clear that the characteristics of all these various sources are quite different concerning source size, geometry, produced photon flux and temporal behavior. Comparison are sometime quite difficult and there is a real need, specially due to the increasing links with industry, for measurements in good conditions of absolute characteristics. This directly implies absolute calibration of detectors in the wavelength domain concerned. It also implies the study of response to short (few ns) to ultra short (few ps) pulses from conventional detectors and response versus dose rate. It includes calibration of scintillators, imaging systems, CCDs, Photodiodes, ...

### ***Optics***

For optics and optical systems, while not excluding the 2-20 keV domain, SAXO is mainly interested to have the possibility of characterization and metrology in the XUV range. There is a tremendous development of new coherent X-ray lasers and incoherent sources in that domain and a parallel growth in applications. This is accompanied by huge developments in XUV optics and imaging systems for XUV photon beam shaping. Measurements of mirrors reflectivity, surface roughness, control of surface, characterization of new materials, transmission or focusing optics of small size, are of great importance to determine photon flux or beam shape on target at specific wavelengths.

Beam shaping generally involves complicated geometry multi-optics systems like in EUV lithography. Taking into account reflectivity of single elements, fluxes can be reduced by orders of magnitude from source to target. So, very precise measurements are needed for development and fabrication of research or industrial tools. This, like absolute calibration of detectors, can be done properly only on a dedicated synchrotron beam. The energy domain proposed for the metrology line at SOLEIL is perfectly adapted for our needs. Expected fluxes after the three beamline monochromators will be largely enough for any kind of measurements.

### ***New optics and Detectors***

Last but not least point is the testing and characterization of new optics and detectors over a wide range of energy and the study of their efficiency and response for very different dose rates. Progress in material science, deposition processes, multilayer device or micro- and

nanotechnologies, together with the demand of efficient system at short and shorter wavelength push the development of completely new kinds of products (diamond detectors, scintillators, gas detectors, guided solid detectors, zone plate, new multilayer optics, ...) that must be clearly evaluated for use mostly, in this case, in research. Different teams in SAXO are developing new devices as mentioned above or have collaborations with other groups doing so, for systems concerned by the whole energy range expected at the metrology line at SOLEIL and above.

The GDR "SAXO" strongly supports the development of a dedicated beamline for metrology at SOLEIL. Such beamline will be of crucial importance for the research in the field of new XUV and X-ray systems in the near future. The activity in that field is considerably increasing with strong links with industry. Development of EUV lithography, MEMS, soft X-ray and hard X-ray diagnostics of small size systems with laboratory sources, ... drain a lot of efforts. This must be accompanied by efficient tools as a SOLEIL metrology beamline for absolute measurements and calibrations of sources and optical systems.

*1- a GDR is an official structure of CNRS*

*2- laboratories and companies involved in SAXO:*

*CEA-DAM/DRIF (Bruyères), CEA-DRECAM/SPAM (Saclay), CELIA (Bordeaux), DARC (meudon), GPS-PIIM (Paris), GREMI (Orléans), INEL (Artenay), IOTA (Orsay), LP3 (Marseille), LAC (Orsay), LCP (Paris), LOA (Palaiseau), LPIIM (Marseille), LPTP (Palaiseau), LSAI (Orsay), LULI (Palaiseau), ONERA/DMP (Palaiseau), RSRM (Grenoble), THALES/TTE (Thonon)*

## Calibration of X-ray detectors for Astrophysics

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Experience has been acquired by the French X-ray astrophysics groups with the calibration of the EPIC cameras\* (which have been installed on board the XMM-NEWTON satellite) in the IAS laboratory, using SACO and DCI derived beamlines. The following discussion is based on this experience.

First, let us stress the importance of having at our disposal a dedicated calibration beamline (the LURE derived beamlines for the EPIC calibration were unique in Europe) for the upcoming missions\*\*. XEUS is the most outstanding mission, planned by ESA, to be launched after 2010. It will consist of a mirror spacecraft flying in association with a refurbishable detector spacecraft, with each version of the detector spacecraft needing its own calibration. Additionally, many mini or medium size missions are presently proposed to the CNES (Simbol-X, for instance) and to the other European space agencies, which will carry an X-ray telescope as part or totality of their payload.

The performance of the next generation of instruments is expected to improve largely upon present X-ray CCD performance in terms of quantum efficiency, energy range, spectral resolution and count rate by relying on newly designed X-ray CCD, Superconducting tunneling junction or microbolometer based instruments.

On the other hand, X-ray astronomy can be performed only outside the atmosphere and satellite experiments have specific needs. They have to be once fully and successfully calibrated on ground as no further full calibration can be performed once they have been delivered to be installed on the spacecraft. In general a calibration window is imposed and the duration of this calibration is reduced as schedules are and will remain inevitably very tight in this field. Finally, there are specific and very stringent spatial quality requirements in terms of cleanliness, absence of pollution and safety (a protection of the instruments against over-exposures to X-ray, irreversible pollution and vacuum failures which would destroy very expensive hardware is needed).

### **These needs impose the following requirements:**

A large vacuum chamber to install the instrument subsystem. This vacuum chamber will be located inside or accessed by a clean room (class 100). This vacuum chamber has to be free of pollution (this need imposes requirements on the type of pumping system, on the kind of materials allowed inside the vacuum and on the presence of large cryogenic traps). The Jupiter tank (in the IAS laboratory), which was used for the EPIC calibration, already achieves most of these requirements.

The limited time allowed for calibration, the constraints of the ground support equipment which will involve cryogenics at very low temperature (lower than 100 mK) and the necessity to handle, as few as possible, fragile instruments impose the use of only one beamline to cover the full energy range.

The resetting of the beamline parameters (energy and flux) has to be the least time consuming possible, as time lost for this tuning will not be available for calibration.

Relative and absolute measurements will be performed, requiring the presence of absolute calibration detectors covering the full energy and flux range.

The flux required to calibrate such detectors is usually very low and then difficult to measure, thus the need for fast mechanical choppers is important.

With the advent of new multilayer mirror telescopes with increased focal distances the useful energy range will extend from 100 eV to 30 keV and perhaps more (bear in mind that only a low flux is required at all energies, orders of magnitude lower than the fluxes required by the usual synchrotron radiation user).

The spectral purity has to be better than 1% and with no visible or near infrared contamination as X-ray detectors are visible detectors as well.

The energy spread should be better than a tenth of an eV to fit the sub-eV energy resolution of the next generation detector, at least under 2 keV. This requirement can be relaxed a little at higher energies. The mean energy has to be precisely defined and reproducible to allow the study of edges which could be present in some detectors. Among these, the carbon edge is important, the study of which requires a beamline as free of carbon pollution as possible. The availability of an in situ decontamination of the sensitive optical elements would be of value to avoid a painstaking dismounting, cleaning, reassembling and retuning of the beamline before such measurements.

To calibrate imaging detectors, an homogeneous and well measured X-ray illumination over the full instrument area is required. For the EPIC calibration\*\*\*, these conditions were obtained by defining a narrow linear beam with a slit and translating, in front of it, the focal plane. This solution was very slow, cumbersome and presented many unpleasant side effects. An homogeneous beam covering the full focal plane is by far the best solution (even if very difficult to implement) and should be available at a calibration beamline. Ideally, the diameter of such a beam should extend up to 20 cm to cover the foreseeable focal planes. Such a beam doesn't need to be exactly parallel.

To comply with the different possible observing modes, such as the full frame exposures of CCDs, it is necessary to dispose of a fast neutral shutter (ensuring uniform illumination of the full focal plane). This shutter could be placed at the beginning of the line, close to the neutral densities, and they could be used in conjunction to modulate the flux for detector timing mode calibration.

The polarization is not a parameter which is measured by the present X-ray missions. With the advent of polarization sensitive X-ray detectors, this situation could change in the future and the availability of a well defined linearly polarized beam could become necessary.

The calibration data will be archived and processed off-line, on some occasions a long time after they have been taken. Only a check is performed on-line. It is important to save, along with the detector data, all the relevant information on the beamline parameters (from the beamline optical elements, from the synchrotron itself and from the absolute calibration and monitoring detectors) in an unambiguous way, using an immediate, efficient, fast and reliable informatic connexion.

To conclude, let us say that a beamline suited to the calibration of an X-ray spectro-imager is a complex beamline. Even a limited unreliability of some beam building elements can have important consequences as these unreliabilities will add up to the point where the resulting beam will be hardly available and all the efforts invested to calibrate a flight-model in its calibration window (we stress the fact that this window will always be reduced and imposed) will be lost.

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Proceedings of the SPIE Conference on EUV, X-Ray and Gamma-Ray Instrumentation for  
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# Metrology of X-UV laser produced plasma diagnostics with synchrotron radiation.

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## **Introduction:**

Within the framework of the “Simulation Program” developed by the CEA defense pole (CEA-DAM), the theoretical models of laser-matter interaction and thermonuclear physics will be validated through inertial confinement fusion experiments carried out on the LMJ.

Considering the temperatures and pressures generated during laser-matter interaction, the X-UV radiation is the ideal tool to diagnose plasmas.

The experimental validation of the laser matter interaction models requires absolute measurements with the smallest possible uncertainties. Therefore, a complete metrology of the diagnostics is essential before any experiment.

Because of its temporal stability, strong brightness and broad spectral range (continuously usable from UV to hard X-rays), the synchrotron is an ideal source for metrology.

Since 1985, the CEA defense pole has mainly used the synchrotron radiation produced by SuperACO (LURE, Orsay) for the metrology in the X-UV range [1,2,3].

Our experience in the metrology of optics and detectors has given rise to many collaborations through the use of our experimental station (Laboratoire Primaire des Rayonnements Ionisants, Laboratoire pour l’Utilisation des Lasers Intenses, Laboratoire de Chimie Physique, Laboratoire pour l’Utilisation du Rayonnement Electromagnétique, Laboratoire de Spectroscopie Atomique et Ionique, Institut d’Astrophysique Spatiale,...)[4,5,6,7].

The credibility, development and research of each partner profited from these collaborations.

In 1999, a group of reflection on the X and XUV detectors called POMADE (Pole of Metrology Applied to DEtector) was created under the initiative of three laboratories directly involved : CEA / DIMRI, LURE, CEA / DIF. The main objective of this group is to solve the technological problems related to X-rays detectors, which hamper the scientific community. A collaboration began on the design, realization and metrology of a X-UV bolometric detector in order to improve measurements uncertainties of our calibrations. Moreover, the CEA-DAM also takes part in the PRAXO group (Pole RAYons X d’Orsay) whose goals are similar, but in the X-ray optics field.

***Scientific objectives : Metrology of X-UV diagnostics for laser produced plasma experiments on the SOLEIL metrology beamline.***

Our metrology needs for the coming years concern the X-UV diagnostics of the Laser Integration Line (LIL), prototype of the Laser MégaJoule (LMJ). Later on, these technologies will be transferred on the Laser MégaJoule. These needs will require access to a large spectral field and a high intensity beamline. Due to this project schedule, it is essential for us to perform the metrology of our diagnostics at the start-up of SOLEIL.

The "plasma-laser " characterization experiments in the X-UV range (10 eV-10 keV) require diagnostics with specific instrumentation. The diagnostics that will be set up either on the LIL or the LMJ shall consist of the following parts :

- A collimation and a filtration system which limits the analysis beam size and eliminates the radiation at the undesirable energies.
- One or more optical elements which are used as imager or spectrometer.
- A detector which transforms the X-ray radiation into a visible or electric signal directly exploitable by the physicists.

There are three types of experiments for plasma-laser characterization :

- **Imagery**, using X-ray microscopes whose field is of the order of one mm and resolution of a few microns. These microscopes allow, with a magnification of about 10 to 20, to obtain X-ray images of the hot and dense zones of strongly emissive plasma. The design of these microscopes depends on the spectral band under study.

For photons energies higher than 1 keV, the pinhole cameras, Kirkpatrick-Baez and Wolter microscopes are mainly used [8, 9].

For photons energies lower than 1 keV, the spatial resolution of the pinhole camera is limited by diffraction, so one uses Schwarzschild microscopes similar to those used in the visible range. The mirrors of this device are coated with a metallic multilayer to reflect X-rays under normal incidence.

Recently, it has been possible to combine the Bragg reflectivity of the crystals or multilayer mirrors with the Fresnel diffraction of a grating in the same optical device called "Bragg-Fresnel Lens" [10, 11]. The grating with variable step can be linear (LBFL) to focus the radiation along one dimension or elliptic (LBFE) to focus along both.

- **X-UV Radiography** [12, 13] consists in using an auxiliary radiation source, often created by laser, to radiograph a plasma. Thus we measure the plasma opacity which, associated with numerical simulations and models, allows us to evaluate the temperature and the density of the plasma.
- **X-ray Spectroscopy** is a powerful technique to investigate the plasma characteristics. We use high resolution X-UV spectrometers to measure the emission of plasma characteristic lines. For photons energies higher than 1 keV, we use plane or curved, natural or synthetic crystals [14]. The reticular d-spacing planes of these crystals is close to the analyzed wavelength  
For photons energies lower than 1 keV, these crystals are replaced by etched plane or curved gratings in grazing incidence or by transmission gratings [15].

The design (research and development), exploitation of this instrumentation and validation of the simulation models render the rigorous metrology of the diagnostics essential.

Metrology can be carried out either on the complete measurement device or on some of its parts and, to match its expected working conditions (high intensity plasma), it requires a very intense X-UV radiation source.

In the X-UV range, there is no primary radioactive source which can be used as an absolute reference for metrology. We generally use X-ray tubes which produce X-ray emission lines of

discrete energies depending on the nature of the anode, but the brightness of this type of source is very low (very wide source which emits in a large angular field).

Due to the temporal, high brightness, polarization and spectral characteristics of its radiation, the synchrotron is an ideal source for X-UV metrology.

This metrology must be performed on a specific beamline where it is possible to optimize the intensity, the spectral purity and the size of the analysis beam. It must use reference detectors (SiLi, proportional counter, radiometer, bolometer,...) as standards measurement devices [16,17,18].

In addition to these engineering constraints, it is necessary to add the planning constraints for the "plasma-laser" experiments. Indeed, these will define the planning of when the metrology of the measurement devices must be carried out.

### **Conclusion:**

This document presents the scientific objectives of the metrology of X-ray diagnostics for the realization of "plasma-laser" experiments and specifies the advantages of the synchrotron radiation for their metrology.

Since 1985, the CEA defense pole has completed its requirements in X-UV metrology mainly at the storage ring SUPER ACO at LURE, Orsay.

Taking into account our X-UV metrology needs and those of the scientific community (SOLEIL, LPRI, LULI, LOA, IOTA, LCP, LSAI, IAS, ...) for the future years, it is essential to build a dedicated metrology beamline for optics and detectors in the X-UV range on SOLEIL.

It would be desirable that the means we developed and the competencies we acquired in the framework of the collaboration poles PRAXO and POMADE be used for the design, the realization and the management of this specific metrology beamline.

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# LURE



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## **Needs for the Detector Group on the metrology beamline at SOLEIL**

*Contribution from the LURE detectors group*

A mission of a detector group is to design and realize detectors adapted for the experiments when no commercial device exist. When a commercial realization is available, the detector group has to participate to the choice of a device, verify his performances and test its limits.

Furthermore, beamtime is needed for in-house research and development activity to test prototypes which can become later the heart of future detectors.

The test of detectors involves particularly

- energy response,
- spatial resolution,
- linearity of the spatial localization,
- counting rate linearity,
- image quality obtained with reference samples.

The characteristics of the metrology beam line could then be the following

- variable energy over the same range as used in experiments, i.e. about 100 eV to 25 keV (test of energy response),
- “point” source of about  $50 \times 50 \mu\text{m}^2$  (test of spatial resolution),
- the most uniform possible flux density over a large area (at least  $10 \times 10 \text{cm}^2$ ), (test of spatial linearity),
- variable flux from a few hundreds of photons per second to the maximum accessible flux (test of the counting rate linearity),
- goniometer (test with sample),
  - 2 independent rotations with a vertical axis
  - detector support on a  $2\theta$  arm
    - motorized translations over 2 axis for x,y scan over the detector window
    - motorized translation of detector holder along the  $2\theta$  arm

For information, two detectors for kinetics studies at high X ray flux in diffuse scattering experiments are being realized, one for small angle scattering and the second for

wide angle scattering. These detectors are designed to accept the very high photon flux delivered by a third generation synchrotron source.

More Recent publications

M. Lemonnier, T. Bucaille, J. Charlet, M. Bordessoule, F. Bartol, S. Megtert  
Brevet Français 2 727 525 (25-11-1994)

*Détecteur de rayonnements ionisants à microcompteurs proportionnels*

M. Lemonnier, F. Bartol, M. Bordessoule, G. Chaplier and S. Megtert  
*The C.A.T. pixel proportional gas counter detector"*  
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*Derivation of a simple analytical expression of the gain of pure Argon filled CAT*  
*proportional counters*  
NIM A 440 (2000) p. 466-470



**Centre d'Etudes Spatiales des Rayonnements**

## **A FAST X-RAY TIMING CAPABILITY FOR XEUS**

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### **Science rationale**

The X-rays generated in the inner accretion flows around black holes and neutron stars carry information about regions of the strongly curved space-time in the vicinity of these objects. This is a regime in which there are extreme predictions of general relativity still to be tested. High resolution X-ray spectroscopy and fast timing studies can both be used to diagnose the orbital motion of the accreting matter in the immediate vicinity of the collapsed star, where the effects of strong gravity become important.

The spectroscopic approach is already well covered in the current XEUS detector baseline, but the fast timing one should also be considered. Exciting new opportunities have arisen in the last few years through the discovery with the ROSSI X-ray Timing Explorer of (sub-)millisecond quasi-periodic timing phenomena and brightness oscillations during X-ray bursts in weakly magnetized accreting neutron stars. These discoveries have unambiguously demonstrated that fast X-ray timing provide the potential to accurately measure the motion of matter in strong gravity fields and to constrain masses and radii of neutron stars.

Fast X-ray timing requires extremely good photon statistics. In view of its huge collecting area, XEUS which is foreseen as a potential follow-on to ESA's Cornerstone X-ray Spectroscopy mission (XMM-Newton) could therefore make a major contribution to this field. With a better than one order of magnitude improvement in sensitivity for timing studies, XEUS will enable for the first time the testing of predictions of general relativity in strong gravity fields, such as frame dragging effects and fully relativistic periastron precession. In addition, XEUS will allow, in exquisite detail, the waveform of coherent brightness burst oscillations to be studied. The waveform is directly affected by gravitational light deflection and relativistic Doppler shifts, and yields direct constraints on the mass and radius of the neutron star, and hence the equation of state of dense matter. Similarly, the modeling of the waveform of the high frequency quasi-periodic oscillations (which will for the first time be observed on their coherence time scales) can place important constraints on the mass and spin of the black hole.

A fast timing capability on XEUS would also extend its field of applications, by allowing the study of the X-ray variability of a wide class of objects, such as low-mass X-ray binaries in external galaxies (owing to the zero background, kilo-Hz quasi-periodic

oscillations could be detected up to ~20 keV for the brightest objects), accreting and isolated pulsars, accreting white dwarfs, and X-ray transients, especially the so-called micro-quasars. Accretion and jet formation are crucial in understanding aspects of astrophysics from normal stars to super-massive black holes. The fast timing capability can be combined with good energy resolution and broad band coverage allowing time resolved spectroscopic observations. For instance, the space-time geometry close to black holes could be probed using variability in the iron  $K\alpha$  line.

### **Science requirements**

The science requirements derive from the ability of the fast timing capability to observe the brightest X-ray sources in the sky, mostly X-ray binaries, either transient or persistent. Simulations using the current effective area of the XEUS mirrors show that the Crab (which is our standard reference in X-ray astronomy) would produce about 250 kcts/s and about 800 kcts/s in XEUS-I and XEUS-II respectively. Sco X-1 (the brightest permanent X-ray source of the sky), bright transients and X-ray bursts can be ten times brighter. This leads to a requirement to be able to handle up to 3 Mcts/s (XEUS-I) and 10 Mcts/s (XEUS-II).

The arrival time of each photon should be recorded with a timing resolution of ~10 micro-second.

The energy resolution of the detector should be around 200 eV (i.e. a factor of ~10 improvement over current instrumentation for timing studies).

Finally the detector energy range should cover the high-energy response of the mirrors (as timing signals tend to become stronger at higher energies).

**The best should be to be able to calibrate the detectors from ~0.5 to ~50 keV**

### **Detector implementation**

The detector the most capable of matching our requirements are a matrix of ~10-20 Silicon Drift Detector (SDD) placed out of focus of the XEUS mirrors. SDDs improve over conventional silicon PIN photodiodes by their much lower electronic noise, owing to the very low value of their output capacitance (Gatti & Rehak, 1984, NIMA, 225, 608). Each SDD should have an effective area of 5 mm<sup>2</sup>, which means that the overall size of the detector will remain very small.

For particle physics applications, SDDs have proved to be capable of handling up to 10 Mcts/s with pile-up less than 20%. This is achieved by integrating the first transistor of the amplifying electronics on the detector chip itself. Energy resolution of better than 200 eV (at 6 keV) is readily achieved with moderate cooling (-20° C) (Lechner et al. 2001, NIMA, 458, 281). SDDs are currently produced with thicknesses of 300 microns. Although there are on-going efforts to thicken these devices, the best match of the high energy response of the mirror could be achieved by associating the SDD detector with a higher density semiconductor detector located underneath (e.g. CdTe, CdZTe, GaAs).

### **The opportunity offered by SOLEIL**

Such a fast timing detector will be included in the baseline of the XEUS focal plane. It will have therefore to be tested and calibrated for flight readiness around 2010. This implies that the detector will have to be exposed to very high X-ray photon fluxes (say up to 10<sup>7</sup> photons/s over one pixel *between ~0.5 and ~50 keV*). Although a detailed study is required to determine how this could be best achieved with a metrology line integrated on SOLEIL, one can already claim that there should not be any major problems as the detector and electronics

will have to be very compact. In addition, the SDD detectors are quite robust, do not require any complicated cooling mechanisms and special handling conditions.

Further information:

XEUS: <http://astro.estec.esa.nl/XEUS/>

Barret et al. (2002): <http://xxx.lpthe.jussieu.fr/abs/astro-ph/0202451>

### ***XEUS - Mission Concept***

*XEUS will incorporate many new and innovative technologies to provide a major leap forward in capability. Following launch into low Earth orbit the mission will provide:*

- *6 m<sup>2</sup> of collecting area at 1 keV*
- *Imaging resolution with a goal at 1 keV of 2" HEW (Half Energy Width)*
- *A limiting sensitivity around 200 times deeper than XMM-NEWTON*
- *Spectral resolution of between 1 and 10 eV over the energy range 0.05 to 30 keV*

*After completion of the initial 4-6 year mission phase, XEUS will rendez vous with the International Space Station for refurbishment and to allow the addition of extra mirror area. A new Detector Spacecraft complete with the next generation of focal plane technologies will be added. The final grown mirror will have up to 30 m<sup>2</sup> of collecting area at 1 keV and 3 m<sup>2</sup> at 8 keV. Then, the sensitivity will be 200 times better than XMM-NEWTON.*



**PRaXO**  
**Pôle d'Optique Rayons X d'Orsay**

**At-wavelength interferometry  
on the SOLEIL metrology beamline**

**Denis Joyeux**  
**LCFIO**

**Mourad Idir**  
**LIXAM/LURE**

**François Polack**  
**LURE/SOLEIL**

A dedicated metrology beamline with large spectral brightness should allow at-wavelength interferometric metrology, **without the need of any technical breakthrough**. It is based on *wavefront division interferometry*, which has already been demonstrated in several applications at SuperACO (LURE/Orsay). The transfer on SOLEIL with an increase in spectral brightness should essentially improve experimental conditions and performances, making experiments much more practical, or even in some cases feasible in routine. The following text intend to explain the technical context and expected result of interferometry on Synchrotron Radiation beamlines.

Speaking of optical metrology, two classes of measurements should come in mind: *intensity* measurements, and *phase* measurements. Basically, the first class is related to the *energy* carried by wavefront, which allows in particular to track energy losses during interaction with materials. The second class concerns the detection of wavefront phase profiles, mainly to track wavefront deformations, i.e. spurious or provoked path advances or delays in the propagation. In both cases, energy detectors are used, but, in phase measurements, the optical phase is encoded into interference patterns. In the particular context of very short wavelengths, interferometry was not demonstrated and implemented, until relatively recently. Mainly two reasons are involved, namely the limited *spectral brightness* of sources, and also the technological difficulties related to short wavelengths (large absorption, small reflectivity, required accuracy and stability). To fully understand the way interferometry at short wavelengths developed, it is necessary to provide some technical background.

Generally speaking, two kinds of interferometers can be implemented to perform the necessary generation of the two (or more) wavefront that are made to interfere:

a) by splitting the wave “ray by ray”, so to say, by the means of a *beam splitter*, usually a semi-transparent / semi-reflecting optical component. This is known as *amplitude division* interferometry (Michelson, Mach-Zehnder). There is no need to insist about the technological difficulty of producing such a component at very small wavelengths (although diffraction gratings may have to some extent the same function). Moreover, such components cannot at

present be fabricated for any wavelength, and, if they can, their use is generally restricted to a narrow spectral band.

b) by splitting the wave into “bundles of rays” e.g. by using 2 reflectors, one for each half of the wavefront. This is known as *wavefront division* (WD) interferometry, one prototype of which is Fresnel’s bimirror. WD interferometers are essentially differential interferometers, in which a part of the field to study is interferometrically compared to another part.

The main feature of such wavefront division interferometry is that it can be implemented within a very wide spectral range without major technological difficulty. The simplest way is to use grazing incidence, plane mirrors (which makes multilayer coatings unnecessary). Such interferometers can be designed monolithic (i.e. very stable), or adjustable. No major breakthrough is required to implement it up to about 1 or 2 keV.

Despite this technological relative simplicity, these devices, when properly implemented, allow surprisingly high performances in terms of phase sensitivity, which can be used in many application of metrology, or even of physics. This was demonstrated at super-ACO since 1992 at  $\lambda=3-5$  nm (400-250 eV), and 14 nm (88 eV). In both cases, *optical thickness* were measured directly, with an ultimate sensitivity below  $\lambda_x/300$  in both cases, in transmission in the first case, in reflection in the second case. Besides this “simple” interferometry, we have also demonstrated interferential microscopy with long working distance (500 mm), with a spatial resolution of 1  $\mu\text{m}$  at  $\lambda=14$  nm, and much below  $\lambda_x/100$  sensitivity in phase.

The range of application of such experiments is quite wide. In principle, any object or phenomenon resulting in an optical phase variation (either vs space or vs time) can be accessed through interferometry. This includes the calibration of phase plates (CALCORR ESPRIT project, within the EUV lithography context), but also the determination of optical constants and dispersion, the study of the “phase roughness” issuing from multilayered reflectors (possibly by interferential microscopy), and even the test of aspherical wavefronts.

On slightly different principle, coherent microscopies such as dark field, Zernike phase contrast are also experiments which benefits of an increased brightness.

Obviously the same metrology principles could be performed on any beamline dedicated to metrology. As a matter of fact, a very important gain in performance can be expected from the transfer on SOLEIL. To understand the nature and importance of these improvements, it is again necessary to switch to some technical considerations.

The most important one is that, in opposition to the common opinion, the *spatial coherence* of SR beam is *not* the pertinent source specification for the performance of spatially coherent, interferometry experiments. The pertinent spec is *the spectral brightness*, i.e. the flux per unit area of the normal emitting surface, per unit of emitted solid angle (or of 1D emittances) of the beam, and per unit of spectral band. This is because, whatever the source, the flux allowed as input in a *spatially coherent* experiment cannot exceed the flux carried by the coherent *étendue* (optical extent) for 2-dimensional experiments, (or by the coherent emittance for 1-D experiments): *these are fully determined by the wavelength only*. Therefore, whatever the intrinsic coherence of the source, the maximum flux is entirely given by brightness, and the wavelength. The consequence is that the improvements to be expected are related to the gain in brightness. The large expected gain around 20 allows to compromise on several parameters of the experimental configuration: the detected flux (better detection s/n), the interferometric field (larger field), *and the geometrical separation between arms*, even with the classical Fresnel bimirror configuration. This latter point is of some importance, considering for instance that samples may include a “dead zone”, requiring the reference wavefront not to be adjacent to the sample wavefront.

Finally, the use of high sensitivity image detectors (cooled, back illuminated, slow scan

CCDs), together with the increased brightness, might allow increased imaging flexibility, by using a composite detection system. The latter consist of an X-to-visible conversion through a practically infinite resolution, grainless fluorescent screen as image detector, followed by an additional magnification by a “good, standard” imaging system. The advantage would be to add flexibility to high resolution image detection, by overcoming the problem of CCD pixel size. Such screens (doped monocrystals) are already used at higher energies (10 keV). We have tested their use at low energies (100 eV), showing a very significant increase of image quality compared to ordinary powder fluo screens, despite a moderate loss in detection efficiency. (Note that in the spectral range considered no image blurring should occur from photon penetration).

**To summarize: WD interferometry can be implemented on the SOLEIL metrology beamline, for high sensitivity phase determinations, possibly with spatial resolution.**

All necessary technologies, which rely on top-level optical know how, are demonstrated. Fields of several millimeters, with arm separation of the same order can be expected even with a simple bimirror interferometer. More sophisticated systems can be considered, to provide similar fields, but with a true separation of several centimeters.

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**EU-funded ESPRIT-4 project #28146 « CALCORR » (*Interferometric CALibration of a CORRective phase layer for diffraction-limited EUV mirror optics*)**

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