Scanning CCD Detector for X-ray Powder Diffraction

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Abstract We discuss the design, fabrication and use of a custom CCD detector for x-ray powder diffraction measurements. The detector is mounted on a diffractometer arm, where line-by-line readout of the CCD is coupled to continuous motion of the arm. As the arm moves, the data from the CCD detector are accumulated and can be viewed as if it were a “film strip” with partial powder diffraction rings. Because of the unique design of the camera, both high-resolution and rapid measurements can be performed. Powder diffraction patterns are collected with speeds of a few minutes, or less, with many of the advantages of large area position-sensitive detectors, for example amorphous silicon flat panels, such as high sensitivity, direct evidence of grainy samples and freedom from low-angle asymmetry, but with resolution better than linear position-sensitive detectors and nearly as good as the ultimate in resolution, analyser-crystal detection [2,3].

1. Introduction
Powder diffraction is a simple form of X-ray diffraction performed on a collection of microcrystals, often in the form of a powder. Because it is advantageous if the crystals have random orientation and to have many crystals in the beam, a sample is often spun on a motor stage as it is exposed to x-rays to better randomize the crystal orientations and to improve particle sampling statistics. Because ideally all crystal orientations are represented, the diffracted x-rays from all Bragg peaks manifest as cones that project as concentric circles. Powder diffraction can be used to determine the structure of even complex materials, and is a vital crystallographic technique [1].

There are two types of powder diffraction instruments at Advanced Photon Source. At beamline 11-BM, a crystal analyser system, in which 12 photomultiplier tubes (PMT), each with an analyser crystal, are mounted on a goniometer arm; it offers the highest resolution powder diffraction data in the United States. The analyser-PMTs are moved on the goniometer arm to scan the projected powder diffraction rings [2]. Scans take typically one hour. For faster measurements at APS, several sectors use a stationary area detector such as a General Electric or Perkin-Elmer amorphous silicon flat panel detector, or less commonly a MAR image plate. In this setup, a single image is taken with a 0.1s to 30s exposure. This fast measurement is not nearly as high resolution as the crystal analyser system, but the fast measurement time allows for many otherwise impossible experiments [3]. It would be advantageous to build a detector that could take measurements with resolution comparable to the analyser system but speed closer to the area detector; such a detector, the Scanning CCD, is presented here.
2. Scanning CCD Detector

2.1. Synchronizing CCD Readout with Detector Movement

To understand how the Scanning CCD works, recall that an image is projected onto the active area of the CCD and incident photons are integrated as electronic charge in the CCD pixels. Once sufficient signal has been integrated over time, the image is read out by shifting it line by line. Each line is subsequently read out pixel-by-pixel to an output amplifier and digitized for storage in a computer. Generally the CCD is stationary and captures a stationary projection before being read out. In the Scanning CCD camera, the CCD is moved on a goniometer arm as it is read out. The shifting of the image line-by-line is exactly synchronized with the movement of the CCD. In this way, the image projected on to the CCD surface moves exactly with the image being read out. Furthermore, the CCD integrates the image while it is being readout. In a powder diffraction experiment, the CCD camera is moved on a goniometer arm through projected powder diffraction rings, and the camera returns a filmstrip, similar to that produced by a Debye-Sherrer Camera [4]. A diagram of a CCD moving up through a series of powder diffraction rings and returning a film strip is shown in Figure 1.

![Figure 1. CCD moving though series of rings, generating a film strip. Readout of CCD is synchronized with camera movement.](image)

2.2. Observations on Scanning CCD

One can observe several design considerations when building a Scanning CCD camera. The CCD must have very small pixels to preserve instrument resolution. Indeed, because the CCD is structured as discrete lines, CCD line shifting will slightly blur the image even if the device is smoothly moved on the goniometer arm. Obviously, a very accurate and repeatable motor system is needed. Because the motor system will move in steps unequal in size from the CCD line shifts, an algorithm must be designed to reconcile this difference. The slower the CCD moves, or the larger the CCD area, the larger the acquired signal. Because the CCD is moving, sensitivity variations in phosphor or optics in front of the CCD will be integrated over columns. To assure that image features are integrated over columns and not smeared across columns, the camera optics must have little or no spatial distortion and be accurately oriented with the direction of motion. Most commonly, data will be radially integrated once the filmstrip is recorded. The combination of radial and columnar integration means that every CCD pixel contributes to every point in the resulting 1-D powder diffraction pattern, rendering pixel-by-pixel sensitivity calibration unnecessary. Spatial distortion in the optics widens the peaks in the 1-D powder diffraction pattern.
2.3. Synchronization Algorithm
The synchronization algorithm accepts pulses from the motor system at the beam line. There will be \( M \) motor pulses corresponding to the spacing between each CCD line; the design requires that \( M \) be greater than or equal to 1, but allows this to be a real number. A digital accumulator integrates the number \( 1/M \) as each motor pulse is noted. Whenever the accumulator reaches 1.0, the integer portion of the accumulator is dropped, and a single CCD line is shifted out of the imager. In this way, the CCD readout is synchronized with the goniometer motor system, but a strict integer relationship between the angular motion and the CCD spacing is not required. A diagram of the algorithm is shown in Fig. 2.

![Diagram of Synchronization Algorithm](image)

Figure 2. Algorithm for CCD readout synchronization with motor system. Motor pulses cause integration of \( 1/M \) in the accumulator; overflow causes a CCD line to be read out.

2.4. The APS Scan CCD Detector
The APS detector group designed and built a prototype Scanning CCD detector utilizing a Kodak KAF-16801 CCD with pixel dimensions of \( 9\mu m \times 9\mu m \) coupled to a Scint-X structured scintillator via a fiber-optic faceplate. The electronics features all-digital double correlated sampling of the CCD signal and runs the motor synchronization algorithm on an on-board FPGA. Data is transferred from the camera to computer via Camera Link interface. The camera spatial resolution was measured to be \( 60\mu m \) FWHM. A photograph of the detector is shown in Figure 3.

![Photo of APS Scanning CCD Detector](image)

Figure 3. APS Scanning CCD Detector

3. Performance of the APS Scanning CCD Detector
A NIST standard (SRM 660a LaB\(_6\)) was measured at APS Sector 11-BM with four different detectors: the 11-BM crystal analyzer system, a Perkin Elmer Area Detector, a Mythen linear detector, and Scanning CCD [5]. To compare detectors, a measurement of resolution as \( \Delta Q/Q \) was taken. The quantity \( Q \) is related to the diffraction angle, \( 2\theta \), and the crystal lattice spacing, \( d \), by \( Q=2\pi/d=(4\pi\sin\theta)/\lambda \). The smaller the \( \Delta Q/Q \), the better the resolution of the powder diffraction
measurement. Figure 4 shows plots of $\Delta Q/Q$ for the four detectors. As can be seen, the scanning CCD outperforms the Mythen and Perkin Elmer in resolution and is close to the 11-BM crystal analyzer system. These differences are not small, as can be seen in the comparison of widths for a single peak, shown in the same figure.

![Graph showing $\Delta Q/Q$ for four detectors](image)

Figure 4. On left, comparison of $\Delta Q/Q$ for four detectors. On right, a single peak from all four detectors.

4. Conclusion and Acknowledgements

The APS Scanning CCD Detector provides resolution nearly of the quality of the best in U.S. 11-BM crystal analyser system, but can take data 10 times faster than the analyser system. With future improvements to the CCD detector, we expect to achieve a 20x improvement in speed over the 11-BM system. If multiple CCDs are employed, this speed gain can be further multiplied.

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