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The nanotomography endstation at the PETRA III Imaging Beamline

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Abstract. The Imaging Beamline (IBL) operated by the Helmholtz-Zentrum Geesthacht (HZG) at the newly refurbished DESY PETRA III storage ring is dedicated to radiography and tomography and provides two experimental endstations, one for micro tomography and one for nano tomography. The technical specifications aim for 3D imaging with a spatial resolution of below 100 nm. This nanometer resolution will be achieved by using different combinations of compound refractive lenses as X-ray optics. In addition, a microscopic optic for magnifying the images after the converting in visible light will be used, too.

The overall setup is designed to be very flexible, which allows also the implementation of other optical elements (e.g. Fresnel zoneplates, KB mirrors) as well as the application of different magnifying techniques like cone-beam tomography or X-ray microscopy. The accessible energy range for the nano tomography is 10 - 30 keV but the beamline is designed for an energy range of 5 - 50 keV and we aim to allow the same energy range for the nano tomography in the long run.

1. Introduction

Developments of novel materials and production technologies are significantly linked to the fundamental understanding of materials characteristics, especially the three dimensional inner structures. Characteristic lengths influencing these structural properties are often in the range of some micrometers down to a few nanometers [1, 2]. For example, the material design of ultra–leight weight materials is strongly dependant on knowledge about the structure on a sub–micrometer scale to correlate mechanical behaviour and nanostucture [3]. Micro and nano tomography are non–destructive techniques allowing the 3D characterization of these properties. To meet this demand, other nanotomography endstations are already in operation at the APS (HXN) and SLS (TOMCAT) or under construction (NSLS-II). They offer resolutions of down to 40 nm in the X–ray regime around 10 keV [4, 5].

The Imaging Beamline (IBL) [6, 7] is one of 14 beamlines at the refurbished PETRA III storage ring at DESY. Running at 6 GeV and with a small emittance, it is one of the most brilliant X–ray sources worldwide for hard X–rays. IBL and the High Energy Material Science Beamline (HEMS) [8] are operated by the Helmholtz–Zentrum Geesthacht. Whereas the accessible energy range at IBL lies between 5 and 50 keV, a second microtomography experiment is installed at HEMS, allowing energies of up to 150 keV for tomographic studies of high–Z materials.

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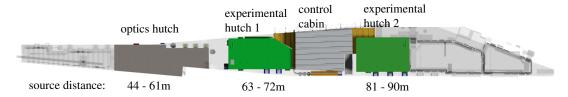


Figure 1: Schematic drawing of the beamline layout. The large optics hutch allows the installation of two monochromators as well as all other required beamline optics. Two separate, large experimental hutches exist for the micro (EH2) and nano tomography (EH1) experiments.

2. Beamline layout

The insertion device of the imaging beamline is one two meter part of an canted undulator, the two x-ray beams are assigned to the Imaging Beamline and to the Micro and Nano Probe Beamline P06, which is located on the same sector. Both beamlines work independently and simultaneously.

A schematic sketch of the beamline layout is given in Figure 1. Because of the shared sector, the Imaging Beamline is limited to 90m length from the source. The beam size requirements of the micro tomography combined with the small divergence force a large source distance for the micro tomography hutch, leading to the nano tomography hutch being the first behind the optics hutch.

The optics hutch is equipped with two monochromators: A double crystal monochromator (DCM) equipped with silicon single crystals (111 and 311) and designed by DESY will be used for tomographic methods, which need a very high monochromatization ($\Delta E/E \approx 10^{-4}$, e.g. absorption edge tomography, vector tomography). For applications, which need particularly high flux (e.g. fast in situ experiments) a double multilayer monochromator equipped with different flat and bent substrates will be used ($\Delta E/E$ up to 10^{-2}). Both monochromators are cryogenically cooled and can be used alternatively or in series to reduce the heat load on the Si crystals. The energy range for both types will be tunable between 5 and 50 keV.

Apart from standard optics like apertures, beam position monitors etc. a large vacuum tank for X-ray lenses and a related aperture is included in the optics hutch. These optics are required for creating a virtual source necessary for the nano tomography option. The tank is designed large enough to mount also a diffuser, should this be necessary to reduce beam inhomogeneity or to decrease the degree of coherence [9]. In addition, enough space is held back in the optics hutch to allow the installation of higher-order suppressing mirrors, should this become necessary.

The micro tomography hutch with its length of 9m houses the micro tomography experiment and spare room of $3 \times 3\text{m}^2$. For example, this room can be used for setting up large, stand–alone *in situ* experiments that are not compatible with the standard setup.

The nano tomography hutch of 9m length is situated at a distance of 63–72m from the source. While this smaller source distance impedes the X–ray focusing, it has other benefits. The virtual source which can be created in the optics hutch is located approximately 5m from the first experimental components in the experimental hutch. In addition, the detector in the microtomography hutch can be used. An evacuated beam pipe with diamond windows allows to use the X–ray optics and sample stage in the nano tomography hutch in combination with the detector in the micro tomography hutch, giving an additional propagation distance of 18m to the detector for very high magnifications.

3. Experimental setup

The nanotomography endstation is designed to allow 3D resolutions of below 100nm. The experiment is designed to be as flexible as possible while still allowing for the best resolution technically achievable. In the first setup stage, all X–ray optical components are compound refractive lenses (CRLs). These lenses are made from a polymer using the LIGA process (German abbreviation for X-ray Lithography, Electroplating, and Molding) at the Institute of Microstructure Technology at the KIT, Karlsruhe

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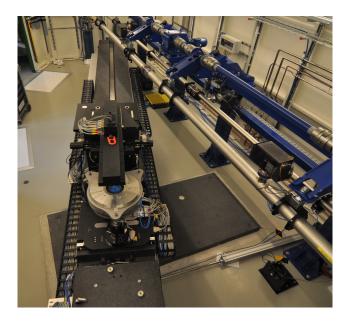


Figure 2: View of the complete experimental hutch 1 with the nano tomography experiment. The large granite is visible to the left as well as the motorized beam pipe to the right. The second granite plate on the floor is polished to allow the optical bench to be moved inside the beam on air pads.



Figure 3: View of optics alignment unit (left) and sample unit (right). The optics is mounted on a 6-axis kinematic mount; the rotational axis is moveable in the horizontal, perpendicular to the beam as well as in the height and tip/tilt with a three pod construction.

[10, 11, 12, 13]. This process allows to produce crossed 1D focusing lenses on one wafer, i.e. the combination of both orientations yields a point focus with a single optical element.

One of the major problems is gaining the required X-ray focusing and resolution with comparable large focusing distances. Because of the needed lens aperture—which limits the field of view—, the lens design is limited to larger radii of curvature and a smaller number of elements than would conventionally be used for the sole creation of a nanofocus.

To allow small spot sizes and high resolutions, a tiered optical approach is necessary. There are three positions at which optics can be introduced. The first position is situated in the optics hutch, approximately 59m from the source. It consists of a large vacuum tank with a 6-axis mount for optical components and a long linear translation to include apertures, knife edges or similar equipment. In addition, linear stages for photo diodes in front and behind the lenses and apertures are included to analyze flux and focusing performance. The second and third position are in front and behind the sample unit and will be further explained below.

A short transfer pipe of 2.8m connects the optics hutch with the first experimental hutch. With walls, beam shutter and vacuum components, an overall length of roughly 5m is inaccessible for experimental setups.

The experimental hutch of 9m length is furnished with a 6.8m long granite base that is used as experimental substructure. Figures 2 and 3 show photographs of the experimental hutch. By linking the complete setup through the large granite, the influence of vibrations is minimized and the equipment is stabilized with respect to each other. On this substructure, 4 air—bearing granite sliders are used to position the individual parts of the experiment. The sliders are each able to travel the complete length of the axis, giving a high flexibility in the positioning.

The sample unit is assembled on the second stage and consists of a precise horizontal translation stage for moving the complete unit in and out of the beam and three pods for height adjustment and tip/tilt

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correction of the axis. The air-bearing rotational axis is equipped with a large bearing surface to minimize rotational errors. Unavoidable statistical errors in axis positioning leave an error of approximately 80nm maesured 20mm above the axis surface. The axis is mounted on a tip-tilt stage to adjust the rotational axis normal to the beam. A tripod stage with travel ranges of 3mm in translation and 6° rotation is installed inside the aperture of the axis, allowing a sample positioning in all 6 degrees of freedom on the axis. Due to the very compact design, the sample position on the tripod will be very near the bearing surface of the axis.

The two sliders in front and behind the sample stage are reserved for the optics and make up for the second and third position for optics mentioned above. This allows maximum spaces of up to 5.3m between optical components and the sample or detector, respectively. The only limitation is the length of the overall granite length of 6.8m.

The mechanics assembly group includes a high precision tripod robotics for positioning and alignment of the optics in the beam. This allows for an easy change of the optical elements, as the mechanics is flexible enough to accommodate and align CRLs and other optics like Fresnel lenses as well. As KB-mirrors typically come with their own mechanics, this setup allows the use of all of the most common X-ray optical elements available while still being flexible enough to allow the adaptation of new developments. A full xyz scanning stage with two high-precision piezo xz scanning tables is installed behind the optics mount point to allow positioning of aperture foils or absorbers for beam characterization.

For a full-field microscopy setup, the optics assembly group is mounted on the third slab, downstream of the sample. The free space on the first slider is needed to include condenser optics and the corresponding mechanics. For a cone beam setup, the optics assembly group is moved to the first slider (with inverse orientation), allowing for a geometrical spot size of below 50nm.

The fourth slider is equipped with a xz stage and reserved for the detector unit. The detector system consists of a microscope optics mounted on a CCD camera. The optics is calculated as diffraction limited optical system and designed for a 4096x4096 pixel CCD chip with a size of 61x61 mm. The microscope is equipped with an automated lens changer for 4 different magnifications (5x, 10x, 20x, and 40x), a scintillator changer, a fast shutter, a filter wheel, and an aperture. The scintillator changer, the filter wheel, and the aperture are also motorized. Following the scintillator, a mirror deflects the visible light and allows for the optics to be installed out of the path of the X–ray beam, protecting the objectives from radiation damage.

4. Conclusion

The setup for the nano tomography is installed and first beam commissioning will start soon. Due to the very flexible setup, an adaptation to new advances in X–ray optics or the implementation of additional components, for example for phase imaging, is very simple. The expected performance is a spatial resolution of below 100nm.

While X-ray optics already allow for much lower resolutions [14, 15], the challenge remains for mechanical stability. Current air-bearing rotational are limited in their accuracy to above 50 nm, higher accuracy has to be achieved by measurement systems and active feedback. The same applies for the mounting of the X-ray optics. Furthermore, for reaching a mechanical stability on the scale of 10 nm, the surroundings have to be controlled as well. Especially temperature changes and ground vibration are typical problems for stability. An improvement in X-ray optics needs to go hand in hand with mechanical optimization of the experiment to achieve the desired resolution and improve it in the long run.

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