Hard X-ray nanoprobe at beamline P06 at PETRA III

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\begin{abstract}
We describe the hard X-ray scanning microscope planned for the new synchrotron radiation source PETRA III at DESY in Hamburg, Germany. It is based on nanofocusing refractive X-ray lenses and is designed for two-dimensional mapping and scanning tomography. It supports X-ray fluorescence and (coherent) diffraction contrast, yielding elemental and structural information from inside the sample. Spatial resolutions down to well below 50 nm are aimed for in direct space. A further increase in spatial resolution is expected by applying ptychographic scanning schemes. The optical scheme with a two-stage focusing optic is described.
\end{abstract}

1. Introduction

Hard X-rays are a very attractive probe for microscopy, as their large penetration depth in matter allows one to investigate the interior of an object without destructive sample preparation, even inside special sample environments, such as chemical reactors or pressure cells. In scanning microscopy, different X-ray analytical techniques, such as fluorescence and absorption spectroscopy or diffraction, can be used as contrasts, yielding elemental, chemical, or structural information on the sample, respectively. This makes hard X-ray scanning microscopy a very attractive probe in many fields of science, including physics and chemistry, bio-medicine, materials, geo- and environmental science, archeology, and nanotechnology.

Hard X-ray scanning microscopy has evolved quickly with the advent of synchrotron radiation sources of the third generation, such as the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, the Advanced Photon Source (APS) near Chicago, United States, or SPring-8 in Japan. These sources generate hard X-rays with unprecedented brilliance, which is the key figure of merit for generating a small and intensive microbeam for scanning microscopy. Today, many new synchrotron radiation facilities have come to operation, are under construction or in the planning phase. PETRA III at DESY in Hamburg, Germany, is such a new facility that is currently under construction and commissioning. Due to its large circumference and special positron optics it has a particularly small horizontal emittance of 1 nm rad making it the most brilliant synchrotron radiation source in the hard X-ray range. Hard X-ray scanning microscopy exploits the high brilliance particularly well and was identified as one of the techniques to be implemented at this new storage ring.

Besides the source, highly specialized optics are required to generate a small hard X-ray beam. In the last decade, significant advances have been made in this field. While Fresnel zone plates are the predominant optic for soft X-rays, in the hard X-ray range, diffractive [1–5], reflective [6], and refractive optics [7–9] are used in scanning microscopy. At the PETRA III Hard X-Ray Micro/Nano-Probe beamline P06, all three types of optics will be used. The beamline is scheduled to become operational in 2010.

In this article, we introduce the nanoprobe activity at beamline P06. The hard X-ray nanoprobe is set-up in collaboration of the Technische Universität Dresden and DESY and financed by the German Department of Education and Research (BMBF). In Section 2 we give a short overview over the new machine and the Hard X-Ray Micro/Nano-Probe beamline P06. The concept for the hard X-ray nanoprobe is given in Section 3, explaining the optical scheme and the instrument.

2. PETRA III and beamline P06

PETRA III is the new low emittance storage ring at DESY in Hamburg, Germany [10]. It is built on the site of the former PETRA storage ring that was mainly used for high energy physics. While seven-eighths of the ring tunnel and other building infrastructure were reused for the PETRA III project, the whole machine with a circumference of about 2300 m was built from scratch. It will store positrons with a ring current of around 100 mA in top-up mode at
an energy of 6 GeV. The large circumference and a total of 80 m of damping-wigglers allow for a low horizontal emittance of 1 nm rad that has been reached recently and that is ideal for micro- and nanofocusing.

On one-eighth of the ring, a new experimental hall was built that accommodates eight straight sections with a total of 14 undulator beamlines. Most straight sections are shared by two beamlines with two independent undulators canted by 5 mrad. The Hard X-Ray Micro/Nano-Probe beamline P06 [11] shares a low-β section (Sector 4) with the imaging beamline P05. The nominal source size in this section is $35 \times 6.9 \mu m^2$ (rms).

A 2-m spectroscopy undulator ($\lambda_u = 31.4 \text{ mm, } K_{\text{max}} = 2.7$) is installed at P06 and is continuously tunable in the hard X-ray range starting from $E = 2.4 \text{ keV}$. The beam divergence is expected to be $29 \times 3.9 \mu \text{rad}^2$ at 10 keV.

Fig. 1 shows the beamline layout of Sector 4. The optics hutch starts at 31.5 m from the undulator source. The first optical element is a cryogenically cooled double-crystal monochromator located at 38.5 m from the source. It holds pairs of Si (111) and (3 1 1) crystals and can cover an energy range from 2.4 to 100 keV, generating a fixed vertical offset of 21 mm from the storage ring plane. Alternatively, a two-bounce multilayer monochromator will be available in the future to generate monochromatic radiation with a band pass in the percent range. A pair of flat horizontally deflecting mirrors based on total external reflection serves to suppress higher harmonic radiation. Three different coating materials (Si, Cr, Pt) are available to adjust the energy cut-off in a range from 4 to 30 keV. The last optical elements in the optics hutch are refractive X-ray lenses for prefocusing located at 43.3 m [12].

As the sector is shared with the imaging beamline, the optical path between the optics hutch, ending at nearly 44 m, and the first experiments hutch (microprobe hutch), starting at 87.5 m from the source, are not available for further optical elements.

The first experiments hutch (cf. Fig. 1) accommodates the Hard X-ray Micro-Probe station. Microfocusing is done with a Kirkpatrick–Baez mirror system and by parabolic refractive X-ray lenses at lower and higher energies, respectively. Hard X-ray beam sizes down to the 300 nm level are to be used for scanning microscopy with fluorescence, absorption spectroscopic and diffraction contrast.

The nanoprobe hutch follows the microprobe hutch and starts at 96.2 m from the source. It is reserved for a hard X-ray scanning microscope based on nanofocusing refractive X-ray lenses that is described in detail in the next section. Both experiments hutches are climate controlled to within 0.1 K and are accessible through an air lock.

3. Hard X-Ray Nano-probe at beamline P06

The hard X-ray nanoprobe is based on nanofocusing refractive X-ray lenses [8,9]. The instrument is located at 98.2 from the source (sample position) and is shown in Fig. 2. It is designed to generate nanobeams with a lateral size of 50 nm and below and supports transmission, fluorescence, and diffraction contrast. It is made of three separate units, a scanner unit and two detector units, each accommodated on a separate optical table.

The scanner unit will be mounted on a granite block that is directly deposited on a concrete base. In this way, maximal stiffness of the support will be given and the seismic motion of the ground will be transferred to the top of the table without amplification. The scanner unit is designed for high stiffness, but has to be sufficiently versatile to allow for a wide range of experiments. Therefore, the sample will be mounted on a stage with nine degrees of freedom. At the bottom, three linear stages allow for the rough alignment of the rotation axis with respect to the beam. A rotation stage for tomographic scanning and diffraction experiments allows for full sample rotation with minimal eccentricity and wobble. On top of the rotation will be a high-precision and high-accuracy piezo-driven flexure stage with three translational degrees of freedom (range 100 $\times$ 100 $\times$ 10 $\mu$m$^3$ with $< 2$ nm repeat accuracy). On top of this stage, the sample will be centred on the rotation axis using two translational piezo-driven slip-stick stages. In a high stability mode, the number of degrees of freedom of the sample stage is reduced.

In a first phase, nanofocusing refractive X-ray lenses (NFLs) made of silicon will be used to generate the small beam. Fig. 3(a)

![Fig. 1.](image-url) (Colour online) (a) Layout of Sector 4 at PETRA III. The sector is shared between beamline P05 (imaging beamline) and P06 (Hard X-Ray Micro/Nano-Probe). (b) Experiments hutches of beamline P06.
shows an array of NFLs made of silicon fabricated with optical lithography and deep reactive ion etching. Each of these optics generates a line focus. In order to obtain a point focus, two such lenses with appropriately matched focal lengths need to be aligned in crossed geometry as shown in Fig. 3(b). A rectangular slit system in front of the lenses and a pinhole behind the lenses remove unfocused radiation. At a later stage, other optics can be used in the setup, such as NFLs made of a more transparent material, i.e., diamond, or Fresnel zone plates [13].

A drift-diode is used as energy dispersive detector to record the fluorescence from the sample. It is placed on a separate table on the side and faces the sample under 90° with respect to the optical axis [Fig. 2(a,b)]. It can be aligned in three translational degrees of freedom. The other detectors are mounted on a third table placed behind the setup [Fig. 2(a,b)]. The table has four degrees of freedom (three translations and one rotation), allowing to position a diffraction camera from the wide- to the small-angle-scattering regime, i.e., from 100 to 2000 mm behind the sample. The camera can also be positioned off-axis in the horizontal plane with its sensitive area facing the sample, allowing one to analyse the shape of Bragg-reflections [14]. Besides the diffraction camera, a high-resolution X-ray camera, a visible light microscope, and two diodes are mounted on the detector table. All these detectors are mutually exclusive but can be automatically moved into the beam. In a next phase, it is planned to install a single-photon counting pixel detector in addition.

With the high-resolution X-ray camera, projection images of the sample can be recorded in the flat beam allowing for tomographic imaging with absorption and phase contrast at a resolution in the µm range [15,16]. The visible light microscope...
gives complementary information about the sample and mainly serves for sample alignment. The diodes, a PIN-diode in current mode and an avalanche photo diode (APD), allow for accurate transmission measurements. With the APD, photon-correlation spectroscopic measurements become feasible.

4. X-ray optical design

In order to achieve diffraction limited focusing at maximal flux, the lateral coherence length at the entrance of the focusing optic must match its aperture. If the coherence length is larger than the aperture, the geometric image of the source is smaller than the size of the Airy disc and the latter dominates the focus. Consequently, only a small part of the total coherent flux is focused. If the lateral coherence length is smaller than the aperture of the optic, the geometric image of the source dominates the focus size at larger flux. In the case of aperture matching, the image size matches that of the Airy disc, resulting in a nearly Gaussian focus that is \( \sqrt{2}/2 \)-times larger than the diffraction limit and the coherent flux from the source is efficiently focused as shown in Fig. 4.

At PETRA III—like at all synchrotron radiation sources of the third generation—the source size is very different in the vertical and horizontal direction. At the position of the nanoprobe at 98 m from the source, the lateral coherence area after free propagation is expected to be 74 \( \times \) 400 \( \mu \text{m}^2 \) \( (E=20\text{keV}) \). To focus the coherent fraction of the beam, the effective aperture of the refractive lens would approximately have to have this size. Largest apertures and smallest Airy discs are, however, reached for NFLs with much smaller apertures. Therefore, appropriate prefocusing is required to focus the coherent fraction of the beam into the aperture of the NFLs.

As an example consider the nanobeam at \( E=20\text{keV} \). At this energy, two NFLs with \( N=100 \) (horiz.) and \( N=50 \) (vert.) single lenses \( (R=2.2\mu\text{m}) \) generate a common focus about 6 mm behind the horizontally focusing lens. The Airy disc size for this optic is 33 \( \times \) 49 nm\(^2 \). Without prefocusing, the focus size is expected to be largely diffraction limited, with a beam size of 34 \( \times \) 49 nm\(^2 \) and a flux of 1.1 \( \times \) 10\(^8 \) ph/s. With astigmatic prefocusing using \( N_{rot}=13 \) rotationally parabolic lenses \( (R=500\mu\text{m}) \) and \( N_{srot}=3 \) horizontal parabolic cylinder lenses made of beryllium \( (R=500\mu\text{m}) \), however, the flux can be increased to more than 8 \( \times \) 10\(^8 \) ph/s while only slightly increasing the focus size to 44 \( \times \) 65 nm\(^2 \).

The prefocusing optic at P06 is realized using an automatic lens changer that allows for a very flexible adjustment of the coherence length at the entrance of the nanoprobe. In this way, the coherence length can be matched to the nanofocusing optic in a large energy range and the beam size can be changed without the need for adjustments in the nanoprobe. For example by reducing the number of prefocusing lenses to \( N_{rot}=12 \) and \( N_{srot}=1 \), a beam with a lateral size of 140 \( \times \) 225 nm\(^2 \) and a flux of 1.5 \( \times \) 10\(^9 \) ph/s can be tailored without the need to realign the nanoprobe. Switching between the large and the small beam is possible in seconds.

The coherence properties in the diffraction limited beam have been shown to be sufficient for coherent diffraction imaging experiments [17]. Besides fluorescence, transmission, and diffraction contrast, ptychographic scanning techniques [18] are planned to be implemented. The instrument is currently being assembled and commissioned and will be installed at PETRA III early in 2010.

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References