Refractive Optics for Modifying X-Ray Wavefronts

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Introduction

Diamond Light Source (DLS) is the UK synchrotron light source with over 30 beamlines, many operating in the X-ray part of the spectrum. The facility has an Optics Group with active research in areas such as modelling of beamlines, development of X-ray optics, and X-ray beam characterization using techniques such as X-ray wavefront measurements. Many optics developments have benefited from using the DLS test beamline, B16 [1], a versatile dipole magnet beamline, for developing techniques and for optics test experiments. A recent project has been the development of microfabricated refractive structures for modifying the X-ray wavefront to extend and improve the performance of beamline optics. At a synchrotron radiation source, X-radiation is emitted by relativistic electrons in the storage ring passing through regions with high magnetic fields. The electromagnetic (em) radiation field emitted by different electrons in the source is uncorrelated and if the spatial distribution of the source is sufficiently large, perfect X-ray focusing optics will produce a direct image of the electrons in the source.

With the latest generation of SR facilities—the so-called 4th generation—the electron spatial distribution at the source is small (of order 10 µm rms in both horizontal and vertical directions) and the radiation field at beamline optics becomes highly correlated in the transverse direction. At a focal plane, where these correlated fields overlap, the intensity distribution is determined by interference and for “ideal” focusing optics, the focus size is determined by the diffraction limit. In this highly correlated regime, a useful concept is that of the wavefront [2]. The wavefront is a surface given by the locus of the point at which the phase of the em field is constant. For slowly varying field amplitude and a wavefront that is locally smooth, the wavefront propagates along its normal direction.

The wavefront error can be defined as the displacement along the wavefront normal of the actual from the ideal wavefront. In Cartesian coordinates \((x, y, z)\) with the \(z\) axis pointing along the optical axis, the wavefront error can be expressed as a function of the two transverse coordinates \(w(x, y)\). Wavefront error is generated as the em wave encounters optical elements in the beamline such as, for example, monochromators, X-ray mirrors, refractive lenses, and X-ray windows and is given by the variation in the accumulated X-ray path-length. In the case of reflecting optics, the wavefront error is directly related to the figure error \(h(x, y)\) as shown in Figure 1, by

\[
w(x, y) = 2h(x, y)\sin(\theta(x, y))
\]

where \(\theta(x, y)\) is the local incidence angle on the mirror. For reflecting optics, the wavefront error is given by the path-length change \(w(x, y) = \delta(E)l(x, y)\) where \(\delta(E)\) is the X-ray energy-dependent real part decrement of the refractive index and \(l(x, y)\) is the error in the thickness profile of the refractor.

For a constant amplitude monochromatic beam, the complex em field can be obtained directly from the wavefront error up to a constant phase factor by \(E = E_0 \exp[i2\pi w(x, y)/\lambda]\) where \(\lambda\) is the wavelength of the radiation. The field obtained can be propagated to

![Figure 1: Pathlength change on reflection. An incident wave reflects from the surface A at an angle \(\alpha\). If the surface is offset by distance \(w\) due to figure error, the path length increases by \(2w \sin(\alpha)\). For X-ray mirrors operating at incident angle \(\alpha\) of a few millirad, nanometer figure errors translate into picometer scale wavefront errors. For a monochromator crystal operating at about 100 millirad angle, the wavefront is about 30 times more sensitive to the figure error.](https://example.com/fig1.png)
the focal plane using the Fresnel-Kirchhoff equation [3] as is done by wave propagation modelling programs such as SRW [4]. The wavefront after the final focusing optical element propagates to the focal plane and determines the wave amplitude and hence the X-ray intensity distribution.

Wavefront measurement

The wavefront can be directly measured by a number of methods, such as the grating method, Hartmann sensor, speckle tracking method, knife-edge method, and ptychographic reconstruction. We have employed the ptychography method [5, 6] and also the knife-edge method [7].

For the knife-edge method, a precision knife edge is scanned through the focal plane of a focusing optical element and the X-ray intensity is measured on an area detector located in the far field, downstream from the focal plane. The layout is shown schematically in Figure 2. Each detector pixel is treated as a separate point detector and the measured pixel intensity vs knife position is fitted by an error function that gives the knife position where the detector pixel intensity is cut by the knife-edge. For an ideal wavefront (with zero wavefront error), the scanned knife-edge cuts the intensity from each pixel on the detector at the same knife-edge position and therefore any deviation in that position gives the error in the wavefront slope. In addition, as the focal spot is small (typically sub-micron), the wavefront slope at the detector pixel can be projected back to a unique position on the wave propagation plane at the focusing optical element. So the wavefront error slope (in the direction of the knife-edge scan) is obtained by this method. The wavefront error slope can therefore be converted to the wavefront error by integration. Measurements by this method indicate that the wavefront error slope can therefore be converted to the wavefront error by integration. The accuracy of this method is limited by the spatial resolution of the detector. The wavefront error slope can therefore be converted to the wavefront error by integration. The accuracy of this method is limited by the spatial resolution of the detector. The wavefront error slope can therefore be converted to the wavefront error by integration. The accuracy of this method is limited by the spatial resolution of the detector.

Using refractors to modify the X-ray wavefront

Modification of the X-ray wavefront can be done to compensate for imperfect optics, the so-called wavefront correction. This is done using a correcting optical element that can apply a controlled pathlength variation to reduce the wavefront error on the field propagating to the sample. The correcting optic need not be positioned downstream of the optical element causing the wavefront error and often the best position is immediately upstream. Often the wavefront error for the horizontal (x) and vertical (y) directions are decoupled—$w(x, y) = w_x(x) + w_y(y)$—for example, for KB mirrors or crossed planar lenses. In this case, the applied correction can also be decoupled with separate one-dimensional corrections for horizontal and vertical directions. Another application of wavefront modification is to control the profile of the focus beam; for example, to provide a variable beam size.

Adaptable mirrors in which the curvature along the surface is varied dynamically have been successfully used for wavefront correction [8] employing a bi-morph mirror in which a number of piezo elements are incorporated into the mirror to allow the local curvature to be altered [9]. A second method is to use specially designed refractive structures made using micro-fabrication techniques. The use of refractors for this purpose was first demonstrated using refractive elements fabricated by the LIGA process to correct the wavefront from an X-ray mirror with a known surface modulation [10]. Later applications followed using refractive structures fabricated by various methods to correct the wavefront from X-ray lenses [11, 12].

Refraction of X-rays is very weak compared to visible light so that, for example, a practical 2D X-ray lens must be made using hundreds of individual paraboloid profile refractors; that is, a compound refractive lens (CRLs) [13]. The perturbation of the wavefront required for focusing is much larger than is required for these applications of wavefront manipulation. For example, for wavefront correction a single refractive structure with feature thicknesses of order 100 µm is sufficient. In this case, the weakness of X-ray refraction is an advantage as changes to the wavefront of 1 Angstrom may be achieved by features with a thickness on the scale of 10–100 µm, which is easily achievable using present-day fabrication methods. Other advantages of using refractive wavefront manipulating elements are that they do not cause a change in the optical axis (unlike a reflecting corrector) so that the element can be withdrawn from the beam without changing alignment of the optics. A disadvantage is that refraction is X-ray energy dependent so if the X-ray energy is changed, the corrective element must be also be modified; however, fabrication techniques allow a large number of accurately positioned structures to be made on a single substrate.

The LIGA fabrication process is a method for making planar structures laid down on a substrate [14]. The features are that the side walls are perpendicular to the substrate and are very smooth. Structure depths of order 1 mm or more are possible to fabricate and they can be fabricated using the polymer SU-8. This makes the structures ideal for use...
for refractors for X-rays. The refractive structures were designed at DLS and fabricated at the lithography beamline BL-7 at the INDUS-2 synchrotron [15].

Modifying the wavefront to vary the X-ray focus size

At SR facilities, there has been much interest in wavefront correction for applications such as micro-probe experiments that require the smallest possible focus; to achieve this, the wavefront error should be minimized. There are applications, however, where it is required to generate a specific wavefront error. An example is macro-molecular crystallography (MX) experiments where a small beam size relative to the sample size results in a higher rate of radiation damage to the sample [16], and therefore, ideally for these experiments, the focused beam profile should be matched to the sample dimension in order to reduce the X-ray power density. Our initial work on manipulating the wavefront was done in order to obtain a variable beam size at a MX beamline at Diamond Light Source [17]. The beamline has a pair of elliptical mirrors focusing horizontally and vertically onto the sample. To provide a variable focus beam size in the vertical direction, we designed a mirror with seven parallel lanes running the length of the mirror, each with a different surface profile, and by transversely translating the mirror a different lane could be illuminated with switching times of order 1 second being feasible. Initial wavefront simulations were done using one-dimensional wavefront propagation to determine the best mirror surface profile for obtaining a range of beam sizes with smooth focus profiles [18]. The mirror surface profiles were the basic elliptical profile with a superimposed profile consisting of parabolic arcs with different amplitudes to obtain the seven focal spot sizes with fwhm values 0.4, 0.8, 1.4, 2.3, 4.0, 5.9, and 7.4 µm. This mirror was recently commissioned on the beamline and is now used routinely.

Following on from this, we investigated the use of refractive elements to achieve the wavefront modulation. The method chosen for fabrication of the refracting structures was the LIGA technique because it produces planar structures laid down on a silicon wafer substrate with structure depths to 1 mm or more and with highly perpendicular and smooth structure side walls. Using a 4-inch wafer, thousands of individual structures could be created, allowing a range of beam spot sizes at a range of X-ray energies simply by translating the required structure into the X-ray beam upstream of the focusing mirror. Fabricated structures were tested on the Diamond Test Beamline and showed that the focused beam could be changed in size from 0.4 µm to 20 µm.

Adaptable wavefront correction with refractors

While correction of the wavefront from a focusing mirror by custom-designed refractive structures has been demonstrated [10], the correction in general requires an initial measurement of the wavefront error in order to specify the thickness profile of the refractive structure. A further complication is that the measured wavefront is taken downstream of the mirror but the corrective element is positioned upstream of that mirror where there is sufficient space available for this additional optical element and where the wavefront is closer to being planar. The measured wavefront error must therefore be projected across the highly curved mirror surface to the plane of the corrector. The assumption is that, following the initial measurement, the wavefront error stays constant in time so that a fixed profile corrector can be used. By measuring the wavefront after the final focusing optical element, rather than inferring the wavefront error from the mirror figure error, we can be sure that the correction will also include upstream contributions such as from window, mirrors, and monochromators.

In practice, it is difficult to ensure that the optical alignment does not change between the initial wavefront measurement and the installation of the fabricated refractor, and this leads to incomplete wavefront compensation. It is also expected that there will be a drift in the wavefront error caused by effects such as instability and heat loading on the optics. In particular, distortion of monochromator crystals caused by thermal effects can have a serious effect on the wavefront and compromise the achievable correction with a fixed correcting optic. This is apparent from Eq. (1), which shows that, for a reflecting optic, surface distortion is proportional to the sin of the incident angle. For X-ray mirrors, this angle is of order 0.003 rads, but for monochromators this is typically of order 100 times higher.
Heating effects are also likely to lead to time-dependent wavefront errors that cannot be fully corrected with a single static refractive correcting element. For these reasons, we have developed refractive optical elements that are adaptable.

The adaptability is achieved by using a pair of refractors so that the total path-length is given by their combined path-lengths. The design of the two refractors is shown in Figure 3. Each refractor has an identical sinusoidal thickness profile.

\[ t = t_0 + t_1 \sin\left(2\pi \left(y - y_0\right) / \Lambda\right) \]

When both refractors are aligned, \( y_0 = 0 \) and the path-length is given by a sinusoid with period \( \Lambda \), amplitude \( 2t_1 \) and phase of zero. When the offset \( y_0 \) is changed by the same amount for each corrector, the phase of the combined sinusoid is changed but the amplitude remains constant (at \( 2t_1 \)). When the offset \( y_0 \) is changed by the same amount but in opposite directions for each corrector, the phase of the correction stays constant, but the amplitude is changed as the sinusoids move out of phase with each other. This gives a device capable of providing a sinusoidal correction to the wavefront with variable amplitude and variable phase. In order to also allow the sinusoidal period (\( \Lambda \)) to be varied, a sequence of these structures are laid down along a line on the substrate with the sinusoid period incrementing in small steps from one structure to the next. This then gives an adaptable refractor that can provide a one-dimensional sinusoidal perturbation to the X-ray wavefront with variable phase, amplitude and period. Such a corrector was fabricated and tested on the Diamond test beamline with a selection of optical components with measurable wavefront errors. The optical elements were two elliptical mirrors—part of a Kirkpatrick-Baez mirror pair with focal lengths of 125 mm and 235 mm—and two planar CRL stacks. For each element, the wavefront was measured prior to the insertion of the adaptable refractive corrector. Using this measured wavefront error, the optimum correction period was estimated and the adaptable corrector was aligned to this period. Using this period of adaptable corrector, a small amplitude of wavefront modulation was selected (by offsetting both sinusoidal refractors a small distance in opposite directions) and then a series of wavefront measurements were performed with the correction phase being varied from \(-\pi / 2\) to \(\pi / 2\) (by translating both refractors together). By plotting the rms value of the measured wavefront, a minimum showed the optimum value of phase. At this phase, a series of wavefront measurements were performed as the amplitude of the correction was varied between 0 to \(2t_1\) (by varying the offset of each corrector in opposite directions). Again, the rms wavefront error was plotted and the optimum amplitude selected. By iterating the phase and amplitude, the optimum correction is achieved usually after two cycles.

For the two mirrors, the wavefront error was reduced by a factor of about 3 and for the lenses by a factor of about 7 to leave residual rms errors of between 0.6 and 4.5 picometers. For the horizontal mirror, the correction was applied at two different X-ray energies and it was found that the optimization could be achieved as expected simply by changing the correction amplitude. The measured wavefronts are shown in Figure 4. The focus intensity profile is obtained from the knife-edge measurement using the integrated detector intensity and is shown in Figure 5 where the corrected focus shows a reduction in fwhm.

![Figure 4: The wavefront errors measured using the knife-edge method for the horizontal KB mirror (HKB), the vertical KB mirror (VKB), and two different planar X-ray lens stacks (CRL1 and CRL2). Using the adaptable refractor, wavefront error was reduced by a factor of 7.0 for the lenses and a factor of 3.0 to 3.5 for the mirrors.](image-url)
Conclusions

With the building of new 4th generation synchrotron radiation facilities and the upgrade of existing facilities to 4th generation, many beamlines will have fully coherent beams of X-rays available. At DLS, we are presently investigating an upgrade of the ring lattice to 4th generation combined with an increase in the ring energy from 3 to 3.5 GeV. This will give the opportunity with high numerical aperture optics to achieve 10 nanometer scale focal spot sizes. This will require optics capable of preserving the X-ray wave-front but, at the same time, the X-ray power density will rise due to the increased beam energy and the reduced source emittance. A main concern is the effect of the power loading on double crystal monochromators. Power absorbed by the first crystal results in a distortion of the surface (heat bump), a change in the Bragg angle of the incoming X-rays, and a change in the crystal lattice parameter. The main effect seen is therefore a loss of beam intensity due to mismatch between the two crystals; however, there is also a large effect on the wave-front from distortion of crystal optics, as indicated by Eq. (1). We are presently planning wave-front measurements to get additional information on these effects. The use of adaptable wavefront correcting optics is also likely to play a large role in the future.

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Figure 5: Comparison of the focused beam profiles with correction (left) and without correction (right) for the HKB mirror. The full benefit of the wave-correction is not seen due to the large source size on the test beamline (~45µm fwhm); however, a reduction in the focused beam size by a factor of approximately 1.6 was measured.