MANAGING BACKGROUND PROFILES USING A NEW X’CELERATOR DETECTOR

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ABSTRACT

The new Philips X’Celerator detector provides for rapid acquisition of x-ray diffraction patterns. The X’Celerator is a position sensitive detector requiring radial divergence control to manage a smooth background profile at low angles. This paper discusses the instrument configuration necessary to obtain a satisfactory background profile useful for Rietveld modeling as well as other analytical applications.

INTRODUCTION

The new Philips X’Celerator [1] detector provides for rapid acquisition of x-ray diffraction patterns. The X’Celerator detector is a proprietary solid-state position sensitive detector that operates at room temperature. The detector is not a high-resolution energy dispersive detector, but is very sensitive to x-ray exposure with excellent linearity at high count-rates. Since the X’Celerator is a position sensitive detector with enhanced sensitivity, optical focus on the sample is necessary to minimize spurious scatter contributing to the background profile. A good background profile is required for samples that include amorphous components and for experiments that utilize Rietveld modeling.

EXPERIMENTAL

Instrumentation: All x-ray diffraction measurements were taken using a Philips X’Pert Pro [1] diffractometer equipped with a long fine focus copper x-ray source powered at 40 kV & 40 mA; X’Celerator detector and beam tunnel; 0.04 Radian Source and Receiving Soller Collimators; 20 mm Source Mask; ½ Degree Primary Slit; and ½ Degree Anti-Scatter Slit.


Specimen: A 32 mm silicon metal low background holder [3] was used as a specimen to measure all background profiles.

RESULTS AND DISCUSSION

The general optical arrangement for a Bragg-Brentano parafoosing x-ray Diffractometer [4] is shown in Figure 1. In this configuration, the source and detector collimators control the axial-divergence[5] of the x-ray beam, Figure 2. Radial divergence is not collimated using a Soller slit system, but by employing collimator masks along with primary, receiving, and anti-scatter slits.
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Conventional detectors, i.e., proportional counters, scintillation counters and Peltier devices, work well under the type of axial and radial divergence control shown in Figure 1.

Figure 1. General optical arrangement of a Bragg-Brentano parafocusing x-ray diffractometer.

The standard configuration for a diffractometer equipped with an X‘Celerator detector is shown in Figure 3. The X‘Celerator position sensitive detector installed in this configuration exhibits a background profile that contains features at low angles. The non-ideal condition of the background profile at low angles makes some phase identification and modeling techniques difficult to employ.

Figure 2. Soller collimator functions to collimate the x-ray beam in the axial direction of the x-ray diffractometer. The x-ray beam remains divergent in the radial direction.
Figure 3. General optical arrangement of a Bragg-Brentano parafocusing x-ray diffractometer equipped with an X’Celerator detector.

Figure 4 shows an x-ray diffraction trace from a silicon wafer specially cut to produce a nice smooth background profile. The low angle region below 20 degrees 2Θ shows features that are related to optical artifacts introduced from various sources [5]. Scattered light from the specimen, air above the specimen, sample holder, beam tunnel, etc. are some of the more likely sources.

Figure 4. Silicon metal low background holder showing artifacts below 20 degrees 2Θ.
The X'Celerator detector is very sensitive. Minor optical artifacts in the background measured with more conventional diffractometer configurations, e.g., monochromator / proportional counter or Peltier detector, may stand out significantly. Figure 5 shows a new optical configuration with a radial divergence Soller slit in place at the end of the beam tunnel located in front of the position sensitive detector. The location of the radial divergence Soller collimator away from the position sensitive detector allows the collimated x-ray beam to diverge and overlap as a smooth continuous beam across the channels that make up the position sensitive detector. This prevents a saw tooth effect to the background profile as shown in Figure 6.

Figure 5. General optical arrangement of a Bragg-Brentano parafocusing x-ray diffractometer.

Figure 6. Saw tooth effect to the background profile.
Figure 7 shows a comparison of the background profile with and without the radial divergence Soller collimator. Placing the radial divergence Soller collimator in front of the specimen away from the detector improves the background in the low angle region of the diffraction pattern. Figure 8 compares an x-ray diffraction pattern of LaB$_6$ taken with and without the radial divergence Soller collimator to show the improvement in background profile along with the impact to the intensity of the pattern. Radial divergence control of the x-ray beam improves the background profile by focusing the channels in the detector onto the sample surface eliminating spurious scatter.

![Figure 7](image1.png)

**Figure 7.** Background profile with a radial divergence slit (bottom trace) and without a radial divergence slit (top trace).

![Figure 8](image2.png)

**Figure 8.** LaB$_6$ patterns with a radial divergence slit (bottom trace) and without a radial Divergence slit (top trace).
SUMMARY

The new generation position sensitive detectors can enhance the effect of artifacts in the background profile at low angles. This effect can be improved by incorporating a radial divergence Soller collimator in front of the sample and away from the position sensitive detector. This configuration allows for the collimated x-ray beam to diverge and overlap as a smooth continuous beam across the channels that make up the position sensitive detector. The result is a well-managed background profile at low angles suitable for modeling applications and without jeopardizing detector performance.

FUTURE

A capillary optical device [6,7] or a multiple tube collimator [8] should be tested to replace the Soller collimator in this optical configuration because these devices would provide both axial and radial divergence control. This would eliminate the need for either an axial or radial receiving Soller collimator.

REFERENCES