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CMOS flatpanel detectors for SAXS/WAXS experiments

Naoto Yagi* and Katsuaki Inoue

SPring-8/JASRI, Japan. Correspondence e-mail: yagi@spring8.or.jp

A CMOS (complementary metal-oxide semiconductor) flatpanel X-ray detector is a two-dimensional silicon image sensor with a scintillator. We have tested the performance of two types of CMOS detectors in simultaneous small-angle/wide-angle X-ray scattering experiments. Both are active-pixel devices that have an amplifier in each pixel. Wide-angle patterns were recorded with the detector just behind the specimen and very close to the beam. The quality of the images shows that these detectors are suitable for this purpose.

1. Introduction

CMOS (complementary metal-oxide semiconductor) flatpanel X-ray detectors have been commercially available for some years. In this type of detectors, X-rays are converted by a phosphor to visible light and a CMOS photodiode-array sensor records the image. Two manufacturers are currently producing them: Rad-icon Imaging Corp. (Santa Clara, California, USA) and Hamamatsu Photonics K. K. (Hamamatsu, Japan). Specifications of the detectors from these companies are very similar: each pixel is a 50 µm square (a recent product from Rad-icon has 22.5 µm pixels), dynamic range 2000–4000, frame rate 2–3 s^{-1}. Rad-icon produce only 'active-pixel' sensors in which each pixel has a pre-amplifier, while Hamamatsu Photonics produce both 'active-pixel' and 'passive-pixel' sensors. Detectors from Rad-icon employ tiling of 25 mm×50 mm sensor segments, while Hamamatsu Photonics make sensors up to 220 mm×176 mm in size (Fujita *et al.*, 2003). Rad-icon use medical scintillator films as a phosphor, while Hamamatsu Photonics use needle-shaped crystals of CsI (150 µm) directly deposited on the sensor. Depending on the size of the detector, the price ranges from 1000 to 8000 USD.

We tested these detectors in several different synchrotron radiation applications (Yagi *et al.*, 2004*a*,*b*), including imaging, diffraction and scattering experiments. For these experiments, an active-pixel detector is preferable because of its lower readout noise. However, even with a passive-pixel device, which has a higher noise level, an $R_{\rm merge}$ value of 6% was obtained in the processing of diffraction from a lysozyme crystal (Yagi *et al.*, 2004*b*). In this report, active-pixel flatpanel detectors from the two manufacturers were tested for simultaneous small- and wide-angle diffraction/scattering (SAXS/WAXS) experiments, which are increasingly becoming common to investigate the hierarchical structure of soft materials using synchrotron radiation.

2. Methods

X-ray experiments were made at BL40XU in SPring-8 (Inoue *et al.*, 2001). The X-ray energy was either 12.4 or 15.0 keV with a bandwidth of about 3%. The X-ray intensity was adjusted by an aluminium absorber. Some measurements were also made at BL40B2 which has a double-crystal silicon monochromator. For SAXS recordings, a beryllium-windowed X-ray image intensifier (V4554P, Hamamatsu Photonics; Amemiya *et al.*, 1995) was used with a slow-scan cooled CCD (charge-coupled device) camera (C4880-50-24A, Hamamatsu Photonics). Also, an image plate (BAS-SR, Fujifilm, Tokyo, Japan) was used, which was scanned by a BAS2500 scanner (Fujifilm) with a step size of 50 µm. The specimen-to-detector distance was about 3 m. Most of the beam path was evacuated. For WAXS recordings, a CMOS flatpanel detector from Hamamatsu Photonics (C9728DK) or Rad-icon (Shad-o-Box1024) was used.

3. Results and discussion

3.1. Hamamatsu Photonics C9728DK

C9728DK from Hamamatsu Photonics is an active-pixel detector with 1056×1056 pixels (52.8 mm × 52.8 mm). The output is in 14-bits. The full well depth is about 1 000 000 electrons. Thus, each ADU (analogue-to-digital conversion unit) corresponds to about 60 electrons. Readout noise was estimated by taking two blank exposures and calculating variation of pixel values in their difference pattern. The standard deviation was divided by $\sqrt{2}$ to give a root-mean-squared readout noise, which was found to be 4.5 ADU, equivalent to 267 electrons. In another measurement, 200 successive frames were recorded and variation in an ADC value of one pixel was obtained, which was found to be equivalent to 283 electrons. Since the readout noise of the passive-pixel detector C7942 was 1670 electrons (Yagi *et al.*, 2004*a*), it was about six times lower in the active-pixel detector C9728DK. The conversion gain was 170 electrons per incident X-ray photon at 12.4 keV. This is slightly lower than the value obtained with the passive-pixel detector C7942 (Yagi *et al.*, 2004*a*), probably because of the smaller photosensitive area in each pixel due to more complicated electronics in the active-pixel device: part of the light created by an interaction of an X-ray photon with the phosphor was blocked by the electronics and did not reach the photodiode.

To test the noise level in an actual diffraction experiment, a small-angle diffraction pattern was recorded with three detectors: C9728DK, an X-ray image intensifier coupled with a cooled CCD camera, and an image plate. The exposure time was 0.1 s with all detectors. The pixel size of C9728DK and the image plate was the same (50 µm × 50 µm), and that of the X-ray image intensifier was larger (150 µm × 150 µm). Among the images given by these detectors (Fig. 1), the one by the image intensifier looks the smoothest, but this is due to the lower resolution of the detector. It is clear that C9728DK has a sensitivity similar to those of the other two detectors.

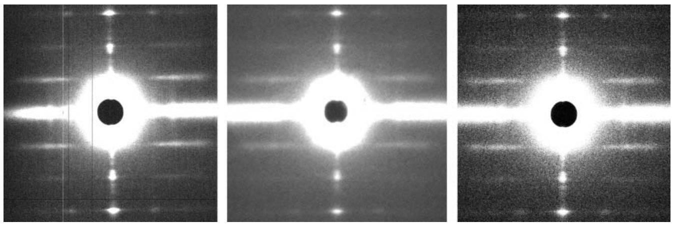

Figure 1
SAXS patterns from a frog sartorius muscle in a Ringer's solution, taken with C9728DK (left), an X-ray image intensifier with a cooled CCD camera (centre), and an image plate (right). These were recorded with the same specimen-to-detector distance and exposure time (0.1 s). Dark image (a pattern obtained in an exposure without X-rays) was subtracted from the pattern taken with C9728DK. The X-ray energy was 12.4 keV. A few detect lines are visible in the image taken by C9728DK.

In order to compare the data quality, linear intensity profiles of meridional diffraction from chicken leg tendon collagen were recorded with C9728DK and an image plate (Fig. 2). The two profiles are very similar, showing that the quality of images recorded by C9728DK is comparable to that of images recorded by an image plate. The spatial resolution is slightly better in an image plate.

The linearity of sensitivity was checked by using monochromatic X-rays with an energy of 12.4 keV at BL40B2. Until the pixel value reached its 14-bit maximum, no sign of non-linearity was observed with an increase in the exposure time. This is in contrast to the active-pixel detector from Rad-icon which has a non-linear response (Yagi *et al.*, 2004*b*).

The dark current was about 141 ADU/s, which corresponds to 8,500 electrons s^{-1}. This is higher than that observed in the passive-pixel detector C7942 (3900 electrons s^{-1}, Yagi *et al.*, 2004*a*). The spatial resolution of C9728DK has not been measured. However, since the design of the phosphor and the pixel size are the same as those of C7942, the resolution is supposed to be similar, 100–150 μm (Yagi *et al.*, 2004*a*).

An active-pixel CMOS image sensor is known to be susceptible to radiation damage by high-energy (> 20 keV) X-rays. Permanent damage was observed in the Rad-icon detector (Yagi *et al.*, 2004*b*). The phosphor (CsI) of C9728DK, which is 150 μm thick, is expected to absorb 90%, 98%, 100% of X-rays with an energy of 15.0, 12.4, 8.0 keV, respectively, assuming a packing ratio of 0.6. The photons that pass through the phosphor may damage the sensor and give rise to shot noises. Although we have not seen any sign of damage in these SAXS/WAXS experiments, and Fig. 1 (left), which was taken with an X-ray energy of 12.4 keV, does not show shot noises, care must be taken when the detector is used at higher energies. In fact, although no damage was seen with a total dose of 300 Gy at 10 keV, we observed a permanent increase of the dark current with 4 Gy at 30 keV (data not shown).

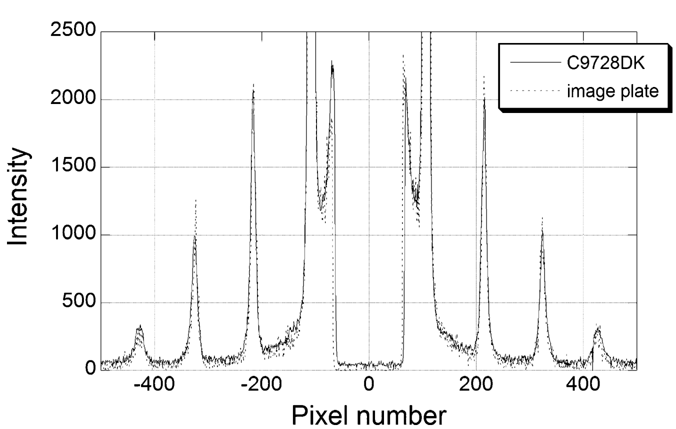

Figure 2
Meridional intensity profiles from chicken leg tendon collagen. The innermost peaks, just outside of the edges of the beam stop, are the first order of the 630 Å repeat along collagen fibrils. The continuous line was recorded by C9728DK and the dotted line by an image plate. The X-ray energy was 12.4 keV. The pixel size was 50 μm and the exposure time 0.3 s for both detectors. The intensity value of C9728DK was multiplied by 3 for comparison.

C9728DK is very compact in size: it is only 14.5 mm thick and weighs only 1.6 kg. Thus, this detector is useful for recording the wide-angle diffraction in a small-angle diffraction experiment. Since its sensor is very close (1.8 mm) to an edge of the detector housing, it is feasible to align the detector so that the edge of the sensor lies close to the main beam (Fig. 3). Fig. 4 shows SAXS and WAXS diffraction patterns from a dried chicken tendon. The WAXS pattern was recorded with C9728DK with an exposure time of 0.4 s.

3.2. Rad-icon Shad-o-Box1024

Shad-o-Box1024 from Rad-icon consists of two sensors (Rad-Eye) with 512×1024 pixels (Yagi *et al.*, 2004*b*) which are aligned side-by-side to form a detector with 1024×1024 pixels. Its characteristics are similar to those of C9728DK except its non-linear response. Since the phosphor is a medical scintillator film (Kodak Min-R) which is too thick for low energy X-rays, it is suitable for experiments using an X-

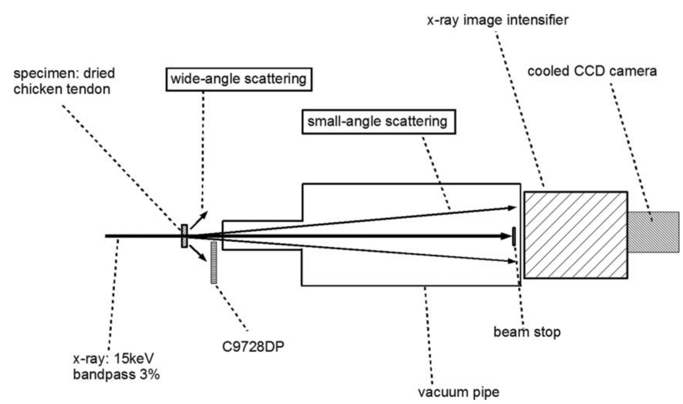

Figure 3
Schematic diagram of the experimental setup for simultaneous SAXS/WAXS measurement using C9728DK.

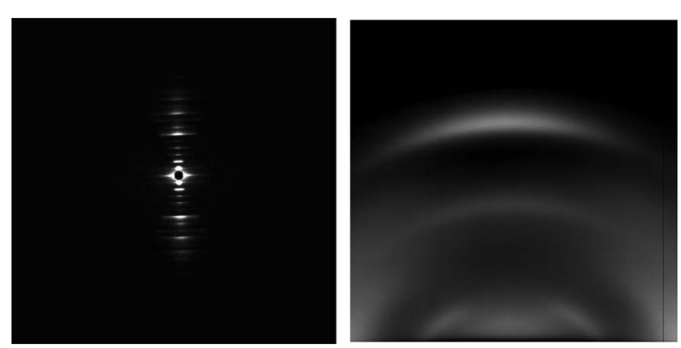

Figure 4
Diffraction patterns from a dried chicken tendon. Left is a SAXS pattern recorded with an X-ray image intensifier (exposure time 60 ms). Orders of the 630 Å axial periodicity of collagen are seen. Right is a WAXS pattern recorded with C9728DK (exposure 0.4 s). The strong arc is the meridional peak at a Bragg spacing of 2.9 Å. The fibre axis of the specimen was vertical. The X-ray energy was 15.0 keV.

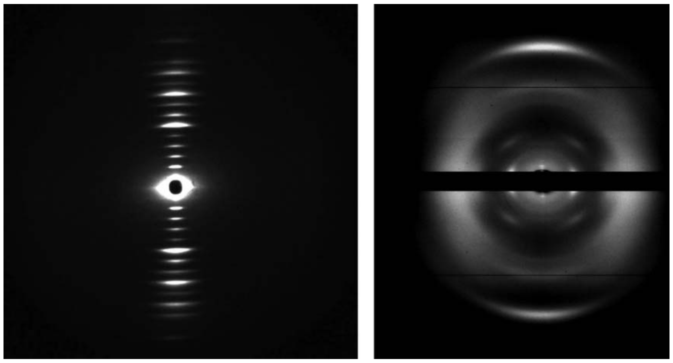

Figure 5
SAXS (left) and WAXS (right) diffraction patterns from a dried chicken tendon. The fibre axis was vertical. The SAXS pattern was recorded with an image intensifier coupled to a cooled CCD camera. The exposure time was 1.0 s. The WAXS pattern was recorded with Shad-o-Box1024 with a 3 mm separation between the two sensors. The exposure time was 6.7 s. The X-ray energy was 15.0 keV.

ray energy higher than 15.0 keV. The tiling was made use of for a WAXS detector in a SAXS/WAXS experiment. The detector was modified to pass X-rays between the two sensors, which were shifted to be separated by about 3 mm. A pipe was placed between the sensors to pass X-rays. With this detector, it is possible to record a two-dimensional WAXS pattern while another detector at the downstream end of the camera records a SAXS pattern, in a similar manner as in Fig. 2.

Fig. 5 shows SAXS and WAXS patterns from collagen (dried chicken tendon) with an X-ray energy of 15 keV. The SAXS pattern was recorded with an image intensifier coupled to a cooled CCD camera. The reflections are orders of the 630 Å periodicity of collagen. Orders up to the 15th are observed. Orders higher than the 15th are not seen because the pipe in Shad-o-Box1024, which has a diameter of 2.8 mm, blocked them from reaching the image intensifier. Thus, q = 0.01–0.15 Å $^{-1}$ ($q = 4\pi\sin\theta/\lambda$, where 2θ is the scattering angle and λ is the wavelength) is covered by the SAXS detector.

The WAXS pattern was recorded by the modified Shad-o-Box1024. The exposure time was 6.7 s. The horizontal gap in the centre, which was artificially inserted after data collection, corresponds to the 3 mm gap between the two sensors. The dark, insensitive lines at the middle of the upper and lower halves are intrinsic to Rad-Eye. The strong meridional arc is at a Bragg spacing (d) of 2.9 Å. At this distance from the specimen, this detector should cover d = 35.3–1.96 Å, that is, q = 0.18–3.2 Å $^{-1}$.

4. Conclusions

Unlike most CCD-based sensors, CMOS flatpanel detectors are currently used without cooling. Since the dark current is high at room temperature, they are most suitable for synchrotron radiation experiments in which exposure time is shorter than a few seconds. In fact, the quality of images is comparable to those obtained by other detectors (Figs. 1 and 2). In this report, the detectors were tested in a simultaneous SAXS/WAXS measurement in synchrotron radiation experiments. The compact size of the detectors makes them suitable for this purpose. C7940DK has already been used as a WAXS detector in SAXS experiments on synthetic polymers by the group of Dr Y. Amemiya (the University of Tokyo). A larger active-pixel detector (C10158DK from Hamamatsu Photonics), which has 2376 × 2376 50μm-square pixels, is now available. However, although it is more suitable for most diffraction experiments than C7940DK, it may not be suitable for the SAXS/WAXS measurement, since its sensor is 13 mm away from an edge of the detector housing.

The experiment was performed under approval of the SPring-8 Proposal Review Committee (2004 A0090-NL3-np).

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