DELIVERABLE REPORT

WP7 JRA1 - Research on In-Operando and high throughput methods

D6.6

Diffractive beamshapers, objective lens, and phase shifters for Zernike full-field X-ray microscopy users with 50 nm resolution or better

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DELIVERABLE DESCRIPTION

Optical setup for full-field X-ray microscopy with Zernike phase contrast

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NATURE

P - Prototype

DISSEMINATION LEVEL

☒ P – Public
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☐ CO - Confidential, only for members of the consortium

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Contents

Executive Summary

We designed a set of diffractive optics for high resolution full-field microscopy and tomography in the range between 6.6 and 18.4 keV photon energy. This high photon energy range will provide 2D and 3D imaging with extended penetration depth. The optics are optimized for taking advantage of Zernike phase contrast imaging.

Combining high resolution imaging at the 50 nm level with reasonable diffraction efficiencies, we employed two methods: the line-doubling technique as reported on in report to Deliverable D6.3, and the fabrication of composite Fresnel zone plates in two lithography steps which are optimized for different structural sizes.

We fabricated beam shapers (condeser lenses), Fresnel zone plates and phase rings for three different photon energies: 6.6 keV, 10.0 keV, and 18.4 keV. The set of optics for 10 keV was commissioned and tested at the Anatomics beamline. First results confirm a microscopic resolution of 50 nm, currently limited by the detector pixel. At the moment, thermal instabilities prevent achieving this resolution in 3D imaging.

After commissioning of newly installed components at the Anatomix beamline at Soleil, the Zernike full-field microscopy setup with 50 nm resolution will be available to users.

1. Concept

X-ray microscopy has potential in imaging thick samples with high resolution due to the large penetration depth and the short wavelength of X-rays. As a drawback, the absorption contrast is low, especially when imaging biological samples with hard X-rays. Zernike phase contrast (ZPC) microscopy can be used to image samples that only cause a phase shift to the incident light [1-4]. The method is routinely applied for imaging various types of samples with visible light and X-rays.

The key point in ZPC microscopy is that the diffracted light from the sample is spatially separated from the non-diffracted part in the back-focal plane (BFP) of the imaging lens (cf. Figure 1). Therefore, the phase shift can be manipulated separately for either of these light paths. A quarter wave plate is generally used to transfer the phase difference of these two waves to amplitude modulation in the detector [3]. The amplitude variation in the ZPC image is directly related to phase shift induced by the sample. For a sharp separation of scattered and unscattered beams, the illumination angles need to be clearly defined. In visible light ZPC microscopes, the illumination is typically hollow cone, resulting in a ring of the unscattered radiation in the BFP. The hollow cone illumination in X-ray microscopes can be achieved by annular aperture with zone plates [5] or capillary condensers [6]. Zone plates with an annulus are inefficient due to the small effective aperture, whereas in the case of capillaries, illumination angles are not well controlled.

Instead of a zone plate as condenser, we use a specific type of condenser, which consists of an array of gratings, which are arranged in a circular fashion. Each grating has its periodicity and the orientation of its line chosen in a way that all diffracted beams interfere constructively in a well-defined field of view that has the size of the individual fields, and on the optical axis (see Figure 2). The optical setup of a Zernike full-field X-ray microscope furthermore consists of a Fresnel zone plate as objective lens, and a set phase rings, which are used to translate the phase information of the object into an amplitude modulation. Note that the condenser and the zone plate usually have comparable outermost line/zone widths.

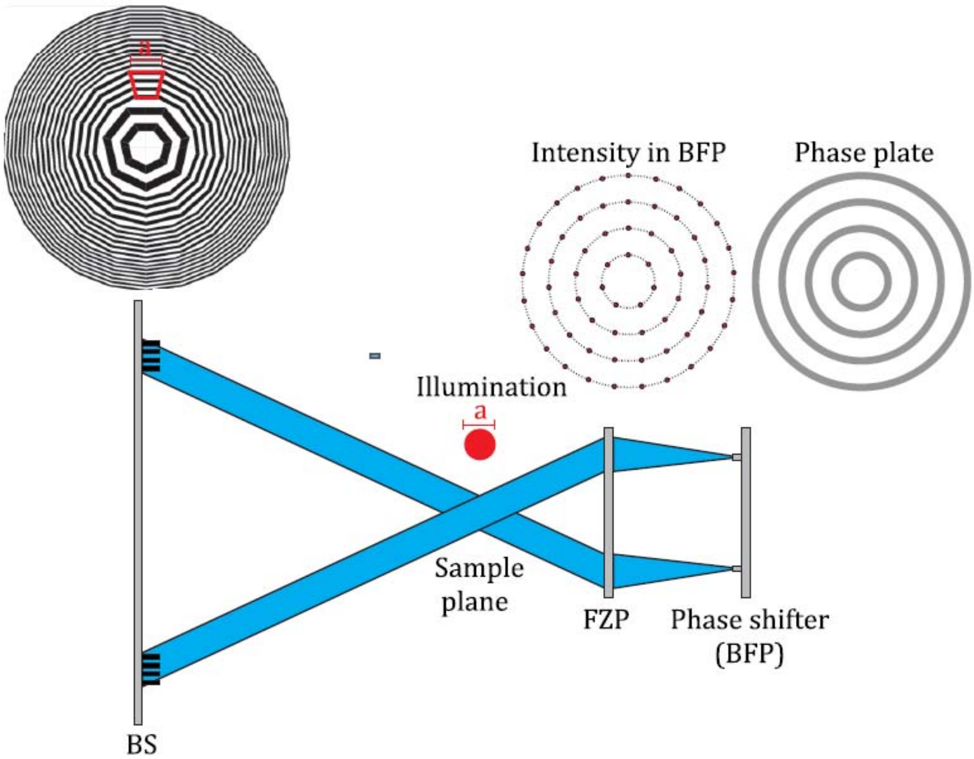

Figure 1: _Schematic illustration of a full-field Zernike phase contrast microscope based on diffractive optics._ A beam shaper (BS) illuminates the sample from discrete angles that overlap in the sample plane, forming a flat top illumination in the sample plane. One of the individual gratings is outlined in red in the BS. Light paths from only two gratings in the BS are shown for simplicity. A zone plate focuses the nondiffracted plane waves to the phase shifter and projects a magnified image of the sample to the detector plane. A phase plate containing concentric rings is used to shift the phase of the zeroth order light by $\pi/2$. Central stop, order sorting aperture, and the detector are neglected from the schematic for brevity. Scheme taken from [4].

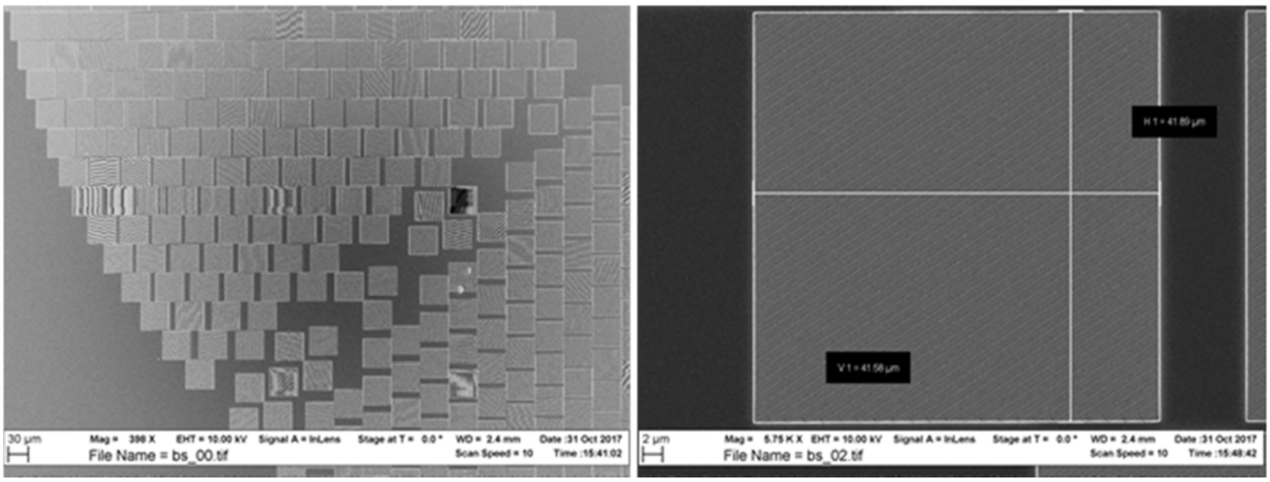

Figure 2: _SEM images of a condenser for Zernike phase contrast microscopy._ The left hand side shows a part of the condenser on its left side. Periodicity and line orientation of each field is calculated in a way that all fields diffract to the same field of view. The right hand side shows a zoom-in on one of the fields.

2. Parameters and Fabrication

2.1 Design Parameters of the Setup

The design parameters have been chosen to match the conditions at the Anatomix beamline at Synchrotron Soleil. A set of optics has been designed for each of three different photon energies with the aim to provide 50 nm microscopic resolution in each case.

Table 1: Design parameters for the optics at three different photon energies.

Design energy [eV]	Distance source-condenser [m]	Outermost zone width condenser [nm]	Field of view [µm]	FZP diameter [µm]	FZP outermost zone width [nm]	Focal length [mm]	X-ray magnification
6.6	170	50	80	282	50	75	50
6.6	170	50	40	141	50	38	100
10.0	170	50	80	200	50	80	50
10.0	170	50	40	100	50	40	100
18.4	170	50	80	110	50	90	50
18.4	170	50	40	171	50	140	100

2.2 Fabrication of Optics

For the photon energies which are to be used, it is crucial to fabricate nanostructures which are sufficiently high to give diffraction efficiencies that are high enough to be used. We therefore employed the line-doubling approach we frequently use for zone plate fabrication (compare report on Deliverable 6.3.1, [7]). This time, the advantage of the method lies not in the fabrication of ultrasmall structure sizes below 10 nm, but on the capability to fabricate nanostructures with 50 nm line width and thicknesses up to 1 µm (Figure 3, left).

Another key point regarding diffraction efficiency has to be considered using the line-doubling approach. As the periodicity in a zone plate gets weaker towards the optical axis with the decreasing line-to-period ratio (because of the constant iridium line width from the atomic layer deposition step), we fabricated the inner part of the zone plate from gold in a second lithography step. With the increasing structure size towards the middle of the zone plate, the lithography step becomes less demanding and can be done in a conventional fashion using PMMA as resist and subsequent electroplating of gold.

We have successfully fabricated beam shapers (condensers), Fresnel zone plates and phase rings for the microscopy setup. The resulting structures for a setup to be used at 10.0 keV are shown in Figure 3.

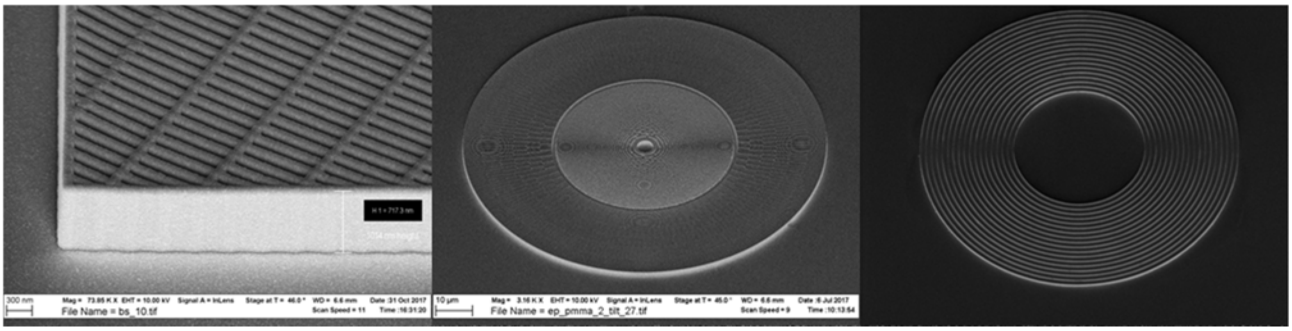

Figure 3: *Set of optics for a Zernike full-field microscope with 50 nm resolution operated at 10 keV*. Left, beam shaper (condenser lens) fabricated by line doubling. In this technique a light material is covered with iridium. The smallest iridium line width is 50 nm, the height 1 µm. Middle, composite Fresnel zone plate. The outer part is fabricated using the line-doubling approach (similar to the condenser). The middle is electroplated gold from a conventional lithography step using PMMA as resist. Right, phase rings with π/2 phase shift at 10 keV to be placed in the back focal plane.

3. First Results of the Setup

The set of optics for a photon energy of 10 keV has been commissioned. For this purpose, the different pieces of optics were installed at the beamline, and tested with a resolution standard and a scientific sample.

Figure 4 (left) shows a transmission X-ray micrograph obtained with this setup on a resolution test chart (model XRESO-50, NTT-AT, Japan; tantalum structures of 500 nm height). The smallest structures in the center of this reference sample of "Siemens-star" type, which are resolved in the image (see inset of Figure 4, left), have a period of 100 nm and are thus just at the Nyquist limit of resolution for the pixel size of 49 nm. This means that the optics work well, and that the microscopic resolution of the setup is limited by the pixel size on the detector at the moment.

Using the same setup, a tomography test was then performed. A rock shale sample with pyrite inclusions served as a test object. The sample was placed on a high-precision rotation stage (model RT150v3, LAB Motion Systems, Heverlee, Belgium) whose runout had previously been characterized to be on the order of 20 nm. Figure 2 shows a vertical reconstructed slice (i.e., a slice parallel to the tomography rotation axis) of a tomography volume of 900×900×900 voxels (field of view 45×45 μm^2) obtained in 4 hours (600 projection angles over 180°, 5 s exposure time per projection micrograph, superposition of 5 scans of this type). The resolution in the tomogram is estimated to around 250 nm, limited mostly by mechanical drift due to temperature variations during the scan.

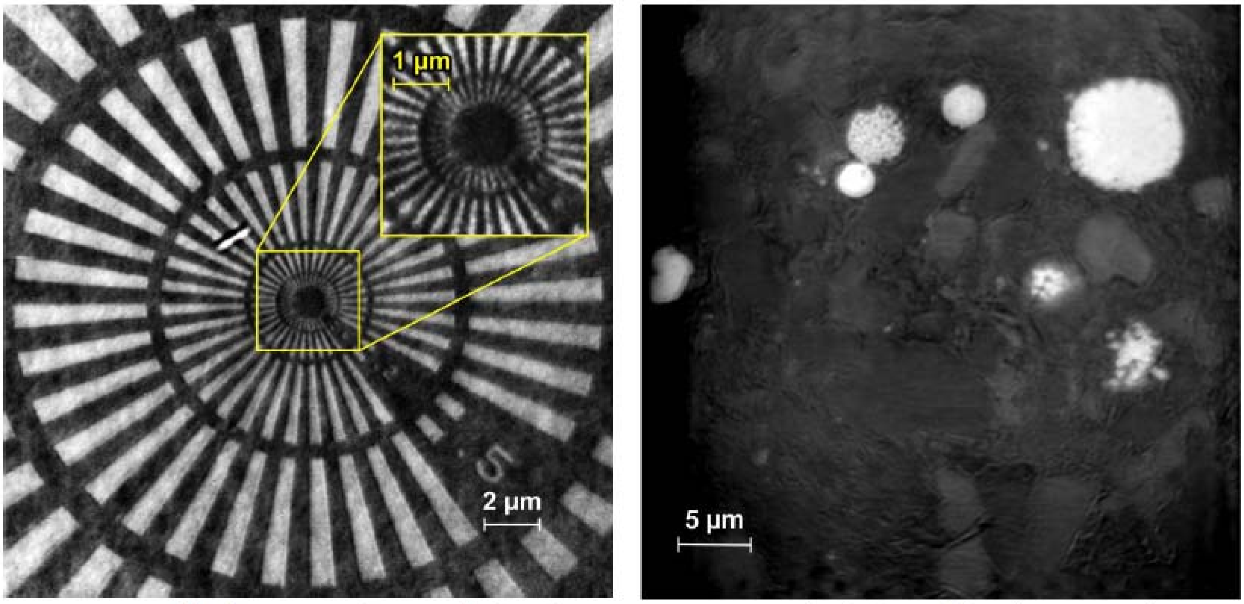

Figure 4: Transmission X-ray micrographs recorded using the Zernike full-field setup. left, Detail of a micrograph that shows a reference sample with smallest lines and spaces of 50 nm (centre of inset). right, Sagittal slice of local tomography on a shale-rock sample with pyrite inclusions (light gray). The sample had a diameter of 160 µm.

4. Conclusions and perspectives

The first tests of optics for the full-field Zernike phase contrast microscopy at the Anatomix beamline have been encouraging. At the moment, the resolution is limited by the detector pixel size, and the thermal stability of the setup. The latter is going to be increased in the near future. We expect that 50 nm resolution can be achieved in tomography in the near future.

Commissioning and testing of the beamline and the optics is ongoing. At the moment, a whole range of components is installed at the beamline, which will further increase the performance of the micrscope. After commissioning of the newly installed components, and testing the optics once more under these conditions, the optics and the setup will be available to users. We expect a microscopic resolution of 50 nm or slightly below to be available in routine operation at three photon energies.

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