Characterization of Sputtered-sliced Fresnel Zone Plate at BL20XU: High-Resolution Hard X-ray Microbeam Experiments

X-ray microprobe/microscopy, which has been extensively developed in the soft X-ray domain, is now being extended to higher photon energies (> 8 keV). This extension will promote the various domains of basic science and technology, such as the observation and characterization of thicker materials, including medical-biological samples and industrial materials. The success of high spatial resolution studies of materials of sub-micron size is due to the X-ray brilliance combined with the availability of various micro-focusing optics. The expected applications of the microprobe in hard/high-energy X-ray regions are microscopy, microanalysis, micro-spectroscopy and micro-diffraction.

The Fresnel zone plate (FZP) fabricated by lithography technique realizes the highest spatial resolution in the soft X-ray domain, it is not thick enough to be used in the hard X-ray domain (aspect ratio (height/width): around 8:1). Compared with the FZP made by lithography technique the sputtered-sliced FZP (ss-FZP) can be made thick enough with no aspect ratio limitation and is proven to work even at quite high X-ray energies (over 100 keV) [1,2].

The ss-FZP composed of alternating transparent (Al) and opaque (Cu) layers (total 50 ~ 100 layers) was fabricated by physical vapor deposition (dc planar magnetron sputtering) on a fine gold wire core with a smooth surface and having a radius of 25 μm at a rotation speed of 15 ~ 50 rpm [3]. After deposition, the wire sample was sliced normal to the wire axis and its thickness was adjusted to 20 ~ 40 μm by mechanical polishing.

Here, two types of ss-FZP with outermost zone widths of 0.25 μm (#FZP1) and 0.1 μm (#FZP2) were fabricated. Figure 1 shows SEM micrographs of these two FZPs. The parameters are given in Table I. They were characterized using knife-edge scanning method (former) and the scanning microscopic method with a test pattern (latter). The experiment was performed at the end station of beamline **BL20XU** (hutch #2). This beamline is a unique beamline, having a 248 m-long beam path equipped with an in-vacuum undulator source and

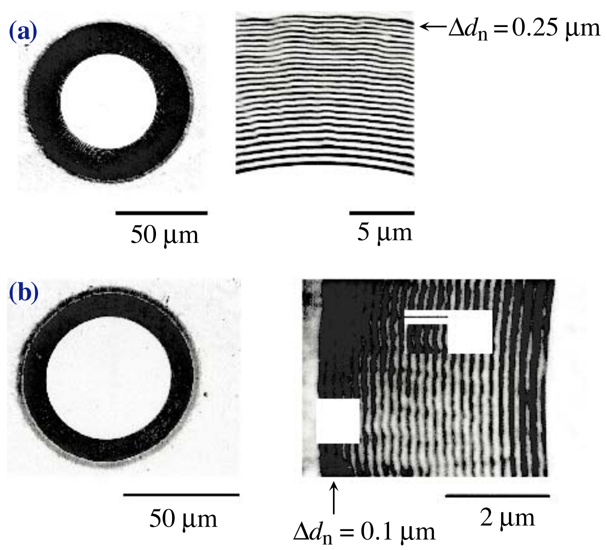

*Fig. 1. SEM micrographs showing cross sections of Cu/Al concentric mulilayers on Au wire core. Fresnel zone plate with an outermost zone widths of **(a)** 0.25 μm and **(b)** 0.1 μm.*

Table I. Zone plate parameters (Cu/Al system)

Zone plate	#FZP1	#FZP2
Outermost zone width	0.25 μm	0.1 μm
Central stop diameter (Gold wire core diameter)	50 μm	50 μm
Primary focal length at 12.4 keV (λ = 1Å)	220 mm	68 mm
Number of zone	50	50
Zone plate diameter	80 μm	70 μm
Zone plate thickness	~20 μm	~40 μm

double-crystal monochromator covering the energy range 8 keV ~ 37.7 keV. The monochromator was placed 46 m downstream from the source point. A liquid-nitrogen cooling system is employed for the monochromator.

First, we performed a characterization of #FZP1. The X-ray energy was chosen to be 12.4 keV. A quadrant slit (50 µm in horizontal width) was installed in the beamline 200 m upstream from the FZP to create a stable pseudo light source, and knife-edge scanning was performed in transmission geometry. The minimum focusing size obtained was 0.3-µm full width at half maximum (FWHM) for the horizontal direction as shown in Fig. 2, and the focal length obtained was 220 mm. A schematic view of the experimental set-up is shown in Fig. 3.

The diffraction limit of the first order focus of the FZP, 1.22 d_n (where d_n is the outermost zone width: 0.25 µm) is 0.3 µm. The focused beam size determined by the geometrical optics, 0.06 µm, is smaller than the diffraction-limited resolution of #FZP1. The focusing size obtained here (0.3 µm) agrees well with the theoretical limit of the FZP with outermost zone width of 0.25 µm. Diffraction

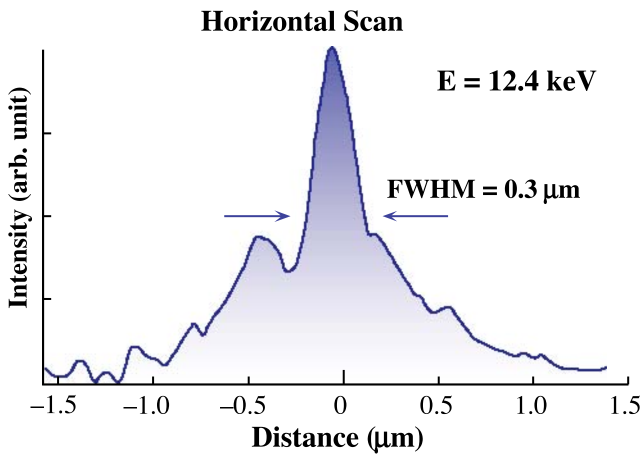

Fig. 2. Focused beam profile measured by a knife-edge horizontal scan at 12.4 keV. The full-line curve is the numerical derivative of the raw intensity data.

efficiency for the first-order light was estimated by comparing the incident beam intensity through the order sorting aperture (OSA) and the total intensity of the focused beam through the OSA. The observed efficiency of approximately 15% agrees well with that of the calculated one at 12.4 keV.

Secondly, a scanning microscopy experiment was conducted on #FZP2. Employing scanning

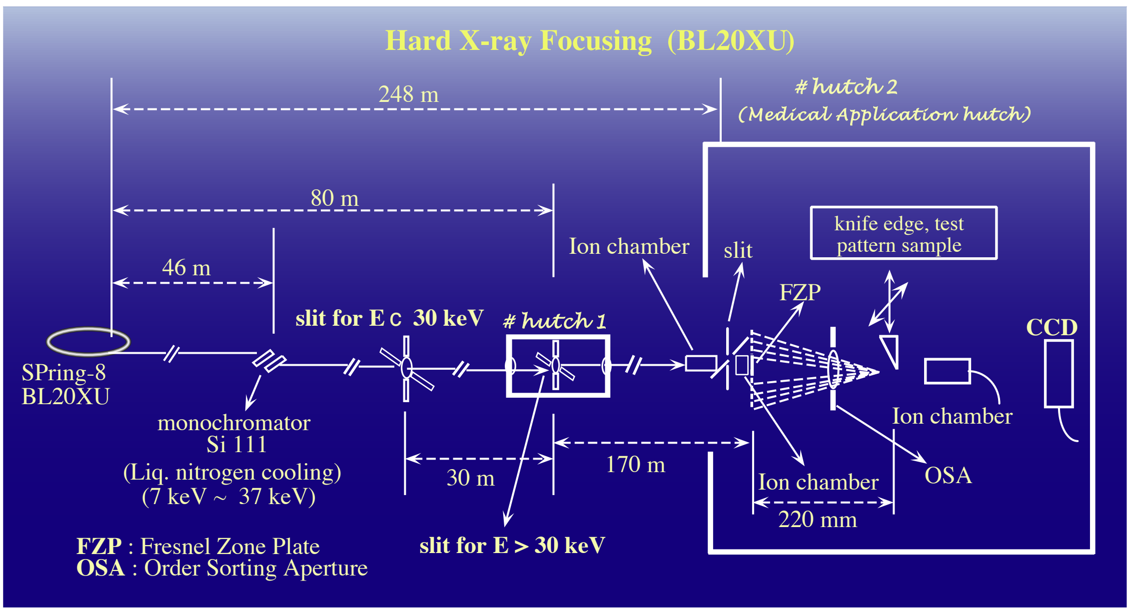

Fig. 3. Schematics of optical system of Fresnel zone plate evaluation and scanning X-ray microscopy.

microscopy to create images of the test pattern with fine structures is one method that may be used for measuring the focal beam size [1]. The X-ray energy was chosen to be 15 keV. A quadrant slit (100 μm × 100 μm) was installed in the beamline 200 m upstream from the FZP.

The designed outermost zone width of #FZP2 used here is 0.1 μm. Thus, the diffraction limit of the first-order focus of the FZP is 0.12 μm. The scanning experiment was conducted using a test pattern made of 0.5 μm-thick tantalum with seven periodic steps of 0.1 and 0.2 μm line-and-space deposited on an Si_3N_4 membrane. The result of the scanning image is shown in Fig. 4. Here, the transmitted intensity was detected using an ion chamber. The fine pattern of 0.1-μm wide was clearly resolved in the measured image. Therefore, the resolution limit of the microscope is estimated to be 0.1 ~ 0.2 μm, which is close to the diffraction-limited resolution of #FZP2. The total flux of the microbeam obtained is ~10^9 photons s^{-1}.

In conclusion, ss-FZPs have now been proven to be a genuine X-ray focusing element. It is possible to work in awide X-ray energy range (8 ~ 100 keV), even though their numerical aperture are quite small (in the order of 10^{-4}).

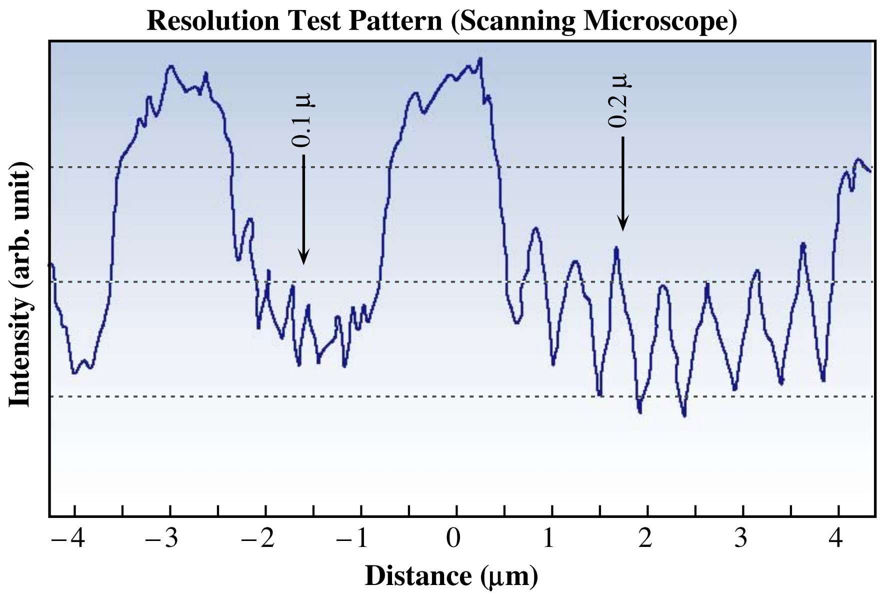

Fig. 4. Scanning microscopy image of a test pattern. Scanning step: 0.025 μm. X-ray wavelength: 0.82 Å.

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