CONFOCAL GRADED d-SPACING MULTILAYER BEAM CONDITIONING OPTICS

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ABSTRACT

A new family of graded d-spacing multilayer optics, called the Confocal Max-Flux® optics, has been designed, fabricated, and tested for several different applications. Utilizing a “side-by-side” Kirkpatrick-Baez scheme, both mirrors of a Confocal Max-Flux® optics can be positioned at the most optimized location. For intensity sensitive applications with limited sample size, the working surfaces are elliptical so more flux can be delivered to the sample. Protein diffraction experiments indicate an intensity increase of 2 to 5 times over total reflection optics. Spectrum purity is also improved. The Cu Kβ component is reduced by more than one order of magnitude comparing with a 15μm thick Ni filter. Small angle diffraction experiments have proven that the beam size in the detector plane is reduced by three times compared with a graphite monochromator. Meanwhile, the flux is increased by more than an order of magnitude.

INTRODUCTION

Graphite monochromator and total reflection mirror (TR) systems have been widely used as x-ray beam conditioning optics for diffractometers and many other kinds of instruments. These optics serve both as intensity enhancement tools and band pass filters. While graphite’s Bragg diffracted beam is monochromatic, the reflected beam is divergent and thus its enhancement capability is poor. As a result, graphite can not meet the requirements of many applications. In some cases, for example protein crystallography, total reflection mirrors with a Ni filter can deliver 2.5 to 5 times more flux than that provided by a graphite monochromator. However, total reflection mirrors pass the full spectrum below the mirror’s cut-off energy. Therefore, total reflection mirror systems must rely on additional filters to remove the remaining unwanted portion of the spectrum.
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Graded d-spacing multilayer collimating optics, introduced in several x-ray diffraction applications over the past few years [1], offer several advantages compared to the classical optical systems. Multilayers have larger reflection angles, resulting in higher collection efficiency. As multilayers also render a beam monochromatic, no additional optical components are necessary. Bandwidth can be customized by optimizing optical and multilayer parameters. A multilayer’s d-spacing is tailored in such a way that the Bragg condition is satisfied at every point. Graded d-spacing multilayer optical systems were first introduced in a cross coupled configuration known as Kirkpatrick-Baez (K-B) optical scheme. However, several limitations greatly affect the performance of this “sequential” K-B multilayer systems.

This paper reports a new family of graded multilayer optics, called the Confocal Max-Flux® Optics. The new optics give pure monochromatic radiation, small beam size at detector plane, and high intensities.

CONFOCAL Max-Flux® OPTICS

The application of high performance multilayers to hard x-rays has brought in significant progress in the development of x-ray optics. The reflection angle is, in general, two to three times larger than the critical angle of a total reflection mirror, resulting in larger capture angle and therefore higher throughput. As a band pass filter, the unwanted continuous and characteristic spectrum are automatically removed, while the filter used in a TR system can effectively remove the spectrum only above the filter’s absorption edge. Therefore, background of the beam conditioned by a multilayer optics is much cleaner. Compared with a single crystal, the band pass is two orders of magnitude wider. Converted into spatial characteristics, this wide band pass yields a wide acceptance angle for a multilayer, which allows multilayers to be used with a source size larger than sub-millimeters. Depending on the application, band pass can be designed by using different coating materials and different d-spacing values. Light materials and small d-spacing values allow more layers to contribute to the constructive interference, resulting in a narrower rocking curve. The reflectivity of a multilayer for higher order reflection can also be suppressed by changing the relative thickness of sub-layers. With suppressed high-order
reflection, the reflected beam is free of high energy contamination. For a curved optics, the d-spacing of a multilayer can be graded to match the curvature point to point, so that Bragg's law is satisfied. Figure 1 schematically shows the reflection from a uniform multilayer as well as graded multilayer collimating and focusing optics.

\[ n\lambda = 2d \times \sin\theta \]

Figure 1. Multilayer and graded d-spacing multilayer optics

The ideal surfaces for collimating and focusing a beam are a paraboloid of revolution and an ellipsoid, respectively. However, these surfaces are extremely difficult to fabricate, especially for hard x-rays. Hard x-ray reflections need very smooth surfaces, usually on the order of angstroms. This is especially true for the substrate of a multilayer coating, as the surface
roughness grows with increased number of layers. The usual way of forming the correct curvature, for example grinding, can not reach the required smoothness. On the other hand, micro-polishing, the procedure used to achieve the required smoothness, would damage the correct curvature obtained. A common approach is using two cylindrical mirrors to deflect the x-rays independently in two perpendicular directions, as shown in Figure 2. This is the optical scheme invented by Kirkpatrick and Baez in 1948 [2]. Compared to parabloid and ellipsoidal surfaces, cylindrical surfaces are much easier to make. The reflecting surfaces can be either parabolic cylinder for collimating optics, or elliptical cylinder for focusing optics.

![Figure 2. Kirkpatrick-Baez Mirror System](image)

A Kirkpatrick-Baez system with two elliptical mirrors can give perfect real point image for a point source at its first focal point. For a field object, the image will be magnified (or demagnified) by the system. Since the two mirrors are at different distances from the object, the magnification will be different in each direction. A Kirkpatrick-Baez system with two parabolic mirrors will give a perfect parallel beam if the source at its focal point is an infinitely small point. For a real source with finite size, the divergence of the reflected beam will be different in each plane. In addition, a Kirkpatrick-Baez scheme has the following characteristics which are unsatisfactory:

- There is no way to install both mirrors at the most optimized position, which results in less flux and larger aberration. Since the mirrors are located at different distances from the detector, for the same angular deviation, the aberration from the mirror closer to the source will be larger.
The alignment hardware is bulky and complicated. The alignment is difficult and time consuming since the alignment includes alignments relative to the source and the alignment relative to each other.

The optical scheme with two mirrors in a "side-by-side" fashion was proposed by Thathachari [3] in 1953. This scheme eliminates all the drawbacks associated with the "sequential" arrangement of two mirrors. In addition, since the optics is much more compact than that in the "sequential scheme", both mirrors can be pre-aligned and bonded, and alignment freedoms can be designed independent from each other. Therefore, alignment is much simpler and easier.

![Figure 3. “Side-by-Side” Kirkpatrick-Baez optical system](image)

Unlike the "sequential" arrangement, in which the working zone is generally in the middle, the working zone of a mirror in the "side-by-side" scheme is along the intersection of the two mirrors. The first and second reflection happens at either mirror. Figure 4 shows the working zones for "side-by-side" optics.

![Figure 4. Working zones for a “side-by-side” optics](image)

a. First reflection zone  
b. Second reflection zone

Although the “side-by-side” scheme was proposed more than 40 years ago, it had not been realized before. Part of the reason is forming the correct curvature for the reflection surface.
Almost all total reflection reflectors rely on bending to achieve the required curvature. It is very difficult, if not impossible, to integrate a bending mechanism into a “side-by-side” set-up.

Confocal Max-Flux® optical system consists of two multilayer mirrors in a "side-by-side" K-B scheme. The mirrors can be either elliptical cylinders or parabolic cylinders. For applications requiring line beam shapes, the optics can be made of one elliptical and one parabolic cylinder. Instead of placing two mirrors in vertical and horizontal directions, the mirrors are arranged at 45 degrees. With this arrangement, both the incident and reflected beams can be kept in the horizontal plane. The alignment assembly features 5 independent degrees of freedoms. The beam path is either helium filled or evacuated to eliminate air absorption and scattering. Both entrance and exit apertures are sealed with Beryllium windows. The mirror housing is connected to the incident beam pipe by flexible metal couplings, and shielded by shielding couplings. Figure 5 is a Confocal Max-Flux® optical system installed on a diffractometer for protein diffraction.

Figure 5. Confocal Max-Flux® optical system installed on a protein diffractometer
OPTICAL SYSTEM FOR PROTEIN DIFFRACTION

Based on different source sizes, different spectrum requirements, and different detecting ranges, various types of Confocal Max-Flux® optics have been developed for protein crystallography. Compared to a graphite monochromator system, the increase of flux ranges from several times to more than 20 times, depending on the resolution requirement. In comparison with total reflection setups, both flux on the sample and spectral purity are improved. For a particular design given in Table 2, which is used for 5kW rotating Cu anode with 0.3 mm focal size, measurement by a pin diode detector showed a five times flux improvement over a total reflection mirror setup given in Table 1.

| Distance between source and the first mirror | 135 mm |
| Length of the first mirror                  | 50 mm  |
| Distance between source and the second mirror | 280 mm |
| Length of the second mirror                 | 100 mm |
| Coating                                     | Nickel |
| Distance between source and sample          | 600 mm |
| Distance between source and detector        | >720 mm (depends on bending) |
| Pinhole size                                | 0.3 mm |
| Nickel filter thickness                     | 15 micrometers |

Table 1 Parameters of total reflection mirror setup

| Distance between source and optics (center) | 240 mm |
| Distance between source and sample         | 600 mm |
| Distance between source and detector       | 720 mm |
| Curvature error (RMS value)                | < 0.2 arc minutes |
| Length of optics                           | 60 mm |
| d-spacing                                  | 38.6-41.1 Å, 40 Å at center, |
| Multilayer coating (W/B₄C)                 | R ~ 78%, FWHM ~ 4 arc minutes |
| Pinhole size                               | 0.3 mm |

Table 2 Parameters of Confocal Max-Flux™ optics

Protein diffraction intensity was also measured. The same Lysozyme crystal was used for both optical systems at room temperature. The averaged intensity in a conical shell (same 2θ angle)
can be found from the following formula:

\[
\langle I_{\text{obs}}(u) \rangle = \frac{1}{C} e^{-2B \frac{\sin^2 \theta}{\lambda^2}} \sum_j f_j^2
\]

where \( \sum_j f_j^2 \) is the sum of square of atomic factors. The intensities were plotted in Figure 6, which is the Wilson's plot of the following formula:

\[
\ln\left( \frac{\langle I_{\text{obs}}(u) \rangle}{\sum_j f_j^2} \right) = -2B \frac{\sin^2 \theta}{\lambda^2} - \ln(C)
\]

![Graph of Wilson's plot of protein diffraction](image)

Figure 6. Wilson's plot of protein diffraction

A near five times flux improvement is calculated from the following the table:

<table>
<thead>
<tr>
<th>Omega Scans</th>
<th>C</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Scal, Normalized to 50kV/100mA</td>
<td></td>
</tr>
<tr>
<td>Total reflection setup</td>
<td>124.59</td>
<td>14.43</td>
</tr>
<tr>
<td>Multilayer optics</td>
<td>132.52, 26.50</td>
<td>16.67</td>
</tr>
</tbody>
</table>

Table 3. Lysozyme Data for High Resolution Bragg Maximum Reflections (3.0 Å-2.0 Å)
For flux comparison, the scale factor is normalized to the power setting of the total reflection mirror setup at 50kV/20mA. For Confocal Max-Flux\textsuperscript{®} optics, the power setting is decreased to 50kV/20mA to avoid detector saturation.

In addition to flux improvement, the optics reduces K\(\beta\) and Bremsstrahlung spectra. An Energy Dispersive X-ray Spectrometer (EDS) was used to compare the spectral purity. Figure 7 shows the measurement results with the K\(\alpha\) peaks normalized to show the relative background intensities, where CFM stands for the Confocal Max-Flux\textsuperscript{®} Optics and TR for the total reflection mirror with 4 \(\mu\)m thick Ni filter.

![Spectrum comparison](image)

Figure 7. Spectrum measurement by EDS

Due to the Cu K\(\alpha\) flux gain and the decrease of Cu K\(\beta\), I/\(\sigma\) at high resolution is improved by 20%-30% compared to the total reflection mirror setup, where \(\sigma\) is the standard deviation of the intensity. Also, R-factors, particularly Rsym and Rmerge values, are improved. R-factors are common crystallographic parameters indicating the correctness of a model structure. Rsys is the factor for comparing the intensity of symmetry-related reflections, and Rmerge is the factor for
comparing N data sets after merging. For a diffraction experiment, all these improvements mean that exposure time can be further reduced. Figure 8 shows two images taken with the Confocal Max-Flux® optics (left) and total reflection setup (right). Bremsstrahlung tails and Kβ spots can be easily seen from the image taken with the total reflection setup.

![Figure 8. Lysozyme w/Confocal Max-Flux™ optics (a)
Vs. w/Total Reflection Mirrors + Ni filter (b)](image)

**OPTICAL SYSTEM FOR SMALL ANGLE SCATTERING**

An optical system was developed and installed on a diffractometer for Small Angle X-ray Scattering at Osaka University, Japan. The previous optical system consisted of a graphite monochromator and a slit collimating system. The system layout with Confocal Max-Flux® optics is shown in Figure 9.

The distance is 490 mm between the source and the optical system, 450 mm between the optical system and the sample, and 565 mm between the sample and the detector. The d-spacing at the center is 25 angstroms. The optical system was made of two elliptical multilayer mirrors. X-ray beam is focused on the detector. The focused beam increases flux on the sample and flux density at the detector, while the decreased beam width at the detector improves the resolution.
Figure 9. System layout of the Small Angle Scattering System with a Confocal Max-Flux® optics

Measurement from an image (Figure 10) of the beam spot at the focal plane, which is registered by an image plate, shows 0.7 mm for the beam width. The beam width given by the original graphite monochromator is 2.1 mm. Therefore, the resolution is improved by three times.

Figure 10. Beam width at detector

Intensity measurements were taken with a polyethylene sample. Compared to the previous graphite-pinhole system, flux is increased by more than one order of magnitude. Figure 11 shows the measurement results.
SUMMARY

Performance evaluation of the Confocal Max-Flux® Optical System applied to protein crystallography shows great improvements in flux and resolution over a graphite monochromator system and a TR+Ni filter system. The spectral purity is also greatly improved over a TR+Ni filter system. As in protein crystallography, both flux and resolution are also greatly improved for an x-ray small angle scattering system.

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REFERENCE