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DELIVERABLE REPORT

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D12.1
Fabrication of hard X-ray aberration correctors

Due date

M15

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DELIVERABLE DESCRIPTION

We report on the fabrication of hard X-ray aberration correctors to improve the performance of nanofocusing X-ray lenses by correcting the shape deviations that arise due to fluctuations and anisotropies during the manufacture of X-ray optics. After carefully characterizing the aberrations of the focusing X-ray lens, the tailored profile of a correction phase plate can be inferred. In this deliverable, we have developed the micro and nanofabrication processes for the production of the refractive phase plates by two-photon polymerization based 3D printing. The refractive phase plate is made of polymer, which introduces the required phase shift corrections while hardly introducing any extra absorption. The process parameters have been optimized to obtain the tailored aberration correction profiles. The refractive phase plates have been produced on multiple substrates such as silicon, silicon nitride and diamond membranes and they can be fabricated reliably and on-demand. The methods and procedures to easily align the focusing optics and the refractive phase plate are currently under development.

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INTRODUCTION

X-ray nanofocusing [1, 2] has become a key requirement for many X-ray techniques with broad applications in many fields of scientific research [3, 4]. The fabrication of X-ray nanofocusing optics requires the most advanced technologies such high-resolution e-beam lithography, state-of-the-art nanostructuring techniques and specialized thin-film methods. Any small fluctuations or process anisotropies in these techniques can cause shape deviations in the X-ray optical elements that will result in a significant reduction of its nanofocusing performance.

In the recent years, it has been shown that the aberrations in an X-ray optical element [5] can be corrected by a refractive phase plate that compensates its phase deviations. So far, the correction of aberrations in mirrors [6], refractive lenses [5] and diffractive multilayer optics [7] have been demonstrated. To date, several techniques such as LIGA, laser ablation, focused ion beam etching and additive manufacturing have been demonstrated for the fabrication of refractive phase plate correctors. In this context, the development of a reliable fabrication approach to produce the refractive phase plate for aberration correction is essential to exploit the full potential of X-ray nanofocusing methods and their scientific applications.

Here, we have developed and optimized a reliable and on-demand production of refractive phase plates, that is aberration correctors, by two-photon polymerization based 3D printing. In our case, the correction phase plate is made of polymer, which introduces the required phase shift correction while hardly introducing any extra absorption of the X-rays. The refractive phase plates can be produced in multiple types of substrates such as silicon, silicon nitride and diamond membranes according to the requirements of the X-ray experimental station. Thus, aberration correctors for nanofocusing X-ray optics in the hard X-ray energy range (6 to 15 keV) can now be routinely fabricated. Currently, the methods and procedures to simplify the alignment of the focusing X-ray optics and the refractive phase plate are under development.

FABRICATION METHODS

The steps for the production and implementation of the refractive phase correctors for nanofocusing X-ray optical elements is schematically depicted in Figure 1. Prior to the fabrication of the refractive phase plate, the wavefront of the X-ray focusing elements was obtained by coherent X-ray diffraction imaging, namely ptychography [8]. The reconstructed wavefield is then used to calculate the wavefront aberrations and the required phase correction. The thickness profile of the phase plate material is then obtained to prepare the actual design of the refractive phase plate corrector. The fabrication starts with the computer aided design (CAD) of the refractive phase plate required by the two-photon polymerization 3D printing system. The printing is done directly on the X-ray transparent membrane, which is located in the center of a thick frame, which acts as a holder. Finally, the fabricated element is implemented and aligned at the X-ray beamline to achieve the aberration-corrected X-ray nanofocusing.

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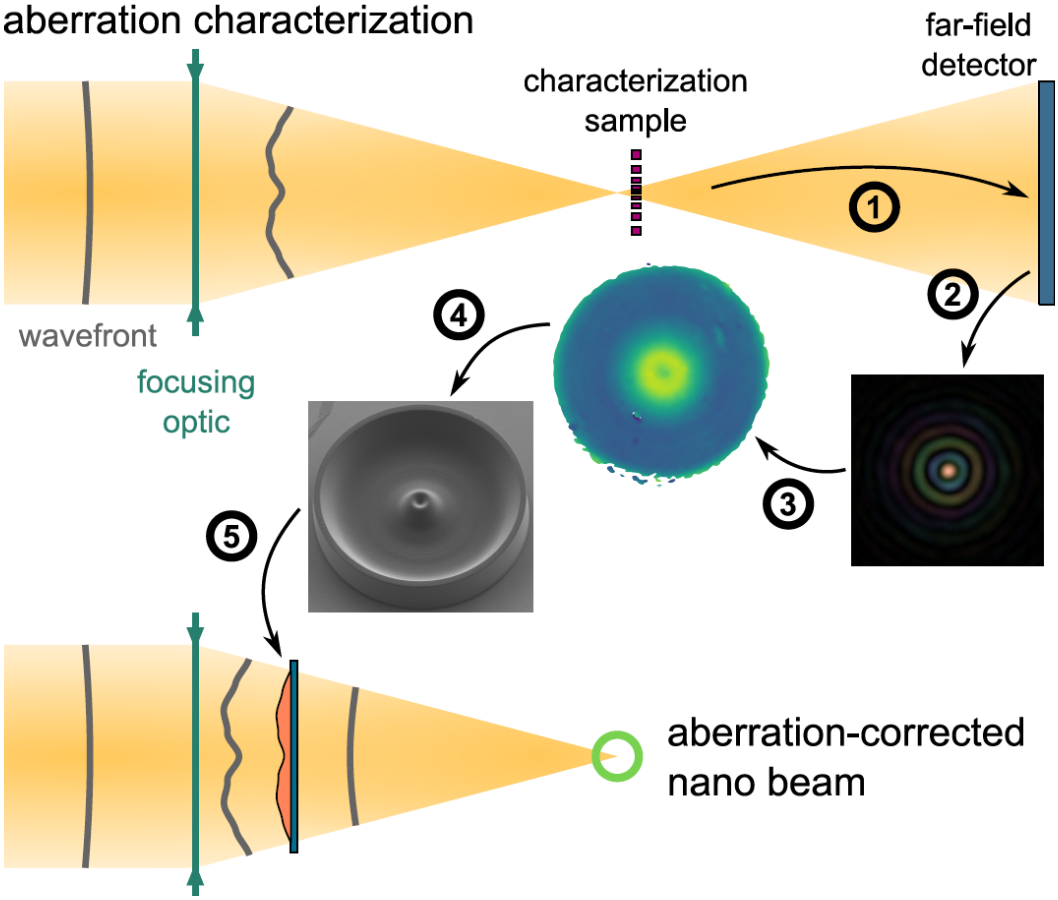

Figure 1: Schematic showing the principle of wavefront characterization with ptychography and consecutive aberration correction with a refractive phase plate. After steps 1 to 3 that allow the propagation of the wavefield to the exit pupil of the lens, the phase error can be calculated by fitting and subtracting a spherical wave from the aberrated wavefront. Then, the thickness profile that cancels the wavefront error by refraction in the phase plate material is calculated. In step 4, the refractive phase plate is fabricated by two-photon photon polymerization based 3D printing. In the final step 5, the refractive phase plate is aligned with X-ray optical element to obtain aberration-correction nanofocusing.

Wavefront Characterization

Prior to the fabrication of the refractive phase plate, the wavefront of the nanofocusing X-ray element is characterized via ptychography [8]. The focusing optic introduces some aberrations in the coherent X-ray beam due to shape inaccuracies. A strongly scattering sample (such as a Siemens star) is placed in the vicinity of the focal plane and several tens or hundreds of diffraction patterns from overlapping sample positions are acquired in the far-field by a spatially resolving detector. Then, the ptychographic reconstruction iteratively retrieves the complex-valued wavefield at the sample position from the recorded diffraction patterns. The wavefield at the sample position can be propagated to the exit pupil of the focusing optics. After that, the phase error is obtained by fitting and subtracting an ideal spherical wave from the aberrated wavefront at the exit pupil of the focusing

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optics. Finally, this phase error is transformed into the corresponding thickness profile that cancels it by refraction in the phase plate material. This thickness profile is then used to design and fabricate the refractive phase plate corrector tailored to the particular nanofocusing X-ray element.

Nano-Fabrication

In this work, the fabrication of the hard X-ray aberration correctors was realized by two-photon polymerization based 3D printing. This nanofabrication approach enables the production of submicrometer polymeric structures in a monomer resist by a non-linear optical process based on the simultaneous absorption of two photons. Such an approach has been successfully applied in the past for the production of X-ray focusing lenses [9]. At the Paul Scherrer Institut, the two-photon polymerization 3D printing was performed with a Photonic Professional GT2 system from Nanoscribe GmbH.

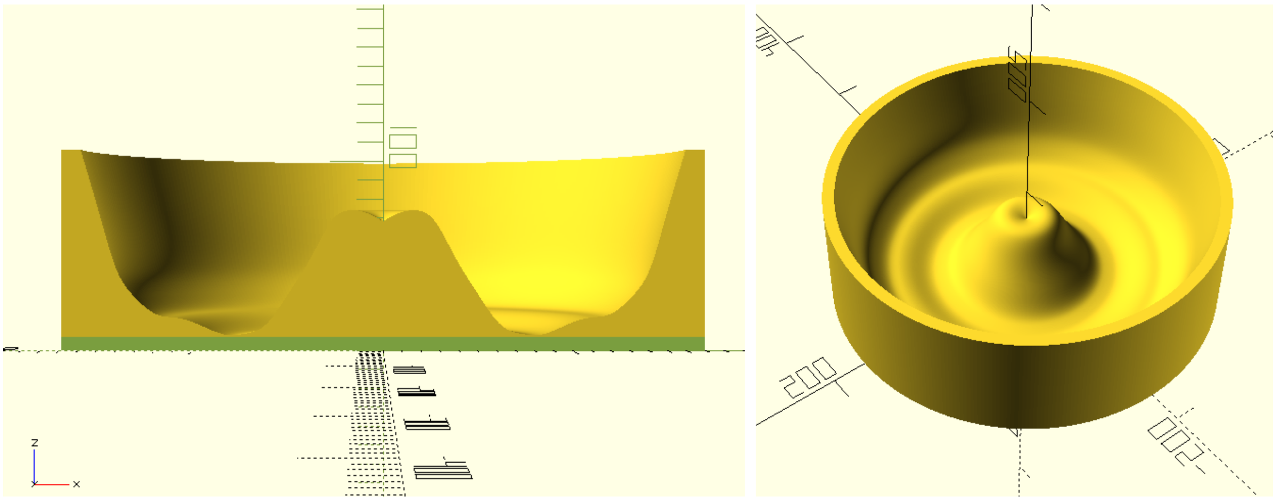

Figure 2: Volume of the refractive phase corrector generated in OpenSCAD taking into account the required thickness profile derived from the phase error in the nanofocusing X-ray optic measured by ptychography. (a) Thickness profile of the refractive phase plate with maximum height of 100 µm. (b) 3D representation of the refractive phase plate with a diameter of 340 µm.

The first step in the production of the hard X-ray aberration corrector is the preparation of the volume for the 3D printing using the thickness profile that has been derived via ptychography and that compensates the phase errors in the nanofocusing X-ray optic. Typically, the circular symmetry of the X-ray lens is used to reduce the complexity in the production of the refractive phase plate, which is also produced with circular symmetry. In addition, this choice reduces substantially the complexity during the alignment of the nanofocusing X-ray optic and the aberration corrector plate without compromising the effectiveness of the aberration correction. The volume of the refractive phase corrector is generated using an open-source CAD software, OpenSCAD, as shown in Figure 2. In this particular case, the aberration corrector has a diameter of 320 µm and requires a polymer resist thickness ranging from 0 to 100 µm. A thin polymer base of 10 µm was added to the volume of the refractive phase plate to improve the adhesion to the substrate surface of the 3D printed structures. From the required phase shift correction, the polymer thickness was calculated taking into account its chemical formula, $C_{14}H_{18}O_7$, and its density of 1.2 g/cm^3 as well as a photon energy of 10.0 keV at which the nanofocusing X-ray lens is used for this particular case.

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After the design preparation, the two-photon polymerization based 3D printing was realized using the commercial Nanoscribe IP-S resist in dip-in lithography mode with a 25× objective lens. This choice of parameters guarantees the fabrication of the refractive phase plate with sufficient resolution as well as ensuring very smooth surfaces of the 3D printed volume. Before the exposure, the substrate surfaces were, in some cases, coated with 20 nm Cr or treated with an Oxygen plasma to enhance the adhesion of the 3D printed structures during the development step. The refractive phase plate were produced in silicon, silicon nitride and diamond thin membranes (thickness ranging from 250 nm up to 10 micrometer). The parameter of the exposure, such as the laser power and speed, were optimized for each substrate material. The two-photon polymerization 3D printing of a refractive phase plate typically lasted from 2 to 10 hours, depending on the actual size of the tailored structure. After the exposure with the Photonic Professional GT2 system (Nanoscribe GmbH), the development was performed by immersing the exposed chip in PGMEA solution for 15 to 30 minutes. The samples were led to dry slowly under laminar flow and were then ready for characterization.

CHARACTERIZATION AND RESULTS

After the fabrication, the characterization of the refractive phase plates was realized by optical and scanning electron microscopy. After that, the aberration correctors were implemented and aligned with the nanofocusing X-ray optics.

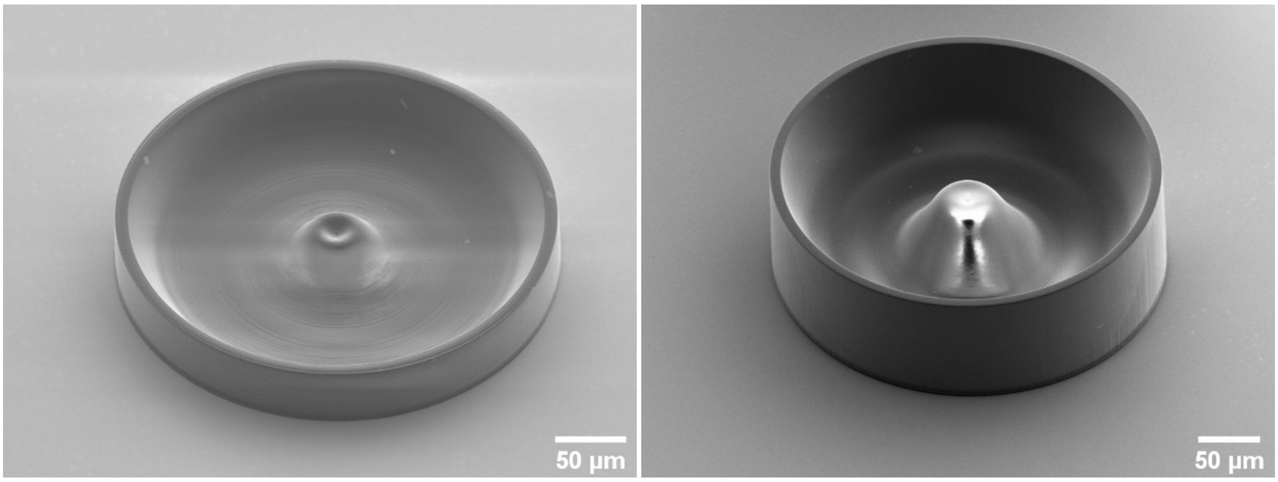

Figure 3: Example of two refractive phase plates produced by two-photon polymerization based 3D printing. After the fabrication, the aberration corrector elements were inspected by scanning electron microscopy. Refractive phase plate with a diameter of 300 µm and a maximum height of 50 µm (left). Refractive phase plate with a diameter of 320 µm and a maximum height of 100 µm (right). This design corresponds to the 3D volume depicted in Figure 2.

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Scanning Electron Microscopy Inspection

Figure 3 shows scanning electron microscopy images of two different refractive phase plates on a silicon membrane substrate. It can be seen that the required 3D volume of polymer resist has been produced accurately to match the thickness profile of the aberration corrector. The surface of the refractive phase plate has a smooth finish that is important to introduce the exact phase shift correction in the wavefront exiting the nanofocusing X-ray optic. On the other hand, Figure 4 shows scanning electron microscopy images of the same refractive phase plate design produced on two different substrates, namely on silicon (left) and diamond (right) membranes.

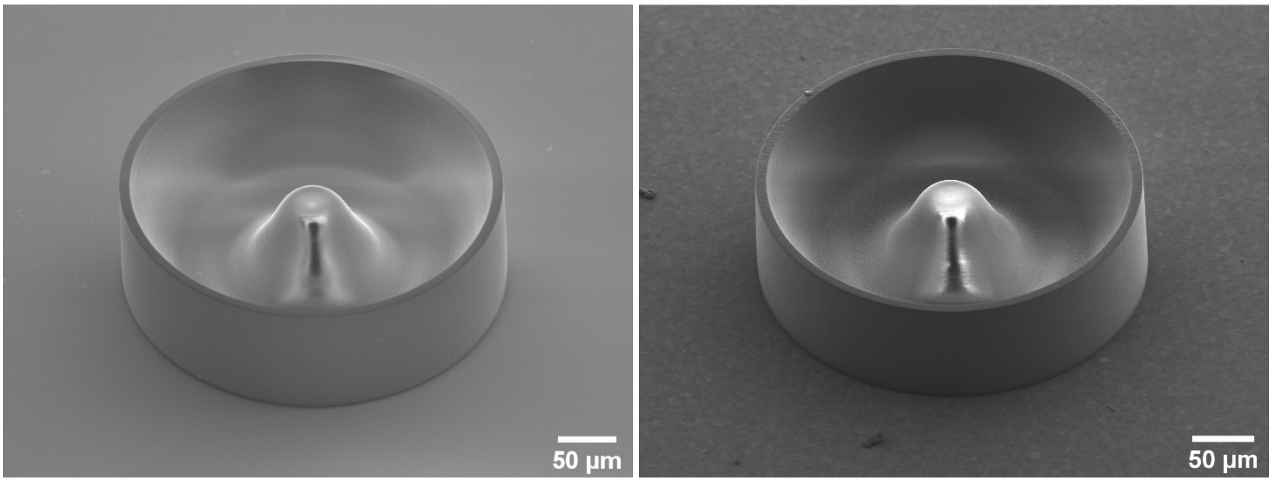

Figure 4: Example of the same refractive phase plate design fabricated on silicon (left) and diamond (right) membrane substrates. The aberration corrector has a diameter of 310 µm and a height of 100 µm.

Implementation of the Refractive Phase Plates

After the fabrication and characterization the tailored refractive phase plate in the laboratory, they were implemented with the nanofocusing X-ray optics at a synchrotron beamline. In this particular case, the nanofocusing X-ray optics consisted in an assembly of 25 beryllium lenses. A photon energy of 10 keV was used. Figure 5 (top) displays an aberrated X-ray focus reconstructed by ptychography before the introduction of the refractive phase plate. To facilitate the alignment of the hard X-ray corrector, the substrate chip containing the refractive phase plate was glued to a disk-shaped frame holder. Such a holder is especially manufactured to be easily aligned with the other components nanofocusing X-ray optics. It was designed with a square recession which was slightly larger than refractive phase plate substrate, and a micro manipulator has been assembled in the laboratory to position the refractive phase plate in the center of the disk holder with an accuracy better than 5 µm. Such pre-alignment step greatly reduces the complexity of the final alignment at the synchrotron beamline. The combination of the nanofocusing X-ray optic and the hard X-ray corrector delivered an aberration free X-ray focus with an approximated size of 150 nm (FWHM), as shown in Figure 5 (bottom).

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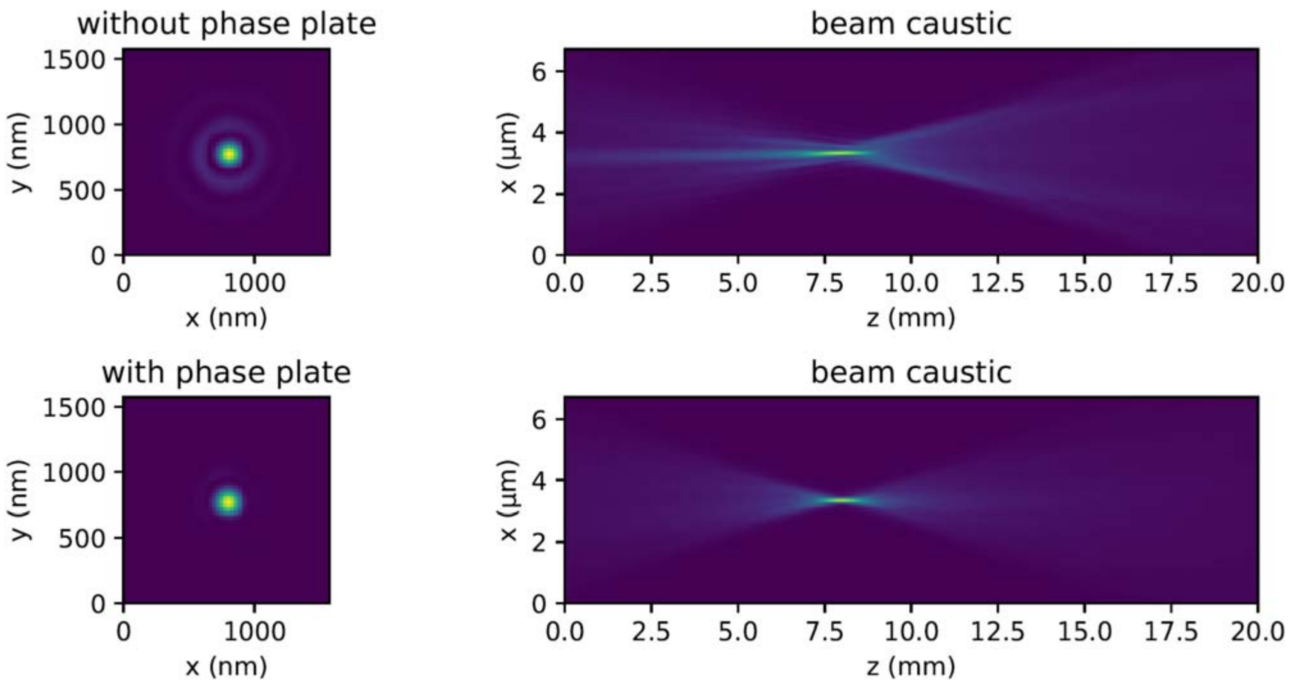

Figure 5. Example of X-ray nanofocusing at 10 keV combining 25 beryllium lenses (r = 50 µm) with a hard X-ray aberration corrector made by two-photon polymerization based 3D printing. (top) Aberrated X-ray focus in absence of the refractive phase plate. (bottom) Aberration-free X-ray focus after the introduction of the refractive phase plate.

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