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Characterization of CCD-based Imaging X-ray Detectors for Diffraction Experiments

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High resolution CCD-based imaging detectors are successfully used in X-ray diffraction experiments. Some of the detectors are commercially available, others have been developed by research groups around the world. Reliable comparison of the performance must be based on thorough testing of all relevant characteristics of these detectors. We describe methods of measurements of detector parameters such as conversion gain, linearity, uniformity, point spread function, geometrical uniformity, dark current, and detective quantum efficiency. As an example for the characterization, test results of a single module fiberoptic taper/CCD X-ray detector will be presented. The projected performance of a large area, array detector consisting of 9 CCD's and fiberoptic taper modules, will be given. This new detector (the "Gold" detector) will be installed on Beamline X8C at the Brookhaven National Laboratory at the NSLS Synchrotron.
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1. Introduction

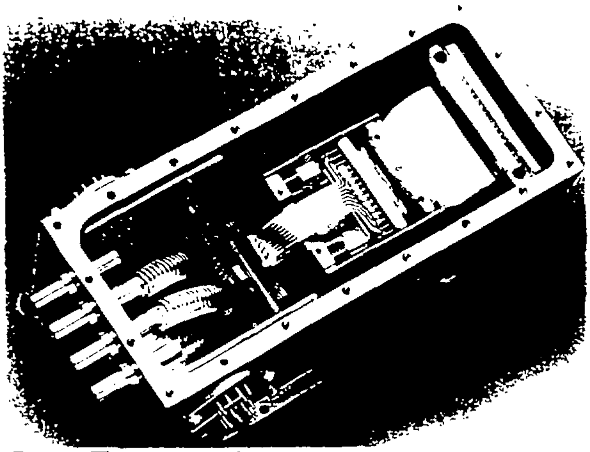
X-ray imaging cameras based on charge coupled devices (CCD's) are being used on synchrotron beamlines for crystallography, where they measure the position and intensity of Bragg diffraction peaks. With the spread of CCD based integrating type position sensitive detectors, it becomes important to establish a standard set of measurements and test procedures by which one can evaluate different detectors for different applications. In this paper we discuss a typical architecture of an X-ray detector, summarize the important detector parameters, giving a procedure by which these parameters may be measured, and finally give results specific to the Argonne National Laboratory "Green" CCD detector, built for protein crystallography applications.

2. Typical CCD-based X-ray detector.

The detector parameters discussed in this paper are general to all X-ray imaging detectors. However these detectors may actually be implemented in different ways, with optional gain and demagnification allowing for trade-offs for example of sensitivity with active area. An X-ray detector typically consists of a window to stop visible light while passing X-rays, followed by a scintillator or phosphor material to convert X-rays to visible light, coupled to a CCD imager. In addition, after the phosphor, it may be desirable to amplify the visible light with an image intensifier and/or to expand the sensitive area with a fiber optic taper or lenses. The photoelectrons generated in the CCD are then read off by digital clocking circuitry, amplified, digitized and processed by computers. As an example, the detector head of the Argonne "Green" detector is shown in Fig. 1. In this detector, the X-ray image is formed on a thin phosphor layer, deposited on the larger end of a demagnifier fiberoptic taper. The CCD imager is coupled optically to the smaller end of the taper.

3. Important detector parameters.

All X-ray imaging detectors share the following parameters: conversion gain, non-linearity, non-uniformity, point spread function, geometrical distortion, dark current and detective quantum efficiency (DQE). In this section we review these parameters and present standard test procedures. The test procedures will share many elements, such as a collimated X-ray beam (or Fe^{55} source) and attenuators. (Fig. 2). Where applicable, the measurements are performed by simulated signals similar to the ones under normal operating conditions.



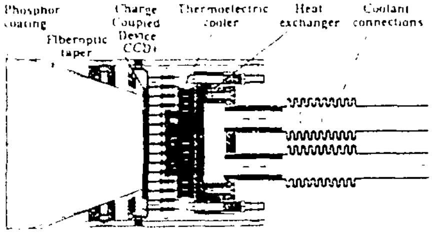


Fig. 1. The "Green" detector.

3.1. Conversion Gain.

Conversion gain of the detectors defined as the output signal in digital numbers per X-ray photon. To measure it we need a monochromatic X-ray source (e.g. a synchrotron and associated X-ray monochromators), a transmission *ion chamber* whose absolute X-ray response has been calculated from its geometry, and an X-ray attenuator calibrated using a phosphor/photomultiplier tube. The conversion gain of the detector is then the ratio of the detector output in analog-to-digital converter units, (ADU) to incident X-ray photons measured by the ion chamber. The X-ray intensity at the detector under test must be similar to the intensity of the Bragg peaks in a typical diffraction experiment. This will require that the intensity of the direct beam be reduced by a calibrated X-ray attenuator The ion chamber should be placed in the unattenuated beam to reduce the statistical error of the measurement.

3.2. Non-Linearity.

Non-linearity is defined as the RMS deviation of a given detector pixel's conversion gain from a linear response. To measure it we need a fast X-ray shutter, a calibrated transmission ion chamber and an X-ray attenuator. We then take a series of measurements with different exposure time settings and/or with different X-ray attenuation values to cover the dynamic range of the detector. Fast, differential pneumatic X-ray shutters, controlled by high accuracy electronics usually give acceptable

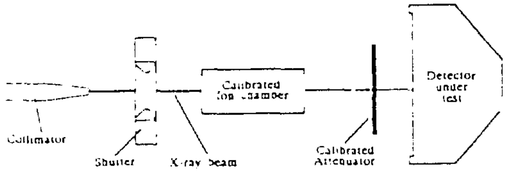


Fig. 2. Test setup for detector parameter measurement.

precision and repeatability for exposure times longer then 100ms. If the dynamic range of the detector is larger then 1000, the linearity measurement series should be repeated with different X-ray attenuations (e.g.: exposure times from 0.1s to 10s, and attenuations of 10 to 1000) to reduce uncertainties in shutter timing and to avoid very long exposure times. A large area (e.g. 200-1000 pixels) should be uniformly illuminated on the detector surface to improve the statistical accuracy of this measurement. The average value of these pixels should be normalized for each frame to the X-ray intensity by using the ion chamber readings. The measured deviation from a fitted linear response is shown in Fig. 3. for the "Green" detector.

3.3. Non-Uniformity.

Non-Uniformity is defined as the RMS deviation of a detector's conversion gain across the sensitive

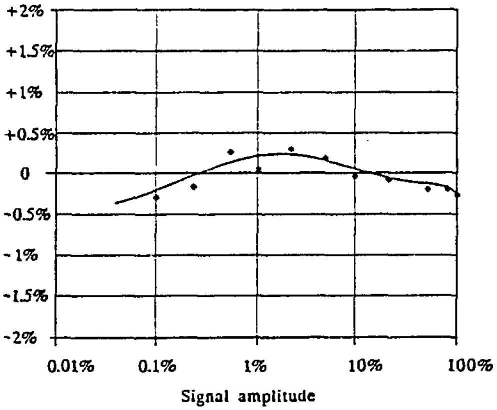


Fig. 3. Deviation from the linear response of the "Green" detector.

area in a given frame. To measure it we need an Fe^{55} X-ray source placed in front of the detector with adequate intensity to illuminate the detector. The flood field should be normalized by $\cos^2 \alpha$ function to remove solid angle effects, where α is the angle between the normal to the detector and the X-ray. The distance from the Fe^{55} source to the surface of the detector should be as large as possible while still maintaining adequate signal. Since the X-ray energy of this source is lower then the usual X-ray energy in protein crystallography experiments (5.9 keV vs. 12 keV), this measurement may give results slightly different then that taken at the synchrotron.

3.4. Point Spread Function.

The image of a finite size source is never as precise as the source itself. The point spread function is a measure of the blurring of a point source and is expressed in terms of full width half maximum (FWHM) of the image. Thus to measure this parameter we require an X-ray pinhole, and an X-ray attenuator. The pinhole is placed close to the sensitive surface of the detector to avoid the effects of scattered X-rays. The attenuated X-ray beam is aligned with the hole. The detector PSF is the image PSF deconvolved with the pinhole PSF (its Fourier transform), or approximately the PSF of the image, if the pinhole is much smaller than the expected detector PSF. In Fig. 4. the measured PSF curve of the Green detector is shown. The point spread function curve usually has wide "tails" at low signal levels because of the photon scattering in the phosphor and along the light path. In high dynamic range detectors the width of the point spread function curve at small fractions (0.1 to 1%) of the maximum should be defined.

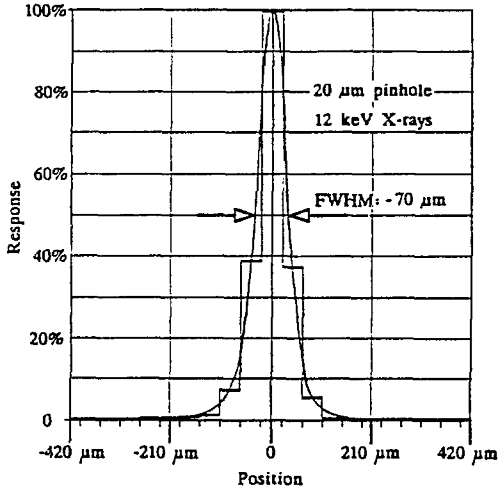


Fig. 4. Measured point spread function of the "Green" detector.

3.5. Geometrical Distortion.

Geometrical distortion is a measure of the change in magnification across the image. An image of a regular grid of lines or points would yield a pin cushion or barrel pattern. Thus to measure geometrical distortion, we need a known, uniform pinhole mask to be illuminated by a sufficiently strong Fe^{55} X-ray source. The size of the holes on the one-two millimeter thick copper mask should be approximately equal to the FWHM of the point spread function of the detector and the distance of the holes should be hundred to twohundred times the hole diameter.The plate must be placed in close proximity to the face of the detector to minimize image blur

3.6. Dark Current.

The main source of the background signal with no X-ray exposure is the thermally generated background charge in the CCD and/or in the image intensifier. Dark current in the CCD detector is a function of the temperature, decreasing by a factor of 2 for every 6 to 9 °C drop in sensor temperature.

To measure the dark current, several exposures are taken within the range of the useful exposure time with no X-ray signal. The background or dark signal of the detector consists of two components:

$$\text{Dark signal (t)} = \text{Offset} + \text{Dark current (t)}$$

The offset is a constant value added by the readout electronics and must be subtracted from the measured pixel values. The offset can be measured by taking zero (or very short) integration time exposures. The dark current of the detector is proportional to the exposure time. An average value of large number of pixels should be taken to reduce the uncertainty of the measurements.

3.7. Detective Quantum Efficiency.

The DQE of a detector is defined as the square of the ratio of the output signal-to-noise to the input signal-to-noise. It is measured by taking multiple exposures over a series of different X-ray intensities, covering the useful dynamic range of the detector. We prefer to use a signal that simulates a Bragg diffraction spot because the conditions during the characterization of the detector are then similar to the actual crystallography operating conditions. Simulated Bragg peaks are created by a pinhole with an image a few pixels in diameter. The DQE can be computed by following the steps:

1) Integrate the measured values within the simulated Bragg-peak area for each exposure.

2) Subtract the offset value.

3) Normalize simulated Bragg-peak values to the ion chamber readings for each exposure (x_{out}).

4) Calculate standard deviation of the normalized values (σ_{out}).

5. Calculate the average of the corrected values.

6. Calculate the number of X-ray photons in the simulated Bragg peak using the measured conversion gain (S_i).

7. Calculate DQE from:

$$DQE = [(x_{out\ avg} / \sigma_{out})^2] / S_i$$

where

$x_{out\ avg}$: average of normalized measured values (integral of the simulated Bragg peak area)

σ_{out} : standard deviation of normalized measured values

S_i : number of photons in the simulated Bragg peak area, calculated from conversion gain

The precision of this measurement increases as the square root of the number of data points. The DQE of the detector is also depends on the signal level as it is shown in Fig. 5. for the Green detector.

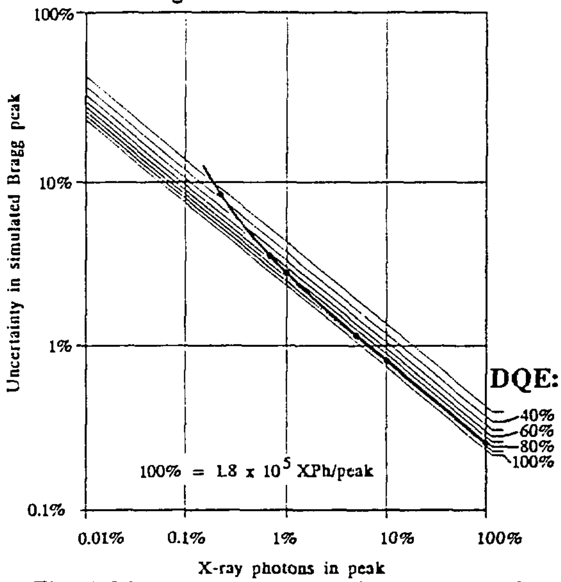


Fig. 5. Measured uncertainty of the intensity of a simulated Bragg reflection.

4. The Argonne National Lab "Green" detector.

This detector consists of a $Gd_2O_2S:Tb$ doped phosphor approximately 25 microns thick (12 mg/cm^2) deposited onto a 2:1 fiberoptic taper. A thermoelectrically cooled Tektronix TK-1024 front side illuminated CCD, with 1024 x 1024 24 μm^2 pixels is attached to the small end of the taper, and read out over several seconds into a computer data acquisition system. The Green detector is a prototype for the more advanced Gold detector,

Table 1.
Parameters of the "Green" and "Gold" detectors.

Parameter:	Green Detector	Gold Detector
Sensitive area:	43.2 x 43.2 mm^2	150 x 150 mm^2
Resolution:	1024 x 1024 pixel	3072 x 3072 pixel
Pixel size @ input:	42 μm	49 μm
Point Spread Function (FWHM):	70 μm	75 μm
CCD read noise:	18 e-rms/pixel (at 6.8 μs/pixel readout)	22 e-rms/pixel (at 3.4 μs/pixel readout)
Digitization noise:	18 e-rms/pixel	10 e-rms/pixel
CCD Dark current:	< 10 e-/s/pixel	< 10 e-/s/pixel
Max. signal:	430,000 e-/pixel	400,000 e-/pixel
Nonlinearity (integral):	<0.8%	<0.8%
Nonuniformity:	6% rms	6% rms
Analog-to-digital converter resolution:	15 bits	16 bits
Readout time (1024 x 1024 pixels):	1.8 sec	1.8 sec
Conversion gain (Energy = 12 keV):	≈ 16e-/XPh	≈ 12e-/XPh
Detective Quantum Efficiency:	≈ 89 %	≈ 82 %

currently being tested at Argonne National Laboratory. The Gold detector consists of a 3 x 3 array of modules, similar to the "Green" detector. In Table. 1. the parameters of the Green detector, measured using the above procedures is presented, along with the predicted performance of the Gold detector.

5. Conclusion

The uniformity is largely limited by the phosphor coating, deposited by a liquid settling technique and by the defects in the fiberoptic taper. The point spread function is mainly affected by the thickness of the phosphor layer (25 - 30 μm)and the grain size of the phosphor (4 - 10 μm). The barrel geometrical distortion is due to the fiber optic bundle not producing a perfect mapping of the front of the bundle to the back. It is easily observed with the fiber bundle alone, looking at a grid with one's eye. The detective quantum efficiency is essentially governed by the demagnification of the fiberoptic taper since the light loss is proportional to the square of the ratio of the input and output size of taper. At high signal levels the DQE is also limited by the noise factor of the phosphor.