Using the MAXIPIX detector for coherent X-ray scattering applications

A Schavkan\textsuperscript{1}, F Westermeier\textsuperscript{1}, A Zozulya\textsuperscript{1}, S Bondarenko\textsuperscript{1}, G Gr"ubel\textsuperscript{1}, C Schroer\textsuperscript{2} and M Sprung\textsuperscript{1}

\textsuperscript{1} HASYLAB, Deutsches Elektronen-Synchrotron, Hamburg, Germany
\textsuperscript{2} Institute of Structural Physics, Technische Universit"at Dresden, Dresden, Germany

E-mail: alexander.schavkan@desy.de, fabian.westermeier@desy.de

Abstract. Possibilities to use the MAXIPIX 2\times2 detector for coherent scattering applications are investigated. This study focuses on X-ray Photon Correlation Spectroscopy experiments. Flatfield measurements show that the MAXIPIX detector pixels respond very inhomogeneously in the X-ray range between 5 and 10\,keV most commonly used for coherent scattering experiments. Two colloidal model sample systems are investigated using the MAXIPIX and a direct illumination CCD for comparison. It is demonstrated that the MAXIPIX 2\times2 detector is applicable for static and dynamic coherent scattering experiments after flatfield correction.

1. Introduction

An interference ('speckle') pattern is produced by coherent X-rays scattered from a disordered sample. In a static coherent scattering experiment, the detector system must only be able to spatially resolve individual speckles. However, dynamic processes in the sample system lead to changes of the speckle pattern. This creates an additional requirement for the detector system in order to perform X-ray Photon Correlation Spectroscopy (XPCS) experiments. The detector system must be able to capture speckle patterns faster than the dynamical processes of the investigated sample system change it. While conventional 2D X-ray detectors have often frame-rates of seconds, photon counting area detectors are able to collect data with frame-rates of a few hundred hertz, making it possible to investigate dynamical processes in the sub-second time regime. The 2D Pixel Array Detector (PAD) MAXIPIX 2\times2 is such a photon counting detector system with the potential to extend the accessible time window for XPCS measurements.

The MAXIPIX \cite{1,2} was designed by the ESRF based on the MEDIPIX2/TIMEPIX \cite{3,4} readout Application Specific Integrated Circuits (ASICs) developed by CERN and the MEDIPIX2 collaboration \cite{5}. Among positive properties (e.g. fast read-out and high quantum efficiency), there are important disadvantages. Like all PAD detectors, the MAXIPIX has a relatively large pixel size of 55\times55\,\mu m^2. Also, the thresholding of the MAXIPIX is limited to a minimum X-ray energy of 5\,keV resulting in an optimum working energy range of 10 – 20\,keV. This is outside of the standard X-ray energies for coherent scattering experiments of 5 – 10\,keV.

The purpose of the experiments described in this paper was to test the applicability of the MAXIPIX for XPCS experiments in the range of 5–10\,keV. In the first part the flatfield response of the detector pixels is examined. In the second and third part the MAXIPIX performance is tested in static and dynamic coherent scattering experiments. The experimental results of the
2. Beamline set-up
The experiments were performed in the second experimental hutch of the Coherence Beamline P10 at PETRA III. The two detectors, MAXIPIX and LCX, were placed on the same translation stage at a distance of \( R_{\text{sample-det}} \approx 5.06 \text{ m} \) downstream of the sample position. The static and dynamic experiments were carried out in Small Angle X-ray Scattering (SAXS) geometry at a photon energy \( E_{\text{ph}} = 8 \text{ keV} \). The beam was collimated by a set of slits placed \( R_{\text{slit-sample}} = 820 \text{ mm} \) upstream of the sample, while another pair of slits was placed \( R_{\text{guardslit}} = 285 \text{ mm} \) upstream of the sample. The SAXS patterns of the samples were evaluated using the program \( \text{XPCSgui} \). The program calculates \( I(Q) \) as well as \( g^{(2)}(Q,t) \)-functions.

3. Flatfield measurements
To investigate the homogeneity of the pixel response of the MAXIPIX, flatfield images were measured at different X-ray energies. The detector was positioned at a 90\(^\circ\)-angle respective to the incident beam to minimize the scattering background of the incident beam compared to the fluorescence signal. Three foils of Cu, Se and Zr were used to investigate the flatfield response at different energies using X-ray fluorescence. To excite the fluorescence radiation, the photon energy for the Cu, Se and Zn measurement was set to \( E_{\text{Cu photon}}^\text{Cu} = 9 \text{ keV}, E_{\text{Se photon}}^\text{Se} = 13.2 \text{ keV} \) and \( E_{\text{Zn photon}}^\text{Zn} = 19.2 \text{ keV} \), slightly higher than the corresponding absorption edges of Cu, Se and Zn at \( E_{\text{Cu abs}}^\text{Cu} = 8.98 \text{ keV}, E_{\text{Se abs}}^\text{Se} = 12.66 \text{ keV} \) and \( E_{\text{Zn abs}}^\text{Zn} = 17.99 \text{ keV} \). The fluorescence emission lines for Cu, Se and Zn of strongest intensity are at \( E_{\text{Cu em}}^\text{Cu} = 8.05 \text{ keV}, E_{\text{Se em}}^\text{Se} = 11.22 \text{ keV} \) and \( E_{\text{Zn em}}^\text{Zn} = 15.78 \text{ keV} \). The flatfield was recorded for all four quadrants of the detector chip. To normalize the flatfield, first hot and cold pixels were identified and masked out if they showed less than 0.3 or more than 3.0 counts than the mean counts in the quadrant with the least deviations. The flatfield was normalized by the mean intensity value of the most homogeneous quadrant (visual estimation). Figure 1 shows the normalized detector response at the three energies in an identical region of interest (ROI) of 21 × 21 pixels of the detector. The response of the MAXIPIX pixels is clearly not homogeneous. Comparing the scale bars of the normalized images it is obvious that the pixel response becomes more inhomogeneous with decreasing energy. At \( E_{\text{em}}^\text{Cu} = 8.047 \text{ keV} \) (see fig. 1 (a)) the response is varying by more than 50\%, even in neighboring pixels. However, it seems that the response of every pixel is similar. To illustrate this, two example areas of similar response are marked in fig. 1 by black ellipses. The response of every single pixel scales...
The contrast of autocorrelation functions $g^{(2)}$ from SAXS diffraction patterns of a static colloidal sample consisting of dried spherical particles with a radius $R \approx 60$ nm was evaluated as a function of beam defining slit size. Data sets were taken under identical conditions with the LCX and the MAXIPIX detector for comparison. The beam defining slit size was varied between $5 \times 5 \mu m^2$ and $100 \times 100 \mu m^2$ and autocorrelation functions $g^{(2)}$ were calculated for 18 $Q$ values in the range from $0.0014 \text{Å}^{-1}$ to $0.012 \text{Å}^{-1}$. The auto-correlation functions were constant as the static sample exhibited no dynamics. The mean of the auto-correlation functions as a function of delay time was determined to calculate the contrast as $\beta = g^{(2)} - 1$. The resulting contrast values are plotted as a function of the beam defining slit size in figure 2. The LCX shows higher contrast levels than the MAXIPIX due to the smaller pixel size ($20 \times 20 \mu m^2$ instead of $55 \times 55 \mu m^2$).

The contrast $\beta$ can be modeled as $\beta = 1/(1 + P_{eff}^2/S^2)$, where $P_{eff}$ is an effective pixel size (accounting for charge sharing and other effects) and $S$ is the speckle size approximated by $S = \lambda \times R_{det}/d_{illu}$. Here, $\lambda$ is the X-ray wavelength and $R_{det}$ is the sample to detector distance. The size of the illuminated spot on the sample, $d_{illu}$, is given by $d_{illu} = d_{slit} + A \times \lambda \times R_{slit}/d_{slit}$, where $d_{slit}$ is the beam defining slit size, $R_{slit}$ is the slit to sample distance and $A$ is a fitting parameter. The model formula was obtained using a Gaussian approximation from contrast calculations [6, 7, 8]. Both contrast measurements can be explained using $A = 1.22$ (airy disk prefactor) and effective pixel sizes of $P_{LCX} \approx 35 \times 35 \mu m^2$ and $P_{MAX} \approx 40 \times 40 \mu m^2$. The effective pixel sizes of the MAXIPIX and the LCX detectors can be explained by a pixel size reduction due to a high threshold of 5 keV for the MAXIPIX and a pixel size increase due to charge spread in the deep depletion silicon layer of the small pixels for the LCX.

5. Dynamic experiments

A solution of colloidal tracer particles suspended in the molecular glass-forming solvent polypropylene glycol (PPG4000) was used for the XPCS experiments. Spherical SiO$_2$ particles with a radius of $R = 28 \pm 0.6$ nm were covered with 3-(trimethoxysilyl)-propyl methacrylate to yield a hard-sphere like particle interaction behavior. The volume fraction of the sample system was 0.06. A beam defining slit size of $20 \times 20 \mu m^2$ was used resulting in average contrast values of $\beta \approx 0.33$ for the MAXIPIX and $\beta \approx 0.42$ for the LCX detector. The results using the two different detector systems were compared for a temperature of $T = 250$ K.

To avoid beam damage of the sample, the total exposure time at one position on the sample was limited to a maximum of 100 s. The LCX data series consisted of 200 frames with an

![Figure 2: Contrast $\beta$ as a function of the beam defining slit size for the LCX and MAXIPIX detector at $Q = 0.0106 \text{Å}^{-1}$. The fitting curves (solid lines) are calculated using $\beta = 1/(1 + P_{eff}^2/S^2)$.](image)
Figure 3: (a) $g^{(2)}$-functions at 250 K at $Q = 0.00296$ Å$^{-1}$. The curves are shifted vertically for better visibility. The solid lines are single exponential fits to the data. (b) Relaxation rates as a function of $Q^2$.

exposure time of $t_{exp} = 0.4$ s. Two data series were taken with the MAXIPIX, one of 1000 frames using $t_{exp} = 0.1$ s and the second with 5000 frames of $t_{exp} = 0.02$ s. For all data series the auto-correlation functions $g^{(2)}(Q, t) = 1 + \beta(Q)e^{-2\Gamma(Q)t}$, where $\Gamma(Q)$ is the relaxation rate, were calculated and are displayed in figure 3a for a $Q$ value of 0.00296 Å$^{-1}$.

The advantage of the MAXIPIX detector for observing faster dynamics is clearly visible in figure 3a. The dynamics of the sample is too fast, so the LCX can only provide the information about the complete decay of the $g^{(2)}$-function at low $Q$ values. At high $Q$ values, the LCX measurements are catching just the last portion of the autocorrelation function, while both MAXIPIX measurements apparently provide information of the contrast value. The obtained fit results for the relaxation rates $\Gamma(Q) = D(Q)Q^2$ of the exponential decay are plotted as a function of $Q^2$ in figure 3b. With all three measurements similar results are obtained. The almost linear dependence points to Brownian motion of the tracer particles.

6. Conclusions

It is demonstrated that the MAXIPIX needs a flatfield correction to be used in XPCS experiments, in particular at lower X-ray energies. The analysis of the contrast of static scattering patterns shows that the effective pixel size of the MAXIPIX 2 × 2 detector below 10 keV is reduced from its physical pixel size. The analysis of speckle pattern from dynamical samples shows that the MAXIPIX is well suited for XPCS experiments. Both, MAXIPIX and the well established LCX detector give identical results. As the MAXIPIX is much faster than the LCX camera, it expands the accessible time window for multi-speckle XPCS experiments. The detector improves experimental conditions for the Coherence Beamline P10.

References

[1] Ponchut C, Rigal J M, Clément J, Papillon E, Homs A and Petitdemange S 2011 Journal of Instrumentation 6 C01069
[2] ESRF/ISDD 2010 MAXIPIX User Manual ESRF/ISDD
[3] Llopart X, Campbell M, Segundo D S, Pernigotti E and Dinapoli R 2002 IEEE Transactions on Nuclear Science 49 2279–2283
[4] Llopart X, Ballabriga R, Campbell M, Tlustos L and Wong W 2007 Nuclear Instruments and Methods in Physics Research A 581 485–494
[5] MEDIPIX-Collaboration http://medipix.web.cern.ch/medipix/
[6] Grübel G and Schüssler-Langeheine C 2010 International Workshop on the Materials Imaging and Dynamics Instrument at the European XFEL, Report of Working Group II on X-ray Photon Correlation Spectroscopy
[7] Abernathy D, Grübel G, Brauer S, McNulty I, Stephenson G, Mochrie S, Sandy A, Mulders N and Sutton M 1998 Journal of Synchrotron Radiation 5 37–47
[8] Lurio D, Lurio L, Mochrie S and Sutton M 2000 Review of Scientific Instruments 71 3274–3289