Northern Illinois University

Introduction to X-ray Photon Correlation Spectroscopy

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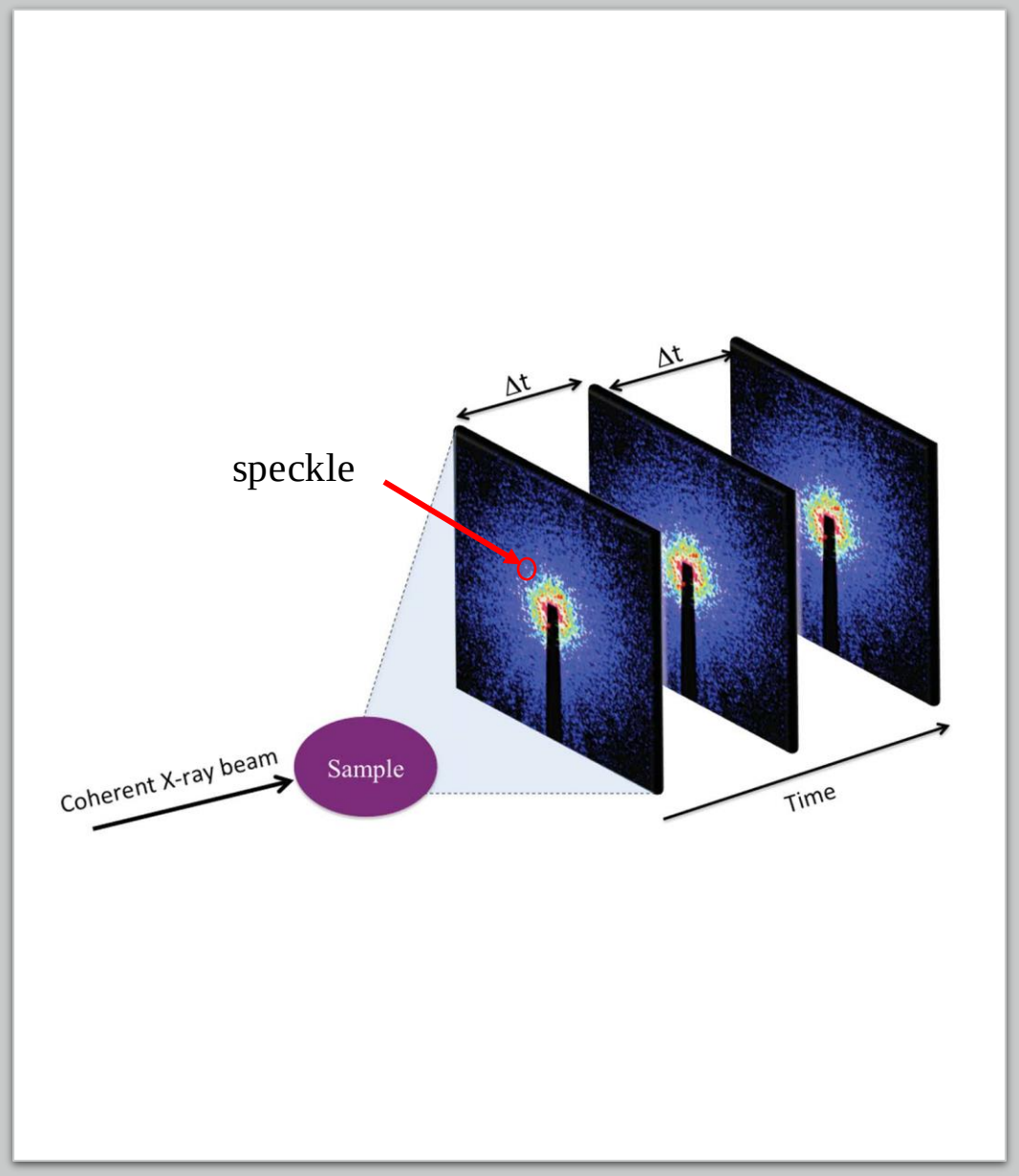
Outline

1. Overview of XPCS
2. A prototypical XPCS experiment (colloidal diffusion)
3. Review of XPCS applications

XPCS in a Nutshell

- XPCS uses a coherent x-ray beam to produce speckles
- The time correlation function of the intensity in the speckles is measured: $g_2(\tau, q)$
- This yields the spectrum of density fluctuations in the sample: $f(\tau, q)$



In Equations

- The measured quantity, g_2, is the average correlation of the intensity within individual speckles over time.
- $g_2(q,\tau) = \langle I(q,t)I(q,t+\tau)\rangle / \langle I(q)\rangle^2$

- It is related to the sample intermediate scattering function
- $g_2(\vec{q},\tau) = 1 + \beta|f(\vec{q},\tau)|^2$
- $f(\vec{q},\tau) = S(\vec{q},\tau)/S(\vec{q},0)$

- This is a measure of how the sample electron density fluctuates in time:
- $S(\vec{q},t) = \int e^{i\vec{q}\cdot\vec{r}} S(\vec{r},\tau)$
- $S(\vec{r},\tau) = \langle \rho(r,\tau), \rho(0,0)\rangle_{eq}$

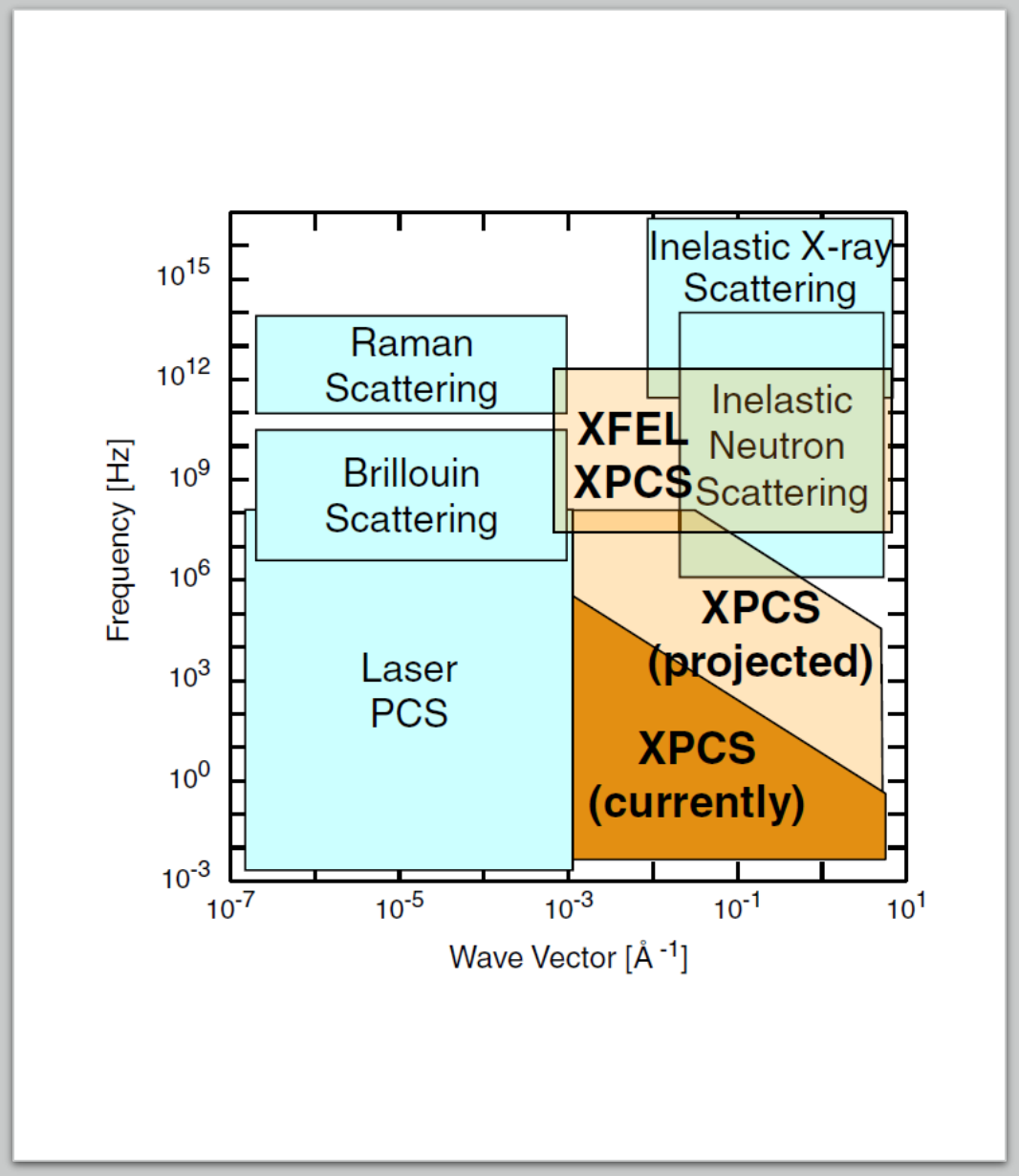
Significance of $f(q,\tau)$

- The behavior of soft materials in thermal equilibrium is governed by the Langevin equation
- $m\dot{v} + \alpha v = \zeta(t)$. Here $\zeta(t)$ is a random force
- $\langle \zeta(t) \rangle = 0$ but $\langle \zeta(t)\zeta(t+\tau) \rangle \neq 0$
- The useful information is in the correlation function!
- $\langle \zeta(t)\zeta(t+\tau) \rangle \sim \langle x(t)x(t+\tau) \rangle$
- We can measure how fluctuating thermal forces lead to molecular rearrangement in materials.

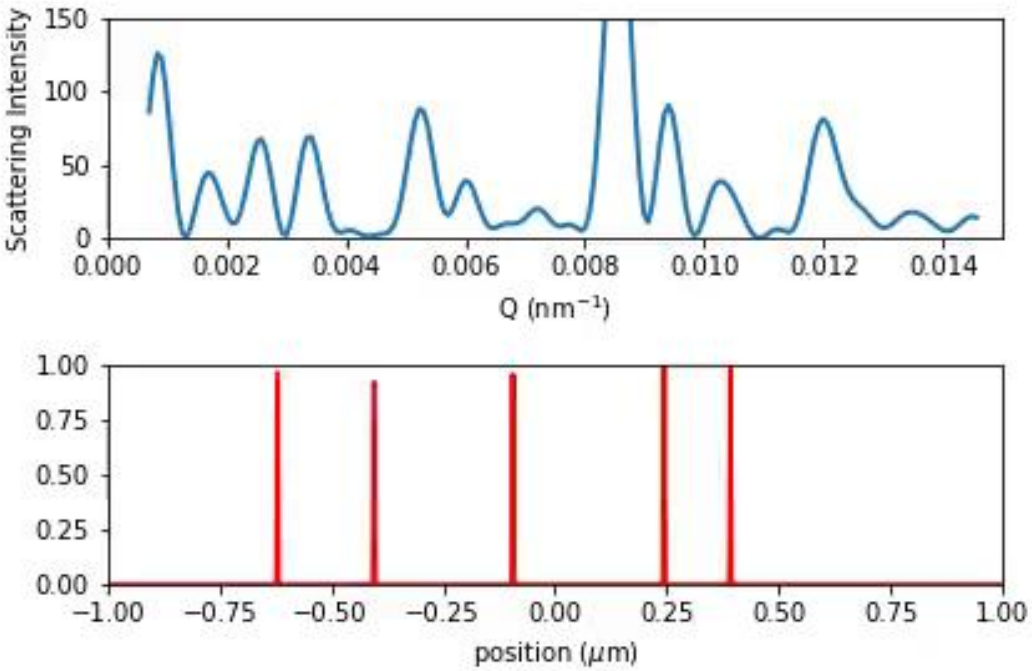
Uses of $f(q,\tau)$

- Brownian Motion and Diffusion
- Surface and Membrane Fluctuations
- Density Fluctuations
- Concentration fluctuations in a binary fluid or solid
- Fluctuations of the order parameter of a crystal.
- Atomic fluctuations about lattice positions
- Diffusion within sheer flow
- Magnetic domain fluctuations
- Aging of glasses
- Battery aging

Fluctuations about the average structure can be measured, which are invisible to incoherent x-rays.



$$I(q) \propto \left| \sum_m e^{iqx_m(t)} \right|^2$$



(1) Random intensity variations (speckle)
(2) Speckle intensity fluctuates with time
(3) Dynamics faster at higher Q

Why do you need coherence?

$$I(\boldsymbol{q}) \propto \left| \int \rho(\boldsymbol{r}) e^{i\boldsymbol{q}\cdot\boldsymbol{r}} \right|^2 = \iint \rho(\boldsymbol{r})\rho(\boldsymbol{r}') e^{i\boldsymbol{q}\cdot(\boldsymbol{r}-\boldsymbol{r}')} d\boldsymbol{r} d\boldsymbol{r}'$$

If the beam is incoherent, then this integral can be approximated as the average over a large number of independent parts of the sample so that:

$$I(\boldsymbol{q})_{incoherent} = \left\langle \iint \rho(\boldsymbol{r})\rho(\boldsymbol{r}') e^{i\boldsymbol{q}\cdot(\boldsymbol{r}-\boldsymbol{r}')} d\boldsymbol{r} d\boldsymbol{r}' \right\rangle \sim g(r)$$

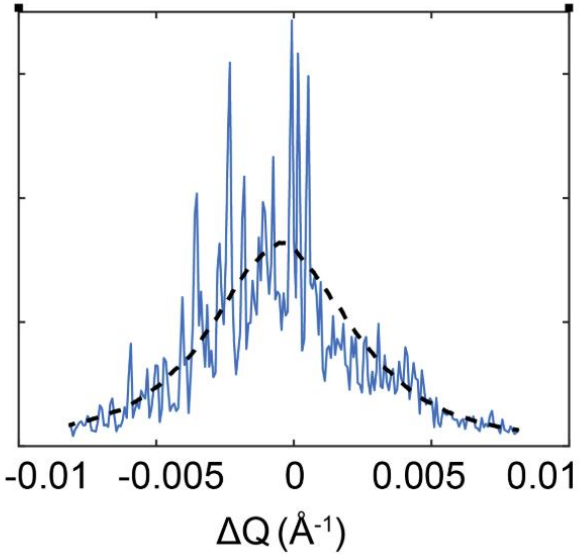
Dynamics information can be obtained, but only by perturbing the system so that $g(r)$ changes in time.
Equilibrium dynamics is invisible.

Coherent Beams

We still have:

$$I(\boldsymbol{q}) \propto \iint \rho(\boldsymbol{r})\rho(\boldsymbol{r'})e^{i\boldsymbol{q}\cdot(\boldsymbol{r}-\boldsymbol{r'})}d\boldsymbol{r}d\boldsymbol{r'}$$

But … this is no longer equal to g(r)



The intensity depends on the exact distribution of density, not the statistically averaged correlation function of the intensity.

These two intensity functions are almost the same, but the difference results in a speckle pattern superimposed on top of the average scattering for the case of a coherent beam. The speckle pattern changes in time, even for an equilibrium system.

What do we mean by coherence?

- Plane electromagnetic waves are completely coherent

$$\vec{E} = \hat{\epsilon} E_0 e^{i\vec{k}\cdot\vec{r} - \omega t}.$$

- However, most sources emit x-rays over a range of angles and energies. This spread destroys coherence. The x-rays behave like a large number of independent modes.

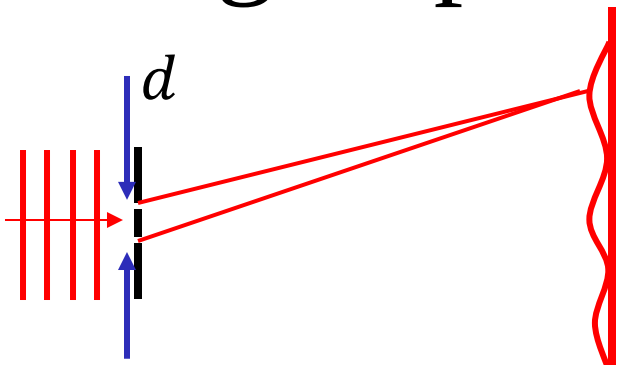
- Coherence can be regained by collimation and monochromatization.

- New sources (e.g. APSU and FELs) are coherent or partially coherent without any modification. This means that coherent x-ray scattering is the norm rather than the exception.

Transverse coherence and angle spread

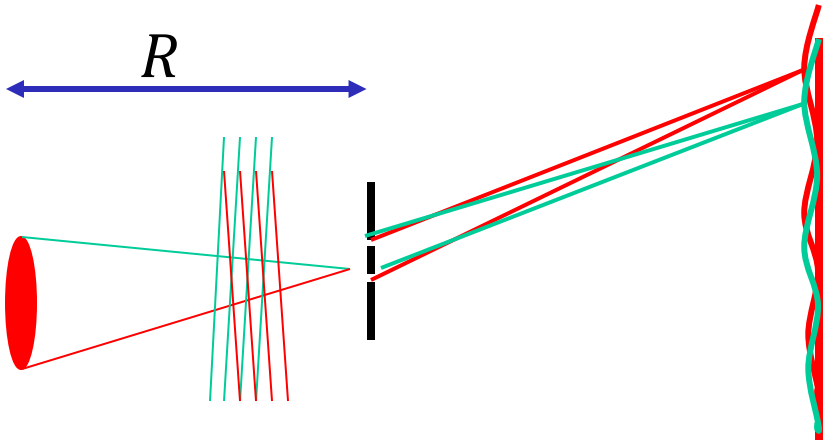
Ideal Young's double slit experiment

$$I = 2I_0[1 + \cos(2\pi d \sin(\theta)/\lambda)]$$



Real Young's double slit experiment

$$I = 2I_0[1 + \beta \cos(2\pi d \sin(\theta)/\lambda)]$$



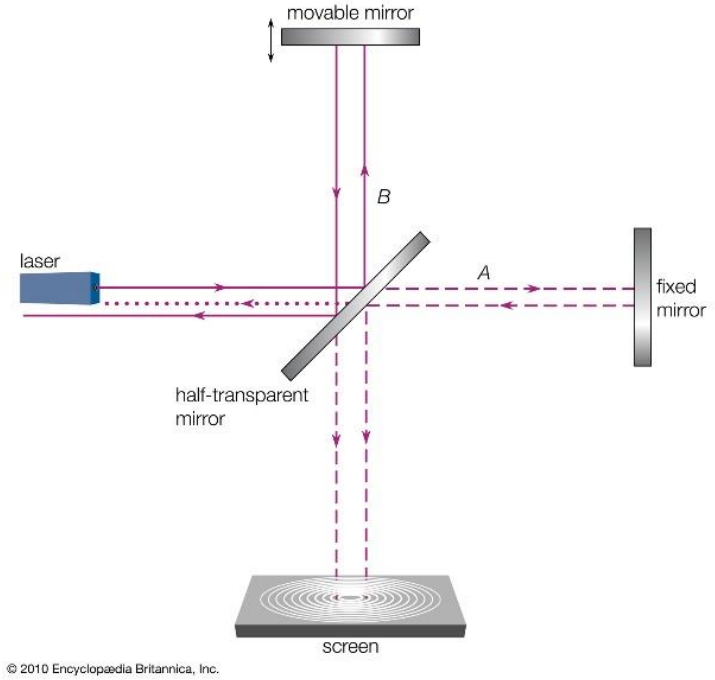
β represents the contrast.

One can define a transverse coherence length, ξ, largest distance between slits which still yields interference pattern.

For gaussian source distribution of width σ: $\xi = \lambda R/2\sqrt{\pi}\sigma$

Longitudinal coherence and energy spread

In optics this is typically understood in the context of a Michaelson Interferometer. By how much can the path lengths in the two arms differ and still produce an interference pattern?



$$\Lambda \approx \lambda(E / \Delta E)$$

Speckle Contrast

The speckle contrast is the degree of intensity difference between the minima and maxima of a speckle pattern (after intensity variations due to the structure factor have been divided out)

The contrast, β, can have a maximum possibly value of 1, and goes to 0 for an incoherent beam.

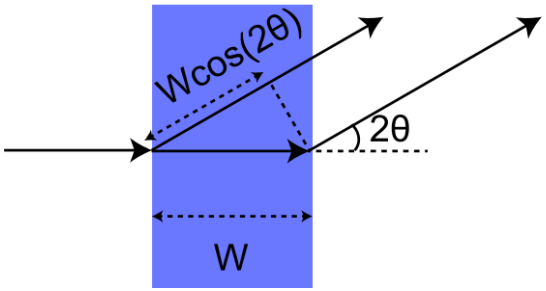
Contrast is what allows you to "see" the intermediate scattering function

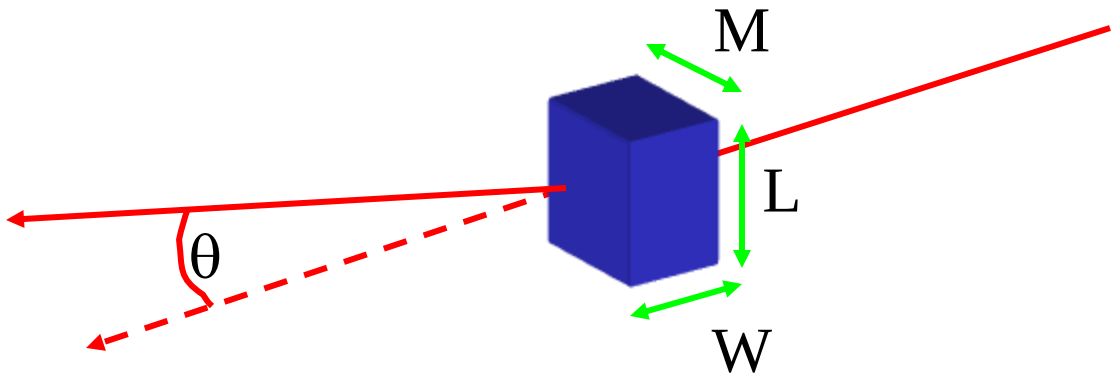
$$g_2(\vec{q},\tau) = 1 + \beta|f(\vec{q},\tau)|^2$$

Speckle Contrast

Speckle contrast is approximately the ratio of the coherence volume to the scattering volume

$$\beta = \beta = \frac{\langle I^2 \rangle}{\langle I \rangle^2} - 1 = \left(\frac{\Lambda \xi_x \xi_y}{MLW} \right) \frac{1}{2\sin^2 \theta}$$

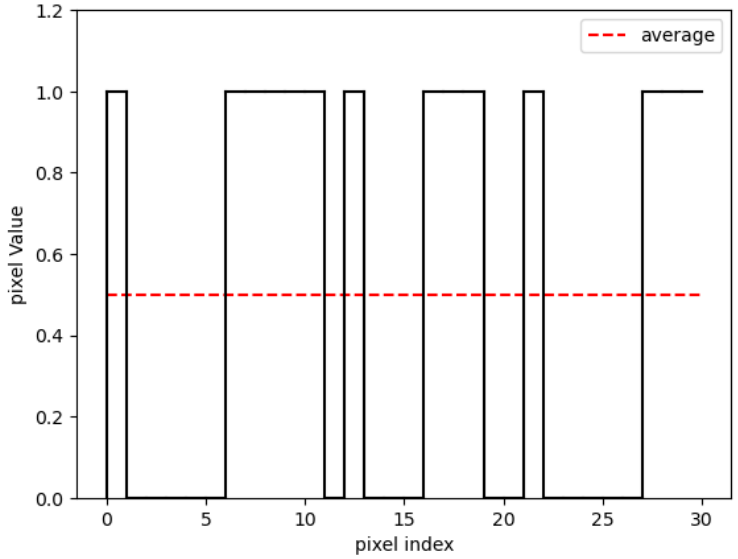




Why does $\beta = 1$?

(just for a flavor of how these calculations are done)

Consider a simplified speckle pattern, consisting of a random sequence of 0's and 1's measured as flux on a linear detector



$$\langle I \rangle = .5$$

$$\beta = \frac{\langle I^2 \rangle}{\langle I \rangle^2} - 1$$

$$I^2 \in \{1 \times 1, 0 \times 0\}$$

So $\langle I^2 \rangle = \frac{1+0}{2} = \frac{1}{2}$ and $\beta = \frac{\frac{1}{2}}{\frac{1}{4}} - 1 = 1$

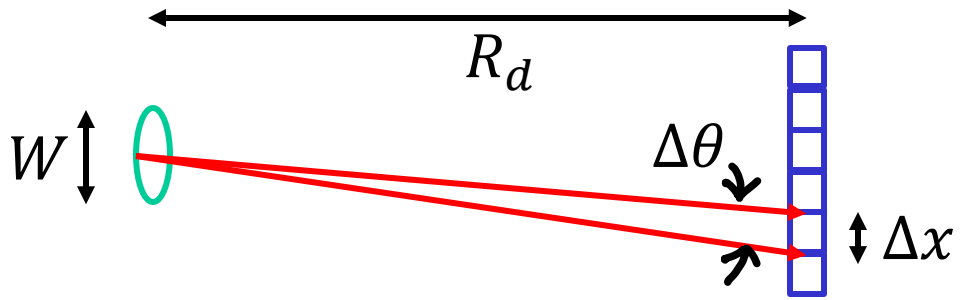
For a proper treatment of realistic speckle patterns see Joseph W. Goodman "Statistical Optics"

Speckle Size

The speckle widths are approximately the size of the diffraction pattern from a slit the size of the sample:

$$\Delta x = \frac{R_d \lambda}{W}$$

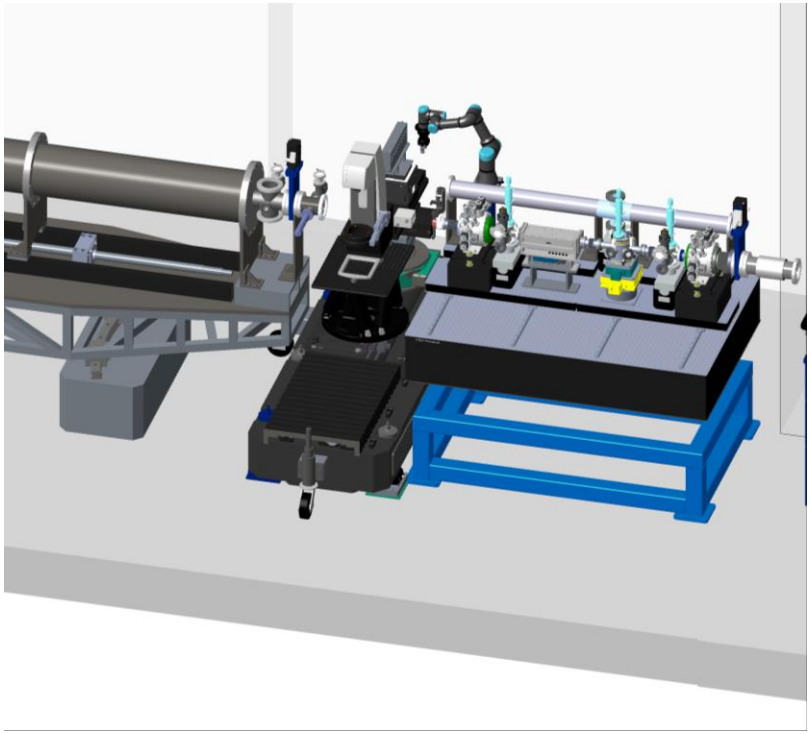
Typically, a few tens of microns

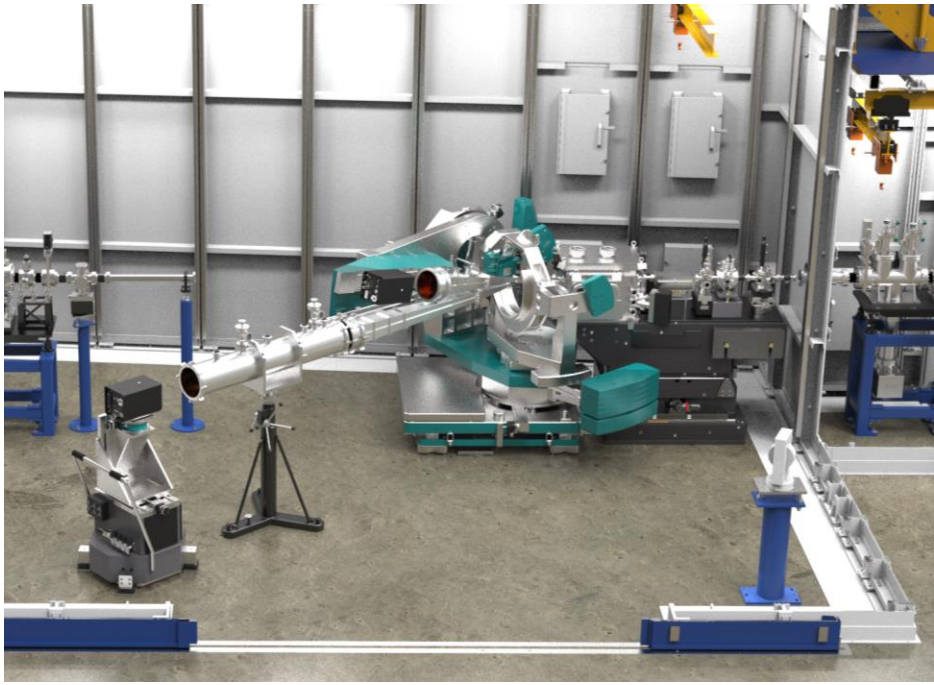


If detector cannot resolve speckles, contrast is reduced.

Sometimes the incident x-ray beam is focused onto the sample to reduce the spot size, leading to bigger speckles which better match the camera


10-ID





Coherence at the APS (8-ID-I)

$\sim 10^{12}$ Photons/Coherence Area/s

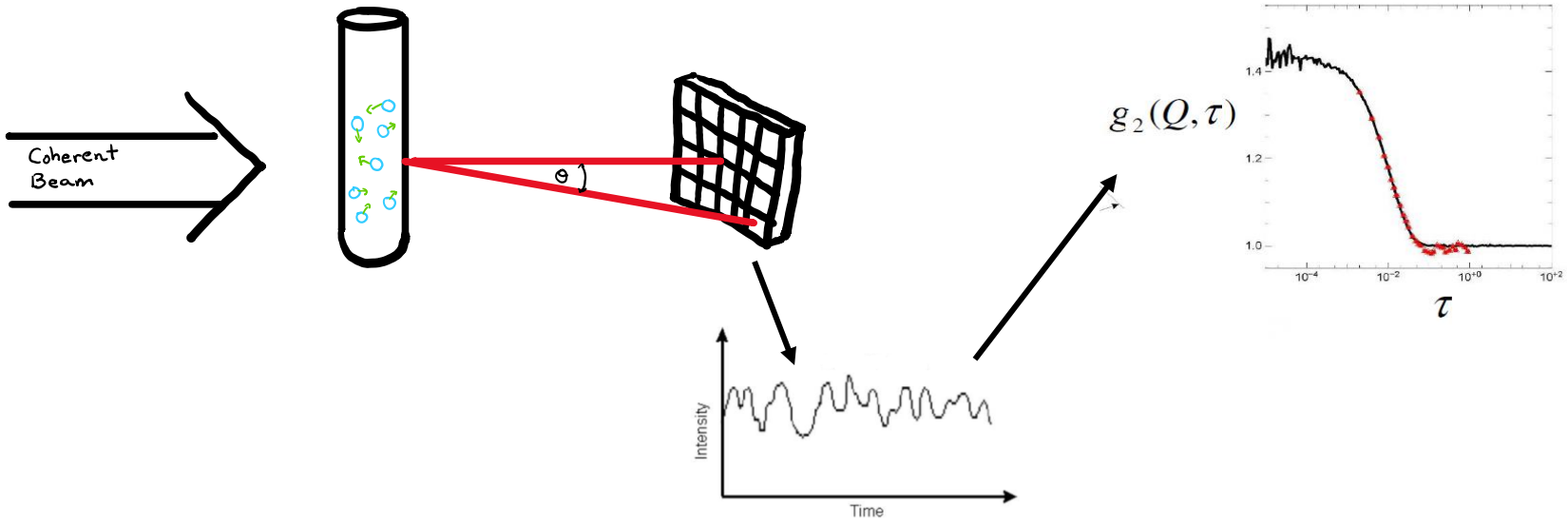
Horizontal coherence length (with slits) $\xi_x = 136$ μm

Vertical coherence length $\xi_y = 806$ μm

Longitudinal coherence length $\Lambda = 1.2$ μm

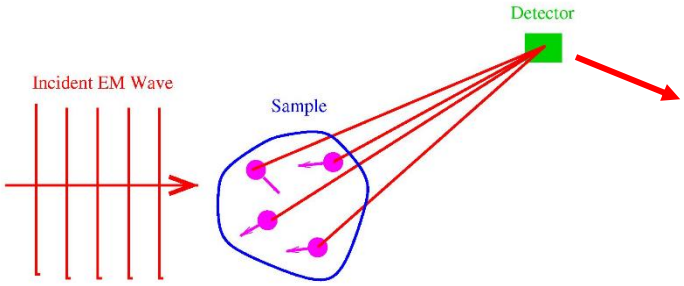
Typically, the coherent beam will be focused on the sample to yield a smaller spot size and larger speckles (Dufresne et. al, JSR, 2020 1528)

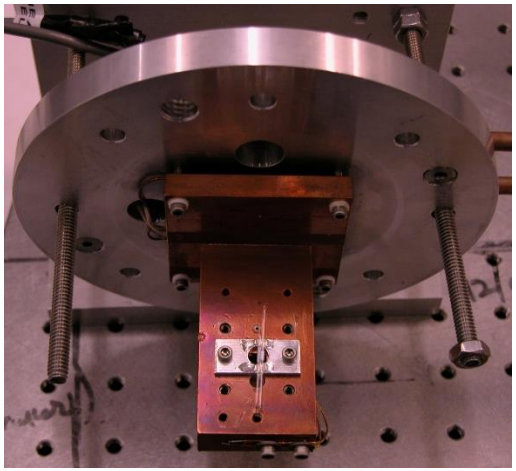
Prototypical system: diffusion of spheres in a fluid (Brownian Motion)

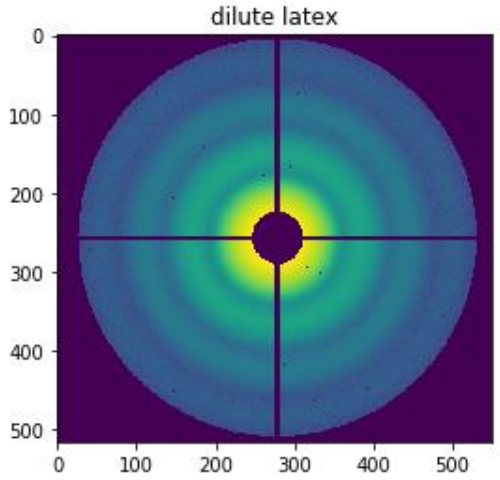


$$g_2(Q,\tau) = \frac{\langle I(Q,t)I(Q,t+\tau)\rangle}{\langle I\rangle^2} = 1 + \beta|f(Q,\tau)|^2$$

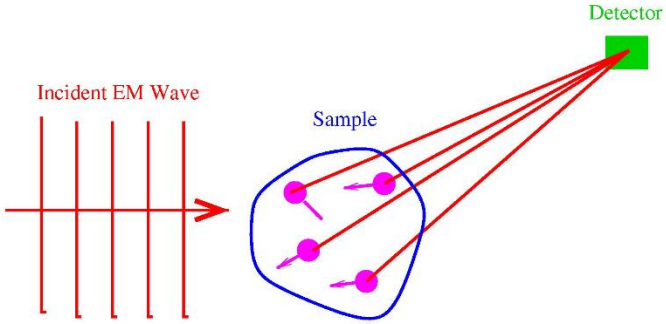
- Look at XPCS from colloidal suspensions of 67 nm radius latex beads in glycerol
- Compare XPCS for dilute and concentrated colloids.
- 2 concentrations in glycerol (to slow dynamics) in 2 glass capillaries on separate mounting plates
- ≈ 2% vol. frac.
- ≈ 40% vol. frac.
- PRL Lurio, 2000







Point Particles



$$f(\mathbf{Q},\tau) = \frac{1}{N}\sum_{i,j} e^{i\mathbf{Q}\cdot\left(\mathbf{r}_i(0)-\mathbf{r_j}(\tau)\right)} \approx \frac{1}{N}\sum_{i} e^{i\mathbf{Q}\cdot\left(\mathbf{r}_i(0)-\mathbf{r}_i(\tau)\right)}$$

Take equilibrium average assuming a gaussian random distribution of phases*:

$$\left\langle \sum_i e^{i\mathbf{Q}\cdot\boldsymbol{\Delta}\mathbf{r}} \right\rangle = e^{-\frac{1}{2}\left\langle(\mathbf{Q}\cdot\boldsymbol{\Delta}\mathbf{r})^2\right\rangle} = e^{-\frac{1}{6}Q^2\langle\Delta r(\tau)\rangle^2}$$

*Baker-Hausdorff theorem, see Jens Als-Nielson appendix D

Particle Diffusion

One dimensional diffusion: $\langle \Delta x \rangle^2 = 2\mathrm{D}\tau$

$$\langle \Delta r^2 \rangle = \sqrt{\Delta x^2 + \Delta y^2 + \Delta z^2} = 6\mathrm{D}\tau$$

Stokes-Einstein Relation: $D = k_B T / 6\pi\eta r$

$$f(Q,\tau) = \exp(-DQ^2\tau)$$

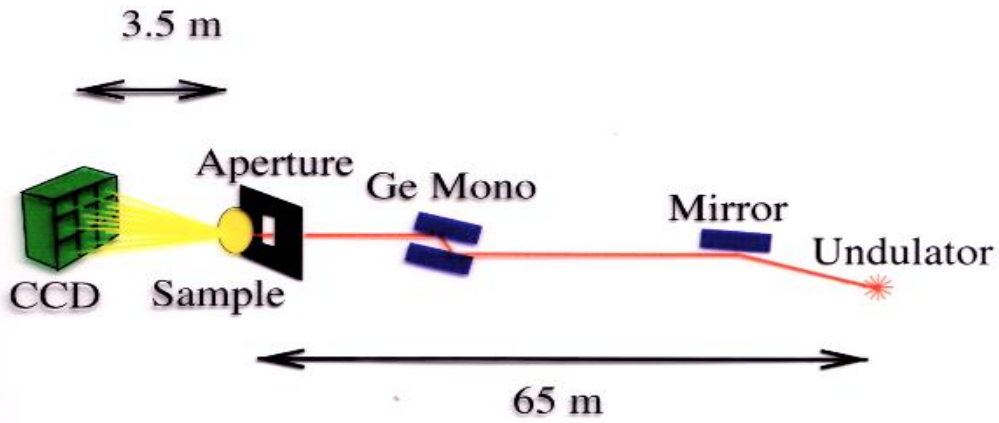
$$g_2(Q,\tau) = 1 + \beta e^{-2\Gamma\tau}$$

$$\Gamma = DQ^2$$

r radius, η viscosity, D diffusion coefficient, T temperature

- XPCS gives you the particle radius or the viscosity.
- For concentrated systems there is a variation of viscosity with length scale (caging).

Static Scattering



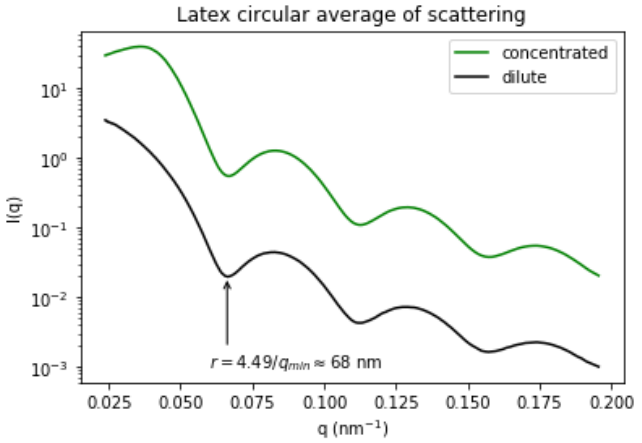
- Sphere form factor $x = qr$

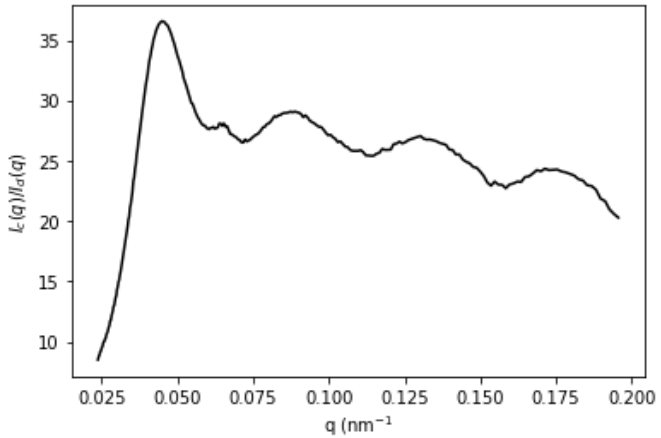
$$F(Q) = \left[3\frac{\sin x - x\cos x}{x^3}\right]^2$$

- At high concentration, scattering modified by interparticle correlations:

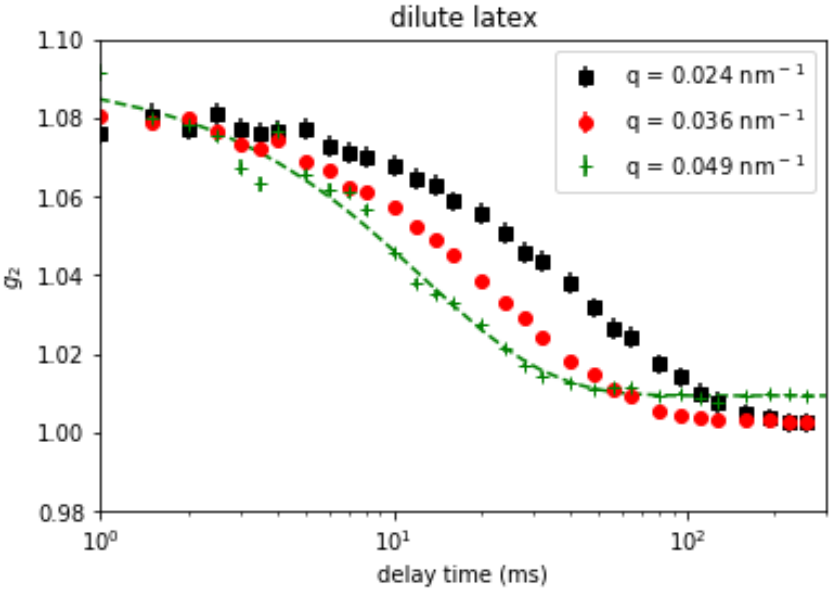
$$I(Q) \propto F(Q)S\,(Q)$$

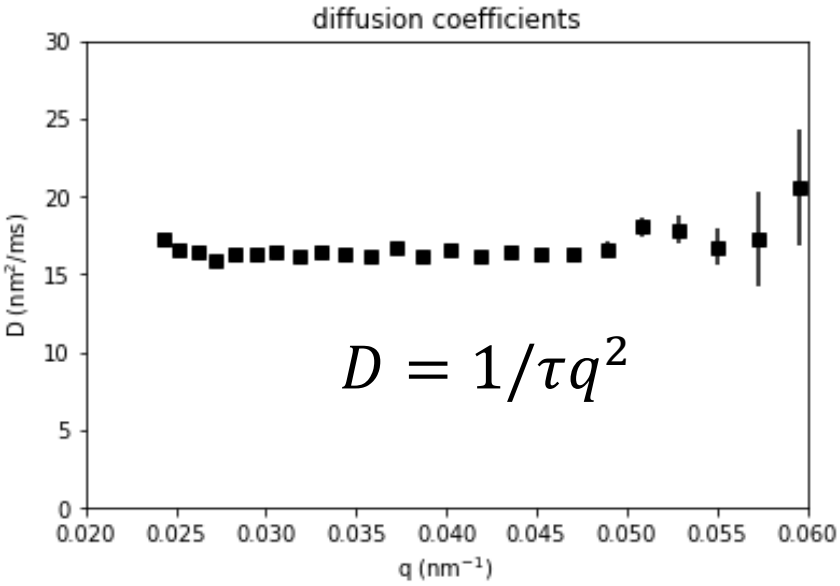
$$S(Q) \approx \left(\frac{\phi_{dil}}{\phi_{conc}}\right) \times I_{conc}/I_{dil}$$

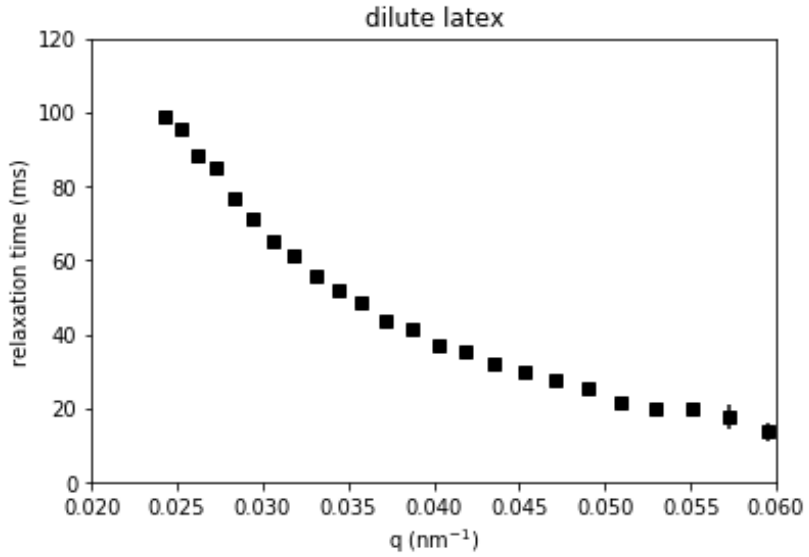




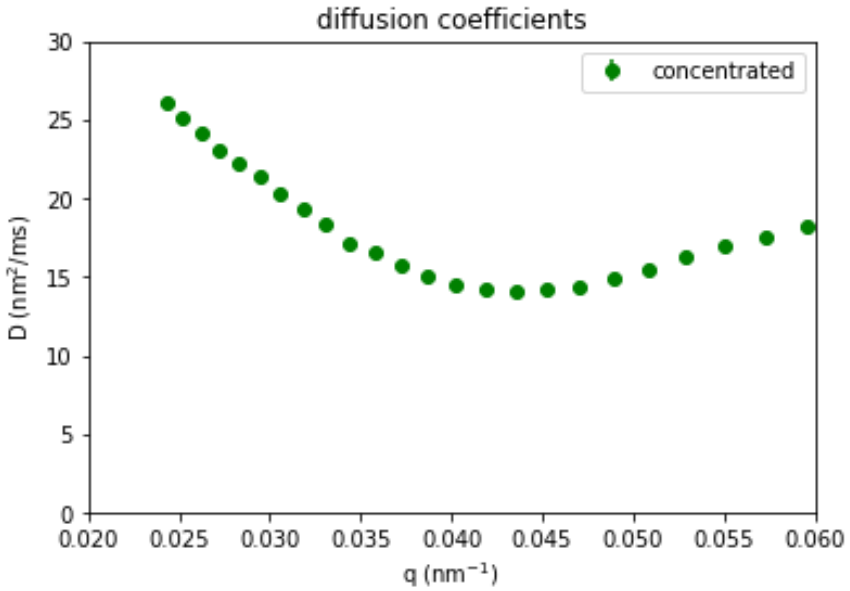
Dynamics Results: Dilute

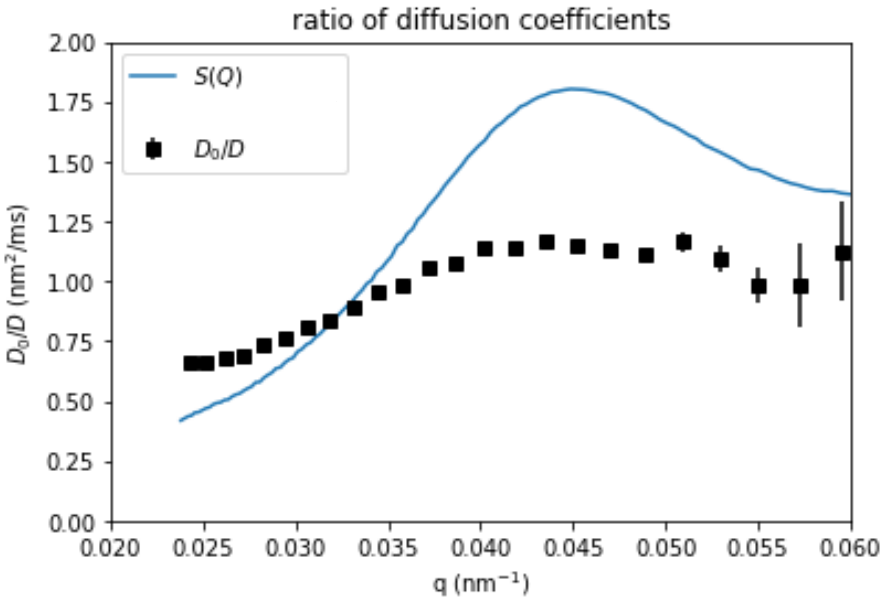






Dynamics Results: Concentrated





$$\frac{D_0}{D(Q)} = S(Q) \ldots \text{de Gennes Narrowing}$$

Assume velocities are uncorrelated but hydrodynamic flow creates correlations.

Length scale dependent deviations from diffusion tell you something new about correlated motion!

Atomic jumps in a binary alloy

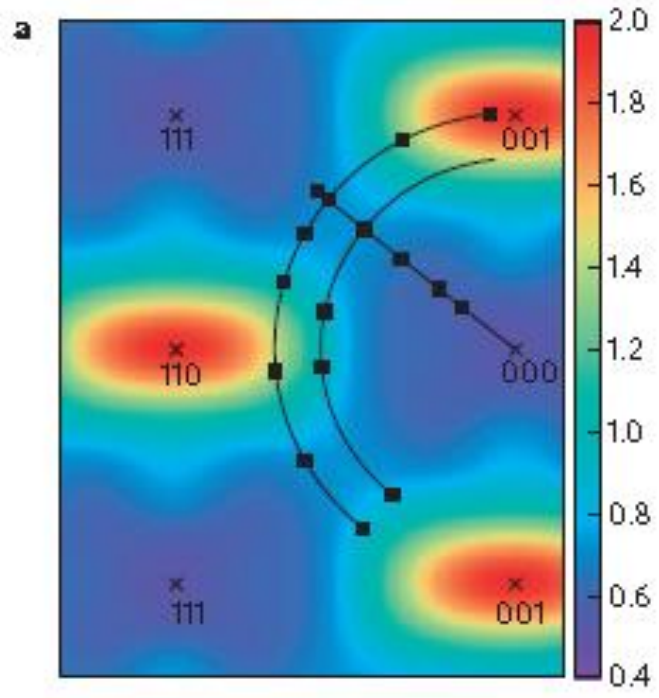
M. Leitner, B. Sepiol, L.-M. Stadler, B. Pfau, and G. Vogl, Nature Materials **8, 717-20 (2009).**

Study $Cu_{90}Au_{10}$ alloy.

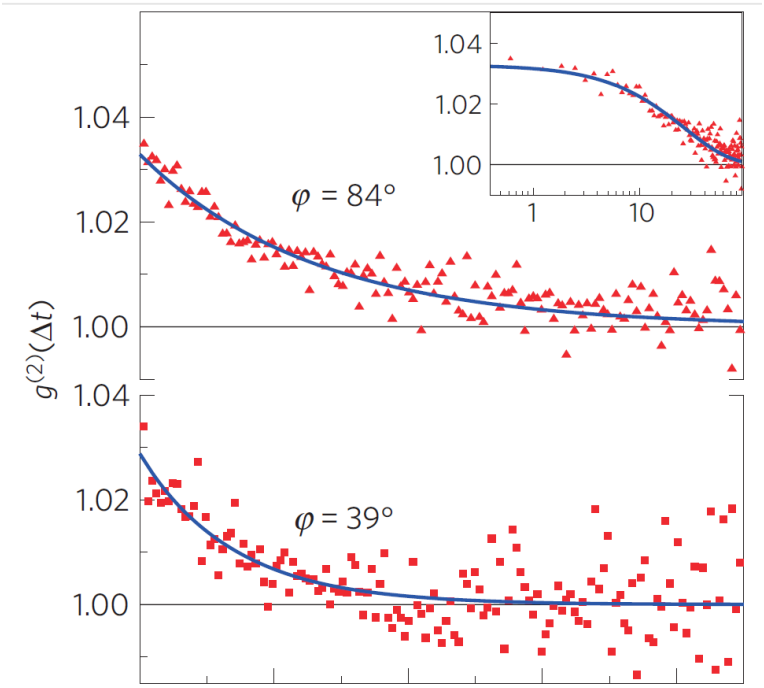
Measure XPCS from thermal diffuse scattering between Bragg peaks.

Dynamics corresponds to Cu-Au place exchange.

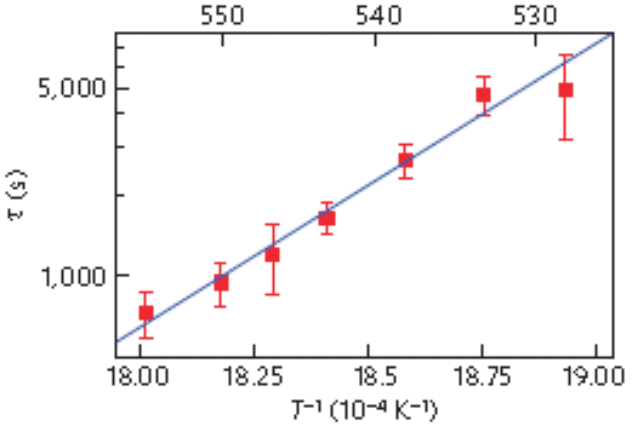
For this system: $f(q,t)$ is the F.T. of the place exchange probability



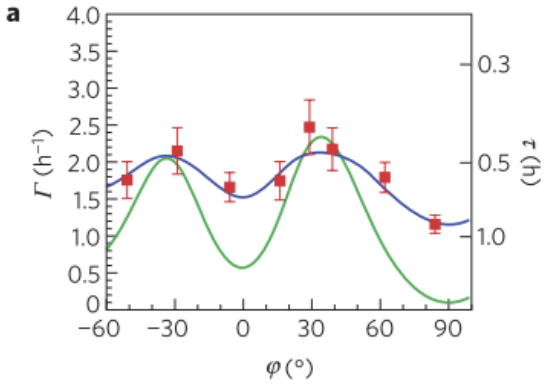
Atomic Dynamics



See simple exponential decay with dynamics varying as a function of azimuthal angle and temperature.



T dependence



Angle dependence

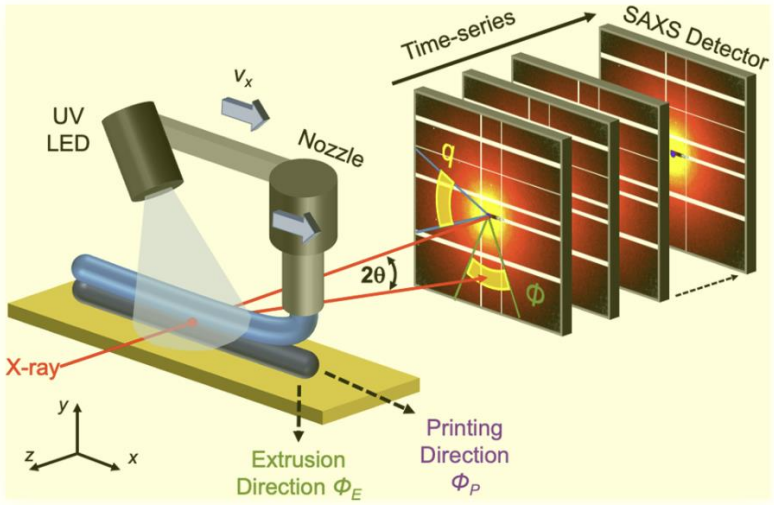
Significance

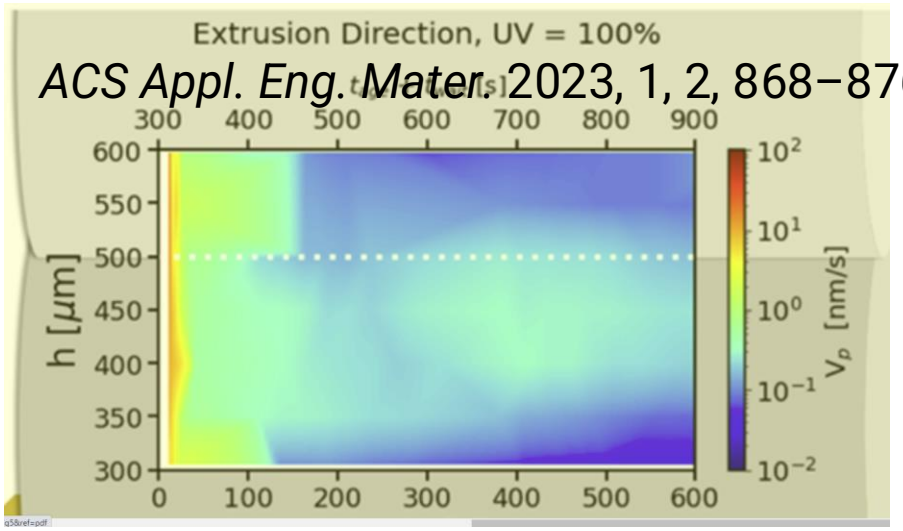
- Can find atom place exchange time (2340 s)
- Can find diffusion coefficient ($2.6 \times 10^{-24} \mathrm{m}^2 \mathrm{s}^{-1}$)
- Temperature dependence gives activation energy for a place exchange jump (2.09 eV)
- Need strong scatterer (gold) and very slow dynamics. With the APSU these sorts of experiments could become routine!

Imaging Dynamics in Additive Manufacturing

Spatial-Temporal Dynamics at the Interface of 3D-Printed Photocurable Thermoset Resin Layers

Benjamin M. Yavitt, Lutz Wiegart, Daniel Salatto, Zhixing Huang, Leonidas Tsapatsaris, Maya K. Endoh, Sascha Poeller, Manuel Schiel, Stanislas Petrash*, and Tadanori Koga*





ACS Appl. Eng. Mater. 2023, 1, 2, 868−876

Look at 2 μm Barium Sulfate tracer particles

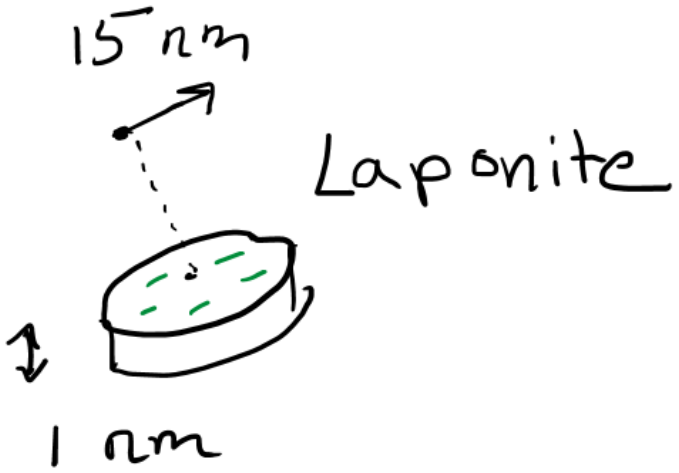
Map positional dependence of diffusion coefficient at interface between beads of resin

Beyond simple diffusion

- Diffusion: we expect $f(q,\tau) = e^{-\frac{\tau}{Dq^2}}$.
- Dense colloids $D \rightarrow \frac{D}{S(q)}$
- Consider gelation transition. (Laponite Clay)
- Dynamics have non-diffusive character
- PRL Bandyopadhyay, 2004

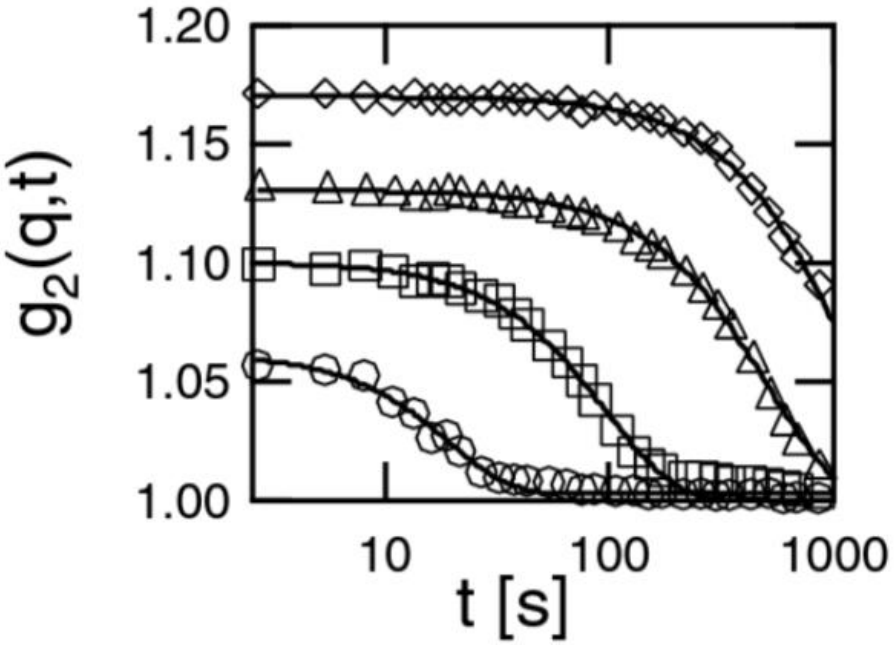
Gelation of Laponite

Dilute clay suspension $\phi > .007$ slowly gel with time over around 1000 s



Correlation functions do not show exponential decay

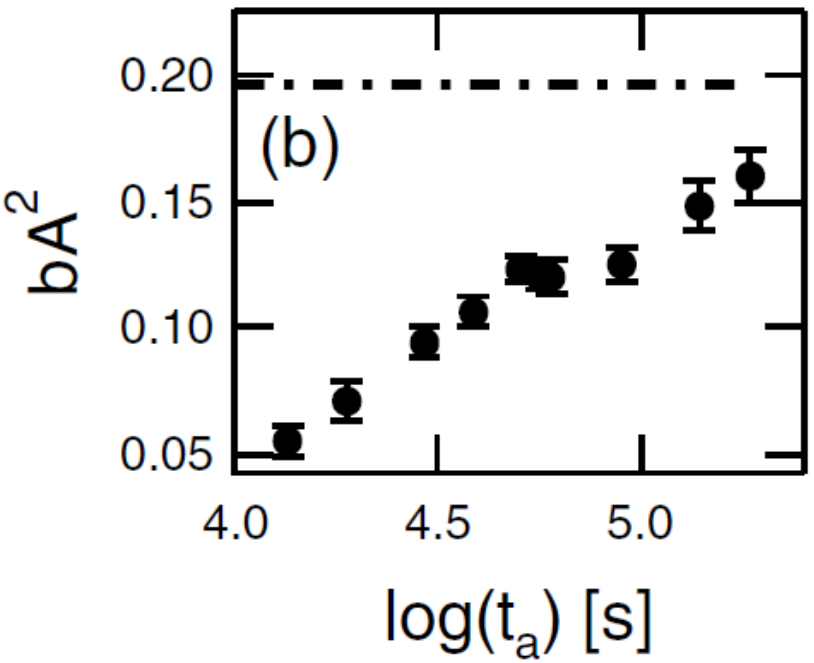
- $g_2 = 1 + b\left[Ae^{-\left(\frac{t}{\tau}\right)^{\beta}}\right]^2$



g_2 vs. time, circles: 13 ks, squares: 30 ks, triangles: 90 ks diamonds: 200 ks

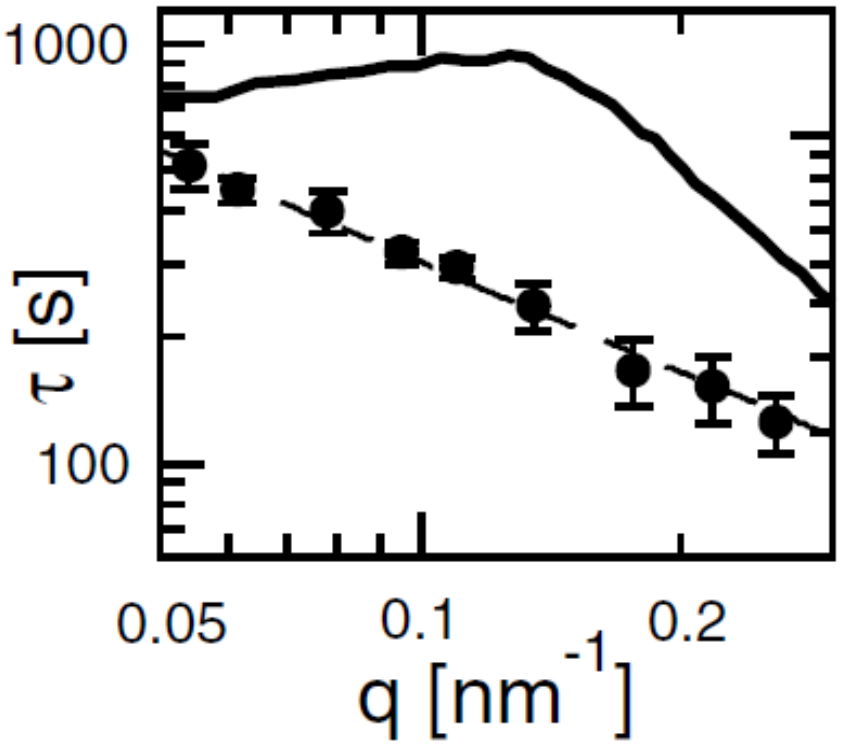
Contrast (bA^2) changes with time

- $g_2 = 1 + b\left[Ae^{-\left(\frac{t}{\tau}\right)^{\beta}}\right]^2$



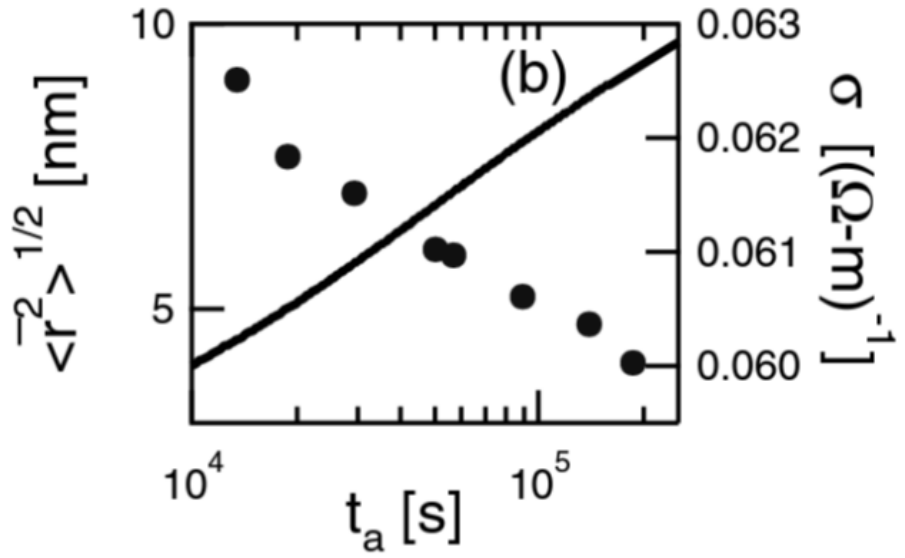
Relaxation time $\sim q^{-1}$

- Expect $\tau \sim q^{-2}$ for diffusion



What is going on?

- Particle caging changes with time, hence contrast decreases. Can represent the fast mode as a Debye-Waller factor $A = e^{-\frac{q^2\langle r\rangle^2}{3}}$



Ballistic Motion

- Stress drives ballistic motion.
- Ballistic motion gives $\tau \sim \frac{1}{q}$
- Shape of $\beta = 1.5$ seems to be generally associated with ballistic motion as well.

Two Time Correlation Functions

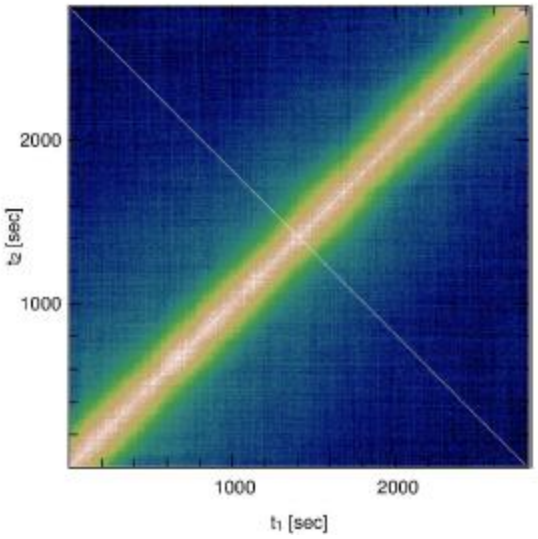
- For processing showing aging, you can approximately measure $g_2(\tau)$ at different absolute times from your starting point.
- More generally, you can define a new correlation function that depends on two times, t_1 the start time of your correlation and t_2 the time you are correlating with t_1

Two-time correlation function

- $C(t_1, t_2) = \frac{\langle I(t_1) I(t_2) \rangle}{\langle I(t_1) \rangle \langle I(t_2) \rangle}$

Equilibrium dynamics

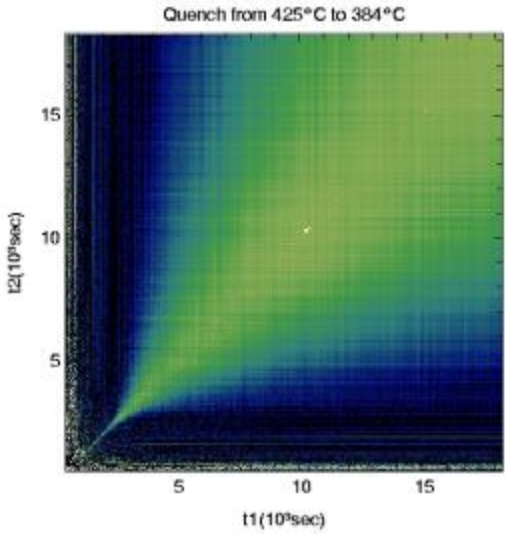
Au in polystyrene



Steady state diffusion

Nucleation and growth

Cu_3Au

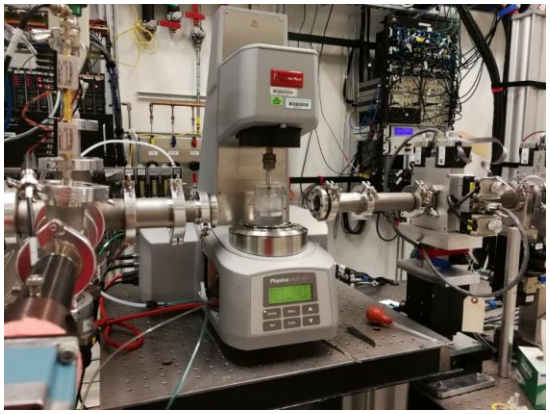


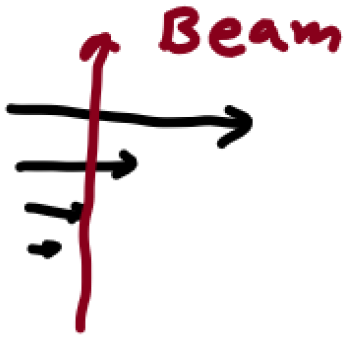
Mark Sutton

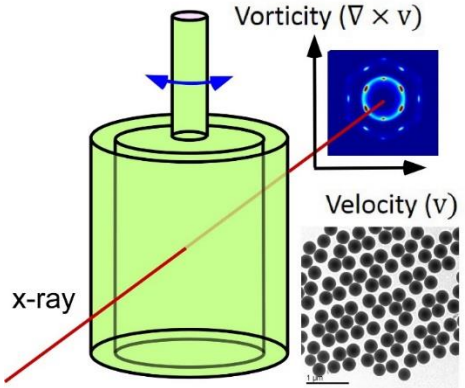
Stress relaxation in colloidal glasses

PRM Chen et. al. 2020

Measure dynamics of colloidal glass under sheer using in-situ sheer cell. σ is stress, γ is strain.

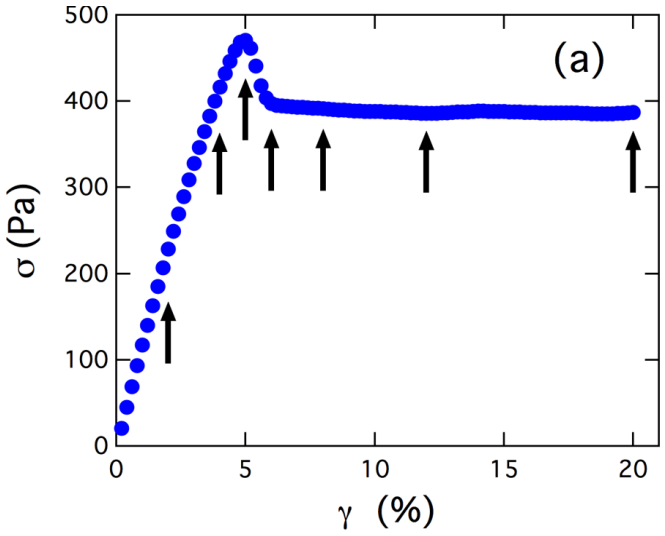






Relaxation after sheer

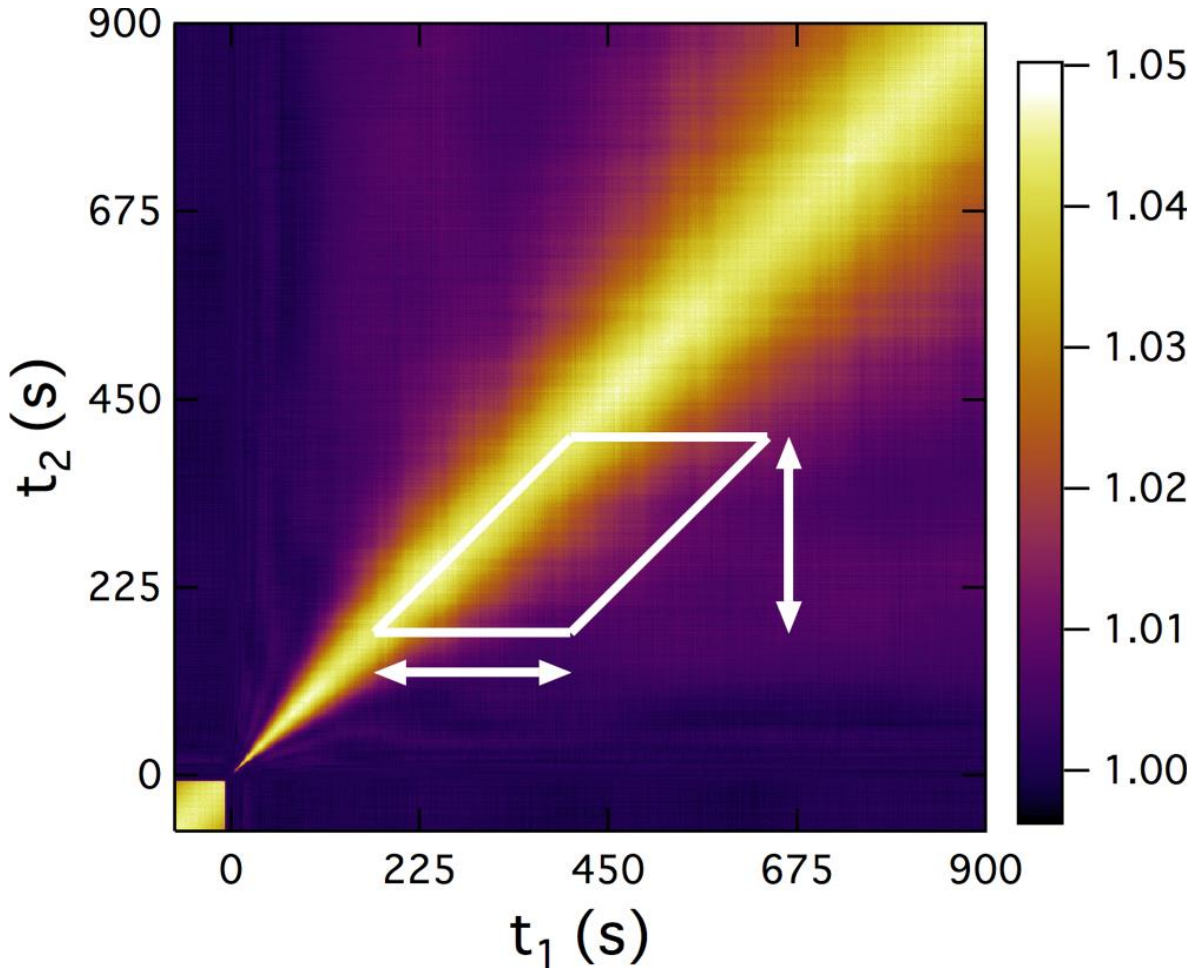
- Similar to the clay experiment, there is an initiating even and then the system evolves
- In this case, the dynamics is initiated by a step strain the rheometer
- Stress relaxes as a function of time and the dynamics are measured using XPCS



Stress strain curve

Relaxation along flow direction

Relaxation times change with overall wait time since step flow.

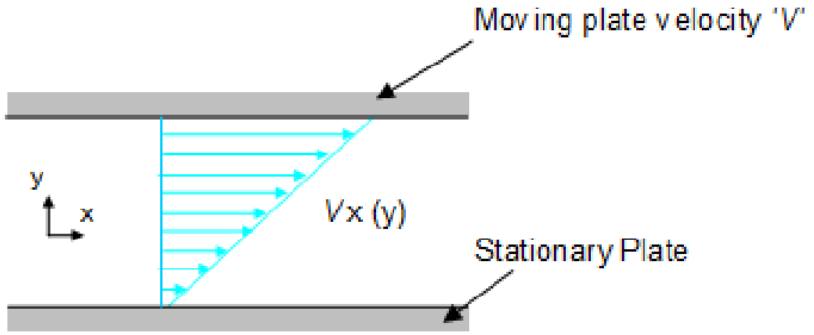


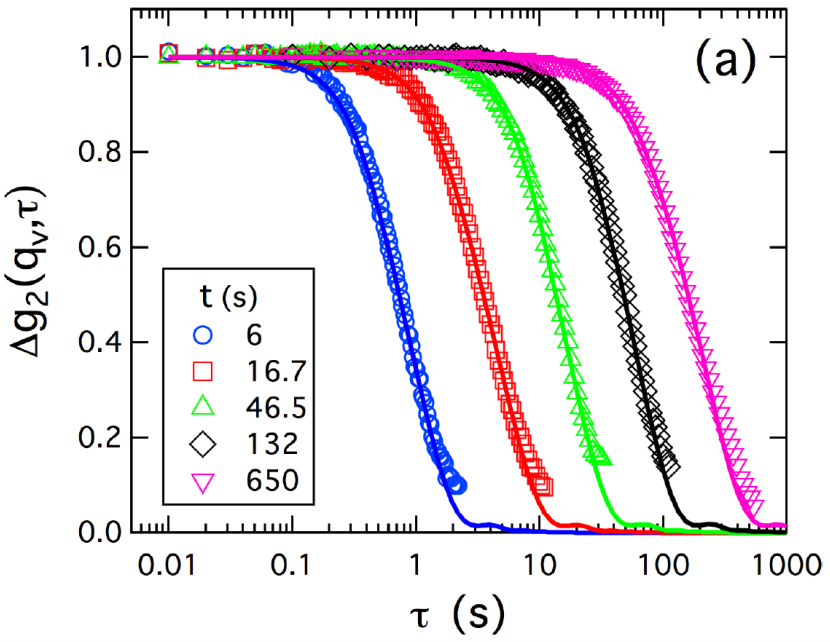
Non diffusive relaxation

- Dynamics is not diffusive, but shows flow with a range of velocities. Measuring relaxation of flow induced by residual stress

$$\Delta g_2(\vec{q},\tau) = \frac{\sin^2(qv_0\tau/2)}{\left(\frac{qv_0\tau}{2}\right)^2}$$

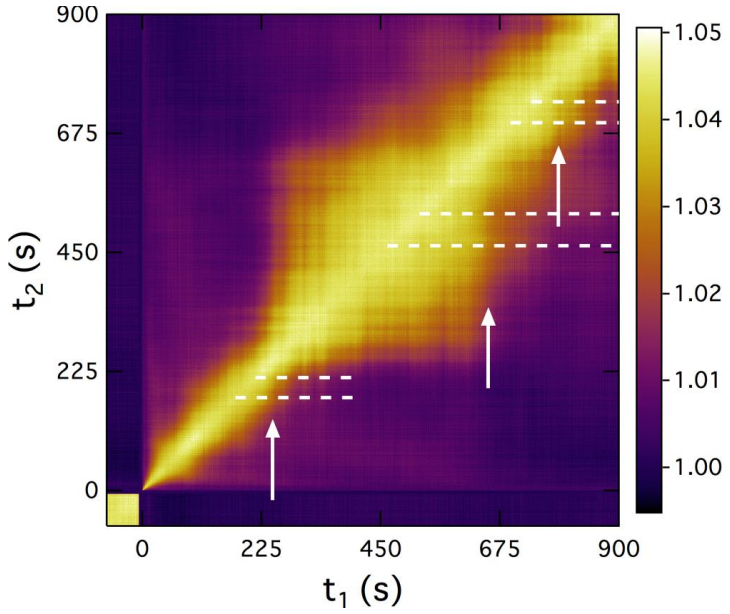
- Here Δg_2 has the constant subtracted and is normalized by contrast.
- v_0 is the peak velocity between the two walls.
- XPCS only sees velocity gradient, not absolute velocity.





What about dynamics along vorticity direction?

- Dynamics are complex and depend on both waiting time and time delay.
- New feature, “avalanches”, discontinuous events that destroy correlations.



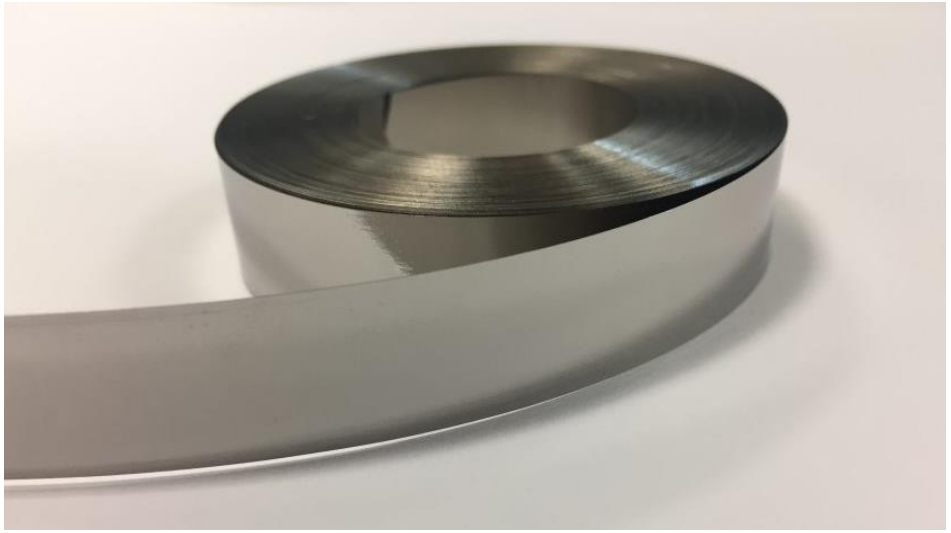
Vorticity direction

Some observations

- XPCS used to study a non-equilibrium system.
- In flow direction, observe quasi-equilibrium behavior around evolving dynamics
- Directional dependence of $\vec{q}$ used to distinguish very different dynamics in two directions.
- Two-time correlation functions can be used to identify avalanche like events which cause decorrelation.

Relaxation in Metallic Glasses

- Metallic glasses are formed by rapid quenching of metal alloys.
- They have high strength, large elastic elongation and high corrosion resistance.
- Downside is they are only metastable, so they slowly transform with time.
- Understanding the dynamics of aging and how to counteract aging effects is crucial for this technology



Antoine Cornet and Beatrice Ruta

Comptes Rendus

Physique

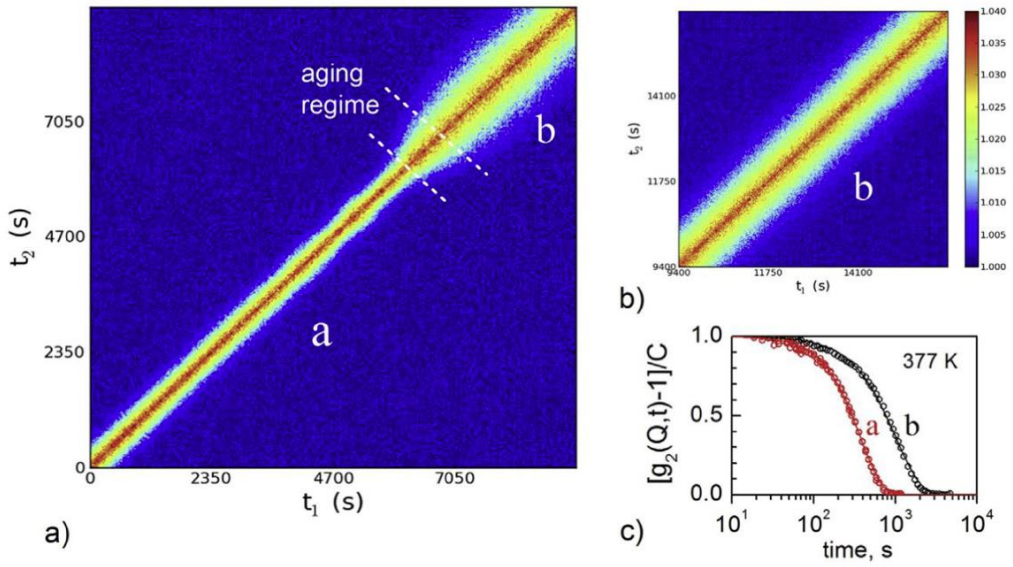
New pathways to control the evolution of the atomic motion in metallic glasses

Published online: 20 April 2023

https://doi.org/10.5802/crphys.149

Aging in metallic glasses occurs in short bursts with long quasi-static regimes interspersed.

$Au_{49}Cu_{26.9}Si_{16.3}Ag_{5.5}Pd_{2.3}$

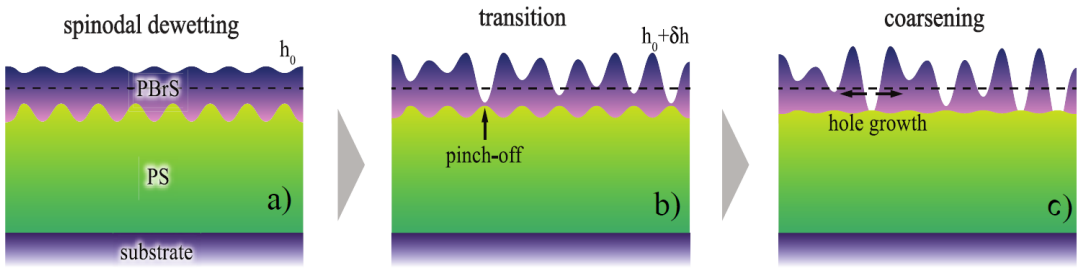


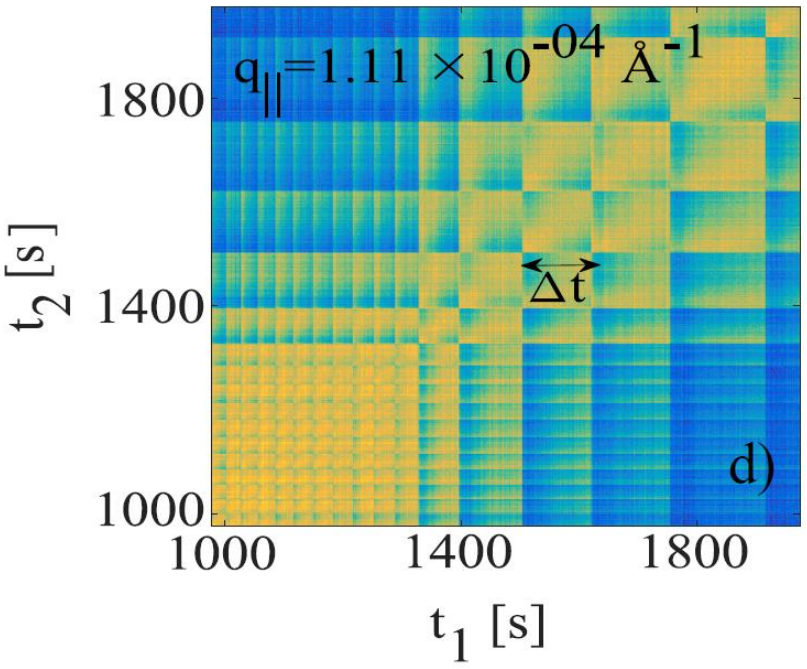
Stress in polymer films

Unstable bilayer film of PBrS on PS on a Silicon Crystal

Cycles of stress growth and sudden relaxation lead to disconnected two-time functions

Very complex two-time curves. Could AI analysis yield insights?



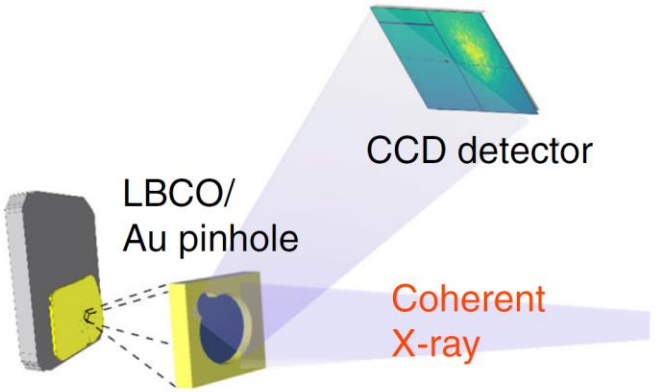


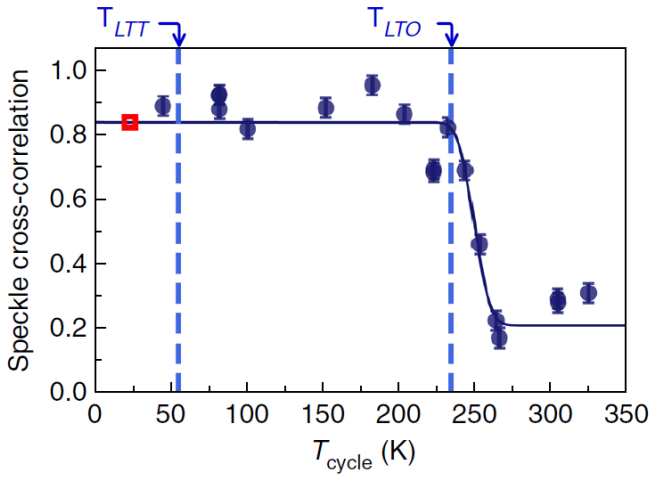
Lal, et. al. PRL 043162, 2021.

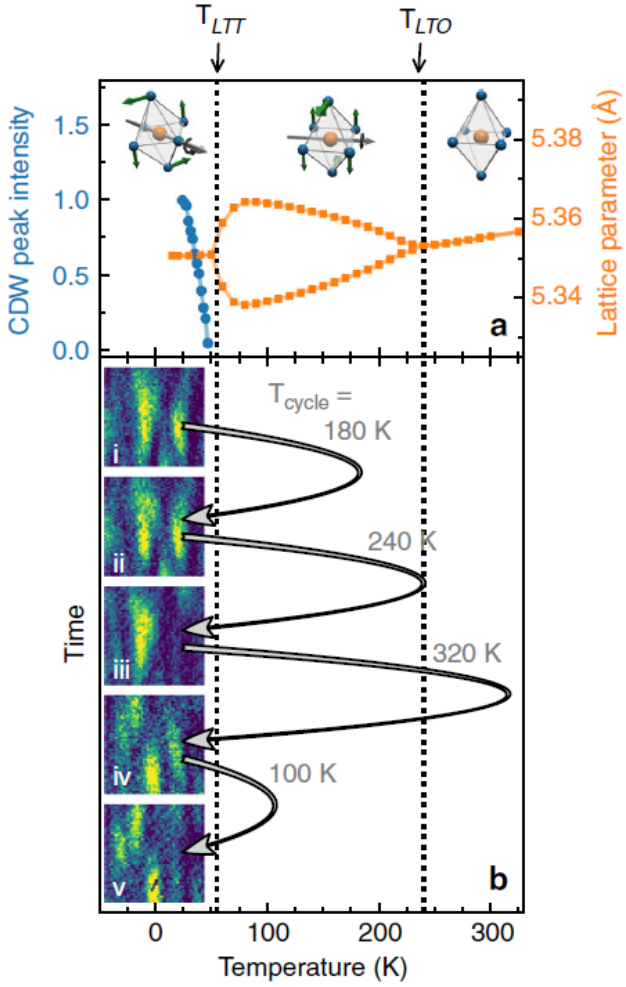
Speckle in Charge Density Waves

- Use speckle to measure persistence of structure.
- Superconductivity in LBCO 1/8 is suppressed relative to similar compounds due to the formation of charge density waves.
- Measure low energy diffraction at Cu L edge (931 eV) to enhance contrast.
- Speckles disappear after annealing, but then re-emerge with correlations upon cooling.
- Only heating above a second phase transition removes correlations.

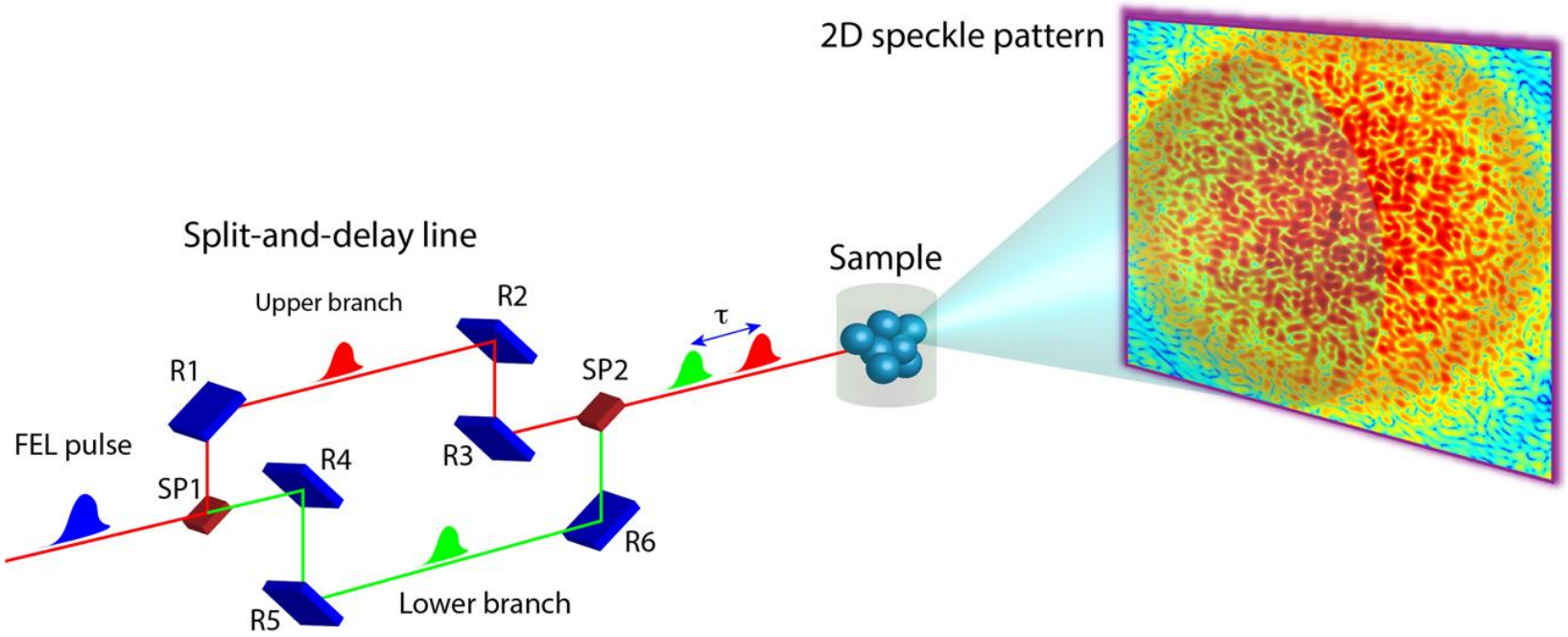
(Nature Comm: Chen et. al. 2019)







XPCS with free electron lasers

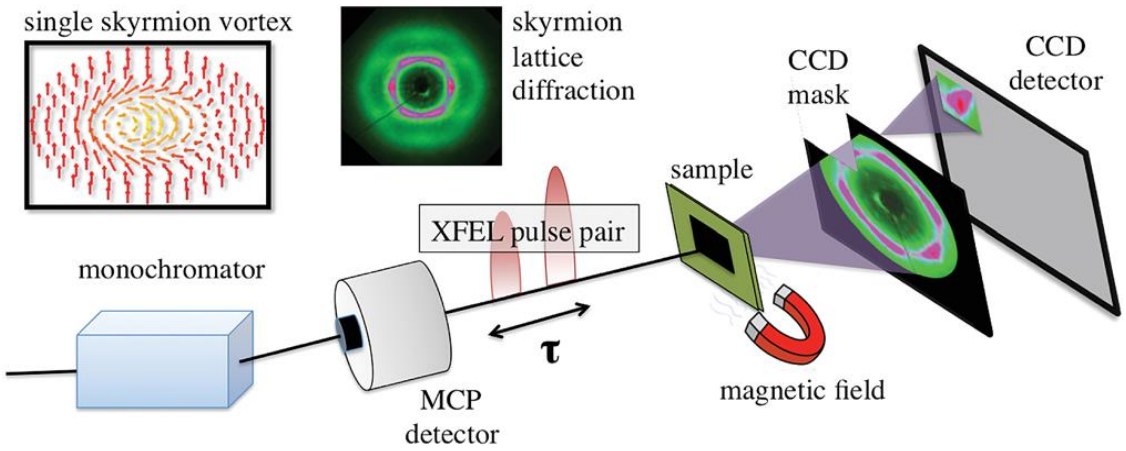


Alternately, a double pulse can be generated in the accelerator rather than splitting a single pulse.

Magnetic Skyrmion Dynamics

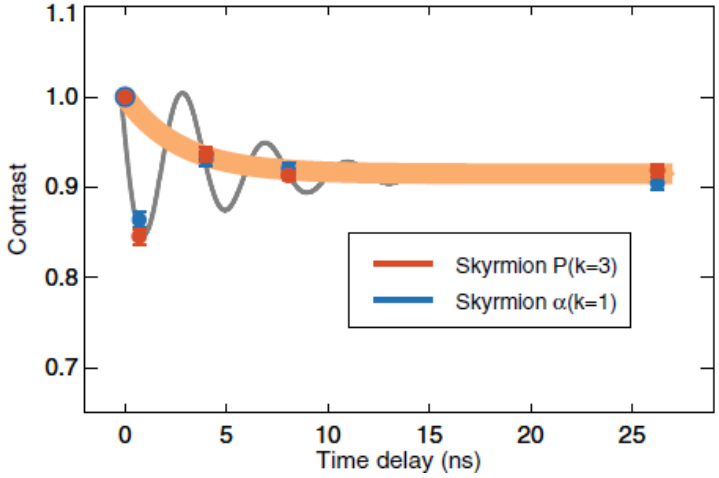
PRL Seaberg, et. al. 2017

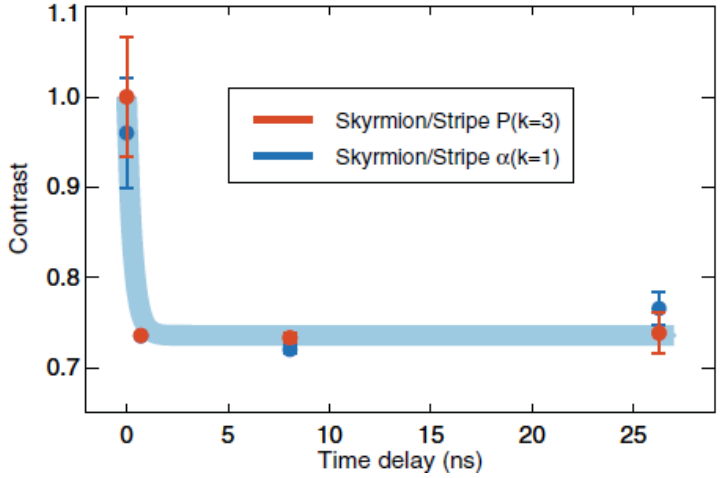
- Use resonant L-edge energy to gain sensitivity to magnetic structure.
- Use free electron laser pulses to measure with nanosecond time resolution
- Rather than split pulse, use pulse pair created by hitting electron source with two closely spaced laser pulses.



Dynamics

- Skyrmion phase dynamics speed up near the skyrmion/stripe phase boundary.
- Still unexplained dip at short times





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How to Calculate a Correlation Function

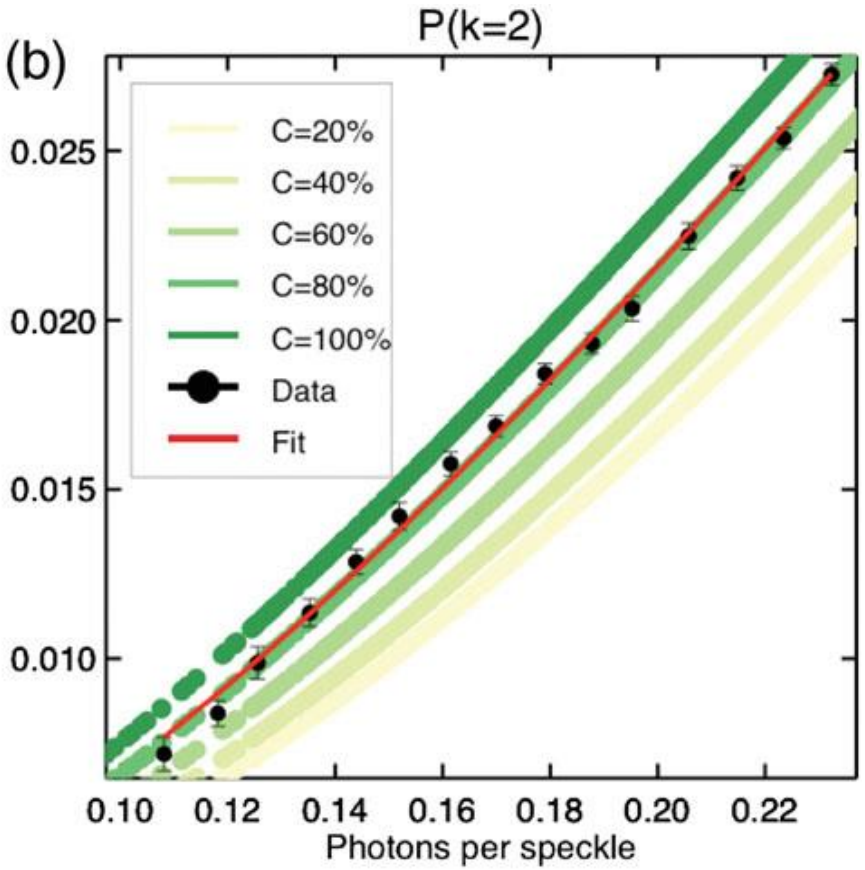
- We want to find $g_2(\tau) = \frac{\langle I(t+\tau)I(t)\rangle}{\langle I\rangle^2}$
- Each pixel is a sequence of N intensity measurements I_j
- $g_2(\tau = \mathrm{m}\Delta t) = \frac{1}{N-k}\frac{1}{\langle I\rangle^2}\sum_{j=1}^{N-k} I_j I_{j+k}$
- Interesting feature: The signal to noise ratio scales as I, rather than $\sqrt{I}$ since g_2 is second order in I
- Shortest accessible times scales as $\frac{1}{B^2}$, where B is the synchrotron brilliance.
- APS-U should see 10^4 faster times!

Extra Slides

Use photon statistics to extract contrast

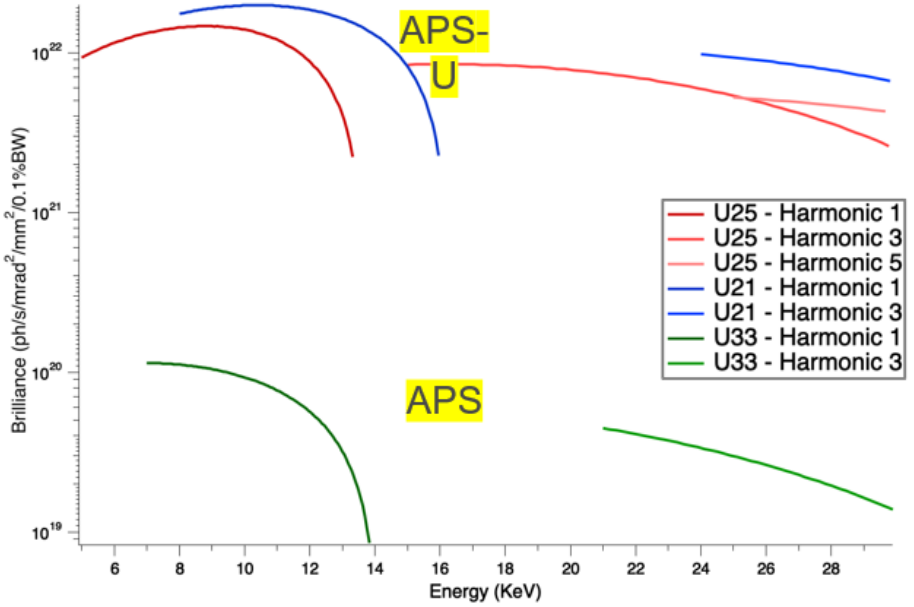
$$P_M(I) = \frac{M^M I \langle I \rangle^{M-1} \exp\left(-\frac{MI}{\langle I \rangle}\right)}{\Gamma(M)\langle I \rangle}$$

Probability to scatter 2 photons into one pixel as a function of the average counts/pixel and the contrast, C



Upgraded APS

- 8-ID XPCS Beamlines : Suresh Narayanan, Eric Dufresne, Qingteng Zhang
- ~100x more brightness
- Or ~20x more flux but 100% coherent instead of 20%
- Or 500x less x-ray damage for the same SNR



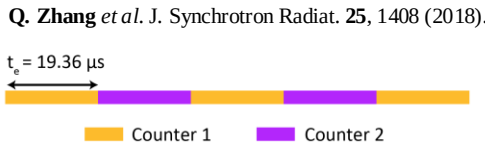
Single-Photon-Counting Detectors

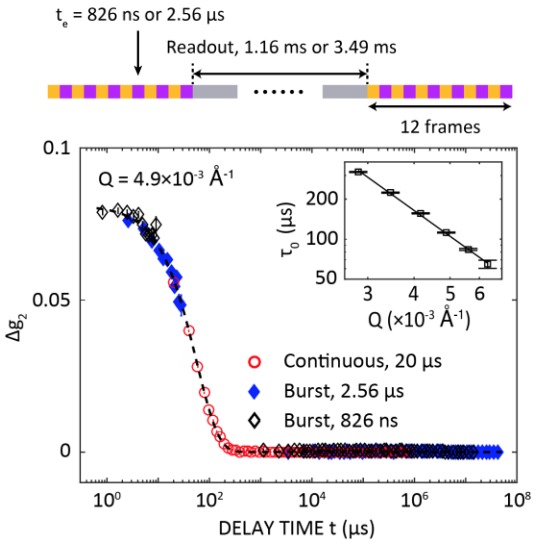
2048 (H) x 2048 (V) pixels, Pixel size = 75 µm

- Eiger 4M CdTe sensor for XPCS with max photon energy of 25 keV at 8-ID-I.
- Max frame rate = 4 kHz (250 µs).

2048 (H) x 1536 (V) pixels, Pixel size = 76 µm

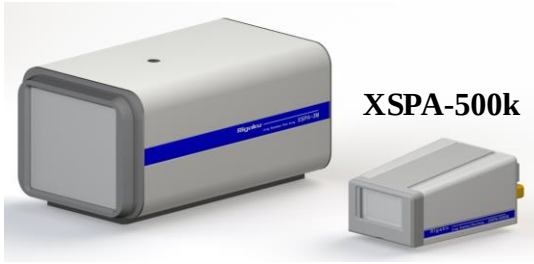
Q. Zhang *et al.* J. Synchrotron Radiat. **25**, 1408 (2018).





XSPA-3M

XSPA-500k



- Rigaku XSPA 3M (6x larger than 500k) Si sensor for XPCS with photon energy < 12 keV.
- Gapless coverage from 1 µs to 10^3 s by stitching different modes.