Zernike-type X-ray imaging microscopy at 25 keV with Fresnel zone plate optics

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A Zernike-type imaging microscope using a sputtered-sliced Fresnel zone plate (SS-FZP) has been developed and tested at an X-ray energy of 25 keV. The SS-FZP was used as an objective. A copper (Cu) phase plate was placed at the back focal plane of the SS-FZP in order to produce phase contrast. The performance of the Zernike-type imaging microscope was tested with a gold (Au) mesh and a resolution test pattern at undulator beamline 47 of SPring-8. The Au mesh and the resolution test pattern could be imaged in transmission with a magnification of ×10.2. Owing to the Cu phase plate, different image contrast was observed compared with the bright-field image contrast. Tantalum microstructures down to 0.5 μm line-and-space have been observed on spatial resolution test patterns.

Keywords: Zernike-type imaging microscopy; sputtered-sliced Fresnel zone plates; phase plates; diffusers; partial coherent illumination.

1. Introduction

Hard X-ray microscopy makes it possible to observe the inside of a living body and thick materials with high image contrast and high spatial resolution. High-energy X-rays of several tens of keV are needed to observe materials with thicknesses of the order of cm. However, it is easily predicted that structures composed of light elements, such as biological soft tissues, have very little absorption contrast for such high-energy X-rays. Even if the biological structures have a small X-ray absorption coefficient, phase change for image contrast can be expected because of the relation $\delta >> \beta$, where the complex refractive index of the light elements n is expressed as $n = 1 - \delta - i\beta$. Therefore, research into phase contrast imaging techniques in the hard X-ray region has been growing rapidly.

Recently, several methods for phase contrast imaging have been reported using an X-ray interferometer (Bonse & Hart, 1965; Momose *et al.*, 1996), mirrors (Watanabe *et al.*, 2000) and zone plates (Schmahl *et al.*, 1995; Wilhein *et al.*, 2001). Among these methods, Zernike's phase contrast imaging with zone plate optics allows the possibility of achieving sub-μm spatial resolution with high-energy X-rays. Schmahl *et al.* (1995) developed a transmission soft X-ray microscope with zone plate optics and a phase plate at an energy of 0.5 keV, and studied the phase contrast of biological specimens. However, a lack of lens systems for hard X-rays has prevented the development of Zernike-type imaging microscopy, especially with a photon energy over 20 keV.

The sputtered-sliced Fresnel zone plate (SS-FZP) proposed by Rudolph & Schmahl (1981) is one of the promising candidates for high-energy X-ray optics because a thick FZP with a narrow zone width can be fabricated by the sputter-slice technique. Therefore, the SS-FZP is considered to be suitable as a high-energy X-ray focusing and imaging element. In this paper we introduce a Zernike-type phase contrast X-ray imaging microscope using the SS-FZP as an objective, and show the results at 25 keV.

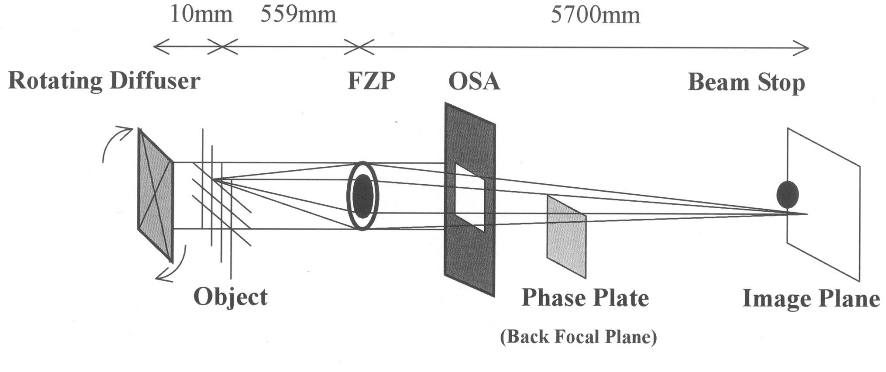


Figure 1
Scheme of the Zernike-type hard X-ray imaging microscope with the SS-FZP.

2. Zernike-type imaging microscopy and results

The imaging experiment has been carried out at undulator beamline 47XU of SPring-8. Fig. 1 shows the scheme of the Zernike-type imaging microscope. The SS-FZP fabricated at AIST (Tamura *et al.*, 1997) was employed as the objective (phase zone plate). It consisted of 50 layers of alternating copper (Cu) and aluminium (Al) deposited on a gold (Au) core. The thickness of the SS-FZP is about 36 μm and the outermost zone width is 0.25 μm. The measured focal length was 508 mm, and the measured diffraction efficiency of the first order was about 15% for 25 keV X-rays. As it is difficult to control and measure the thickness of the SS-FZP precisely at present, the X-ray energy was selected such that the diffraction efficiency of the first-order was high enough to perform this experiment. Here the calculated diffraction efficiency of the first-order had its peak value (~30%) at a thickness of about 40 μm.

The X-rays emitted by an in-vacuum-type permanent-magnet planar undulator (Kitamura, 1998) were monochromated with an Si(111) double-crystal monochromator. The photon flux density of the monochromated X-rays at the exit plane of the monochromator was 4.2×10^{12} photons mm^{-2} s^{-1} at 25 keV. The sample-to-source distance was 43 m and the estimated source size was about 50 μm in the vertical and about 900 μm in the horizontal directions. The angular spread of the X-ray beam, D/L, where D is the source size and L is the sample-to-source distance, is estimated to be 1.2 μrad in the vertical and 21 μrad in the horizontal directions. The spatial coherent area at the object plane was estimated to be about 43 μm in the vertical and 2.4 μm in the horizontal directions. Therefore the coherently illuminated area in the vertical direction is 18 times as large as that in the horizontal direction.

In visible-light microscopy under coherent illumination using a laser light, so-called ‘speckle’ noise is usually observed. We also observed speckle noise in the hard X-ray region under coherent illumination using undulator radiation, and the noise structures could be mostly seen in the vertical direction (Awaji *et al.*, 2000). To eliminate the speckle noise we assumed that if the spatial coherence in both the vertical and the horizontal directions can be reduced then the speckle noise would disappear. In recent years, White *et al.* (1995) used a kind of diffuser to extinguish the speckle noise in imaging optics for EUV light from a synchrotron light source. We developed a diffuser for the 25 keV hard X-rays to reduce spatial coherence, and eliminated the strong speckle noise (Awaji *et al.*, 2001). In this experiment, P 800 (Japanese Industrial Standard) abrasive cloth with SiC powder of average grain size 21 µm was used as the diffuser (the total thickness was 0.45 mm) to disturb the wave front of the incident beam. As Zernike’s phase contrast method cannot be accomplished if the incident beam is perfectly incoherent, the degree of coherence was reduced adequately. Fig. 2 shows the measured angular spread of the 25 keV X-rays that passed through the diffuser. The values of angular spread (FWHM) in the vertical and the horizontal directions were about 11 µrad and 31 µrad, respectively, and the difference between the values in the vertical and the horizontal directions was due to the instrumental function without the diffuser. The transmissivity of the 25 keV X-rays to the diffuser was 71%.

The diffuser was set 10 mm upstream of the sample, and was rotated to remove the speckle noise from the diffuser itself. Here, the fill factor, the ratio of the angular spread length of the illuminating beam, which passed through the diffuser at the FZP plane to the diameter of the FZP, was 0.078 in the vertical and 0.22 in the horizontal directions. A spatial filter (50 µm × 50 µm square aperture) was used as the order-sorting aperture (OSA) to select the first-order diffracted beam from the SS-FZP to form the objective image. Then a Cu foil about 5 µm thick was placed as a phase plate at the back focal plane of the objective. This phase plate covers half of the back focal plane. Thus this imaging method is a little different from the general Zernike’s phase contrast method. The phase difference caused by the Cu phase plate is 1.7 rad ($\simeq \lambda/4$ at 25 keV). The transmission images of the samples were magnified 10.2 times with the FZP optics. A beam stop with a diameter of about 400 µm, made of Sn–Pb alloy, was set just in front of the image detector [a cooled charge-coupled-device (CCD) camera coupled with relay lens and phosphor screen] in order to avoid saturation owing to the zeroth-order (direct) beam. We tested the Zernike’s phase contrast method only in the vertical direction.

An Au mesh with 1500 lines per inch, a wire diameter of 5.6 µm, a nominal aperture of 11 µm and a thickness of about 3.8 µm (28% absorption at 25 keV) was used as a test sample to check the image formation characteristics. Fig. 3(*a*) shows a bright-field image of the Au mesh, and Fig. 3(*b*) shows an image taken using the Cu phase plate. In Fig. 3(*a*) the mesh image is successfully formed without serious noise and aberration. In Fig. 3(*b*) the Cu phase plate covers half of the back focal plane, and the contrast of the Au wires marked by arrows was changed. This is due to the phase plate. As the beam size, which is due to the angular spread in the vertical direction (11 µrad), is adequately small compared with the size of the OSA (spatial filter) which is upstream of the back focal plane, only the horizontal image contrast of the Au wires was changed, and there were no remarkable changes in the vertical image contrast.

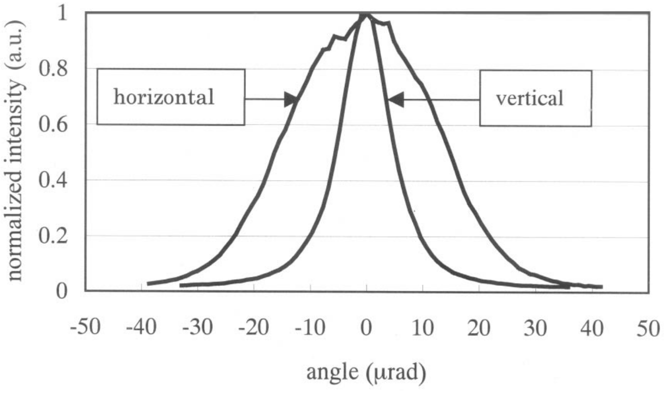
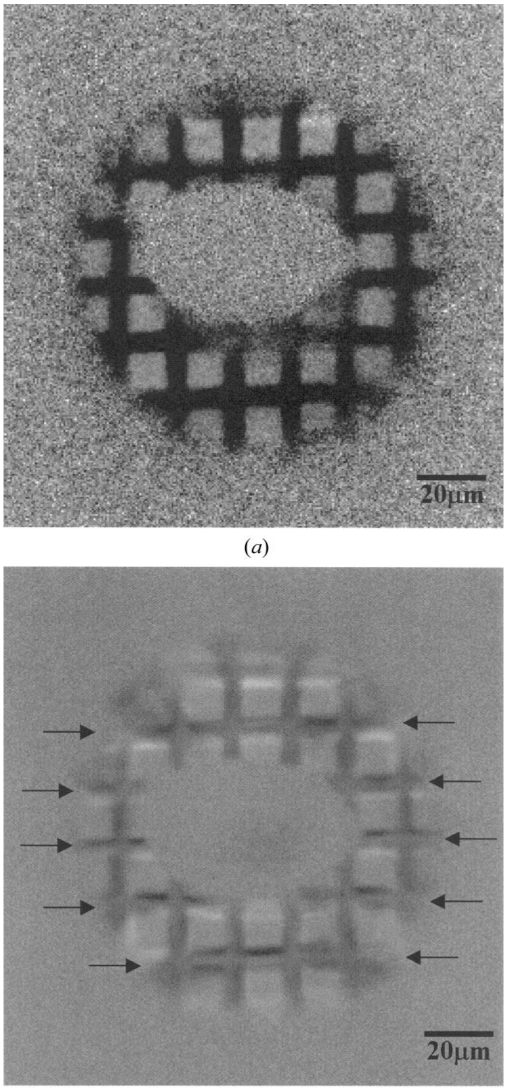


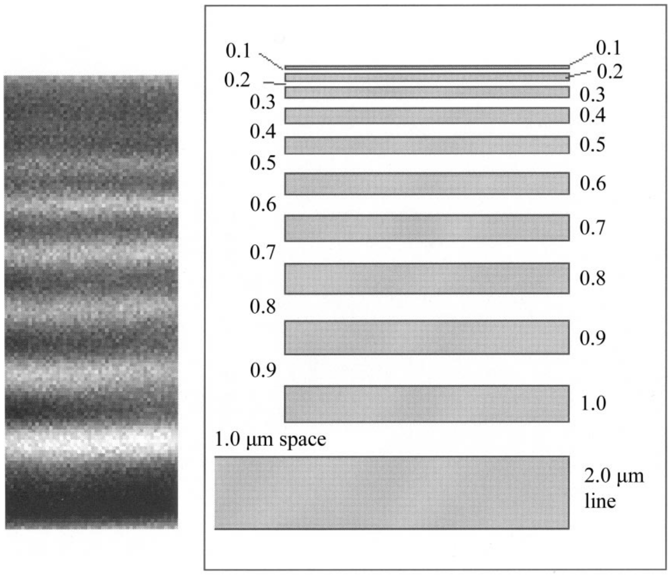
Figure 2
Measured angular spread of the illumination beam.



(*a*)

(*b*)

Figure 3
(*a*) Bright-field image of the Au mesh under incoherent illumination (magnification 10.2, exposure time 200 ms), and (*b*) the image taken by using the half-plane-type Cu phase plate at the back focal plane (magnification 10.2, exposure time 600 ms).



(*a*) (*b*)

Figure 4
(*a*) Measured image (magnification 10.2, exposure time 100 s) and (*b*) the corresponding drawing of the resolution test pattern.

Next, the spatial resolution of the Zernike-type imaging microscope was checked. A resolution test pattern, which has a tantalum (Ta) microstructure of thickness 0.5 µm (3% absorption at 25 keV) on an Si_3N_4 membrane (thickness 2 µm, 0.1% absorption at 25 keV) whose finest structure was 0.1 µm line-and-space, was used. Fig. 4(*a*) shows the measured image of the resolution test pattern and Fig. 4(*b*) shows a schematic drawing. In Fig. 4(*a*) the black lines correspond to the Ta lines. The measured image contrast of the 1 µm line-and-space is 4.3%; this is larger than the measured absorption contrast (3.6%) at the same position of the bright-field image. Therefore, the observed image of the test pattern can be thought to be a kind of Zernike-type phase contrast image. The spatial resolution test patterns down to 0.5 µm line-and-space have been observed in the vertical direction under these experimental conditions.

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