Rapid aberration correction for diffractive X-ray optics by additive manufacturing

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Abstract: Diffraction-limited hard X-ray optics are key components for high-resolution microscopy, in particular for upcoming synchrotron radiation sources with ultra-low emittance. Diffractive optics like multilayer Laue lenses (MLLs) have the potential to reach unprecedented numerical apertures (NA) when used in a crossed geometry of two one-dimensionally focusing lenses. However, minuscule fluctuations in the manufacturing process and technical limitations for high NA X-ray lenses can prevent a diffraction-limited performance. We present a method to overcome these challenges with a tailor-made refractive phase plate. With at-wavelength metrology and a rapid prototyping approach we demonstrate aberration correction for a crossed pair of MLL, improving the Strehl ratio from 0.41(2) to 0.81(4) at a numerical aperture of 3.3 × 10⁻³. This highly adaptable aberration-correction scheme provides an important tool for diffraction-limited hard X-ray focusing.

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1. Introduction

Diffractive X-ray optics like multilayer Laue lenses (MLLs) have the potential to achieve numerical apertures (NA) in the hard X-ray regime that would enable focal spot sizes down to 1 nm [1] with current fabrication techniques [2,3]. A low-aberration X-ray lens with such high NA would enable scanning X-ray microscopy and full-field imaging experiments with unprecedented resolution. Efficient focusing requires to fulfill Bragg’s law over the whole aperture of the multilayer, leading to a wedged architecture [4]. However, minuscule inaccuracies in the layer deposition, lamella cutting, and also final lens alignment at the experiment can introduce optical aberrations [5]. While some errors can be compensated within the manufacturing process if well characterized, it was found by Chapman and Bajt that a so called ‘oblique spherical aberration’ prevents a diffraction-limited performance of a crossed pair of one-dimensionally focusing MLLs to NA<0.016 [5]. The resulting wavefront error could be overcome by an additional optical element, such as a corrective phase plate [6]. Recent experiments showed focusing below 10 nm for MLLs [7] and below 20 nm for a multilayer zone plate [8]. Although the achieved focal spot sizes correspond to the NA of the lens, the wavefield is affected by aberrations and the focusing...
performance is not diffraction limited. With a suitable at-wavelength metrology technique like ptychography [9] these errors can be characterized and corrected for with an aberration-correcting refractive phase plate. As the lens alignment process will get more critical with increasing NA, a reproducible lens alignment can be challenging. A tailor-made optical element with a sufficiently short manufacturing time, implemented within a short time span of the experiment, could help to resolve the challenge of diffraction-limited X-ray focusing at high NA beyond $1 \times 10^{-3}$.

In this work, we demonstrate an aberration-correction approach for X-ray optics via 3D printing of a refractive phase plate made-to-measure. With the availability of commercial 3D printers using two-photon polymerization, high-quality X-ray optical components [10–12] can be manufactured on site in only a few hours. We show that a complex three-dimensional phase plate structure, capable to correct complicated high-order aberrations in current diffractive X-ray optics, can be produced with high fidelity. With printing heights of several millimeters even optically thick phase plates can be manufactured. This opens up the possibility to correct aberrations in cutting-edge X-ray optics and achieve diffraction-limited focusing performance for spot sizes below 30 nm, where optics often are hampered by manufacturing tolerances and a difficult alignment process [13]. Thus, aberrations might vary in between production runs and even the same optical system might show different aberrations if the alignment process can not be repeated faithfully. While adaptable aberration-correction schemes were demonstrated in the past [14,15], they require significant instrumentation or are limited to specific aberration types. A tailor-made phase plate on the other hand, which can be rapidly printed at low cost as a consumable, is very compact, can be inserted during the experimental campaign, and is not limited to certain aberrations. A turnover time between initial optics characterization and ready to use device can be short enough to correct optics with high NA for diffraction-limited performance within the time constraints of typical experimental campaigns at synchrotron radiation facilities.

2. Method

2.1. Experimental configuration

The PtyNAMi instrument [16] at beamline P06 at DESY provides a platform for high-resolution X-ray imaging on the nanometer scale with the flexibility to employ various X-ray optical configurations with diverse focusing optics such as nanofocusing refractive X-ray lenses, Fresnel zone plates, or MLLs. Here, we use a pair of MLLs [17] in a crossed geometry with a focal length $f = 12.150$ mm at an X-ray energy of $E = 16.2$ keV and wavelength $\lambda = 0.765$ to focus the beam to below 30 nm in the focal plane. The two MLLs were mounted individually on two hexapods (custom SmarAct SmarPods), each providing six degrees of freedom to align the MLLs relative to each other and the experiment. The multilayer consisted of 3000 four-layer periods of Mo/C/Si/C [18] with a fixed C layer thickness of 0.4 nm and a 50:50 ratio between the Mo absorber and the C/Si/C spacer. The innermost zones are $d_{\text{in}} = 15.48$ nm thick with a distance of $r_{\text{in}} = 30.03$ µm to the optical axis. For the outermost zones $d_{\text{out}} = 5.78$ nm and $r_{\text{out}} = 80.50$ µm, which results in a theoretical geometric aperture of 50.47 µm. The MLLs were fabricated in the same deposition run, yielding in theory the same multilayer and thus also the same focal length. However, by selecting MLLs from different regions across the wafer, the focal length can be tuned slightly due to varying deposition rates across the substrate. The MLL lamellae cut out of the multilayer had a thickness of 10 µm and a width between 150 µm and 200 µm. Two of them were mounted in a crossed geometry. The vertically focusing MLL (vMLL) was mounted at the nominal focal length $f_{\text{vMLL}} = 12.15$ mm upstream of the focal plane. For the horizontally focusing MLL (hMLL) we determined the optimal position to be 45 µm upstream from the vMLL, yielding a focal length of $f_{\text{hMLL}} = 12.195$ mm.

The experimental setup is depicted in Fig. 1(a). The geometrical aperture was defined by a pair of slits to 40 µm × 40 µm, resulting in a NA of $3.29 \times 10^{-3}$ for the vMLL and $3.28 \times 10^{-3}$
for the hMLL. A rectangular order-sorting aperture (OSA) with a size of 20 μm × 20 μm was positioned 3 mm upstream of the focus to clean the beam.

**Fig. 1.** Experimental setup for wavefield reconstruction. (a) X-rays with \( E = 16.2 \) keV (selected by a Si-111 monochromator) were collimated with a pair of slits to the MLL aperture. The horizontally focusing MLL (hMLL) was positioned 45 μm in front of the vertically focusing MLL (vMLL). The phase plate (PP) was placed 3 mm downstream from the vMLL and a square-shaped order-sorting aperture (OSA) was positioned 3 mm upstream of the focal plane. The test sample for beam characterization was 0.28 mm out of focus in order to increase the probe size on the sample. A photon counting pixel detector was collecting far-field diffraction patterns at a distance of 3.5 m downstream. (b) An example far-field diffraction pattern, as used for ptychographic phase retrieval. The residual intensity from the direct beam and the individual diffraction orders of each MLL were masked numerically. The scale bar represents 0.3 nm\(^{-1}\). (c) Reconstructed object phase shift of the Siemens star test pattern. The squared box indicates the scanned area. (d) Reconstructed complex probe function at the position of the test pattern. Scale bars in (c) and (d) correspond to 1 μm.

### 2.2. Optics characterization

For an initial wavefield characterization, which will be used to design the corrective phase plate, a coherent scanning X-ray diffraction method called ptychography [9] was utilized. By scanning an area of 2 μm × 2 μm on a grid of 21 × 21 scan points (step size: 100 nm), we collected a set of 441 far-field diffraction patterns from a Siemens star test object from NTT-AT with 50 nm smallest features in a 500 nm thick Tantalum layer. It was placed 0.28 mm out of focus and data was recorded on a Dectris Eiger X 4M photon-counting pixel detector [19] with a pixel size of 75 μm at a distance of 3.5 m downstream from the focal plane. One of the diffraction patterns is shown in Fig. 1(b), where the central square area is the divergent beam originating from diffraction of the two crossed MLLs. Despite the use of an OSA, stripe features next to the central cone can be seen. On the right side they originate from diffraction of the vMLL only, on the top from diffraction of the hMLL only. The undiffracted beam can be seen on the top right corner. These residual intensities were masked during data processing and are not considered for phase retrieval. As the test sample was placed slightly out of focus, parts of the Siemens star structure are directly visible in the central cone of the divergent beam with strong phase contrast. This out-of-focus placement enlarges the illumination on the sample to roughly 0.7 μm × 0.7 μm, so that a high probe overlap between scan points is achieved with a moderate step size of 100 nm. An area of 2 μm × 2 μm, highlighted by the orange square in Fig. 1(c), was
raster scanned with a dwell time of 0.5 s per position. The 441 diffraction patterns were cropped to a size of 512 px × 512 px, as shown in Fig. 1(b), resulting in a pixel size of 6.98 nm for the reconstructed data. Ptychographic phase retrieval with the ePIE algorithm [20] was performed to characterize the wavefield [10,21–23]. The ePIE algorithm performed 500 iterations in total with brute-force position refinement [24] in order to correct for positioning errors and instabilities. As a result, both the complex-valued object and probe function at the sample plane are reconstructed. The object phase shift of the Siemens star pattern is shown in Fig. 1(c) and the complex-valued wavefield of the probe is shown in Fig. 1(d).

2.3. Phase plate design and fabrication

In a next step, the wavefield was numerically propagated to the desired position for the refractive phase plate, in this case to a plane 3 mm downstream of the vMLL, as indicated in Fig. 1(a). The location was chosen so that the fabricated phase plate could be mounted and positioned freely, without any manipulation of the already aligned MLL pair. To retrieve the wavefield at this plane, the reconstructed wavefield from ptychography, shown in Fig. 1(d), was numerically backpropagated with the Fresnel-Kirchhoff diffraction integral [25]. Initially, a spherical wave with the nominal radius of the focal plane distance was subtracted [26]. As the MLL aperture is square, the circular Zernike polynomial basis $Z_j^i$ was used to compose a new set of Zernike-like basis functions $S_j^i$ on a square aperture through the Gram-Schmidt orthonormalization process [27], where the Zernike unit circle circumscribed the entire square aperture. The residual wavefront error, compensated for tilt and defocus, can be retrieved by fitting the first 4 Zernike-like basis functions to the wavefield. It is shown in Fig. 2(a) and the considered region was selected by a mask based on an intensity threshold above 3 % of the maximum intensity. To model the phase error to be corrected by the phase plate we fitted the first 153 Zernike-like basis functions to the aberrated wavefield. The number was chosen to cover basis functions up to $S_{16}$ to accurately model the phase error and to remove high-frequency features visible in Fig. 2(a). The modeled phase error for compensation by the phase plate is shown Fig. 2(b), where in addition the first 6 Zernike-like terms were dropped. This implies that basic aberrations of tilt, defocus, and basic astigmatism were omitted. These phase errors originate either from data handling, e.g. choosing an artificial optical axis by cropping the diffraction patterns, or can be corrected for by adjusting the vMLL to hMLL distance and angle. In this way we reduced the overall printing height of the phase plate to a minimum, concentrating on aberrations that cannot be corrected for by lens adjustments alone. To fill the masked areas and to expand the phase plate beyond the MLL aperture in order to avoid scattering effects on its edge, the wavefield shown in Fig. 2(b) was convolved with a Gaussian kernel, where only the masked parts were replaced. The design of the printed phase plate height is shown in Fig. 2(c), where the orange square highlights the geometrical MLL aperture.

The structure was printed on a silicon nitride membrane with 250 nm thickness that can be mounted without manipulating the already aligned MLL pair within the PtyNAMI instrument. It was printed on a Nanoscribe Photonic Professional GT with the IP-S resist in dip-in lithography mode and a 25× objective with NA = 0.8. The height profile was estimated from the chemical composition of the acrylic resin (C$_{14}$H$_{24}$O$_7$) and its cross-linked density of 1.2 g cm$^{-3}$, resulting in an assumed refractive index decrement $\delta = 1.007 \times 10^{-6}$ at $E = 16.2$ keV [28]. These assumptions are based on previous experiences on designing x-ray optics with this resin [10,11,29–31]. With a theoretical attenuation coefficient $\mu = 1.006$ cm$^{-1}$ the phase plate transmission is 99.6 % within the optically relevant area shown in Fig. 2(c). The printing was performed with 100 nm slicing and hatching to produce a smooth surface. Larger values might be used to increase printing speed. The final phase plate with additional stabilizing elements at the tallest features is shown in Fig. 2(d–f). The printing time for this structure with a volume of $160 \times 10^3 \mu m^3$ was approximately 45 min.
Fig. 2. Wavefront error and phase plate design. (a) Residual wavefront error 3 mm downstream of the vMLL with a root-mean-square deviation (RMSD) of 0.21 λ. (b) Modeled phase error to be compensated by the phase plate with 0.15 λ RMSD. (c) The height profile of the phase plate is obtained by expanding the wavefield via convolution. Additional markers for orientation and alignment are added. The optically active area is marked by the orange square box. (d–f) Scanning-electron-microscope images of the printed polymer phase plate. Additional stabilizing elements were added on the tallest phase plate structures. The scale bars in all subfigures represent 10 μm.
The phase plate is aligned in the X-ray beam with linear translations in $x$, $y$, $z$, and a rotation around the optical axis $z$ with the help of the printed alignment guides visible in Figs. 2(d–f) and a scintillator-coupled high-resolution X-ray microscope (Optique Peter with 10x/NA 0.4 objective, 8 $\mu$m thick LSO:Tb scintillator and PCO 4000 camera). Typically, additional refinement of the phase plate position with at-wavelength metrology is not required, as the transverse position and rotation can be adjusted with the high-resolution X-ray microscope within 2 $\mu$m by the alignment guides. All results presented here were achieved with this alignment process. Rotations around $x$ and $y$ were not implemented in the setup. Assuming an angular misalignment of 1° and a mean thickness of 43 $\mu$m for the phase plate results in a deviation of 0.7 $\mu$m. As features of the phase plate are much larger, as shown in Fig. 2, we neglected the influence on wavefront correction.

No further improvement but a degradation in the root-mean-square deviation (RMSD) of the wavefront error was observed by scanning the phase plate position laterally in 1 $\mu$m steps for $\pm 2$ $\mu$m in both directions and evaluating the aberrations via ptychography as described in Section 2.2.

3. Results

Finally, the aberration-corrected MLL pair is quantitatively characterized by ptychography in the same fashion as before. A comparison of the residual wavefront error, calculated by removing tilt and defocus from the measured wavefield, is presented in Fig. 3(a) and Fig. 3(b) without and with the phase plate, respectively. Especially the strong errors in the lower left and upper right corner were reduced, as well as the strength of vertical stripes. The RMSD wavefront error reduced from 0.27 $\lambda$ without to 0.17 $\lambda$ with the phase plate. The strength for the fitted Zernike-like basis functions, excluding piston, tilt, and defocus, are shown in Fig. 3(c). Basic coma ($S_{3}^{-3}$, $S_{3}^{+3}$) and oblique trefoil ($S_{3}^{1}$) are reduced. Higher order aberrations appear slightly enhanced. A reduction in astigmatism, represented by $S_{2}^{-2}$ and $S_{2}^{+2}$, is clearly visible. Since we omitted basic astigmatism for the calculation of the phase plate as described in Section 2.3, this reduction can not be explained by the phase plate alone. As six hours lie between the measurement of Fig. 3(a) and Fig. 3(b) the MLL alignment might have changed slightly, potentially influenced by mounting the phase plate. To demonstrate that the phase plate still performs as intended, the phase difference between Fig. 3(a) and Fig. 3(b), modeled by the best fitting 153 Zernike-like basis functions, is shown in Fig. 3(d). Evaluating this phase shift against the design values of the phase plate in Fig. 3(e) shows good agreement, as can be seen by the phase difference between them shown in Fig. 3(f). Large parts of the wavefront are flat, except for the mentioned basic astigmatism and two horizontal stripe features in the lower part of the wavefront. Overall, the printed features of the phase plate match with the aberrations of the MLL and correct phase errors on the intended spatial frequencies.

When looking at the horizontal and vertical beam caustic for the aberrated MLL pair in Figs. 4(a) and 4(b), respectively, strong asymmetric tails are visible upstream and downstream of the focal plane that is marked by the dashed line. The X-ray beam further away from focus has strong fluctuations in intensity, visible by the bright and dark stripes in the beam caustic in that region. These intensity variations result from the horizontal and vertical stripe features in the phase error, shown in Fig. 3(a). Within the focal plane, shown in Fig. 4(c), this manifests in strong speckles surrounding the focal spot. After the phase plate was installed, the tails of the beam caustic in Figs. 4(e) and 4(f) appear more homogeneous and speckles around the focal plane in Fig. 4(g) are suppressed. Out of focus the X-ray beam also achieves a more homogeneous intensity distribution, shown in Fig. 4(h), in comparison against the uncorrected case in Fig. 4(d). The higher-frequency phase stripes visible in both Figs. 3(a) and 3(b) still cause higher-frequency intensity stripes, visible in both Fig. 4(d) and Fig. 4(h). If the phase plate design would include higher-frequency features, some of these aberrations could potentially also be corrected for.
Fig. 3. Aberration-corrected wavefront. (a) Residual wavefront error 3 mm downstream of the vMLL with a root-mean-square deviation (RMSD) of 0.27 λ, measured four days after the initial characterization shown in Fig. 2(a), prior to any correction. (b) Residual wavefront error 3 mm downstream of the vMLL with 0.17 λ RMSD, after the phase plate has been aligned to compensate aberrations. (c) Comparison of the amplitudes of Zernike-like basis functions. The base term notation $S_{mn}$ follows the common Zernike polynomial notation $Z_{mn}$, starting here with astigmatism ($S_{22}^{2}$, $S_{22}^{1}$) and ending with pentafoil ($S_{55}^{5}$, $S_{55}^{1}$). The gray marked terms were not considered in the phase plate design. (d) Phase shift difference between (a) and (b), modeled by Zernike-like basis functions with 0.22 λ RMSD. (e) Designed phase shift of the phase plate for direct comparison, representing the area inside the orange square in Fig. 2(c) and 0.18 λ RMSD. The outer boundaries from (d) are traced in black for visual guidance of the phase plate position within the wavefield. (f) Phase difference between (d) and (e), showing components in the wavefield not originating from the phase plate with 0.17 λ RMSD. The scale bars in (a–b) and (d–f) represent 10 µm.
A precise phase plate alignment would be required, as a mismatch by half of the aberration frequency would actually enhance phase errors.

![Fig. 4. Beam caustic calculated from ptychography reconstructions.](image)

(a) Horizontal beam caustic for the aberrated MLL pair. (b) Vertical beam caustic for the aberrated MLL pair. (c) Focal spot created by the aberrated MLL pair. The focal plane is indicated by the dashed line in (a) and (b). (d) Beam profile 1 mm downstream of the focus (uncorrected). (e) Horizontal beam caustic for the MLL pair with phase plate. (f) Vertical beam caustic for the MLL pair with phase plate. (g) Focal spot formed by the MLL pair with phase plate. The focal plane is indicated by the dashed line in (e) and (f). (h) Beam profile 1 mm downstream of the focus (corrected). The scale bars in z-direction represent 100 µm and in x/y-direction 200 nm.

In horizontal direction an improvement in the full-width at half-maximum (FWHM) focal spot size from 33 nm down to 25 nm was observed, as shown in Fig. 5(a). For the vertical direction the FWHM spot size remained unchanged at 27 nm. In Fig. 5(c) the radially integrated relative intensity is shown, which gives an indication of the relative intensity within a certain radius compared to the total X-ray beam intensity. The aberrations of the uncorrected MLL pair cause an intensity spread, whereas the aberration-corrected MLL closely follows an ideal lens. As a result, the Strehl ratio improved from 0.41(2) to 0.81(4) due to aberration correction achieved with the refractive phase plate, transforming the MLL pair into a close to diffraction-limited optical system. The ratio was determined by comparing the reconstructed on-axis beam intensity in the focal plane (cf. Figure 5(g)) with an ideal focus of a square-aperture lens, represented by a sinc² function. Due to the low spatial sampling with a pixel size of 6.98 nm we used the peak value of a fitted Gaussian to the central speckle of the data shown in Fig. 5(g) to calculate the on-axis intensity. The intensities in both cases were normalized to the total intensity within a 200 nm radius around the optical axis.
Fig. 5. Focusing performance. (a) Horizontal focal spot size (FWHM) for the MLL without (blue line) and with (orange line) phase plate in comparison to an ideal sinc² profile (green line). (b) Vertical focal spot size (FWHM) for the MLL without (blue line) and with (orange line) phase plate in comparison to an ideal sinc² profile (green line). (c) Radially integrated intensity distribution up to a radius of 200 nm for the MLL without (blue line) and with (orange line) phase plate in comparison to an ideal lens with a sinc² profile (green line).

4. Discussion and conclusion

In summary, we have demonstrated the rapid aberration correction of X-ray optics from the initial lens characterization, over the device manufacturing with commercially available 3D printing technology, to an aberration-corrected lens achieving a FWHM focal spot size of 25 nm × 27 nm with close to diffraction-limited performance at a Strehl ratio of 0.81(4), within the time frame of an experimental campaign at a modern X-ray synchrotron radiation facility. With typical printing times below 5 hours for a volume of $1 \times 10^6 \mu m^3$ and low computational requirements for the phase-plate design, an implementation over night or even within a few hours appears feasible if a suitable 3D printer is available on site. The typical printing resolution of 200 nm allows to correct also higher-frequency phase variations. As the printing height can exceed a few millimeters, in theory large phase errors can be compensated. At a photon energy of 16.2 keV with an assumed phase plate thickness of 2 mm this would correspond to over 26 $\AA$. In these extreme cases the phase plate might become a thick optical element and a placement upstream of the MLL will be beneficial. Not only can the short working distance for high-NA MLLs pose a problem with the implementation between MLL and focal plane, also the X-ray beam will converge significantly within the phase plate due to its NA, drastically impacting the phase plate design.

The compact size of the phase plate and its low production cost allow for a fast implementation at the instrument and, as demonstrated here, without the need to manipulate the existing optics and a loss of alignment. Hence, even high-NA optics that might not show a reproducible aberration pattern due to variations in the fabrication process and alignment tolerances can be corrected for the specific application. In addition, physical limitations of crossed MLLs for $\frac{\lambda}{NA} > 0.016$ [5] can be overcome by this approach, enabling diffraction-limited X-ray focusing towards 1 nm spot sizes in the future.

An X-ray optical degradation of the polymer structure itself could not be observed, even when using the phase plate repeatedly over several beamtimes at an intensity of $1 \times 10^8$ ph/s impinging on the phase plate. However, we could observe an optical discoloration when the printed polymer phase plates are used at X-ray free-electron laser (XFEL) facilities such as LCLS or EuXFEL with pulse energies around 2 mJ. Although no change in X-ray optical performance,
assessed by the focal spot intensity throughout the experiment, could be observed, we note that the silicon nitride membrane is prone to break in XFEL use, which we attribute to induced stress due to repeated heat load cycling of the polymer structure adhering to the membrane. Choosing a different support structure such as a diamond or silicon membrane should resolve this issue. As the phase plate is a static optical element it can not account for shot-to-shot phase variations of the impinging XFEL beam.

The evolution of 3D printing techniques in recent years provides many possibilities to manufacture tailor-made structures for X-ray optics [10,11,29–31] on short time scales and at low cost. Optical properties, surface quality, and radiation resistance are suitable for use at modern X-ray synchrotron radiation facilities and potentially even XFELs. Thus, refractive X-ray optics and especially corrective phase plates could become widely available as a consumable optical element to improve focusing and imaging performance or to implement experiment specific optical configurations.

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**Disclosures.** A.K. is currently employed at XRnanotech GmbH, which can manufacture phase plates presented in this work.

**Data availability.** Data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon reasonable request.

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