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X-ray photon correlation spectroscopy using the Mythen 1D detector

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Abstract. X-ray Photon Correlation Spectroscopy (XPCS) is an experimental technique to measure the dynamics of materials on nano- and microscales. Often, the maximum frame rate of the detector limits which dynamical processes can be investigated. This study examines the applicability of the Mythen 1D detector for coherent scattering applications with special focus on XPCS experiments.

1. Introduction

If coherent photons are scattered by a disordered sample, an interference (‘speckle’) pattern is created. One of the techniques exploiting the coherence properties of modern synchrotron sources is X-ray Photon Correlation Spectroscopy (XPCS), which makes it possible to investigate dynamical processes of samples by calculating auto-correlation functions from the fluctuations of the scattered intensity \cite{1, 2}. To be able to measure dynamical processes of a sample, the maximum frame rate of the detector must be faster than the time scales of the investigated dynamics. Nowadays, CCD detectors have typically frame rates of order 1 Hz, while pixel array detectors have frame rates of a few hundred Hertz. To investigate faster dynamical processes, point detectors in combination with hardware autocorrelators are frequently used. While these detector systems are able to probe dynamics up to the nanosecond timescale, they can only probe one point in reciprocal space per measurement. A detector probing a range of momentum transfers simultaneously thus reduces the experimental time needed. In addition, the time evolution of heterogeneous samples can be probed on different length scales at the same time. Therefore we started to investigate the possibility of using 1D detectors, namely the Mythen detector (version of Paul Scherrer Institute), for XPCS.

The Mythen detector is a single photon counting, high framing rate, 1D detector developed by the Paul Scherrer Institute (PSI), Switzerland, and is based on Silicon \cite{3, 4}. The sensor consists of 1280 strips (each 8 mm long). The pitch between neighboring channels is 50 µm and the strip thickness of the used detector is 320 µm. The dynamic range of the detector can be set to 4, 8, 16 and 24 bit. The Mythen detector version explored here is a custom version by PSI equipped with an additional RAM memory buffer, which allows to achieve frame rates of up to 2000 Hz using the lowest dynamic range of 4 bit. This makes it possible to extend the time window of multi-speckle XPCS experiments by an order of magnitude towards faster time scales.
2. Experimental details

In an XPCS experiment, the dynamics of a sample are analyzed by means of the normalized intensity autocorrelation function $g_2(Q, \tau)$ at a given momentum transfer $Q$ [5]:

$$g_2(Q, \tau) = \frac{\langle I(Q, 0) I(Q, \tau) \rangle}{\langle I(Q) \rangle^2}.$$ (1)

The investigated samples were model colloidal systems of spherical particles suspended in polypropylene glycol. The colloidal SiO$_2$ particles were coated with 3-methacryloxypropyl-trimethoxy-silane, resulting in a hard-sphere like interaction potential.

The experiments were performed at the Coherence beamline P10 at PETRA III. The experiments were carried out in Small Angle X-ray Scattering geometry at a photon energy of $E_{ph} = 8$ keV. A beam of 200 $\mu$m in the vertical and 100 $\mu$m in the horizontal was focused by a set of compound refractive lenses to a focal point size of approximately $3 \times 5 \mu m^2$ (VxH).

In order to resolve speckles, a slit in front of the Mythen detector decreases the strip length from 8 mm down to 50 $\mu$m, resulting in an apparent square pixel size. To control the detector, a device server has been implemented using the Tango control system [6]. It makes it possible to control the detector via PERL scripts from Online [7], the beamline control software at PETRA III. As an additional feature, this Tango device server allows for the automatic merging of individual frames taken by the Mythen detector in a continuous series. The merged frames are stored as a 2D ASCII matrix. The Mythen detector is equipped with an additional 128 MB RAM memory buffer, making it possible to store data in RAM without having to wait for data transfer. Using a dynamic range of 8 bit, a total of 80000 frames can be stored in memory, before the data must be transferred.

To analyze the performance of the Mythen detector for XPCS, the experimental results obtained using the Mythen 1D detector were compared to results of i) an avalanche photo diode (APD), a point detector and ii) a MAXIPIX $2 \times 2$ detector with an array of 512 by 512 pixels. The three detectors were placed $\approx 5.1$ m downstream of the sample position.

3. Results

3.1. Static scattering experiments and dynamic range

In fig. 1, panel a), a series of 10000 individual Mythen frames taken with a frame rate of 1667 Hz is displayed as a 2D picture. A region of interest of 300 strips of the Mythen detector are displayed in the vertical, while in the horizontal the individual frames are shown. The intensity
Table 1: Minimum hardware read-out time, safe minimum read-out time and corresponding frame rates using a 1 to 1 ratio of exposure time to safe minimum read-out time.

<table>
<thead>
<tr>
<th>Dynamic range</th>
<th>24 bit</th>
<th>16 bit</th>
<th>8 bit</th>
<th>4 bit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minimum hardware read-out</td>
<td>240 µs</td>
<td>186 µs</td>
<td>125 µs</td>
<td>94 µs</td>
</tr>
<tr>
<td>Safe read-out</td>
<td>2000 µs</td>
<td>1600 µs</td>
<td>800 µs</td>
<td>300 µs</td>
</tr>
<tr>
<td>Frame rate [Hz]</td>
<td>250</td>
<td>312</td>
<td>625</td>
<td>1667</td>
</tr>
</tbody>
</table>

Variations for a given strip are the result of the interference due to the coherent illumination of the sample. As the frame rate is high enough to capture the sample dynamics, the 2D picture shows the evolution of a speckle pattern with experimental time.

Due to the low concentration of particles, the static (time averaged) scattering intensity is directly proportional to the form factor \( P(Q, R) \) of the particles, as given for example in reference [8]. In fig. 1, panel b), the time-averaged scattering intensity is displayed. The data obtained using a dynamic range of 24 bit, 16 bit or 4 bit with a frame rate of 1667 Hz matches the model fit almost perfectly. A fit of a spherical particle form factor model yields a mean radius of \( R = 242 \) nm and a size polydispersity of the colloidal particles of 4%.

Measurements of the same sample using identical illumination conditions were performed for different dynamic ranges of the Mythen detector. As displayed in fig. 1, panel b), apart from the cut-off at higher count rates for the lower dynamic ranges, the measurements for 4 bit, 8 bit, 16 bit and 24 bit give identical results. By increasing the frame rate from 2 Hz to 1667 Hz, also the low \( Q \)-values with high count rates can be measured using a dynamic range of 4 bit.

3.2. Dynamic scattering experiments and read-out time

The read-out time of the Mythen detector was measured at the four different dynamic ranges using an oscilloscope. The resulting values are summarized in table 1. A lower dynamic range reduces the read-out time of the detector, thus increasing its maximum framing rate. The data acquisition system is able to measure \( \approx 1000 \) frames using these minimum read-out times. However, frame drops occur for longer data series which cause an interruption of the data acquisition. This is a serious problem for XPCS measurements, which rely on continuous uninterrupted data sets. To circumvent these frame drop events, safe minimum read-out values have been identified by performing repeated data series of \( \geq 20000 \) frames using the different dynamic ranges of the detector. The values, which worked in the case of the investigated setup, but can not be guaranteed, are displayed in table 1.

Using the XPCS technique, results obtained by the the Mythen detector system (1D) have been compared to i) a point detector (APD) connected to a hardware autocorrelator and ii) a MAXIPIX 2 × 2 detector (2D). 20000 frames have been measured with a frame rate of 100 and 1666 Hertz for the Mythen and a frame rate of 200 Hertz for the MAXIPIX 2 × 2 detector, resulting in a total exposure time of 200, 13 and 100 seconds respectively. The APD was accumulating data for 300 s. The comparison between APD and Mythen detector is displayed in fig. 2, panel a), while the comparison between Mythen and MAXIPIX 2 × 2 detector is shown in fig. 2, panel b). All resulting intensity autocorrelation functions show a single exponential decay, and a single exponential fit to the data resulted in identical relaxation rates.

The different visibility of the autocorrelation functions is a result of the different effective pixel sizes of the detectors. For the APD, a slit in front of the detector reduces the effective pixel size to \( 100 \times 100 \) µm². The observation of a slightly lower speckle visibility for the Mythen detector when compared to the MAXIPIX 2 × 2 detector having a nominal pixel size of \( 55 \times 55 \) µm² is a result of the used discrimination threshold of 5 keV for the MAXIPIX. Any 8 keV photons shared between two pixels will not be detected, resulting in an effectively smaller pixel size.
4. Conclusions and Outlook

The comparison of the measured auto-correlation functions between the Mythen, the MAXIPIX $2 \times 2$ and the APD detector shows that all 3 detectors are suitable for XPCS. By adjusting the dynamic range of the Mythen detector, it is possible to increase the speed of the measurement when staying well below the maximum count-rate possible at the chosen dynamic range. The Mythen detector is able to measure multiple $Q$ values at the same sample position at the same time, which is important if radiation sensitive heterogeneous samples are investigated with small beams. In these systems different dynamics can be observed at each spot of the sample. In addition, the Mythen detector offers the advantage of an increased frame rate when compared to the MAXIPIX $2 \times 2$ detector.

The Mythen detector, as a 1D detector, is only able to probe one direction in reciprocal space at one time and can therefore not be used for XPCS experiments of anisotropic sample systems. In addition, the maximum frame rate achieved using the Mythen detector is only up to 2kHz. The development of faster 2D pixel array detectors such as the Medipix3 chip with a frame rate of 4kHz at 6bit counter depth and the Eiger detector with a framing rate of 3kHz will be advantageous for future XPCS experiments.

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References