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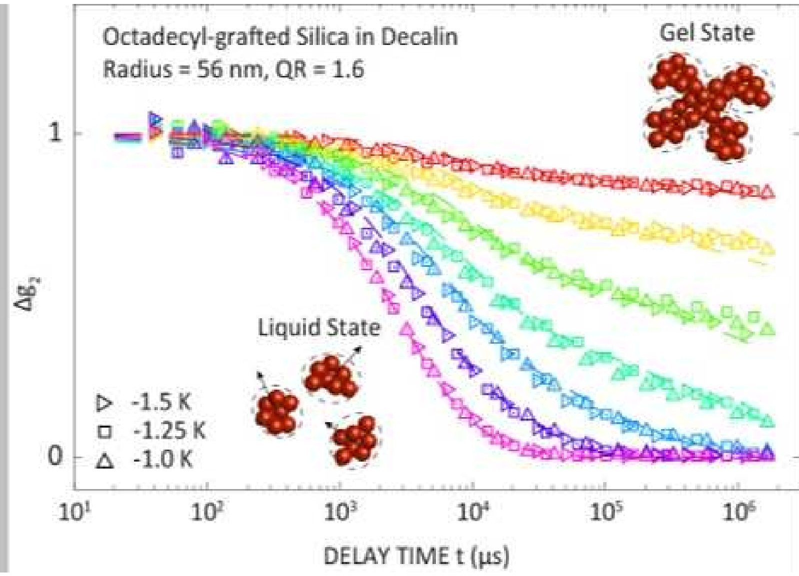
PUSHING THE SPEED OF X-RAY PHOTON CORRELATION SPECTROSCOPY EXPERIMENTS AT 8-ID-I WITH FAST PIXEL-ARRAY DETECTORS



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U.S. DEPARTMENT OF ENERGY | Office of Science

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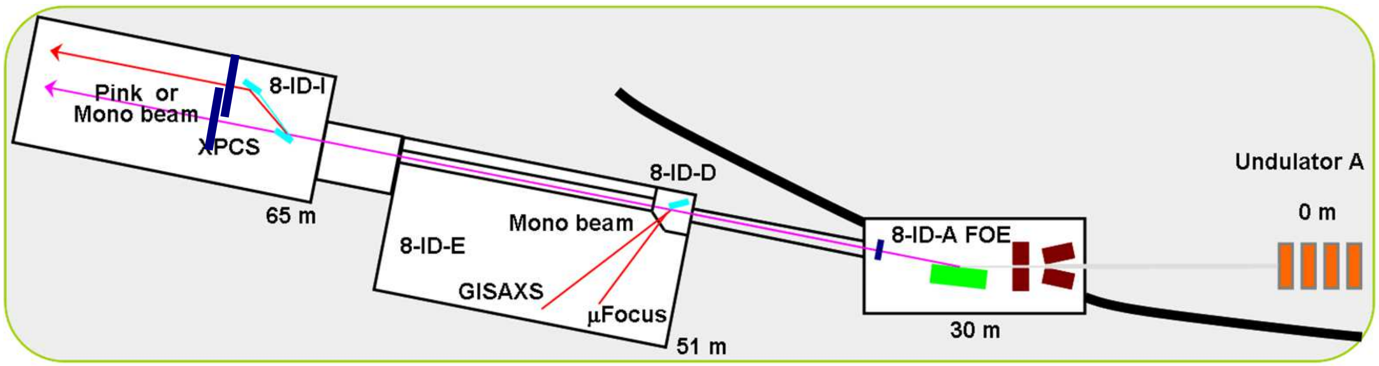
OUTLINE

- UFXC32k introduction and performance
- Brief example of XPCS study of gelation dynamics with unprecedented speed.
- Recent commercial detector results from Rigaku, UHSS500k
- Upgrade plans of APS 8-ID.

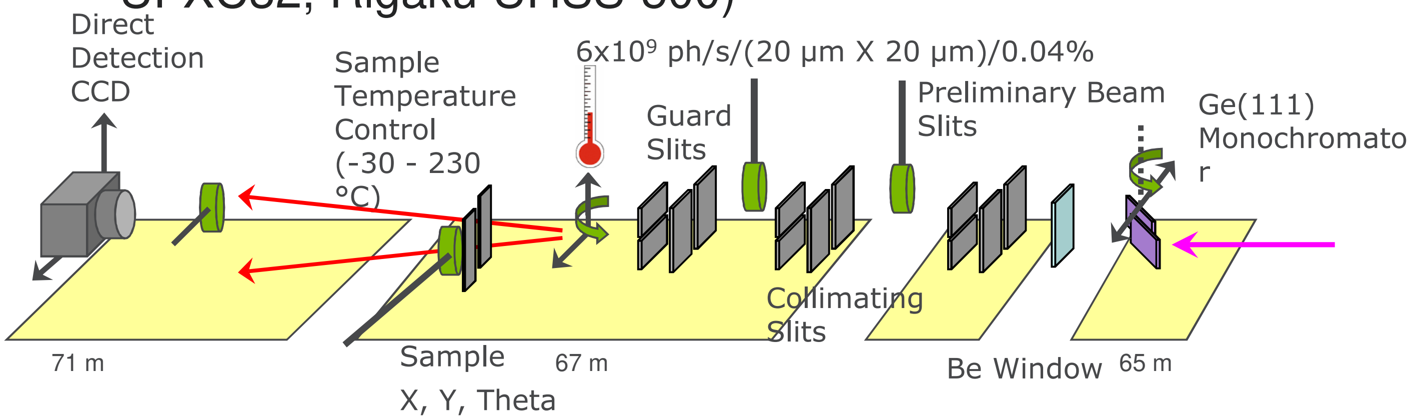
XPCS AT BEAMLINE 8-ID

- How is XPCS realized at 8-ID?
  - Simple undulator beamline with water-cooled optics → improved stability
  - Minimal beam size – only central cone into optics enclosure
  - 2 phased undulator A using the full straight section (X 2.5 at 7.35 and 11 keV)
  - Split beam with 8ID-D side-bounce Si (220) for WAXPCS operation at 7.35 keV or 11 keV with Si (111).



SMALL-ANGLE SCATTERING XPCS AT 8-ID

- 8-ID-I Station Features
  - Ge(111) monochromator horizontal
  - 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**, new Pixel-Array Detector (Lambda, 2 kfps, UFXC32, Rigaku UHSS-500)



**In 2014, when I joined 8-ID, our Fast CCD ran at 100 Mpixels/s, with a kinematic mode of 1 kfps for 100x1000 pixels.

DUAL COUNTER PADS ENABLES NEW MODES OF OPERATION SUCH AS DEAD-TIME FREE OPERATION, TWO-FRAME MODE

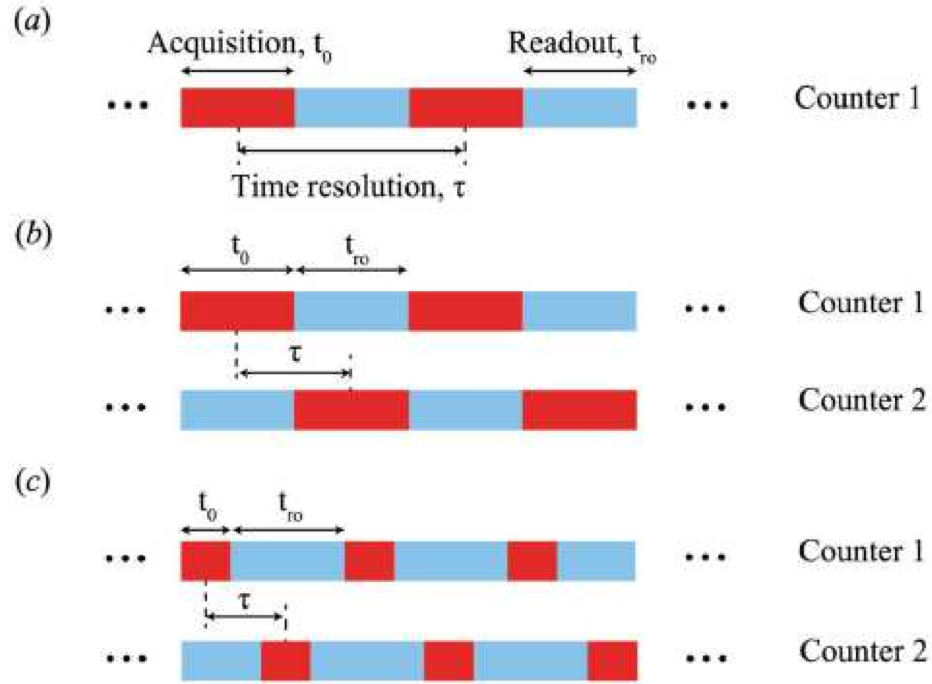


Figure 1
Time infrastructure of (*a*) continuous acquisition with one counter per pixel, (*b*) dual counter acquisition discussed in this study with no readout dead-time between the frames and (*c*) future upgrade of dual counter acquisition where the separation between the frames is smaller than the digitization time associated with each counter.

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

UFXC32K TESTS ON 8-ID-I: TWO COUNTERS DEADTIME FREE.

Test chip from Krakow University
Digital Hybrid PAD
128 x 256 75 μm pixels
320 μm thick Si sensor
2 counters, here timed for deadtime free operation
2 bit operation tested up to 50 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

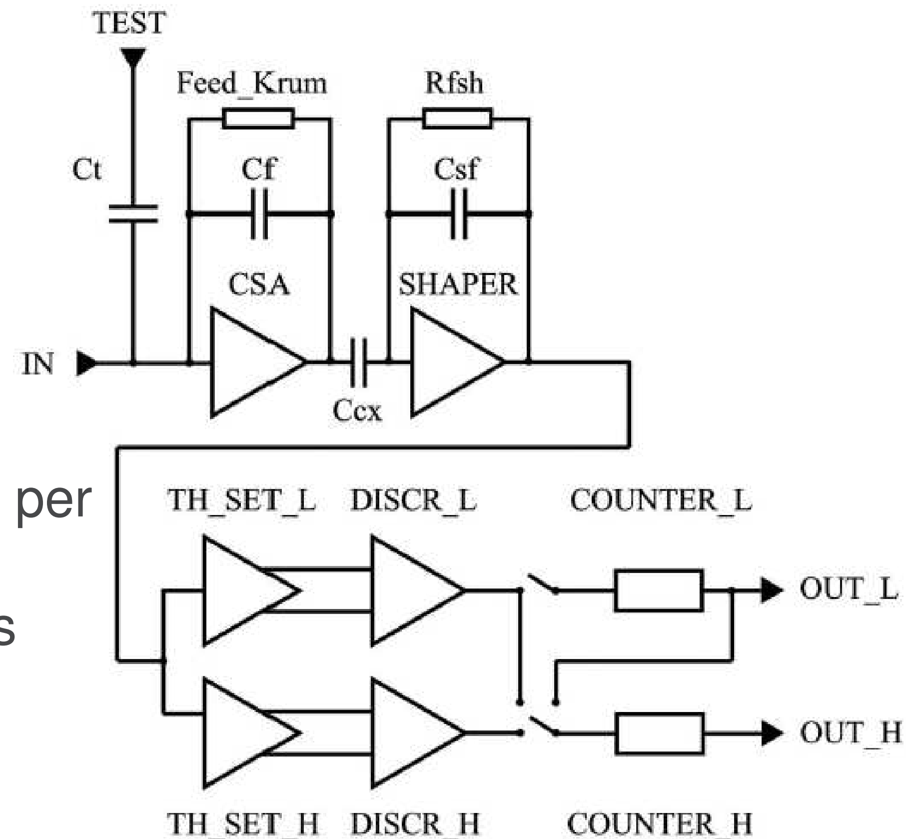


Figure 3
Schematic of the readout circuit of a single pixel.

AN XPCS EXAMPLE: UFXC32K TESTS ON 8-ID-I.

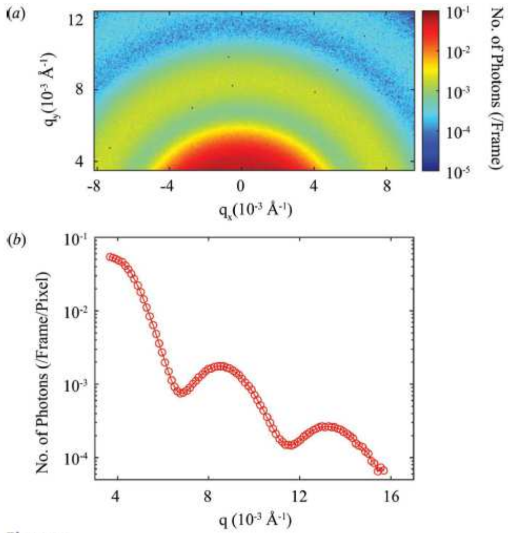


Figure 5
(*a*) Time-averaged scattering from the latex nanoparticle suspension. The scattering intensity is indicated by the logarithmic color bar. (*b*) Azimuthal average of Fig. 5(*a*).

70 nm latex spheres
in glycerol

Prototype detector now used in beamline operation at 50kHz. Data below at 12 kHz.

Data is sparsified saving time in the data saving.
Recently used detector in a novel burst mode with 830 ns frame period.

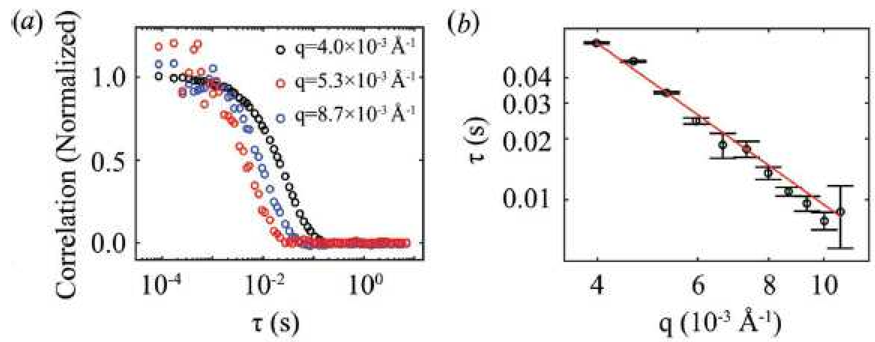
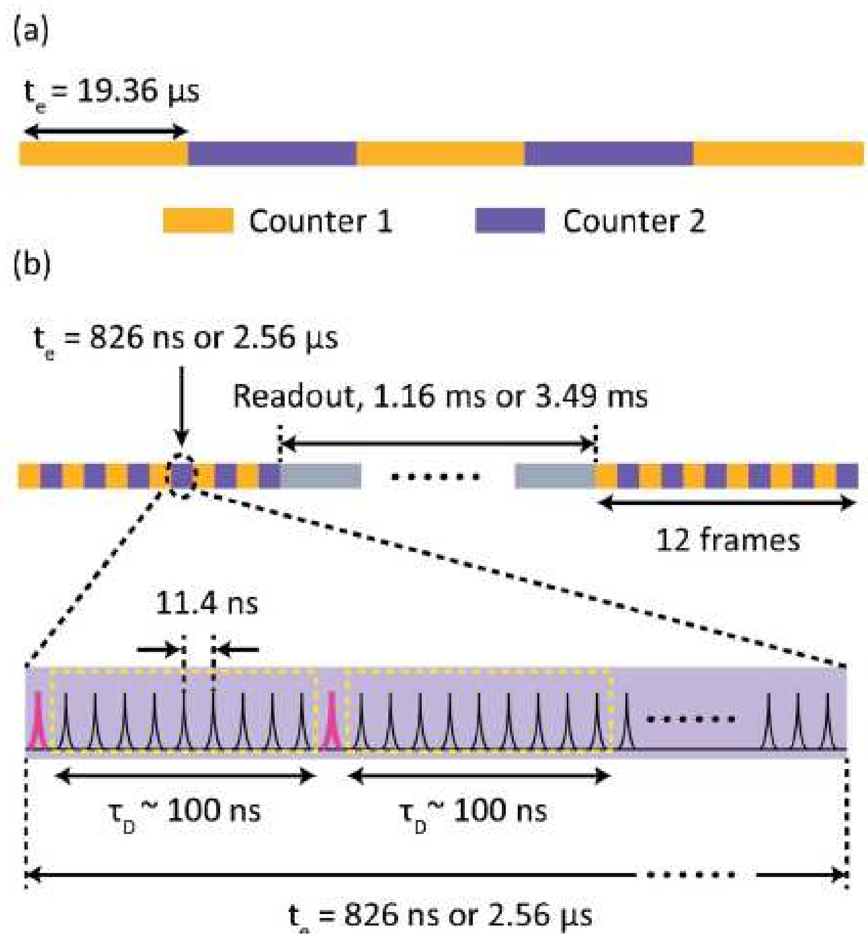


Figure 6
(*a*) Dynamics of latex nanoparticles indicated by $g_2(\tau)$ at different q. (*b*) Decorrelation time $\tau(q)$ *versus* q. The red line shows the inverse-square decay of the correlation time.

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

BURST MODE TIMING

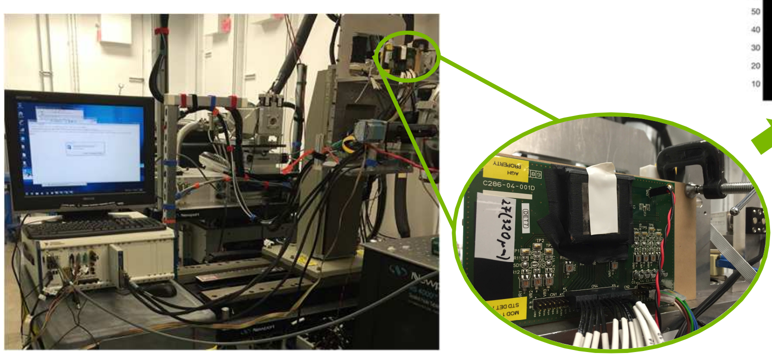


Q. Zhang et al., J. Synchrotron Rad. **25**, 1408-1416 (2018).

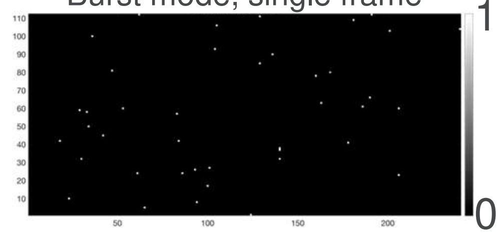
NEW BURST MODE DEMONSTRATION

UFXC pixel array detector

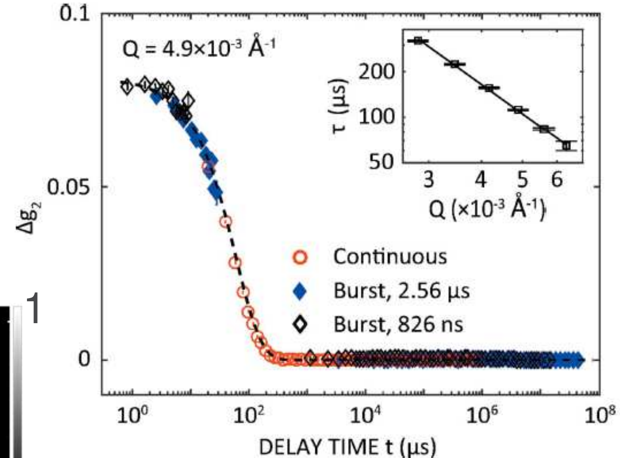
- Ultrafast x-ray camera (UFXC) ☺
  - Collaboration with AGH University
  - 128 × 256 75-µm pixels, 2 bit counter
  - World-record XPCS frame rates of
    - 50/70 kHz ("continuous")
    - 1200 kHz (burst)



Burst mode, single frame



Dynamics of R=10 nm colloids in water



Q. Zhang *et al.*, J. Synch. Rad. **23**, 679 (2016)

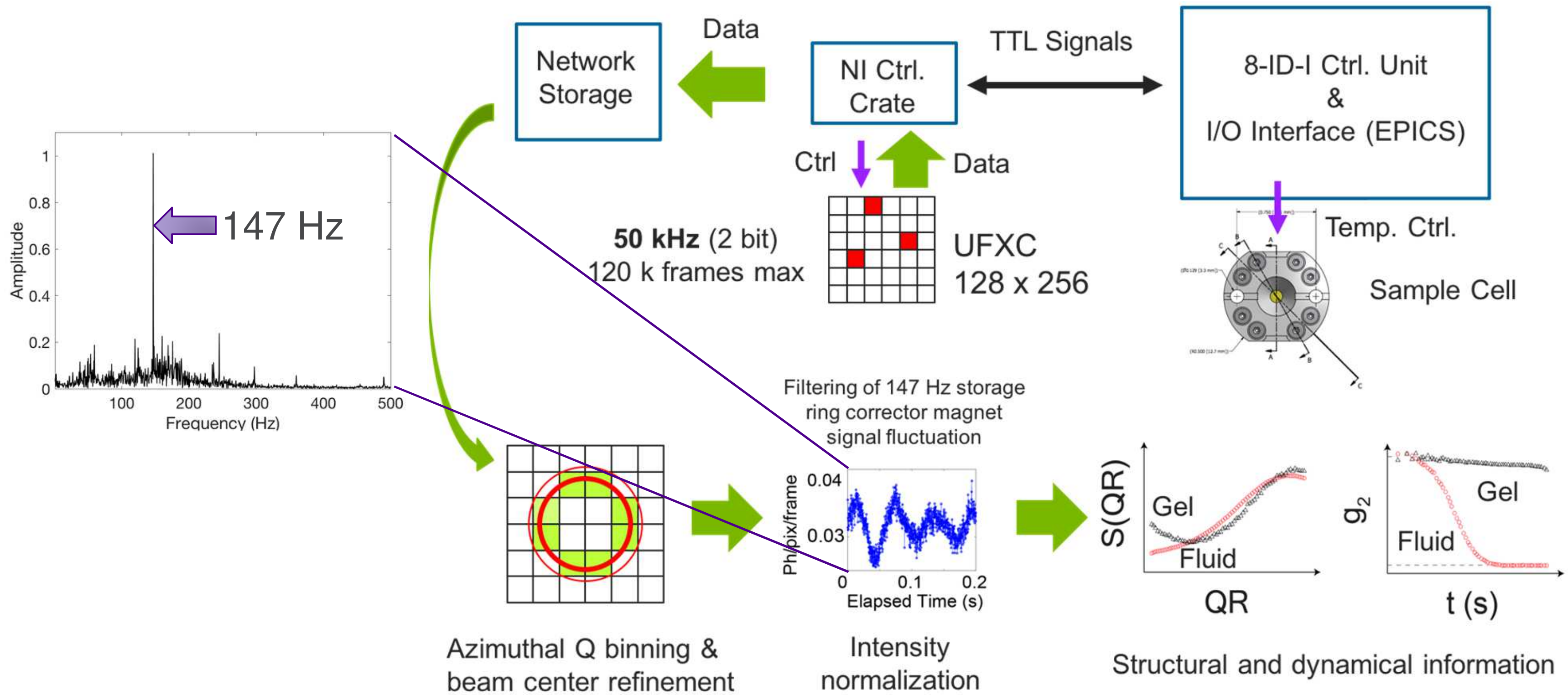
P. Grybos *et al.*, IEEE Trans. Nucl. Sci. **63**, 1155 (2016)

Q. Zhang et al., "Sub-microsecond-resolved multi-speckle X-ray photon correlation spectroscopy with a pixel array detector," J. Synchrotron Rad. **25**, 1408-1416 (2018).

NEW DETECTORS FOR FAST XPCS

UFXC pixel array detector data treatment for gel dynamics

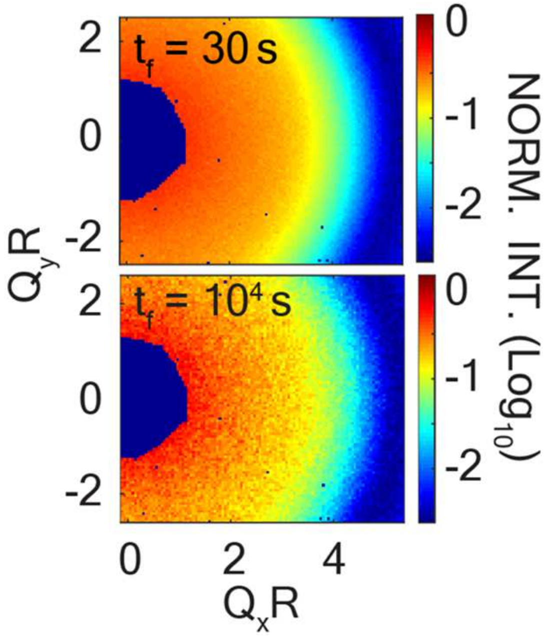
- UFXC operation



3. SCIENCE: THERMO-REVERSIBLE GELATION

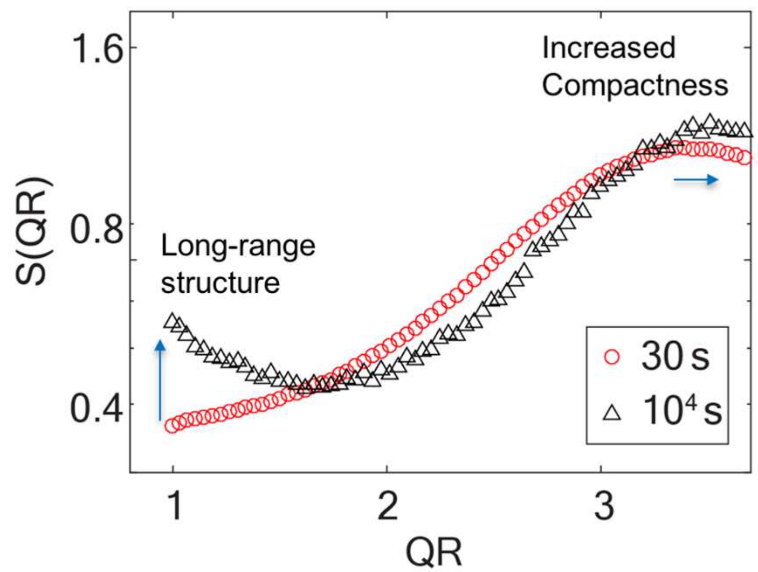
Dynamic scaling in colloidal gel formation

Time-average scattering after different formation times, t_f



ΔT = -1 K

Static structure factor S(QR)



- Dynamic arrest seen from persistent speckles after long t_f

- Gel structure spans large length scales
- Increased compactness in the attractive gel state

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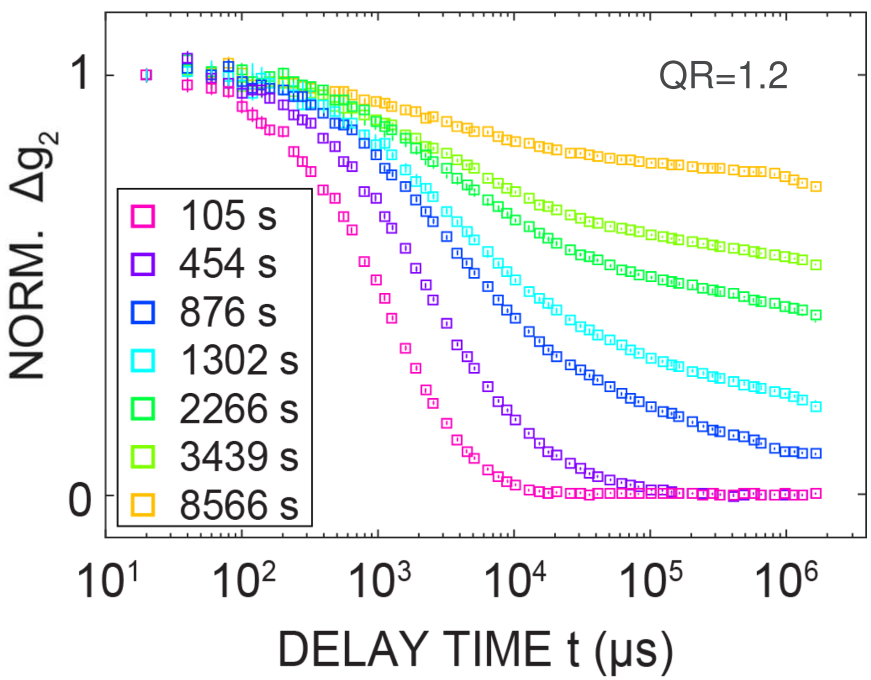
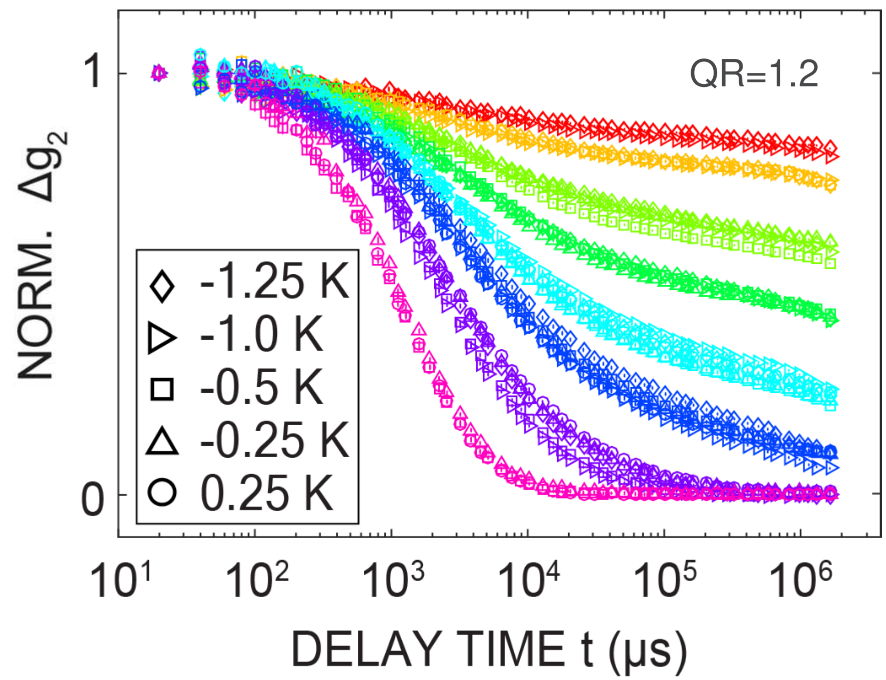
3. SCIENCE: THERMO-REVERSIBLE GELATION

Dynamic scaling in colloidal gel formation

Dynamics versus formation times after a quench to ΔT = -0.5 K



Dynamics at different formation times and quench depths



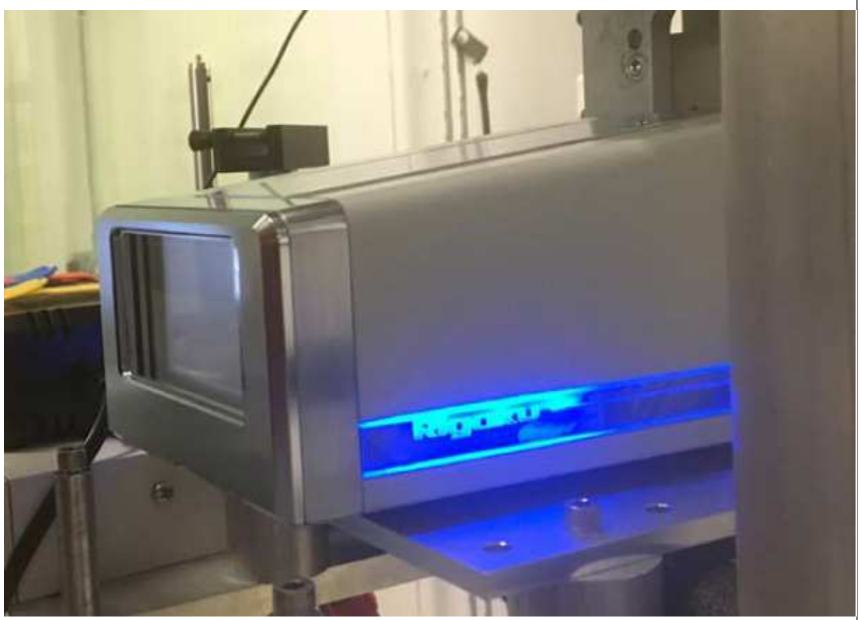
- Dynamic behavior at particular combinations of quench depth and formation time overlap
- Qingteng Zhang et al. Phys. Rev. Lett. **119**, 178006 (2017).

RECENT SCIENCE ENABLED BY UFXC32K

Note the biophysical studies of colloids in water

- *Evolution of Structure and Dynamics of Thermo-Reversible Nanoparticle Gels - A Combined XPCS and Rheology Study*, Divya Bahadur, Qingteng Zhang, Eric M. Dufresne, Pawel Grybos, Piotr Kmon, Robert Leheny, Piotr Maj, Suresh Narayanan, Robert Szczygiel, James Swan, Alec Sandy, and Subramanian Ramakrishnan, J. Chem. Phys. 151, 104902 (2019) DOI: 10.1063/1.5111521 . (Gel work)
- *α-Synuclein Sterically Stabilizes Spherical Nanoparticle-Supported Lipid Bilayers*, Peter J. Chung,Qingteng Zhang, Hyeondo Luke Hwang, Alessandra Leong, Piotr Maj, Robert Szczygiel, Eric M.Dufresne, Suresh Narayanan, Erin J. Adams, and Ka Yee C. Lee, ACS Appl. Bio Mater. (publishedFebruary 28)2, 1413–1419 (2019) DOI: 10.1021/acsabm.8b00774. (Biophysics)
- *Hard-sphere like dynamics in highly concentrated alpha-crystallin suspensions*, Preeti Vodnala, NuwanKarunaratne, Laurence Lurio, George M. Thurston, Michael Vega, Elizabeth Gaillard, Suresh Narayanan,Alec Sandy, Qingteng Zhang, Eric M. Dufresne, Giuseppe Foffi, Pawel Grybos, Piotr Kmon, PiotrMaj, and Robert Szczygiel, Phys. Rev. E 97, 020601(R) (Feb. 2) (2018), DOI: 10.1103/Phys-RevE.97.020601. (Biophysics)

RIGAKU UHSS-500K RECENT TESTS AT 8-ID-I



SPECIFICATIONS

- Active area: 77.824 x 38.912 mm^2
- Sensor material compatible with Si, CdTe, CdZnTe, GaAs etc…
- 1024 x 512 76 x 76 μm^2 pixels
- Counter length 14-bit (x2) or 28-bit (x1)
- Max count rate > 2 x 10^6 ph/s/pixels
- Max frame rate: 56 kfps (ZeroDead 2-bit / pixel) 970 kfps (2-bit BurstMode: Duty ratio 1.12%) **8500 fps** (ZeroDead 16-bit (**14-bit counter**) / pixel)

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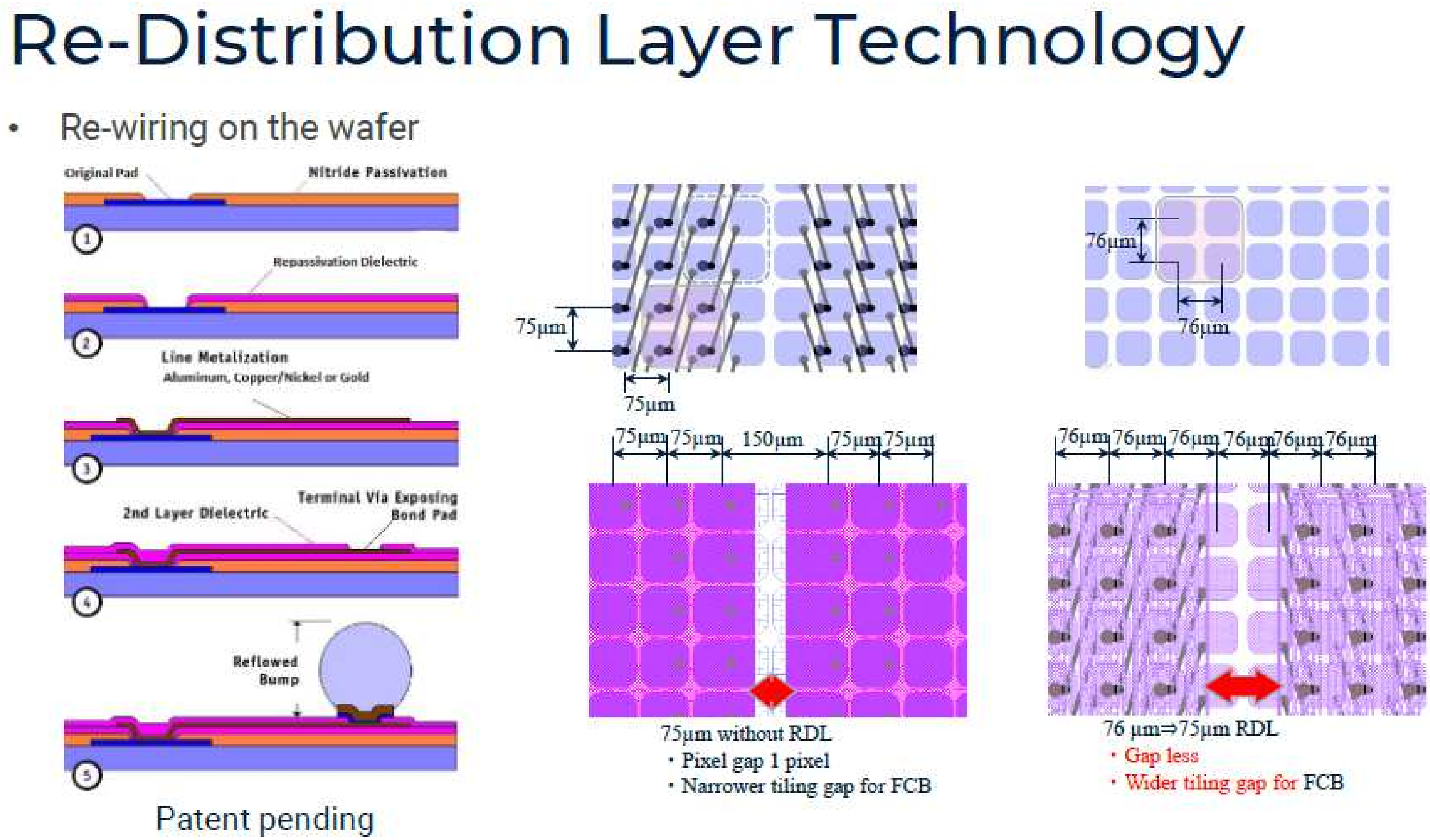
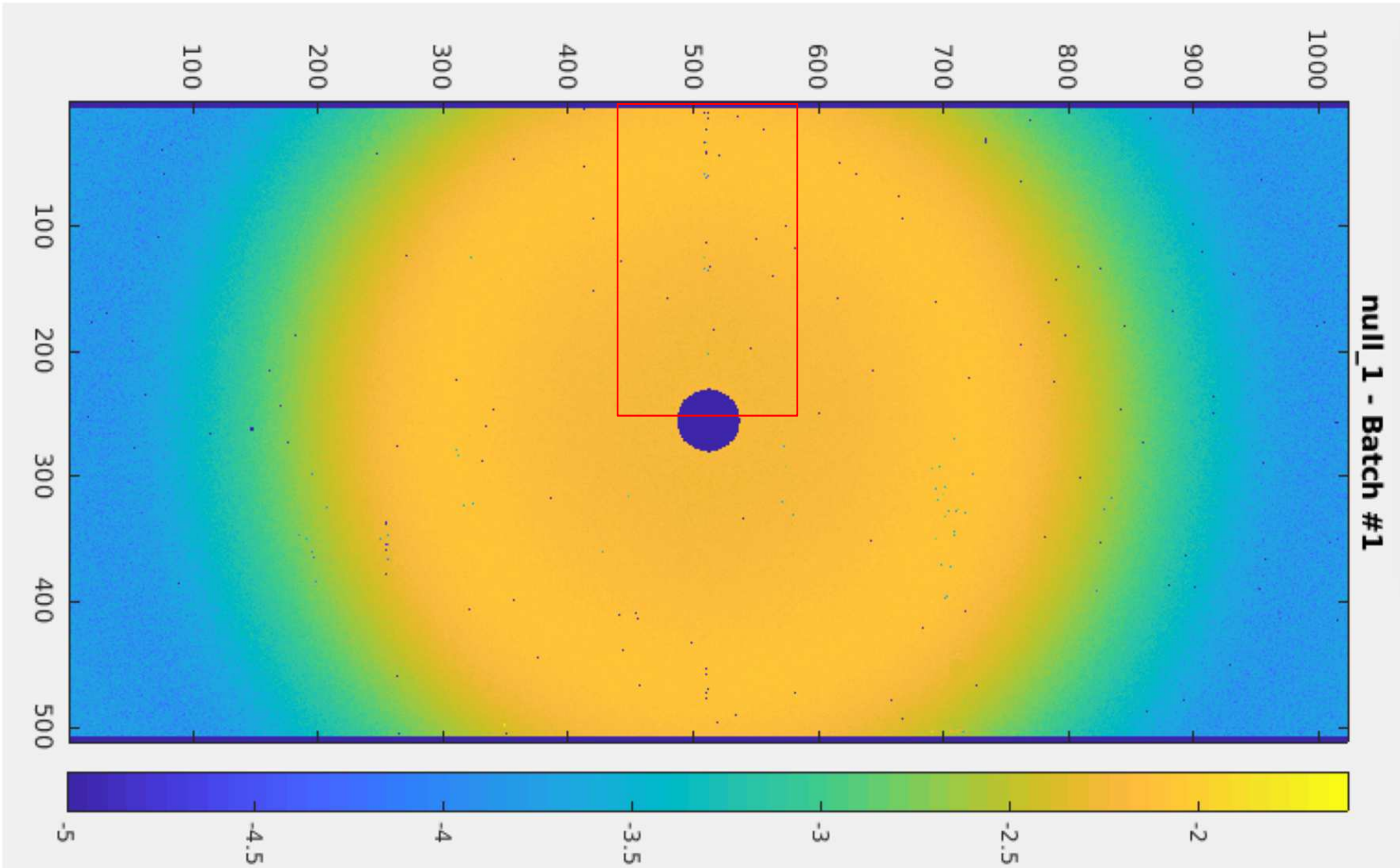
GAPLESS HYBRID PAD DETECTOR

Possibly interesting for CDI application as well

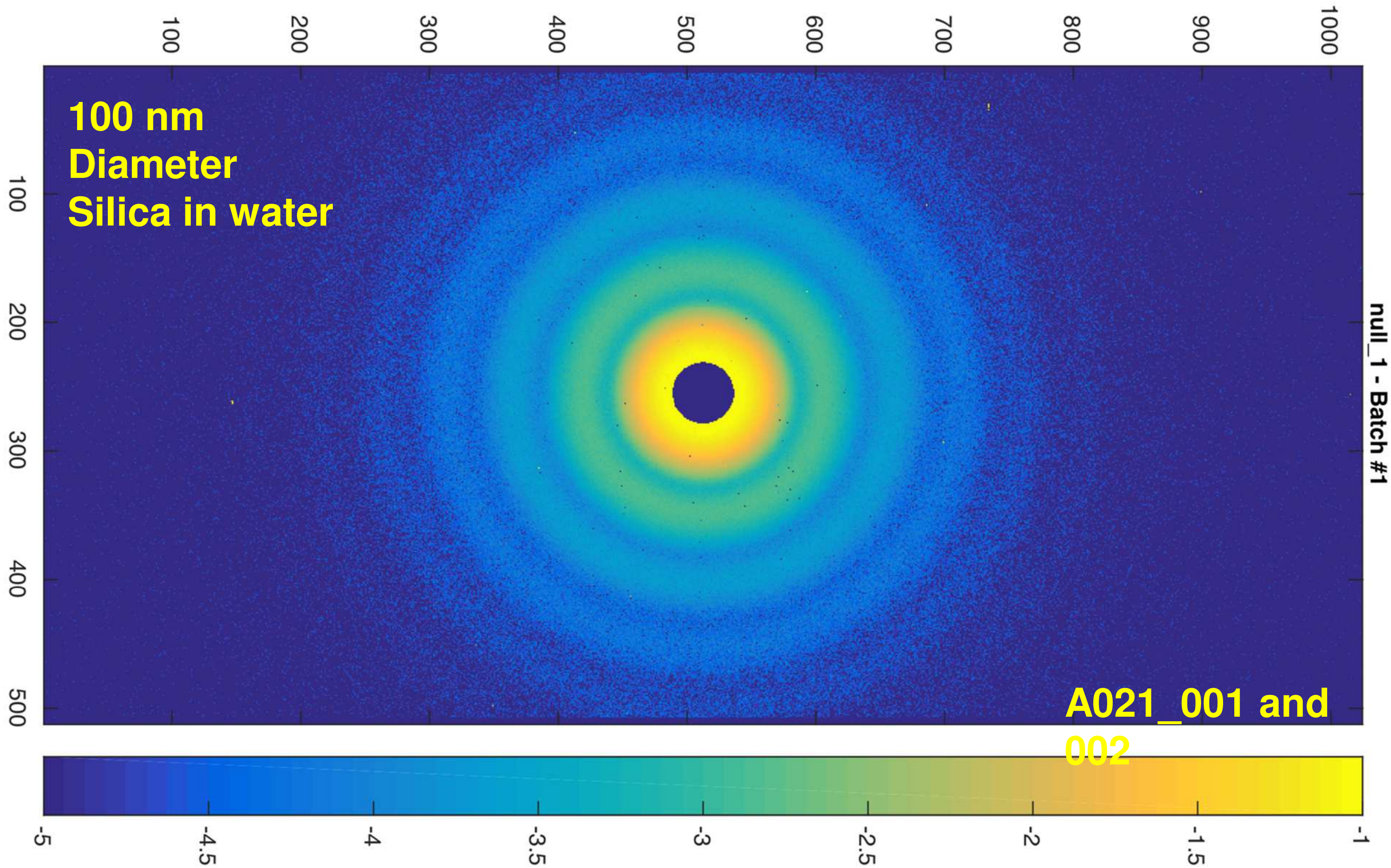


High Frame Rate X-ray Hybrid Pixel Array Detector with No Inter-chip Pixels
Yasukazu Nakaye, Yasutaka Sakuma, Satoshi Mikusu, Takuto Sakumura,
Submitted to IEEE NSS 2019.

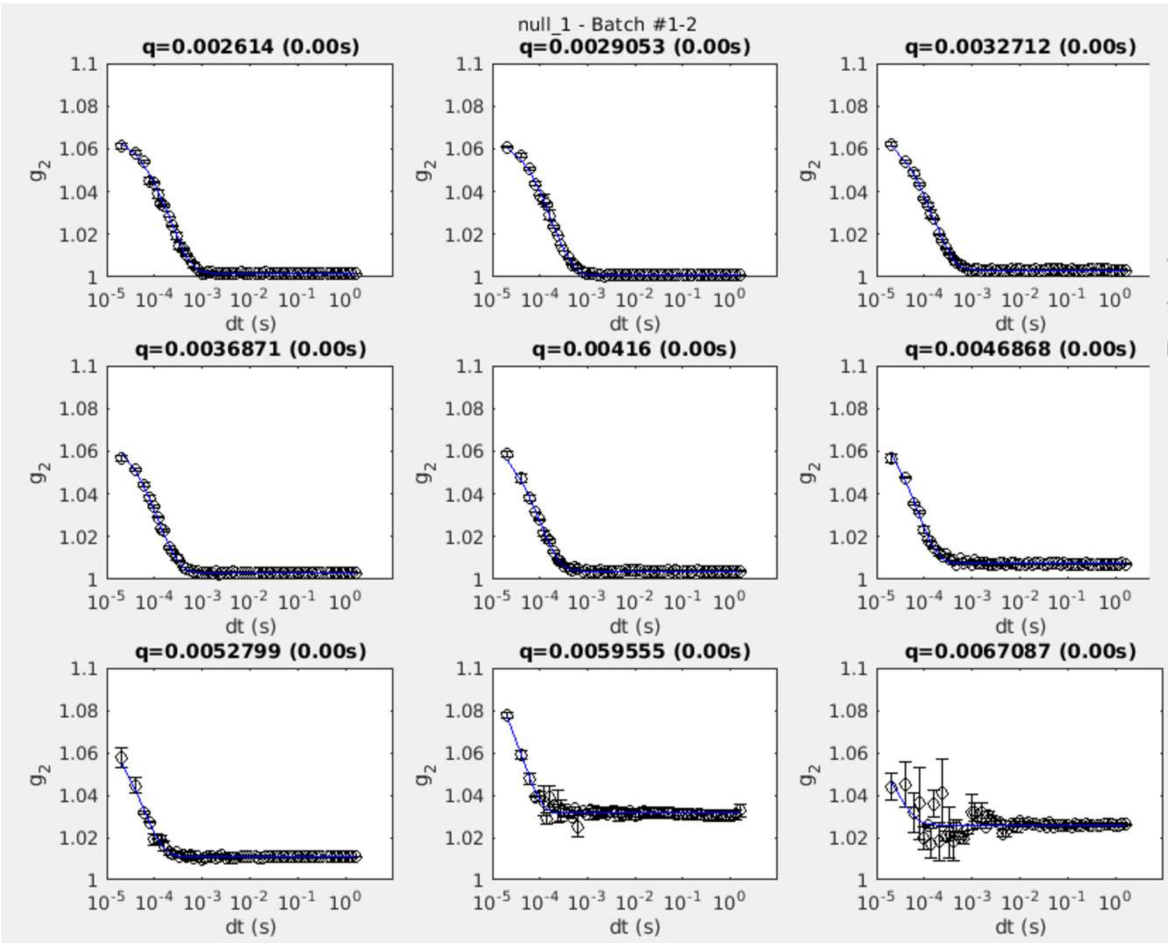
Silica in Decalin 20% vol., D = 20 nm, 33°C, Avg. 50

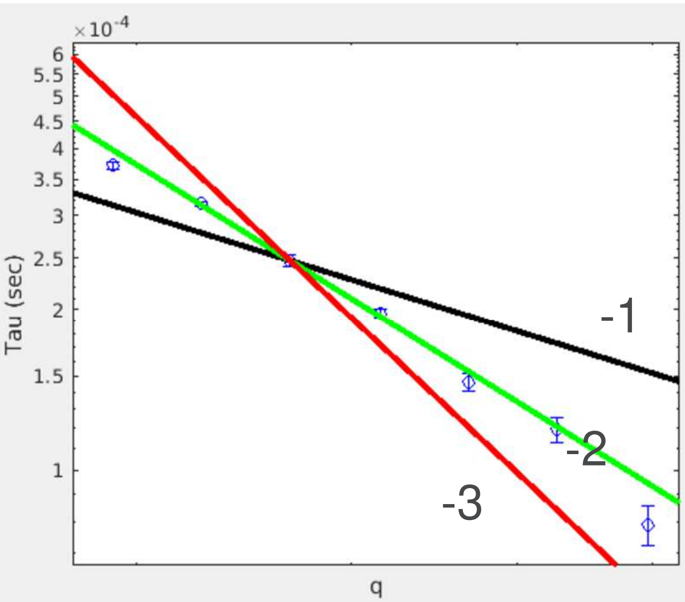


Red rectangle is one single UFXC32k module


100 nm
Diameter
Silica in water
null_1 - Batch #1
A021_001 and
002

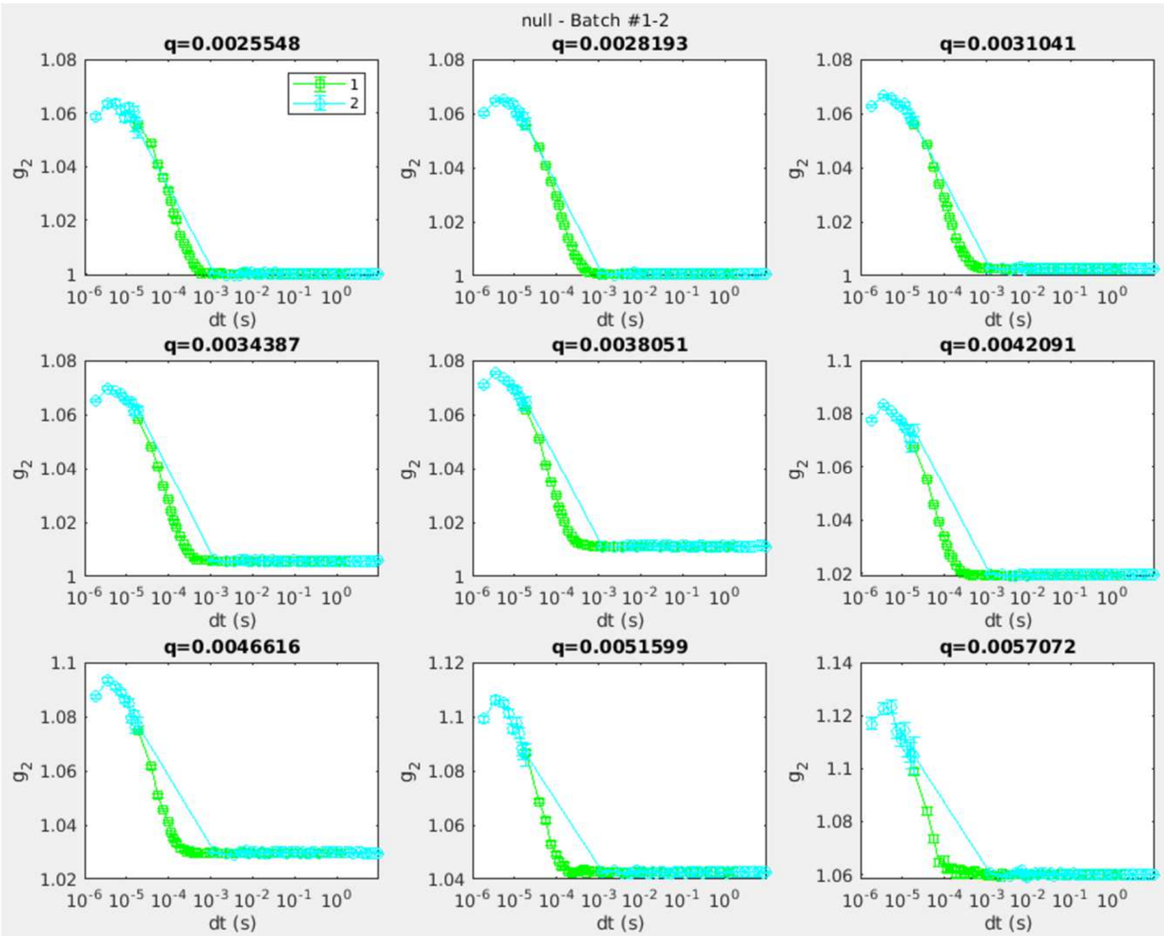
g2, A021, att = 3, Ave. of 2





g_2 for the first 6 ROI's follow tau vs. Q^{-2} quite nicely

Stitching of 50 kHz and 2 bit, 520 kHz Burst Mode



C134_ZDT_Silica_avg.mat
C138_Burst_Silica_avg.mat

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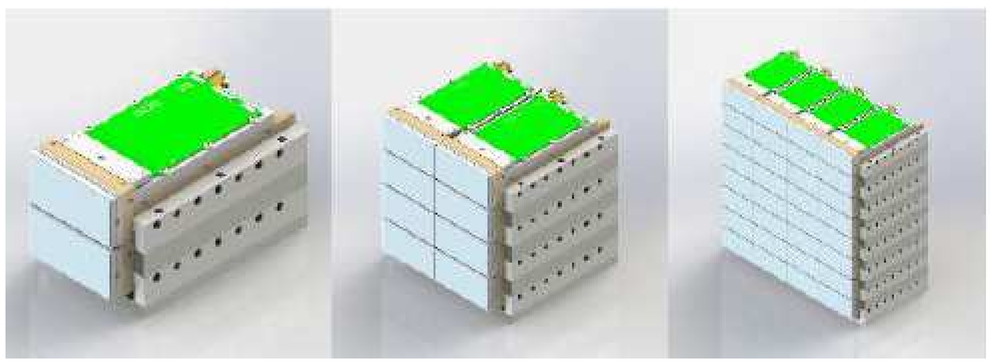
RIGAKU UHSS-500K SUMMARY

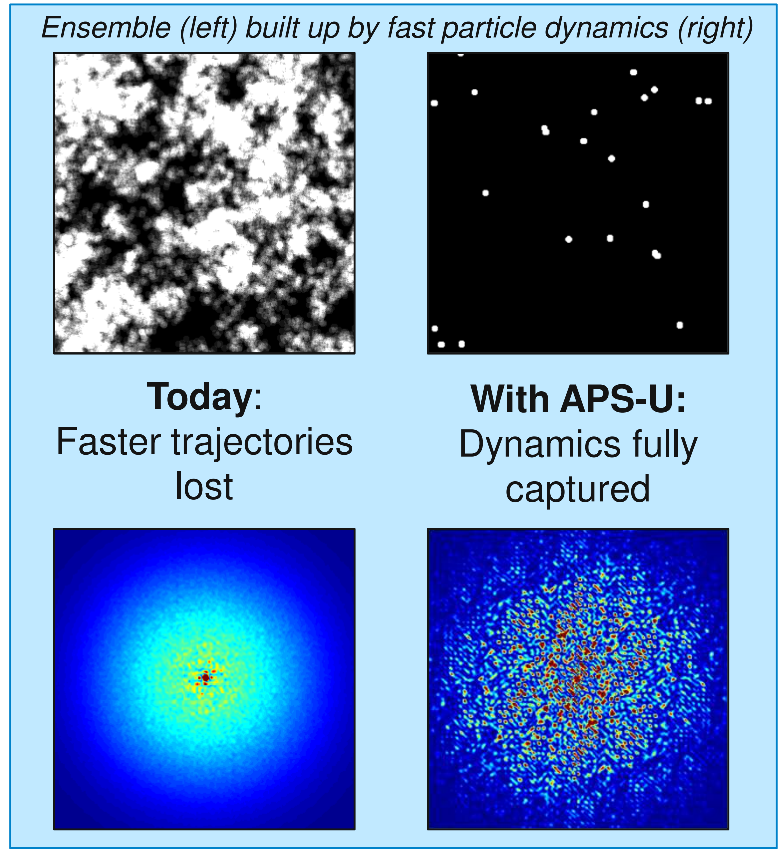
- 1 st prototype works well and was recently purchased by the APS detector pool.
- We will use it in the fall for experiments at a detector distance of 8 m.
- Some work remains on the burst mode to complete the commissioning.
- Larger system are already in planning i.e. 1M, 4M and 16 M.
- The detector is available from Rigaku.



APS-U WILL ENABLE NEW SCIENCE

- New dedicated general purpose XPCS beamline that will advance understanding of dynamics across a broad range of time and length scales
- Two instruments, SAXS and WAXS.
- Fully-leverages APS-U features:
  - World-leading coherent flux especially at higher energies

- Ring dark period around mid-2022 for a year
- Current 8-ID beamline will be rebuilt sometimes earlier.



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ACKNOWLEDGEMENTS

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  - Suresh Narayanan
  - Qingteng Zhang
  - Alec Sandy

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  - Yasutaka Sakuma
  - Takuto Sakumura
- Florida State University and Florida A&M University
  - Divya Bahadur
  - Subramanian Ramakrishnan
- Johns Hopkins University
  - Robert L. Leheny

RECENT PICTURE