New Pixel Array Detector Techniques for X-ray Photon Correlation Spectroscopy Experiments

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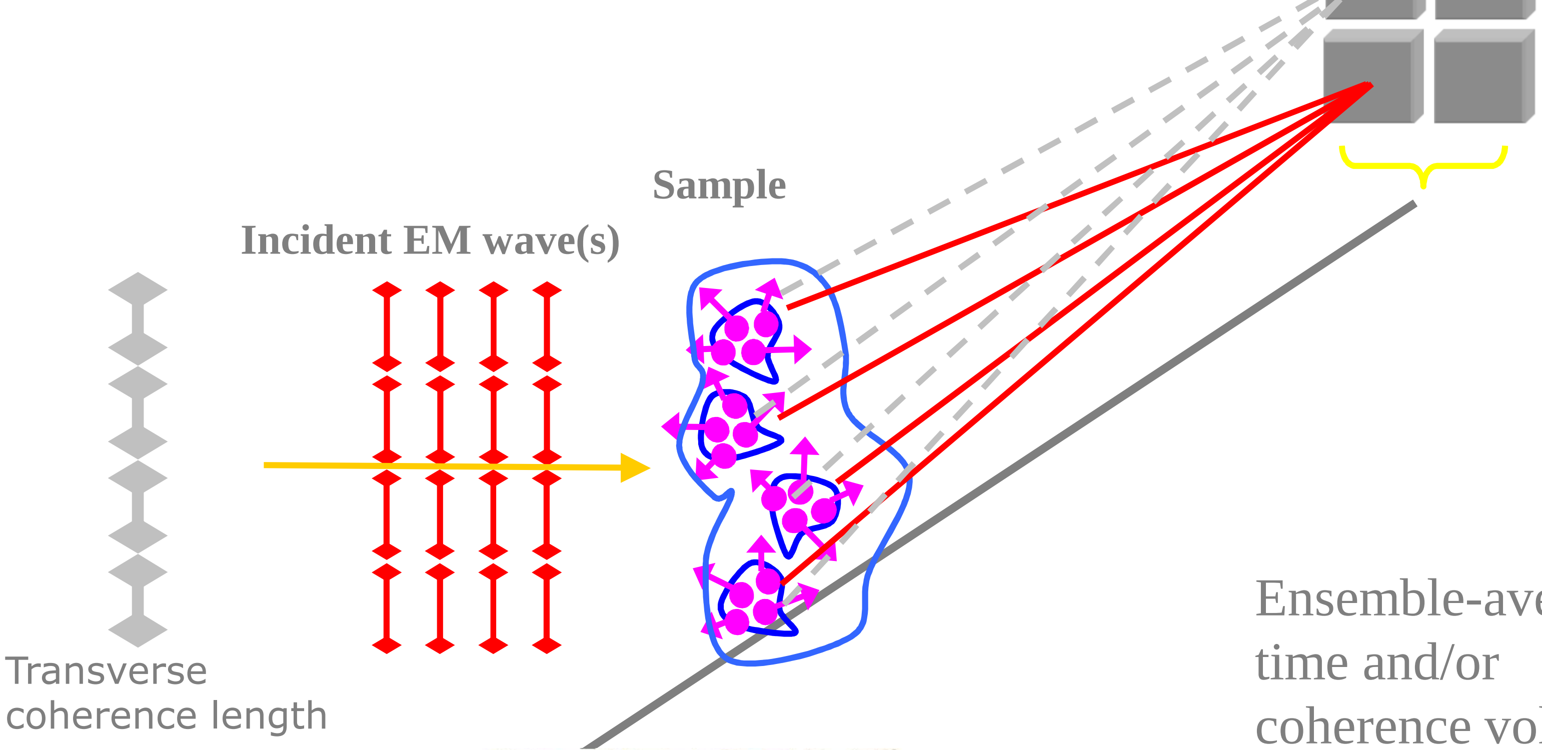
APS Technical Working Group, Argonne, October 20, 2016

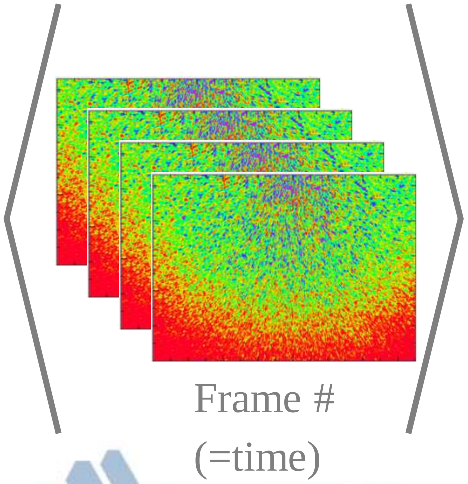
Outline

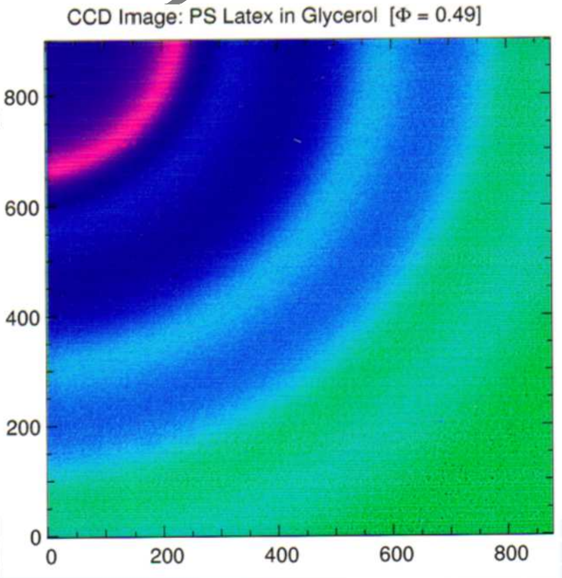
- Very brief background on XPCS
- Background of use of Pixel Array Detector (PAD) in X-ray Photon Correlation Spectroscopy (XPCS) in the literature
- Timing and integration with PAD
- 8-ID-I Beamline overview
- Three example of new capabilities in PAD:
  - dead-time free 11 kHz operation with UFXC32k PAD
  - two-frame XPCS with Voxtel
  - Sparse data acquisition with VIPIC prototype
- Conclusions

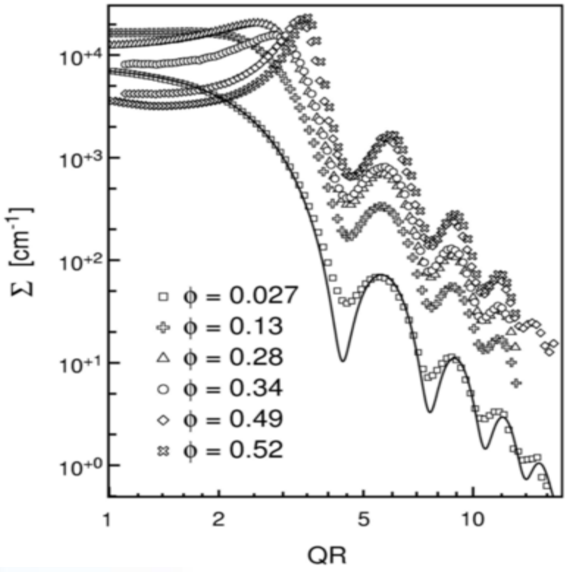
Measuring Static/Time-Averaged Properties



Ensemble-average:
time and/or
coherence volumes

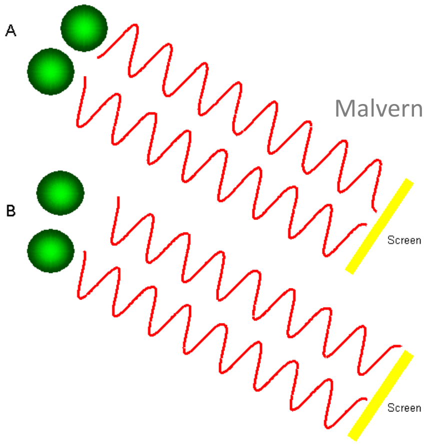



CCD Image: PS Latex in Glycerol [Φ = 0.49]

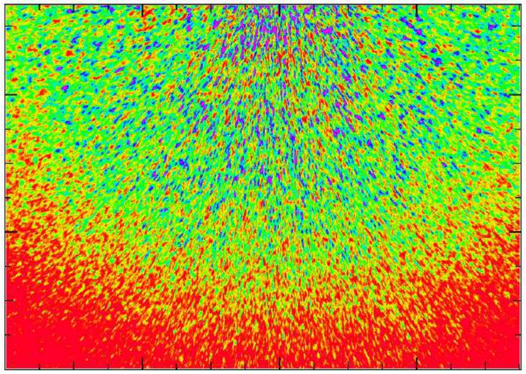


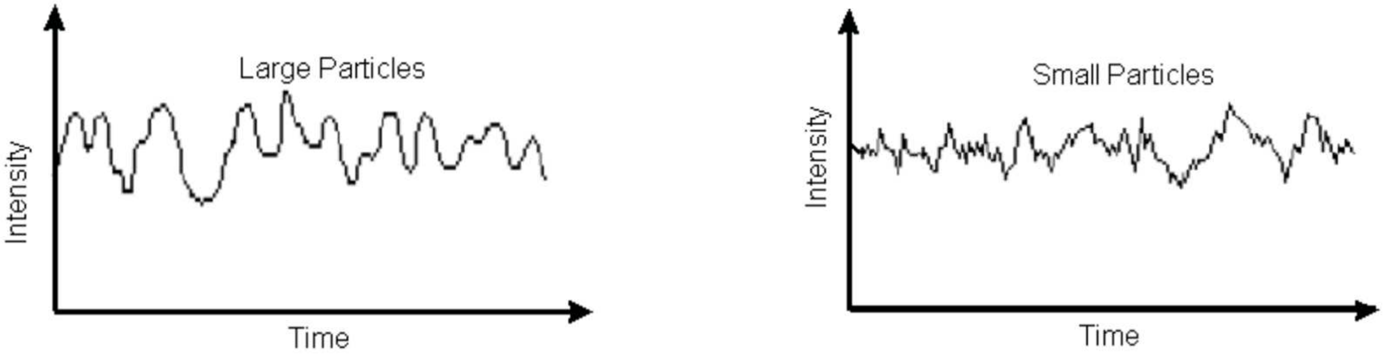
XPCS

- Dynamic light scattering (DLS) or photon correlation spectroscopy (PCS) but with x-rays rather than laser light:
  1. Illuminate a disordered sample with a (partially) coherent x-ray beam
  2. Collect the speckled scattered beam with a high resolution detector
  3. Monitor speckle pattern as a function of time so that changes in the speckle pattern can be observed



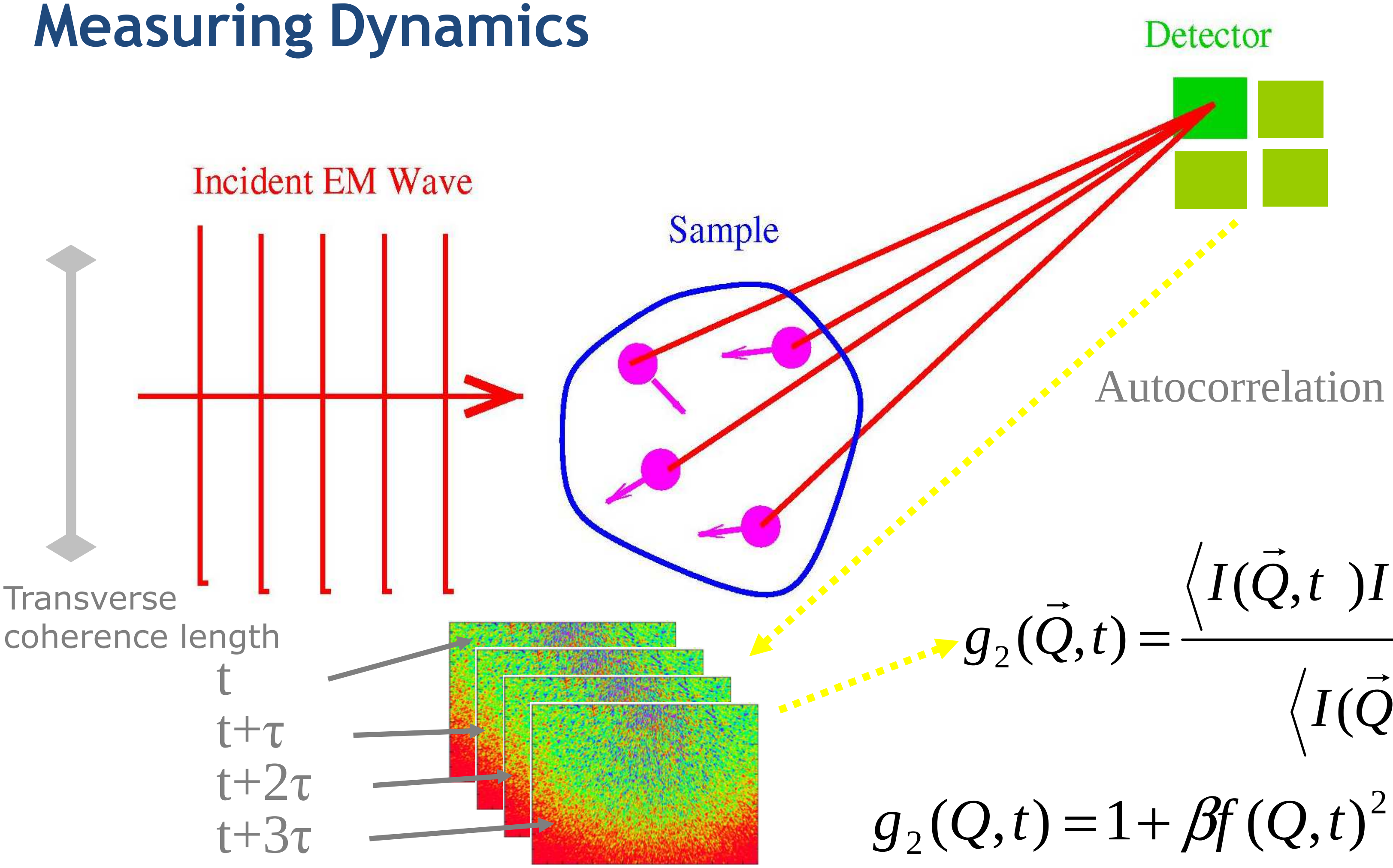
Malvern Technical Note MRK-656-01





Malvern Technical Note MRK-656-01

Measuring Dynamics



$$g_2(\vec{Q},t)=\frac{\left\langle I(\vec{Q},t)I(\vec{Q},t+\tau)\right\rangle_\tau}{\left\langle I(\vec{Q},\tau)\right\rangle_\tau^2}$$

$$g_2(Q,t)=1+\beta f(Q,t)^2$$

$$f(Q,t)=S(Q,t)/S(Q,0)$$

$$=e^{-\Gamma t}$$

$$\Gamma=DQ^2=(k_BT/6\pi\eta R)Q^2$$

Dilute suspension-"Brownian" motion

- S(Q,t) is the dynamic structure factor
- Γ is proportional to the diffusion constant and has a Q^2 dependence (dilute)
- β, varying from 0 →1, expresses how coherent the incident beam is

Background of Pixel-Array detector use in XPCS

- XPCS made great strides since 1995 with the pioneering use of small-pixel (20 microns) direct detection Charge Coupled Device (CCD). The direct detection CCD is the workhorse of many XPCS beamlines in the world and provides excellent performance for slow dynamics with time scales from a few milliseconds (ms) to hours.
- A second generation of PAD with pixels < 100 microns is slowly replacing CCDs in XPCS due to their good quantum efficiency, high speed, multiple counters, and flexible timing capabilities.
- The Eiger is one of the fastest XPCS detector in the world. A Swiss Light Source version demonstrated 22 kfps operation in 2012 (Johnson et al, JSR 2012) , and the commercial Dectris Eiger operates around 1 kfps.
- Other commercial detector (Lamda Gmbh) offers a Medipix3-based PAD with 2 kHz operation in burst-mode. Our beamline is commissioning a system which we hope will save data at 1000 fps in continuous operation mode, and 2000 fps in burst mode.

Dual counter PADs enables new modes of operation such as dead-time free operation, two-frame mode

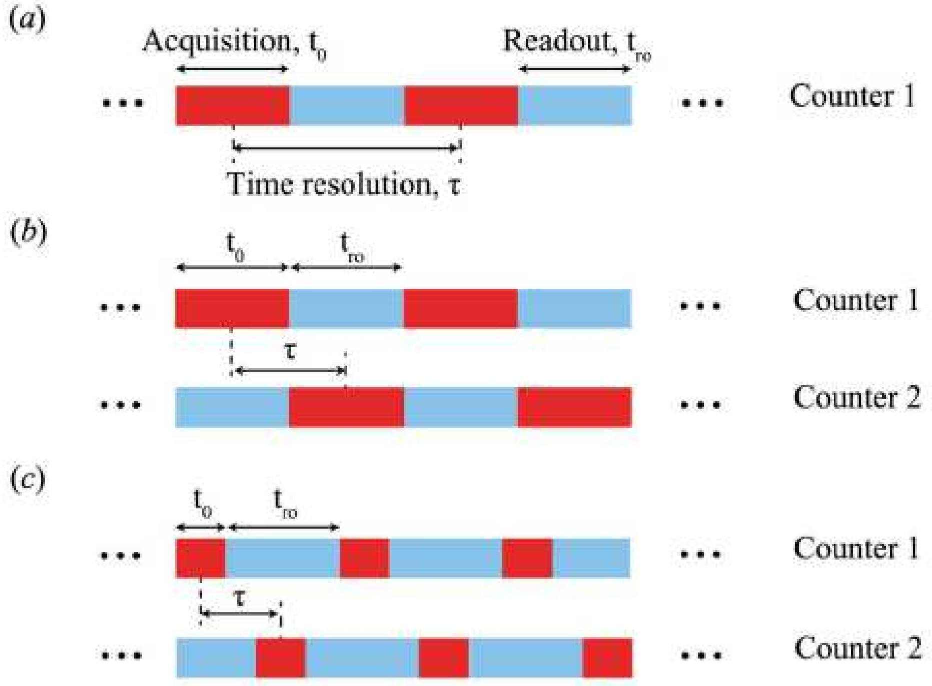


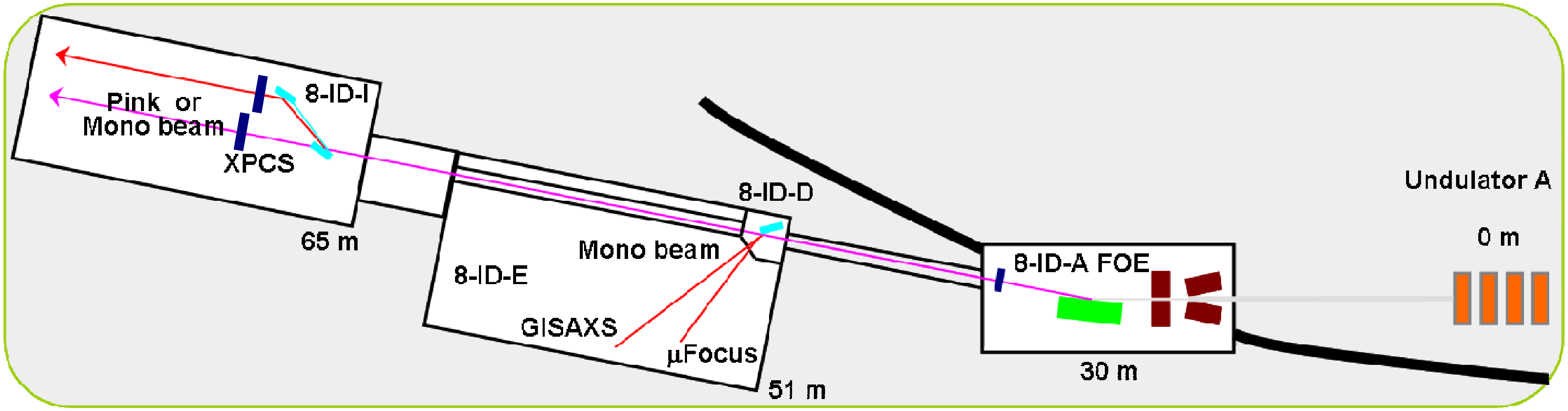
Figure 1

Time infrastructure of (*a*) continuous acquisition with one counter per pixel, (*b*) dual counter acquisition discussed in this study with no readout dead-time between the frames and (*c*) future upgrade of dual counter acquisition where the separation between the frames is smaller than the digitization time associated with each counter.

Q. Zhang et al., J. Synchrotron Rad. 23, 679-684 (2016)

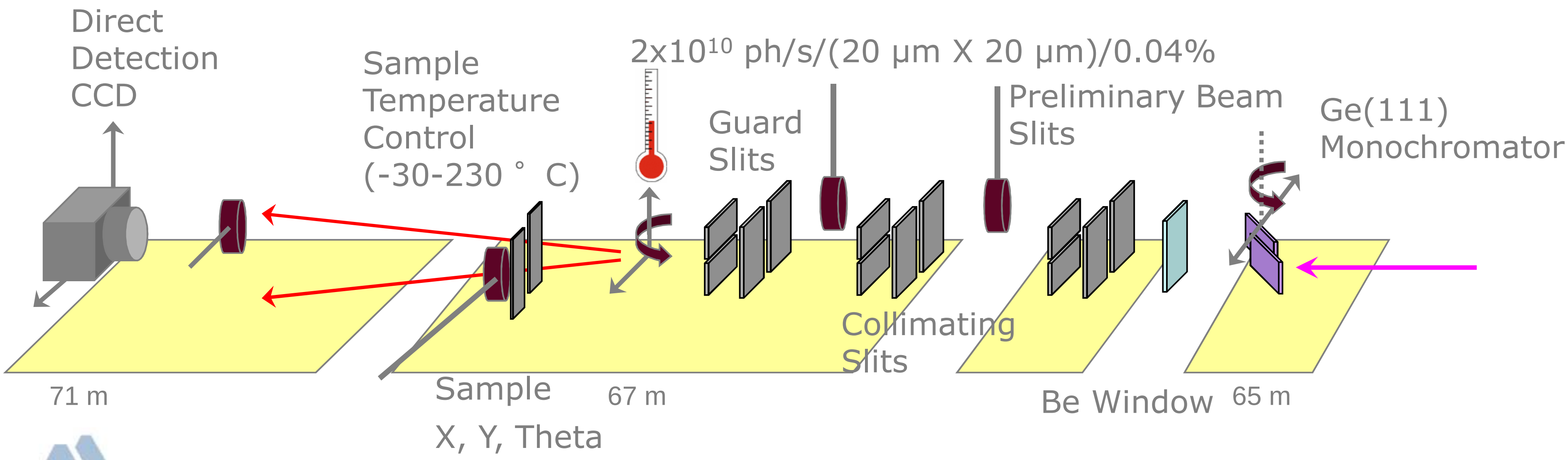
Overview of Small-Angle XPCS at 8-ID-I

- Simple undulator beamline, all water cooled optics → improved stability
- 2 phased undulator A (at 7.35, and 11 keV) using the full straight section
- Minimal beam size – only central cone into optics enclosure
- Mirror first optics in 8-ID-A
- Beam splitting monochromator in 8-ID-D for 8ID-E, DCM in 8-ID-I



Small-Angle XPCS in 8-ID-I

- 8-ID-I Station Features
  - Ge(111) monochromator DCM
  - Polished Be window
  - In-vacuum slits- preliminary, collimating and guard slits (X2)
  - In-vacuum sample “oven”
  - In-vacuum alignment detectors and beam stops
  - Direct-detection CCD, commissioning Lambda Mpixel PAD.
  - New environment such as rheometer under commissioning



UFXC32k tests on 8-ID-I: two counters deadtime free.

Test chip from Krakow University
128 x 256 75 μm pixels
320 μm thick Si sensor
2 counters, here timed for deadtime free operation
2 bit operation tested to 12 kfps (cps per speckle well below saturation)
Experiment at 7.4 keV with 2 x 10^{10} ph/s with 3 μm FWHM vertical focusing
20 μm horizontal coherence slit
4 m detector distance.

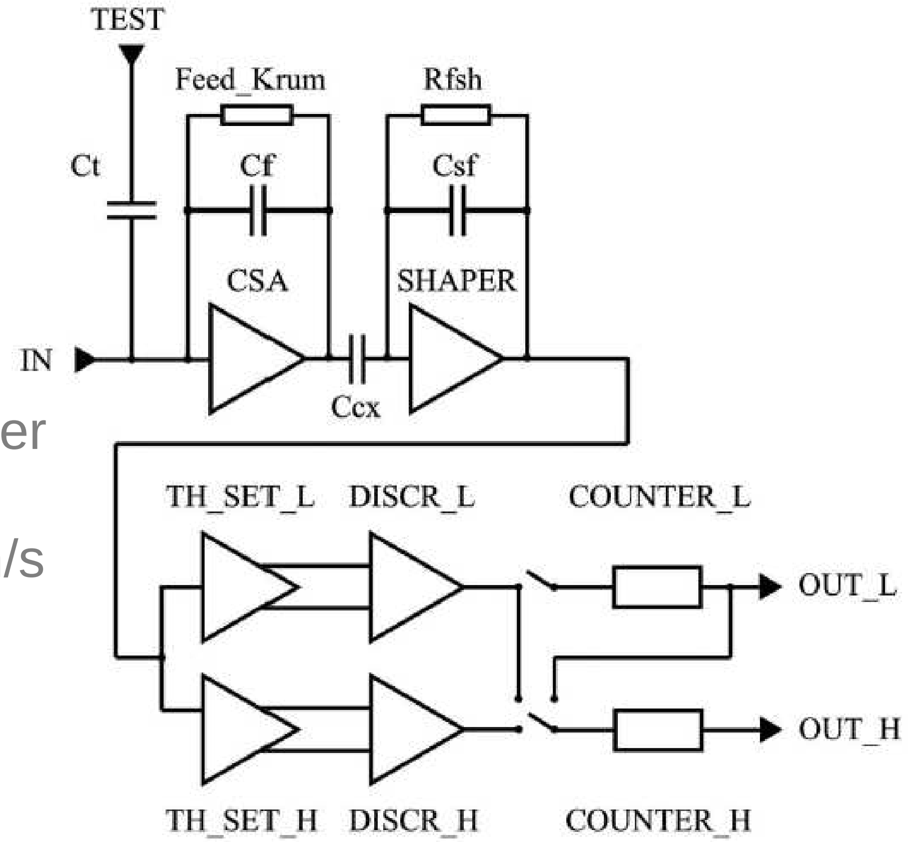


Figure 3
Schematic of the readout circuit of a single pixel.

UFXC32k tests on 8-ID-I (cont)

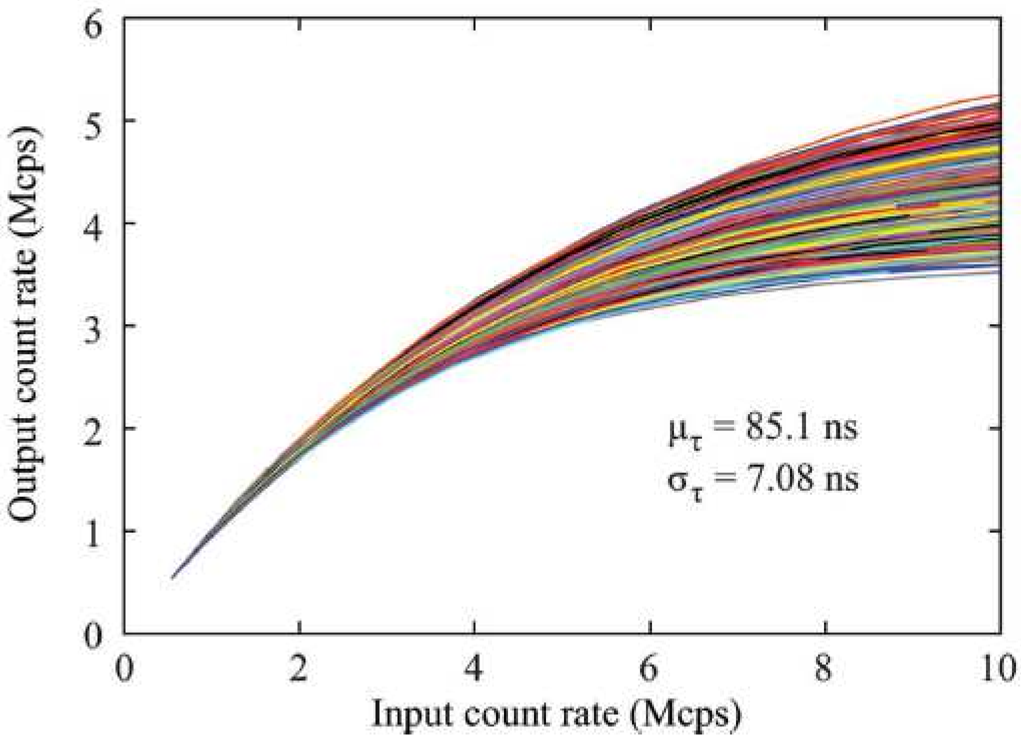


Figure 4
Dead-time measurement for about 1200 pixels of UFXC32k.

Lab source tests show uniform deadtime over all pixels

UFXC32k tests on 8-ID-I (cont)

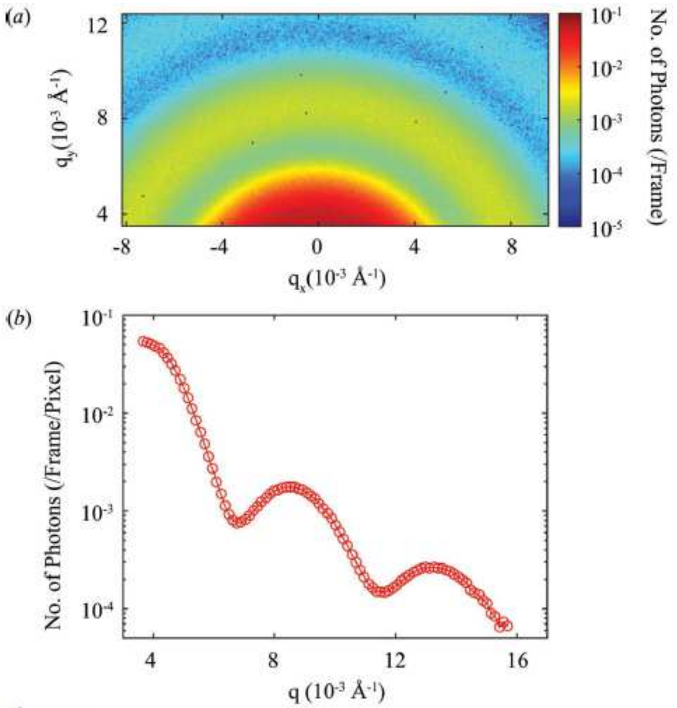


Figure 5
(*a*) Time-averaged scattering from the latex nanoparticle suspension. The scattering intensity is indicated by the logarithmic color bar. (*b*) Azimuthal average of Fig. 5(*a*).

Prototype detector tested successfully in July 2016 at 50kHz.

Plan to sparsify the data saving.
Possible to use detector in future in two-fast-frame mode

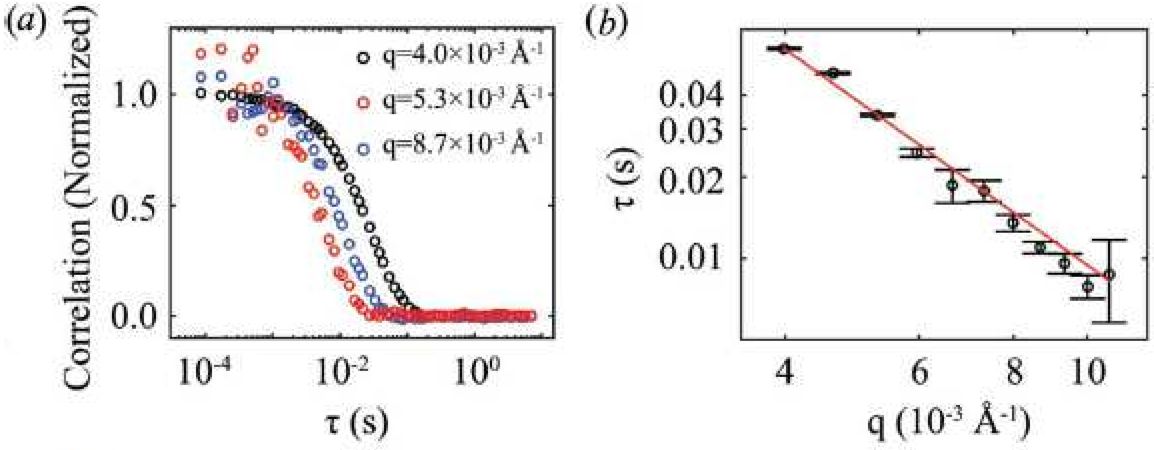


Figure 6
(*a*) Dynamics of latex nanoparticles indicated by $g_2(\tau)$ at different q. (*b*) Decorrelation time $\tau(q)$ *versus* q. The red line shows the inverse-square decay of the correlation time.

70nm latex spheres
In glycerol

Q. Zhang et al., J. Synchrotron Rad. 23, 679-684 (2016)

Two-fast-frame XPCS

Prototype from Voxtel Inc. with
48x48 130 μm pixels, 520 μm Si sensor,
660 eV resolution around 10 keV

Very fast detector designed for pump-probe experiments capable of resolving the APS 153 ns bunch mode (see S. Ross et al. JSR 23, 196 (2016)).

Slow data saving to disk with ~8 frames per second.

Dual counter PAD with two externally triggered gates. Gate widths and delay scanned with width = delay.

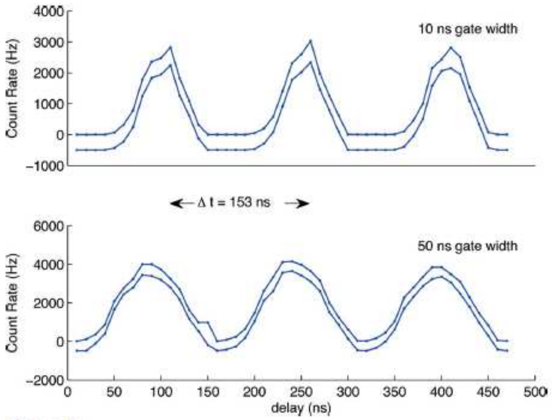


Figure 11

Above from S. Ross et al. JSR 23, 196 (2016)

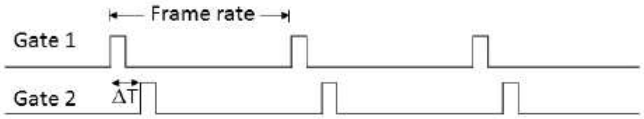


Fig. 1. The novel detector timing diagram applicable to two pulse XPCS. Varying the time delay ΔT between the gate 1 and 2 signals allows measurement of time correlation functions at small delay times. Note that the time difference between pulses in the two channels can be much smaller than the overall frame pair rate.

$g_2(t+\delta t) = \langle I(q,t)I(q,t+\delta t)\rangle / \langle I(q,t)\rangle^2$
where <> is a per-pixel average over ~5000 pairs of frames

Example 1: two-fast-frame XPCS (cont.)

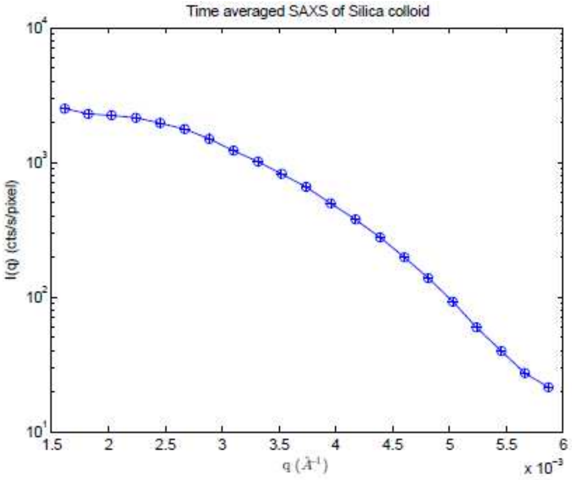


Fig. 2. The time-averaged and azimuthal average of I(q) versus q for the Silica nanoparticle in water. Two data sets are shown, the average from the low counter (o) and high counter (+). The solid line is a guide to the eye.

Time-averaged SAXS of dilute (5.25 vol %) 150 nm Silica nanoparticle in water.
6 x 10^9 ph/s at 7.38 keV
unfocused beam 20 x 20 μm

E.M. Dufresne et al., Opt. Express **24**, 355 (2016).

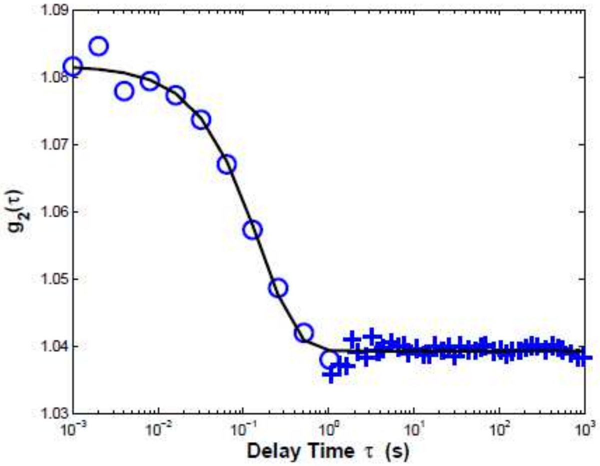


Fig. 3. A typical time correlation function for one wavevector from the latex particle in glycerol at 25 °C. Open circle data are from the two fast-frame technique, data displayed with + are from sequential XPCS, and the solid line is a fit described in the text.

Two-frame time correlation of 70 nm latex particle in glycerol for a typical wavevector sampled as
fast as 1 ms with 4 frame pairs per second.
+ sequential XPCS
o two-frame crosscorrelation

Two-fast-frame XPCS (cont)

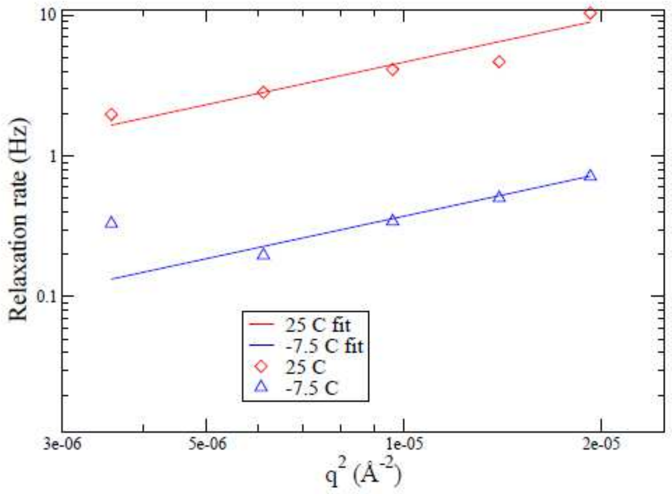


Fig. 4. The relaxation rate of g_2 versus q^2 for the latex sample in glycerol for two temperatures, -7.5 and 25 °C.

Temperature dependence of relaxation rate of latex in glycerol

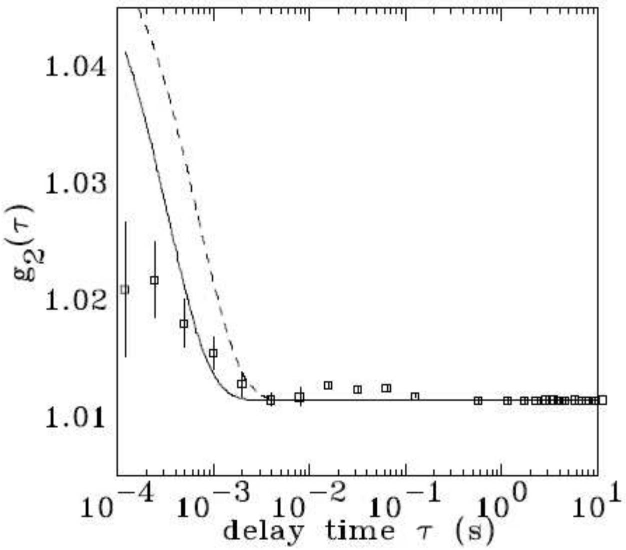


Fig. 5. The time correlation of the Silica colloid versus time delay at 25 °C. The solid and dashed lines are from an estimate of the relaxation rate in the range of wavevector sampled. Note the minimum sampling time of 0.12 ms.

Dilute silica nanoparticle in water sampled down to 0.12 ms.

Voxtel detector no longer available.

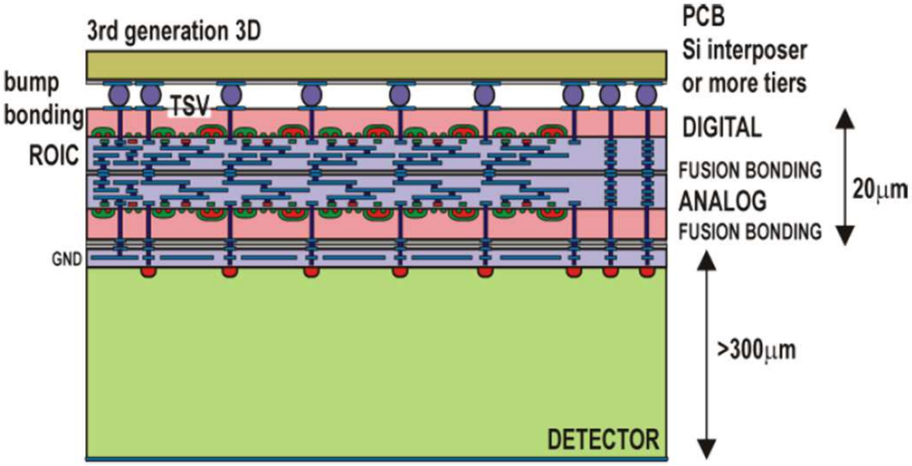
E.M. Dufresne et al., Opt. Express **24**, 355 (2016).

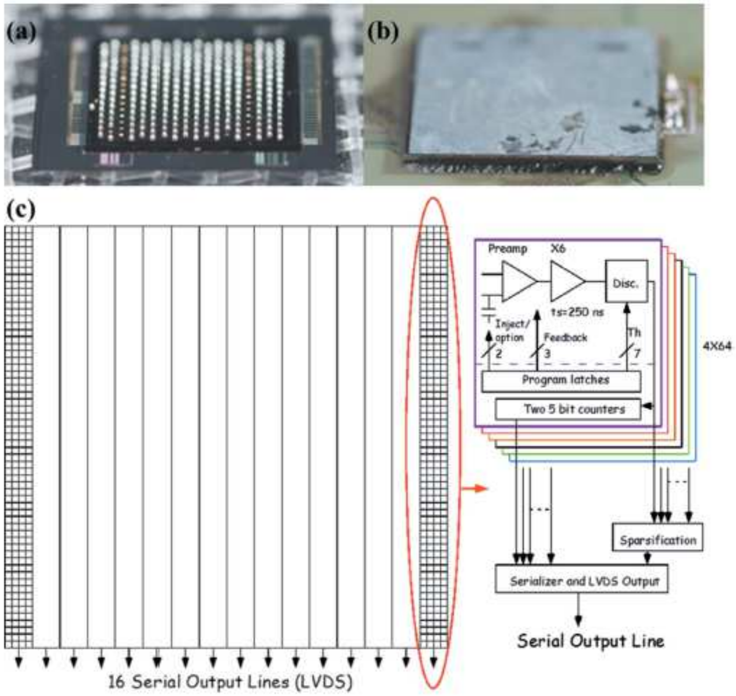
Two frame XPCS: why bother?

- True time correlation measurement, so compared to Speckle Visibility Analysis doesn’t need a model of $g_1(\tau)$. Likely less sensitive to confuse spatial variance with time variance.
- Can break the frame rate limit with time-based correlation. Here we sampled ms fluctuations with 4 frame-pair per second.
- If the product of the number of pixel and average frame rate is over ~10 Mpixel/s, it outperforms our single-channel APD while providing 2D information.
- Recently procured a modified Pixirad-1 detector with this capability, with 100x more pixels and 20x faster average frame rate (42 Mpixel/s).

VIPIC prototype chip tests on 8-ID-I

Demonstrated proof-of-concept speckle visibility and XPCS measurements via the prototype VIPIC (vertically integrated photon imaging chip) (64 X 64 pixels) detector





VIPIC (cont.)

Sparsified output with x,y and time stamp of detected x-ray

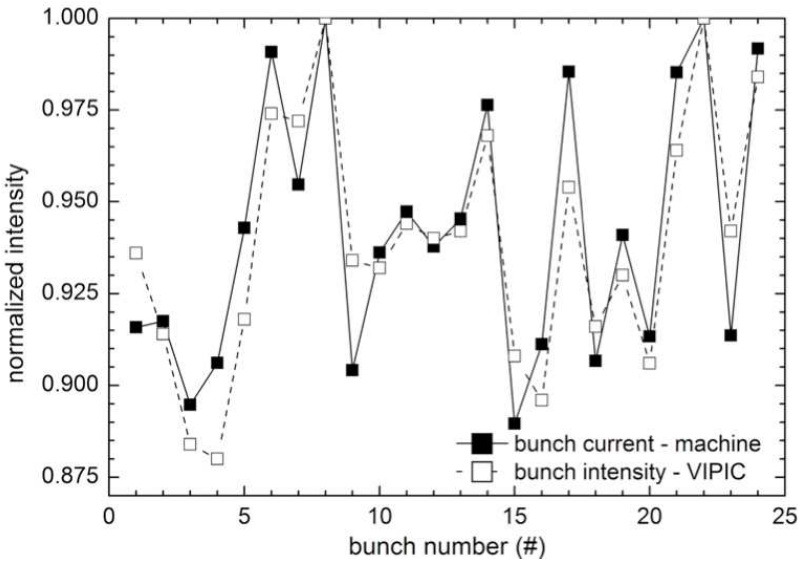
100 MHz clock, fast response

5-bit counter limit count rate per pixel to 40 kcps.

Detector synchronized to ring clock and exposure 1 SR turn typically at 3.6 µs for up to 20 min.

1 ID used at 10 keV,

20x20 µm coherence slit



Detector can resolve 24 bunch mode of APS.

First demonstration of VIPIC

- Fast colloidal dynamics recorded with the VIPIC prototype, latex sphere in glycerol.
- Time constant temperature dependent due to change of glycerol viscosity
- Prototype is no longer functioning

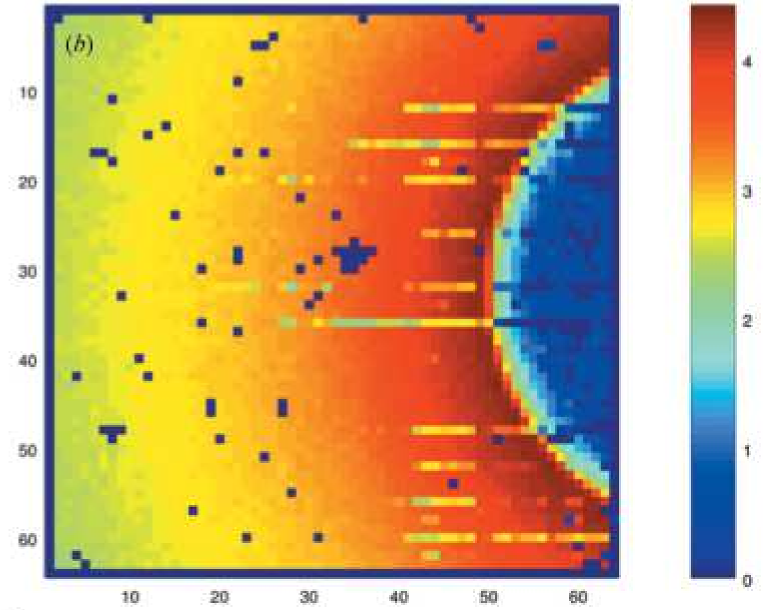


Figure 3
(a) Histogram, on a logarithmic scale, of the number of hits in one serial stream within one integration frame. (b) The accumulated intensity (logarithmic intensity scale). The number of dead pixels was about 5%.

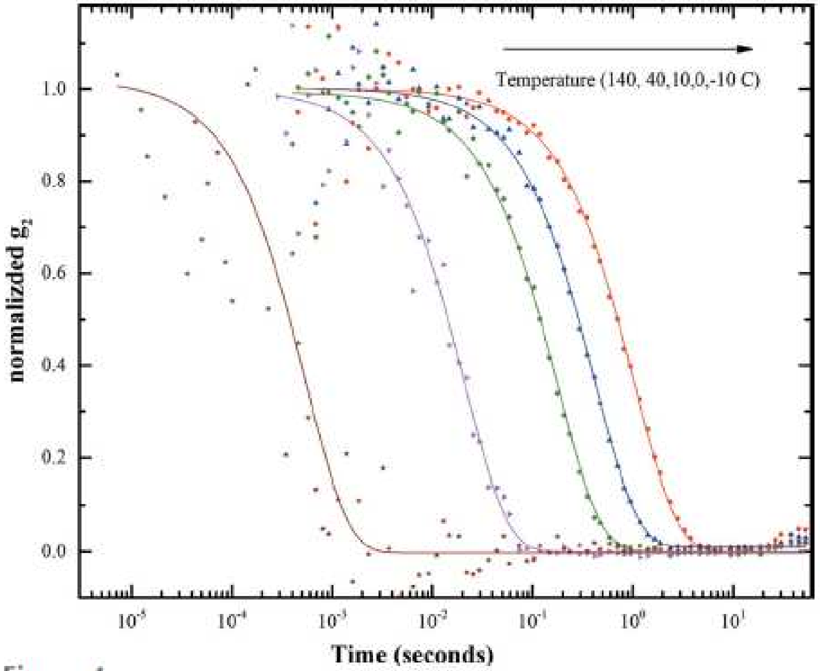


Figure 4
Measured intensity autocorrelation function of polystyrene particles suspended in glycerol at different temperatures.

Near term future

- Two new VIPIC based detector with 1 Mpixel will be delivered to APS and NSLS-II as part of the final phase of this DoE detector grant. The chip has been submitted to the factory recently, and the detector should be ready for test in 2018.
- We recently tested successfully the UFXC32k detector at 50 kfps. The speed limit has likely been reached, and thus future improvements will focus on the data storage compression, and test new features such as two fast frames.
- As part of preparing for the MBA, we will also develop experimental techniques to handle the large flux increase by using pink beam in simple tests experiments with fast detectors.

Partners

Argonne: Steve Ross, David Kline, John Wezorik, Bob Bradford, Qingteng Zhang, Suresh Narayanan, Alec Sandy
DePaul University: Eric Landahl
McGill University: Mark Sutton
FNAL: Gregory Deptuch, Anthony J. Kuczewski, Scott Helm
SLAC: Gabriella Carini
BNL: Abdhul Rumaiz, Andrei Fluerasu, Peter Siddons.
AGH University: Piotr Maj, Piotr Kmon, Robert Szczygiel, Pawel Grybos

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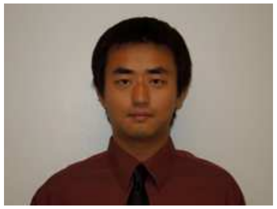
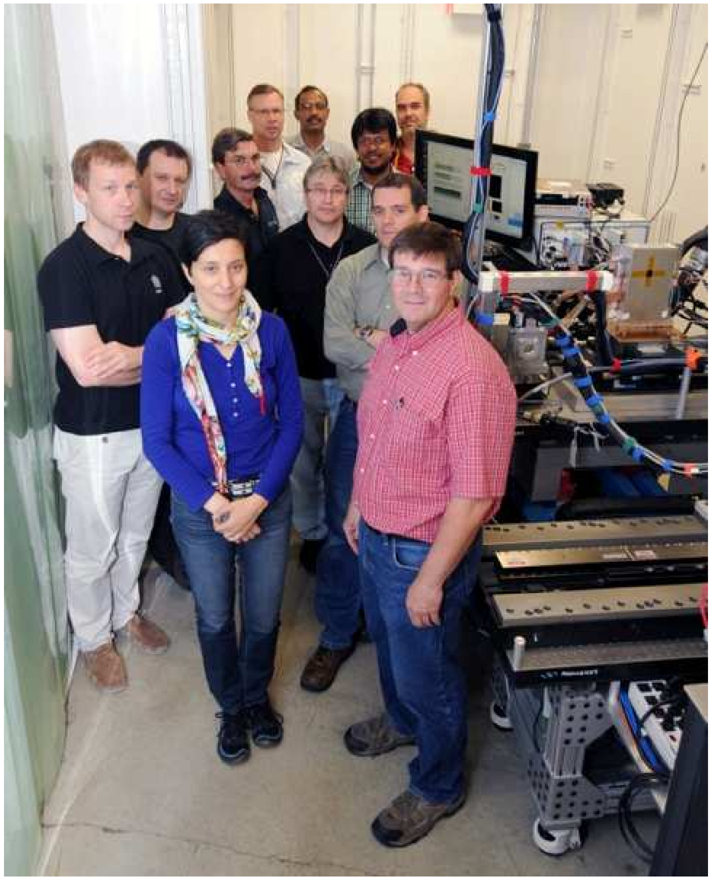
AGH University of Science and Technology was supported by the National Center for Research and Development, Poland, PBS1/A3/12/2012.

Postdoc
Qingteng
Zhang



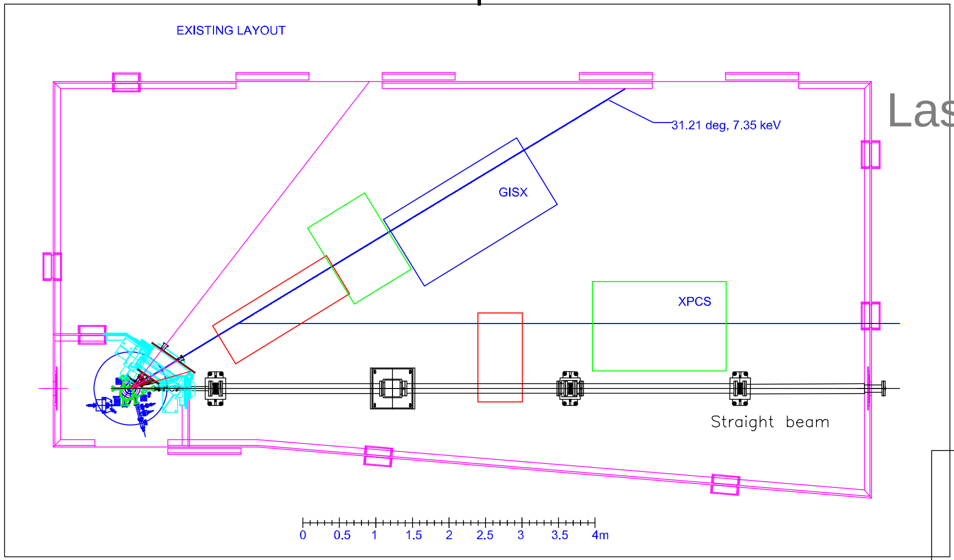
VIPIC and a portion of the commissioning team at 8-ID-I (4X national labs!)



8-ID E and I recent changes

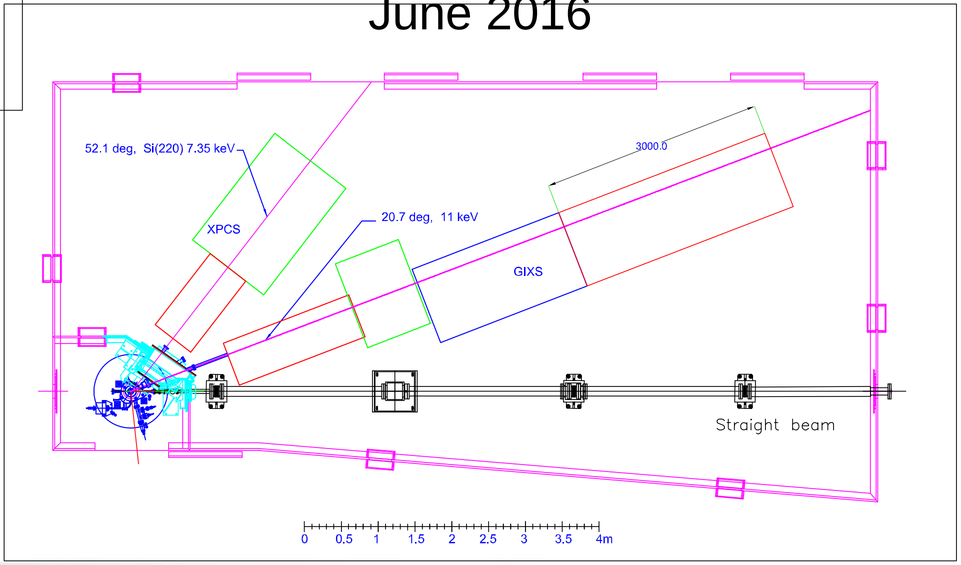
2., 3. CSI resolution and positioning

- Resolution gains via
  - Increased sample to detector distance



Last run 2016-1

June 2016



Relocation of WA-XPCS to Si (220) side-bounce at 7.35 keV and GISAXS beamline on Si (111) at 11 keV in May 2016.