ESRF	**Experiment title:** **Slow dynamics and aging of concentrated colloidal gels**	**Experiment number**: SC 980
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Shifts: 18	**Local contact(s)**: Aymeric Robert	*Received at ESRF:*

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Report:

Experiment SC980 was originally proposed to investigate by XPCS the slow dynamics of polystyrene colloidal gels, a jammed system. Preliminary measurements during experiment SC982 (feb. 2002) have shown that the scattering from this system is too low to use the CCD detector. Since the CCD camera is essential to access the ultraslow dynamics, we decided to test several other jammed systems during SC980.

The analysis of the data is still in progress. Here, we report preliminary results on some of the systems together with some general remarks. A more complete report will be presented as soon as possible.

The samples that we investigate are (in the following a is the particle radius and ϕ the volume fraction):

Reversible aggregation

1) Carbon black particles (CB) ($a = 250$ nm, $\phi = 0.08$, suspended in mineral oil, sample initialized by applying a shear [ultrasounds])
2) Zinc oxide (ZO) ($a = 100$nm, $\phi \sim 0.1$, suspended in mineral oil with different amounts of dispersant, sample initialized by applying a shear [ultrasounds])

Irreversible aggregation

1) Aluminum oxide particles (AO) ($a = 225$ nm, $\phi = 0.3$, suspended in water, destabilized by changing the ion concentration in the surrounding medium)
2) Silica particles (Si) ($a = 225$nm, $\phi = 0.2$, suspended in water, destabilized by changing the pH)

All these systems form colloidal gels because of the attractive interactions between the particles.

We find that the scattering intensity for all these systems is high enough to allow CCD-based XPCS measurements of their dynamics. As a general behavior, all systems exhibit an ultraslow relaxation of the intensity correlation function $g_2(t)$, whose characteristic time is as long as thousands of seconds. To our knowledge, these measurements are the first example of ultraslow dynamics measured by XPCS, thus demonstrating the applicability of this technique to dynamical processes lasting hours.

Figures 1 and 2 show the q-dependent correlation functions of a CB- and a AO-sample at a fixed age. For the CB [1], we find that the correlation functions are well described by an unusual faster-than-exponential decay,

$g_2(q,t) - 1 \sim \exp\{-(t/\tau)^p\}$, with $p \sim 1.2$ and with the relaxation time τ decreasing linearly with scattering vector q. A similar behavior has been previously observed, on much larger length scales, for completely different systems: highly diluted, strongly aggregated colloidal gels [2] and concentrated surfactant phases

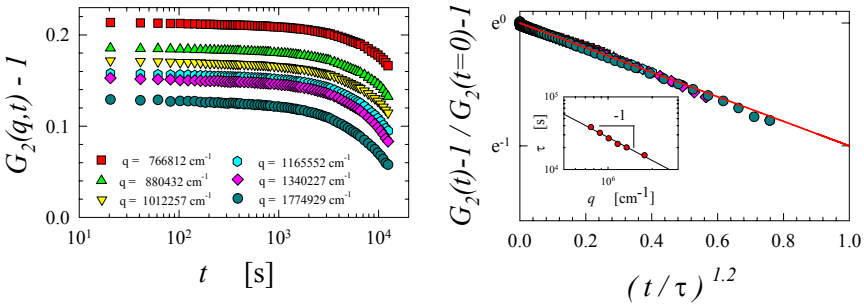

Figure 1: g_2-1 for various q for CB. As demonstrated in the right hand plot all functions are well described by a stretched exponential decay (p=1.2); the relaxation time decreases linearly with q (inset).

[3]. The q^{-1} dependence of τ reveals that a ballistic-type motion determines the long time relaxation. A tentative interpretation suggests that internal stresses cause the rearrangement of the constituents of the systems, which leads to the final relaxation of g_2.

For the AO sample, we find a similar shape of the correlation functions ($g_2(q,t) - 1 \sim \exp\{-(t/\tau)^p\}$, p=1.5), although almost no q dependence of the relaxation time is observed. This very peculiar behavior is still

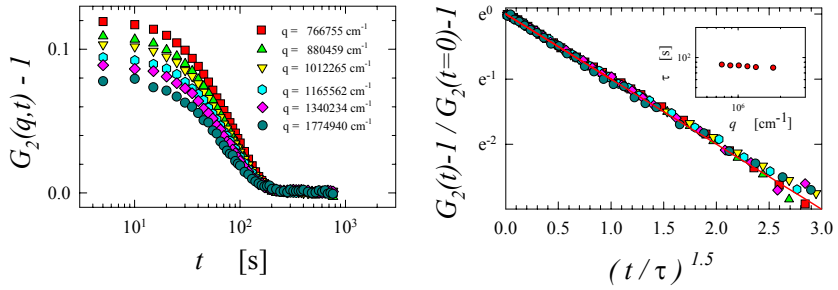

Figure 2: g_2-1 for various q for AO. As demonstrated in the right hand plot all functions are well described by a stretched exponential decay (p=1.5); the relaxation time is nearly independent of q (inset).

poorly understood. The particle motion may be determined by a reaction rate. In the course of destabilization the particles first aggregate in the secondary minimum of the interparticle potential and then hop to the primary minimum; the hopping time could set a length- and hence q-independent time scale for the dynamics. Unfortunately, this q-independent dynamics could also be an artifact due to beam instabilities.

To test this issue, we have measured g_2 for a static sample (Vycor glass). As can be seen in fig. 3, a q-independent relaxation is observed, with a relaxation time of the order of 2000 s. We point out that much longer relaxation times have been measure for the CB, setting a lower limit for beam stability much longer than that observed in fig. 3. Therefore, beam instability appears to vary from run to run: this issue will require a more complete investigation in order to asses the limits of XPCS ultraslow measurements at ESRF.

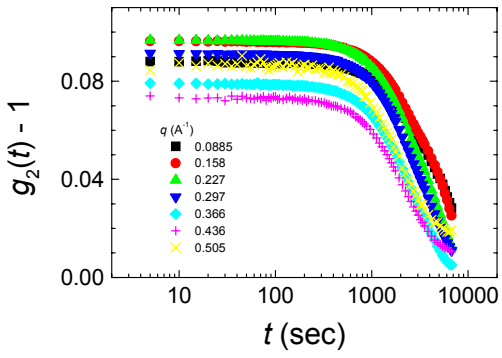

Figure 3: g_2-1 for various q for a static sample, Vycor glass. The decay of the correlation function indicate beam instabilities on the time scale of 2000 sec.

[1] Trappe, V.; Weitz, D. A. *Phys. Rev. Lett.* **2000**, *85*, 449
[2] Cipelletti, L.; Manley, S.; Ball, R. C.; Weitz, D. A. *Phys. Rev. Lett.* **2000**, *84*, 2275.
[3] Ramos, L.; Cipelletti, L. *Phys. Rev. Lett.* **2001**, *87*, 245503