INVESTIGATION OF THE EFFECT OF USING VARIOUS OPTICS CONFIGURATIONS WITH PROPORTIONAL AND POSITION-SENSITIVE DETECTORS

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ABSTRACT

XRD patterns of NIST standard reference materials were collected using a gas proportional detector and a linear Position Sensitive Detector (PSD). Incident beam optics used for data collection consisted of a polycapillary X-ray lens, a graded multilayer parabolic X-ray mirror, and programmable divergence slits. Peak position, width, and intensity were studied with respect to the different experimental configurations. Bragg-Brentano parafocusing optics resulted in the best resolution and peak position accuracy, though low intensity. The mirror with the PSD resulted in higher intensity, without losses in resolution and accuracy as significant as those seen with the polycapillary lens and the PSD. In addition, when combining incident parallel optics with the PSD, sample surface displacement resulted in considerable peak shift.

INTRODUCTION

Because of the ability to measure a range of 2θ angles simultaneously, position sensitive detectors (PSDs) have many uses in time-dependent studies, for example to determine reaction kinetics. In addition, PSDs allow increased scan rates in standard, time-independent X-ray diffraction. However, PSDs are designed for use with parafocusing optics [1]. Recent developments in incident beam optics that produce nearly parallel X-ray beams change the parafocusing geometry. Therefore, using incident parallel optics with a PSD could result in data where the intensity, resolution, and/or peak position are compromised.

EXPERIMENTAL

X-ray diffraction measurements were made on a PANalytical X’Pert Pro using a 0/θ goniometer and CuKα radiation. Ray diagrams for the different focusing optics employed are shown in Figure 1. The incident and diffracted beam configurations are shown in Table I. The Prefix capabilities of the diffractometer allowed the optics to be quickly reconfigured, without alignment between changes. A 0.04 radian Soller slit assembly was mounted on the parallel-plate collimators. A curved-crystal graphite monochromator was used with the parafocusing optics. The peak full-width half-maxima (FWHM) and lattice parameters were determined using profile fitting [2] with a Pearson VII profile. Lattice parameters were also obtained from Rietveld refinements [3, 4]. Data were collected on NIST SRMs LaB₆ (SRM 660a) and Si (SRM 640c) contained in deep-well sample holders and an Al₂O₃ sintered plate (SRM 1976). Additional X-ray diffraction data were collected for SRM 640c, using an adjustable sample stage to vary the sample height.
Figure 1. Variations of the Bragg-Brentano goniometer circle with different focusing optics.
Table I. Incident and Diffracted beam configurations.

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<thead>
<tr>
<th>Incident beam configuration</th>
<th>Diffracted beam configuration</th>
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<tr>
<td><strong>Optics</strong></td>
<td><strong>Accessories</strong></td>
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<tr>
<td>Polycapillary Optics</td>
<td>Mask 8 x 2mm</td>
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<td></td>
<td>Mask 8 x 2mm</td>
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<tr>
<td>X-ray Mirror</td>
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$^1$Programmable Divergence Slits  
$^2$Programmable Receiving Slits

RESULTS AND DISCUSSION

Figure 2 shows the (110) peak of the LaB$_6$ sample for the various configurations. The polycapillary lens and the mirror resulted in significantly more intensity than the parafocusing optics. The polycapillary lens resulted in more intensity than the mirror, due to its larger acceptance angle (3.5° versus 0.8° for the mirror). Combining incident divergent optics and receiving parallel optics resulted in non-standard peak shapes, which could not be well fitted with a profile function.

The peak widths are quantified in Figure 3, which shows the average FWHM for each configuration and sample. Among the configurations with the proportional detector, the best resolution resulted from the parafocusing optics and from the mirror with the 0.09° collimator. The mirror resulted in better resolution than the polycapillary lens (approximately 60% for the 0.09° collimator), since it gives 0.04° beam divergence as compared to 0.3° for the polycapillary lens. As expected, the 0.27° collimator resulted in worse resolution than the 0.09° collimator for both the polycapillary lens and the mirror (approximately 40% and 100%, respectively).

With the divergence slit and PSD, high resolution was achieved, comparable to that with the proportional detector. The polycapillary lens with the PSD resulted in large scatter and poor resolution, comparable to that with the 0.27° collimator and the proportional detector. Notably, combining the mirror with the PSD resulted in low scatter and a resolution comparable to that from combining the polycapillary lens and the 0.09° collimator, or the mirror and the 0.27° collimator, with the proportional counter.
Figure 2. X-ray diffraction pattern of the (110) peak of LaB6 for each configuration.

Figure 3. Average FWHM as a function of configuration for each sample.
The refined lattice parameters were compared with the NIST standard values as shown in Figure 4, which plots the relative error in the refined lattice parameters for each configuration. The relative error is defined as the ratio of the difference between the refined value and the NIST value to the NIST value. The error bars were obtained from the estimated standard deviations given by GSAS. Among the configurations with the proportional detector, parafocusing optics resulted in both the best precision and accuracy. The polycapillary lens resulted in less scatter in the lattice parameters than the mirror. For each incident optic, combination with the PSD resulted in larger scatter than with the proportional detector. The divergence slit gave accurate results with the least scatter. With the mirror, the results were still accurate, but more scattered. When combining the polycapillary lens with the PSD, the precision was worst, and the refined lattice parameters were consistently larger than the NIST values.

When data were collected with the mirror and PSD while varying the height of the silicon sample, a peak shift was evident. When the same data were collected with the 0.09° collimator and proportional detector, no peak shift was evident. Figure 5 demonstrates the effect of sample height on the displacement refined with JADE for both configurations. When using the PSD, the relationship is linear. Therefore, the use of the PSD negated one of the primary advantages of incident parallel optics, insensitivity to sample height.
Figure 5. Refined displacement versus sample height for the mirror combined with the PSD and with the 0.09° collimator and proportional detector.

CONCLUSIONS

Parafocusing optics resulted in the best resolution and peak position accuracy, though low intensity. Combining the polycapillary lens with the PSD resulted in greater intensity, but considerable losses in resolution and accuracy. The mirror with the PSD resulted in higher intensity, without losses in resolution and accuracy as significant as those seen with the polycapillary lens and the PSD. In addition, when combining incident parallel optics with the PSD, sample surface displacement resulted in considerable peak shift.

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