Development of a multilayer Fresnel zone plate for high-energy synchrotron radiation X-rays by DC sputtering deposition

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Hard X-ray microscopy with high spatial resolution (≤ 0.1 μm) using a high-energy and high-brilliance X-ray microprobe is expected to be a promising technology for various types of analysis, imaging *etc.* in materials science, biology and medicine. A multilayer Fresnel zone plate (FZP) could be a promising approach to focusing optics in the high-energy X-ray region (≥ 20 keV) because a large thickness (aspect ratio) can be available. Various types of multilayer FZPs have been fabricated by DC sputtering deposition. Their focusing characteristics have been evaluated at the high-brilliance undulator beamline BL47XU of SPring-8. An optical system using a Cu/Al multilayer FZP (with an outermost zone width of 0.25 μm) as the focusing optics fabricated by the optimum deposition condition with precise film (zone) thickness control has attained an almost diffraction-limited microbeam of 0.3–0.35 μm at 8.9 keV. A line-and-space resolution test pattern has been observed: fine structures up to 0.2 μm were clearly observed in the measured image. This FZP has been working since 1995, keeping good focusing characteristics. It can be said from these results that a spatial resolution better than 0.1 μm in the high-energy X-ray region is in prospect by the development of a multilayer FZP with a narrower outermost zone width in the near future.

Keywords: Fresnel zone plates; multilayer Fresnel zone plates; hard X-ray microbeams; scanning hard X-ray microscopy.

1. Introduction

The third-generation synchrotron radiation facilities have enabled us to utilize X-rays with high brilliance, small source size and high spatial coherence. Hard X-ray microscopy with high spatial resolution below 0.1 μm using a high-energy and high-brilliance X-ray microprobe will contribute to the advanced research such as various types of analysis, imaging *etc.* in materials science, biology and medicine. With the great advance of the microfabrication process during the past decade, hard X-ray microbeams with submicrometre spot size have been formed using various optics: Fresnel zone plates (FZP) (Lai *et al.*, 1992, 1998; Kamijo *et al.*, 1997; Suzuki *et al.*, 1997, 2001; Yun *et al.*, 1999; Kagoshima *et al.*, 2000; Kamijo, Suzuki, Awaji, Takeuchi *et al.*, 2001), Bragg Fresnel lenses (BFL) (Snigirev *et al.*, 1995, 1997), Kirkpatrick-Baez (K-B) mirrors (MacDowell *et al.*, 1998; Eng *et al.*, 1998), refractive lenses (Lengeler *et al.*, 1999; Kohmura *et al.*, 2001) *etc.*

The FZPs have mainly been fabricated by a lithography-based technique or by a sputtered-sliced technique. A multilayer (sputtered-sliced) FZP should have the following advantages for focusing optics in the high-energy X-ray region: (i) a large aspect ratio can easily be realised; (ii) the kinoform-type FZP with high focusing efficiency can be fabricated (Fujisaki & Nakagiri, 1990). The large aspect ratio enables us to utilize the FZP in a higher-energy region up to 100 keV (Suzuki *et al.*, 2000; Kamijo *et al.*, 2000). The idea of the multilayer FZP was proposed by Hart *et al.* (1966) and Rudolph *et al.* (1981); later, a WSi$_2$/C FZP was fabricated and tested by Saitoh *et al.* (1988, 1989) and a Cu/Al FZP by Bionta *et al.* (1990). It can be said that such a concentric coating for a FZP is a new application of multilayer optical thin-film coatings similar to the interference coatings such as antireflection or high-reflector coatings for high-power lasers.

We have also developed various types of multilayer FZPs (Tamura *et al.*, 1998, 2000) which have proved experimentally to be effective in the high X-ray energy region (25–100 keV) at the high-brilliance undulator beamline BL47XU of SPring-8 (Awaji *et al.*, 2001; Suzuki *et al.*, 2001). A resolution test pattern with 0.2 μm line-and-space has been resolved in a scanning microscopy experiment at 27.8 keV and a pattern with 0.5 μm line-and-space has been resolved in an imaging microscopy experiment at 25 keV. In this paper, the fabrication process of the multilayer FZP is examined and an X-ray focusing test of forming a diffraction-limited microbeam is described.

2. Experimental

2.1. Coating of concentric multilayer

The outline of the fabrication process of the multilayer Fresnel zone plate is shown in Fig. 1. This process consists of two parts: a coating process and a mechanical manufacture process. Various concentric multilayer samples (W/C, Cr/C, Ag/C, Cu/C, Cu/Al, NiCr/Al *etc.*) have been deposited onto rotating Au wire substrate by DC magnetron sputtering apparatus with two DC sputtering guns (76 mm) placed at 90° from each other. Among these samples the Cu/

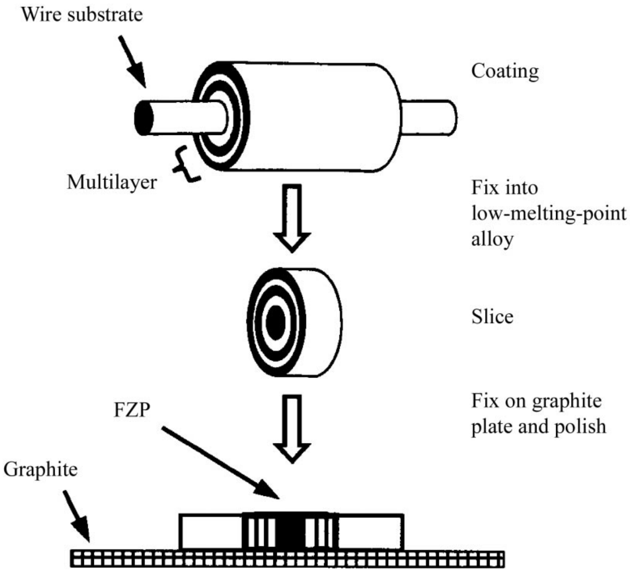


Figure 1
Outline of the fabrication process of the multilayer Fresnel zone plate.

Al multilayer has been superior because of its productivity, the smoothness of both the multilayer interface (zone) and the surface (Tamura *et al.*, 1994, 1997). We therefore describe the Cu/Al multilayer coatings in this paper. A schematic diagram and a photograph of the sputtering apparatus are shown in Fig. 2. The rotating speed of the wire substrate was 15 r.p.m. The substrate temperature was not controlled. The substrate temperature at the substrate holder measured by a thermocouple was 373–383 K. The diameter of the Au wire was 18, 25, 50 or 100 µm. The gun-to-substrate distance was 50 mm. The layer thickness was monitored using a quartz thickness monitor. A linear slit, opened on the surface of a stainless cylindrical shield, was set between the target and the substrate, which was controlled automatically so that it faced the operating sputtering gun (Yasumoto *et al.*, 2001). This cylindrical slit reduced the oblique component of the deposition flux, which resulted in forming smooth zones (multilayer interface) with a 'mirror-like surface' (Thornton, 1986) with good repeatability.

The zone width decreases gradually from the centre to the outer edge. The radius r_n of the nth zone is given by

$$r_n^2 = r_0^2 + n\lambda f, \qquad (1)$$

where r_0 is the radius of the central zone (Au wire), λ is the X-ray wavelength and f is the focal length. The spatial resolution is comparable with its outermost (minimum) zone width. The representative deposition parameters are as follows: the sputtering powers were 6 W cm^{-2} for Cu and 10 W cm^{-2} for Al, the total gas pressure was 0.2 Pa and the coating rate was 0.3 nm s^{-1} for both Cu and Al layers. An overcoat protective layer (Cu, 3 µm) was also deposited.

2.2. Fabrication of the multilayer Fresnel zone plate

After the deposition process, the multilayer sample was fixed into a low-melting-point alloy (Sn: 60%; Pb: 40%; melting point 453 K) and sliced into a plate of thickness 1 mm perpendicular to the wire axis by using a band saw microcutting machine (BS-3000; Meiwa Shoji Co.). One sliced surface was polished mechanically and the layer structure was examined by a scanning electron micrograph (SEM: S-510, Hitachi), because distortion of the zone area sometimes appears. Next, this polished surface was fixed onto the graphite plate (20 mm × 20 mm × 1 mm) by using resin glue (Loctite 420) and polished until the desired thickness was attained. Mainly a wet polishing process was implemented by using a microgrinding machine (MG-4000; Meiwa Shoji Co.). Finally, the surface was finished by a polishing suspension of 0.3 µm Al_2O_3. A dry polishing process using an Ar atom beam etching machine (806; Meiwa Shoji Co.) was also tested. The representative design parameters of the FZP are shown in Table 1, where Δr is the outermost zone width.

Table 1
Parameters of the multilayer (Cu/Al) Fresnel zone plates.

Samples	Number of zones	Diameter of substrate (µm)	Δr (µm)	Thickness (µm)	Diameter (µm)
FZP#1	50	50	0.25	20	80
FZP#2	50	50	0.15	40	65
FZP#3	40	50	0.14	30	62
FZP#4	60	25	0.10	30	40

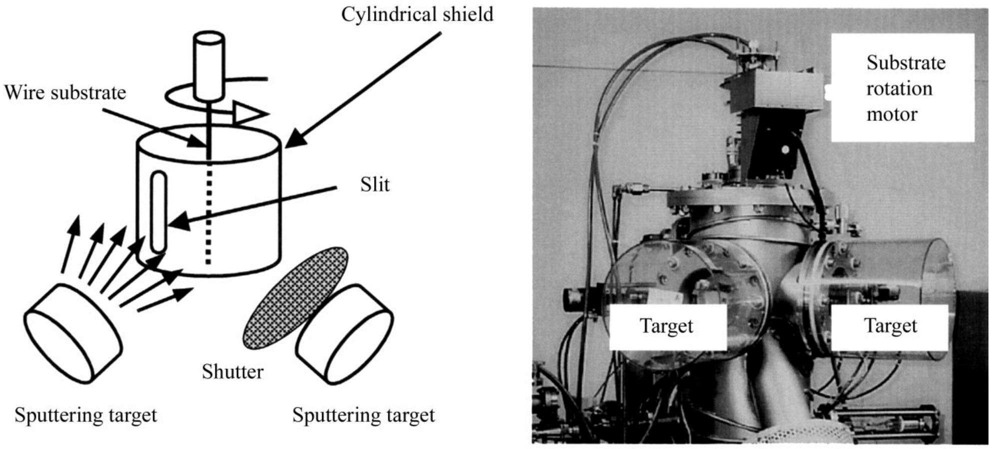
2.3. Experimental set-up and microbeam forming

The focusing test of the FZP was performed at the BL47XU undulator beamline of SPring-8. The high-brilliance synchrotron radiation beam was monochromated by an Si(111) monochromator. A slit or a pinhole (10, 20 µm) was installed at 9 m upstream of the multilayer FZP as a pseudo source. The distance between the synchrotron radiation source and the FZP was 47 m. The first-order focal beam sizes were measured and the beams were applied to the scanning X-ray microscopy experiment. The results of FZP#1 are presented in this paper. The results of FZP#2 have already been presented elsewhere by Kamijo *et al.* (2000) and Suzuki *et al.* (2000, 2001). The characteristics of FZP#3 and FZP#4 will be measured in the near future. Details of the experimental set-up and the X-ray focusing test have been reported by Suzuki *et al.* (2000, 2001).

3. Results

3.1. Cu/Al multilayers and multilayer Fresnel zone plate

The scanning electron micrographs (SEMs) of Cu/Al polished concentric multilayers for the FZPs (FZP#1, FZP#3 and FZP#4 in Table 1) are shown in Fig. 3. Each thickness was ~0.5 mm. Amplification of the zone roughness towards the top of the layer stack is observed. Düvel *et al.* (2000) have reported that a wire substrate of diameter smaller than 50 µm was not suitable for forming smooth multilayer interfaces (zones). As a preliminary experiment, we have also confirmed that a wire substrate with a larger diameter forms smooth zones. Since the W/C multilayer has formed rough zones compared with other multilayers (Tamura *et al.*, 1994), the W/C multilayer was fabricated as a typical example on both the Au wire substrate of diameter 18 µm and the glass wire substrate of diameter 0.3 mm. The effect of the substrate shape is clearly shown in Fig. 4. The oblique component of the deposition flux may have been the cause of forming rough zones (Thornton, 1986).



(*a*) (*b*)

Figure 2
DC sputtering apparatus for multilayer coating: (*a*) schematic diagram, (*b*) photograph of apparatus.

The configurations of the polished surfaces of some sliced multilayer samples were observed by atomic force microscopy (AFM; Nanopics 1000, Seiko Instruments). The AFM images of the sample polished by the dry process and the wet process are shown in

Figs. 5(*a*) and 5(*b*), respectively. The smoother polished surface was formed by the dry process, though a step was observed between the zone area and the Au wire substrate. The steps between the zone area and the Au wire substrate polished by the wet process and by the dry process were 3 µm and 1.5 µm, respectively.

These polished (observed by the SEM) surfaces were fixed onto the graphite plate using resin glue, and then the other surfaces were polished. The FZPs were fabricated from these multilayer samples by the process shown Fig. 1. The surface of FZP#1 by the wet polishing process was observed by laser microscopy (OLS 1000; Olympus Optical Co.). The SEM observation requires setting samples in a vacuum. As mentioned in §2.2, our FZPs were fixed onto the graphite plate by using resin glue, which might pollute the SEM. We therefore used laser microscopy, though high magnification (>1000) was impossible. As shown in Fig. 6, the zone area and the Au stopper area configurations were observed; fatal defects such as large distortion were not observed in spite of the mechanical thinning process down to 20 µm. We have also succeeded in thinning the multilayer FZPs to below 10 µm (Kamijo *et al.*, 1997; Tamura *et al.*, 2000).

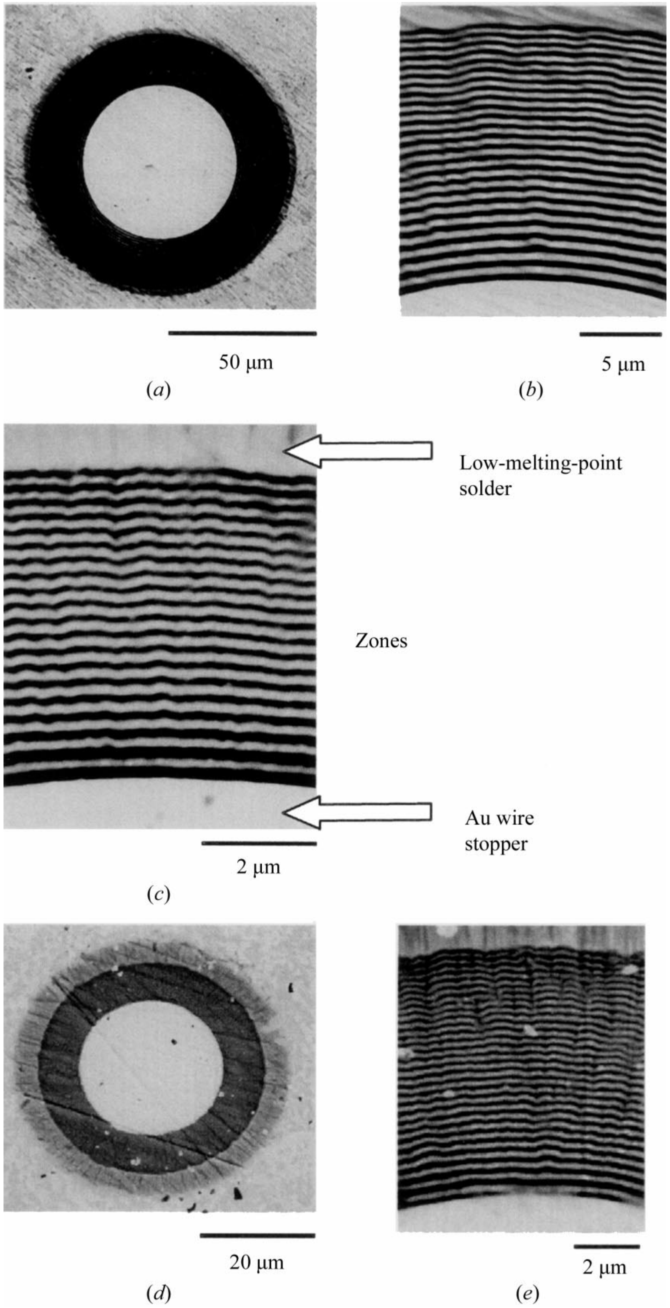


Figure 3
Scanning electron micrographs of various Cu/Al concentric multilayers prepared by DC sputtering deposition. (*a*) Full view of a multilayer with 50 layers and an outermost zone width of 0.25 µm on an Au wire substrate of diameter 50 µm. (*b*) A close-up view. (*c*) Close-up view of a multilayer with 40 layers and an outermost zone width of 0.14 µm on an Au wire substrate of diameter 50 µm. (*d*) Full view of a multilayer with 60 layers and an outermost zone width of 0.1 µm on an Au wire substrate of diameter 25 µm. (*e*) Close-up view. Black and white rings are Al and Cu layers, respectively.

3.2. Focusing characteristics of FZPs

The focused beam size [full width at half maximum (FWHM)] measured by knife-edge scanning was 0.3–0.35 µm at 8.9 keV for FZP#1, which was almost a diffraction-limited microbeam in spite of the zone roughness shown in Fig. 3(*b*). The difference between the measured data and theory ($1.22\,\Delta r = 0.3$ µm) may mainly be a result of the periodic zone roughness shown in Fig. 3(*b*). The measured focal length was 158 mm. The details of the microbeam formation experiment have been presented elsewhere (Kamijo, Suzuki, Awaji, Tamura *et al.*, 2001). The line-and-space resolution test pattern was

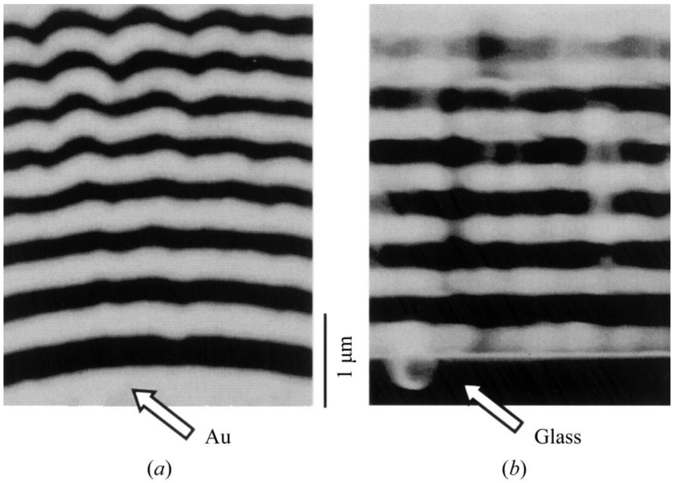


Figure 4
Scanning electron micrographs of W/C concentric multilayers: (*a*) layers on an Au wire substrate of diameter 18 µm; (*b*) layers on a glass wire substrate of diameter 0.3 mm. Black and white zones are C and W layers, respectively.

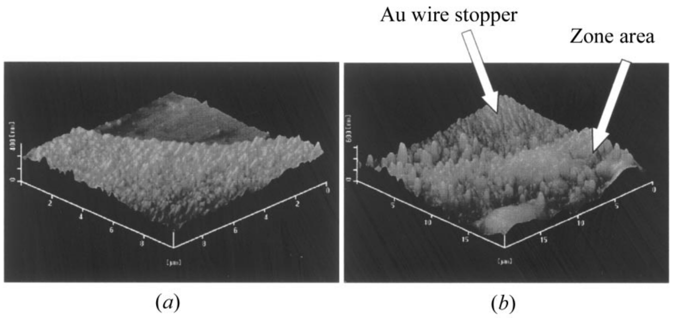


Figure 5
Atomic force microscopy images (close-up view) of the polished surface of a Cu/Al multilayer Fresnel zone plate with 50 layers and an outermost zone width of 0.25 µm on an Au wire substrate of diameter 50 µm: (*a*) finished by the dry polishing process; (*b*) finished by the wet polishing process.

observed by using this microprobe: fine structures up to 0.2 µm line-and-space have been clearly observed in the measured image (Fig. 7). This test pattern is made of Ta microstructure deposited on an Si_3O_4 membrane. This FZP has been working since 1995 with good focusing characteristics: the measured diffraction efficiency of this FZP has been 25% at 8.9 keV. This is the highest diffraction efficiency among the multilayer FZPs (Suzuki *et al.*, 2000, 2001). The representative focusing characteristics by various focusing optics, mainly for the hard X-ray region, are summarized in Fig. 8. It can be said that the multilayer FZP is the most promising optics, especially for the higher-energy X-ray region.

3.3. Consideration of synchrotron-radiation-induced damage on FZPs

As mentioned in §2.2, our recent FZPs were fixed onto graphite plates. No damage has been observed at these FZPs up to now. For example, FZP#1 has been working since 1995. However, at the first stage of our FZP development history, an acrylic acid resin plate (20 mm × 20 mm × 1 mm) was used, instead of the graphite plate, to fix the sliced multilayer sample. In other words, the FZPs were supported by pasting them onto the acrylic acid resin plates. These FZPs have been damaged by the synchrotron radiation irradiation at SPring-8, though they have worked well and one of them has presented superior focusing characteristics at the MR-BW-TL beamline of the TRISTAN main-ring of the High Energy Accelerator Research Organization, Japan (Kamijo *et al.*, 1997; Suzuki *et al.*, 1997). Strictly speaking, the acrylic plate was damaged which resulted in the exfoliation of the FZP. An 0.8 mm × 3 mm area changed from transparent to opaque and became rough; in particular, the centre of the damaged area had risen. A three-dimensional image of the risen area using laser microscopy is shown in Fig. 9. The FZP could be observed at the risen area using Nomarski microscopy. A Nomarski microscopic image around the FZP is shown in Fig. 9(*a*). It seems that this oval configuration of the damaged area reflects the shape and size of the synchrotron radiation source (vertical: 40 µm; horizontal: 900 µm). At this time, the pinhole or the slit was not used. The synchrotron radiation irradiation time was 30 min. Since the same damage (the roughness and the change from transparent to opaque) has been observed on the reverse side of the plate, this was the synchrotron-radiation-induced damage on the acrylic acid resin plate. It can be said from these results that the graphite plate is a more suitable support for the multilayer FZP than the acrylic acid resin plate.

4. Conclusions

Cu/Al multilayer FZPs have been developed by DC sputtering deposition and their focusing characteristics have been evaluated at the undulator beamline BL47XU of SPring-8. An almost diffraction-limited microbeam of 0.3–0.35 µm at 8.9 keV has been attained by the optical system using a FZP (with an outermost zone width of 0.25 µm) fabricated by the optimum deposition condition with precise thickness control. With this FZP a clear image of a fine line-and-space resolution test pattern up to 0.2 µm line-and-space was observed. It can be said from this and other recent results (Suzuki *et al.*, 2001) that a spatial resolution better than 0.1 µm in the high-energy X-ray region is in prospect by the development of a multilayer FZP with a narrower outermost zone width in the near future. We are planning to develop a multilayer FZP with high resolution below 0.1 µm for use in the high-energy X-ray region up to 100 keV, and to develop high-brilliance hard X-ray microscopy at SPring-8. A focused beam intensity of 10^{10} photons s^{-1} has already been achieved, which should be sufficient for most applications in fluorescent X-ray scanning microscopy (Suzuki, 2000). In the near future, great progress is expected in the field of focusing optics for use in the high-energy X-ray region, including recent novel optics such as a silicon refractive optics (Aristov, Firsov *et al.*, 2000), silicon planar parabolic lenses

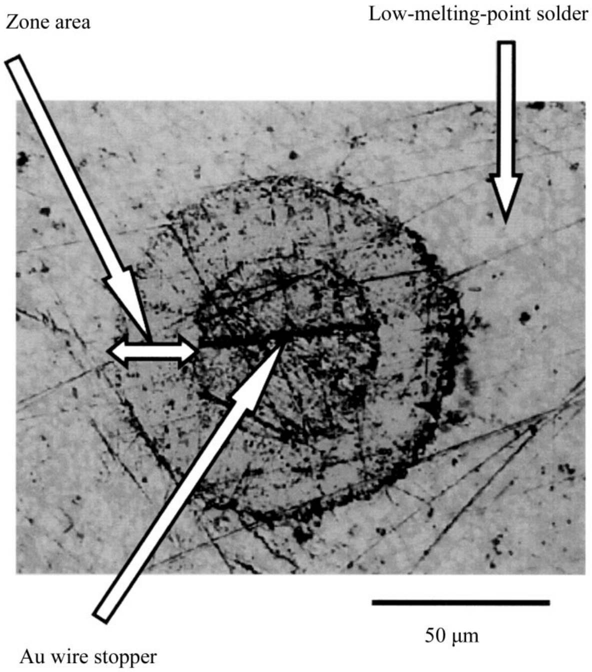


Figure 6
Laser microscopy image of a multilayer Fresnel zone plate (Cu/Al, 50 layers, 20 µm thickness).

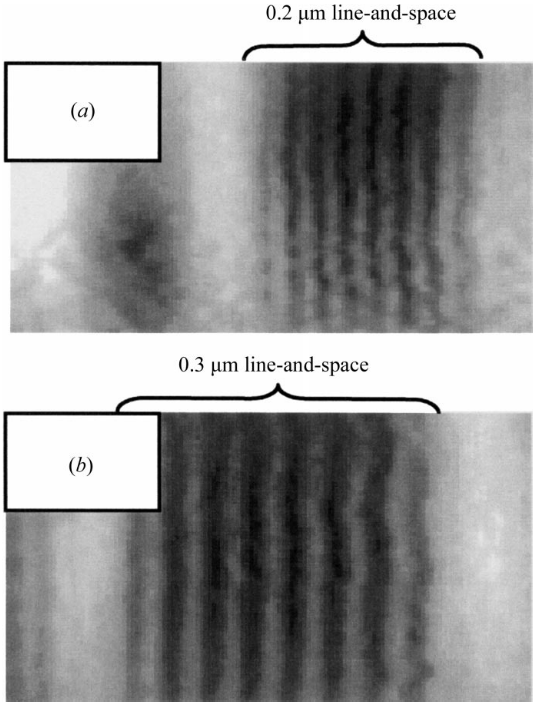


Figure 7
Scanning microscopic image of a resolution test pattern: (*a*) 0.2 µm line-and-space pattern; (*b*) 0.3 µm line-and-space pattern.

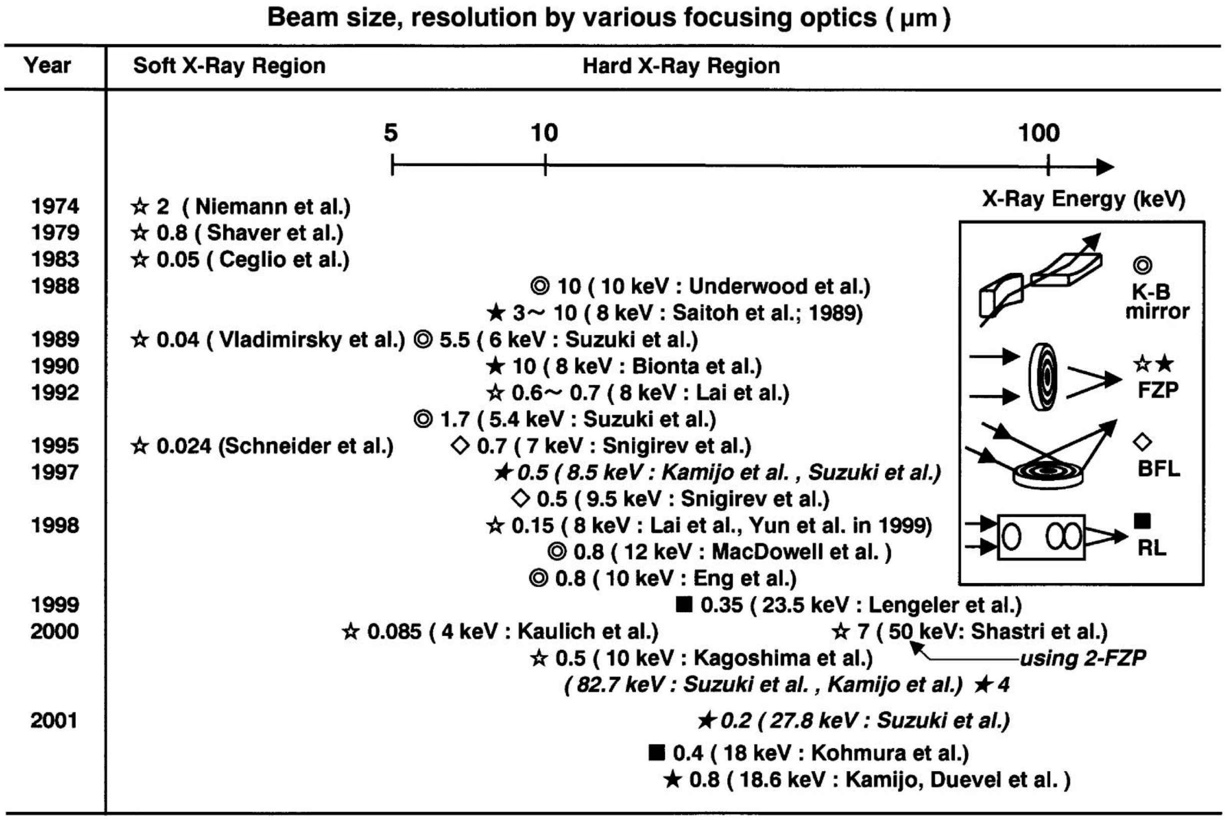
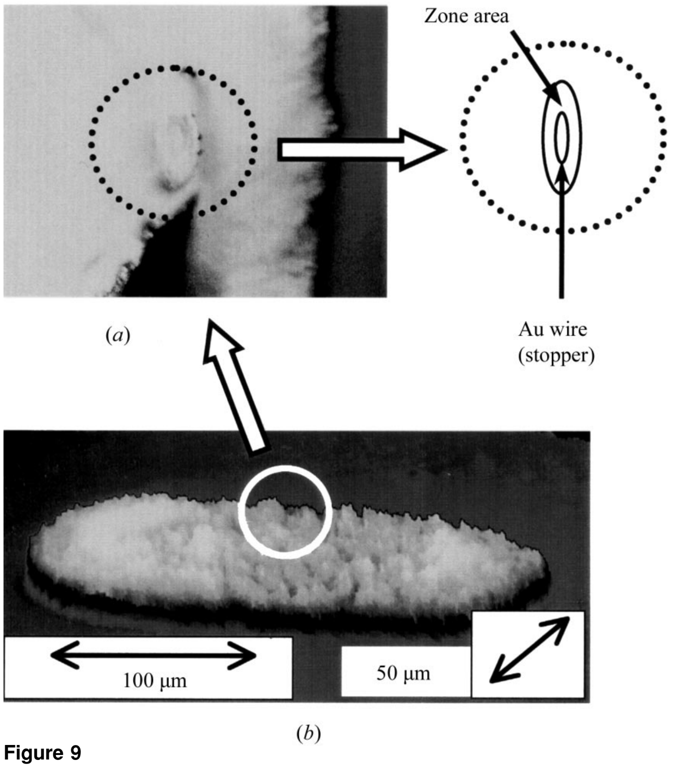


Figure 8
Focusing characteristics for the hard X-ray region by various focusing optics: Fresnel zone plates (open stars), multilayer FZP (filled stars), Bragg Fresnel lenses (BFL: open diamonds), Kirkpatrick-Baez (K-B) mirror (circles) and refractive lenses (RL: filled squares). Representative data of soft X-ray region are also shown.



(*a*)

(*b*)

Figure 9
Synchrotron-radiation-induced damage of an acrylic acid resin plate with multilayer FZP: (*a*) Nomarski microscopic photograph around the FZP; (*b*) three-dimensional image of the resin area by laser microscopy.

(Aristov, Firsov *et al.*, 2000, 2001), a bifocal lens (Aristov, Chukalina *et al.*, 2000), a single refractive lens (Zhang *et al.*, 2001) *etc.*

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