



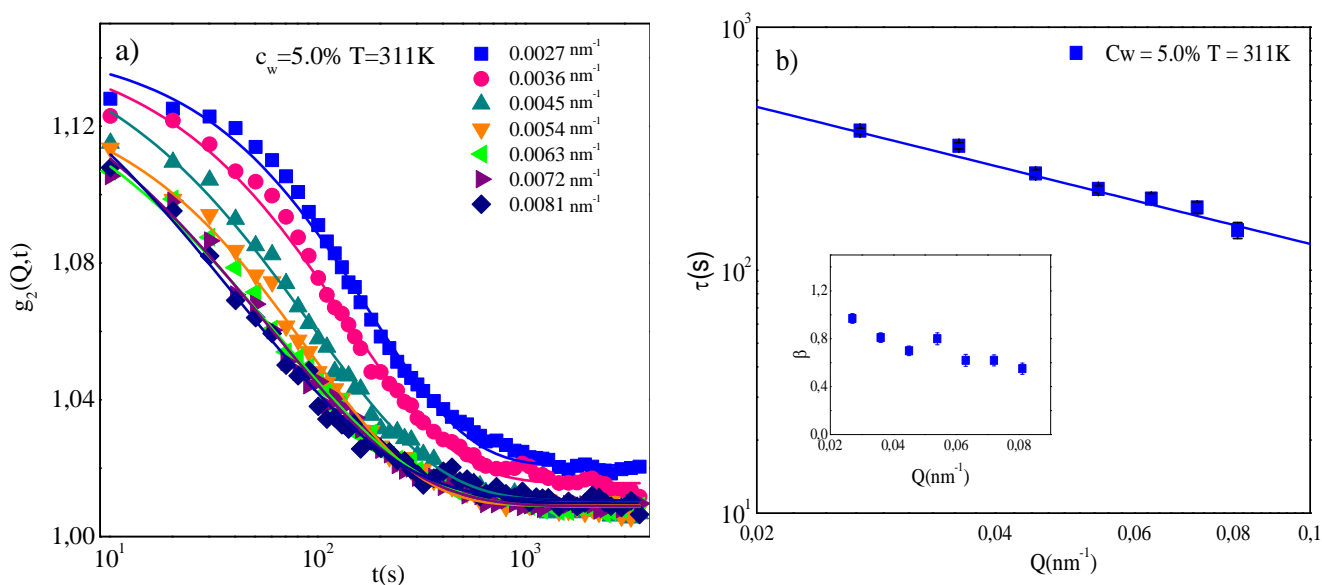
	<b>Experiment title:</b> Fragility and dynamical arrest in soft colloids: the case of an IPN microgel	<b>Experiment number:</b> SC-4655
<b>Beamline:</b> ID10	<b>Date of experiment:</b> from: 08-11-17 to: 14-11-17	<b>Date of report:</b> 09-03-18
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## Report:

X-ray Photon Correlation Spectroscopy (XPCS) measurements have been performed on thermo- and pH-sensitive colloidal microgels. In particular we investigated microgels composed of Interpenetrating Polymer Networks (IPN) of poly(N-isopropylacrylamide) (PNIPAM) and poly(acrylic) acid (PAAc). PNIPAM microgels are strongly thermo-sensitive while PAAc provides an additional pH-sensitivity to the IPN microgel leading to a complex phase behaviour [1]. By increasing temperature, at around 305 K, IPN microgels undergo a volume phase transition (VPT) from swollen to shrunken particles, due to the expulsion of the water absorbed with a swelling capability remarkably reduced as the AAc concentration is increased. This property makes possible to control directly the microgel softness and to vary the fragility of the systems, as recently proposed by Mattson et al. [2]. Previous Dynamic Light Scattering (DLS) measurements were performed by our group [3-5] on low concentrated IPN microgels ( $C_w = 0.1-0.3\%$ ) in a scattering vector  $Q$  range  $0.0067 \div 0.021 \text{ nm}^{-1}$  and in a time scale  $10^{-6} \div 1 \text{ s}$ , during the experiment SC-4655 we investigated concentrations up to  $C_w = 5\%$  and extended time and length scales. Several tests were performed to find the optimal experimental conditions to avoid beam damage on the sample. Finally, we used a 21Kev beam, a 2mm capillary, an attenuator (attGe75) with an exposure time of 0.1s and 10s of sleeping time between an image and the next. In Fig.1 a) an example of the intensity autocorrelation function at  $C_w = 5\%$  and at  $T = 311 \text{ K}$  above the VPT is reported as a function of  $Q$ . The data have been fitted using Kohlrausch-Williams-Watts (KWW) expression

$g_2(Q,t) = b[(A \cdot \exp(-t/\tau_Q)^\beta + 1)]$  where  $\tau_Q$  is an "effective" relaxation time and  $\beta_Q$  measures the distribution of relaxation times (associated with simple exponential decays). The fits are superimposed to the data. The fitting parameters  $\tau_Q$  and  $\beta_Q$  have been plotted as a function of  $Q$  in Fig1 b), in this  $Q$ -range  $\tau_Q$  scales like  $Q^\alpha$  with  $\alpha = (-0.80 \pm 0.03)$ , similarly to what found in many others colloidal systems where the  $1/Q$  dependence suggests a ballistic-like motion of the particles. In this case, at the investigated temperature and concentration, it is accompanied by values of the exponent  $\beta \approx 1$  that decreases with increasing scattering vector.

A comparison with the results obtained through dynamic light scattering measurements performed at lower concentrations and time scales provided a clear picture of the IPN microgel behaviour at the liquid to arrested state transition [6].



**Fig.1** a) Intensity autocorrelation function for an IPN microgel at  $C_w=5\%$ ,  $T=311\text{K}$  and as a function of the scattering vector  $Q$  (symbols). Full lines represent a fit through a KWW expression. b) Relaxation time and  $\beta$ -exponent obtained from the fit procedure as a function of  $Q$

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