A Hard X-ray KB-FZP Microscope for Tomography with Sub-100-nm Resolution

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ABSTRACT
An instrument for high-resolution imaging and tomography has been built at the APS beamline 34 ID-C, Argonne National Laboratory. In-line phase contrast tomography can be performed with micrometer resolution. For imaging and tomography with resolution better than 100nm a hard X-ray microscope has been integrated to the instrument. It works with a Kirkpatrick-Baez (KB) mirror as condenser and a Fresnel-Zone plate (FZP) as an objective lens. 50 nm-features have been resolved in a Nickel structure operating the microscope at a photon energy of 9keV. Phase objects with negligible absorption contrast have been imaged. Tomography scans were performed on photonic crystals.

Keywords: hard x-rays, tomography, full-field microscopy, Fresnel Zone Plate, Kirkpatrick-Baez mirror

1. INTRODUCTION
Synchrotron-based hard X-ray tomography is nowadays a standard technique for structural analyses on the micrometer lengthscale. Scientific applications cover a large range of scientific areas such as materials sciences, biomedicine, planetary science etc.. The high coherence of third generation synchrotron sources gives access to new phase contrast imaging techniques. In-line phase contrast imaging with micrometer resolution can be achieved readily.

With the growing interest in nano-science the need to resolve smaller structures has risen. One example to mention is about the investigation of photonic crystals. Also for other applications any improvement in spatial resolution is of large impact. For biomedical applications, it would be for example possible to study cochlea dynamics, as the displacement of the membrane is very small.

There are several ways to achieve this goal. Imaging in fan or cone beam geometry has been shown recently, with a small source spot generated by an electron microscope1. Since high quality optics are also available for the hard X-ray regime, it is possible to build X-ray microscopes working much similar to a visible light microscope.

Starting from an instrument built for in-line phase contrast imaging, a hard X-ray microscope has been developed with 50nm spatial resolution at the APS beamline 34 ID. The condenser is a highly reflective Kirkpatrick-Baez mirror system and the high spatial resolution is achieved with a Fresnel-Zone plate. Tomography is performed with this instrument. Other from visible light microscopy known techniques are going to be carried out.

The experimental capabilities include complementary in-line phase contrast imaging and hard X-ray microscopy. They cover a range of resolution from 50nm to 5µm and a field of view with a sidelength from 50µm to 1mm.
2. EXPERIMENTAL

The instrument is located at the APS undulator beamline 34 ID. The source size is 600 µm by 40 µm (horizontal x vertical) and the beam divergence is 40 µrad by 12 µrad (horizontal x vertical). The beam is split between the E- and the C- hutch of the beamline by a horizontal deflecting mirror. The mirror is nitrogen-cooled. The deflection angle to the C-hutch is 5 mrad, cutting off photon energies above 15keV. The fixed-exit double crystal monochromator is water-cooled and works over an energy range of 6-30keV. Using Si(1 1 1) crystals the energy bandwidth is $\Delta E/E = 10^{-4}$. The monochromator can be moved out of the beam for pink beam imaging. The instrument is placed 55 m from the source in the C-hutch. The beamsize is at this location about 1 mm$^2$ and the photon flux about $10^{13}$ photons/s. The theoretical value for the coherence length is 10x50µm$^2$ (horizontal x vertical).

It is possible to perform in-line phase contrast imaging or to use alternatively the hard X-ray microscope. Details about the in-line phase experiment can be found elsewhere. The stages and detectors from the latter are also used for the hard X-ray microscope setup and are described in detail in the text below.

The hard X-ray microscope works much like a visible light microscope. A condenser converges the incoming light into a spot, where the sample is placed. The latter is then imaged via an objective lens on the detector. The illumination is crucial in order to achieve the full resolution of the microscope.

For the condenser a Kirkpatrick-Baez mirror system has been chosen, because of its high reflectivity, which is above 63%. The two orthogonal palladium coated mirrors have a focal length of 20cm and 10cm respectively. The numerical aperture of the KB-system is matched to the objective lens. The focal spot is a 3x2 µm$^2$. In order to increase the illuminated field the condenser can be scanned and be adapted to the spatial extension of the sample. The sample itself is mounted on high precision stages, in particular the air-bearing rotation stage has a run-out of 20nm. The sample is then projected via a Fresnel-Zone plate as objective optics on the detector plane. The zone plate is made of gold and has a thickness of 500nm and an efficiency of approximately 5% at a photon energy of 9keV. The focal length of the lens is at this energy 23mm. The outermost zone width of several zone plates to chose between is 40nm to 70nm for a resolution of between 50nm and 85nm. Considering the accessible energy range between 6-12keV at the beamline, the depth of focus is for the low resolution zone plates 50-100µm and still 15-30µm for the high resolution zone plates. The distance between lens and detector is 50-100cm for a 20-40x X-ray magnification. The CCD chip of the detector records via a visible light objective lens the image on a scintillation screen. Materials of the screens are europium doped YAG or CsI. The resolution of the detector system depends on the experimental configuration and is within 1 to 4µm. Single images are recorded within seconds over a field of view of 20x40µm$^2$. All images are corrected by additional projections (“darkfield” and “flatfield”). Tomographic scans with up to 180 projections are carried out within an hour. Image series with a defocused X-ray microscope are recorded by driving the sample along the axis of the X-ray beam.
Figure 2: Image of a photonic crystal, taken with the hard X-ray microscope (left) and with in-line phase contrast imaging (right).

Figure 2 shows a typical image achieved with the hard X-ray microscope (left side) and for comparison an in-line phase contrast image. The sample is a photonic crystal, consisting of hollow spheres in a nickel matrix. The diameter of the spheres is about 1.5µm. The magnification of the X-ray microscope is 26x. The illuminated field of view was 40x20µm² and the exposure time a minute using a YAG crystal with a 26µm thick europium layer. The exposure time is reduced to seconds when using a CsI crystal. The latter configuration is used for tomography experiments. Although the quality of the microscope image is somewhat compromised (caused by the detector system) much more details can be identified in comparison to the in-line phase contrast image. Different layers and the shape of the hollow structures (spheres) can be identified.
Figure 3: Image of photonic crystal made of hollow nickel spheres, taken with microscope at 9 keV.

Another more detailed image of the photonic crystal, taken under detector-optimized conditions, is shown in figure 3. The detector was equipped with a 6µm-thick doped europium YAG crystal and the exposure time 500s. Shorter exposure times maintaining the same resolution are achieved with higher X-ray magnification and a light efficient configuration of the detector. In the image small details such as the separating walls with an extension of 50nm can be visualized with a high contrast of about 10%. Also a line going on the left side through the sample can be identified easily as much as the precise position of individual spheres.

Figure 4: Single projection from a tomographic scan of photonic crystal.

A single projection of an entire tomography scan is shown in figure 4. The sample is again the same photonic crystal. The scan of 180 projections took about 1 hour. A CsI scintillation crystal was chosen for the detector system. For first data reconstruction, the data is realigned manually on a smaller subset of the data.
Figure 5: Image of a phase object. The sample is a boron fibre with 100\(\mu\)m diameter. The upper left shows a horizontal profile line through the sample. An image series with the defocused X-ray microscope has been taken of a phase object. The object was a 100\(\mu\)m diameter boron fiber. In figure 5 the sample is close to be focused. A sharp line separates the boron fiber and the air, although the intensity on both sides of the edge is very similar. The sharp edge splits into two parts, when the sample is driven in either direction out of focus. An explanation of the observations should take into account the chromatic aberration of the lenses, the bandwidth of the radiation and the different orders of the zone plate.

4. OUTLOOK

Currently the resolution of the microscope is limited to 50nm, which depends on the outermost zone width of the zone plate. When using the third order of the zone plate the resolution can be improved to 15nm but the efficiency of the zone plate is reduced by a factor of nine. Another way to achieve this goal is by reducing the width of the outermost zone width of the zone plate. The difficulty lies in the fabrication of lenses with such a high aspect ratio and furthermore detailed considerations have to be made about the physics of these optical devices. Simulations in the recent past show that the minimal achievable focal spot with optics for hard X-rays is in the nanometer range, and it is an open question, what the resolution limit for full-field imaging is.

In order to reduce the acquisition time, pink beam can be used for magnified tomography. Pink beam is 50-100 times more intense than monochromatic radiation, because a larger bandwidth of the radiation is used. When using Fresnel zone plates under these conditions, the total number of zones should be not more than 100. The focal distance is then in the range of millimeters. It is not sure that such the imaging quality of such a zone plate will still be satisfying and an intermediate solution might be an option.

5. CONCLUSION

A KB-FZP microscope has been built for sub-100 nm imaging and tomography. Features of 50 nm have been visualized at 9 keV photon energy. A 40x20 microns field of view can be imaged in seconds. Tomography experiments have been performed with this device. Phase objects have been visualized taking image series. Phase contrast techniques, such as the Zernike method will be tested in the future. Both the efficiency and the resolution of the instrument can be further improved. Together with the instrument for In-line phase contrast imaging the nano- and micrometer lengthscales are covered.

ACKNOWLEDGEMENTS

The UNICAT facility at the Advanced Photon Source (APS) is supported by the U.S. DOE under Award No. DEFG02-91ER45439, through the Frederick Seitz Materials Research Laboratory at the University of Illinois at Urbana-Champaign, the ORNL (U.S. DOE contract DE-AC05-00OR22725 with UT-Battelle LLC), the NIST (U.S. DOE contract DE-AC02-05CH11231 with the National Institute of Standards and Technology), and the Office of Basic Energy Sciences of the U.S. DOE.
Department of Commerce) and UOP LLC. The APS is supported by the U.S. DOE, Basic Energy Sciences, Office of Science under contract No. W-31-109-ENG-38. CPR is supported by a grant from the NSF (IBN-0415901).

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