High-speed detector for time-resolved diffraction studies

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Abstract. There are a growing number of high brightness synchrotron sources that require high-frame-rate detectors to provide the time-scales required for performing time-resolved diffraction experiments. We report on the development of a very high frame rate CMOS X-ray detector for time-resolved muscle diffraction and time-resolved solution scattering experiments. The detector is based on a low-afterglow scintillator, provides a megapixel resolution with frame rates of up to 120,000 frames per second, an effective pixel size of 64 μm, and can be adapted for various X-ray energies. The paper describes the detector design and initial results of time-resolved diffraction experiments on a synchrotron beamline.

1. Introduction
Demand is increasing for high-frame-rate detectors to meet the growing needs of various time-resolved synchrotron applications that require low-noise, high-sensitivity detectors capable of acquiring X-ray diffraction imaging data at 1,000 frames per second (fps) or faster. While CCDs offer low-noise performance, they lack high-speed capability beyond a few tens of frames per second. CMOS sensors offer parallel high-speed readouts, making it possible to design high frame rate detectors, but detector noise has so far been a hindrance in using them for low-noise applications. Recent developments in scientific grade CMOS sensor technology have addressed the detector noise and related limitations, resulting in a growing interest in using CMOS sensors for time-resolved synchrotron applications. Furthermore, high frame rate imaging is inherently light-starved due to the short exposures needed to avoid motion blur. This imposes stringent requirements on the performance of the individual system components, such as the X-ray-to-light converter, imaging optics and readout. Each of these system components must be designed to maximize the signal-to-noise ratio, and to improve image resolution and contrast.

This paper briefly describes the detector design, and discusses the results of time-resolved muscle diffraction experiments performed at the BioCAT 18-ID beamline at the Advanced Photon Source (APS) at Argonne National Laboratory (Argonne, IL).

2. Time-resolved small-angle scattering detector requirements
The time scales of interest in muscle and small- and wide-angle scattering experiments range from 1 to 5 ms [1],[2], with some requiring resolutions in the hundreds of microseconds [3]. Small-angle scattering and diffraction features are generally weak, diffuse, and hard to distinguish from background. Thus, in addition to high speed, an ideal scattering detector should have high sensitivity,
with single X-ray counting capability (at 12 keV), good dynamic range (4,000 or more X-ray photon equivalents per pixel), and high spatial resolution (point spread functions of ~50-100 µm FWHM) with low noise, and should permit the readout of at least 1k×1k pixels at 1,000 fps (or faster).

3. The detector design

Our detector consists of a high-performance, low-cost film of a patented microcolumnar CsI:Tl,Sm scintillator developed at RMD [4], coupled to a high frame rate FASTCAM Ultima APX-i2 CMOS imager from Photron (San Diego, CA) [5] via a 25 mm diameter single stage MCP125 image intensifier (II) from Photek (East Sussex, UK). The schematic of the detector is shown in Figure 1(a), and a photograph of the detector head is shown in Figure 1(b). While Matsuo et al [6] have reported using an intensified CMOS detector at SPring-8 synchrotron facility with effective pixel size of 100 µm [7], large area (6" diameter) image intensifiers of the type they used are no longer commercially available. The current detector design has an effective pixel size of 64 µm, and also delivers higher frame rates that are important for time-resolved synchrotron applications. Besides, unlike the powdered Gd2O2S screen used by Matsuo [6], our detector is based on a 7 cm × 7 cm film of a high-resolution, low-afterglow, bright microcolumnar CsI:Tl,Sm scintillator coupled to a fiberoptic taper, resulting in the higher sensitivity needed for time-resolved experiments. Co-doped CsI:Tl,Sm is a newly developed scintillator from RMD that reduces the afterglow and hysteresis associated with conventional CsI:Tl by several orders of magnitude [8]-[10] without sacrificing its excellent scintillation properties, thereby making it a material well suited for high-speed, high-resolution imaging [11],[12]. The microcolumnar nature of the screen suppresses lateral light spread in the scintillator, resulting in a high spatial resolution, even when the scintillator is thick to increase absorption of the incident X-ray flux, offering the flexibility to tailor the scintillator thickness for a particular X-ray energy. For example, using a 150 µm thick CsI:Tl,Sm film stopping over 99% of the 12 keV X-rays used in our experiments, we had earlier demonstrated over 16 lp/mm of spatial resolution [11],[12] and over 275 fps imaging of small-angle X-ray scattering at a synchrotron beamline [13]. In this paper, we have demonstrated the suitability of CsI:Tl,Sm for up to 2,000 fps imaging, which is also the frame rate of interest for our time-resolved scattering experiments.

Table 1. Specifications of the detector.

| Parameter                              | Specification                       |
|----------------------------------------|-------------------------------------|
| Image area                             | 7 cm × 7 cm                         |
| Pixel resolution                       | 1024 (H) × 1024 (V) pixels          |
| Effective pixel resolution with 3.78:1 FO taper | 64 µm (H) × 64 µm (V)               |
| Frame rate at full pixel resolution    | 2,000 fps                           |
| Frame rate in a selected region-of-interest | 120,000 fps                        |
| Sensitivity for 12 keV photons         | > 99%                               |
| SNR for 12 keV photon (image intensifier gain of 250) | 5.76                              |
An optical image of the USAF 1951 resolution target demonstrating the high resolution of the detector is shown in Figure 1(c). The technical specifications of the detector are shown in Table 1. An important feature of the detector is that it can operate at up to 2,000 fps with full 1024×1024 pixel resolution and up to 120,000 fps with a reduced imaging area, without any binning.

4. Dynamic radiographic imaging
To test the effectiveness of the reduced afterglow CsI:Tl,Sm scintillator for dynamic imaging, a 50 µm thick film was integrated into the CMOS detector and X-ray images of a steel nut held by a mechanical wrench were acquired using a 300 kVp pulsed X-ray source with a pulse duration of 20 ns. The camera was operated at 1,000 fps and the object was exposed to X-rays only during the first integration period, with the subsequent frames acquiring only the residual image provided by the afterglow. For comparison we also evaluated our commercial CsI:Tl screen, whose thickness was ~150 µm. The resulting data for a conventional CsI:Tl screen and the new CsI:Tl,Sm film are shown in Figure 2. As can be seen from these figures, with conventional CsI:Tl a residual image is visible even after 30 frames, corresponding to a time interval of about 30 ms. In contrast, co-doped CsI:Tl,Sm shows virtually no residual image after only the second frame, corresponding to a 2 ms interval.

5. Time-resolved diffraction experiments at BioCAT beamline
Time-resolved muscle diffraction experiments were performed using the standard SAXS instrument on the BioCAT beamline 18-ID at the APS using 0.103 nm (12 keV) X-rays. The detector was mounted on the beamline (Figure 3(a)) and diffraction patterns were acquired for different detector gain settings and at various frame rates, ranging from 125 fps to 2,000 fps. As described earlier, the detector can operate at up to 2,000 fps with full resolution. A single frame of 500 µs duration (corresponding to 2,000 fps) showing diffraction from an insect flight muscle sample is shown in Figure 3(b). This clearly demonstrates the high sensitivity, even at such a high frame rate of operation.

The performance of the detector was also compared to that of the PILATUS 100K detector Dectris [14], and the graph comparing the averaged intensities per millisecond from the two detectors is shown in Figure 3(c), indicating that the CMOS detector is almost 1,000 times more sensitive. This high sensitivity is due to the high gain (~10,000) provided by the image intensifier, but at the expense of reduced dynamic range. However, these are preliminary results, and further evaluations are planned at the BioCAT beamline in the next few weeks.
6. Discussion
We have developed a high-frame-rate CMOS detector for time-resolved diffraction experiments using a high-resolution commercial sensor and a microcolumnar co-doped CsI:Tl,Sm scintillator developed at RMD. Solution scattering and time-resolved muscle diffraction experiments were performed at the BioCAT beamline at up to 2,000 fps (500 µs time resolution) to demonstrate its suitability for time-resolved applications. While these experiments clearly demonstrate the sensitivity and speed of the detector, further tests are planned to calibrate its linearity and response uniformity using a series of calibrated flood field exposures. Spatial distortions will be corrected using images of pinhole masks.

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Figure 3. (a) The experimental setup at the BioCAT beamline. (b) A single 500 µs frame of time-resolved diffraction from an insect flight muscle. (c) Comparison of averaged intensity per millisecond using the CMOS and DECTRIS PILATUS 100K detectors.
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