

# Status of the Nanoscopium Scanning Hard X-ray Nanoprobe Beamline of Synchrotron Soleil

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**Abstract.** The Nanoscopium 155 m-long scanning hard X-ray nanoprobe beamline of Synchrotron Soleil (St Aubin, France) is dedicated to quantitative multi-modal 2D/3D imaging. The beamline aims to reach down to 30 nm spatial resolution in the 5-20 keV energy range. Two experimental stations working in consecutive operation mode will be dedicated to coherent diffractive imaging and scanning X-ray nanoprobe techniques. The beamline is in the construction phase, the first user experiments are expected in 2014. The main characteristics of the beamline and an overview of its status are given in this paper.

## 1. Introduction

The Nanoscopium scanning hard X-ray nanoprobe beamline is dedicated to quantitative multi-modal imaging on the submicron and nano-length scales. Two experimental stations, CSI and Nanoprobe, are dedicated to coherent scatter imaging and X-ray nanoprobe techniques, respectively, in the 5-20 keV energy range. Typical scientific applications cover fields such as biology and life sciences, and earth sciences, environmental sciences and geobiology. All of these scientific communities are aiming to study heterogeneous samples at multiple length scales; to obtain information on the specimen structure, its major, minor, and trace element content, and the chemical speciation. We will enable this multiscale and multitechnique imaging by scanning in fast continuous and high precision step scan modes. Cryogenic cooling of radiation-sensitive, fragile samples will be implemented at the beamline.

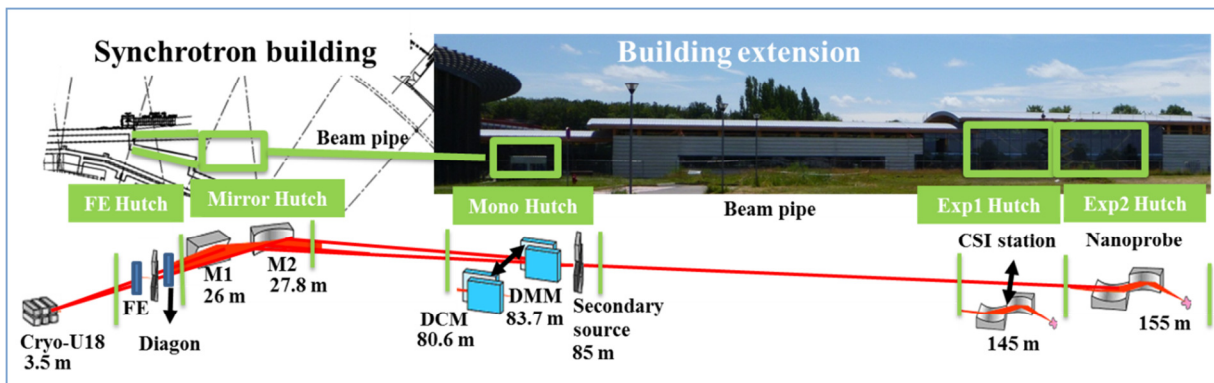
## 2. Characteristics and status of the Nanoscopium beamline

### 2.1. Source, frontend and first beam-shaping elements

The Nanoscopium beamline [1] shares a 12 m-long straight section of the Soleil storage ring with the future Tomography beamline. The dedicated source optics providing double low vertical beta functions [2] was installed in 2011. This was followed by the implementation of a prototype cryo-cooled U18 Pr<sub>2</sub>Fe<sub>14</sub>B permanent magnet undulator [3] for the commissioning period of Nanoscopium, in canted geometry, with the undulator source of Tomography.

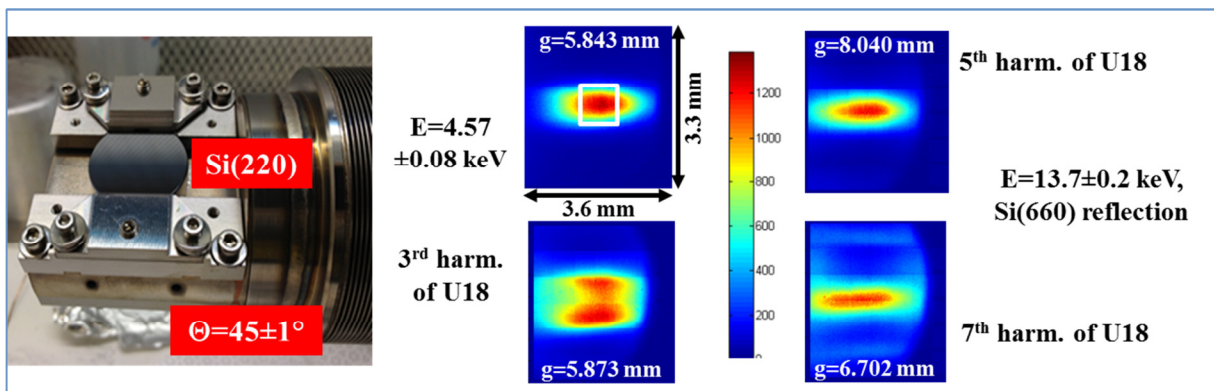
The optical scheme of the beamline can be seen in figure 1. The 800 x 800  $\mu\text{m}^2$  fixed-opening front end (FE) aperture, the primary slits, and the beamline filters are situated in the FE Hutch. The size of the FE aperture is matched to the maximum beam acceptance of the nano-focusing optics of the experimental stations.





**Figure 1.** Scheme of the Nanoscopium beamline. The distances of the beamline components are given from the center of the straight section. The source is a cryocooled U18 undulator, on the left. Proceeding downstream, the other components are the front-end (FE), filters and slits, a Diagon, white-beam focusing mirrors M1 and M2, double-crystal and double-multilayer monochromators (DCM and DMM, respectively) and then the experiment hutches CSI and Nanoprobe. See text for details.

The FE aperture was centered on the emission axis of the undulator using a hard X-ray version of the Diagon device [4], which images Bragg diffraction from a water-cooled Si220 crystal at an angle of  $45 \pm 1$  degrees relative to the incoming white beam. The emission cone of the undulator is imaged at  $4.57 \pm 0.08$  keV or at  $13.7 \pm 0.2$  keV defined by the 1st- or 3rd-order Si(220) reflections, respectively (figure 2). This also allows a comparison between the theoretical and measured undulator emission, for diagnostic purposes.

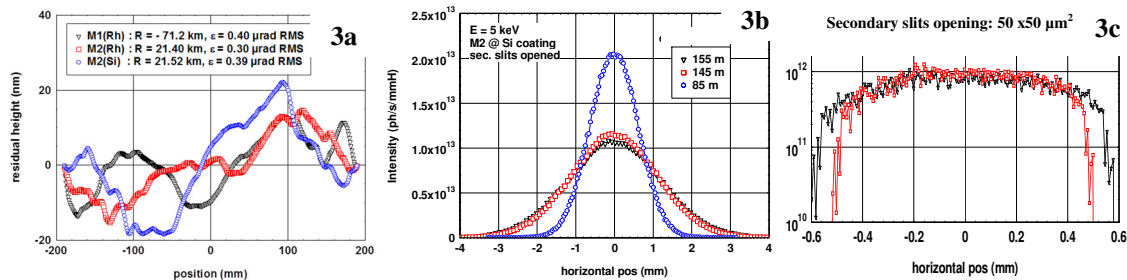


**Figure 2.** Te “Diagon” device and the measured 3<sup>rd</sup>, 5<sup>th</sup>, and 7<sup>th</sup> undulator harmonics. The corresponding gap values,  $g$ , are indicated in the figure. The  $\sim 4.6 \times 4.6 \mu\text{m}^2$  effective pixel size is determined by the Basler camera of the Diagon. The white rectangle shows the size and position of the  $800 \times 800 \mu\text{m}^2$  FE aperture aligned at the beam axis. The beam shape was obtained by scanning the FE diaphragm in the  $\pm 2$  mm range.

## 2.2. Main optical elements of the beamline

The two 400 mm long, fixed curvature mirrors, M1 and M2 (Winlight), work at 2.5 mrad grazing incidence angle. They are situated in the Mirror Hutch inside the synchrotron experiment hall (figure1). The mirrors will create an image of the source at the location of the secondary source, 85 m from the center of the straight section. M1 is a sagittally-focusing mirror with 81.7 mm sagittal radius with a Rh coated optical surface. The Si and Rh coatings of the M2 tangentially-focusing mirror (with 21.4 km measured tangential radius) ensures the rejection of higher order harmonics from the undulator spectrum. The cumulative effect of the measured tangential residual shape-errors of M1 and

M2 [5] (figure 3a) on the horizontal beam-profile (figure 3b) was calculated by ray-tracing calculations [6] at the secondary source (85 m) and at the positions of the focusing optics of the CSI (145 m) and Nanoprobe (155 m) stations. The homogenous beam profile shown in figure 3c overfilling the apertures of the focusing optics (typically 200-500  $\mu\text{m}$ ) is one of the requirements for stable beamline operation.



**Figure 3a.** Measured (by long trace profiler, LTP) tangential residual height of the coated M1 and M2 prefocusing mirrors [5]. **3b.** The horizontal beam profile at 85 m (secondary source), at 145m (CSI station) and at 155 m (Nanoprobe) calculated by ray tracing taking into the measured mirror quality. **3c.** Horizontal beam profile at the position of the focusing optics of the CSI (red curve) and Nanoprobe (black curve) stations with  $50 \times 50 \mu\text{m}^2$  secondary source size. See text for details

The two interchangeable monochromators, a Si(111) double-crystal (DCM, CINEL) and a double-multilayer (DMM) one, together with the secondary source will be situated in the Monochromator Hutch in the building extension (figure 1). In keeping with the all-horizontal geometry, both monochromators will be used in  $\pi$ -polarization mode. The secondary source will be determined by high precision slits and pinholes, in the  $\mu\text{m}$ -mm opening range. They will be situated after the monochromators, 85 m from the center of the straight section. All the equipments, except the DMM, are under construction; their installation is expected for the beginning of 2013. The DMM, allowing for high flux measurements is in the design phase.

### 2.3. Experimental stations and methods

Two consecutive experimental stations will be situated in the 10 m-long concrete hutches in the external building at  $\sim 60$  and  $\sim 70$  m distances from the secondary source (figure 1). The experimental setup optimized for scanning coherent scatter imaging methods, such as ptychography [7] can be found in the first, CSI, Hutch. Quantitative 2D/3D images based on electron density contrast, and nanostructure orientation, will be offered by these coherent imaging methods. Scanning X-ray spectro-microscopy methods will be situated in the second, Nanoprobe, Hutch offering elemental mapping at trace levels (by X-ray fluorescence, XRF), speciation mapping (X-ray absorption near edge structures, XANES), phase gradient mapping (scanning differential phase contrast imaging) with down to  $\sim 30$  nm spatial resolution.

The use of achromatic total-reflection Kirkpatrick-Baez (K-B) mirrors at both end-stations will ensure high flux in the probing nanobeams, and facilitating easy energy tuning in the full energy range without realignment of the beamline. The ultra-high optical quality of these elliptically figured K-B mirrors is crucial both for wave-front preservation and for diffraction limited focusing. Wave-optical calculations [8] have been carried out to specify the required surface quality and alignment tolerances of the KB mirrors in the 5-20 keV energy range using four figures-of-merit to characterize the focused wavefield; namely, focal spot intensity full-width at half-maximum (FWHM) in the lateral (beam size) and longitudinal (depth-of-field) directions, integrated intensity in the FWHM, and finally the presence of significant features, or “side-lobes” in the vicinity of the focus. The measured optical and mechanical characteristics of the Rh-coated K-B mirror-pair (mirrors manufactured by JTEC Corp., Osaka, Japan [9]), and the ultra-high precision UHV positioning system (designed and manufactured by Bruker ASC) indicate tolerances ensuring diffraction-limited focusing at the Cryo-Nanoprobe station. This system is to be delivered at the end of 2012.

In order to offer multiscale and multimodal imaging, both continuous scanning with millisecond dwell times and step-scan mode operation with nm positioning precision will be implemented at the beamline with simultaneous multiple detections. This will enable imaging large fields of view with moderate spatial resolution and sensitivity, followed by high spatial resolution and high sensitivity mapping of small volumes of interest. Fast multi-technique experiments, namely scanning X-ray fluorescence spectrometry combined with absorption, differential phase contrast, and dark-field imaging will provide statistically significant quantitative imaging possibilities. The corresponding distributed fast-acquisition system for synchronized multi-technique experiments (FLYSCAN) is being prototyped at SOLEIL (see K. Medjoubi *et al* [10]).

### 3. Conclusion and Outlook

The Nanoscopium beamline is in the final phase of construction. The external building will be finished in Nov. 2012, which will be followed by the installation and commissioning of the optical elements, such as monochromators and secondary slits, and the experimental stations in 2013. The beamline will be open for users in 2014.

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