APPLICATION OF POLYCAPILLARY OPTICS FOR PARALLEL BEAM POWDER DIFFRACTION

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ABSTRACT

The use of polycapillary optics for powder diffraction has been evaluated based on intensity, profile shape, and accuracy of lattice parameters for materials with a wide range of absorption coefficients. Measurements of powder samples were made using Bragg-Brentano, traditional parallel beam, and polycapillary/parallel beam instrument configurations. In the case of the polycapillary optic, several configurations of the receiving side optics were tested. The results demonstrate that the intensities obtained using the polycapillary optic range from 0.4 to ~7 times that obtained in Bragg-Brentano geometry. In addition, the polycapillary optic provides profile shapes that are essentially gaussian and the small divergence of the beam allows accurate measurements of lattice parameters without calibrating the diffraction peak locations.

INTRODUCTION

Polycapillary x-ray optics, which use hollow capillaries as waveguides for x-rays, have been used to control x-ray beams for various applications. The principle behind the operation of these optics is the multiple total external reflection of x-rays from the smooth inner walls of capillary channels. When an x-ray photon strikes the reflecting surface of a capillary at a grazing angle smaller than the critical angle of the material, it undergoes total external reflection. X-rays satisfying the total reflection condition can be effectively transported through the capillary channels. Appropriate design of a single or polycapillary device can produce a focusing or parallel x-ray beam. For example, the optic used in the present work was a standard x-ray collimating lens, model X-8-L10 from XOS, Inc. This lens is designed to collect x-rays from a standard line source in point focus orientation over a cone angle of 4.1° and collimate the x-rays into a quasi-parallel beam with FWHM divergence of 0.25° for Cu Kα radiation. The beam size is 10 by 10 mm, and the optic exhibits a transmission efficiency of 30% at Cu Kα when measured with a 250-mm diameter point source. Beam collimation is in two dimensions, therefore eliminating the need for axial collimators.

Although the use of polycapillary optics has been investigated for powder diffraction, a direct experimental comparison of polycapillaries with traditional instrumental configurations can only improve our understanding of their applicability. One expects that the use of the polycapillary optic, which provides a beam with only ~0.25° divergence, will eliminate most of the sample-related systematic errors found in parafocusing instruments. In addition, a parallel beam will allow studies of irregularly shaped samples.
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EXPERIMENTAL PROCEDURES

A Siemens D-500 diffractometer fitted with a Cu fine focus tube and scintillation counter was used in all of the measurements. The diffractometer was aligned to provide a 6° tube takeoff angle for each instrumental configuration. Three diffraction geometries were employed to evaluate the use of the polycapillary optic, including:

- **Bragg-Brentano (B-B)** geometry with 12 mm line focus, 1° divergence slits, 0.05° receiving slit and 200 mm radius. The diffracted beam side was equipped with standard 3° axial divergence slit, a diffracted-beam graphite monochromator, and 13 mm diameter detector aperture. All measurements were made as coupled Θ-2Θ scans.

- **Parallel-Beam (P-B)** geometry using 2Θ scans with Θ fixed at 2.0° with the goniometer radius set to 200 mm and line focus. The P-B arrangement included divergence slits set to 0.14° to illuminate 15 mm of the specimen at Θ = 2°. An incident beam 3° axial divergence collimator was used in this configuration. A thin film attachment (long Soller collimator) with divergence of 0.15° was installed on the diffracted beam side of the diffractometer, with a detector aperture 13 mm in diameter. A 500:1 Ni beta filter was installed on the diffracted beam side of the diffractometer.

- **Polycapillary Optic** with several different configurations on the diffracted beam side, with the tube in point focus. The goniometer radius was not symmetrical, but rather approximately 300 mm on the incident beam side and 200 mm on the receiving side. No axial divergence collimator was used because of the inherent axial collimation of the optic, and all measurements were run as coupled Θ-2Θ scans. The receiving optics included:
  - Only a thin film attachment (long Soller collimator) with divergence of 0.15°,
  - a 0.15° thin film attachment with a 500:1 Ni beta filter,
  - only a LiF diffracted beam monochromator, and
  - a 0.15° thin film attachment with a LiF diffracted beam monochromator.

All experiments were run as step scans, using 0.02° step size and 2 second count times with the x-ray tube running at 1200 W. Each powder specimen was mounted in a 15 x 15 mm side-drifted sample holder after mixing with an appropriate quantity of NBS SRM 640b silicon to attain equal intensity strong peaks in the patterns. The powder samples run included urea powder, silver powder, and NIST LaB$_6$. In addition, a polycrystalline quartz plate with dimensions of 50 x 50 mm was measured. The samples were chosen to span a wide range of absorption coefficients for evaluating sample transparency effects on the profiles.

Data analysis was completed using two programs, Jade+ and NBS*LSQ. Jade+ was used to determine the backgrounds and background-subtracted peak heights and peak positions for each pattern. Peak heights and positions were determined as the peak summit, after applying a 7-point modified Savitsky-Golay filter. NBS*LSQ was used to refine the lattice parameters for each phase of interest, and provided lattice parameters with estimated standard deviations, as well as Smith-Snyder figures of merit.
RESULTS AND DISCUSSION

To take advantage of the photons available from the polycapillary optic, the specimen size and diffraction experiment need to be considered relative to the large beam size. For example, a powder diffraction measurement can be performed using the full beam from the optic at angles above ~24° 2Θ if the sample is ~50 mm. Texture and residual stress, however, are typically run at higher angles and the use of a significantly smaller sample, for example 12 x 12 mm, is practical.

To evaluate the diffracted intensity from a sample large enough to intercept the entire incident beam, the large quartz plate sample was used. Figure 1 presents a comparison of data obtained in the different instrumental configurations. Table 1 presents a numerical description of the data shown in Fig. 1 as well as the relative count time required to detect a peak at two standard deviations above background. When using the polycapillary optic, different receiving side optics yield intensities ranging from 0.4 to ~7 times that obtained in B-B geometry (note, however, that a 1° divergence slit was used in B-B, and therefore a significantly smaller sample area was illuminated than in the case of the polycapillary optic). In addition, the count time required to
Table 1: Comparisons of intensity and count time required to detect a peak at two standard deviations above background. All values are relative to the Bragg-Brentano configuration.

<table>
<thead>
<tr>
<th>Configuration</th>
<th>Peak Intensity relative to B-B</th>
<th>Relative count time to detect peak</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bragg-Brentano</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Traditional P-B</td>
<td>0.96</td>
<td>7.0</td>
</tr>
<tr>
<td>LiF Crystal</td>
<td>1.4</td>
<td>3.3</td>
</tr>
<tr>
<td>Soller + LiF Crystal</td>
<td>0.4</td>
<td>2.3</td>
</tr>
<tr>
<td>Soller + Ni filter</td>
<td>3.1</td>
<td>1.3</td>
</tr>
<tr>
<td>Soller only</td>
<td>7.3</td>
<td>0.95</td>
</tr>
</tbody>
</table>

Figure 2: Powder patterns for urea powder using three instrumental configurations. The patterns are scaled and offset for clarity. Note the symmetric peak shape in the polycapillary data.

detect a diffraction peak when using the polycapillary optic ranges from 0.95 to 3.3 times that for B-B geometry.

Figure 2 includes data for urea powder, which is a low-absorption material with $\mu = 10$ cm\(^{-1}\). Note that the B-B data show the typical sample transparency error that produces a highly asymmetric diffraction profile. In contrast, the P-B measurement produces significantly more symmetrical lines, while the polycapillary optic produces a symmetrical line. As shown in Fig. 3, the symmetrical profile shape and small divergence of the polycapillary measurement allows one
to accurately determine the lattice parameters of urea without using an internal standard to correct for specimen related errors. Each lattice parameter refinement shown in Fig. 3 included data from 19 to 90°2Θ, including 29 diffraction peaks. The refined lattice parameters for all the samples measured using the polycapillary optic were within 3 estimated standard deviations of the ‘ideal’ values obtained from calibrated B-B results.

Although the profile shapes obtained using the polycapillary optic have not yet been fully quantified, profile fitting using a pseudo-Voigt function tends to pure gaussian profile shapes for all of the samples investigated. The peak full width at half maximum is approximately 0.25°, and the peak width remains in the range of 0.25 to 0.30° over the 2Θ range of 20 to 100°2Θ.

SUMMARY

The use of polycapillary optics for powder diffraction has been evaluated based on intensity, profile shape, and accuracy of lattice parameters for materials with a wide range of absorption coefficients. Polycapillary optics may be successfully used for powder diffraction, where the primary advantage of the optic is beam collimation in two directions, which significantly reduces systematic peak shape and peak location errors, especially for low absorption materials. The intensities obtained using an instrument equipped with a polycapillary optic is determined by the
receiving side optics, and can be varied over a wide range from 0.4 to ~7 times those obtained using a Bragg-Brentano instrument with 1° divergence slits.

REFERENCES


