Recent progress of hard x-ray imaging microscopy and microtomography at BL37XU of SPring-8

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Recent Progress of Hard X-ray Imaging Microscopy and Microtomography at BL37XU of SPring-8

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Abstract. A hard x-ray imaging microscopy and microtomography system is now being developed at the beamline 37XU of SPring-8. In the latest improvement, a spatial resolution of about 50 nm is achieved in two-dimensional imaging at 6 keV x-ray energy using a Fresnel zone plate objective with an outermost zone width of 35 nm. In the tomographic measurement, a spatial resolution of about 100 nm is achieved at 8 keV using an x-ray guide tube condenser optic and a Fresnel zone plate objective with an outermost zone width of 50 nm.

INTRODUCTION

In imaging microscopy, the field of view (FOV) is as important as the spatial resolution. The large FOV is especially required for computer tomography (CT) applications, because the diameter of object must be smaller than the FOV of the microscope. A simple and effective way for achieving a large-FOV and high-resolution the x-ray imaging microscopy is a large magnification factor by using a long distance between objective lens and image plane. The beamline 37XU of SPring-8 has an experimental station whose length is about 30 m by combining the three experimental hutches. Therefore, by exploiting the long experimental station at BL37XU, we have developed an imaging microscope and microtomography systems with a large magnification, high spatial resolution, wide FOV, and high throughput. By using a Fresnel zone plate (FZP) objective having an outermost zone width of 50 nm, a spatial resolution better than 100 nm in 2D image has already been achieved at 6 keV, and a spatial resolution better than 300 nm has been achieved in CT imaging at 8 keV with a FZP of 100 nm outermost zone width [1]. Here, some recent improvements of the optical systems for x-ray microscopy at the beamline 37XU are described, and results on characterization of the imaging system are also shown.

EXPERIMENTAL SETUP

A schematic diagram of the hard x-ray imaging microscopy system is shown in Fig. 1. Undulator radiation form a 139-pole planar undulator installed in the SPring-8 8 GeV electron storage ring is monochromatized by passing through a liquid-nitrogen-cooled Si 111 double-crystal monochromator, and a double Rh-coated total-reflection mirror system is used to suppress the higher-order x-rays from the crystal monochromator. A hollow cone illumination by a rotating sector condenser zone plate (CZP) is employed to achieve quasi-Köhler illumination [2, 3]. An x-ray guide tube (conical-shape total-reflection mirror optics) is also used to improve the throughput [4]. The tomography scan is performed in on-the-fly scan mode using a high-precision slide-guide rotation stage (SPU-1A, Kohzu Precision Co., Ltd., Japan). The eccentricity of the rotation stage is estimated to be less than 100 nm. The magnified transmission image is acquired with an indirect-sensing x-ray imaging detector consisting of a fine scintillator powder screen (Gd_2O_2S:Tb+), an optical lens system, and a CMOS image sensor with 2048 x 2048 pixel format.

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The sector CZP is a combination of constant pitch gratings. By rotating the sector CZP, the illuminating beam has a half angle of convergence defined by λ/d_C, where λ is wavelength and d_C is the pitch of the condenser grating. When the illuminating beam is parallel to the optical axis, and the object is placed at a center of image circle, the limit of the spatial resolution for a periodic structure is expressed as λ/NA, where NA is numerical aperture of objective lens. Using the outermost zone period, d_O, of the objective FZP, the spatial resolution limit for parallel beam illumination is simply rewritten as $\lambda/NA = d_O$. Considering the annular illumination aperture of the rotating sector CZP, the spatial resolution limit for the periodic structure, d_S, is expressed as

$$1/d_S = 1/d_C + 1/d_O$$

$$= NA_C/\lambda + NA_O/\lambda.$$

On the contrary, the FOV of bright field image for the annular hollow-cone illumination is restricted by the following inequalities, as

$\text{FOV} \leq 2R\,(1 - NA_C/NA_O)$, limitation by the acceptance of objective lens,

$\text{FOV} \leq 2R\,NA_C/NA_O$, limitation by overlap of direct beam at the image plane,

where R is the radius of the objective FZP, NA_C is the half convergent angle of the illuminating beam, and NA_O is the numerical aperture of the objective FZP. Therefore, the bright-field FOV is optimized at $NA_C = NA_O/2$, while the best spatial resolution is achieved at $NA_C = NA_O$. The maximum FOV of the bright-field imaging is equal to the radius of the objective FZP. We usually employ the optimized FOV condition for the microscopy CT. Then, the spatial resolution limit for periodic structure is $2\lambda/(3NA_O) \sim 0.67\lambda/NA$, instead of Rayleigh's criterion, $0.61\lambda/NA$.

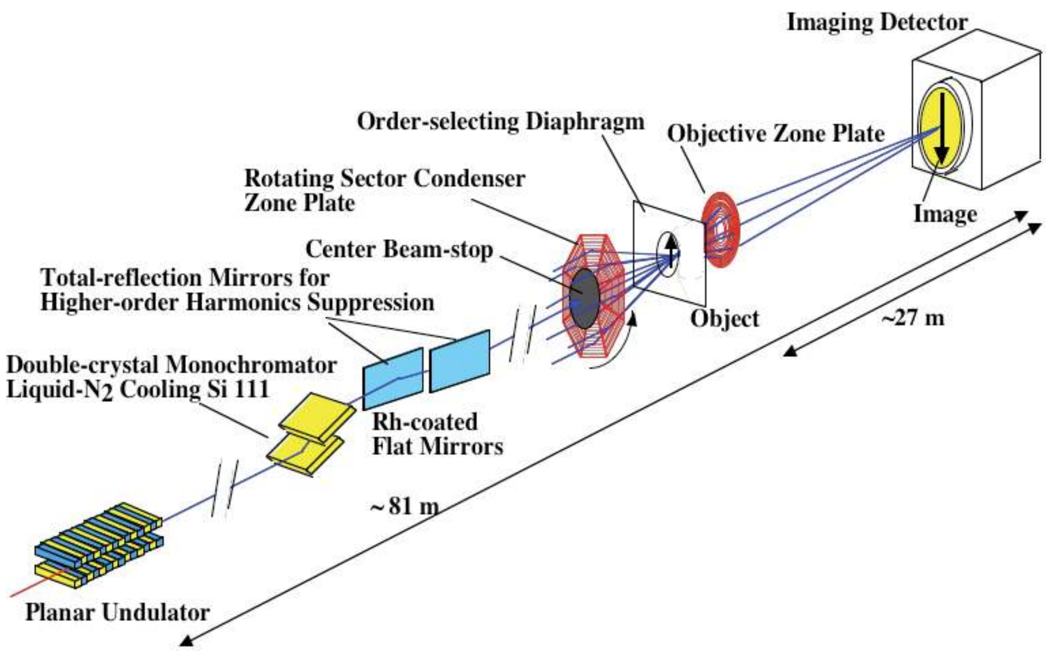

FIGURE 1. Schematic diagram of x-ray microscopy and microtomography system at BL37XU of SPring-8

RESULTS and DISCUSSION

The spatial resolution in the tomographic imaging is evaluated using fine periodic structures fabricated on an aluminum wire by focused ion beam (FIB) milling as a resolution test object [5]. The typical micro-CT results are shown in Fig. 2. Here, a FZP objective with an outermost zone width of 100 nm and diameter of 310 µm is combined with an octagon sector-CZP with a grating period of 400 nm. The focal length of the FZP is 200 mm at an x-ray energy of 8 keV, and the magnification of x-ray optics is about 135. The FOV is 123 µm, and the effective pixel size is 60 nm. The CT image is reconstructed from 1800 projections taken in on-the-fly scan mode with a step angle of 0.1º, a frame rate of 1 Hz and an exposure time of 0.75 s for each projection at an x-ray energy of 8 keV. The limiting resolution estimated from the visibility of the periodic structure is 160 nm, which agrees with the theoretical resolution of the optical system (133 nm period) and with the sampling pitch of the projection image (120 nm). The 160 nm periodic structures are resolved both in the transversal slice image and in the sagittal slice image, as shown in the figure.

High-resolution CT imaging is also carried out using an objective FZP with an outermost zone width of 50 nm. The diameter of the FZP is 250 µm, and the zone material is tantalum with a thickness of 0.5 µm. The focal length is 80 mm for 8 keV x-rays, and the optical magnification is about 350. Here, the condenser zone plate optics is replaced with an x-ray guide tube (XGT) for achieving the higher flux and higher throughput, and a rotating diffuser (a sheet of paper) is placed in front of the XGT for suppressing the speckle noise from the XGT condenser optics. The XGT is conical-shape total-reflection mirror optics. The convergence angle of the XGT is 1.9 mrad, which is matched to the *NA* of the objective FZP with an outermost zone width of 50 nm at 8 keV. Figure 3 shows a result of the resolution test with the model object. The effective pixel size is 24 nm at the object plane, and the FOV is 49 µm. 1800 projections are taken at 0.1º-interval in on-the-fly scan mode with a frame rate of 1 Hz. The exposure time for each projection is 0.8 s. Fine periodic structures of 120 nm period are clearly observed, and the some traces of 100 nm-period patterns are barely visible, as shown in the figure. Therefore, it is considered that the present resolution limit of the micro-CT system is about 100 nm in the full-pitch of the periodic structure. The theoretical resolution limit is 62 nm in this case. However, the measured resolution is worse than the theoretical value. This discrepancy is mainly attributed to the wobbling of the rotation stage.

The performance test for two-dimensional imaging was also carried out using a resolution test chart (XRESO-20, NTT-AT, Japan) that has fine structures up to 20 nm lines and 20 nm spaces. Figure 4 shows the result of the spatial resolution test for 2D imaging. Here, a FZP with an outermost zone width of 35 nm is used as an objective lens, and a sector zone plate with a grating period of 200 nm is combined with the objective FZP. The diameter of the objective FZP is 177.12 µm, and the focal length for 6 keV x-rays is 30 mm. The optical magnification is 916. The FOV and the effective pixel size is 17.8 µm and 8.67 nm, respectively. In the measured image, a periodic structure of 25 nm line and space in the star pattern test chart is visible. The resolution limit of 50 nm in the full pitch of periodic pattern agrees well with the theoretical resolution limit (51.9 nm in the full period).

As a result, in the present hard x-ray imaging microscopy system at BL37XU of SPring-8, the three-dimensional resolution of 100–120 nm is achieved by tomography method, and the spatial resolution of about 50 nm is attained in the two-dimensional imaging.

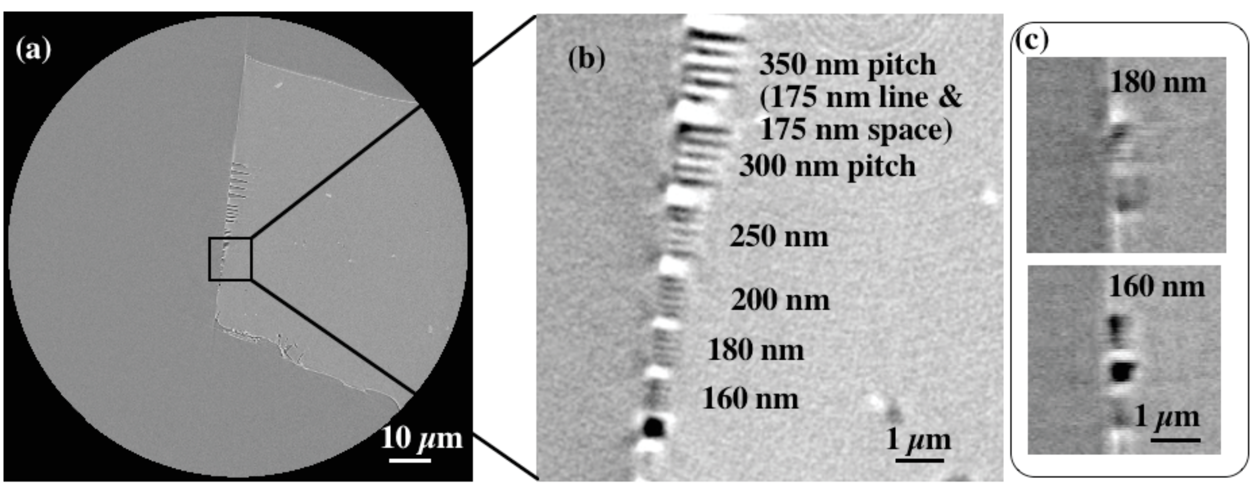

Figure 2. CT images of resolution test pattern. (a) Full-field image of transversal slice, (b) magnified view of central area of (a), (c) sagittal slice images. X-ray energy is 8 keV. The field of view is 123 µm, and the pixel size is 60 nm. 1800 projections are taken with 1 Hz frame rate. The exposure time of each projection is 0.75s. The total scan time is about 1800 s.

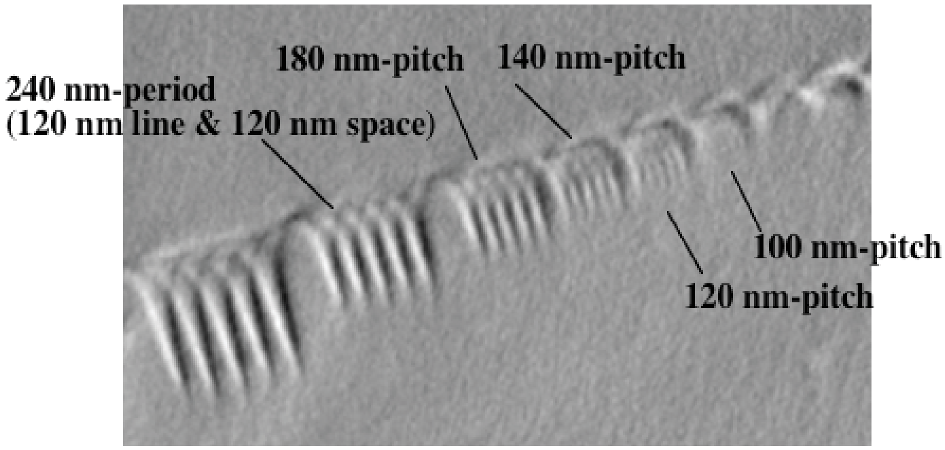

FIGURE3. Tomography images of resolution test patterns. Transverse slice. X-ray energy is 8 keV. The field of view is 49 μm, and pixel size is 24 nm. 1800 projections are taken at 0.1˚ step in the on-the-fly scan mode with a frame rate of 1 Hz. The exposure time of each projection is 0.8 s.

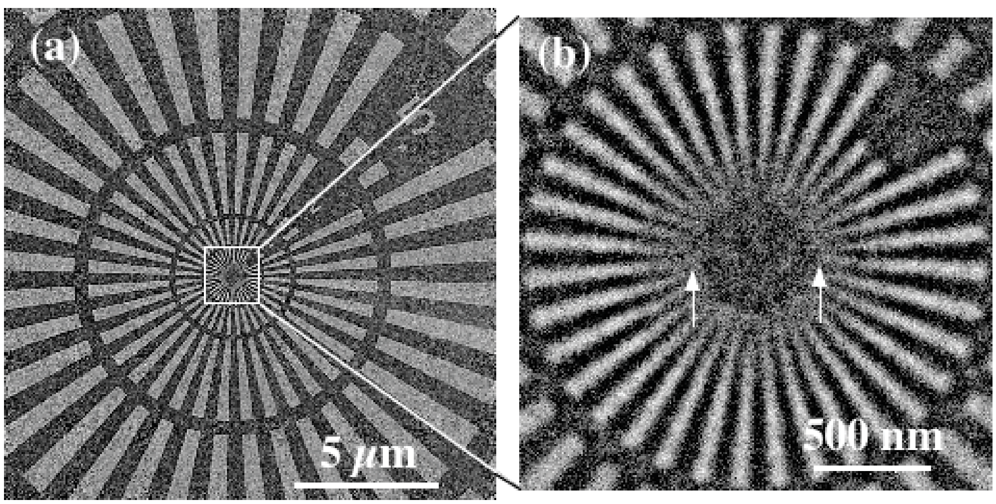

FIGURE 4. Two-dimensional microscopy image of resolution test chart (ATN/XRESO-20, NTT-AT, Japan) measured with absorption contrast mode at an x-ray energy of 6 keV. The pixel size is 8.67 nm, and the FOV is 17.8 μm. The exposure time is 1000 s. (a) Full-field image and (b) magnified display of the central area of (a). The arrows in (b) indicate the region of 25 nm line and space patterns.

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