A Full-Field KB-FZP Microscope for Hard X-Ray Imaging with Sub-100 nm Resolution

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A full-field hard X-ray microscope has been built at the UNICAT/APS beamline 34ID-C. A Kirkpatrick-Baez mirror is used for the condenser and a micro-Fresnel Zone Plate (FZP) as the objective lens. The zone plates available give access to 50-85 nm spatial resolution operating the microscope between 6-12keV photon energy. The first tomography experiments have been performed with this device.

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

1. Introduction

Hard X-rays are advantageous for imaging and tomography, due to their high penetration depth and the ease of sample handling. Customized sample environments can be realized while preserving the integrity and functionality of the object. With respect to Full-field X-ray microscopy, the depth of focus of the objective lens becomes more important with increasing photon energy. The advantage for tomography applications is, that large volumes can be visualized sharply. For many scientific applications with hard X-rays, micro-tomography has become a well established technique. In particular, instruments built at third generation synchrotrons make use of the high coherence, the high intensity of the radiation and the possibility of tuning the incident energy. In-line phase contrast imaging renders not only to features with low absorption contrast visible, but also features smaller than the detector resolution. Often scientific problems require higher spatial resolution as features of interest are spread over several length scales. Since high-resolution optics like Fresnel Zone Plates (FZPs) are also suitable for the hard X-ray regime, it has become possible to build a hard X-ray microscope that significantly overcomes the micron limit given by the spatial resolution of the detection system. Sub-100 nm resolution was demonstrated with a setup including a Kirkpatrick-Baez multilayer-mirror (KB) as a condenser followed by a micro-FZP as an objective lens. The microscope at UNICAT/APS is working in the energy range of 6-12 keV with the capacity to provide 50 nm resolution. We performed first tomography scans and plan in addition to apply phase contrast imaging, such as the Zernike method. For applications in material science the absorption contrast is likely to be sufficient to render features visible, whereas the phase contrast techniques have to be considered for biomedical studies.

2. Experimental

The experiment was performed at the Advanced Photon Source (APS), a synchrotron radiation source of the third generation. The station 34ID-C is dedicated to coherent diffraction and imaging, operated by the University of Illinois at Urbana-Champaign (UIUC) within the UNICAT consortium.

The X-rays are generated with an undulator in a high-beta section of the electron storage ring. 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). At 34 ID the beam is shared between two different hutch(es). When the settings of the undulator are controlled by the 34 ID-E station this operation mode called "parasitic mode". The beam splitting is realized by inserting a platinum-coated silicon single-crystal mirror into the beam, deviating the main cone of the x-ray beam to the C hutch. The liquid nitrogen-cooled mirror rejects higher undulator harmonics above 15 keV at an incidence angle of 5 mrad. The fixed-exit double crystal monochromator is water cooled and yields an energy bandwidth of ΔE/E=10⁻⁴ using Si (111) crystals over an energy range of 6-30 keV. Optionally it can be removed for experiments with pink beam. The beam size is ~1 mm² at a distance of 55 m from the source and the flux is about 10¹³ Photons/second. The theoretical value for the vertical coherence length is about 50 µm and about 10 µm for the horizontal coherence length. The imaging and tomo-
graphy setup is placed 55 m from the source. It is possible to perform in-line phase contrast imaging or alternatively to use the microscope. Details about the in-line phase contrast imaging can be found elsewhere). The table and all stages are chosen for their high stability and precision. The air-bearing stage for the sample rotation has a run out below 20 nm. Figure 1 represents the scheme of the beamline and the hard x-ray microscope. A Kirkpatrick-Baez mirror system is used as condenser because of its high reflectivity. It consists of two orthogonal mounted palladium-coated mirrors. The focal length of the mirrors are 20 and 10 cm respectively. About 63% of the incoming intensity is focused to a 3x2 μm² spot onto the sample. A large field of view is obtained by scanning the KB-system. The sample illumination matches the aperture of a Fresnel-zone plate, placed about 23 mm behind the sample. The zero order of zone plate is eliminated by blocking the direct beam with a beam stop, placed in front of the KB-mirror system (not shown in graph). The outermost zone width of the gold micro-zone plates varies between 40-70 nm, delivering under these conditions a resolution between 50-85 nm. In the considered energy range of 6-12 keV the depth of focus is 15-30 μm (50 nm resolution) and 50-100 μm (85 nm resolution). The efficiency of the 500 nm-thick lens is about 5% at 9 keV photon energy. The zone plate projects the magnified image of the sample on the detection system, placed at 50-100 cm distance. The X-ray magnification is 20-40x in the given geometry. The detector consists of a CCD camera coupled via an optical microscope to a scintillation screen, with a resolution of 1-3 micron depending on the configuration.

Additional images without sample ("flatfield image") and without X-rays ("darkfield image") are taken for corrections. Most experiments are carried out in "parasitic mode" at an energy of about 9 keV and with tapered undulator, so the intensity is reduced to 1/3 of its maximal value. Exposure times were up to 500 s, depending on the intensity and the illuminated field of view. For example a field of view of 20x40 microns² was scanned in 60 s at a photon energy of 9 keV and with non-tapered undulator. Further, first tomographic scans were performed within 10 minutes (20 projections) and 1 hour (180 projections) respectively.

3. Results

Figure 2 shows an image taken with the microscope at 9 keV photon energy and with 26x X-ray magnification. The exposure time was 500 sec. The object is a photonic crystal. It consists of an inverted nickel matrix containing hollow spheres of 1.2 micron diameter. The spheres are arranged in a hept-structure and the thickness is of 2-3 layers. The thickness of the walls between the spheres is below 200 nm. In the image 50 nm features of can be clearly identified and the contrast is about 10%. Dust deposit during the experiment on the detector system could not be removed by simple flat-field correction of the images and might be only possible by additional image treatment. For this reason a more detailed analysis is difficult, as the artifact modifies the modular transfer function. The reconstruction of the tomographic data is still under evaluation.

4. 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 of can be imaged in a minute. The first tomography experiments have been performed with this device. Further, it is planned to apply phase contrast techniques, such as the Zernike method. Both the efficiency and the resolution of the instrument can be further improved. A more efficient zone plate and an improved detector will reduce the exposure times and the use of the 50x100 times more intense so called "pink-beam" is possible. To improve the resolution, the zone plates deliver in their third order a resolution of 15 nm.

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