nffa.eu
PILOT 2021 2026

DELIVERABLE REPORT

WP12 JA2 - X-ray Wavefront Metrology, Correction and Manipulation

D12.4

First user experiment with corrected hard X-ray nanobeam

Due date
M30

This initiative has received funding from the EU's H2020 framework program
for research and innovation under grant agreement n. 101007417, NFFA-Europe Pilot Project

PROJECT DETAILS

PROJECT ACRONYM	PROJECT TITLE
NEP	Nanoscience Foundries and Fine Analysis - Europe|PILOT
GRANT AGREEMENT NO:	FUNDING SCHEME
101007417	RIA - Research and Innovation action
START DATE	
01/03/2021	

WORK PACKAGE DETAILS

WORK PACKAGE ID	WORK PACKAGE TITLE
12	JA2 – X-ray Wavefront Metrology, Correction and Manipulation

WORK PACKAGE LEADER

Dr. Christian David (PSI)

DELIVERABLE DETAILS

DELIVERABLE ID	DELIVERABLE TITLE
D – 12.4	First user experiment with corrected hard X-ray nanobeam

DELIVERABLE DESCRIPTION

We report on a first user experiment at beamline P06 at PETRA III with the newly developed and integrated compound refractive lens focusing unit with integrated aberration correction by a refractive phase plate. The updated phase plate kinematic as well as at-wavelength beam characterization available to users is presented.

DUE DATE	ACTUAL SUBMISSION DATE
M30 – 31/08/2023	31/08/2023

AUTHORS

Dr. Frank Seiboth (DESY)

This initiative has received funding from the EU's H2020 framework program for research and innovation under grant agreement n. 101007417, NFFA-Europe Pilot Project

PERSON RESPONSIBLE FOR THE DELIVERABLE

Dr. Frank Seiboth (DESY)

NATURE

☒ R - Report

☐ P - Prototype

☐ DEC - Websites, Patent filing, Press & media actions, Videos, etc

☐ O - Other

DISSEMINATION LEVEL

☒ P - Public

☐ PP - Restricted to other programme participants & EC: (Specify)

☐ RE - Restricted to a group (Specify)

☐ CO - Confidential, only for members of the consortium

This initiative has received funding from the EU's H2020 framework program for research and innovation under grant agreement n. 101007417, NFFA-Europe Pilot Project

REPORT **DETAILS**

ACTUAL SUBMISSION DATE	NUMBER OF PAGES
31/08/2023	9 (right-click and select "update the field")

FOR MORE INFO PLEASE CONTACT

Frank Seiboth
Center for X-ray and Nano Science
CXNS
Deutsches Elektronen-Synchrotron
DESY
Notkestr. 85
22607 Hamburg
Germany

email: frank.seiboth@desy.de

VERSION	DATE	AUTHOR(S)	DESCRIPTION / REASON FOR MODIFICATION	STATUS
1	14/08/2023	Frank Seiboth		Draft
2	21/08/2023	Frank Seiboth	Input from co-authors	Draft
				Choose an item.
				Choose an item.
				Choose an item.
				Choose an item.
				Choose an item.

CONTENTS

INTRODUCTION

Compound refractive lenses (CRLs) are widely used at synchrotron radiation facilities for X-ray beam shaping [1, 2] and focusing [3]. They are mainly made by pressing a parabolic lens profile into a thin foil of aluminum or beryllium via a coining process. Their focusing capability depends on the manufacturing quality of the stamp and mechanical alignment during the coining process, both of which are limited by today’s technology [4]. Recently, diamond CRLs made by laser ablation and mechanical polishing emerged, which exhibit similar shape errors [5].

It has been shown that each lens shows a typical shape deviation of 500 nm from an ideal paraboloid of rotation [3]. When many of these lenses are stacked in order to create sub-micrometer X-ray beams, these shape errors add up and lead to spherical aberration, impacting the resolution and imaging capabilities of X-ray microscopes.

A solution to overcome these challenges is the correction of aberration by an additional optical element, called a refractive phase plate [6]. It is tailor-made for the specific lens configuration and needs to be aligned with respect to the optical axis to within a few micrometers, requiring a motorization within a plane perpendicular to the optical axis.

Here, we present the further development of the lens unit with integrated phase plate kinematic and the application at a high x-ray energy of 32 keV in a user experiment at beamline P06 at DESY. The performance of the focusing unit was evaluated at-wavelength in the high-coherence mode with ptychography and in the high-flux mode with a fluorescence knife-edge. Both methods are available to users at the beamline.

IMPROVED PHASE PLATE KINEMATIC

In order to correct aberration of the CRL stack, a refractive phase plate needs to be positioned on the optical axis with an accuracy of a few micrometers with respect to the focusing lens. In the deliverable report D12.2, we showed a new integrated solution based on PIrest piezos and flexure hinges. Due to the limited travel range of the PIrest piezos, the pretension of the piezos and lever design are crucial to realize a usable travel range. A travel range of > 50 µm is required to align the phase plate, taking manufacturing and printing tolerances for the refractive structure into account. The old design with a maximum travel range of 40 µm is shown in Fig. 1a next to the new flexure design in Fig. 1b, increasing the travel range to 85 µm. However, these travel ranges can only be reached if the piezo system is operated without creep compensation, leading to a strong drift, as shown in Fig. 1c, after the piezo has reached the dialed position. With creep compensation on, the positioning of the device is stable, but the travel range reduces further to roughly 60 µm, also shown in Fig. 1c. Due to the new flexure design the target travel range of > 50 µm can be reached even with the necessary creep compensation for position stability.

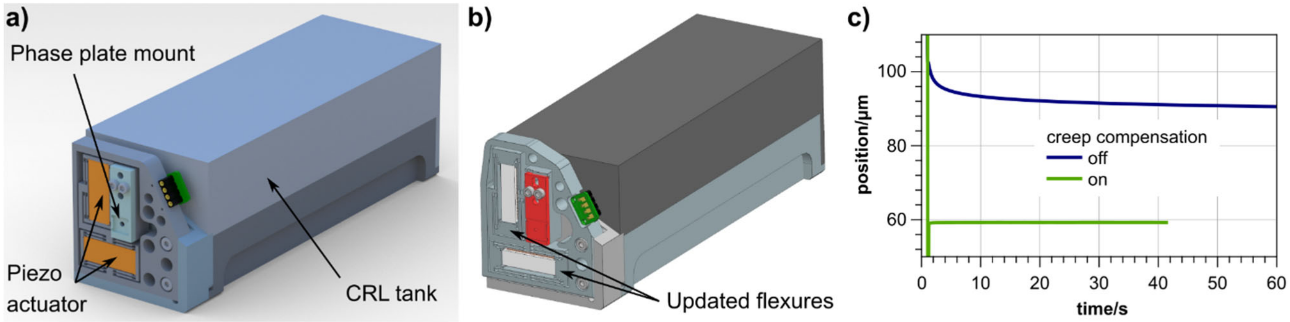

Figure 1: a) CAD model of the previous phase plate kinematic design, as reported in D12.2. b) CAD model with improved flexure design to increase travel ranges. c) Position drift along a single axis for the phase plate without and with creep compensation. The shown position is the maximum travel range of the kinematic.

USER EXPERIMENT AT BEAMLINE P06

In the previous report for MS16 we presented the correction of aberration for a stack of 20 individual bi-concave beryllium lenses for energies between 8.2 keV – 12 keV with an upstream mounted phase plate. For higher x-ray energies between 25 keV – 35 keV another lens stack out of 150 individual bi-concave lenses is available at the beamline. For these high x-ray energies, the KB mirror system of the beamline is not suited due the extremely shallow angle of external total reflection. Here, beryllium CRLs are the only option for users to focus the beam below 1 µm for scanning x-ray microscopy applications. In the following we will present the results for the implementation of the phase plate device at 32 keV and a subsequently performed user experiment.

High-energy x-ray focusing at 32 keV

The lens stack is assembled out of 150 individual bi-concave lenses, each with a radius of curvature of 50 µm and a geometrical aperture of 300 µm. It was characterized in high-coherence mode (front-end slits closed to < 50 µm) using ptychography [4]. We performed measurements at two x-ray energies of 25.7 keV and 35 keV. In contrast to previous work [7], the goal was to design an upstream mounted phase plate to be used across the full energy range of 25 keV – 35 keV. We performed numerical beam propagation simulations through the 150-lens stack to design a corrective phase plate suited for 25 keV as well as for 35 keV and in between, similar to the results presented in report MS16. The approach is to model the propagation through the lens stack to fit both measured data sets. Once the model of the lens stack converged, an upstream phase plate can be designed for these energies. The phase plate was printed by two-photon polymerization in IP-S photoresist at PSI using a Nanoscribe Professional Photonic GT, as reported previously in D12.1. Its diameter is roughly 300 µm with a height of 245 µm. An example of 3D printed phase plate is shown in the Fig. 2. By using a mean phase plate design, the aberration correction can be applied for the whole energy range, but with slightly reduced performance when compared to a phase plate designed for a specific energy. As the beamline operates in high-flux mode most of the time, the negative effects to the incoherent beam are minor and represent a good trade-off for better usability.

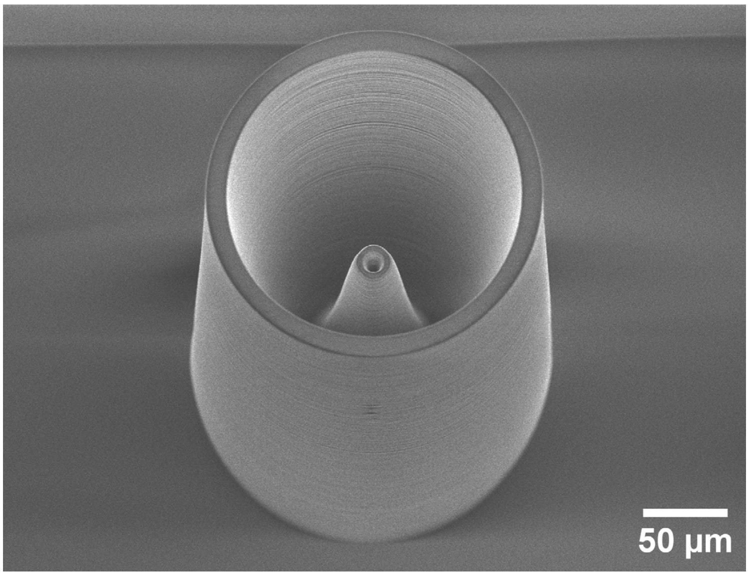

Figure 4: Scanning electron microscopy image of a polymer phase plate made by two-photon polymerization 3D printing for 27.5 keV.

The achieved results are shown in Fig. 3. Before aberration correction, the beam caustic at 35 keV in Fig. 3a shows strong side bands. The phase error of the beam directly behind the exit of the lens had a standard deviation of σ_{noPP} = 2.54 rad. With the fabricated phase plate aligned in the new piezo kinematic the beam caustic improved visibly, shown in Fig. 3b. As the user experiment was asking for 32 keV we have not recorded data at 35 keV for a direct comparison. The phase error, shown in Fig. 3d, improved to σ_{PP} = 1.00 rad. The visible residual ring aberration in the center of the wavefield indicates a slight missmatch of the phase plate, which we accept for a wider energy bandwith, where the phase plate can be used at. The achieved beam size measured 81 nm x 80 nm FWHM (H x V) with a flux of 5e7 photons/s.

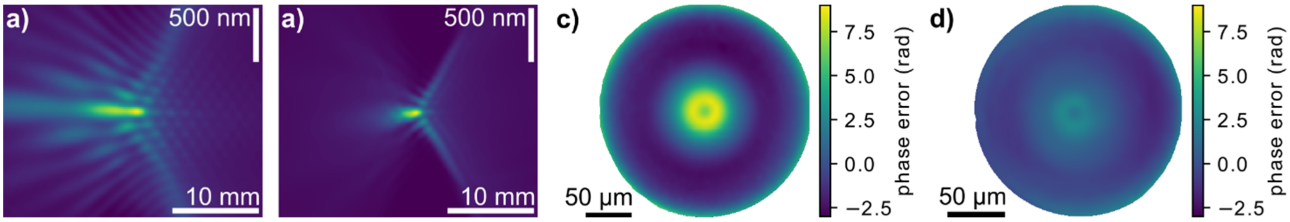

Figure 3: a) Beam caustic for 150 beryllium lenses at 35 keV. b) Beam caustic for 150 beryllium lenses at 32 keV with upstream mounted phase plate. c) Reconstructed wavefield error at the exit of the beryllium lens stack at 35 keV. d) Reconstructed wavefield error at the lens exit of the beryllium lens stack with phase plate.

Cd-K fluorescence mapping

After characterizing the diffraction-limited spot size of the lens stack, the beamline switched to high-flux mode by opening front-end slits for fast fluorescence mapping. With this, the flux increased to 3e9 photons/s. The beam size was measured with fluorescence knife-edge scans on a thin Pt marker with 170 nm line width. The data is shown in Fig. 4. Neglecting the marker width, the beam size was determined to 800 nm x 600 nm FWHM (H x V).

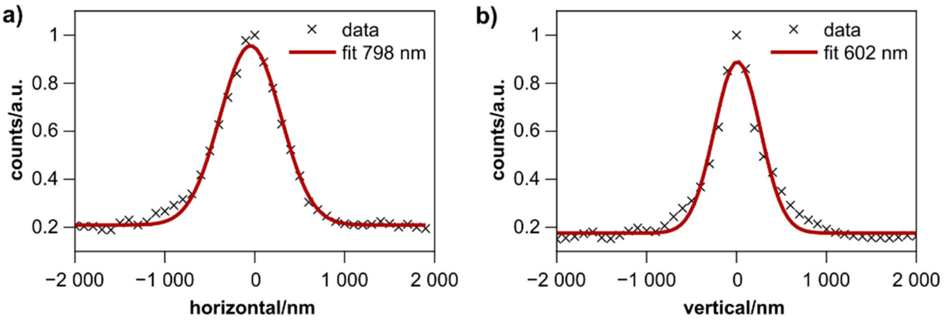

Figure 4: a) Fluorescence knife-edge scan on a 170 nm wide Pt marker in horizontal direction. b) Fluorescence knife-edge in a 170 nm wide Pt marker in vertical direction.

The subsequent user experiment focused on mapping transport pathways of Cd in hyperaccumulator plants [8]. As the Cd-L lines around 3 keV lie between Ar-K and K-K, it is hard to distinguish the Cd signal. Measuring Cd-K lines with high-energy x-rays enabled the mapping of Cd in these plants. The data is currently being analyzed. The experiment was performed in June 2023 at the microhutch endstation of beamline P06.

Aberration-corrected CRL focusing at beamline P06 is available to the NFFA user community in a wide energy range from 8 keV – 35 keV with beam sizes around 100 nm in high-coherence mode. At-wavelength characterization is routinely performed by ptychography and knife-edge methods to characterize the x-ray beam prior to a user experiment.

REFERENCES

[1] Heimann et al., "Compound refractive lenses as prefocusing optics for X-ray FEL radiation", Journal of Synchrotron Radiation **23**(2) (2016)

[2] Zverev et al., "X-ray refractive parabolic axicon lens", Opt. Express **25**(23) (2017)

[3] Seiboth et al., "Nanofocusing with aberration-corrected rotationally parabolic refractive X-ray lenses", Journal of Synchrotron Radiation **25**(1) (2018)

[4] Schropp et al., "Full spatial characterization of a nanofocused x-ray free-electron laser beam by ptychographic imaging", Sci. Rep. **3** (2013)

[5] Celestre et al., "Polished diamond X-ray lenses", Journal of Synchrotron Radiation **29**(3) (2022)

[6] Seiboth et al., "Perfect X-ray focusing via fitting corrective glasses to aberrated optics", Nature Communications **8** (2017)

[7] Seiboth et al., “Hard X-ray wavefront correction via refractive phase plates made by additive and subtractive fabrication techniques”, Journal of Synchrotron Radiation **27**(5) (2020)

[8] Küpper and Leitenmaier, “Cadmium-Accumulating Plants”, In: Sigel, A., Sigel, H., Sigel, R. (eds) Cadmium: From Toxicity to Essentiality. Metal Ions in Life Sciences, vol 11. Springer, Dordrecht. (2022)