FOCUSING POLYCAPILLARY OPTICS FOR DIFFRACTION

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ABSTRACT
In this paper, we describe two low power systems using polycapillary focusing optics designed to collect Cu Kα radiation from Oxford ultra-bright micro-focus sources to produce a convergent beam for X-ray powder diffraction and protein diffraction measurements. A collimator with two apertures was used to block high-energy X-rays. A still data set for chicken egg-white lysozyme was collected with the low power source focusing optic combination. This data exhibits high quality when processed with a special software program. Several powder samples were also measured and compared with measurements taken with an Enraf-Nonius FR590 sealed-tube source based system.

1. INTRODUCTION
Polycapillary optics are made of thousands of bundled bent hollow glass capillaries to focus and control X-rays or neutrons over broad capture angles (up to 30°) and energy ranges (200 eV - 80 keV) with high efficiency (10-50%). These tiny hollow glass tubes typically have diameters ranging between 5 and 50 micrometers\textsuperscript{1}. X-ray beams are deflected by total external reflection from the capillary inner surface at very small angles\textsuperscript{2}. Total external reflection for small angle scattering at the capillary walls occurs because the index of reflection \(n\) of glass is slightly less than the index of refraction from air. X-rays incident at angle less than a critical angle \(θ_c\) are totally reflected.

Polycapillary optics have been successfully applied for protein diffraction applications recently.\textsuperscript{3-7} This paper concerns the development of low power systems using a polycapillary focusing optic combined with a low power micro-focus source for convergent beam X-ray powder diffraction and protein diffraction measurements.

It is customary to use parallel or weakly focused X-ray beams for protein structure and powder diffraction measurements. However, the possibility of using a convergent X-ray beam for protein crystallography was first suggested by Wycoff and Agard in 1977\textsuperscript{8}. In this theoretical paper it was shown that the use of a one-dimensional convergent beam would not seriously impede structure determinations. More recently, it has been shown that convergent beams in two dimensions can also be used\textsuperscript{9-11}. In such measurements the sample is fixed (not oscillated) during each exposure. Use of a convergent X-ray beam for diffraction, results in tangentially elongated diffraction spots, the basic theory for which is well understood\textsuperscript{10-11}. The diffraction patterns from such measurements cannot be analyzed with standard software packages. Therefore, a measurement and analysis procedure and a special software package, CBMPRO has been developed specifically for processing data collected with the Converging Beam Method (CBM)\textsuperscript{11}.

A double-aperture collimator was used to block high-energy X-ray penetration and to define the beam intensity and convergence. A still data set for chicken egg-white lysozyme was collected with a low power source-focusing optic combination. These data give high quality results when processed with the CBMPRO program. Several polymer powder samples were also measured with this system and their qualities are compared with measurements of the same samples taken with an Enraf-Nonius FR590 sealed-tube source system. This investigation shows that the application of polycapillary focusing optics in powder diffraction and protein diffraction has a promising future.
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2. SYSTEM SETUP

Figure 1 shows photos for the powder diffraction system and protein diffraction system.

![Figure 1](image1)

Figure 1. The systems using polycapillary optics for powder diffraction (left) and protein diffraction (right).

The X-ray sources used in the two systems were Oxford ultra-bright micro-focus Cu sources. Following is the data sheet of the powder diffraction system source from Oxford Company:

<table>
<thead>
<tr>
<th>Anode Voltage (max):</th>
<th>60 kV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anode Current (max):</td>
<td>2 mA</td>
</tr>
<tr>
<td>Measured Spot size at 20 kV/20 W:</td>
<td>18 µm</td>
</tr>
<tr>
<td>Measured Spot size at 60 kV/20 W:</td>
<td>15 µm</td>
</tr>
</tbody>
</table>

A double-aperture collimator was used to block high-energy X-rays, which pass directly through the optic and produce undesirable background. Figure 2 shows the combination of source, optic and collimator.

![Figure 2](image2)

Figure 2. Source, optic and collimator combination.

Table 1 shows the parameters and transmissions for the two systems. Focusing optic 1106 was used for powder diffraction. Focusing optics 1265 and 1291 were used for protein diffraction.

<table>
<thead>
<tr>
<th>Focusing Optic #</th>
<th>F-input (mm)</th>
<th>F-output (mm)</th>
<th>D-input (mm)</th>
<th>D-output (mm)</th>
<th>Capture Angle</th>
<th>Length (mm)</th>
<th>Transmission. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1106</td>
<td>6.9</td>
<td>98</td>
<td>1.56</td>
<td>~4.0</td>
<td>13°</td>
<td>32.6</td>
<td>19.5</td>
</tr>
<tr>
<td>1265</td>
<td>10.0</td>
<td>90</td>
<td>1.88</td>
<td>5.30</td>
<td>11°</td>
<td>48.8</td>
<td>20.0</td>
</tr>
<tr>
<td>1291</td>
<td>7.0</td>
<td>88</td>
<td>1.70</td>
<td>4.37</td>
<td>14°</td>
<td>48.8</td>
<td>18.8</td>
</tr>
</tbody>
</table>

Table 1. Optics parameters and transmissions.
3. EXPERIMENTAL RESULTS AND ANALYSIS

3.1. Protein diffraction
Still diffraction patterns of lysozyme were taken with the source-focusing optic combination. Figure 3 shows one of these images with an enlargement of part of it. These images show that the diffraction spot shapes are tangentially elongated due to the strongly convergent beam. However they can still be analyzed separately by using the special software package.

![Figure 3. Still diffraction image taken with source-strongly focusing optic combination.](image1)

The source-focusing optic combination system produced a high intensity beam in a small spot. For optic 1291, a whole data set of chicken egg white lyzosyme crystal with 42 still frames were taken, each frame was exposed 30 minutes. The overall R-factor was 9.5%. The beam divergence was about 15 mrad, and the crystal size was less than 0.2 mm.

3.2. Powder diffraction
Several powder diffraction sets were taken with this low power powder diffraction system for organic powder samples provided by National Institutes of Health. A schematic of the setup for such measurements is shown on Figure 4 and results are shown in Figure 5 through Figure 8.

![Figure 4. Setup for powder diffraction measurements.](image2)
These diffraction patterns have been integrated by a FIT2D program that was developed at the European Synchrotron Research Facility\textsuperscript{12}.
Table 2. Diffraction pattern analysis results by Gaussian fitting the diffraction peaks for 2 theta 5.5o to 10.5o of the Nonius system and the focusing optic system.

<table>
<thead>
<tr>
<th>System</th>
<th>Collection Time (min)</th>
<th>$Y_0$</th>
<th>$X_c$</th>
<th>W</th>
<th>A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nonius system</td>
<td>30</td>
<td>16.7</td>
<td>8.4</td>
<td>2.5</td>
<td>66</td>
</tr>
<tr>
<td>Optic 1106 system</td>
<td>5</td>
<td>21.3</td>
<td>8.4</td>
<td>3.1</td>
<td>169</td>
</tr>
<tr>
<td>Optic 1106 system</td>
<td>7</td>
<td>36.3</td>
<td>8.4</td>
<td>3.3</td>
<td>260</td>
</tr>
</tbody>
</table>

In Table 2, $Y_0$ is the Gaussian fitting base height to indicate the background level. $X_c$ was used to determine the width of the diffraction ring 2 theta which was used to determine the unit cell space. The reason that the $X_c$ values were not exactly the same was because the distance between the image plate and the sample and the direct beam position were not precisely measured. $W$ is the Gaussian fitting width which for the optic 1106 system was a little larger than that of Nonius system. This is because, compared to the Nonius system, the polycapillary optic beam had more high energy X-rays as well as some white radiation. $A$ is the integration of the diffracted peaks which was used to compare the beam raw intensity. Compared with the Nonius system, 1106 system provided **15 times higher intensity**, at 4.5% of the x-ray source power.

Measurements were also made with a polycrystalline polymer powder sample. The diffraction pattern for this measurement is shown in Figure 9 and the radial integration is shown in Figure 10.

Although this pattern has not been analyzed, it is clear that fine features in the pattern are clearly resolved.

Figure 9. Diffraction pattern of polycrystalline polymer powder sample with focusing optic and 40 W Ultrabrite source.

Figure 10. Radial integration of polymer powder diffraction pattern.
4. CONCLUSIONS
Strongly focusing polycapillary optics can be used for protein diffraction. This will have particular application for screening of small crystals for purity, quality and to obtain preliminary structural information, for example, during development of the crystal growth process. The high X-ray intensity and small spot size could reduce the data collection time as well as the crystal size requirement. These results also provide the basis for considering convergent beam diffraction for X-ray microdiffraction studies of strain and texture distributions with low power sources. In addition they suggest that convergent beam neutron diffraction may be viable for diffraction of proteins and other large molecules and for X-ray and neutron diffraction of small samples at low temperature or high pressure. This kind of optic can also be used for solving simple structure powder samples with lattice space less than ~100 Å (this is constrained by the system beam divergence.

5. REFERENCES

6. ACKNOWLEDGEMENTS
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