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Lens-Coupled CCD Detector for X-ray Crystallography

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Abstract

An x-ray crystallography detector (Blue-1) has been built based upon a Fairchild 486 back-illuminated CCD and a custom lens system designed by Optics One Inc. The advantages of our Blue-1 lens system over more conventional fiber-optic tapers are: lower noise and higher efficiency; improved point spread function; negligible spatial distortion; and lack of “chicken-wire” patterns. Also, the engineering is simpler because the CCD is not bonded to the fiber-optic taper. A unique mechanical design has been employed to accurately focus the image on the CCD. The detector software is based on MATLAB and takes advantage of its powerful imaging and signal processing libraries. The CCD timing can be updated on the fly by using a “CCD controller language” to specify timing.

I. Introduction

In the field of molecular biology, crystallized biological materials are studied at synchrotrons by X-ray diffraction, in which intensities of Bragg peaks incident upon the face of a detector must be measured efficiently and accurately [1]–[5]. Typically, the X-ray field intensity is converted to visible light in a phosphor film. This light is transferred to a CCD, usually via a fiber-optic taper. Fiber optic tapers are normally used for this application to maximize detector sensitivity and readout speed [6]–[9]. Several fiber-optically coupled detectors can be found in the literature. A particularly good list of detectors appears in [10].

Although fiber-optic detectors have proven to be successful, there are several drawbacks with these systems. First, fiber-optic tapers exhibit *chicken wire*, the projection of a honeycomb pattern in the images due to the inherent design of the taper [11]. Second, the CCD must be mechanically bonded to the fiber-optic glass (which is a complex process). Third, the fiber-optic tapers introduce spatial distortion into the images [12]. In large area fiber-optic systems, several tapers are bonded together into a mosaic. These detectors are insensitive to light (or X-rays) at the seams where the tapers are bonded.

Though the light transfer efficiency of a lens system is less than that of a fiber-optic taper, we have overcome this deficit by using a very large, back-illuminated CCD. Combining this with a modern lens design, we have successfully developed a fine detector with lens optical coupling [10].

Lenses do not exhibit several negative properties of fiber-optics, such as chicken wire. Lenses can be designed to have finer point spread function than tapers, and much less spatial distortion. Also unlike tapers, lenses can be tiled to have no insensitive areas in the detector face. Some of us were involved in building several lens-coupled detectors in the past [13]-[15]. One of these, a very successful lens detector called the “Noir-1,” is currently installed and operational at Lawrence Berkeley National Laboratory. It has been in essentially continuous use for over three years in service for a wide crystallographic community, and has taken thousands of high quality datasets that have been used to solve hundreds of crystal structures.

We have recently built a sister to the Noir-1, called the “Blue-1.” Both detectors feature identical lens systems designed by OPTICS One Inc. [16]. The main difference between the Blue-1 and the Noir-1 is that, while the Noir-1 utilizes a Fairchild detector head, the Blue-1 features a custom detector head designed at Argonne National Laboratory. The purpose of the Blue-1 design was to improve noise performance and to allow the detector to be tiled into a 2×2 system, because it is advantageous for a detector to have a large area in crystallography applications [17], [18].

II. General Detector Features

The Blue-1 detector (Fig. 1) features a custom lens system, designed and built by OPTICS One, and a Fairchild back-illuminated 486CCD, enclosed in a dewar filled with continuous flowing dry nitrogen [19]. The nitrogen is supplied by a bottle of gas and is necessary to keep the CCD dry. At a proper flow rate, the bottle lasts for about a month of continuous use. The CCD is cooled to -27°C with a thermo-electric cooler (TEC), powered by a low noise temperature controller. To keep the TEC cool, the TEC is attached to a metal plate through which 10°C chilled water flows (temperature controlled by a Neslab RTE7).

The phosphor is $\text{Gd}_2\text{S}_2\text{O:Eu}$, deposited on a light-tight aluminized mylar sheet. The phosphor sheet is fastened to the lens system by drawing a vacuum with a continuously running cheap, small vacuum pump (the Air Admiral).

The CCD dewar features a front non-reflective window, which is bolted directly to the lens. Thus, the CCD is fixed to the lens to preserve focal length. The electronics are connected by short cables and are mechanically separate from the dewar. The camera is focused by moving the CCD via a *flex plate* design, described below.

The detector head contains Argonne-designed analog and digital electronic circuit boards attached to water-cooled plates, which remove heat and reduce thermal drift effects. The electronics contain four analog-to-digital converters to simultaneously digitize each of the four analog outputs from the Fairchild CCD. Digital circuitry is included that generates CCD clock signals, and interfaces to the computer. The detector is connected to a Windows computer via a National Instruments 6534 parallel interface card [20]. The computer runs a custom software system created at Argonne for the detector. This software can run either standalone with a MATLAB GUI, or with an EPICS interface [21], [22].

III. Optical Design

The lens system was designed and built by OPTICS One Corporation in West Lake Village, CA. The design utilizes nine lenses, two of which are aspherically ground (Fig 2). The main

objectives of the lens design were: to provide a fine point spread function, to maximize sensitivity, to allow tiling of lenses into a 2×2 array, and to properly focus the entire color spectrum emitted by the phosphor. Table I summarizes the lens design parameters.

The lens system was designed to provide a sufficiently small point spread function that 99% of the light from a point source on the phosphor falls within a diameter of 150μm in the image. Figure 3 illustrates the point response diagrams simulated by OPTICS One, from a point source on axis, and 10mm, 20mm, etc. radially away from the axis, on the image plane (the edge of the image is 112.48mm from the axis).

In the 12 diagrams of Fig. 3, the differently colored rings represent the shapes of the foci of different light wavelengths. The black circle in each diagram represents a 100μm diameter. The lens is designed to be tiled as a 2×2 system, to create a detector of 317mm × 317mm active area. Because the imaging fields of each lens overlap (by ~0.5mm), the resulting detector has no dead areas at module boundaries. To implement the 2×2 large array (Blue-4), a single large phosphor would be installed on the detector to be imaged by the four lenses. At the time of this publication, the tiled system has not yet been built. A model of the tiled lens system is shown in Fig. 4.

IV. Detector Sensitivity

It has been argued in the literature that lens systems are too inefficient for X-ray detector applications [10]. The efficiency of our lens, calculated in (1) from the numerical aperture (NA) of 0.16 [15]:

$$E = \tau \times (NA)^2 \quad (1)$$

(with $\tau = 0.8$, the approximate total transmission coefficient of the lens system), is about 2%. We kept the NA high by reducing the demagnification factor (2.589×), since the Fairchild CCD486 is very big (61.44mm). About 500 optical photons are generated from a 12keV X-ray hitting the phosphor [23]. This means that, for each X-ray photon, about 10 optical photons make it to the CCD from the phosphor. The back-illuminated 486CCD has a very high quantum efficiency: about 90%. Thus, of the 10 photons reaching the CCD, 9 of them generate an electron in the CCD. This sensitivity has been verified by our measurements and is comparable to commercial fiber-optic taper detectors, such as that of Area Detector Systems Corp (ADSC), which gives 8.5e- for a 12keV X-ray photon [24]. With a read noise of 12e-, the Blue-1 signal-to-noise ratio is about 0.75×. In comparison, the ADSC Q315 read noise is 18e-, so its signal-to-noise ratio is about 0.47.

V. Focusing System

The detector is focused by moving the CCD relative to the lens. Because the depth of the focal plane is less than 100μm, the CCD must be moved very finely. Also, because the CCD must be kept cold and dry, it must be moved while keeping it sealed in a dewar. This is accomplished by mounting the CCD on a “flex plate,” a thin aluminum plate that moves like a drum head. Three micrometers push on the flex plate to allow CCD movement with micron precision. This arrangement allows the CCD to be moved along the lens axis, and it can also be tilted in any direction. A photograph of the detector head showing the dewar and micrometers is shown in Fig. 5.

VI. System Specifications

General specifications of the detector system are shown in Table II.

In the Blue-1 detector, the Fairchild CCD486 is operated in 2-by-2 binning mode, so the total number of pixels is 2048×2048 . All four outputs of the CCD are read simultaneously at 500kHz to give a total pixel rate of 2MHz.

The point spread function was measured by irradiating a $10\mu\text{m}$ -diameter hole in a tantalum film with a 12keV X-ray beam, after the detector had been focused. Images were dark-subtracted and corrected for nonuniform response. The response function fitted very well (correlation coeff: 0.99) to a Gaussian function with $\sigma=0.10\text{mm}$, down to a noise floor of $\pm 2\text{ADU}$, where the peak response at the center was 25,000ADU [25].

Read noise was measured to be 12e- rms by reading only the serial register with no vertical shifts. The electronic gain was measured by the mean/variance method [26], applied to a series of dark images.

VII. Electronics Design

The detector electronics feature three major circuit boards and several minor circuit boards. Major boards include the *analog board*, *drive board*, and *digital board*. Other boards include the *Interface Board* and CCD socket assembly. A photograph of the detector head showing the major circuit boards appears in Fig. 6.

The drive board has a field programmable gate array (FPGA) generating CCD signals as TTL pulses. These pulses are converted to proper voltages acceptable by the CCD. To reduce noise, the CCD clocks are filtered. The analog board contains four analog signal paths, one for each CCD output. The digital board has enough RAM to store two image frames such that, as one frame is downloaded to the computer, a new frame is read from the CCD creating a pipeline. At the time of this publication, the pipeline operation has not yet been implemented. The digital interface board has buffers to drive a parallel cable to the National Instruments card installed on the PC. The CCD plugs into a socket assembly including a low noise preamplifier. The CCD socket assembly resides in the gas-filled dewar. The analog and drive boards are fastened to water-cooled aluminum plates for cooling and thermal regulation.

VIII. Analog Signal Path

The analog signal path was designed so that the CCD is the largest noise source. This was accomplished by selecting a very high quality analog-to-digital converter (ADC), the AD7677 from Analog Devices, and by using a very low noise operational amplifier, the Texas Instruments THS4032, in the first stage of the signal chain [27], [28]. The signal path is diagrammed in Fig. 7.

The analog signal path has eight gain settings to set the gain from 5e-/ADU to 10e-/ADU in eight steps. This is accomplished by switching the feedback resistance in an operational amplifier circuit. To optimize read noise performance, the signal path has two selectable bandwidths to allow reading the CCD at two different pixel rates. At the time of this publication, only the 500kHz pixel rate has been used.

The analog signal path utilizes correlated double sampling (CDS) to reduce the noise added by the CCD reset gate [29]. This is accomplished by shunting the capacitively coupled signal to a programmed DC voltage after the reset. This DC voltage sets the background bias in the captured image from the detector. The ADG201 from Analog Devices is used for the CDS circuit. Because the ADC is differential, the CCD signal is converted from single ended to differential before feeding to the ADC.

IX. CCD Control Language

An FPGA was designed for generating CCD timing as logic pulses. Because the CCD timing must be altered for different readout speeds, binning, and CCD timing optimization, it was desirable to alter timing without recompiling the FPGA. For this reason, the FPGA was designed to function like a very simple microprocessor, complete with its own assembly language. We call this language “CCD Control Language.” Using CCD Control Language, it is possible to quickly reconfigure the CCD timing on the fly. In practice, writing Assembly-like language is timing-consuming. Therefore, a higher layer of software was created allowing the use of Tcl scripts to create CCD timing sequences [30]. The steps to create CCD timing are as follows. First, create Tcl arrays of 1s and 0s representing the CCD timing. Second, convert the Tcl arrays into CCD timing language using custom Argonne-designed Tcl scripts. Third, use Argonne-designed scripts to assemble the CCD Control Language into a binary file to download to the FPGA. In this way, CCD timing is updated on the fly, with automatically-generated CCD Control Language.

X. Calibration Process

The Blue-1 detector was shipped from Argonne National Laboratory to Lawrence Berkeley National Laboratory for testing on beam line 4.2.2 at the Advanced Light Source synchrotron.

We calibrated the Blue-1 detector by standard procedures [31], [32]. Flood images were recorded by tuning the beamline X-ray energy to 12keV and irradiating a thin (10 μ m) gold foil. Gold absorbs X-rays with energy above 11,919keV, its absorption edge. The subsequent gold fluorescence is primarily at 9,713keV, and it is truly isotropic. However gold foil, being essentially a polycrystalline powder, also exhibits Bragg powder diffraction rings, the narrowest of which is the (111) reflection (gold is a cubic crystal, Fm3m, $a = 4.0786\text{\AA}$), scattering from the gold foil at a Bragg θ angle of 12.68°. To avoid this ring and also avoid the direct X-ray beam, the detector was placed 40cm from the gold foil and offset vertically 18cm. Strong ($\sim 20,000\text{ADU/pixel}$) fluorescence signals were obtained with a 1,000s exposure. Sixteen flood images were recorded, averaged, subjected to outlier (“zinger”) suppression, and re-averaged with zingers removed to give a consensus flood image. Sixteen dark images were also recorded (identical geometry used for flood images, but without X-ray exposure). These also were averaged, zinger-suppressed, and reaveraged. The consensus dark image was subtracted from the consensus flood image, and this X-ray field intensity image was compared algebraically to the theoretical reference field, i.e., the expected field strength in this geometry, which follows a $\cos^2(\theta)$ functional form. In this calibration procedure we calculate an image file that is, for each pixel, the multiplicative factor that corrects for nonuniform sensitivity/response over the entire detector face.

Sixteen dark images were recorded at recording times of 0.1s, 1s, 10s, 100s, 200s, . . . , 1,000s. The 0.1s dark image was taken to be the “DC Offset” image. For each recording time, the sixteen images were averaged, zinger-suppressed, and reaveraged. To determine electronic gain, the variance (square of the standard deviation) of these dark images was compared to their averages, pixel by pixel. The average ratio of ($\sigma^2/\langle\text{Dark}\rangle$), taken to be the electronic gain of the system [26], was 4.89 e-/ADU.

To study spatial distortion of the detector image, we fabricated a 1mm-thick brass plate covering the entire detector face. We machined a square raster of holes (each 1/32” diameter), 10 holes/inch, in the brass plate, put this plate over the detector face, and exposed the assembly to the same fluorescence flood field used above. Good image contrast of the spots due to holes was obtained in 100s images. A computer program was written to find the centroids of these spots, fit a polynomial function to each vertical and horizontal line of spot centroids, and

calculate the conformal mapping between the observed spot positions and their expected positions in a square matrix. The essential bottom-line of these tests was that there was minimal distortion of the image.

XI. Crystallographic Testing

The first complete dataset of a protein crystal recorded by the Blue-1 detector was from a routinely-used benchmark protein crystal, hen egg-white lysozyme. One hundred eighty data frames, each recording diffraction from the crystal as it rotated 1° , were recorded. Each data frame was recorded for 1 second. One such diffraction image is shown in Fig. 8.

At the time we recorded this dataset, the Blue-1 detector had not yet been perfectly focused, as can be appreciated here, since the Bragg spots are slightly blurred in this image.

As a measure of data quality, crystallographers use a quantity called the “R-merge” to measure the quality of the diffraction data [33]. The overall R-merge for this first dataset was 3%, for 209,211 Bragg spot measurements out to the edge of the detector (1.5Å resolution). This is quite good. R-merge values are never better than 2%. Typically for delivery of a new commercial detector system, an acceptance requirement would be that a lysozyme dataset be recorded with an R-merge better than 5%, so this detector is already good enough to be used routinely at a synchrotron beamline. We anticipate that the Blue-1 detector will perform even better once it is focused properly, and once we have had the opportunity to truly tune it up.

The data frames recorded by our lens system are exceptionally clean; they have no chicken wire patterns because they lack fiber-optics. Also, we verified that no spatial correction is needed for solving crystal structures because the lens essentially does not distort the image. In fact the lysozyme dataset discussed above was processed without any distortion corrections at all.

XII. Conclusion

By building the Blue-1 detector, we have demonstrated that an excellent crystallographic detector system can be developed in which the light transfer medium is a lens, rather than a fiber-optic taper. A drawback to the lens system is that, in order to achieve the required sensitivity, a large back-illuminated CCD is necessary, and the lens must be long. Indeed, the lens system on the Blue-1 detector is 750mm long, taking up room in the hutch. The performance of the lens system is comparable or superior to conventional fiber-optic systems in terms of point spread function, noise, and sensitivity. The lens data are more uniform, and they are cleaner than those from a fiber-optic system. Furthermore, no software spatial correction is needed.

We continue testing and evaluating the Blue-1 detector. It is already excellent. Over the next several months we expect to be able to demonstrate that it will perform even better.

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Fig. 1. Argonne Blue-1 detector. The detector head was designed and built at Argonne National Laboratory. Lens was designed and built at OPTICS One.

Fig. 2. Diagram of lens design. Nine lenses are included with seven spherical and two aspherical. The total length from object (phosphor on left) to CCD at far right is 750mm.

Fig. 3.
Encircled energy plots of lens design. Simulated image on CCD of a point source.

Fig. 4.
Four lenses stacked as a tiled array.

Fig. 5.
Detector head showing dewar and micrometers.

Fig. 6.
Photograph of detector head showing major circuit boards.

Fig. 7. Diagram of analog signal path. The CCD signal is capacitively coupled to the preamplifier, in the dewar. The preamplifier feeds to the selectable gain stage, filtering stage, and DCS circuit. The signal is converted to differential before sending to the ADC.

Fig. 8.
Sample diffraction image from Argonne Blue-1 detector.

Table I

General Lens Specifications

Specification	Value
Active Imaging Area	160mm × 160mm
Demagnification Factor	2.589×
Image on CCD	61.44mm × 61.44mm
Numerical Aperture	0.16 (at phosphor)
Efficiency	>2%
Sensitivity at Edge	57% of sensitivity at center
Spot Size/Resolution	99% encircled energy within a 150μm diameter in the image plane
Spatial Distortion	<0.5%

Table II

Detector Specifications

Specification	Value
Frame Rate	0.44 frames/s
Pixel Rate	500,000 pixels/s
Read Noise	12e- rms
Full Well	~400,000 e-
Dark Current	32 e-/sec-pixel, @ -27°C
Dark Current Noise	6 e- rms for 1 second image
Pixel Size(at phosphor)	78 μm $\times$ 78 μm
Number of Pixels	2048 $\times$ 2048 pixels
Conversion Gain	9e-/12keV X-ray photon, measured
Electronics Gain	5 e-/ADU, measured
Dynamic Range	~33,000
Point Spread Function	Gaussian, $\sigma=0.1$ mm
FWHM	180 μm (2.3 pixels), measured
Phosphor	Gd ₂ O ₂ S: Eu