X-ray photon correlation spectroscopy (XPCS) in microfluidic systems

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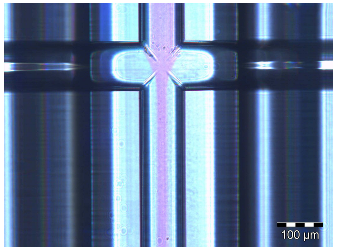
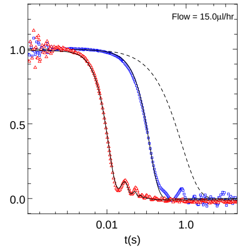
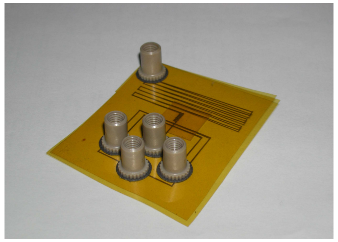






Outline

- Introduction to X-ray Photon Correlation Spectroscopy (XPCS)
  - $\longrightarrow$ Coherence and Speckles
  - $\longrightarrow$ Diffusive dynamics of colloidal suspensions
  - $\longrightarrow$ “Standard” (single-speckle) and multispeckle XPCS
- *XPCS-microfluidics*: measuring the mesoscale dynamics in sheared complex fluids
  - $\longrightarrow$ Motivations
  - $\longrightarrow$ SAXS-compatible microfluidic devices
  - $\longrightarrow$ XPCS in a shear flow
  - $\longrightarrow$ “transverse” ($\mathbf{Q} \perp \mathbf{v}$), “longitudinal” ($\mathbf{Q} \parallel \mathbf{v}$) scattering geometry
- Slow dynamics and aging in colloidal gels
  - $\longrightarrow$ Colloid / polymer mixtures: phase behaviour
  - $\longrightarrow$ Non-equilibrium and non-stationary dynamics: two-time correlation functions
  - $\longrightarrow$ Jamming phenomena; jamming transition in aging colloidal gels
  - $\longrightarrow$ Colloidal gels in microfluidic devices
- Conclusions & Outlook
  - $\longrightarrow$ XPCS-flow experiments in soft-matter model systems and biological systems

Coherent XRD: speckle, XPCS

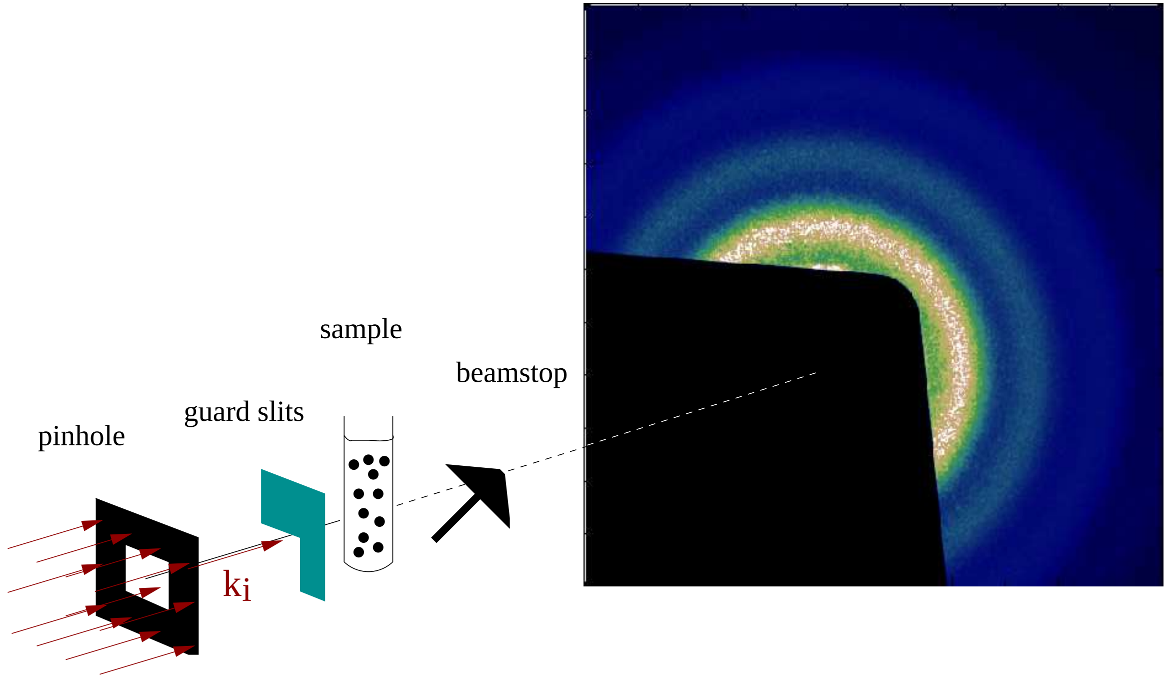
$Q = \left(\frac{4\pi}{\lambda}\right) sin\left(\frac{\Theta}{2}\right)$

$I(Q,t) = N_p \Delta\rho^2 V_p^2 P(Q) S(Q,t)$

$P(Q)$ - particle form factor

Structure factor:

$S(Q,t) = \frac{1}{N}\sum_{i,j=1}^{N}\left\langle e^{-i\mathbf{Q}(\mathbf{r_i}(t)-\mathbf{r_j}(t))}\right\rangle$



Coherence

What interferes? Probability amplitudes Φ regarding microscopic states compatile with the macroscopic state of the system!

P=$|\Phi|^2$: probability for a single event

I $\longrightarrow$P if the event is repeated several times (good statistics).

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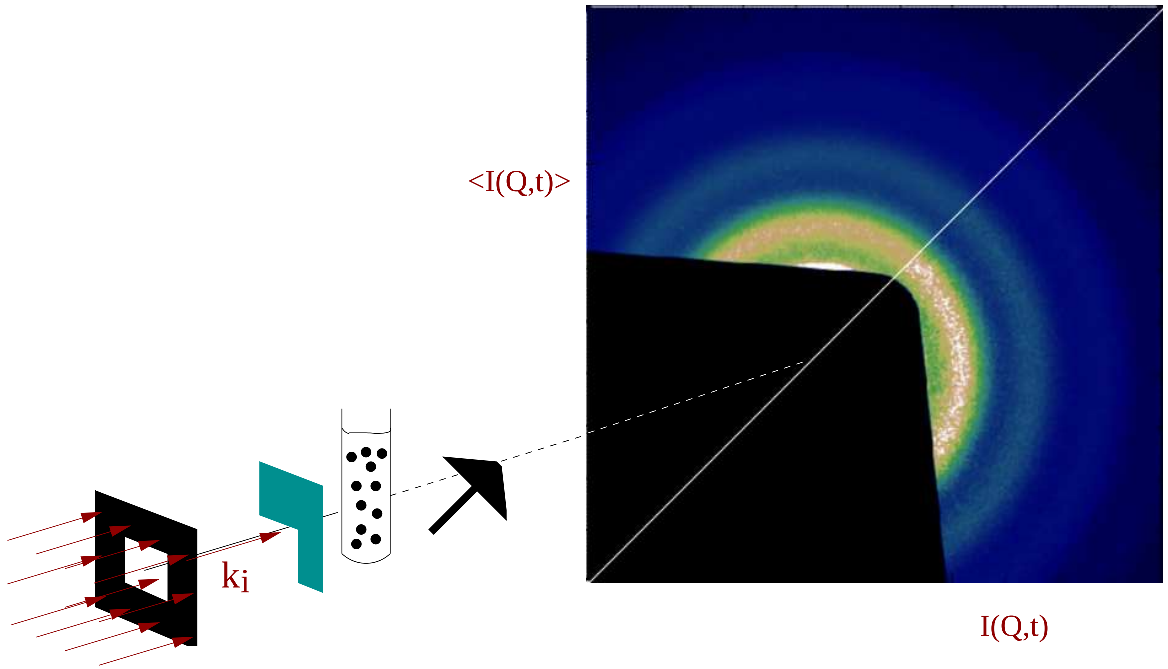


The speckle pattern is observed only if the event is repeated several times (good statistics). The re-occurence of an event under non-ideal conditions leads to the loss of the speckle pattern (phase information). E.g.:

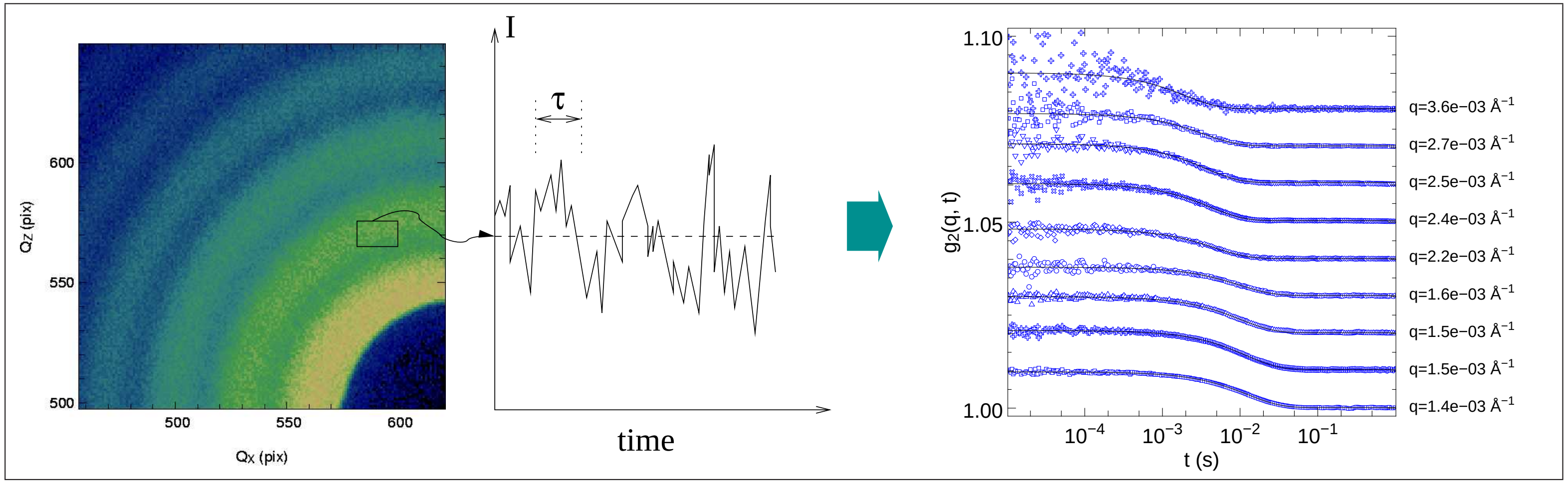
$\longrightarrow$ E_{in}, E_{out}, $\mathbf{k}_{in}$, $\mathbf{k}_{out}$ are not well defined

$\longrightarrow$ disorder in the sample

$\longrightarrow$ unsufficient detector resolution

$\longrightarrow$ chaotic source . . .

X-ray Photon Correlation Spectroscopy, XPCS

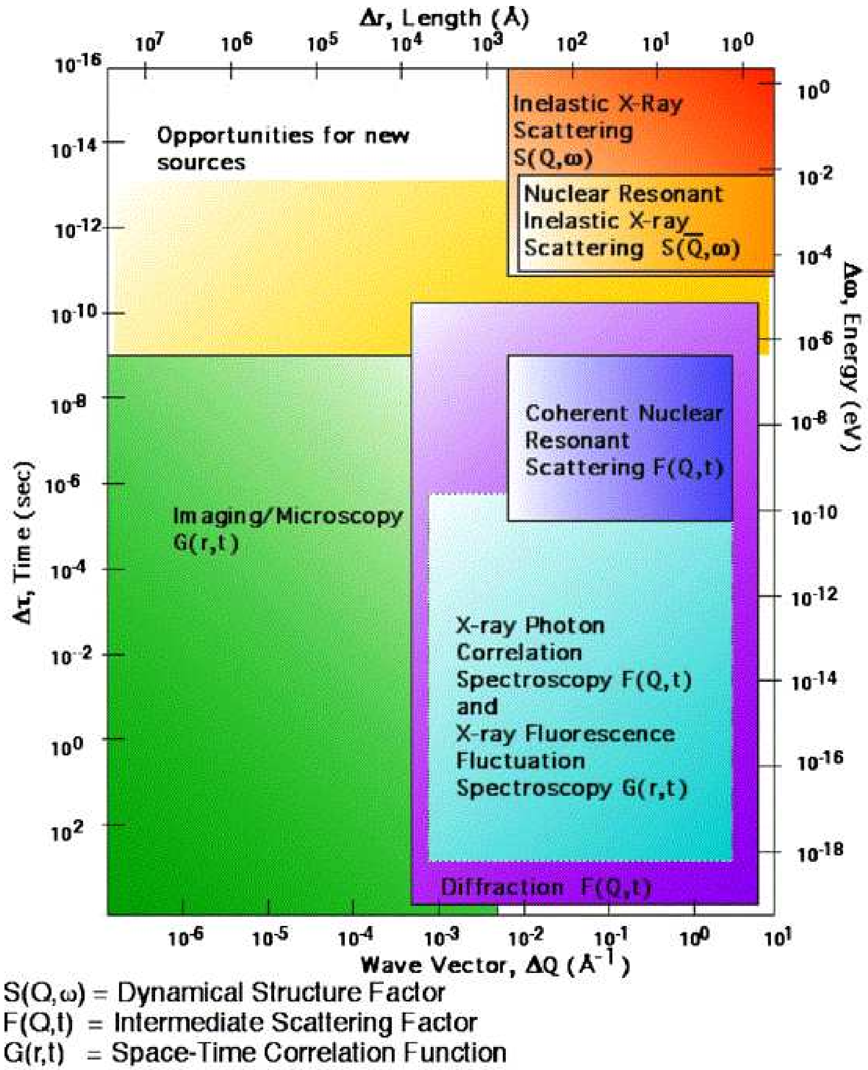


- Intensity fluctuation autocorrelation functions:

$$g_2(q,t) = \frac{<I(q,0)I(q,t)>}{<I>^2} = 1 + \beta \left| \frac{S(q,t)}{S(q,0)} \right|^2$$

- X-ray photon correlation spctroscopy - XPCS
  X-ray Intensity Fluctuation Spectroscopy - XIFS
- complementary to Dynamic Light Scattering - DLS

XPCS: length and time scales



Can be used in a large variety of experimental configurations:

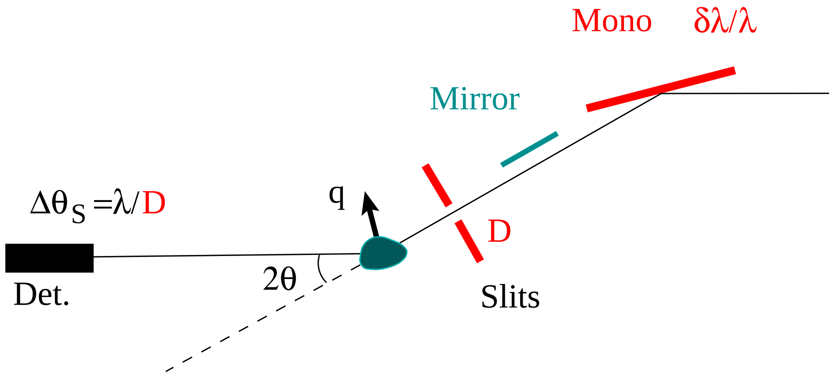
→SAXS, WAXS, GID, ...
→transmission / reflection

$l \simeq 1\mu$m - 10Å
$t \simeq 10^6$ - 10^{-3} Hz

from G. K. Shenoy, *Nucl. Inst. and Meth. B* **199** (2003), 1–9

Troïka I beamline @ E.S.R.F.

Troïka (ID10A) side station



Undulator source

U27, U35, revolver U27/U35

source size 928 x 23 μm^2 (h x v) FWHM

Multi-crystal single bounce monochromator

Si(111), $\Delta\lambda/\lambda = 10^{-4}$

Filtering and focussing

Si mirror in the monochomatic beam

Be CRLs

Coherent source

Highly polished rollerblade slits

flux @ sample 10^9-10^{10} ph/s (8 keV, 10x10 μm^2)

brill. > 10^{20} ph/s/mm^2/mrad^2/0.1%bw/100mA @ 8 keV

Medipix 2 pixel detector

Single photon conting

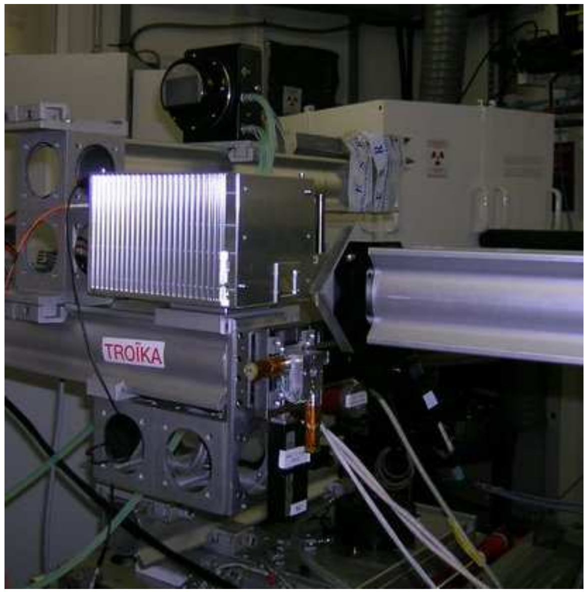
Energy discrimination

55 x 55 μm^2 pixel size

256 x 256 pixels (12 mm x 12 mm)

13.5 bits - 11800 cts/pixel (1 ADU/photon)

1 kHz frame repetition rate

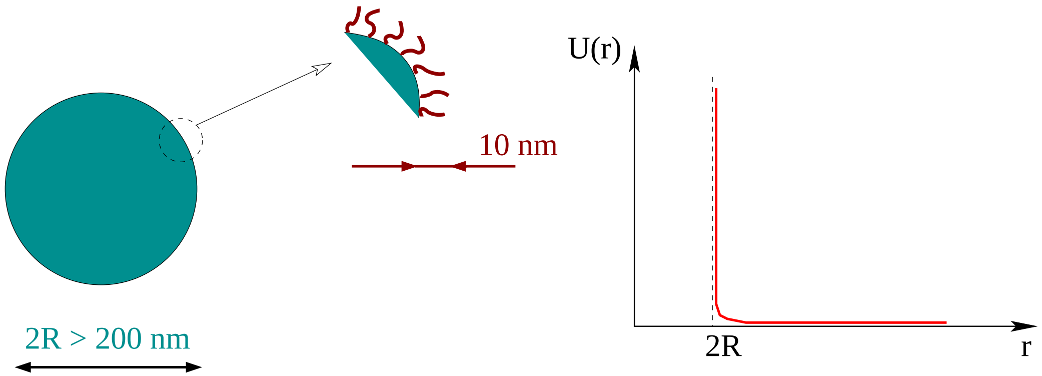


ESRF Detector Sytems

X. Llopart et al.,*IEEE Trans. Nucl. Sci.* **49**, 2279 (2002)

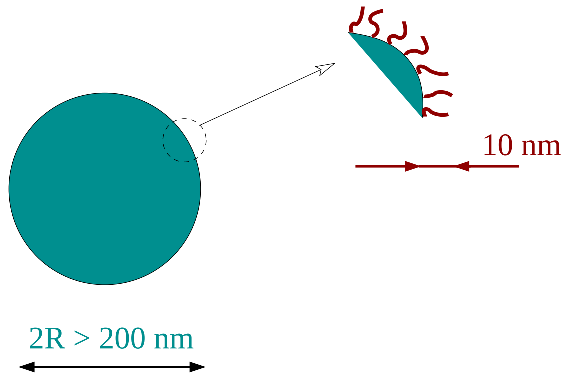
C. Caronna et al., ESRF *Sporlight on Science* **39** (2006)

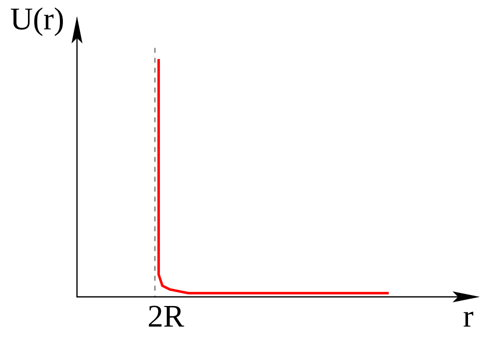
Hard-sphere suspensions



- Spherical Poly(MethylMethacrylate) PMMA particles coated with poly-12-hydroxystearic acid in cis-decalin
- Entropic forces between the polymer layers -> infinite repulsion.

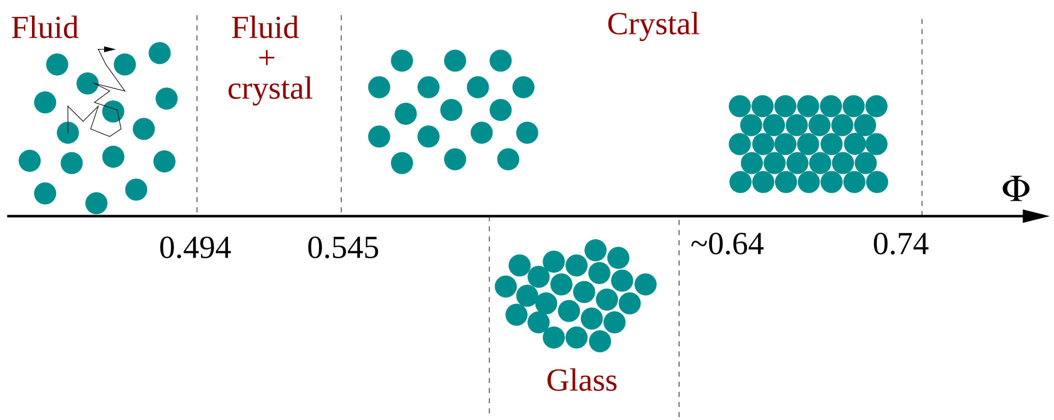
Hard-sphere suspensions





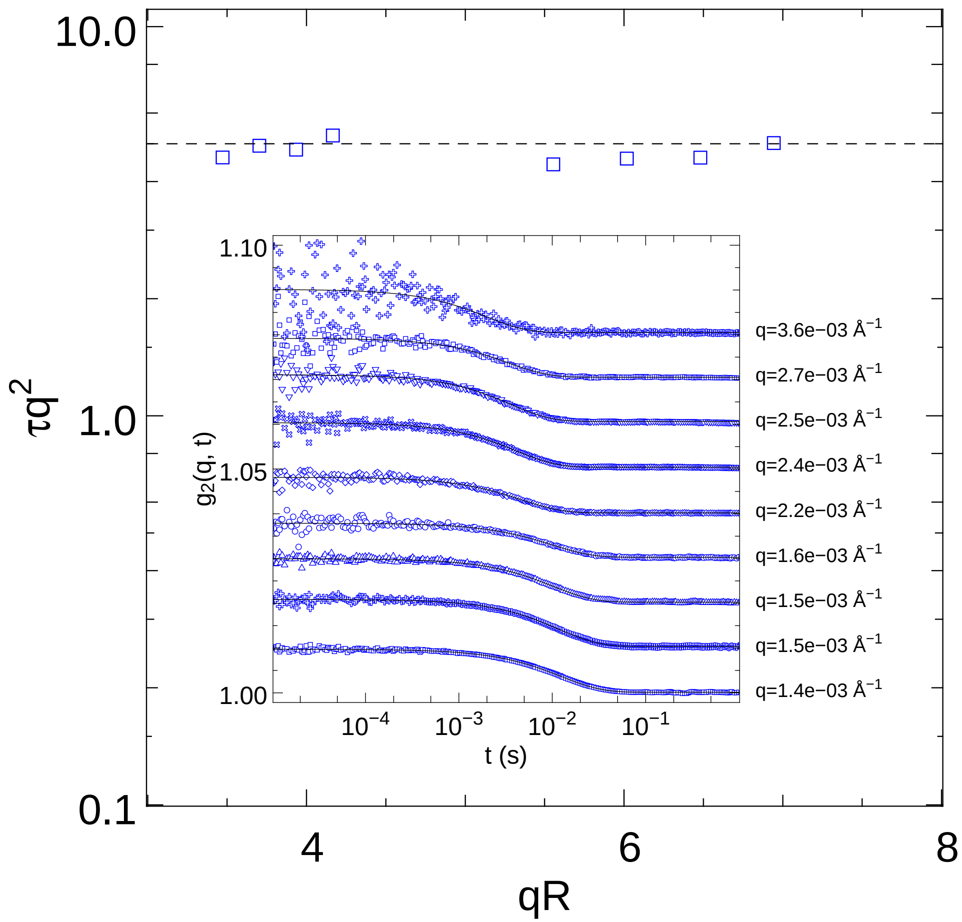
- Spherical Poly(MethylMethacrylate) PMMA particles coated with poly-12-hydroxystearic acid in cis-decalin
- Entropic forces between the polymer layers -> infinite repulsion.
- The phase behaviour depends on the colloid volume fraction

$$\Phi = \frac{4}{3}\pi R^3 \frac{N}{V}$$



P.N. Pusey & W. Megen, Nature **320**, 340 (1986).

XPCS: Brownian dynamics in a PMMA / cis-decalin suspension (Φ=0.16)



- Intensity correlation functions:

$$g_2(q,t) = \frac{<I(q,\tau)I(q,\tau+t)>_\tau}{<I(q,\tau)>_\tau^2}$$

$$= 1 + \beta\,|g_1(q,t)|^2$$

- Intermediate scattering functions:

$$g_1(q,t) = \frac{S(q,t)}{S(q,0)}$$

$$g_1(q,t) = exp(-\Gamma t) = exp\left(-\frac{t}{\tau}\right)$$

- Diffusion

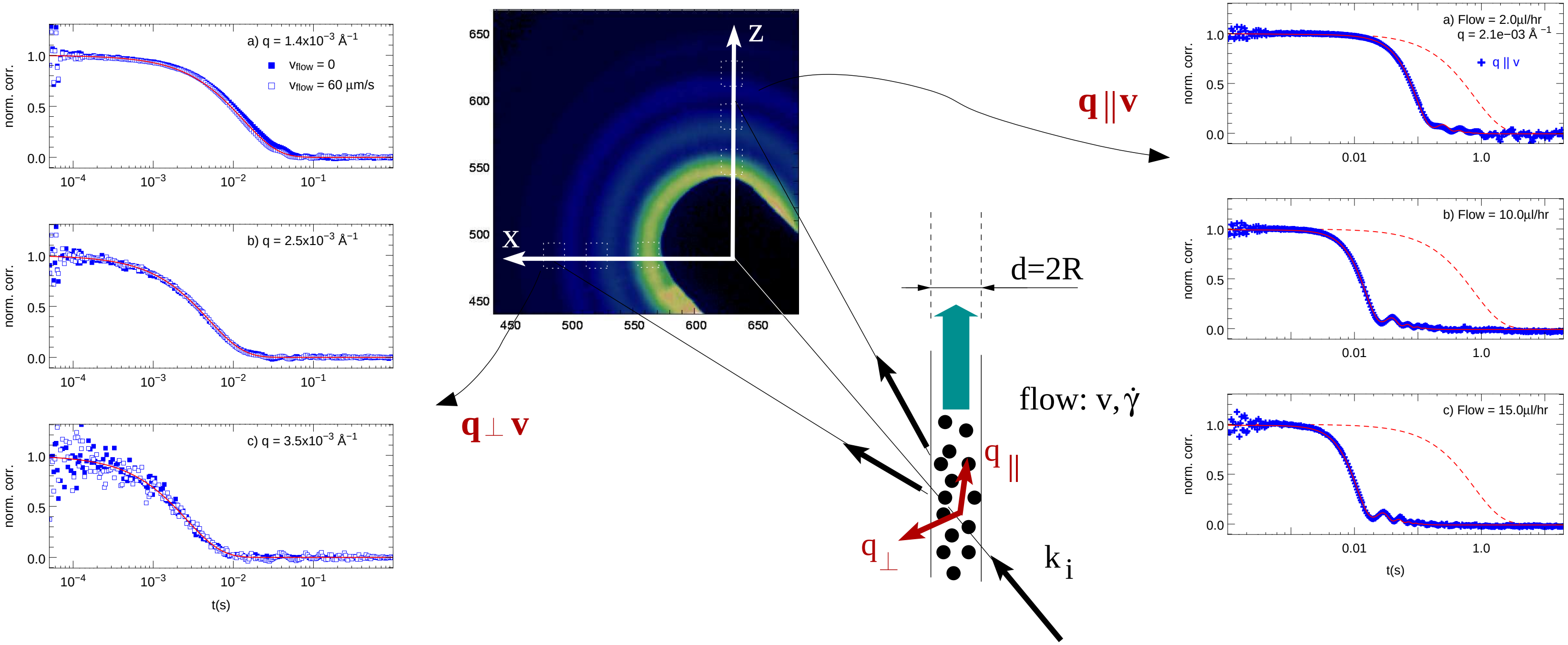
$$\Gamma = \frac{1}{\tau} = Dq^2$$

$$D = \frac{k_B T}{6\pi\eta a} \qquad \text{(Einstein)}$$

$$\langle x(t) - x(0)\rangle^2 = 2Dt \quad \text{(Fick)}$$

L. Lurio et al., Phys. Rev. Lett **84**, 785 (2000)
D. Lumma et al., Phys. Rev. E **62**, 8258 (2000)
F. Zontone, A. Moussaïd, *work in progress*

Measuring dynamics in shear flow by X-ray photon correlation spectroscopy (XPCS)



- the dynamics in anisotropic and measures a combination of diffusive and (flow induced) advective motion of the particles. G.G. Fullet et al., J. Fluid Mech. 1980

  → Heterodyne detection: $g_1(\mathbf{q},t) = \exp(-Dq^2t+\ldots)\cdot\exp(-i\mathbf{qv})\cdot\int \mathbf{q}\delta\mathbf{v}$
  C.Gutt et al., PRL 2004; F. Livet et al., J. Synch. Rad. 2006

  → Homodyne detection: $g_2(\mathbf{q},t) = \exp(-2Dq^2t+\ldots)\cdot\cdot\left[\int \mathbf{q}\delta\mathbf{v}\right]^2$

- the diffusive component can be obtained by using correct experimental conditions (scattering geometry, flow/shear rate)
  Andrei Fluerasu et al., *J. Synchrotron Rad.*, submitted; Sebastian Busch et al., *Eur. Phys. J. E.*, submitted;

XPCS & SAXS + microfluidics: motivations

- offers unique control possibilities over many parameters, and access to a large range of time scales; is an original and movel way to study problems that cannot be studied by conventional means.
- Continuous flow may help preventing sample damage by the X-ray beam.

  → opens new possibilities with biological samples

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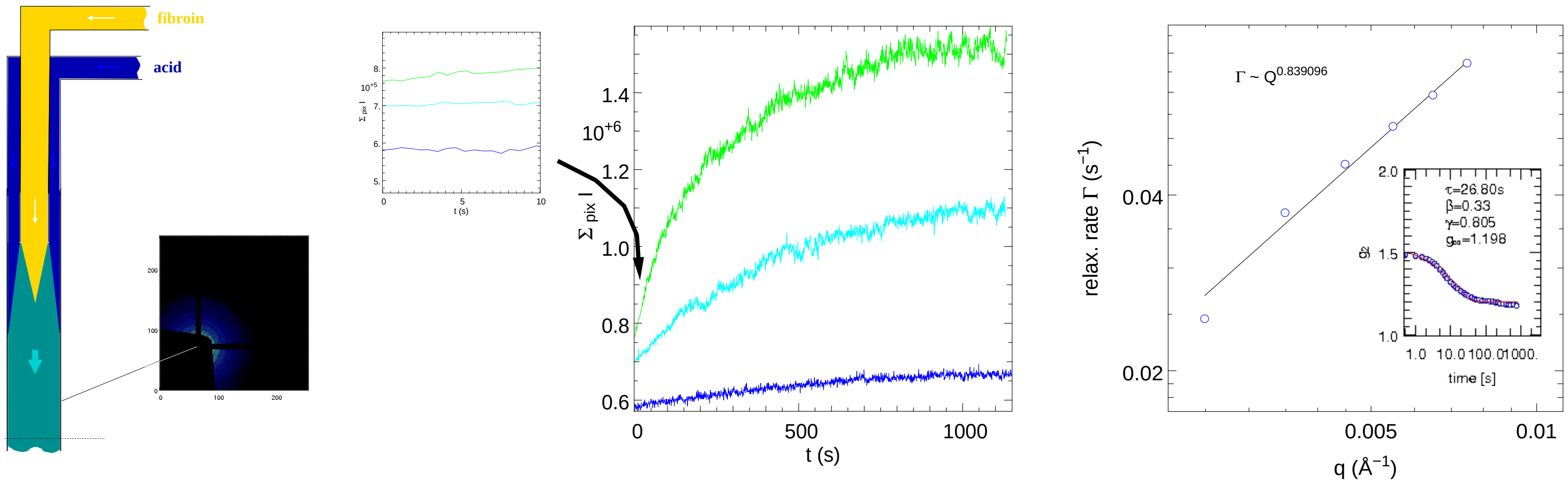
- Continuous flow may help preventing sample damage by the X-ray beam.

  → opens new possibilities with biological samples

E.G. Aggregation of fibroin (Anne Martel, Christian Riekel, ID13 ESRF)

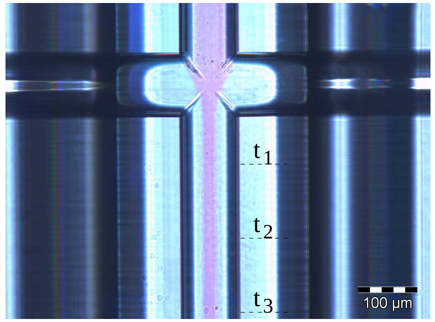


A. Martel, S. Busch, A. Fluerasu, and C. Riekel, work in progress

XPCS & SAXS + microfluidics: motivations

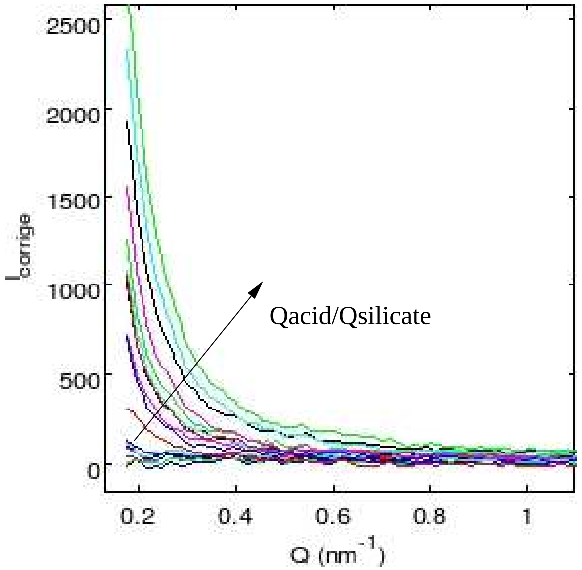
- Microfluidic systems allow time-resolved studies of processes taking place in mixing flowcells. The time-dependance is mapped into a space-dependance!

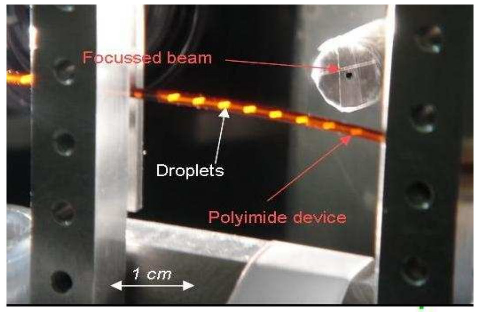
  L.Pollack et.al., Phys Rev. Lett. **86**, 4692, (2001).

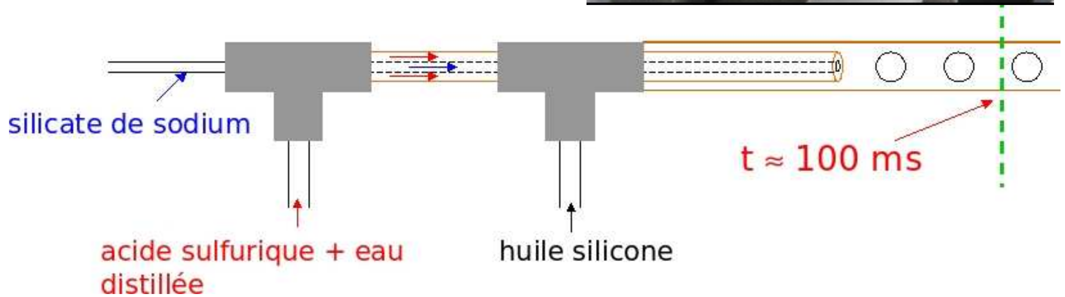


courtesy of Anne Martel, ID 13 − ESRF

e.g. time-resolved studies formation of silica particles via sol/gel processes (ID 18, ID 2, ID 10, ESRF)



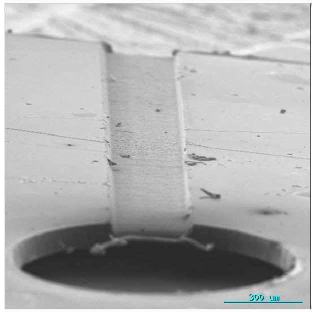


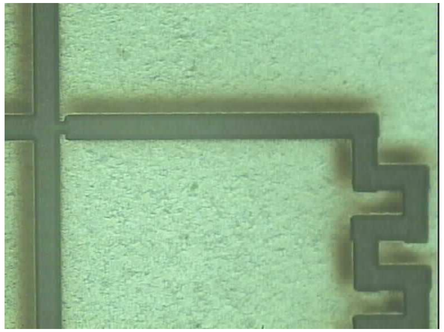


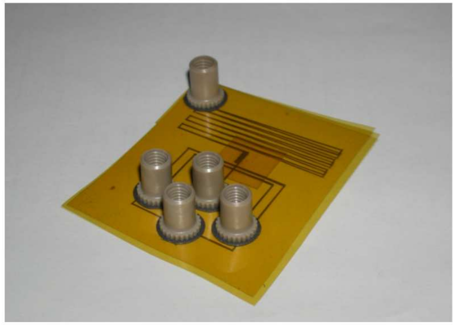
F. Destremaut, J−B. Salmon, J. Leng (LOF CNRS−Rhodia, Bordeaux) A. Fluerasu (ESRF) work in progress

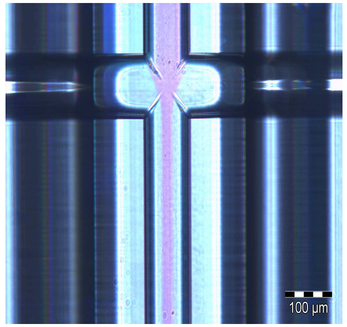
X-ray compatible flow devices

- Microfluidic devices R. Barret et al., *Lab. Chip.* **6**, 494 (2006); A. Martel, PhD thesis (ID 13 - ESRF);

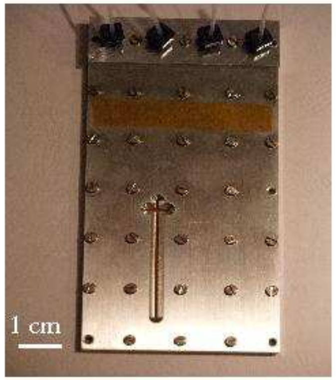


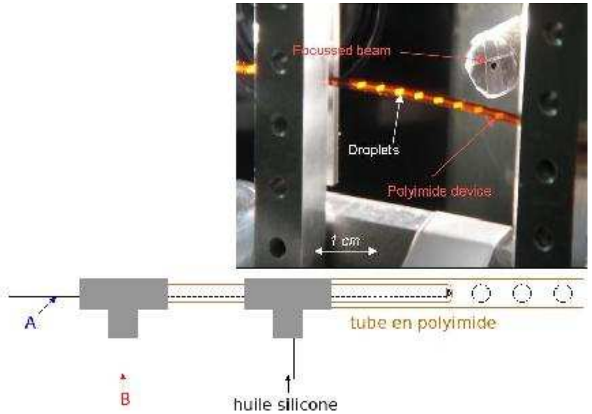






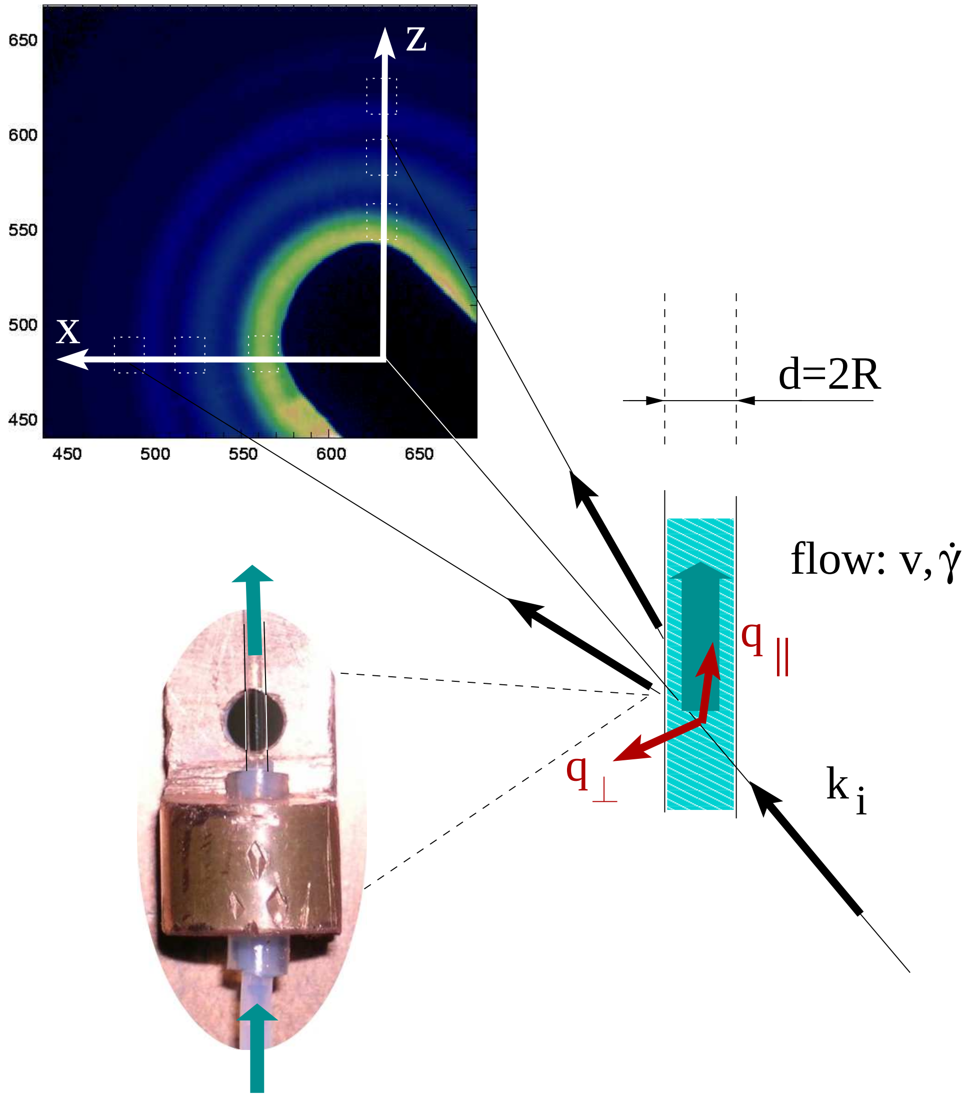
- “Milifluidic” devices - F. Destremaut, J.-B. Salmon, to be submitted; S. Busch et al., *Eur. Phys. J. E*, submitted (2007);





“Table-top” fabrication method and technologies brought to ESRF as a part of the LTP SC-2266 & SC-2329 project (ID 13, ID 2, ID 10, LOF CNRS-Rhodia, Bordeaux).

XPCS-microfluidics: the importance of scale



Diffusion

Shear rate: $\dot{\gamma} = -\frac{dv(r)}{dr}$ (T^{-1})

$\mathrm{Wi} = \dot{\gamma}\tau \ll 1$

Transit time effects

Deborah number:

$\mathrm{De} = \frac{\tau v}{s} \ll 1$

Shear-induced effects

Peclet number:

$\mathrm{Pe} = \frac{\dot{\gamma} R^2}{D} \gg 1$!!!!

Shear number:

$S = \frac{\mathbf{q} \cdot \mathbf{v}}{Dq^2} \gg 1$!!!!

Shear flow

Volume rate of flow
$Q(L^3T^{-1}) = \pi R^2 v$

Shear rate: $\dot{\gamma} = -\frac{dv(r)}{dr}$ (T^{-1})

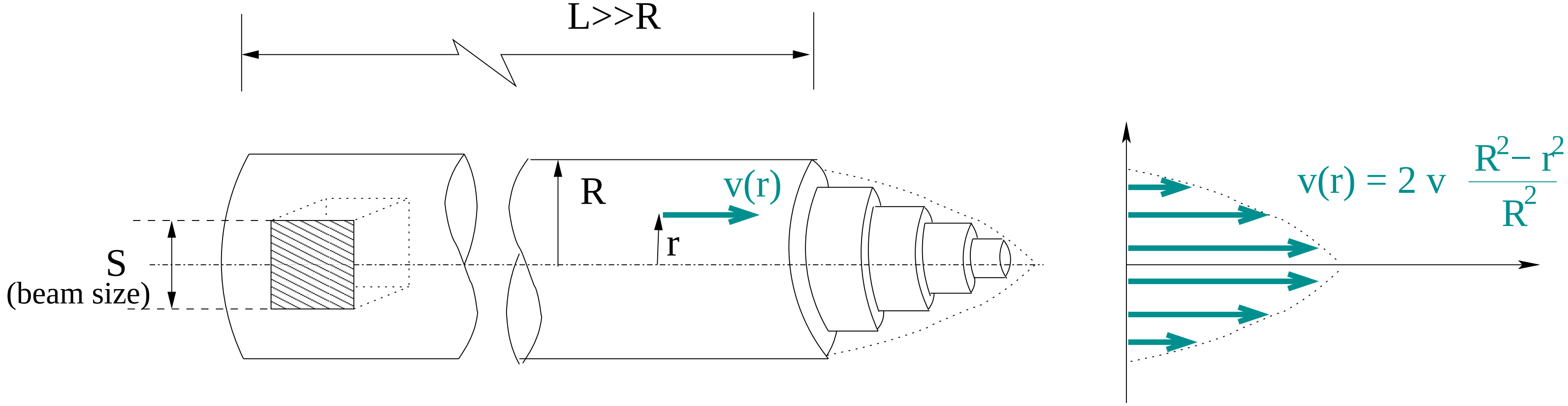
average shear rate $\dot{\gamma} \approx \frac{3v}{R}$

Reynolds number $Re = \frac{\rho v(2R)}{\eta}$

(inertial forces) / (viscous forces)

η = 3.38 cP, $\rho \approx 0.9$ g/cm^3 (cis-decalin)

2R = 980 μm, v = 100 μm/s $Re < 0.1$



XPCS in shear flow

- Brownian dynamics in shear flow   Ackerson, Clark, *J. Physique* **42**, (1981), 929-936;

$$g^{(2)}(\mathbf{q},t)-1=\beta\exp\left[-2\Gamma t\left(1-\frac{q_\perp q_\parallel}{q^2}\dot{\gamma}t+\frac{q_\parallel^2}{q^2}\cdot\frac{(\dot{\gamma}t)^2}{3}\right)\right]\approx\exp\left[-2\Gamma t\right]\qquad(\text{if }\dot{\gamma}t<1)$$

- Doppler velocimetry (w. homodyne detection)   G.G. Fuller et al.,*J. Fluid Mech.*, **100** (1980), 555

$$\mathbf{q}\cdot\delta\mathbf{v}\text{ - self beat frequency}\qquad G(\mathbf{q},t)=\int dr_1 dr_2\cdot\exp[-i\mathbf{q}\delta\mathbf{v}]$$

$$G(\mathbf{q},t)=\left[\frac{\sin(\Gamma_S t)}{\Gamma_S t}\right]^2(\dot{\gamma}\text{=const})=\frac{\left|erf\left(\sqrt{i\Gamma_S t}\right)\right|^2}{\Gamma_S t}\text{ (Poisseuille), with }\Gamma_S\propto q_\parallel v$$

- Transit time effects

neglected if De$\ll$1

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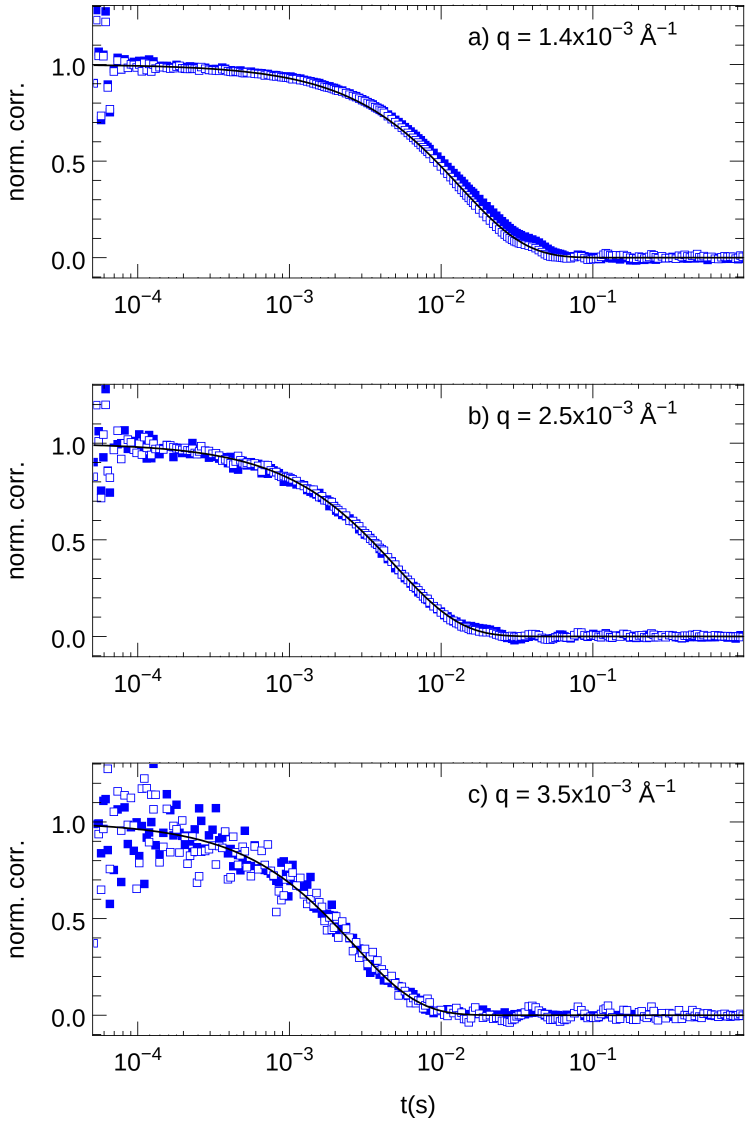
With **homodyne** detection, the signal is modulated by a frequency determined by the Doppler shifts between all pair of particles in the scattering volume ($\mathbf{q}\delta\mathbf{v}$(r)).

$$g^{(2)}(\mathbf{q},t)-1=\beta\exp\left[-2\Gamma t\left(1-\frac{q_\perp q_\parallel}{q^2}\dot{\gamma}t+\frac{q_\parallel^2}{q^2}\cdot\frac{(\dot{\gamma}t)^2}{3}\right)\right]\cdot\left[\frac{\sin(vq_\parallel t)}{vq_\parallel t}\right]^2$$

$\mathbf{q}\perp\mathbf{v}$: $g^{(2)}(q,t)=exp(-2\Gamma t)$ $\mathbf{q}\|\mathbf{v}$: $g^{(2)}(q,t)\approx\left(\frac{sin\Gamma_S t}{\Gamma_S t}\right)^2 exp(-2\Gamma t)$

Transit: $\Gamma_{tr}=v/s$ **Diffusion**: $\Gamma=Dq^2$ **Shear**: $\Gamma_S=vq_\parallel$

XPCS-flow (q⊥v)



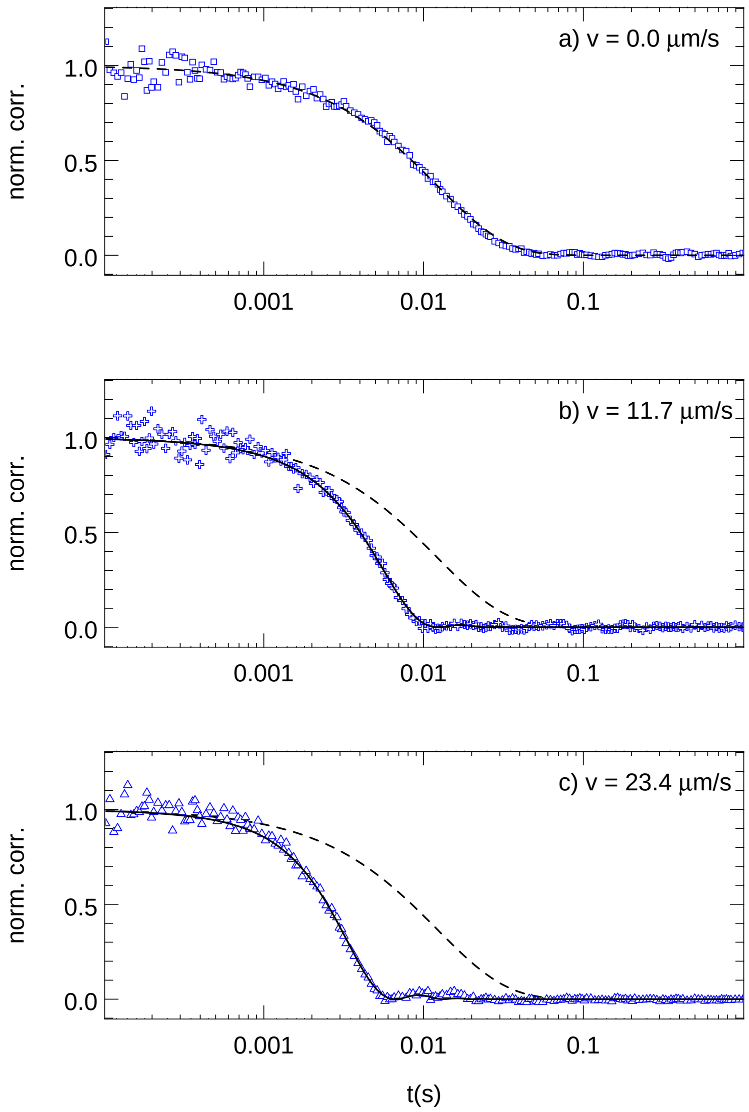
q⊥ v:

Normalized correlation functions $(g^{(2)}(\mathbf{q},t) - 1)/\beta$, obtained in a transverse scattering geometry, shown here for three different values of q at zero flow (filled symbols) and at $v \approx 58.5$ μm/s (empty symbols).

It can be seen that for this flow rate, the influence of the shear flow on the correlation function is, in the first order, negligible. The solid lines show fits to the $v \approx 58.5\mu$m/s data with simple exponential relaxations,

$$g^{(2)} = 1 + \beta \exp(2\Gamma t)$$

XPCS-flow (q||v)



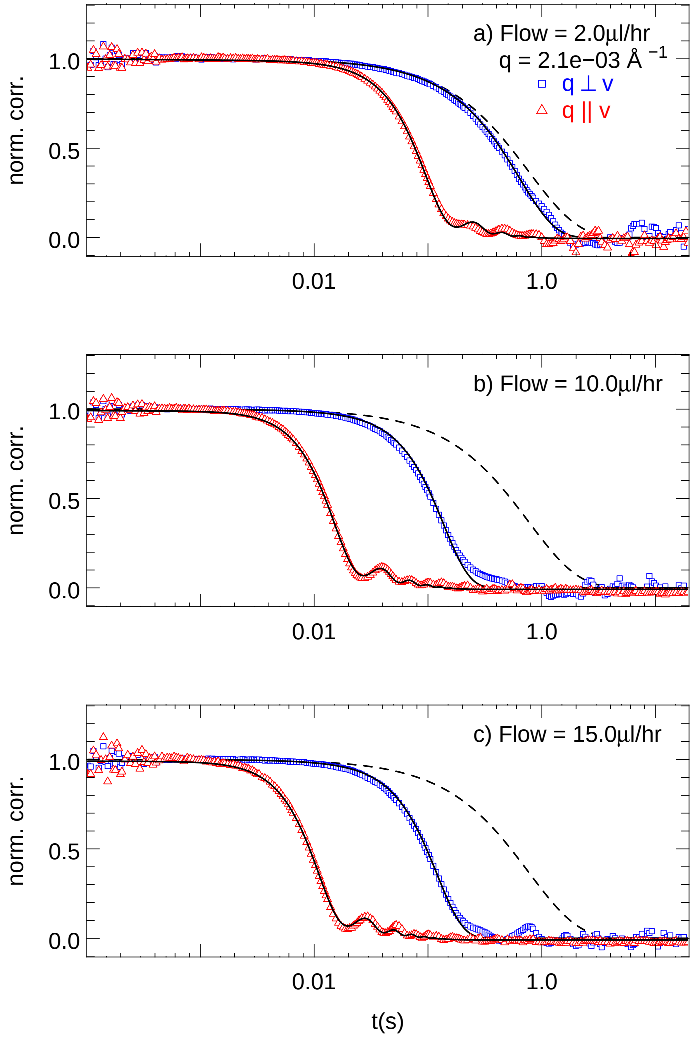
q|| v:

Normalized correlation functions $(g^{(2)}(\mathbf{q},t) - 1)/\beta$, obtained in longitudinal scans, at q=1.3×10^{-3} Å^{-1} and three different flow velocities - (a) $v = 0$, (b) 11.7 μm/s, and (c) 23.4 μm/s.

The dashed lines show the fits to the zero flow correlation function (panel a). Solid lines are least square fits to the non-zero flow data with,

$$g^{(2)}(q,t) = 1 + \beta \exp(2\Gamma t) \cdot \left[\frac{\sin(\Gamma_S t)}{\Gamma_S t}\right]^2$$

XPCS - flow, latex / glycerol



(De≈1)

q⊥v

$$g^{(2)}(q,t) = 1 + \beta \cdot \exp\left[-2Dq^2 t\right] \cdot \exp\left[-(\nu_{tr} t)^2\right]$$

q||v

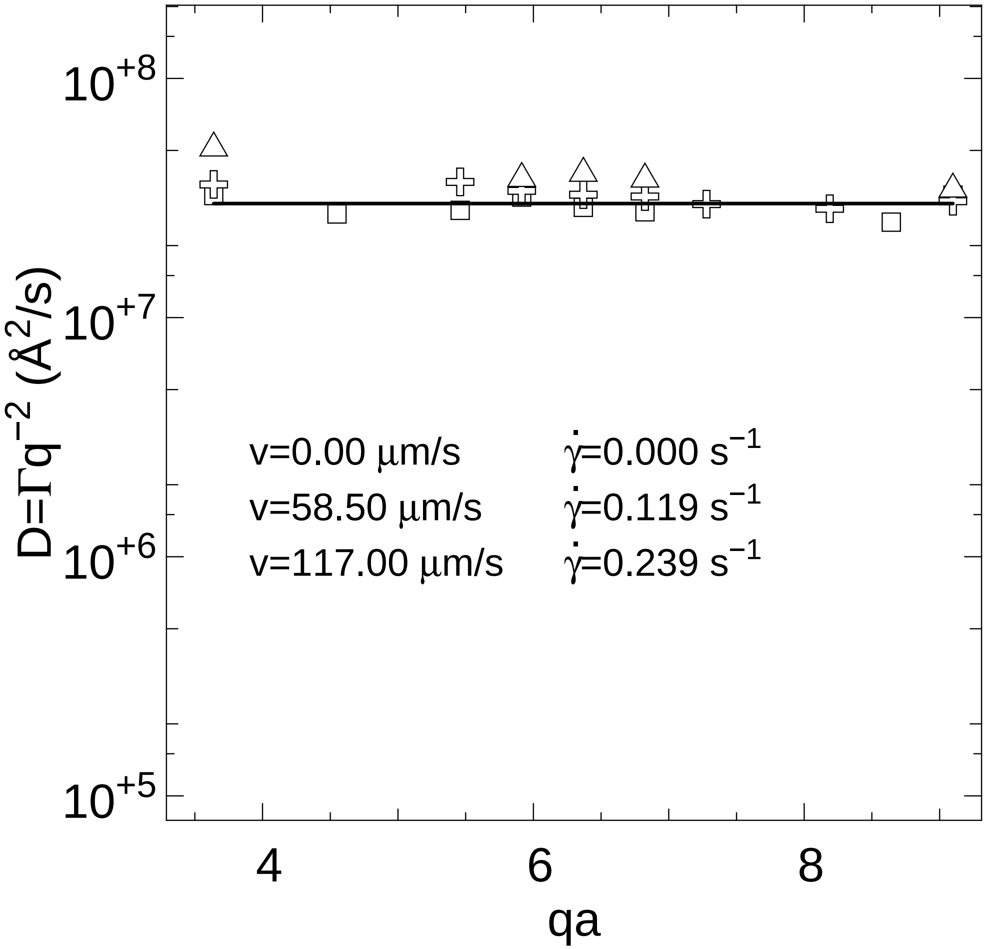
$$g^{(2)}(q,t) - 1 =$$

$$\beta \exp\left[-2Dq^2 t \left(1 + \frac{\dot{\gamma}^2}{3} t^2\right)\right] \cdot \exp\left[-(\nu_{tr} t)^2\right] \cdot \frac{\left|erf\left(\sqrt{i\Gamma_S t}\right)\right|^2}{\Gamma_S t}$$

$$\Gamma_S \propto q_{\|} v$$

S. Busch, T. Jensen, Y. Chushkin, and A. Fluerasu, submitted, EPJE (2007)

XPCS-flow: measuring diffusive dynamics



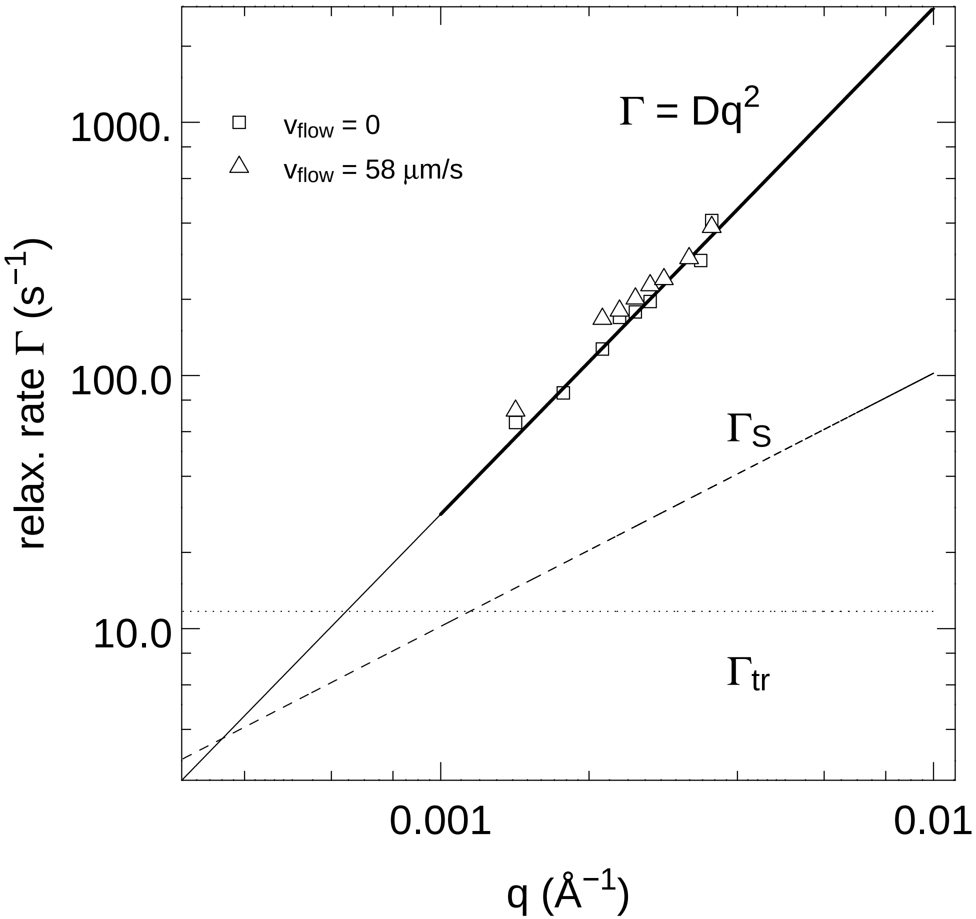
Diffusion constant D associated with the Brownian dynamics of free colloidal particles (radius a) in shear flow as a function of q measured in a transverse ($\mathbf{q}\perp\mathbf{v}$) scattering geometry for three volume flow rates. The solid line shows the value for the diffusion constant calculated by using the Einstein-Stokes equation,

$$D_0 = \frac{k_B T}{6\pi\eta a}.$$

The shear rates are estimated using,

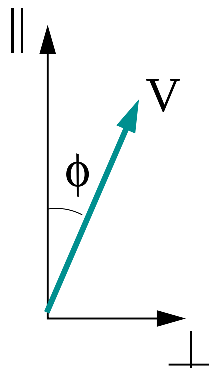
$$\dot{\gamma} = \frac{3v}{R}.$$

XPCS-flow: measuring diffusive dynamics



Dispersion relationships:

- **diffusion** $\Gamma = D_0 q^2$
- **shear** $\Gamma_S = v\phi q$
- **transit** $\Gamma_{tr} = v/s$

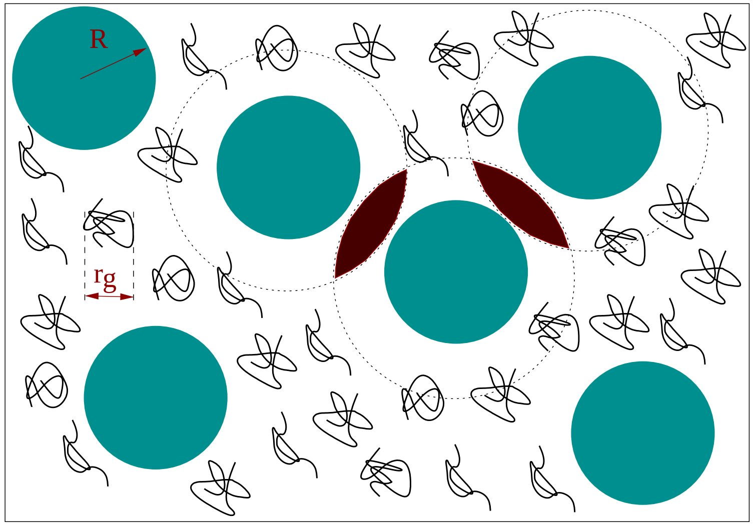


E.g. $D_0 \approx 2.84{\cdot}10^7$ Å/s, $\phi \approx 0.01$ (0.5 deg)
$v = 58,\ \mu$m/s

XPCS-flow: outlook

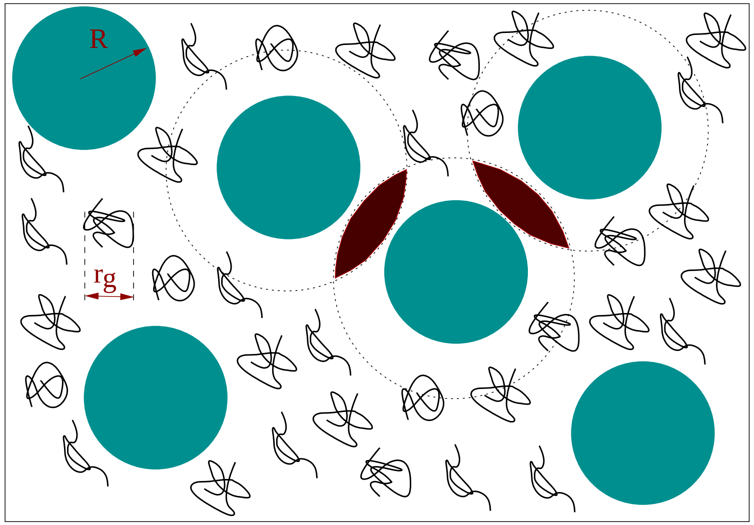
- Dynamics in high concentration suspensions under flow
- Aggregation phenomena in soft matter and biological systems
- Dynamics in attractive suspensions (depletion gels).

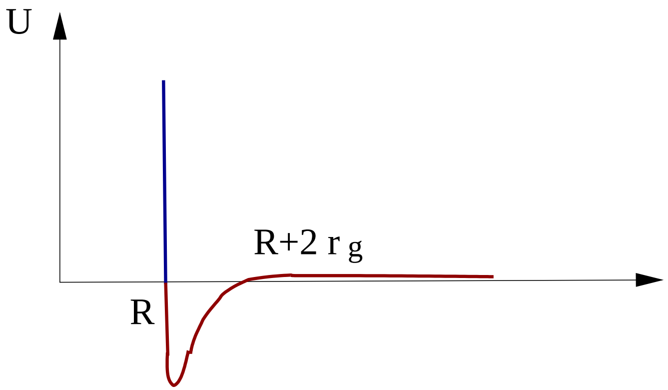
PMMA/PS depletion gels



- Mixture of Poly(MethylMethacrylate) PMMA particles (spherical, R ≈ 1000 Å) coated with poly-12-hydroxystearic acid and free polymer (polystyrene) in cis-decalin
- Entropic forces between the polymer coatings layers → infinite repulsion
- Depletion effect due to the free polymer → attractive potential

PMMA/PS depletion gels





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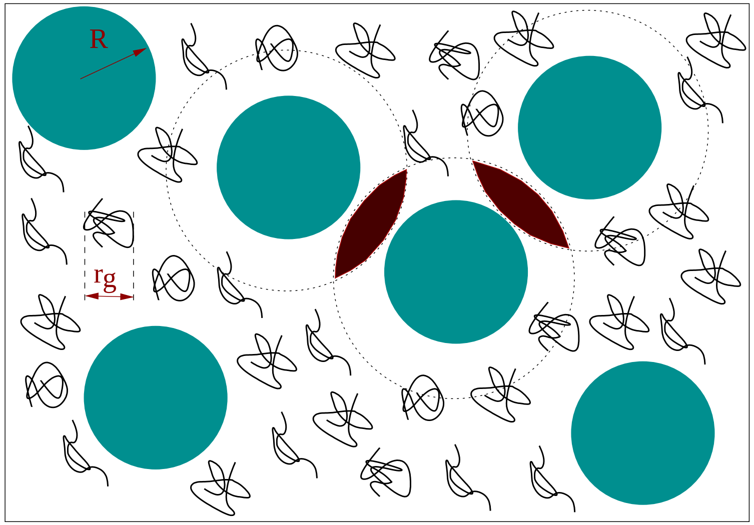

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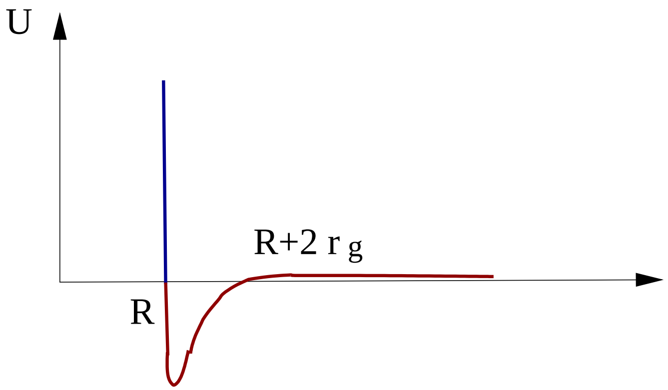
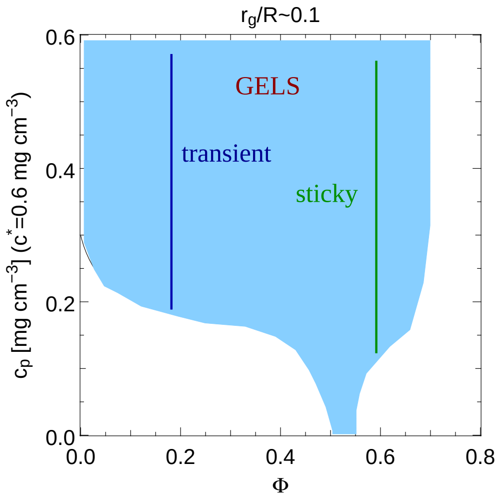
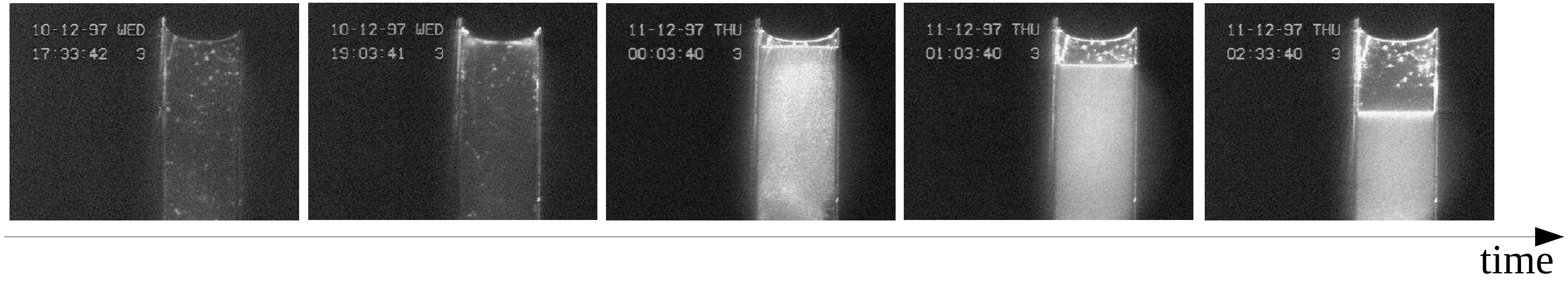
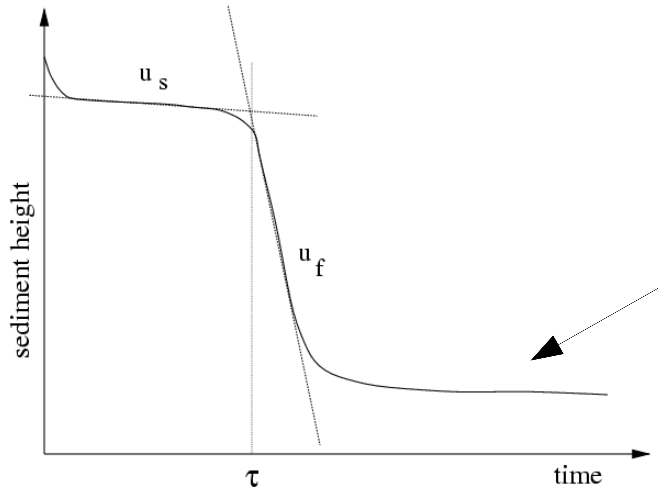




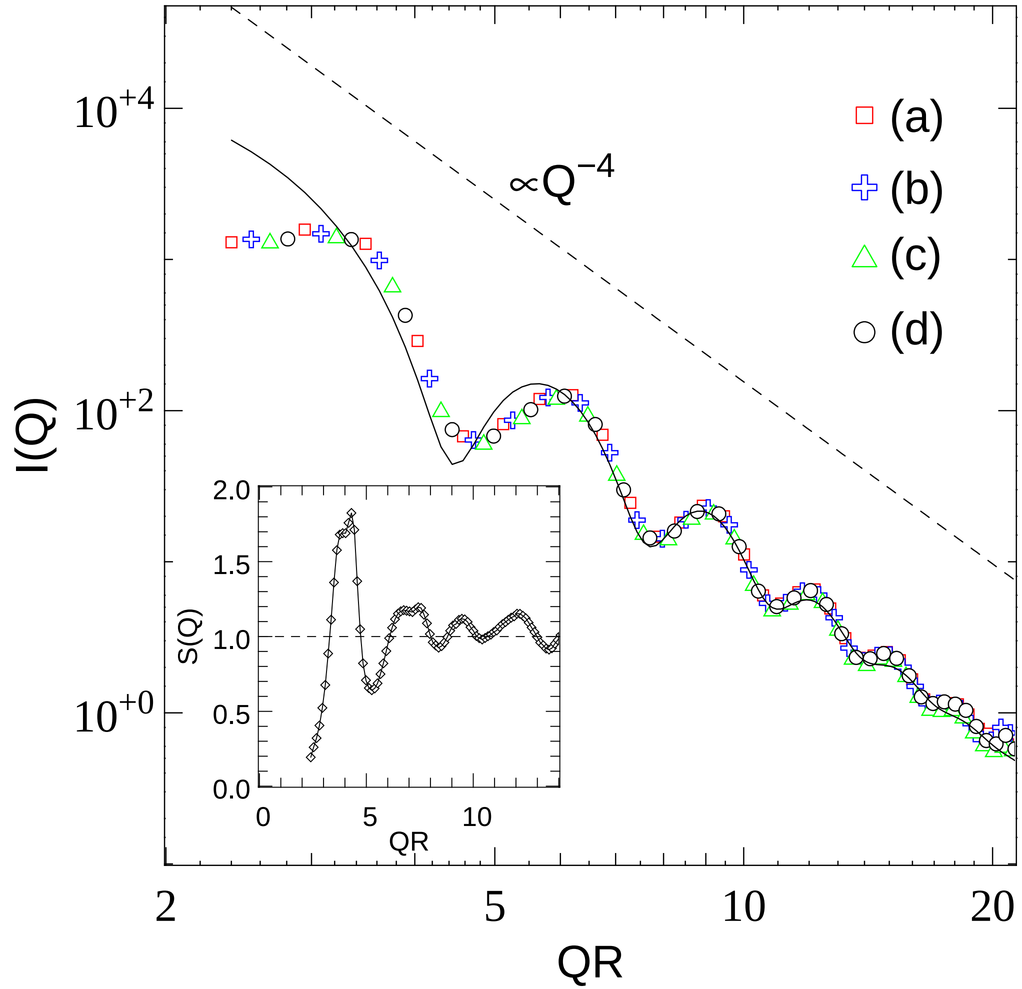
PMMA/PS transient gels



(from W.C.K. Poon et al., Faraday Discuss., 112, 143−154)



Static analysis

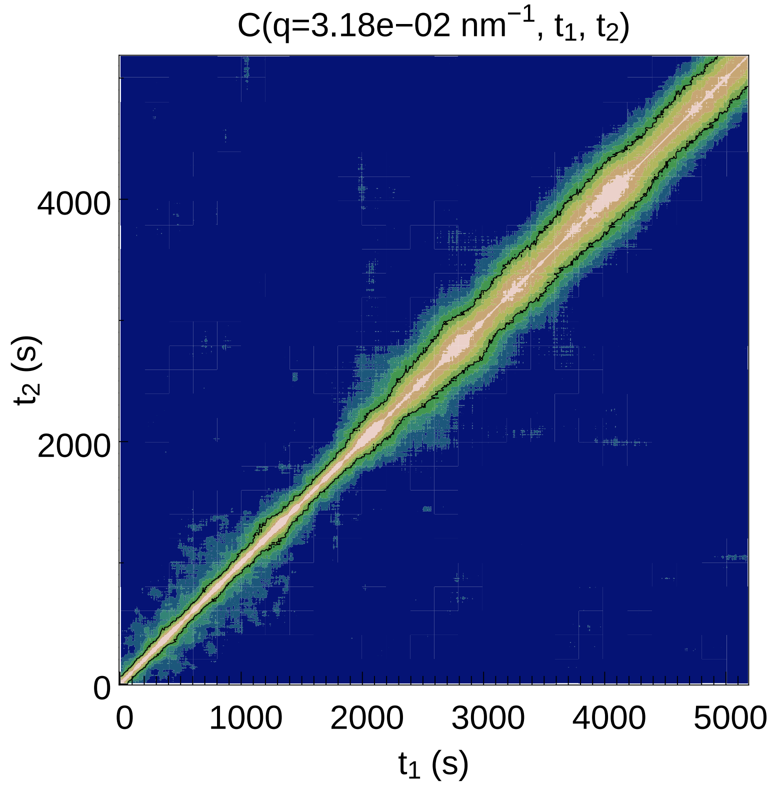


The static scattering is constant during the transient gelation process

SAXS profiles at 4 different times during the transient gelation process . The black line shows the form factor of polydisperse spherical colloids resulting from a least-square fit with a hard sphere form factor and a Schulz particle size distribution. The deviation between data and model at low QR is due to the structure factor (inset) caused by interactions between the colloids. The dashed line is an eye-guide and has slope -4.

Two-time analysis*

Two-time correlation functions: $C(Q,t_1,t_2) = \frac{\langle I(Q,t_1)I(Q,t_2)\rangle_{pix}}{\langle I(Q,t_1)\rangle_{pix}\langle I(Q,t_2)\rangle_{pix}}$



average time ("age"):
$t_a = \frac{t_1+t_2}{2}$

time difference:
$t = \delta t = |t_1 - t_2|$

* M.Sutton et al., Optics Express **11**, 2268 (2003).

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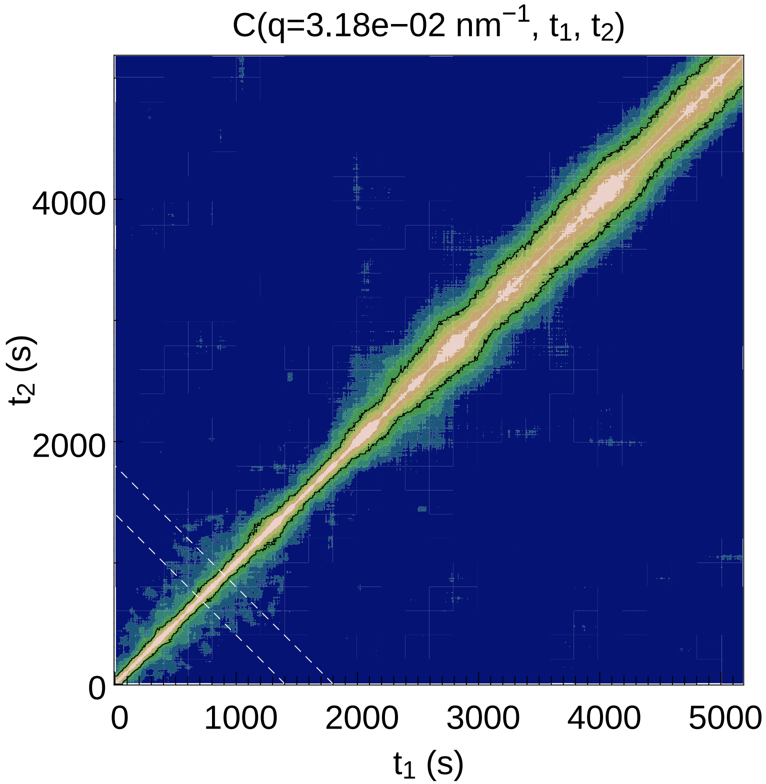

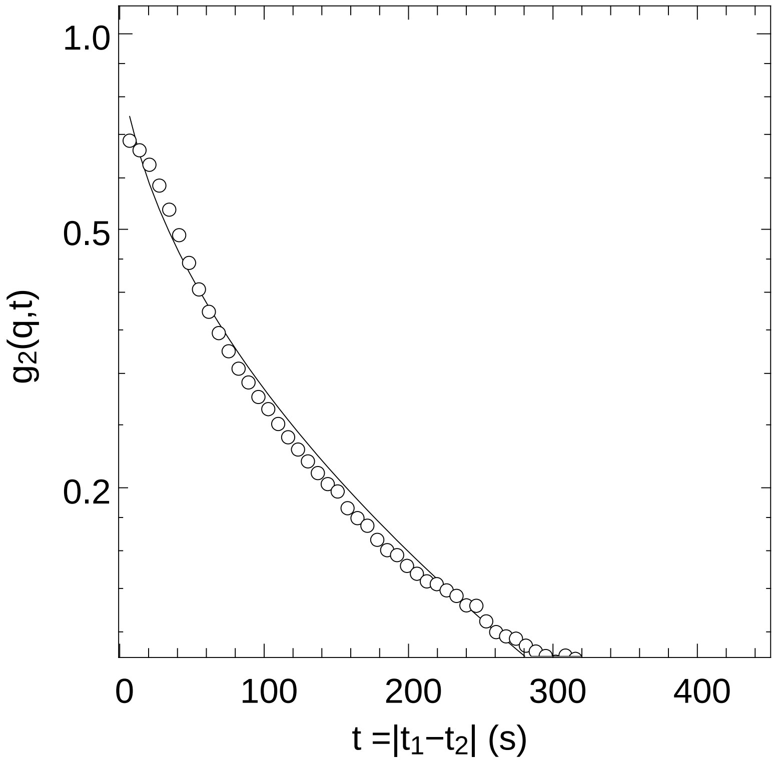

$$g_2 = \beta exp\left(-(\Gamma t)^{\gamma}\right) + g_{\infty}$$

$\Gamma = 4.57\text{e-}3\ s^{-1}$ $\gamma = 0.57$

Two-time analysis

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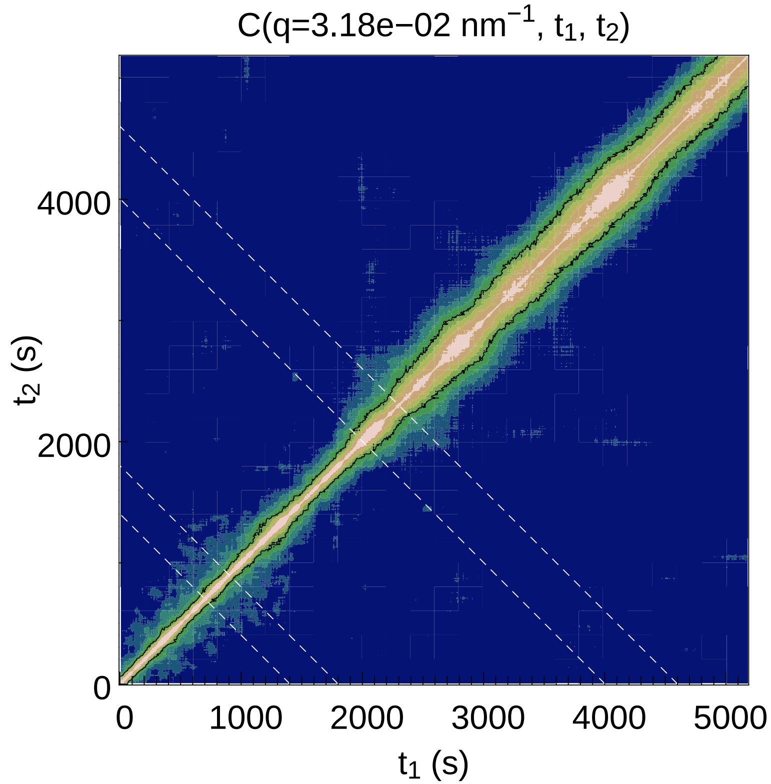
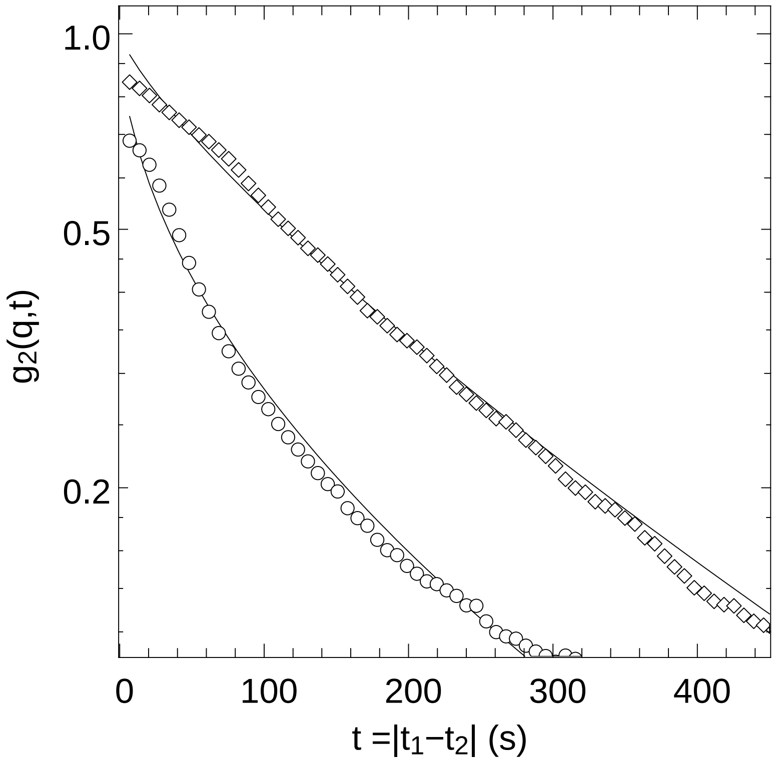


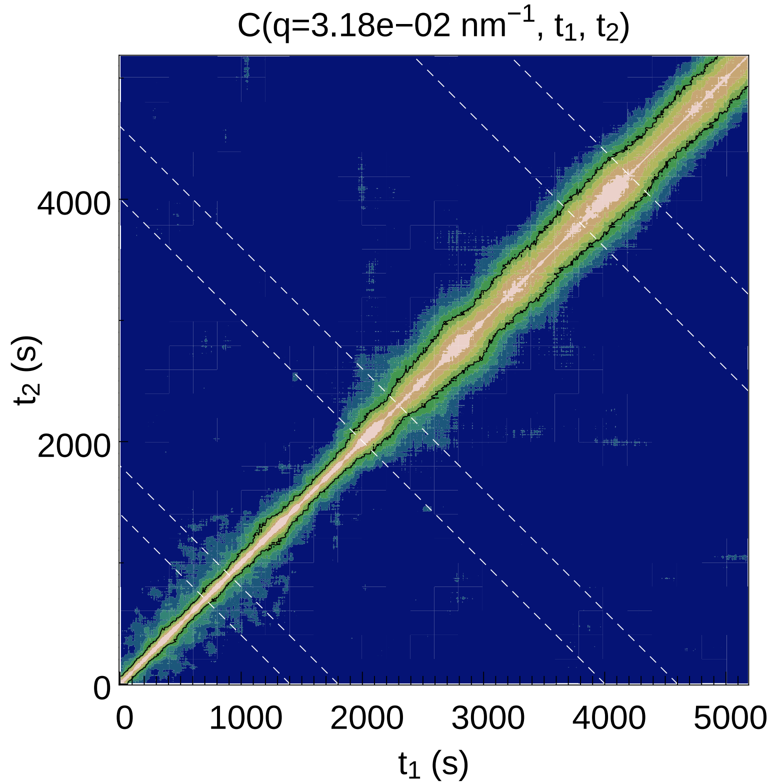


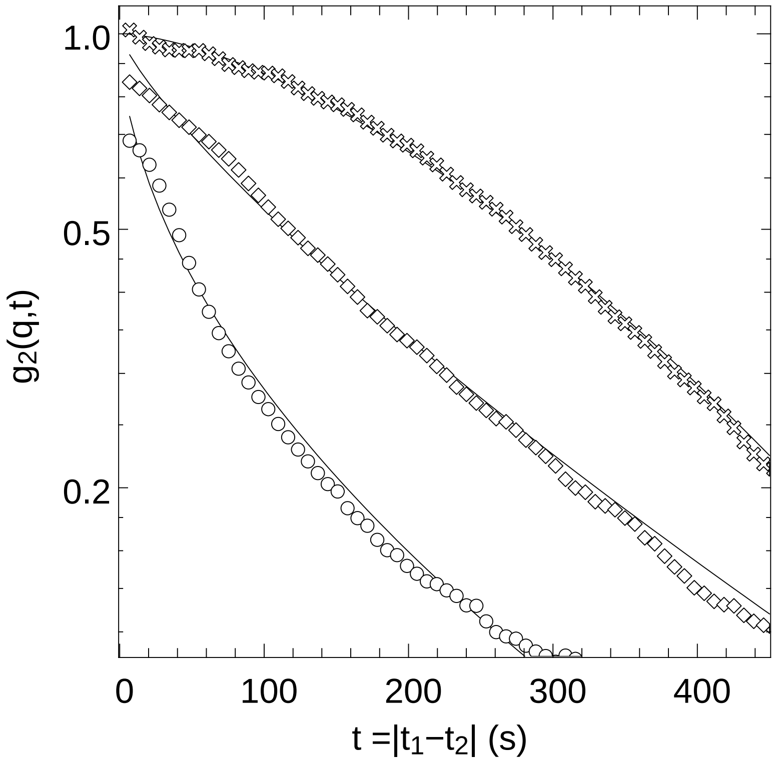
$$g_2 = \beta exp\left(-(\Gamma t)^{\gamma}\right) + g_{\infty}$$

Γ = 2.4e-3 s^{-1} γ = 0.82

Two-time analysis

Two-time correlation functions: $C(Q,t_1,t_2) = \frac{\langle I(Q,t_1)I(Q,t_2)\rangle_{pix}}{\langle I(Q,t_1)\rangle_{pix}\langle I(Q,t_2)\rangle_{pix}}$



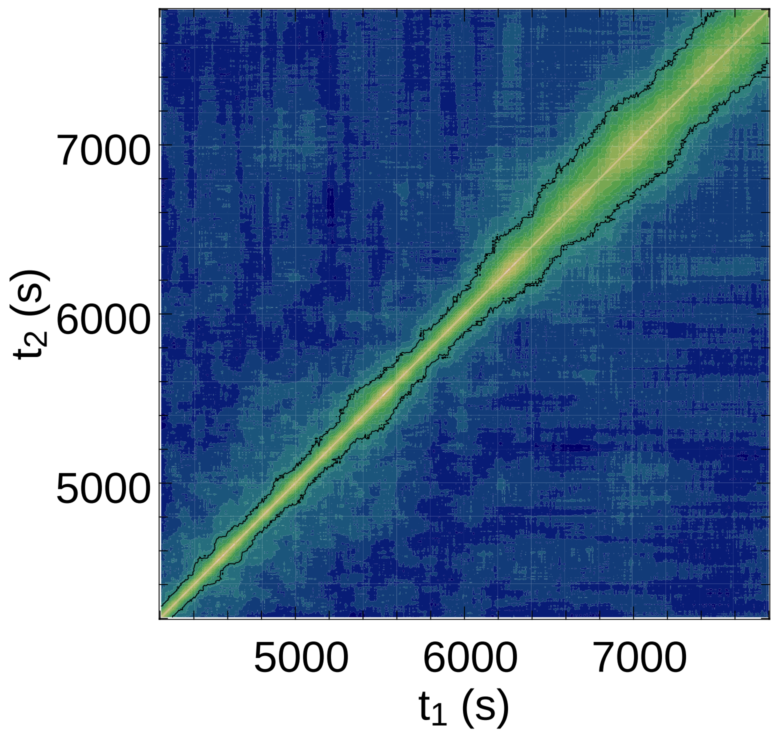


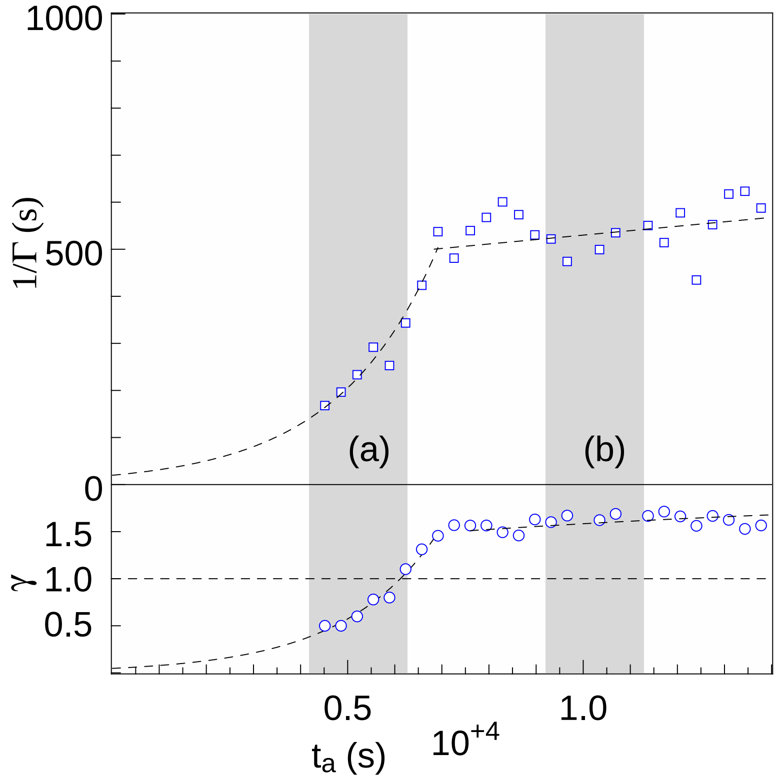
$$g_2 = \beta exp\left(-(\Gamma t)^{\gamma}\right) + g_{\infty}$$

$\Gamma = 1.9\text{e-}3\ s^{-1}$ $\gamma = 1.61$

Two-time analysis

Two-time analysis: $g_2(Q,t_a,t) = \beta exp\left(-(\Gamma t)^\gamma\right) + g_\infty$





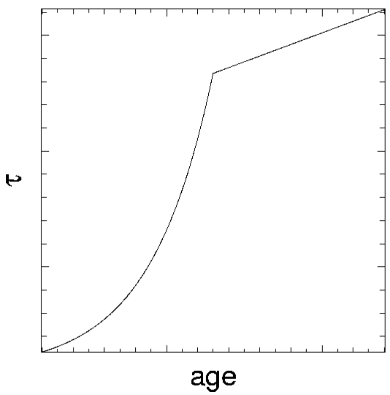
Dynamics in “jammed” systems

“Signature” of a jammed system*:

$g_1(t) = exp\left(-(\Gamma t)^{\gamma}\right)$, with $\gamma \approx 1.5$!

$\Gamma \propto Q$; $(<\Delta r^2> \propto t^2)$

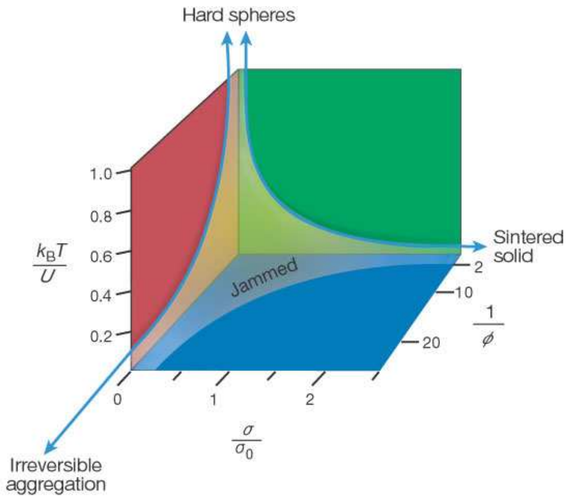
“Aging”



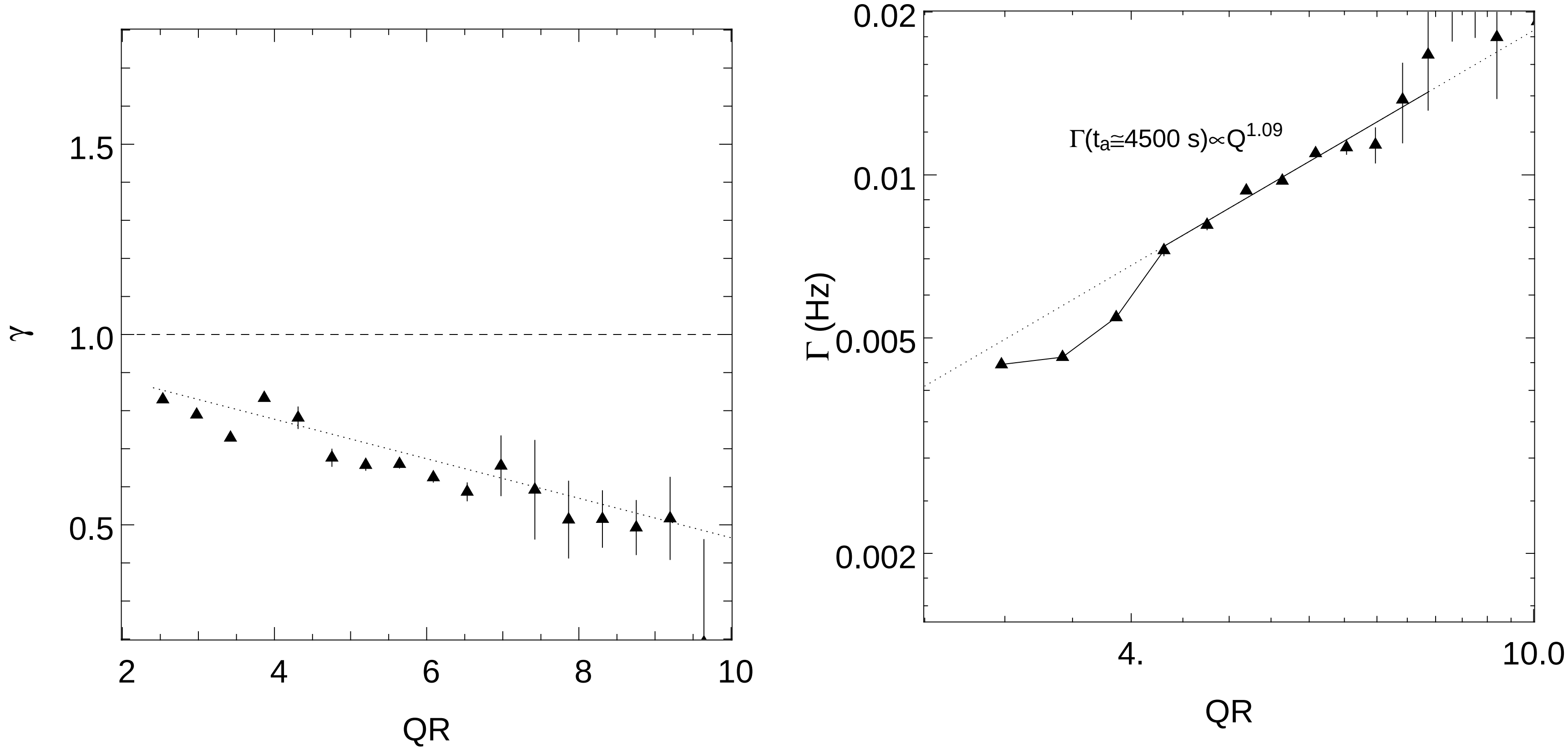
* L. Cipelletti et.al., *Faraday Discuss.*, 2003, **123**, 237
* M. Bellour et al., PRE **67**, 031405 (3003).

C. Caronna et al., PRL, in press (2008)
P. Falus et al., PRL **97**, 066102 (2006)
A. Roshi et al., PRE **74** 031404 (2006)
B. Chung et al., PRL **96** 228301 (2006)
A. Robert et al., EPL **75** 764 (2006)

V. Trappe et. al., Nature **411** 772



Dynamics and aging in PMMA/PS transient gels



A. Fluerasu, A. Moussaïd, A. Madsen, and A. Schofield, Phys. Rev. E **76**, 010401(R) (2007)

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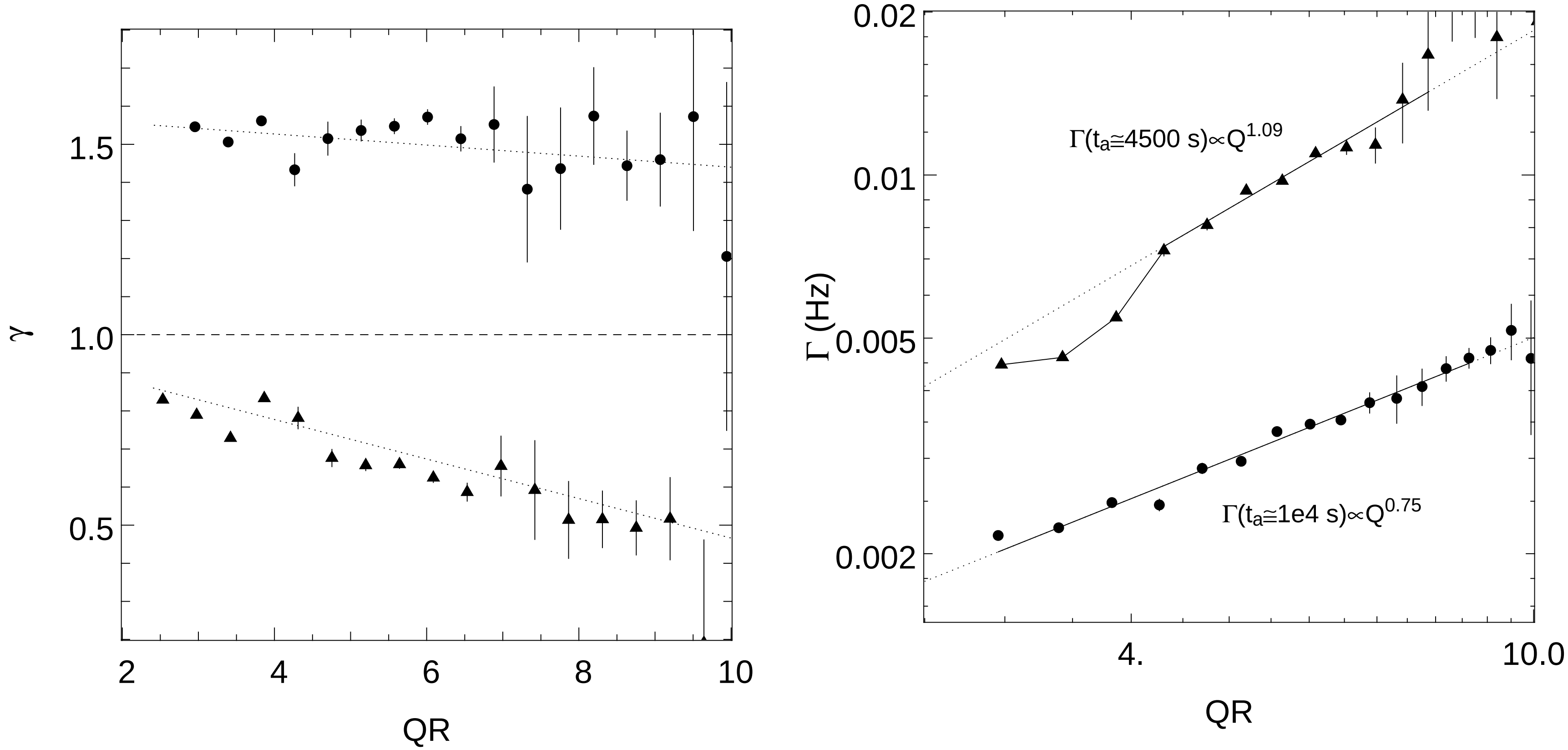

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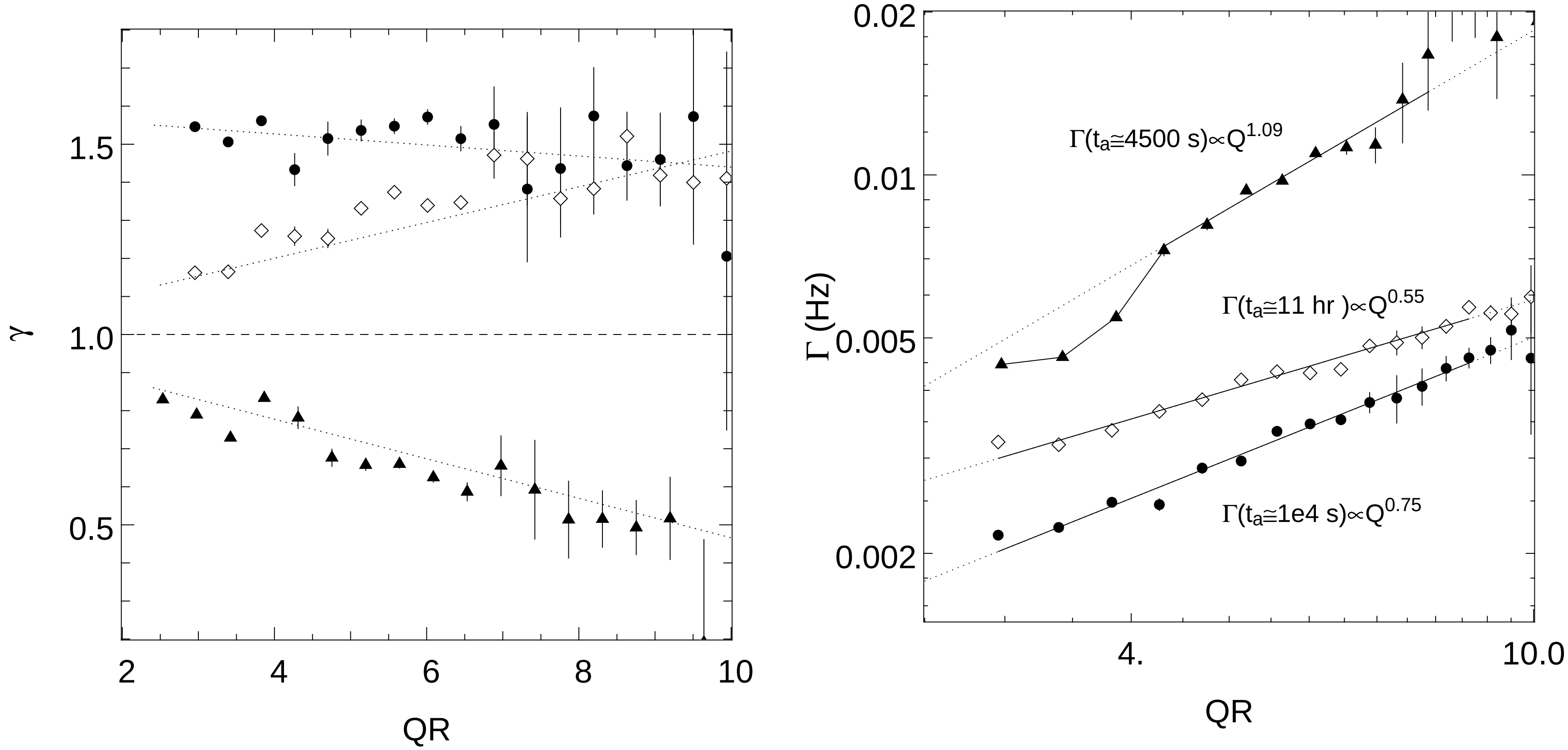

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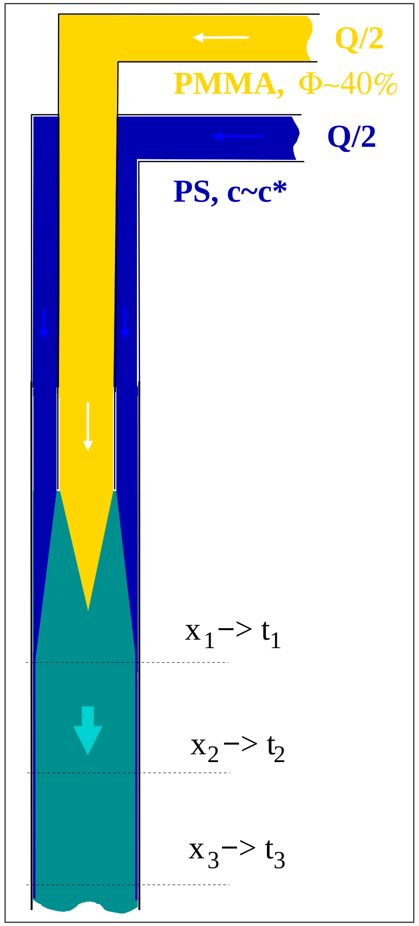
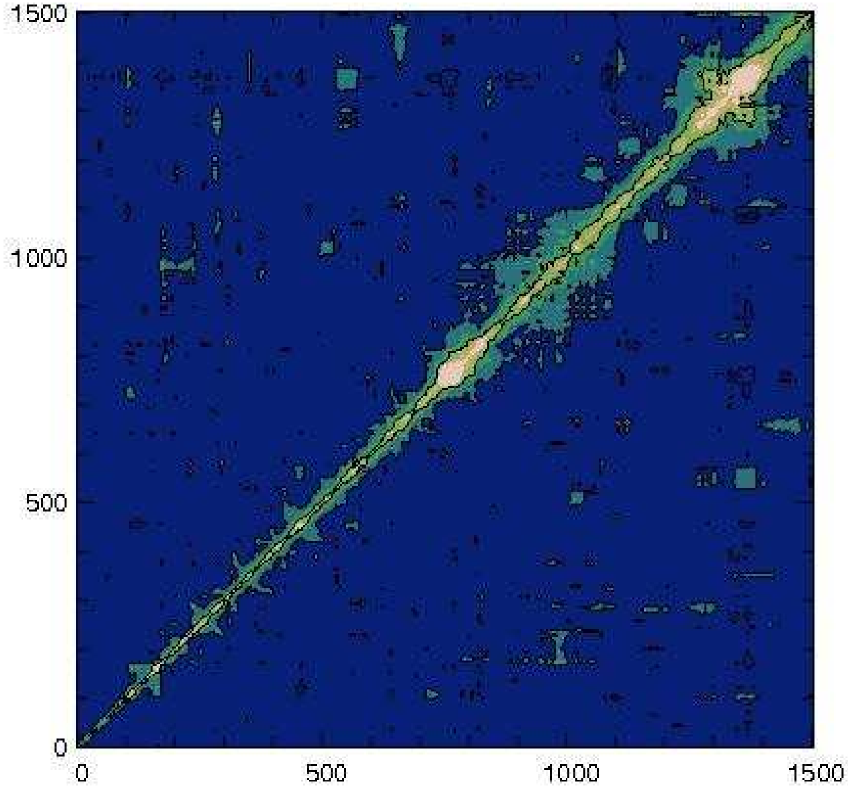
Colloidal gels in microfluidic systems

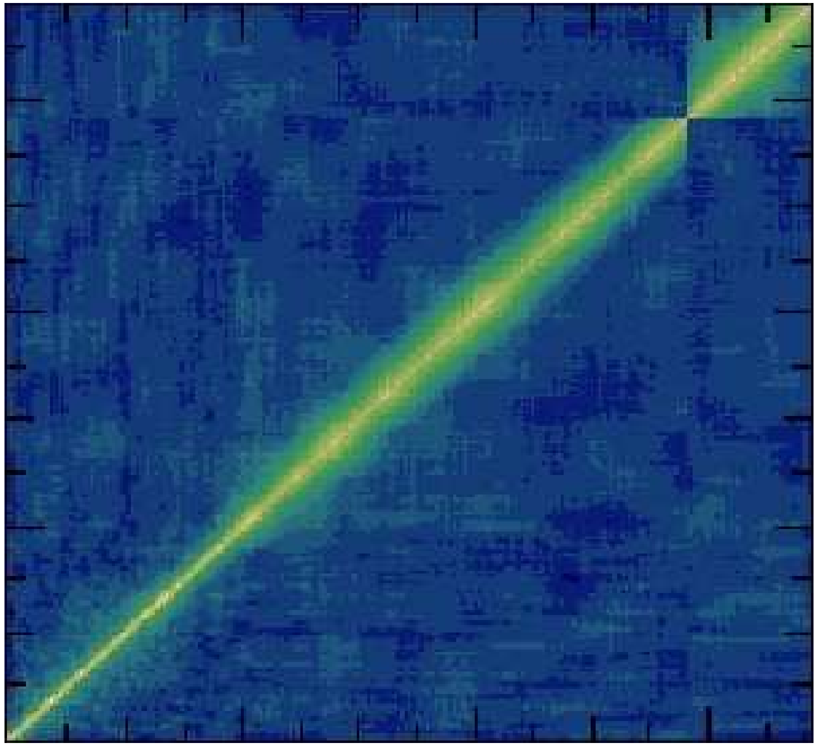


Q=0.5 μm/h, x=0 | x=1 | x=2

Q=50 μm/h, x=0 | x=1 | x=2

Dynamical heterogeneities and “rare events”



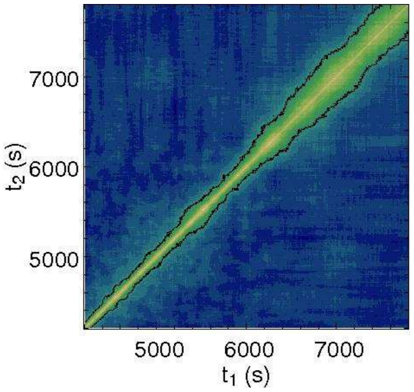


Dynamical heterogeneities, $g^{(4)}, \chi_4, \ldots$, talk by DJD
DLS $\longrightarrow$ L. Cipelletti et al., DJD et al.
XPCS $\longrightarrow$ V. Trappe, A. Robert, L. Cipelletti et al., PRE 2007

“Rare events”

A. Moussaïd, A. Fluerasu, work in progress

Conclusions and outlook

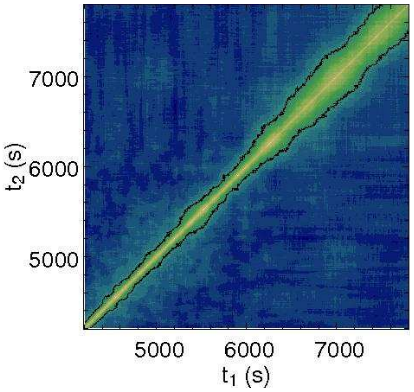


- XPCS + "microfluidics": first studies have been performed on colloidal suspensions undergoing (simple) Brownian motion
  → it is possible to measure the diffusive dynamics
- X-ray + "microfluidics" studies of *nucleation, growth and aggregation phenomena in soft matter*
  e.g.: sol-gel proc. - ongoing LT collaboration ESRF/CNRS-Rhodia Bordeaux
  → Colloidal gels, Aggregation of fibroin, etc.
- these methods mitigate beam damage, which is a major limiting factor in the study of soft matter and biological systems and will become even more important at future light sources

- Changes in the aggregation state of proteins are responsible e.g. for cataract formation in the human eye lens and neuronal cell death involbed in Alzheimer's disease.
  → kinetics and the dynamics of such processes using XPCS + microfluidics.

Technical/operational requirements/wishes:

Conclusions and outlook



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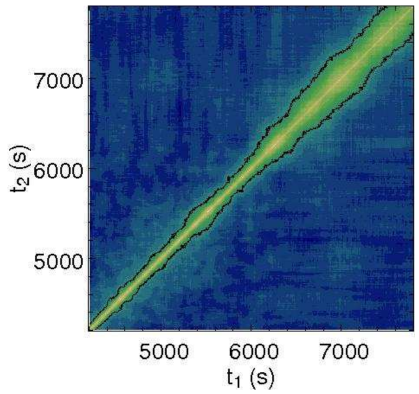
Technical/operational requirements/wishes:

- MORE PHOTONS! higher current; long(er) in-vacuum undulators; lower emmitance; microfocus; faster (2D) detectors . . .

Conclusions and outlook



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Technical/operational requirements/wishes:

- MORE PHOTONS! higher current; long(er) in-vacuum undulators; lower emmitance; microfocus; faster (2D) detectors ...
- complementary methods: direct observation, light scattering, microscopy, rheology, ...

Collaborators

Anders Madsen, Abdellatif Moussaïd,
Yuriy Chushkin, Federico Zontone (Troïka group, ESRF)
Anne Martel, Christian Riekel, Narayanan Theyencheri (ESRF)
Ray Barret, Henry Gleyzolle (ESRF)

Jean-Baptiste Salmon, Jacques Leng, Fanny Destremaut
(CNRS-Rhodia, Bordeaux)
Sebastian Busch (TU München), Torben Jensen (Univ. Copenhagen)
Peter Falus (ILL)
Erik Geissler (Univ. Joseph Fourier, Grenoble)
Mark Sutton (McGill University, Montreal)

http://www.esrf.eu/UsersAndScience/Experiments/SCMatter/ID10A/