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Ulrich Vogt
Karolis Parfeniukas
Tomaš Stankevič
Sebastian Kalbfleisch
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Alexander Björling
Gerardina Carbone
Anders Mikkelsen
Ulf Johansson

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Ulrich Vogt[a], Karolis Parfeniukas[a], Tomaš Stankevič[b], Sebastian Kalbfleisch[b], Marianne Liebi[b], Zdenek Matej[b], Alexander Björling[b], Gerardina Carbone[b], Anders Mikkelsen[c], and Ulf Johansson[b]

[a]KTH Royal Institute of Technology, Biomedical and X-ray Physics, Albanova University Center, SE - 106 91 Stockholm, Sweden

[b]MAX IV Laboratory, Lund University, Fotongatan 2, SE - 225 94 Lund, Sweden

[c]Lund University, Synchrotron Radiation Research, Box 118, SE - 221 00 Lund, Sweden

ABSTRACT

NanoMAX is a hard x-ray nanoimaging beamline at the new Swedish synchrotron radiation source MAX IV that became operational in 2016. Being a beamline dedicated to x-ray nanoimaging in both 2D and 3D, NanoMAX is the first to take full advantage of MAX IVs exceptional low emittance and resulting coherent properties. We present results from the first experiments at NanoMAX that took place in December 2016. These did not use the final experimental stations that will become available to users, but a temporary arrangement including zone plate and order-sorting aperture stages and a piezo-driven sample scanner. We used zone plates with outermost zone widths of 100 nm and 30 nm and performed experiments at 8 keV photon energy for x-ray absorption and fluorescence imaging and ptychography. Moreover, we investigated stability and coherence with a Ronchi test method. Despite the rather simple setup, we could demonstrate spatial resolution below 50 nm after only a few hours of beamtime. The results showed that the beamline is working as expected and experiments approaching the 10 nm resolution level or below should be possible in the future.

Keywords: X-ray microscopy, x-ray nanoimaging, x-ray zone plates

1. INTRODUCTION

X-ray nanoimaging with synchrotron radiation in the 5 – 20 keV hard x-ray energy range is an increasingly popular technique that allows the investigation of matter in 2D and 3D with spatial resolution in the 10 nm range.[1] It is a versatile technique which allows the investigation of samples from many different areas of research such as physics, chemistry, material science, earth and environmental science, biology, medicine and life science. For a measurement an x-ray beam is focused with x-ray optics. The x-ray focus is then used to probe the sample, which is scanned and rotated through the beam. Typical contrast mechanisms that are used in such a scanning x-ray microscope are based on x-ray absorption spectroscopy, x-ray fluorescence or x-ray diffraction. The direct spatial resolution is ultimately determined by the diffraction limit of the x-ray optics used, and best x-ray optics today can offer focal spot sizes around 10 nm.[2]

As an alternative approach, scanning coherent x-ray microscopy, also denoted by ptychography, can be used.[3] It is a combination of coherent x-ray diffraction imaging and scanning x-ray microscopy. The major advantage is that the resolution limit given by the performance of the x-ray optics can be overcome, hence a spatial resolution of below 10 nm is in principle possible. Usually the same experimental arrangement can be used for ptychography or classical scanning microscopy.

Common for both approaches are the coherence requirements for the illumination of the x-ray optic. High degree of spatial coherence is needed, which requires the use of undulator radiation sources at low-emittance storage rings. Nevertheless, available coherent flux often poses limitations to experiments, and an increase of coherent photons would be beneficial in most cases. The development of fourth-generation synchrotron radiation sources with diffraction-limited electron beams holds the promise for a substantial increase of coherent photons.

Further author information:
Ulrich Vogt: E-mail: uvogt@kth.se, Telephone: +46 70 388 08 52

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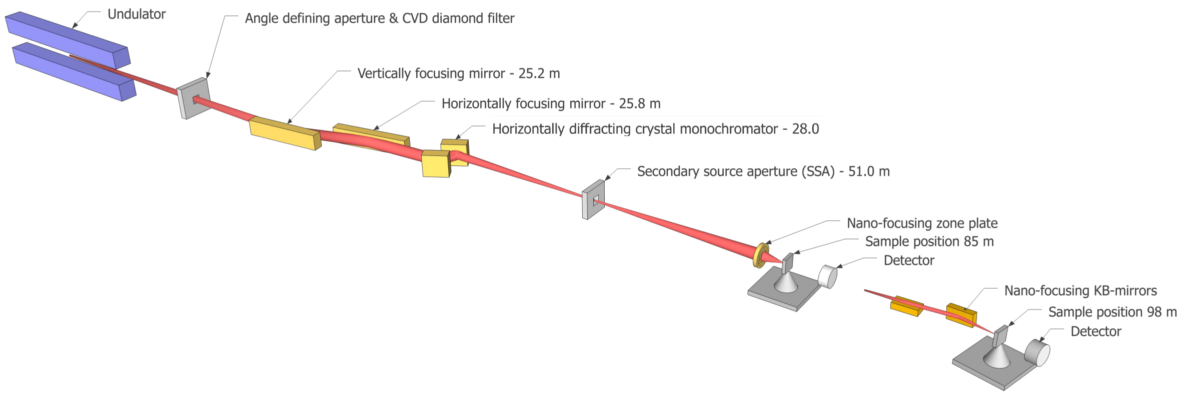

Figure 1. Major layout of the NanoMAX beamline with two experimental endstations at the end. The experiments described in this paper were performed at the position of the first endstation, approximately 85 m from the source.

In this context, the new Swedish synchrotron radiation source MAX IV is the first facility of this new generation with a nearly diffraction-limited beam at least in one direction.[4]

In this contribution, we describe the first x-ray nanoimaging experiments in 2D that were performed at the NanoMAX beamline at MAX IV. We start with a short description of the beamline, followed by the experimental arrangement. We then report the results of our experiments, including investigations on the beamline coherence with a Ronchi method and finally imaging of test objects, both in classical scanning mode and with ptychography.

2. THE NANOMAX BEAMLINE

NanoMAX was one of the two first beamlines to become operational in mid 2016 at the new Swedish synchrotron radiation facility MAX IV in Lund. As a hard x-ray nanoimaging beamline it is located at the 3 GeV electron energy storage ring. A detailed description of the beamline design and expected performance can be found elsewhere,[5] here follows a short summary. A schematic drawing with the major beamline components is depicted in Fig. 1.

NanoMAX has a length of approximately 100 m and extends into a satellite building adjacent to the main ring house. This long construction ensures a high degree of spatial coherence at the experiments as needed for x-ray nanoimaging. Additionally, the beamline design was optimized for stable operation conditions at the 10 nm spatial resolution level.

The beamline employs an in-vacuum undulator with a total magnetic length of 2 m. Using the 3rd – 17th harmonics an x-ray energy between 5 – 30 keV can be fully covered. The beamline optical design was chosen to be rather simple for stability and coherence reasons. Two curved mirrors deflect the beam in horizontal direction and focus it vertically and horizontally, respectively, in order to form a secondary source 51 m away from the undulator. The size of the secondary source and therefore the spatial coherence at the experiment can be adjusted by a secondary source slit system. Temporal coherence or monochromaticity is ensured by a horizontally diffracting Si crystal monochromator situated just behind the focusing mirrors. In this way no optical components that could influence coherence conditions are present between secondary source and nanofocusing x-ray optic, and possible vibrations of optical beamline components are translated into intensity fluctuations at the experiment.

The two experimental endstations of NanoMAX are located 34 – 47 m from the secondary source in a 20 m long concrete hutch. The temperature in the hutch is controlled to ±0.1 K which minimizes temperature fluctuations. A radiation shield wall is located approximately in the middle of the hutch, in this way the first endstation closest to the source can be operated while the second station is accessible for preparations and

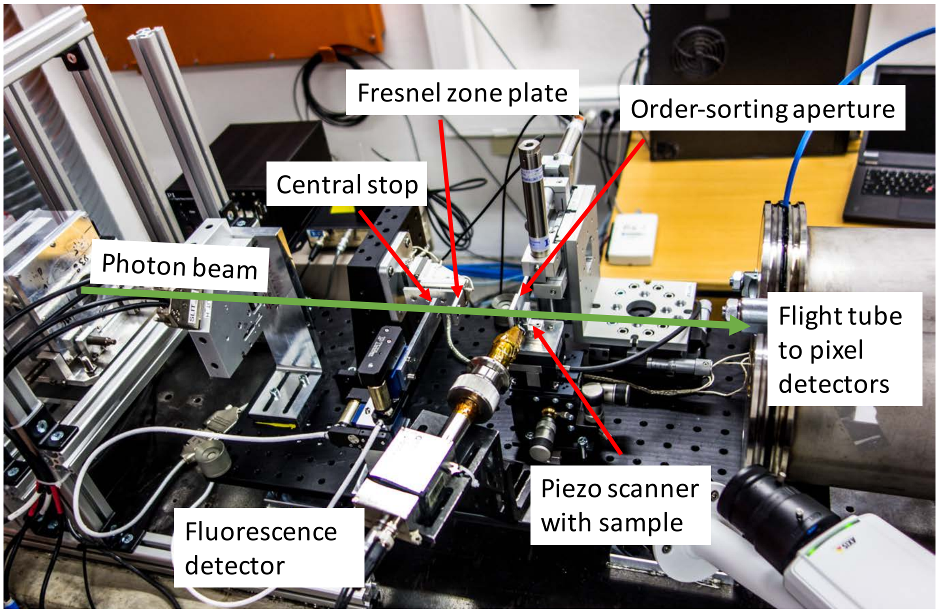

Figure 2. Photography of the experimental arrangement that was used. Major components are indicated.

maintenance. The second endstation uses a Kirkpatrick-Baez mirror optic with spot sizes above 70 nm and is equipped with a goniometer for nano-diffraction experiments. This endstation has already opened for users. The first experimental station is dedicated to highest-resolution experiments with zone plate optics. The experiments described in this paper were done at the location of this endstation.

3. EXPERIMENTAL ARRANGEMENT

Nanoimaging experiments were performed at a fixed photon beam energy of 8.17 keV and a ring current of 50 mA. A photography of the setup is shown in Fig. 2. Major components included zone plate central stop, nanofocusing zone plate optic and order-sorting aperture (OSA) as well as sample and fluorescence and pixel detector.

Central stop and OSA were made out of laser-cut 25 μm thick tungsten foils and mounted on motorized positioners. The same was true for two different zone plates we used for our experiment. The first zone plate was made out of 600 nm thick platinum on 50 nm silicon nitride with a diameter of 75 μm and an outermost zone width of 100 nm.[6] The second zone plate with an outermost zone width of 30 nm was made out of 600 nm thick tungsten on 1 μm silicon nitride with a diameter of 200 μm.[7]

The sample was mounted on a NanoCube piezo-scanner with a travel range of 100 μm (PI Instruments). Larger movements were only possible manually without beam by manual micrometer positioners underneath. As sample itself we mainly used a special test target made out of 1 μm thick tungsten on 100 μm thick diamond. This sample was originally developed for free-electron laser experiments and features rectangular gratings with horizontal and vertical grating line directions and a matrix of Siemens stars.[8] For fluorescence tests a 50 nm thin chromium film with the NanoMAX logo was employed.

Three different detectors were used for the different experiments. A fluorescence detector (Amptek 1 element with PX5 electronics) was placed close to the sample at slightly less than 90 degree angle with respect to the beam direction. For Ronchi tests a Lambda detector (X-Spectrum) with 55 μm pixel size was preferred. Imaging experiments were done with a Pilatus 100k detector (Dectris) with larger 172 μm pixels. Both detectors were situated behind a He-filled flight tube approx. 4 m from the sample position.

It has to be stressed that the above described setup by no means was optimized for highest-possible spatial resolution. It was rather meant to test beamline stability as well as software control and data acquisition. A dedicated zone plate endstation with both 2D and 3D imaging capabilities will become available later.

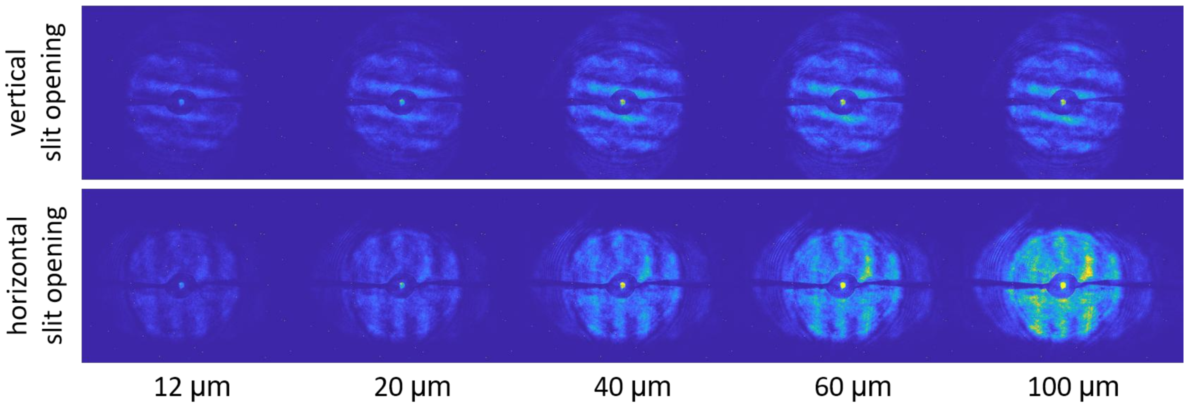

Figure 3. Ronchigrams in vertical (top row) and horizontal (bottom row) direction at different slit openings. The intensity color scaling in all images is identical.

4. RONCHI TEST

We used a simple Ronchi method as a first test of the beam conditions at the experiment. A more detailed explanation about the method can be found elsewhere.[9,10] It is possible to get qualitative information if the spatial coherence is sufficient for nanoimaging experiments. For the measurements we used the tungsten zone plate with 30 nm outermost zone width and the grating test objects with smallest available period of 200 nm. The gratings were placed slightly behind the focal plane, resulting in interference patterns with a few fringes between the zeroth and first grating diffraction orders, also called Ronchigrams (Fig. 3). Looking at the contrast of the interference fringes one can get an idea about the degree of spatial coherence at the experiment. In extreme cases, e.g., when optical components in the beamline or at the experiment are disturbed by vibrations or other instabilities, fringes would vanish completely.[10] We took images at different slit openings in vertical and horizontal position to observe the effect of secondary source size on the coherence. For the vertical series the corresponding grating with horizontal lines was used, and the vertical grating for the horizontal series.

From the beamline design and raytracing simulations, we expected a secondary source size of 13 μm vertical and 115 μm horizontal at our x-ray energy. Accordingly, opening the slit in vertical direction starting from a smallest size of 12 μm should not have a large effect on the spatial coherence at the optic. This was indeed what we observed (Fig. 3, top row), the intensity at larger slit openings was slightly higher but fringe contrast basically remained the same. This also confirmed that we had no stability issues in vertical beam direction.

The situation was slightly different when changing the horizontal slit size (Fig. 3, bottom row). Opening the slits resulted in much higher intensities, as much more photons can pass through, while fringe visibility also was effected in a negative way. Nevertheless, even at the largest opening of 100 μm fringes were still observable. Again, we got confirmation of a good stability in horizontal direction.

To summarize, these tests made us confident that nanoimaging experiments including ptychography should be possible at the beamline even with our rather simple setup.

5. NANOIMAGING RESULTS

We performed a series of experiments with both 100 nm and 30 nm outermost zone width zone plates in classical absorption contrast scanning mode, ptychography and fluorescence mode, as well as in step-scan and fly-scan configuration. Here, we want to present three selected results.

The first result in classical absorption contrast scanning mode was obtained with the 30 nm zone plate and the Siemens star test pattern. For this, the sample was scanned in 10 nm steps through the focus and the total intensity was collected with the Pilatus detector at 1 s dwell time per pixel. Fig. 4 shows the obtained image, together with a line plot through one of the smallest spokes of the star (marked with a black line). An analysis of the line plot indicates a special resolution around 40 nm, close to the diffraction limit of the zone plate optic.

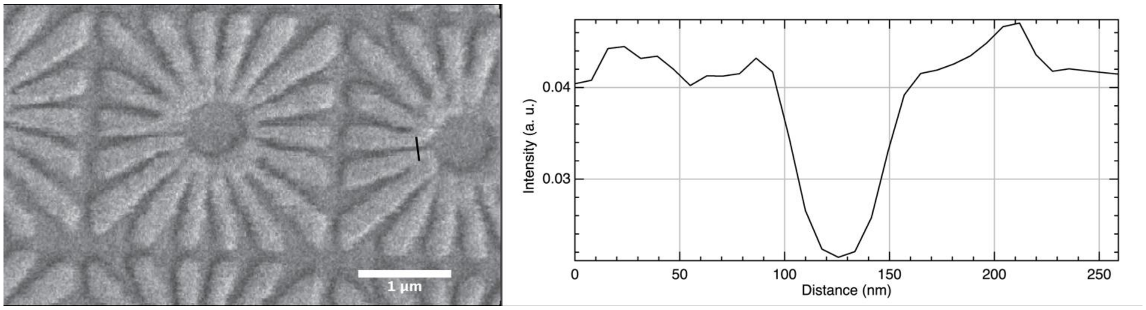

Figure 4. Absorption contrast image of the Siemens test sample obtained in classical scanning mode. The line plot shown to the right was obtained at the position of the black line in the image.

For the second result we switched to the 100 nm zone plate and ptychography mode. An optic with a larger focal spot size was preferred in order to get an easier overlap of neighboring images, and the sample was placed slightly behind the focal plane. Pilatus detector dwell time per pixel was 1 s and the sample was scanned in 100 nm steps. Fig. 5 shows reconstructed amplitude and phase images, together with the probe at sample and focal plane positions. The reconstruction was done with the software PtyPy[11] through 500 ePIE (extended ptychographic iterative engine) iterations with default software parameters. Images are as expected, as well as the shape of the x-ray probe. It has to be noticed that these results were obtained only after one day of beamtime, showing that the beamline is indeed capable of x-ray nanoimaging with high spatial coherence.

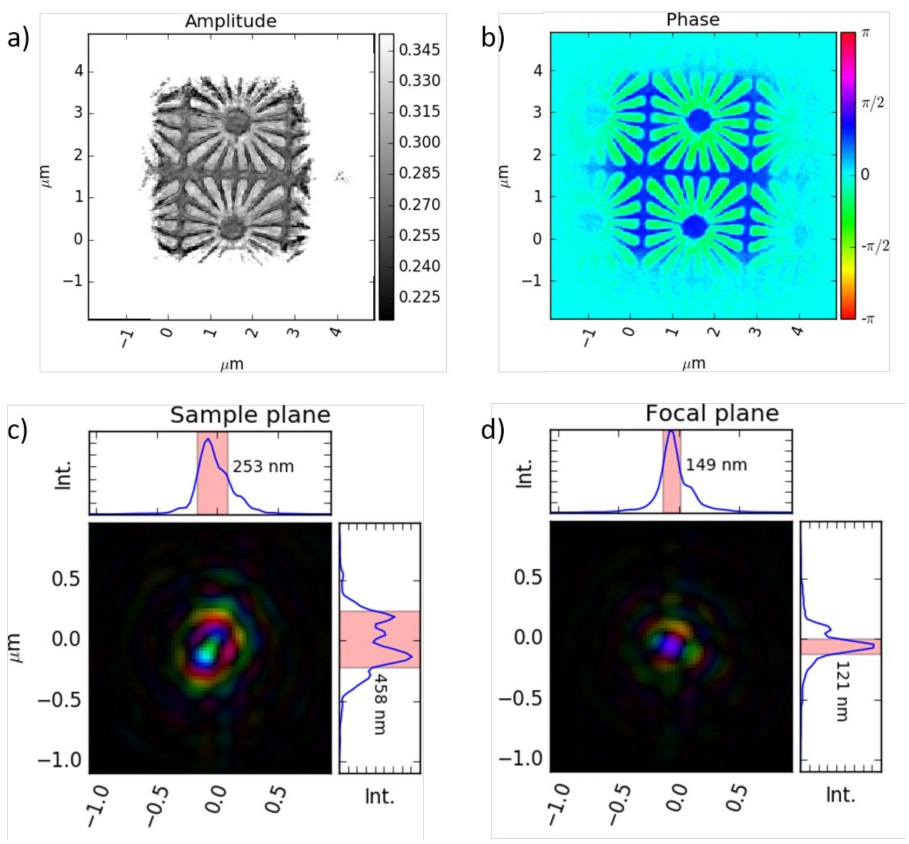

Figure 5. Ptychography imaging results. a) Reconstructed sample amplitude. b) Reconstructed sample phase. c) Reconstructed probe at the sample position. d) Reconstructed probe in the focal plane.

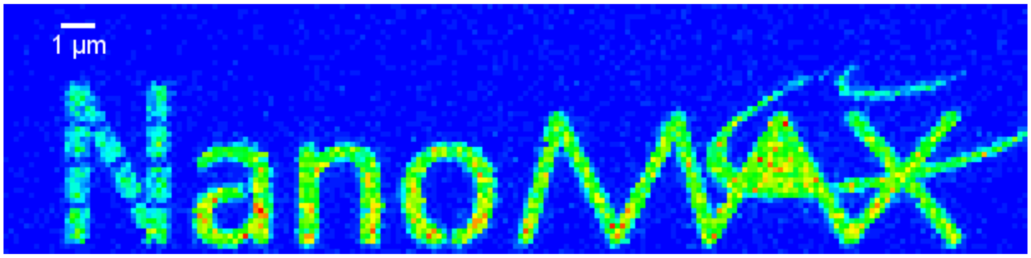

Figure 6. Fluorescence image of the NanoMAX logo made out of 50 nm thick chromium

The last result was again obtained with the 30 nm zone plate, but with a different sample in oder to test the fluorescence mode. The object was a NanoMAX logotype in a small micrometer-sized version made by nanofabrication of a 50 nm thick chromium layer on silicone nitride. For this we used the fluorescence detector and observed the Cr K_α line at 5.4 keV, and Amptek detector exposure time per pixel was 0.5 s. The obtained image is shown in Fig. 6.

6. SUMMARY

To conclude, we have successfully performed first nanoimaging experiments at the NanoMAX beamline. Our efforts were meant as a first real test of the beamline capabilities and the three imaging modes absorption, diffraction (ptychography) and fluorescence. Despite the rather improvised experimental arrangement, limited beamtime and low-current ring operation, we consistently obtained a spatial resolution well below 50 nm. Therefore we believe that with a specialized zone plate endstation we should be able to approach the 10 nm level both for 2D and 3D x-ray nanoimaging and make it routinely available to future users.

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