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高分辨率 X 射线衍射光学元件

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摘要：评述了 BESSY 研制的用于 X 射线聚焦的各种衍射光学元件。基于布拉格-菲涅耳光学元件，设计了高效高分辨率 X 射线聚焦和色散光学元件。描述了对长焦距布拉格-菲涅耳透镜与可变曲率半径反射镜组合所做的实验研究。用一块反射菲涅耳波带板作聚焦和色散光学元件进行了短脉冲 X 射线吸收谱(XAS)的测量。

关　键　词：衍射光学元件；布拉格-菲涅耳光学元件；波带板；同步辐射
中图分类号：O436.1；O434.19　　**文献标识码**：A

High-resolution diffraction X-ray optics

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Abstract：A review of different diffraction optical elements developed at BESSY for X-ray focusing is presented. Bragg-Fresnel optics as a basic element to design effective and high resolution X-ray focusing and dispersive X-ray optics is discussed. An experimental investigation of the combination of a long focal distance Bragg-Fresnel lens with a bimorph mirror is described. A reflection Fresnel zone plate has been tested as a focusing and dispersion optical element for X-ray Absorption Spectroscopy (XAS) on short-time pulse beamline.

Key words：diffraction optics；Bragg-Fresnel optics；zone plate；synchrotron radiation

1　Bragg-Fresnel optics

The interest in Bragg-Fresnel optics is based on their unique properties as dispersive and focusing elements. The first successful demonstrations of the Bragg-Fresnel principle were achieved in 1985-1986 using multilayer[1-2] and crystalline[3] substrates. The general principles of Bragg-Fresnel diffraction were first formulated in the work of Aristov[4]. The use of Fresnel focusing in combination with total external reflection was shown in 1994[5]. Since then Bragg-Fresnel lenses have been used at several synchrotron radiation facilities for construction of microprobes[6-7], imaging beam monitors[8] and time-resolved systems[9]. The theory of Bragg-Fresnel optics has been developed[10-11]. Unfortunately, the main advantage of the Bragg-Fresnel optics, a combination of a monochromator

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and focusing element in one device, becomes the reason for the main limitation of Bragg-Fresnel applications. The necessity to design an optical element for a particular geometry and energy is in conflict with the flexibility of the experimental arrangement and limits the number of possible experimental methods, mainly for μ-fluorescence analysis and μ-diffraction. The situation was further aggravated by the complexity of Bragg-Fresnel optics technology and the very high price of their production. Zone plates as focusing elements and X-ray waveguides as sources for nanometer scales were recognized as the main optical elements in the nano-world. On the other hand, during the last decade conventional zone plate technology has reached the theoretical limit of spatial resolution. Volume diffraction effects in the outer zones with sizes comparable to X-ray wavelengths were found to be the fundamental limitation of zone plate resolution. Shortening the wavelength towards hard X-rays increases the zone plate thickness and leads to the same volume diffraction effects and resolution limitation as for low-energy zone plates.

The results of recent investigations could refocus interest in Bragg-Fresnel optics. During the last two years at BESSY GmbH, a technology for Bragg-Fresnel optics production based on evaporation (sputtering) of metals onto surfaces of crystals and multilayers was established[12]. We reported fabrication and successful tests of a synthesized X-ray hologram made with Ni phase-shift layer on a surface of Si (111) crystal[13]. A linear Bragg-Fresnel lens placed onto the second crystal of a double-crystal monochromator was tested and will serve as a basic sagittal focusing element for the small-angle scattering facility at the BESSY microfocus beamline[14].

According to a ray-tracing analysis, sub-micron spatial resolution can be achieved without considerable loss in intensity only without pre-focusing the mirror, because of the aberrations that it introduces. Its surface profile errors introduce an additional focal spot blurring. The same problems occur in the design of long-focus optics for μ-SAS and μ-XRD methods. One needs to find optical elements that could have an "as-perfect-as-possible" profile providing micro-radian level accuracy in their slope. As a possible candidate, one can consider a combination of a bimorph X-ray mirror and a Bragg-Fresnel lens. Bimorph mirrors provide adaptive zonal control; the mirror surface can thus be tailored to the incoming X-ray wave-front by means of a classical Hartmann test using up to 8 independent electrodes located in the mirror itself. It is then possible to reduce surface profile errors to about 0.2″ rms and also to strongly damp the contribution of low-frequency components of the mirrors surface′s power-spectral-density-function. Moreover, the mirror can correct not only its own slope errors but also distortion of the incoming X-ray wave-front due to other optical elements.

A bimorph mirror with an optical surface length of 550 mm and 8 independent electrodes was used. This device can operate with a large variation in a focal distance range of 1.5～4 m and at 0.15° grazing angle. The vertical angular acceptance is on the order of 0.05 mrad.

The horizontal beam focusing is achieved by means of a sagittal Bragg-Fresnel lens. As shown in Fig. 1, several Bragg-Fresnel lenses are placed on the second monochromator crystal, in our case Ge (111), which provides about 30 %

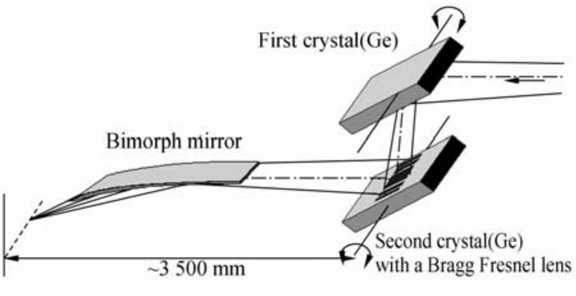
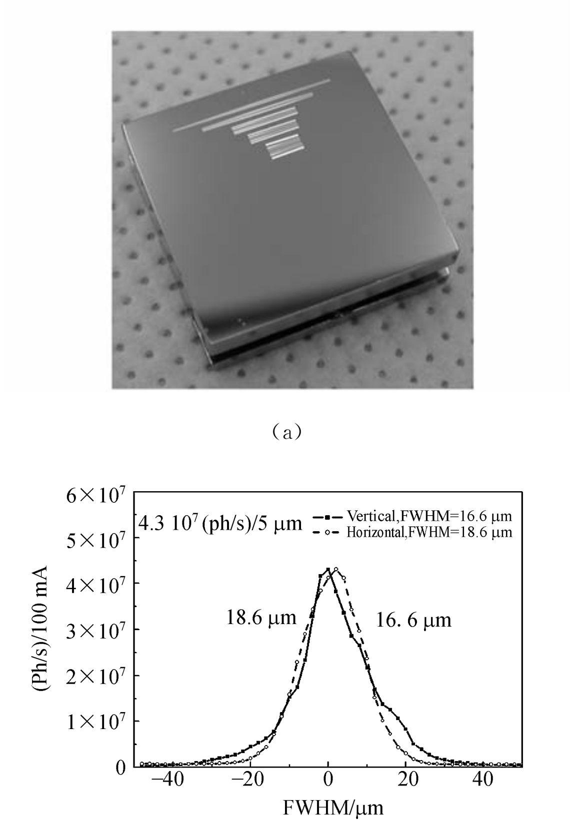


Fig. 1　μ-SAS focusing system employing a Bragg-Fresnel lens and bimorph mirror higher flux in comparison with a Si (111) crystal. A Fresnel structure was fabricated using a metal coating on a perfect Ge crystal in the shape of a Fresnel zone plate. In the case of such a coated zone plate, the beam is transmitted twice through the thickness of a metal coating at the Bragg grazing angle θ_B. In comparison with etched zone plates the value of the optimal thickness is reduced by a factor of 0. 5 sin θ_B. The difference in optimal thickness is most significant for high energies. According to calculations, even with an aspect ratio of about 1 ∶ 1 (layer thickness equal to a groove width) an outer zone width of the Bragg-Fresnel lens for 30 keV can reach 0. 2 μm.

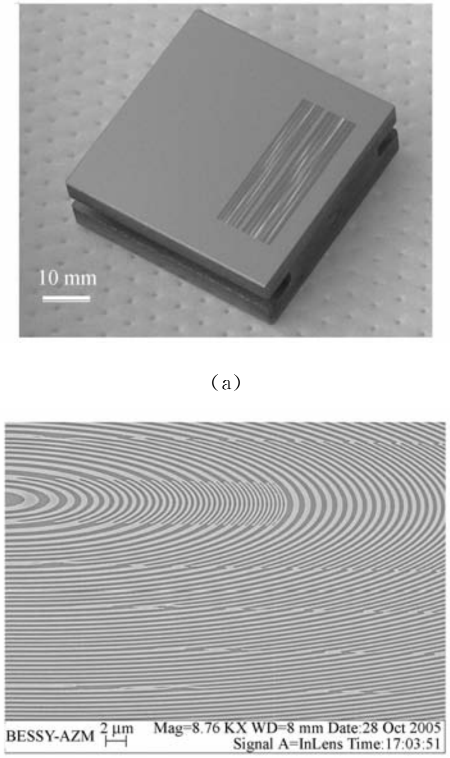
The combination of the linear BFL and bimorph mirror allows a small-range energy scan without reducing the spatial resolution. Several lenses fabricated on the same substrate will cover the entire operational energy range of the beamline. Special attention must be paid to beam position stabilisation for the microfocus experiments. Using the mirror/BFL combination in the direct monochromatic beam, the vertical and horizontal focal size can be decreased as FWHM ~18. 6 μm(h) ×16. 6 μm (v) with maximal divergence of 1 mrad as shown in Fig. 2(b).



(a)

(b)

Fig. 2 Five Bragg-Fresnel lenses optimized at 4, 7, 9, 16, 24 keV photon energy on the surface of a Ge monochromator crystal (a). Horizontal and vertical FWHM of the focal spot measured by 5 μm Pt pinhole at 9 keV (b)



(a)

(b)

Fig. 3 Two elliptical Bragg-Fresnel lenses combined first to fifth order of diffraction (12 keV and 16 keV) on the surface of a Ge(111) crystal (a). Microphotograph in electron microscope of the RZP structure. E-beam lithography is done by BESSY GmbH (b)

The smaller focal spot size with higher flux can be achieved using a so-called "modified zone plate"[15] exploiting the first, third, fifth, seventh and ninth orders of diffraction simultaneously. Fig. 3 shows an elliptical Bragg-Fresnel lens on a Ge (111) crystal with a focal length of 2 700 mm at 16 keV photon energy. The lens has shown a flux density gain on the order of 800 in comparison with direct beam for a focal spot of 8 μm (h)×15 μm (v). The result of the BFL test at 16 keV photon energy is shown in Fig. 4.

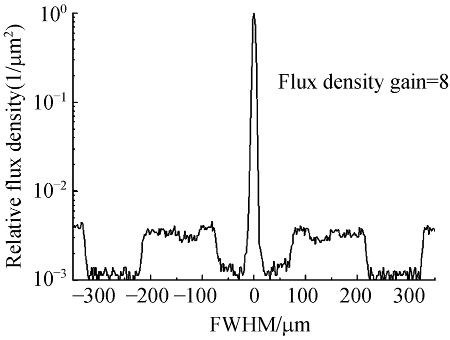


Fig. 4　Horizontal scan of a focal spot at the energy of 16 keV, FWHM=8 μm

In both cases a gold mask placed on the surfaces of a Ge 111 crystal has been used to perform X-ray focusing. The optimal thickness of the gold layer has been found to be of the order of 200 nm for the photon energy range 4～30 keV. In this energy range a theoretically calculated maximum diffraction efficiency of the order of 30% for the linear sagittal lenses and 15% for the elliptical lenses has been measured experimentally.

2　Reflection zone plate monochromator

Elliptical zone plates fabricated on a total external reflection mirror surface, the so-called "Reflection Zone Plate" (RZP), can be effectively used for an X-ray monochromatization and beam focusing at photon energies below 1 000 eV. This element can be applied in the beamline with specific beam conditions, such as very high thermal and radiation load or very low flux, when it is necessary to use only one optical element in the optical design to reduce a loss. It is important that the RZP be off-axis to provide the best energy and spatial resolution.

Development of an off-axis RZP at BESSY is closely connected with a project of the BESSY soft X-ray free-electron laser and ultra-high time-resolved experimental beamline for the "time-slicing" undulator.

The principle design of the RZP monochromator is shown in Fig. 5. As a prototype we took a transmission zone plate monochromator, developed at Advanced Light Source (ALS), Berkeley[16]. This design provides a rejection of the zero-order beam and fine focusing of the monochromatic beam on an optical axis. A small aperture placed in the focal plane provides high energy resolution of the monochromator on the order of $\lambda/\Delta\lambda \sim 1\ 000$. Among important characteristics of the slicing-beamline monochromator are not only energy resolution, but time resolution. The monochromator optics should not "blur" short X-ray pulses, in our case the characteristic time is of the order of 30 fs.

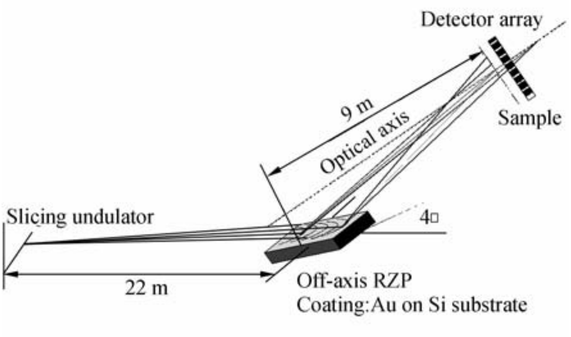


Fig. 5　Optical layout of the RZP monochromator

The number of grooves in the diffraction structure along the beam direction should not exceed:

$$N_{\max} = \frac{\Delta t_{\text{pulse}}}{\Delta t_{\lambda}}\ , \qquad (1)$$

where Δt_{pulse} is the X-ray pulse duration and Δt_{λ} is the time delay for the one-wavelength path. For a Fe K-edge radiation Δt_{λ} is given by

$$\Delta t_{\lambda} = \frac{\lambda_{\text{Fe}}}{c} \approx 0.578 \times 10^{-17}\ \text{s}\ . \qquad (2)$$

Therefore the maximum number of grooves is equal to $N_{\max} < 5\ 200$. This value defines a maximum aperture and maximum energy resolution of the optical element.

A structure of the RZP was calculated using the computer program described, for example, in [15]. The calculation parameters are: source - zone plate distance of 22.5 m, zone plate - sample plane distance of 9.02 m and angle of incidence 2°. The Fresnel lens has an offset of 400 mm with respect to the optical axis which provides an effective rejection of the zero-order radiation and "Bremsstrahlung" from the undulator.

In Fig. 4 an optical scheme for a focusing monochromator exploring off-axis RZP is shown. The lens with size of 86 mm × 10 mm has a focal length of 6.4 m. The groove period in the central part of the lens is of the order of 18 μm and in the outer part about 1.3 μm.

The reflection Fresnel-zone plates were produced in cooperation between BESSY GmbH, Leibniz Institute for Surface Modifications in Leipzig and Microlithography and Consulting (ML&C) Company in Jena. The lenses are designed using the same software as a Bragg-Fresnel lens at BESSY. A single-crystal silicon substrate is covered by a gold layer with a thickness of 50 nm; the depth profile is 10 nm. The technology of the lens fabrication includes laser beam lithography and ion etching. Three Fresnel lenses designed for energies of 715 eV, 785 eV and 861 eV which correspond to the L-absorption edges of Fe, Ni, and Co were fabricated on the same substrate with a diameter of 100 mm. The optical element of the monochromator for the slicing beamline is shown in Fig. 6.

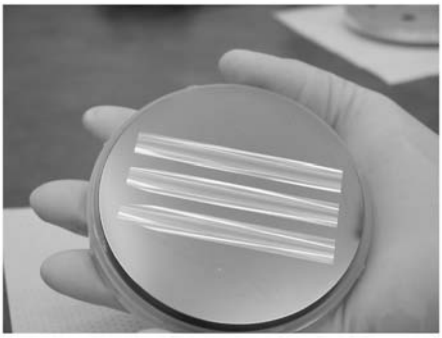


Fig. 6 Reflection Fresnel zone plates for energy 715, 785, 861 eV. Focus distance is 9.02 m

The monochromator was tested both with the direct undulator beam and white light radiation. With direct undulator radiation, a fluorescence screen was used to visualize an intensity distribution in the focal plane of the RZP. The result is shown in Fig. 7 that represents an image obtained with a light containing three undulator harmonics: first at 262 eV; third at 765 eV and fifth at 1 308 eV. The RZP is optimized for an energy of 765 eV and produces a focused spot at this energy. The other lenses produce a similar distribution in a focal plane, optimized for the other two energies. Note that the beamline has only one optical element, therefore total transmission of the beamline of the order of 6% is much higher than a traditionally designed beamline with minimum three reflection surfaces (~0.6%).

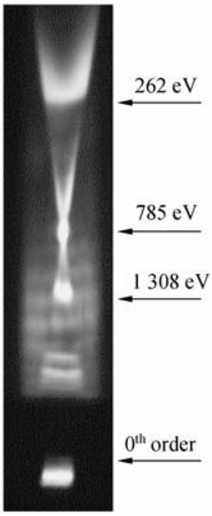


Fig. 7 Intensity distribution in focal plane of a RZP

In Fig. 8, transmission of spectra of Co measured by monochromators using a grating and the RZP are compared. It was found that the RZP monochromator works well for this study.

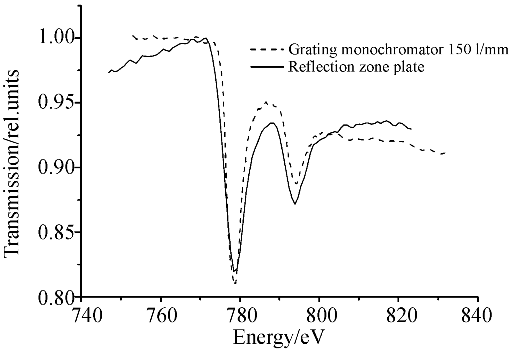


Fig. 8　Transmission spectra of a Co foil of 40 nm thick

3　Conclusions

We have presented two examples of the application of RZP at the BESSY synchrotron radiation facility. First, a hard X-ray linear and elliptical zone plates on the surface of Ge (111) crystal are used for fine X-ray focusing at a long distance. Linear RZP was used in combination with a bimorph adaptive mirror and an elliptical zone plate to produce a two-dimensional focal spot with flux density gain on the order of 800. Both lenses were placed on the second crystal in the double-crystal monochromator at the BESSY microfocus beamline.

Second, a soft X-ray elliptical RZP etched on the surface of a total external reflection mirror was tested in the monochromator of the BESSY slicing beamline. Such optics can be used with existing third-generation synchrotron radiation sources, as well as with X-ray laser sources, *e. g.*, free-electron lasers.

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