Two-dimensional imaging detectors for structural biology with X-ray lasers

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Our ability to harness the advances in microelectronics over the past decade(s) for X-ray detection has resulted in significant improvements in the state of the art. Biology with X-ray free-electron lasers present daunting detector challenges: all of the photons arrive at the same time, and individual high peak power pulses must be read out shot-by-shot. Direct X-ray detection in silicon pixel detectors—monolithic or hybrid—are the standard for XFELs today. For structural biology, improvements are needed for today’s 10–100 Hz XFELs, and further improvements are required for tomorrow’s 10+ kHz XFELs. This article will discuss detector challenges, why they arise and ways to overcome them, along with the current state of the art.

1. Introduction

X-ray free-electron lasers (XFELs) are a novel tool for structural biology, providing intense, coherent X-ray pulses of a sufficiently short duration that the X-rays scatter before the sample is able to appreciably move (‘diffract-before-destroy’). Making use of these new sources requires detectors capable of recording a two-dimensional scattering pattern shot-by-shot. Such a detector should consist of \( N^2 \) pixels, where each pixel records the number of X-rays per pixel shot-by-shot, \( n(x,y) \), with an accuracy of \( \Delta n(x,y) \). The key technical challenges are dynamic range—signal amplitude varies as the fourth power of scattering angle, readout rate—shot-by-shot readout is required for megapixel-scale detectors; along with microelectronics design, interconnect technology and development of sensor materials. The key practical challenge is the investment mis-match between producing, and seeing, X-rays.

This paper aims to briefly describe the detector challenges for biology with XFELs, as a basis for further discussion between users and providers of detectors. Section 1 introduces current detectors (all based on direct detection in silicon) and the significance of how the sensing element is connected to its readout. Section 2 discusses dynamic range, and §3 describes means of achieving a given dynamic range. Finally, §4 discusses options for pixel design, particularly in the light of single photon sensitivity.

(a) (Silicon) sensors and interconnect topology

Current (and proposed) XFEL detectors are based on direct X-ray detection in silicon: X-ray in/charge out. (Indirect detectors create charge through secondary mechanisms, e.g. scintillators, where the visible light is converted to charge.) Electrons produced when an X-ray photoconverts in a material will produce electron-hole (e-h) pairs by ionization. In a sufficiently depleted and properly biased semiconductor, these e-h pairs may then be collected. In silicon, \( \eta = 3.6 \text{ eV} \) is required on average to create an e-h pair, so that a 10 keV X-ray generates around 2800 e-h pairs, which is roughly 0.5 fC. Silicon is a nearly ideal semiconductor sensor for X-rays: a 300 \( \mu \text{m} \) thick sensor absorbs 98% of X-rays at 8 keV, approximately 95% at 10 keV and approximately 85% at 12.4 keV. (For harder X-rays, though, Si starts to become transparent, and this motivates developments of higher Z sensors, such as Ge, GaAs, CdTe/CZT, etc.)

Crudely, the ‘detector’ consists of an array of \( N^2 \) sensor elements, the corresponding electronic readout and a data acquisition system. (A real system is more involved, with mechanics, cooling software, etc.—and often these secondary
elements require more work than the ‘detector’ itself. The sensor array generally consists of a thick (typically 300 μm) sheet of p(n) type silicon, to collect electrons (holes), the back side of which is implanted with a continuous p(n) contact and the front side is patterned with M2 (M ≤ N) n(p) implants (and perhaps additional electronics). The silicon must be of sufficiently high resistivity so that a suitable bias voltage can deplete the thick bulk of free carriers. In this way, electric fields are created in the M2 p−n (n−p) diodes, and charge will drift to the nearest n(p) pixel electrode.

Interconnect topology (how the sensor is connected to the readout electronics) fundamentally drives ‘detector’ capabilities. Figure 1 illustrates the two typical topologies: one-dimensional connection at the periphery of the sensor and two-dimensional connection to individual diodes. The one-dimensional topology is used with monolithic detectors—those that combine the sensor and sufficient readout electronics in order to present the voltage output of each pixel at the periphery. In the two-dimensional topology, each pixel has its own dedicated readout. The most common one-dimensional device is the charge-coupled device (CCD), and CCDs of various forms are in use at all XFELs [1–3]. CCDs accumulate charge in each pixel, and (noiselessly) transport that charge from pixel-to-pixel. Generally, the charge is converted to a voltage through the gate capacitance of an output source follower transistors. CCDs are monolithic and can have a large number of pixels (of small dimension) on a single piece of silicon. In addition, CCDs can provide true correlated double sampling, so that low readout noise can be obtained. As the readout is inherently serial, parallelism is required for high-speed readout. Tens to 100s of megapixels s−1 have been obtained, and two to three orders of magnitude increase in speed may be possible. The practical speed limitation arises from the RC time constant of the CCD gates, which limits how quickly charge can be clocked from pixel-to-pixel. The physical speed limitation arises from the time needed for to transport the charge (with reasonable efficiency) from pixel-to-pixel.

Hybrid pixel detectors have been in development for 30 years and are now quite popular in synchrotron radiation research. Significant investment for large particle physics experiments has advanced the state of the art [4], and several groups worldwide have produced successful X-ray hybrid pixel detectors. As each pixel has its own dedicated readout, faster and more sophisticated readout is possible. To date, pixels are attached to mating readout chips using solder or indium bumps, and this sets a minimum pixel size (pitch) of 50–100 μm. Further, since the readout chip, discussed later, is typically limited to 2 cm in size, hybrid pixels are composed of ‘filed’ detector segments, with a certain amount of dead space between tiles. Although beyond the scope of this article, R&D on ‘3D interconnect’ [5] could enable an order of magnitude decrease in bonding pitch. Similarly, R&D in ‘edgeless’ sensors [6] could greatly reduce the dead space between tiles.

The high density of electronic readout required, necessitates the use of application-specific integrated circuits (ASICs). Invariably today, these ASICs are constructed in a CMOS (complementary metal-oxide-semiconductor) process, characterized by the feature size (the minimum transistor gate length which can be lithographically printed). In order to (try to) continue to follow Moore’s law, constant field scaling is used, so that the feature size determines most of the performance characteristics of the process: speed and noise performance depend on the process used. Further, in a two-dimensional detector topology (unlike the one-dimensional case) any X-rays which penetrate the sensor may result in ionizing radiation in the ASIC. Given the high peak power of XFEL pulses, and the less than 100% absorption of a silicon sensor at approximately 1 Å wavelengths, this imposes radiation hardness requirements on the readout ASIC, which can generally met by the proper process and design. (Of course, hard X-ray FELs can also perforate the detector—which, while not an electronics and sensor design problem per se, imposes constraints on the design of the detector system.)

(b) Dynamic range and noise
‘Dynamic range’ is a loosely defined term, which is used by the X-ray practitioner to indicate the maximum number of photons which a pixel can collect (with an implicit assumption of single photon sensitivity) and by the electronics designer to indicate the ratio of the maximum signal which can be stored to the noise floor. The linguistic difference to these two speakers is in the noise.

Counting detectors, popular at storage rings, amplify individual X-rays and increment in-pixel counters when the energy deposited is above a threshold. (Generally, the threshold is greater than or equal to 5σn, where σn is the RMS electronic noise.) For single-shot experiments, where all of the photons arrive simultaneously, counting is not possible.1 Integrating detectors, which integrate all charge received during a certain period, are thus the readout architecture for XFELs.

For a pixel, the readout electronics generally consists of amplification (with a conversion gain G [V/eV]—so that v X-rays of energy E incident on a given pixel result in a voltage

Figure 1. Examples of detector interconnect topology: (a) the sensor array is connected in one dimension to readout (e.g. CCDs). (b) The sensor is connected in two dimensions to readout (e.g. hybrid pixel detectors).
out of the amplifier of $V = vGE$ followed by quantization (via an analogue to digital convetor (ADC) that converts an input voltage into a (integer) number). Apart from the intrinsic statistical fluctuations in the number of X-rays $\nu(x,y)$ striking a pixel, the error $\delta \nu(x,y)$ depends on fluctuations in the charge collection process (statistics of the number of e-h pairs created and detected), the electronic noise and the quantization error.

For a (normalized) input to an n-bit ADC, the output is an integer $N(V) = \text{int}(2^n V)$. For an ideal ADC, $N(V) = N$ for any input voltage $N/2^n \leq V < (N + 1)/2^n$ (i.e. $N(V) = 1$ for $0 \leq V < 1/2^n$). The mean voltage with an ADC value $N$ is $V = (N + 1)/2^n$, and the statistical quantization error, the RMS uncertainty due to a range of voltages giving the same ADC result, is $1/2\sqrt{2^n}$. In units of X-rays, $v$, the measurement error is $\delta v = \sqrt{(\sigma_n/GE)^2 + (v_{\text{max}}/2\sqrt{2^n})^2}$, where $v_{\text{max}}$ is the maximum number of photons which can be recorded. For the X-ray practitioner, $v_{\text{max}}$ is the dynamic range, and for the electronics designer, $v_{\text{max}}/\delta v$ is the dynamic range.

(c) Readout architecture

For single photon detection, $\delta v \sim 1/m$ (approx. 1/5 in §2).

If $1/m = \sqrt{(1/m)^2 + (\sigma_n/GE)^2}$, then to cover the dynamic range linearly would require (at least) an $n = \log_2 v_{\text{max}} + \log_2 m \rightarrow \log_2 12$ bit ADC. Given that $v_{\text{max}}$ can be quite large for an XFEL, this presents a daunting challenge. Statistics of the illumination, however, are reflected in the detection: $\nu(x,y)$ has a Poisson distribution, so that $\delta \nu(x,y)$ need not be better than $~\sqrt{\nu}$—i.e. for $\nu(x,y) = 0$, one wants $\delta \nu(x,y) \sim \sim 1/m$, and for $\nu(x,y) = v_{\text{max}}$ one wants $\delta \nu(x,y) < v_{\text{max}}$. This problem, detecting a needle in a detectable haystack has presented itself in many different fields, and there are three basic approaches:

1. fully linear—an n-bit front-end and ADC,
2. piecewise linear (or floating point) front-end and n'-bit ADC, and
3. nonlinear front-end and n'-bit ADC.

Figure 2 illustrates how these different approaches perform: here, we consider the case for $v_{\text{max}} = 10^5$ X-rays ($\log_2(10^5/\sqrt{12})v_{\text{max}} = 17.1$). Curve (a) shows the required fractional resolution in units of X-rays, i.e. the maximum value for $\delta \nu$ for a given value of $\nu$. Curve (b) shows $\delta v$ versus $\nu$ for an ideal, fully linear, 18-bit ADC (18 > 17.1). For a fully linear system, the bit size is determined by $m$ (the desired signal-to-noise ratio for one X-ray). Consequently, at full scale, the fractional resolution is approx. 3000 times better than required.

Curves (c)–(e) show how a multi-gain or floating-point system works. A multi-gain approach might have three separate amplifiers with gains, 64 (c), 8 (d) and 1 (e) and selects the highest non-saturated gain to digitize. With a 12-bit ADC, a 12 + $\log_2 64 = 18$ bit range can then be covered. In a floating-point approach, a single amplifier switches gains as a function of input signal to achieve the same functionality.2

Ideally, (nonlinear) amplifier, for which $\delta \nu$ versus $\nu$ could be arranged to be precisely the desired value, would be the most efficient system, as the ADC requirements would then be minimized. For various applications, logarithmic amplifiers, or those based on the square-law response of field effect transistors have been used. Curve (f) shows a logarithmic nonlinear front-end with a 12-bit ADC. In general, the challenge for nonlinear amplifiers is calibration and stability. Often the nonlinear element is temperature sensitive, and for a megapixel detector, going from $10^6$ to $10^{10}$ X-ray calibration constants can be difficult.

(d) Pixel and single photon considerations

Figure 3 schematically illustrates a pixel with its readout (shown here as a floating-point or piecewise linear readout; (as mentioned earlier, screening effects [7] may perhaps slow the development of the input voltage, which (S. Gruner, private communication) may be used advantageously). $C_{\text{DET}} = \varepsilon_0 \varepsilon_{\text{DET}} A/t$ for a pixel of area $A$, thickness $t$ and dielectric constant $\varepsilon_{\text{DET}}$, so that $v_{\text{max}} = (\varepsilon_0 \varepsilon_{\text{DET}} A/t)/e \sim 10^2 A/t$ for approximately $A$ wavelength X-rays (in a silicon detector). As $t \sim 10^2 \mu m$ for efficient absorption of approximately $A$ X-rays, it is hard to surpass approximately $10^4$ X-rays in a pixel of area ($10^2 \mu m)^2$. For a bump-bonded hybrid pixel detector, pixels less than $O(10^2 \mu m)$ are difficult to realize, owing to the limitations of bump-bonding technology (going below the 200 µm industry-standard pitch was a major development in high-energy physics for the Large Hadron Collider pixel detectors).

The input amplifier moves the charge from the pixel capacitance, $C_{\text{DET}}$, on to the feedback capacitance, $C_F$. The feedback capacitance determines the conversion gain, $G = \varepsilon/\varepsilon_{\text{F}}$ (multi-gain and floating-point readouts can have multiple, signal size-dependent, values of $C_F$). For typical CMOS processes, $C_F \sim O(BF/\mu m^2)$, and each X-ray is approximately 0.5 fC, so that for 1V full scale, a 1 µm$^2$ capacitor can store a few X-rays. Large numbers of photons per pixel thus...
require large pixels, or charge-division schemes (so that less than 0.5 fC/X-ray need be stored).

Pixel size not only plays a role in maximum signal, but also in single photon performance. For ideal, Gaussian noise (rarely achieved on the experimental floor) $m = 5$ implies that the probability that an empty pixel will falsely indicate a single photon is approximately $3 \times 10^{-6}$. With a large number of pixels, and a large number of shots, however, even this small number suggests many false ‘hit’ pixels. In photon counting applications (at storage rings, §2), pixels that are much larger than the point spread function (PSF) of an X-ray simplify the detector design (the larger the pixel, the fewer X-rays shared between pixels). For integrating applications, though, smaller pixels are advantageous, as they allow sub-pixel spatial resolution. Figure 4 sketches a single X-ray as seen by large and small pixels. For large pixels, all of the charge is most likely
in only one pixel. For small pixels, charge is shared—so that a single photon deposits charge in more than one pixel, giving an additional constraint. To make use of this constraint requires noise performance better than $n = 5$, but since $\text{noise} \propto C_{\text{DET}} \propto A$, this constraint is easier to meet with a small pixel than a large pixel.

2. Conclusion

The past decade has delivered impressive growth in X-ray detection capabilities for synchrotron radiation research. These new detectors are derivative of (significant) investments in high-energy physics and astrophysics. Truly optimized next-generation XFEL detectors will demand dedicated and coordinated development (which can only happen with the right infrastructure, see [8]), requiring, potentially, greater than or equal to 1% of the cost of the next-generation XFEL per XFEL detector. Modern CMOS processes offer very high performance, but the X-ray detector community is typically a decade behind the state of the art, owing to the high cost of entry (costs are roughly exponential with decreasing feature size, as is design complexity, and the cost of design tools). While many new XFEL detectors are underway [9] given the 5–10 year detector development cycle time, it is not too early to start planning next-generation detectors.

Endnotes

1Simulations and experiments [7] suggest that for large charge deposition, plasma effects modify the local collection field, and that charge collection could be slowed down. This could potentially enable ‘counting-inspired’ detectors for XFELs [8].
2As an example, the five initial detectors for the European XFEL [9] use most of these schemes: AGIPD, FCCD have floating-point readout, LPD, pnCCD have multi-gain readout and DSSC has nonlinear readout.

References