Making hard x-ray micro-focus beam with Fresnel zone plate optics

-SPring-8 summer school text-

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Contents

1. Introduction - a simple and intuitional explanation of Fresnel zone plate (FZP) -

It is difficult to use refractive optics in the hard x-ray regions, while the refractive lens is most popular optics for visible light. This is because the index for refraction in the x-ray region is very near to the index for refraction of vacuum. The discrepancy from unity is only 10^{-5}-10^{-6} for any materials. Therefore it is very difficult to deflect the x-ray beam by refraction at the interface of optical media, and any optical devices that are used in visible light optics, lens or prism, cannot be used for x-rays. Although, some refractive lenses or prisms are developed in the hard x-ray region, they are still rare case. At present, the Fresnel zone plate (FZP) is a widely used and practical optics in the x-ray regions, the x-ray energy around 10 keV or higher energy regions. The micro-focus beam size of about 30 nm is already achieved with the FZP optics in the hard x-ray region.

The Fresnel zone plate is a concentric transmission grating with radially decreasing grating period, as schematically shown in Fig. 1, coarse grating period at central part, and finer pitch at the outer (marginal) area. An optical micrograph of FZP is shown in Fig. 2. This is a real Fresnel zone plate used as micro-focusing optics or image forming optics at the SPring-8 beamlines.

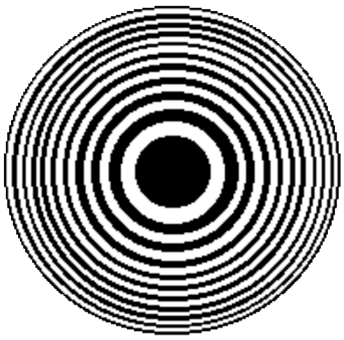

Fig.1. Schematic drawing of FZP structure. The radius of n-th ring is defined by $r_n = \sqrt{(n\lambda f)}$. λ is x-ray wavelength and f is focal length.

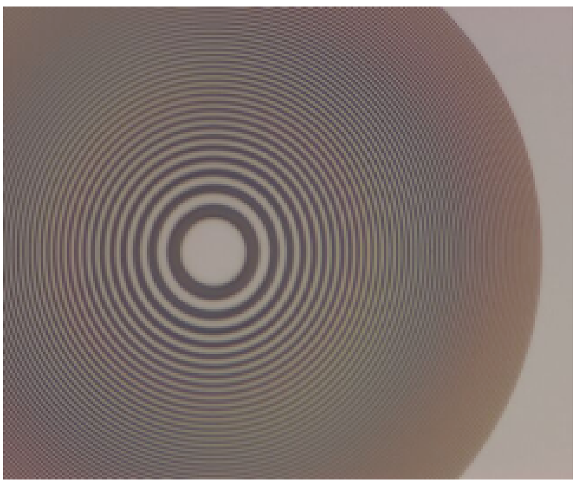

Fig. 2. An optical micrograph of FZP used at the SPring-8 imaging beamlines as microfocus lens and/or

microscope objective lens. The diameter of FZP is 100 μm and the outermost zone pitch (period) is 0.5 μm. The focal length of the FZP is designed to be 248 mm for 1.0 Å (12.4 keV) x-rays. The zone structure is made of 1 μm-thick tantalum deposited on a 2 μm-thick silicon nitride membrane. This FZP will be used in our experimental course.

In order to understand the FZP optics intuitionally, it is useful to start from two-slit optics, so called Young's two-slit. The two-slit is a most simple and basic tool in wave optics. As shown in Fig. 3, when a pair of slit is placed with a distance of *d*, intensity distribution on a screen far from slit has periodic structure. When the observation angle θ satisfy an equation, $n\lambda = d \sin \theta$, the intensity is maximum, and minimum is observed at $(n + 1/2)\lambda = d \sin \theta$. Here, *n* is an integer number called order of diffraction. If the incident radiation is perfectly coherent and the slit width is negligibly narrow, the observed intensity distribution must be sinusoidal! But the perfect coherence is not realistic in general experimental condition. Therefore, the actual interference pattern becomes weak far from optical axis, as shown in Fig. 3.

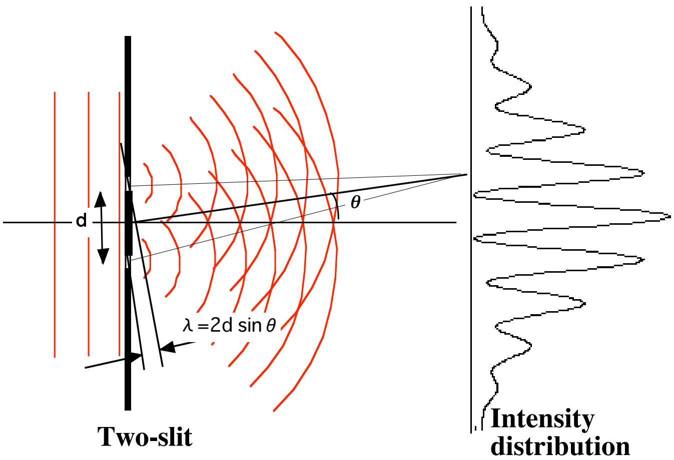

Fig. 3. Schematic diagram of Young's two-slit experiment and its interference pattern,

Next, let us consider diffraction by grating: a periodic slit array. As shown in Fig. 4, the beam deflection angle at a grating with a period of *d* is described as $\sin \theta = n\lambda/d$, where $n = 0, \pm1, \pm2, \pm3$,,,,,. The integer *n* is also called order of diffraction. The $n = 0$ is undiffracted beam.

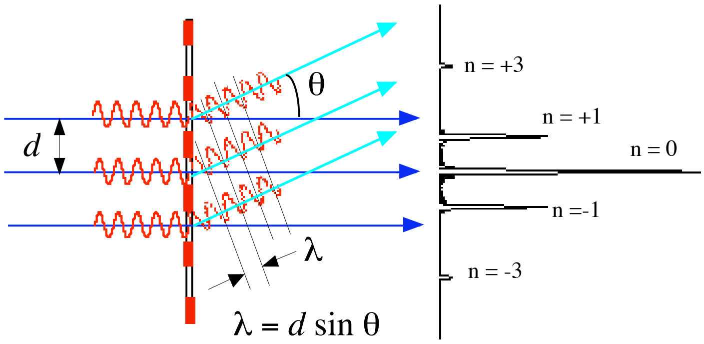

Fig. 4. Linear grating and diffraction pattern by the grating. The intensity distribution (diffraction pattern of grating) is given by the Laue function as $sin^2(\pi N d sin\theta)/sin^2(\pi d sin\theta)$, where N is number of grating period. Even order diffractions, n = 2, 4, 6 ,,, are not allowed for 1:1 ratio of obstacle and transparent area, and higher order diffractions are weaker than the fundamental diffraction.

When the grating period is changed as $d = \lambda/sin\theta$, as shown in Fig. 4, all the diffracted rays are focused at a point, where θ is determined by $tan\theta = r/f$, and the r is radius of circular grating, and f is a specific distance from zone plate.

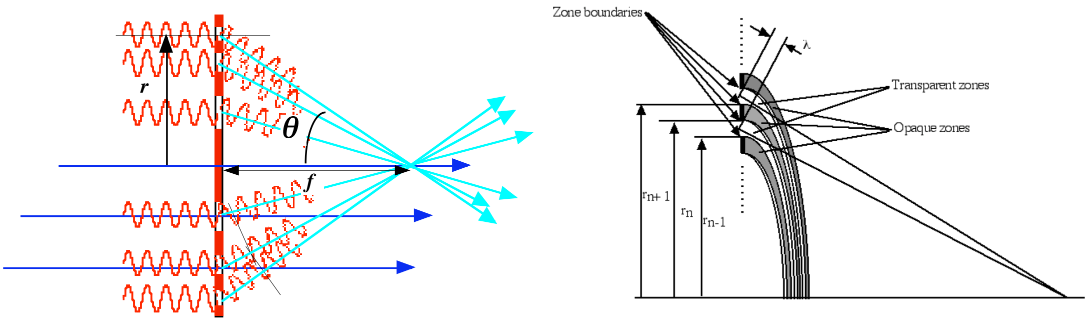

Fig. 5. Diffraction at Fresnel zone plate (schematic diagram), and focus of x-ray beam.

The specific length f is called focal length. Assuming $\theta << 1$, this condition is usually satisfied in the hard x-ray region, diffraction condition, $d\theta \sim \lambda$, for n-th ring can be written as

$$(r_{n+1} - r_{n-1})\, r_n / f \sim \lambda,$$

where, $\theta \sim r_n /f$, and $d \sim (r_{n+1} - r_{n-1})$. Then, regarding the above equation as a differential equation of

$$2\partial r(n)/\partial n = \lambda f / r(n).$$

By solving the above equation, we can get an equation for Fresnel zone plate structure as

$$r_n^2 \sim n\lambda f + \text{Const.},$$

generally Const. = 0, and $r_n = \sqrt{(n\lambda f)}$.

This is only an approximation, because the above discussion is valid only for large n and small diffraction angle. However, this intuitional explanation is very useful for understanding the geometrical meaning of FZP optics.

As is understandable from these considerations, there are positive and negative orders of diffraction. Thus, the zone plate acts as a convex lens for visible light, and it simultaneously works as a concave lens with negative order diffraction. The 0^{th} order and higher order diffractions usually exist. Therefore, a kind of spatial filtering is needed in FZP optics to work as an optical lens. A typical method of selecting a required order of diffraction is putting a small diaphragm near the focal point. When the diameter of aperture is sufficiently smaller than the FZP diameter, the undesired order of diffractions including direct beam are suppressed enough. If the suppression is not sufficient, an additional beam stop disc may be used for diffraction order selection as shown in the figure. This beam stop disc is usually added in fabrication process of FZP, and it is called center stop, or center obstacle. The aperture near the focus is called order-selecting-aperture (OSA), or sometimes order-sorting-aperture.

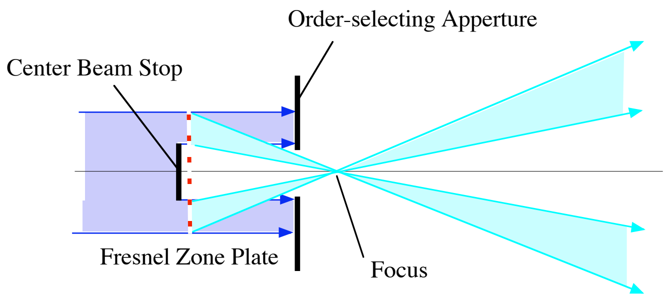

Fig. 6. Diffraction-order-selection in FZP microbeam optics.

2. More precise treatment of FZP optics

The exact equation of FZP structure is obtained by optical path difference and expansion of Fermat's principle. The Fermat's principle is that an actual optical path between any two points should be shorter than any other paths that connect the two points. By considering the finite wavelength, the *n*-th zone boundary of a Fresnel zone plate that focuses the spherical wave emitted from point A to the point B is expressed by the following equation,

$$(R_a + R_b) = n\lambda/2 + (a + b),$$

as shown in Fig. 7, and

$$(a^2 + r_n^2)^{1/2} + (b^2 + r_n^2)^{1/2} - (a + b) = n\lambda/2, \quad (1)$$

where a is a distance from point A to the FZP, b is a distance between B and FZP, and r_n is n-th boundary of zone structure, and n is integer. This formula is a basic and exact equation of Fresnel zone structure. Using a Taylor expansion and neglecting higher order terms, the equation (1) is rewritten as

$$a + r_n^2/2a + b + r_n^2/2b - (a + b) = n\lambda/2. \quad (2)$$

Then,

$$r_n^2/a + r_n^2/b = n\lambda. \quad (3)$$

Again, by defining $1/a + 1/b = 1/f$, the equation (3) is rewritten as

$$r_n^2 = n\lambda f. \quad (4)$$

The equation, $1/a + 1/b = 1/f$, is well-known Newton's lens equation. Thus, an FZP whose zone boundary is defined by the above equation works as a lens with a focal length of f.

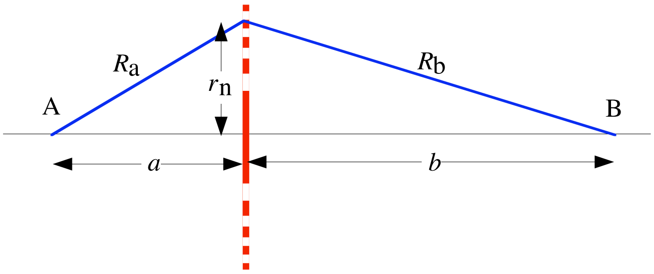

Fig. 7. Optical path description of Fresnel zone plate

3. How is the limit of focused beam size? Diffraction limit and geometrical limit of resolution

Ultimate spatial resolution (focused beam size limit) of imaging optics is determined by the diffraction of light. The diffraction-limited resolution is deduced from the uncertainty principle in quantum mechanics. If a photon is squeezed within a small space of $\langle\Delta x\rangle$, the momentum uncertainty of photon, $\langle\Delta p\rangle$, is given by following formula,

$$\langle\Delta x\rangle \langle\Delta p\rangle \geq h/2\pi,$$

where h is the Plank constant. Consequently, larger spread of momentum is required to focus (localize) a photon in a smaller area. This is a fundamental limitation of microfocus beam size. The momentum of photon is given by the formula, $p = h/\lambda$, and the uncertainty of momentum is related to the angular spread of momentum. As shown in the figure, when a parallel incident beam is focused by a lens, the angular spread of focused-photon's momentum becomes $(h/\lambda)sin\theta$, and momentum uncertainty becomes $\Delta p = \pm (h/\lambda)sin\theta$. Then the limit of focused beam size, Δx, can be written as

$$\Delta x \geq 2h/(2\pi\Delta p) \sim \lambda/(\pi sin\theta),$$

The term, $sin\theta$ is known as numerical aperture (NA) of lens. The *F-number* defined by $F = 1/NA$ is usually used in optical instruments instead of NA. In the hard x-ray region, the $NA << 1$, and $sin\theta$ is approximately equal to r_N/f, where the r_N is radius of outermost zone.

This discussion is one-dimensional condition, and we cannot get an exact form of intensity distribution. The lens optics, including FZP optics, is an axis-symmetric system. For the axisymmetric optical systems, the formula of the resolution limit is slightly deformed as

$$\Delta x = 0.61\ \lambda/NA,$$

This definition is well-known Rayleigh's criterion for spatial resolution of microscope with an objective lens of circular aperture. This formula was obtained by a classical diffraction theory for electromagnetic wave, far before the birth of quantum physics, but essentially the same as that derived from quantum mechanics. This relation comes from a definition of quantum photon momentum, $p = h/\lambda$.

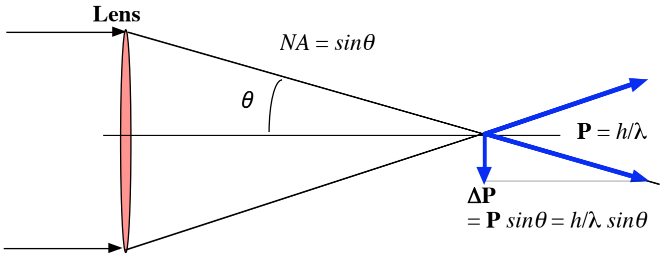

Fig. 8. Numerical aperture of objective lens.

The diffraction limit can also be explained by a simple diffraction theory for single slit, as described below. When a parallel and monochromatic beam is incident on a slit with an opening of w (one dimensional case), the propagating wave through the slit is not a parallel wave anymore.

Using the Kirchhoff-Huygens' principle, the electromagnetic wave field is given by an integral formula of

$$E(\theta) = \int \exp(ik\, x \sin\theta)\, dx$$

Here, k is a wave number defined by $k = 2\pi/\lambda$. Taking a integral over the slit opening, the amplitude of electromagnetic wave far from the slit is given by the following function.

$$E = \sin(kw/2 \sin\theta) / (kw/2 \sin\theta).$$

The function $(sin X)/X$ is the *sinc function* which frequently appears in the diffraction theory. The beam intensity far from slit takes maxima on the optical axis ($\theta = 0$), and first zero (local minimum) at

$$(kw/2 \sin\theta) = \pi,$$

$$\text{or} \quad \lambda = w \sin\theta \sim w\theta,$$

because w is usually much greater than wavelength in the hard x-ray region. Thus, the angular spread (blurring) of the propagating wave is broadened by passing a slit. It is equivalent that a finite angular convergence of incident beam is necessary to focus the beam within a limited area. Saying differently, a finite divergence of outcoming wave is always accompanied by focusing a photon beam. This result of the diffraction theory of electromagnetic wave is essentially the same as the uncertainty principle in the quantum physics.

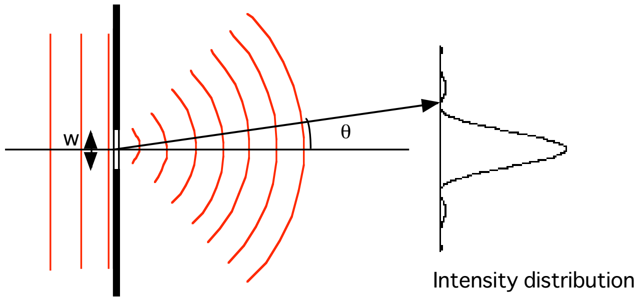

Fig. 9. Diffraction by a single slit. Radiation through the slit is broadened by propagating in a free space.

It is also apparent from the diffraction by a grating that the $sin\theta$ is equal to λ/d, where d is the outermost zone period of FZP. Then, for the circular grating (FZP without center stop), the diffraction-limited resolution is defined as

$$\Delta x = 0.61d.$$

A formula of $\Delta x = 1.22\ dr_N$ is frequently used instead of above equation, where dr_N is a half pitch of the outermost zone period (or width of outermost zone). Then, the ultimate limit of the focused beam size is nearly equal to the width of outermost Fresnel's zone of the objective FZP. This relation can also be derived from the Fresnel zone plate equation and the formula of diffraction-limited resolution.

However, the actual focus is generally limited by the quality of incident beam. We have implicitly assumed a parallel beam for explaining how the FZP works as beam focusing element. But, in any light sources, the emitted radiation is neither a perfect plane wave, nor a perfect spherical-wave. The radiation is emitted from a finite source area, and is emitted in random direction, a chaotic source! In this case, the beam focusing should be considered as generation of a demagnified image of the light source. This limitation of focused beam size is known as the geometrical optics limit. This operation of microfocusing optics is similar to that of telescope optics. As shown in Fig. 10, the light source image is formed at focus by a magnification factor of b/a according to the Newton's equation. Then the focused image size, S_b, is given as

$$S_b = S_a\, b/a,$$

where S_a is source dimension, a is source to lens distance, and b is lens to focus distance. Usually $a >> b$, then $b \sim f$. A long distance from source point or a small source size is necessary to achieve small (micro) focus beam. The factor S_a/a is sometimes called angular size of light source. In most synchrotron radiation facilities, the angular size is not small enough to generate microfocus beam, and the distance is limited by beamline length. So, we need to makeup a virtual source by putting a small pinhole (or slit) at the upstream section of the beamline. The slits equipped at beamline, front-end slit or transport-channel slit, are usually used as the virtual source for micro-focusing experiment. It is also important that there are no disturbances of wave front by the optical elements in the beamline. The monochromator crystals, mirrors, and vacuum windows usually tend to give serous deformation of the x-ray beam direction and the wavefront shape.

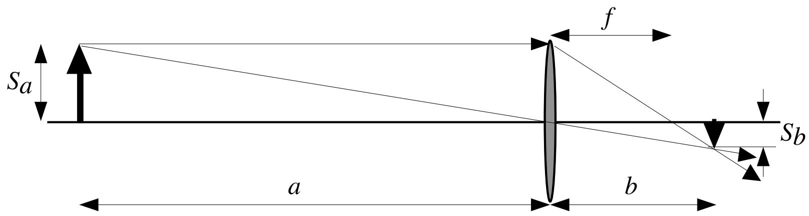

Fig. 10. Geometrical description of beam focusing optics

4. Diffraction efficiency of FZP

The diffraction efficiency is an important characteristic of diffraction-based optics, because the intensity of focused beam is mainly determined by the diffraction efficiency, and the diffractive optics such as FZP or grating can gather only a portion of incident radiation. The diffraction efficiency of FZP is equivalent to the linear grating. Therefore, we can calculate it by solving one-dimensional Kirchhoff's integral for single period of gating. For instance, first order diffraction of opaque and transparent zone structure with 1:1 zone ratio, the intensity of diffracted beam is given by

$$I = |E|^2, \text{ and } E = \int C * \exp(ik\, x \sin\theta)\, dx,$$

Here, the integral is taken for one cycle, d, of grating, and $2\pi/k = \lambda = d \sin\theta$. The constant C = 1 for transparent area, and C = 0 for opaque zone. Then, for the first-order diffraction,

$$E = \int \exp(2\pi i/d)\, dx = 2$$

By normalizing with the incident beam intensity of $(2\pi)^2$, $I = 4/(2\pi)^2$ ($=1/\pi^2$ ~ only 10% for 1st-order diffraction). The general form for n-th order diffraction can be easily calculated by the similar manner. It is known that the n-th order diffraction efficiency for transparent and opaque zone, i.e., black and white zone, with even zone width (1:1 zone ratio) is $1/(n\pi)^2$, where $n = \pm1, \pm3, \pm5$,,,,. The even order diffraction does not exit for the grating with 1:1 groove width.

More efficient grating is realized by the phase-modulated structure that is the utilization of phase shift through the zone, instead of stopping the beam with opaque zone. The maximum diffraction efficiency is attained at a phase shift of half wavelength (phase shift of π). The efficiency of n-th order diffraction for ideal phase grating of 1:1 zone ratio is $4/(n\pi)^2$ ($n = \pm1, \pm3, \pm5$,,,,.). So, the first order diffraction has an efficiency of about 40% in the case of ideal phase-modulated zone plate. However, in the x-ray region, pure phase material without absorption dose not exist. All the optical media is complex of phase and absorption effect.

More efficient FZP can be made by using so-called kinoform structure, that is recently introduced in hard x-ray regions. Optimized kinoform FZP can give nearly 100% efficiency, if the absorption loss can be ignored. Real diffraction efficiency is lower than 100%, but the diffraction efficiency of higher than 50% is already achieved in the hard x-ray region with quasi-kinoform structure.

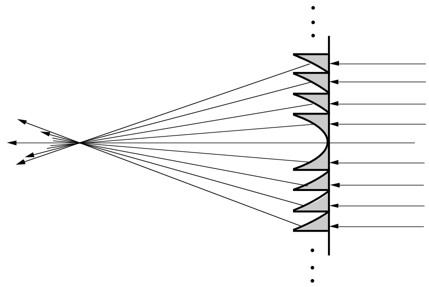

Fig. 11. Schematic diagram of kinoform zone plate.

5. Depth of focus and chromatic aberrations

If the wavelength is slightly different from designed value, the focus slightly moves on the optical axis. That means different wavelength x-rays are focused at a different position. The focal length f varies inversely as the wavelength of x-rays. These phenomena are called chromatic aberrations. Therefore, monochromatic x-ray beam is required for FZP microbeams. Saying differently, the FZP microbeam optics can be used as a monochromator or spectrometer.

Geometrical beam size at a distance of Δf from the exact focus can be calculated to be $2NA\,\Delta f$, as shown in Fig. 12. This value should be smaller than the diffraction-limited resolution in order to achieve the full-performance of FZP optics. Then the condition can be written as

$$0.61\,\lambda/NA \geq 2NA\,\Delta f,$$

or using the FZP equation, we can get a simple and useful formula, as

$$0.61/(2N) \geq \Delta f/f.$$

Here, N is number of zones.

The chromatic aberration is also estimated by similar manner, because $\Delta f/f = -\Delta\lambda/\lambda$ from the FZP equation. So, the chromatic aberration is characterized by the number of Fresnel zones, N.

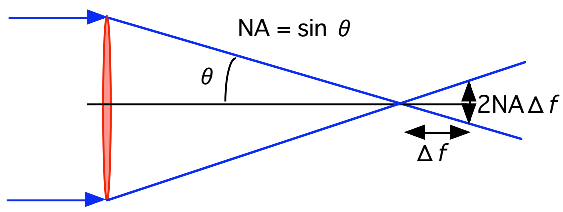

Fig. 12. Depth of focus

6. Fabrication of zone plates:

Most of zone plate for x-ray microscope is fabricated by using a state-of-art technology developed in semiconductor technology, i.e., electron-beam lithography technique. Recent ULSI technology makes it possible to fabricate sub-100 nm microstructure on a silicon tip. This is also a key technology in x-ray microscopy. Schematic drawing of cross sectional structure is shown in Fig. 13. The zone plate pattern is drawn by electron-beam to the resist on thin membrane on silicon wafer. The thin membrane is usually a few μm-thick silicon nitride or silicon carbide. The pattern transfer from photo-resist to zone material is done by reactive dry-etching process or wet electroplating process (usually, dry-etching is used for tantalum, and electroplating is employed for gold pattern). Finally, the silicon wafer of pattering area is removed by chemical etching process, as shown in the figure.

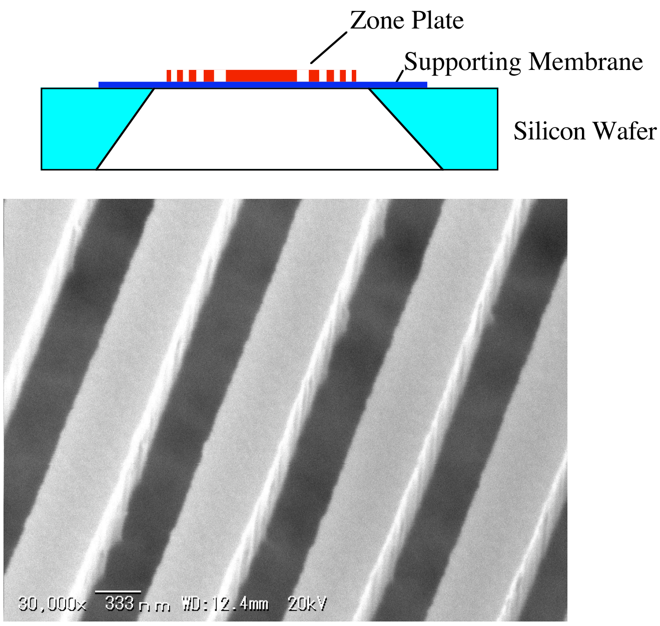

Fig. 13. Structure of Fresnel zone plate. Cross-sectional view and SEM micrograph.

The difficulty on fabrication of x-ray zone plate is high aspect ratio (zone-height/zone-width) of zone structure at the marginal zone area. In the x-ray region, the phase shift is much greater than the absorption contrast even for the high-Z elements. Therefore, most of the x-ray FZPs is designed as a phase-modulation zone plate. Assuming a free electron approximation, the real part of index for refraction, n, for x-rays is written as

$$n = 1 - \delta, \quad (7)$$

$$\delta = 1.35 \times 10^{-6} \rho(\mathrm{g/cm^3})\, \lambda\, (\text{Å})^2, \quad (8)$$

where ρ is density of material, and Z/A = 1/2 is also assumed (Z: atomic number, A: atomic weight). The optimum thickness of zone plate, *t*, is equivalent to the optical path difference of λ/2, that is calculated by $t\delta = \lambda/2$. Then, high dens material is preferable for zone plate. Considering the manufacturing process of microstructure, gold or tantalum is usually chosen as a zone material for hard x-ray FZP. The optimized thickness and δ for tantalum zone plate, for instance, is 2.9 μm and 1.71×10^{-5} at an x-ray wavelength of 1.0 Å, respectively. Then, the aspect ratio should be 29 for the FZP having 100 nm-zone-width. The required aspect ratio is still challenging even in the present nano-fabrication technology. The resolution limit of FZP (width of outermost zone) is being improved year by year, and the finest zone structure of about 20 nm is possible at present. However, the thickness of these high resolution FZPs is far from ideal value for hard x-rays.

7. Preservation of emittance and brilliance

Finally, an important law in beam-focusing optics is described in this section. An emittance preservation rule. As shown in Fig. 14, when a magnified (or demagnified) image of light source is formed by an optical element, there is a relation between the image dimension, S_a, S_b, and angular spread, θ_a, θ_b as

$$S_a\ \theta_a = S_b\ \theta_b\ .$$

The product of beam dimension and angular spread, $S\theta$, is called the beam emittance, and the above formula represents the emittance preservation rule. This emittance preservation is deduced by the geometrical optics. The efficiency of optical system is not 100%, even if a perfect lens (without any aberrations) is used. This is because the focus beam size is limited by the diffraction. Therefore, the equation should be rewritten as

$$S_a\ \theta_a \leq S_b\ \theta_b\ , \text{ for general optics.}$$

If the absorption loss at optical system is ignorable, the flux of focused beam, I_b, is equal to the flux of the incident radiation, I_a, as

$$I_b \leq I_a$$

and $I_b = I_a$ for ideal case.

Then, flux, I, divided by the source dimension, S, and the angular divergence, θ, is constant as

$$I_b, / (S_b\ \theta_b) = I_a / (S_a\ \theta_a), \text{ for the ideal optics.}$$

This relation is called brilliance preservation rule, and the flux density limit of focused beam is determined by this equation. The flux density of the focused beam is generally higher than that of the unfocused beam, where the unfocused beam is a microbeam generated simple by putting a micro-pinhole in front of sample. Although, the beam intensity seems to increase by focusing optics, there is no real gain in beam brilliance. The brilliance is always preserved in any ideal optical systems, and the actual brilliance always decreases by passing through the optical element, because the throughput, reflectivity of mirror or diffraction efficiency of gating and FZP, is always less than 100%. Remember that any optical systems decrease the brilliance, and increase the emittance. The highest brilliance is obtained only at the initial source point! This is an important principle of beam focusing optics.

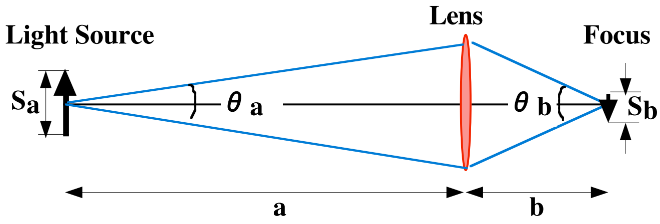

Fig. 14. Schematic diagram of emittance preservation rule

Useful references,

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Basic exercises, before the experiment

1. Calculating the radius of some rings of the FZP in Fig.2, and total number of zone.

2. Calculate the focal length of the FZP in Fig. 2 for 8 keV x-rays.

3. Calculate the exact focal point for a given source to FZP distance (depending on the beamline optics).

--> for example, source to FZP distance of 46 m (BL47XU)

4. Calculate the geometrical spot size, assuming a beamline where you will do the microfocusing experiment.

--> Source size of SPring-8 is ~40 μm vertically and ~600 μm horizontally.

5. The monochromaticity of x-ray beam is sufficient for the FZP in Fig.2 at your beamline (with a conventional crystal monochromator)?

--> Look at the optics of beamline from WEB or ask to beamline staff.

6. Calculate the depth of focus for the FZP shown in Fig. 2.

7. How is the optimized thickness of zone for 8 keV x-rays? Assume the zone material of gold or tantalum.

8. Drawing the sinc function and Laue function for a given slit and grating.

--> 10 μm list, 1 Å wavelength, and 100 line grating.

9. Introducing a relation between required monochromaticity ($\lambda/\Delta\lambda$) and number of zone (N), using the Laue function for N-period grating.

10. Which kind of pin-hole should be used as the order-selecting-aperture? Diameter, mateial and thickness, considering work distance of optics.

--> Once, use 0.5 center stop disc.

11. Calculate the zone plate parameter of the FZP in Fig. 2 with a center stop of 50 μm-diameter.

Practice at the beamlines

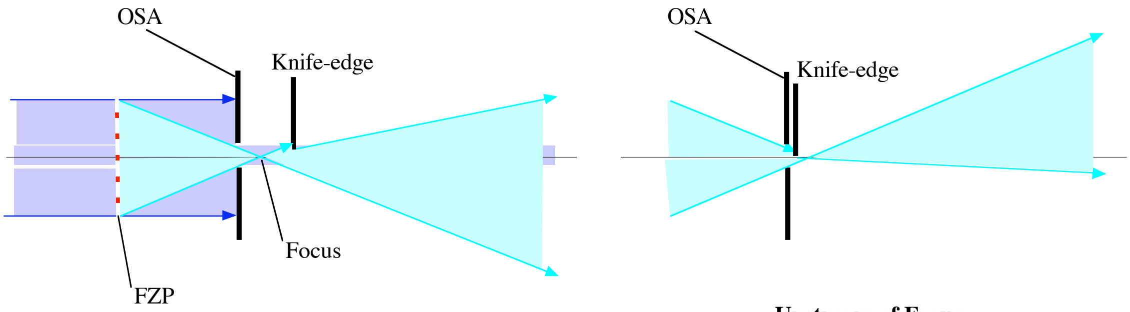
1. General understanding on the beamline optics.

2. Finding FZP and order-selecting-aperture, using x-ray imaging detectors.

3. Alignment of FZP and OSA on optical axis.

4. Searching for the fundamental (first-order) diffraction.

5. Finding the focal point by means of Foucault's knife-edge test.



Downstream of Focus **Upstream of Focus**

6. Measurement of beam size by differential knife-edge scan method, and comparison to theoretical value.

7. Changing the front-end slit size, dependence of focus beam size on spatial coherence condition.

8. Only at BL47XU, measurement of stigmatism for light source, and elimination of the stigmatism by anti-bending of pre-mirror.

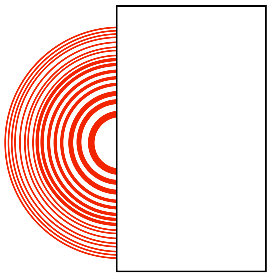
Advanced exercises,

1. Calculate the diffraction efficiency, and comparison to the experimental result.

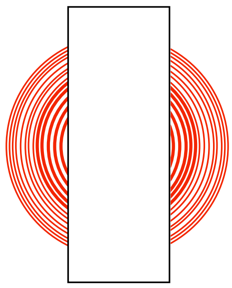
2. Calculating and experimental testing of chromatic aberrations, how is the tolerance of wavelength and bandwidth?

3. Theoretical estimation and experimental testing the depth of focus by changing the position of knife-edge.

4. Influence of mask in front of FZP, shading half of FZP aperture. Both vertical and horizontal focusing properties. This experiment must be done for the diffraction-limited resolution case.



5. Influence of mask in front of FZP, center obstacle. This experiment also requires diffraction-limited resolution.



6. Inserting something in the optical path, how is the disturbance of wave-front, for instance, putting a sheet of paper, Kapton foil, aluminum foils, etc?

7. Inclination of FZP and focusing properties, how is the tolerance for angular misalignment?

8. Focusing properties of higher order diffractions, how is the diffraction-limited resolution, and diffraction efficiencies?

9. The longitudinal source size of SPring-8 undulator is 4.5 m. Does this length give some problem on focusing properties?

10. Focusing properties for off-axis undulator radiation. How is the effective angular size of undulator radiation? and its relation to inclination to optical axis (electron orbit). This experiment should be done at the short beamlines. The BL47XU is preferable.