

# Kodak CCD-Based Detector for Small Angle X-Ray Scattering

Hok-ling Lee, Timothy Madden, *Member, IEEE*, Patricia Fernandez, Byeongdu Lee, Soenke Seifert, John Weizeorick, and Michael Molitsky

**Abstract**--The Beamline Technical Support Group (BTS) at the Advanced Photon Source (APS) has developed two CCD detector systems (the Single and Quad Platinum systems), for x-ray diffraction and imaging experiments. Both of these systems, optimized for sensitivity toward x-ray photons, utilize the Kodak KAF-4320E CCD coupled to fiber-optic tapers, custom mechanical hardware, electronics, and software systems developed at the APS. Each CCD is composed of  $\sim 2k \times 2k$  25- $\mu m$ -sized pixels, providing a total active detector area of 168 mm x 168 mm. In fast mode with 4 x 4 binning, the Quad system can reach a rate of 5 frames per second (fps). The sensitivity of the detector has been enhanced by using tapers with low demagnification ratio (1.78) and CCDs with high quantum efficiency. The sensitivity, resolution, and dynamic range have made the detectors suitable for a wide range of synchrotron experiment, in particular, small-angle x-ray scattering (SAXS). SAXS data from the x-ray scattering of silver behenate powder have been used to demonstrate the performance of the detector.

## I. INTRODUCTION

This paper presents a custom design of a large-area charge-coupled device (CCD) x-ray detector called the Platinum. Two versions of the Platinum have been built: a 2x2 array of fiber-optic-coupled CCDs and a single fiber-optic-coupled CCD. The CCD is currently a work horse for synchrotron x-ray experiments, with applications including single or polycrystalline diffraction, phase contrast imaging, small-angle x-ray scattering (SAXS), and ultra-small-angle x-ray scattering (USAXS). Although different experiments have their own sets of requirements, there are common features in a detector that experimenters want. High pixel count is preferred so large solid angles can be covered. A small point spread function, usually around 1 to 5 pixels, is required so that small features in the signal can be easily resolved. Single-photon detection in the most relevant photon energy is desirable as well. Because a modern synchrotron can produce an x-ray beam with a 10-GHz count rate, a sample scattering x-rays can

produce data with large signal-to-noise ratios in a relatively short time. Therefore, a relatively fast frame rate is desirable in a detector design.

Because of their target markets, commercial CCD detectors are usually tuned toward some but not all of the aforementioned features. Large-area detectors like the Mar225 and the ADSC Quantum target the protein crystallography market. These systems are moderately sensitive in the 10-keV energy range and feature frame rates of around 1fps [1, 2]. The Quad Platinum detector is designed for a maximum frame rate of 5 fps at a relative high pixel count of 2048 x 2048 and single-photon detection at 8 keV. The driving experiment for the Platinum design is SAXS at Sector 12-IDC at the Advanced Photon Source (APS). Specifically, the Platinum was built to replace an older Argonne-designed CCD system called the Gold [3].

## II. MECHANICAL DESIGN

The detector was designed such that the operating temperature could be set as low as  $-40^\circ C$ , though it is typically set at  $-25^\circ C$ . A  $Gd_2S_2O_7:Tb$  phosphor sheet converts incoming x-ray photons to visible light that the CCDs detect. To increase the detection area and allow nearly seamless tiling of CCDs, a 2 x 2 taper array with appropriate demagnification ratio (1.78) is placed between the phosphor and the CCDs. A cross-sectional drawing of the detector chassis is shown in Fig. 1. The detector head consists of a vacuum vessel containing CCDs, tapers, CCD pre-amplifiers, CCD cooling components, and a nitrogen reservoir (pillow) in front. Electronic circuitboards for data capture are distributed around the four sides of the vacuum vessel, outside of vacuum. The vacuum vessel itself is composed of two sub-chambers that are open to each other. The four CCDs, tapers, and phosphor are situated in the front chamber, and the compact cooler is in the back chamber.

The cooling of the CCDs is provided by a Polycold compact chiller (PCC) and cryogenic cold head from Brooks Automation. With the nonflammable NF55 refrigerant, the chiller can provide a temperature ranging from  $-90^\circ C$  to  $20^\circ C$ , with a cooling capacity of up to 50 W [4]. The vacuum vessel, roughly 14.5" x 14.5" x 14.5" in size, is built to provide the 0.005-Torr environment that is needed for operation of the chiller. The cold head, connected by cooling hoses to the chiller, is attached to an OFHC copper plate that

Manuscript received November 13, 2009. This work is supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract no. DE-AC02-06CH11357.

Hok-ling Lee is with XSD of Advanced Photon Source, Argonne National Laboratory, IL 60439 USA (e-mail: jlee@aps.anl.gov).

Timothy Madden, Patricia Fernandez, Byeongdu Lee, Soenke Seifert and John Weizeorick are with XSD of Advanced Photon Source, Argonne National Laboratory, IL 60439 USA.

Michael Molitsky is with the Structural Biology Center, Argonne National Laboratory, IL 60439 USA.

is thermally isolated from the back chamber, and hence room temperature, by a G10 support. The four CCDs are thermally connected to the copper plate by individual spring-loaded copper plungers. The plungers are designed for a thermal resistance of 1 W/K and remain movable at cryogenic temperatures. While the chiller and cold head cool the CCDs, a Lakeshore temperature controller and resistive heater mounted near the CCDs regulates the CCD temperature.

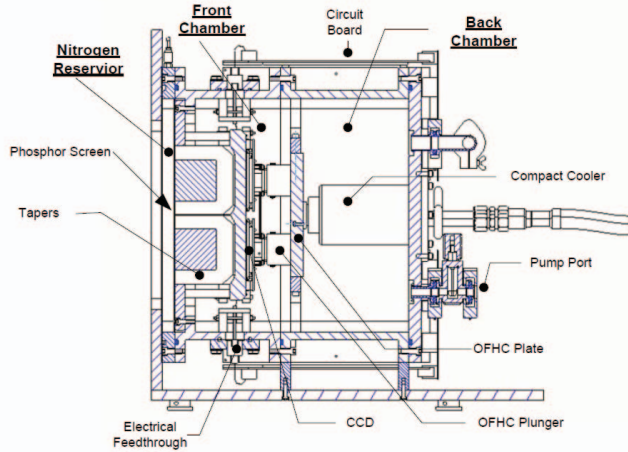


Fig. 1. The figure shows the cross section of the detector head. The detector head consists of a vacuum chamber and a nitrogen reservoir in front of it. The vacuum chamber itself is composed of two sub chambers, namely, the front and back chamber. The compact cooler is housed in the back chamber and it is thermally connected to the backside of the CCDs through an OFHC plate and plungers. The incoming x-ray photons will hit the phosphor screen at front portion of the detector head through the nitrogen reservoir. The generated visible photons will transmit through the optical tapers and landed on the front surface of the CCDs. Electronic circuit boards are placed around the 4 sides of the vacuum chamber. Electrical feedthroughs present at the chamber walls enable electrical connections from the outside to the inside of the chamber.

The phosphor screen is a single sheet of commercially available (Grant Scientific) P43 phosphor material ( $\text{Gd}_2\text{O}_2\text{S:Tb}$ ) deposited on a 1.5-mil-thick aluminized mylar substrate. Because the phosphor screen doubles as a vacuum window, the screen is pressed against the fiber-optic tapers by atmospheric pressure. The density of the phosphor powder is about  $10 \text{ mg/cm}^2$  to provide good stopping power of 8- to 15-keV x-rays [5]. With the demagnification factor  $D$  of the fiber-optic tapers at 1.78, the resulting area of the Quad Platinum is  $164 \text{ mm} \times 164 \text{ mm}$  with around  $350 \text{ }\mu\text{m}$  of dead space between tapers. The small  $D$  value has provided two operational advantages to the detector. Because the efficiency of the fiber-optic tapers decreases as  $D^2$ , a smaller  $D$  value means higher photon transmission efficiency. At the same time, a smaller  $D$  means there is less geometric distortion [6]. Because the tapers are in vacuum and the front vacuum window is mylar, the glass tapers must support the full force of one atmosphere. Given the taper area of  $168 \text{ mm} \times 168 \text{ mm}$ , this means the tapers support 650 lbs. Therefore a metal taper holder supports the glass from behind, ensuring the glass is always under compression and not tension. Teflon and delrin shims have been put between the taper holder and the tapers and between tapers to avoid damage to the glass from

vibration or mechanical forces. To minimize the effect of thermal expansion, the taper holder is made of Kovar, which has nearly the same thermal expansion coefficient as the glass tapers.

When the CCDs are cooled, the front of the tapers and phosphor can get cool enough to form condensation on the vacuum window. To prevent condensation, a second mylar window is put in front of the vacuum window to create a small reservoir to hold dry gas. The gas provides sufficient thermal insulation to prevent condensation on the front of the detector.

### III. ELECTRONICS SYSTEMS

The Quad Platinum detector, having four Kodak CCDs, features four electronic Camera Modules corresponding to each CCD. The module generates CCD clock signals and voltage biases, digitizes CCD output, controls CCD signal gain, and stores images in RAM. The four modules are connected via flex cable to a central Interface Board, which converts image data into Camera Link format for transmission to the computer and manages detector setup and control via serial download from the computer. It also allows for detector and shutter control by synchrotron x-ray beamline equipment. A diagram of the Platinum electronics is shown in Fig. 2.

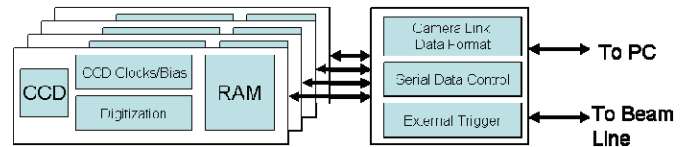


Fig. 2. Diagram of electronics of the Quad Platinum detector. Four camera modules are shown, each including CCD, bias and clock generation, digitization, and image RAM. Four modules feed into single camera link interface board to ultimately connect to PC. The Interface Board also features connectors for exposure and shutter triggering from beam-line equipment at the accelerator.

The APS-designed Platinum detector camera module circuitry is modeled on a previous Argonne CCD detector design, specifically the detector documented in [7]. Each Camera Module is comprised of 5 circuit boards: CCD socket board, preamplifier board, analog board, drive board, and RAM board. A block diagram of the camera module is shown in Fig. 3.

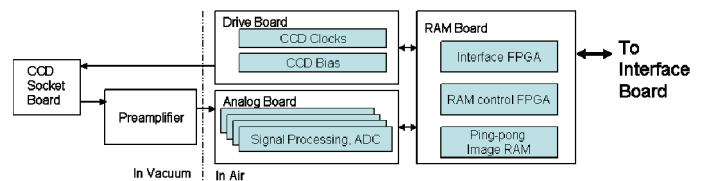


Figure 3. Diagram of Camera Module, including CCD and socket board, preamplifier board, Drive Board, Analog Board, and RAM board. Module connects to Interface board via Flex cable.

Inside the front vacuum chamber the CCD is plugged into the socket board, containing no additional active components to avoid heat generation. The socket board is connected via separate flex cables to two small circuit boards mounted on

the chamber wall, one being a preamplifier connecting to the analog board, and the other being simply a passive connection to the drive board. The preamplifier board, which is heat-sunk to the aluminum vacuum chamber wall, features four differential amplifiers that convert the four CCD outputs to differential signals before being sent through the chamber wall, via hermetic feedthrough, to the analog board. The analog board, residing outside the vacuum, comprises four analog processing paths corresponding to four CCD outputs. Each signal path converts the differential signal to single-ended; adjusts the gain of the signal; removes noise using a low-pass multiple feedback filter topology; and digitizes the signal using double-correlated sampling realized by analog switch, variable DC offset DAC, and ADC. The analog board can sample each CCD channel at 3 MHz (for a total of 12 MHz for the CCD). The gain is adjustable in eight steps from  $3 \text{ e}^-/\text{ADU}$  to  $10 \text{ e}^-/\text{ADU}$ . A block diagram of the analog signal path is shown in Fig. 4.

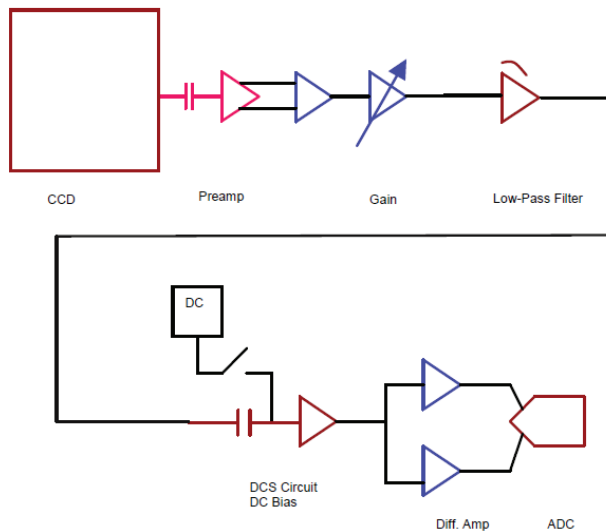


Fig. 4. Analog signal path showing one of four CCD outputs, differential driver and receiver, variable gain and low pass stages, DC bias offset and double correlated sampling with analog switch, and finally differential ADC.

The drive board, residing outside the vacuum and connected to the socket board via hermetic connector, comprises a FPGA that generates CCD clock signals, circuitry to convert TTL clocks to AC voltages suitable for the CCD, and DC bias circuits for the CCD. The CCD clock pattern is programmable on the fly by using a “CCD Timing Language” that allows one to create a series of 1’s and 0’s corresponding to the clock signals and automatically converts to serial data stream for download to the FPGA.

The drive and analog boards both connect to the RAM board via PCI connectors. The RAM board includes memory for storing two images to be read in a “ping-pong” fashion: as one image is uploaded to the PC, a second image is read out from the CCD. Also, the RAM board contains logic for interfacing to the interface board via flex cable.

The camera link interface board houses a single 60-MHz clock oscillator used to generate all clock signals for the entire detector. Because of nonlinearity in ADCs and analog

components, the use of multiple clocks can generate noise in the analog signal path. A single clock minimizes analog noise. The interface board also contains an FPGA implementing a NIOS soft processor to aid in formatting raw image data into the camera link and to manage detector control with serial data streams [8]. The soft processor communicates with the PC via serial interface through the camera link cable. For generating camera link data, the interface board contains a special chip set designed for the camera link format [9].

To interface the detector to beamline equipment, the interface board incorporates seven Lemo connectors used for triggering the detector and controlling an x-ray shutter. The functions of the Lemo connectors can be reconfigured by reprogramming the FPGA on the interface board. A table of basic detector specifications is shown in Table 1.

Table 1. Basic Specifications for Quad Platinum

Read Noise	23 $\text{e}^-$
Gain	3 $\text{e}^-/\text{ADU}$ to 10 $\text{e}^-/\text{ADU}$
Dark Current	0.67 ADU/s, 4x4 binned, -20 °C.
Area	164 mm x 164 mm square
Pixels	1024x1024 (4x4 binned), 2048 x 2048 pixels (2x2 binned)
Pixel size (at phosphor)	178 $\mu\text{m}$ x 178 $\mu\text{m}$ (4x4 binned), 89 $\mu\text{m}$ x 89 $\mu\text{m}$ (2x2 binned)
Sensitivity	Single 8-keV photon generates about 25 $\text{e}^-$
Max Frame Rate	5 fps (binned)
Point Spread FWHM	100 $\mu\text{m}$

#### IV. AN APPLICATION OF THE PLATINUM DETECTOR

SAXS signal intensity decreases with a power law of the scattering vector  $q$ . The order of the power law can vary from 1 to 4, in general. Often the scattering intensity at large  $q$  can be orders of magnitude less than the background scattering signal generated by the solvent, container, vacuum windows, air, and optics. A two-dimensional detector with a large dynamic range to improve data statistics at high  $q$  and with a highly stable dark current are major requirements for a CCD system used in SAXS experiments. High quantum efficiency is also critical to reduce exposure time as much as possible to minimize sample damage by the x-ray beam. As the flux and resolution of x-ray instruments are drastically improved in modern synchrotron facilities, fast data acquisition with low readout noise is highly desirable. In addition, a narrow point spread function and small pixel size are required because, when using SAXS in the study of nanostructures, the sample often produces sharp diffraction peaks. The Quad detector has been used in SAXS experiments with good results. A SAXS image produced by the Platinum detector is shown in Fig. 5.

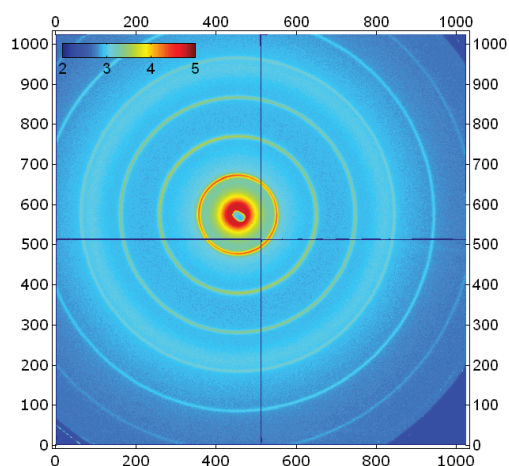


Fig. 5. SAXS image of silver behenate powder, which is a typical scattering vector calibration standard for SAXS, packed in a Kapton envelope with about 0.5-mm thickness. Measurement parameters are: exposure time, 0.5 second; x-ray energy, 12 keV; x-ray beam size, 0.5\*0.1 mm; beamline, 12ID at APS. Data are displayed on a logarithmic color scale.

#### REFERENCES

- [1] Mar Corporate Website: <http://www.marusa.com>
- [2] ADSC Corporate Website: <http://www.adsc-xray.com>
- [3] I. Naday, S. Ross, M. Kanyo, M. Westbrook, E. Westbrook, "Gold detector: modular CCD area detector for macromolecular crystallography," *Proc. SPIE*, vol. 2415, pp. 236-242, 1995
- [4] Brooks Automation Corporate Website: <http://www.brooks.com>
- [5] Sol M. Gruner, Mark W. Tate and Eric F. Eikenberry, "Charge-coupled device area x-ray detectors", *Rev. Sci. Inst.*, vol. 73, no. 8, pp. 2815-2842, 2002
- [6] Masayo Suzuki, Masaki Yamamoto, Takashi Kumasaka, Kazumichi Sato, Hidenori Toyokawa, Ian F. Aries, Paul A. Jerram, Daniel Gullick and Tatzuo Ueki, "A multiple-CCD X-ray detector and its basic characterization", *J. Synchrotron Rad.*, vol. 6, pp. 6-18, 1999
- [7] Timothy J. Madden, William McGuigan, Michael J. Molitsky, Istvan Naday, Alan McArthur and Edwin M. Westbrook, "Lens-Coupled CCD detector for x-ray crystallography", *IEEE Trans. Nucl. Sci.*, vol.2, pp 729-734, Nov 2006
- [8] Altera Corporate Website: <http://www.altera.com>
- [9] Vision One Corporate Website:  
<http://www.vision1.com/pdf/CameraLink5.pdf>