ESRF	**Experiment title:** Layering of fluids in ultranarrow gaps	**Experiment number**: SI-611
Beamline: ID10A	**Date of experiment**: from: 1 Nov. 2000 to: 7 Nov. 2000	**Date of report**: 28 February 2001
Shifts: 18	**Local contact(s)**: F. Zontone	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

J.F. van der Veen[1)]*, J.H.H. Bongaerts[2)]*, M.J. Zwanenburg[1)]*, U. Flechsig[1)]*

[1)] Paul Scherrer Institut, Villigen - Switzerland.

[2)] Van der Waals-Zeeman Instituut, University of Amsterdam - The Netherlands.

Report:

We performed a different experiment than the experiment proposed in the application form with the title 'Layering of fluids in ultranarrow gaps'. The reason for this deviation from the plan is two-fold: 1) The new experimental setup, designed to go to ultranarrow gaps and combine x-ray diffraction experiments with force measurements was not fully tested and calibrated at the time of the experiment. 2) Contrary to what we estimated earlier, the speckle contrast and intensity at the ID10A station proved to be sufficiently high for useful x-ray photon correlation spectroscopy (XPCS) experiments on colloidal particles in confined geometry. Therefore, we took the opportunity to make a start with the XPCS experiment SI-612 that was planned later in the allocation period for the ID10C station (not yet open for users).

We have performed x-ray photon correlation spectroscopy experiments (XPCS) on colloidal suspensions confined within a tunable x-ray waveguide (see Fig. 1). The goal of the experiment is to investigate the effect of confinement within a well-defined geometry (a planar slit) on the dynamics of colloidal particles.
The waveguide consists of two separate flat surfaces which can be positioned opposite to each other at at a distance ranging from a few tens of nanometers to several microns. The experimental setup and the waveguiding technique have been described in refs. [1] and [2].
The colloidal suspension is inserted in the gap between the surfaces, which at the same time serves as the guiding layer for the x rays. The colloidal suspensions used in the experiment are monodispers silica spheres of either 25, 110 or 210 nm in diameter. The solvents used were ethanol, water and a water/glycerol mixture. The area around the waveguide is enclosed by a cylinder with mica windows to prevent evaporation of the solvent. Evaporation of the solvent changes the colloid density and therefore the properties of the suspension. This has been a problem in earlier experiments on colloidal suspensions within the waveguide, especially when ethanol and water were the solvent.
We performed XPCS experiments at 13 keV photon energy with the scattering vector q both parallel and perpendicular to the plane of the confining surfaces (see Fig. 2). For $q_{//} = 0$ and as a function of $q_{\perp}$, the different waveguide modes are resolved. The modes peaks exhibited only a weak dynamic speckle contrast ($\leq$1%). This is caused by the fact that each waveguide mode is is built up from the scattering contribution of many colloidal particles, resulting in an averaging over many positions.

For in-plane scattering ($q_{//} \neq 0$) we measured time correlation functions with dynamic speckle contrasts of 40-50%. An example is shown in Fig. 3. For very large waveguide gaps (bulk fluid), we measured an exponential decay of the time correlation function $g_2(t)$. Plotting the inverse decay time τ^{-1} of the correlation function $g_2(t)$ versus $q_{//}^2$, we obtain a straight line, as is expected for single particle diffusion.
For small gap sizes (less than ~2.5 μm) we observed non-exponential long-time tails in $g_2(t)$ at small $q_{//}$ (see Fig. 3). This indicates a confinement effect. Additional experiments as a function of gap width and over a wider range of momentum transfers are required.

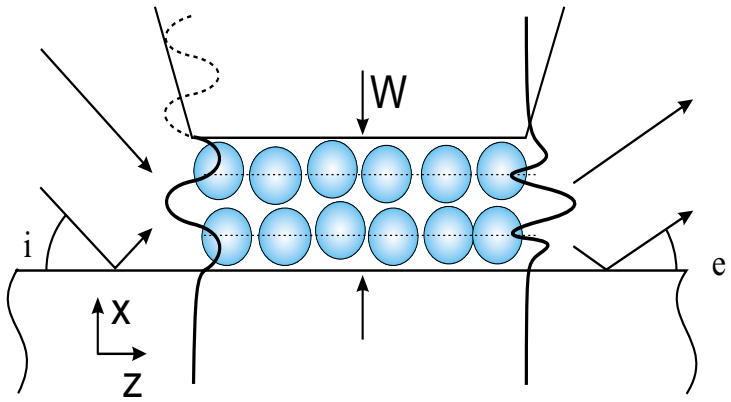

Fig. 1 Schematic drawing of the waveguide setup. The transversely coherent x rays are incident from the side at a grazing angle and propagate between the confining surfaces. The motion of the colloidal particles will result in a fluctuating speckled far field diffraction pattern behind the exit of the waveguide.

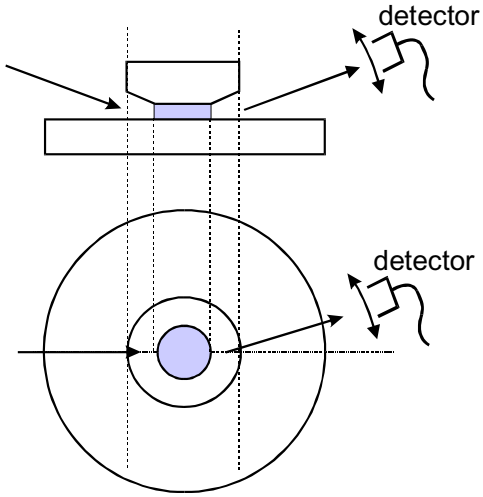

Fig. 2 Schematic of the scattering geometry. Top and bottom panels show the scattering directions perpendicular and parallel to the plane of the confining surfaces. The dark area represents the colloidal suspension.

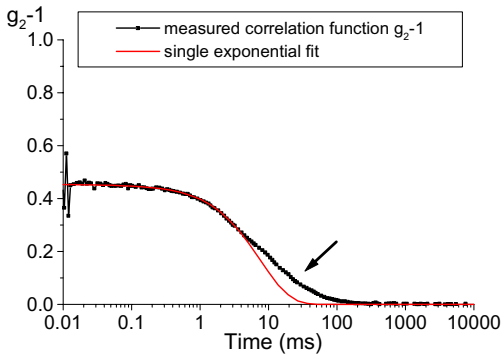

Fig. 3 Time correlation function $g_2(t)$ measured on a solution of 7 wt% 210 nm diameter silica spheres in a 3cP water/glycerol mixture at a gap width of ca. 700 nm. The momentum transfer was $q_{//} = 5.8\ 10^{-3}$ nm^{-1}. Fitted to the first part of the correlation function is a single exponential decay. The deviation from the single exponent (indicated by arrow) cannot be fitted by an exponential decay and is probably an effect of the confinement.

References:

[1] M.J. Zwanenburg, H.G. Ficke, H. Neerings, and J.F. van der Veen, Rev. Sci. Instrum. 71 (4) pp. 1723-1732 (2000).
[2] M.J. Zwanenburg, J.F. Peters, J.H.H. Bongaerts, S.A. de Vries, D. L. Abernathy, and J. F. van der Veen, Phys. Rev. Lett. 82 pp. 1696 (1999).