Preparing for hard x-ray microscopy with Multilayer Zone Plates

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Abstract. With Pulsed Laser Deposition, Multilayer Zone Plates can be fabricated to focus hard x-ray beams into 2D spots smaller than 10 nm. To put these optics into use for imaging applications, we have commissioned a new dedicated sample tower as a high-resolution module for the GINIX instrument, stationed at the P10 beamline at PETRA III. Here we summarise the motorisation and show first imaging benchmark results obtained with a “traditional” Fresnel Zone Plate. The first 2D continuous STXM scan using the new EigerX 4M detector at full 750 Hz speed is shown: a field of view of roughly about 1 μm squared has been recorded with 255 × 255 images within 96 seconds.

1. Introduction

Multilayer Zone Plates (MZPs) are basically an “optically thick” version of Fresnel Zone Plates (FZPs) [5, 6]; the fabrication using a sputter–slice technique – in our case pulsed laser deposition and slicing with a focused ion beam – allow for smooth outer most layers of width well below 10 nm and an optical thickness of several micro metre. Thus, the aspect ratio compared to FZPs based on lithography can be orders of magnitude larger, increasing efficiency even at high x-ray energies above 12.4 keV [3].

In recent years we have achieved sub-5 nm focusing in two dimensions at 7.9 keV [7]; also first imaging experiments have successfully been carried out [8]. Despite the focusing potential of outer most zones below 10 nm, resolution is limited to around 50 nm so far due to vibrations in the used versatile GINIX setup (“Göttingen Instrument for Nano-Imaging with X-rays”): This instrument has been designed for holography measurements with a resolution aim of about 100 nm [1, 2], with an emphasise on flexibility on that scale, not stability on the sub-10 nm scale.

Famous for its vivid and versatile construction, the GINIX setup will keep its basic design, but is currently upgraded for higher resolutions. On one end, sources of vibrations are located and removed; on the other end, the general purpose and tomography sample tower is exchanged with high resolution towers bringing optic (MZP or waveguide) and sample motorisation on a common base. In the next sections we introduce the new MZP tower and report on its commissioning. Current status of MZP fabrication is only touched, see [4] for more details. The imaging capabilities of the GINIX setup are summarised in section 5, before a short summary and outlook.
2. Sample Tower

The new MZP sample tower combines motorisation for optic and sample on a common optical bread board; the beam height is 125 mm above the breadboard. The sample stage consists of an x-y-stage (horizontal) M–686 by Physik Instrumente Karlsruhe (PI) and a z-stage (vertical) N–765 for alignment in the primary and focussed MZP beam; on top, a piezo scanner (P–733) allows for fast and accurate 2D scanning perpendicular to the MZP beam. The scan range is limited to 30 µm, but encoder values show excellent position accuracy of rms less than 0.2 nm for 1 µm scans faster than 1 Hz.

Alignment of the MZP is facilitated by three orthogonal stick-slip positioners by SmarAct (Oldenburg) for translations, and a piezo-driven Gimbal mount for rotations. See Tab. 1 and Fig. 1 for travel ranges and a photograph. Note that in this first iteration, robustness is prioritised over degrees of freedom; especially there are no sample rotations available, yet.

The piezo scanner allows for continuous 2D-movements with a servo cycle of 50 µs and is configured for horizontal forward-backward movements with intermediate vertical steps to fill the 2D area. During linear movements, the Eiger detector (Dectris AG, Dättwill) is triggered...
Figure 2. Continuous 2D scan: (left) encoder positions (vertical ROI) of a 1 µm/340 ms fast scan, showing a vertical rms accuracy better than 0.2 nm at 4 nm step size; (right) final STXM image of a Siemens star test pattern, taken with Eiger detector with frame frequency of 750 Hz. The field of view is about 1 µm.

for 750 Hz acquisitions. During commissioning (see following section), the field of view was set to a square of side length 1 µm, with 255 × 255 acquisitions during 96 s. The rather conservative overhead for acceleration and due to short exposure time of 1 ms was about 48% but could be reduced.

3. Commissioning Results
First, the sample tower itself was tested with the P10 beamline’s compound refractive lenses and a micro-focus at 13.8 keV. Gold colloids and semiconductor nanowires (InP) have been used as test samples. Afterwards, a Fresnel Zone Plate (FZP) with outermost zone widths of 30 nm – a courtesy of Christian David (Swiss Light Source) – was used to commission the MZP tower with a “gold standard” optic. Due to the large working distance of 34.5 mm, these experiments are less prone to collisions than with short-f MZPs. Also, on order sorting aperture (OSA) has been used to absorb higher diffraction orders.

2D raster scans in discrete stepping mode have been carried out on a Siemens star test pattern, alternating with CRL beam and FZP beam to find the overlap, and to find the FZP’s focal plane. These step-by-step scans have been complemented with the full continuous measurements, that allowed for the first time 255 × 255 raster positions to be taken within less than two minutes at the GINIX setup. Parts of the piezo scanner’s encoder positions and the Siemens star STXM image are shown in Fig. 2. Due to limited time during commissioning, the setup’s full potential could not be tapped, yet. The z-encoder shows an rms accuracy of better than 0.14 nm during continuous scans of 1 µm/340 ms per line, which is less than four per cent of the step size (4 nm in vertical direction). Complementary interferometric distance measurements between FZP and sample holder are not yet implemented, but under progress.

4. Current Multilayer Zone Plates
Recent optimisation in MZP fabrication, especially in laser parameters and material choice have enabled apertures of up to 15 µm, an increase by one order of magnitude compared to first lenses,
with a ML area coverage of about 96 per cent. The focal length at 8 keV has been increased to 0.5 mm, also a factor of ten larger than the first lens that was used as proof of concept for focusing. For more details on fabrication, see [3, 4].

One advantage of MZPs over lithographic FZP is the virtually unrestricted optical thickness. The initial glass fibres that are pulled to thicknesses of one to two micron posses a usable length of hundreds of micron; together with fine outer most zones of few nano metres, aspect ratios of several tens of thousand should be possible. This makes MZPs especially interesting for high energy applications: Neglecting absorption and volume effects, the ideal phase shift of $\pi$ between the two neighbouring layers of $\text{Ta}_2\text{O}_1$ and $\text{ZrO}_2$ is obtained for an optical thickness of 0.5 $\mu$m/keV$^2$.

5. Hard X-ray imaging at GINIX

The MZP–sample tower is a new high-resolution module for the flexible GINIX setup (Göttingen Instrument for Nano-Imaging with X-rays), operated by the AG Salditt at the coherence beamline P10 at PETRA III. GINIX is open for user proposals and highly flexible – nevertheless, a couple of routine experiments are offered, like nano-SAXS (scanning SAXS), propagation based (waveguide holographic) imaging extended to holographic tomography, and also WAXS and fluorescence measurements are supported. For more details, see [1, 2].

6. Summary and Outlook

A new dedicated MZP–sample tower has been commissioned; continuous scanning and triggering of the Eiger detector has been carried out with a frame rate of 750 Hz, allowing STXM measurements with $255 \times 255$ images to be taken in less than two minutes. The MZP tower is mobile and will be further evaluated at the GINIX and other instruments.

Acknowledgements

We gratefully thank Michael Sprung for excellent support and granting commissioning beam time, Christian David for the FZP. We thank Bastian Hartmann, Peter Luley, Peter Nieschalk (IRP), Florian Döring and Volker Radisch (IMP) for outstanding support in construction and fabrication, and Tim Salditt for stimulating and encouraging discussions. We acknowledge funding by Deutsche Forschungsgemeinschaft through SFB755 projects C12 and INF, and BMBF through grants 05K13MG4, 05K16MG2.

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