

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

<http://193.49.43.2:8080/smis/servlet/UserUtils?start>

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.



	Experiment title: Surface Dynamics of Colloidal Suspensions	Experiment number: SC 2088
Beamline:	Date of experiment: from: 22.11.2006 to:29.11.2006	Date of report: 14.2.2007
Shifts:	Local contact(s): Yuriy Chuskin	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Christian Gutt, Tina Autenrieth, Lorenz –Mathias Stadler, Olaf Leupold, Agnes Duri, Gerhard Grübel HASYLAB at DESY, Notkestraße 85, D 22603 Hamburg, Germany		

Report:

We performed an XPCS experiment in grazing incidence geometry to study the surface dynamics of colloidal particles in suspension. The investigated sample consisted of SiO₂ particles of 50 nm radius immersed in a polymer melt - polypropylene glycol (PPG) of molecular weight 4000. In order to prevent aggregation, the silica particles were coated by alcy chains resulting in a hard sphere system. A weight fraction of 30 % of silica particles was solved in PPG. The liquid sample was filled in a trough of 10 cm diameter. Cooling was achieved via a flow of liquid nitrogen through a heat exchanger underneath the sample cell.

The incident angle of the x-ray beam was chosen to be below the critical angle of total external reflection. The corresponding 1/e penetration depth of the x-ray beam is around 10 nm leading to a surface sensitive XPCS experiment. Figure 1 shows correlation functions measured at small values of the momentum transfer in the scattering plane at temperatures of 290, 265 and 250 K, respectively. Under those scattering conditions the scattering signal is

due to overdamped capillary waves from the fluctuating surface. The measured relaxation rates Γ allow us to determine the viscosity of the colloidal suspension via $\eta = \gamma q / (2 \Gamma)$ where γ denotes the surface tension and q the wave vector transfer parallel to the surface. Taking the temperature dependent surface tension from ref. 1 we find that within the error bars, the viscosity of the pure polymer melt is identical to that of the colloidal suspension.

In a second step we installed a CCD camera for detecting the weak scattering signal from the colloidal particles out of the scattering plane. Figure 2 displays a summed ccd image taken at a temperature of 250K. The image has been obtained by summing a time series of pictures extending over 20 minutes where each single image was exposed 1 s. The speckle structure of the image is apparent. From the time series of the CCD images we determined via software analysis the intensity autocorrelation functions of the surface dynamics. Figure 3(a) shows a surface correlation function taken at $q = 6 \cdot 10^{-2} \text{ nm}^{-1}$. The observed decay time of $\tau = 17$ seconds is in good agreement with the estimated value of 10 sec., based on the viscosity of the polymer and the radius of the particles. Figure 3(b) displays the logarithm of the intermediate scattering function i.e. $\log(f(q,t))$ versus time. In case of simple Brownian motion the scattering function is exponential which results in a linear slope. Here, however, we observe a so-called compressed exponential intermediate scattering function meaning that the scattering function decorrelates faster than exponential, i.e. $f(q,t) = \exp(-(\Gamma t)^\alpha)$ with $\alpha > 1$. This indicates that the surface motion of the particles is not simple Brownian motion. The exponent α found for the compressed exponential behavior is varying between 1.3 and 1.9. For comparison an inset in fig.3 also displays the intermediate scattering function of the bulk solution as measured with dynamic light scattering (PCS) at $T = 290 \text{ K}$. Obviously the correlation function in the bulk is of a simple exponential form.

In summary, we observed for the first time the surface dynamics of a colloidal suspension. The scattering functions found are different from the correlation functions in the bulk regarding their compressed exponential behavior. In order to establish this result additional beamtime will be necessary.

[1] M. Sprung, T. Seydel, C. Gutt, E. DiMasi, A. Madsen, R. Weber, M. Tolan, Phys.Rev. E 70, 51809 (2004)

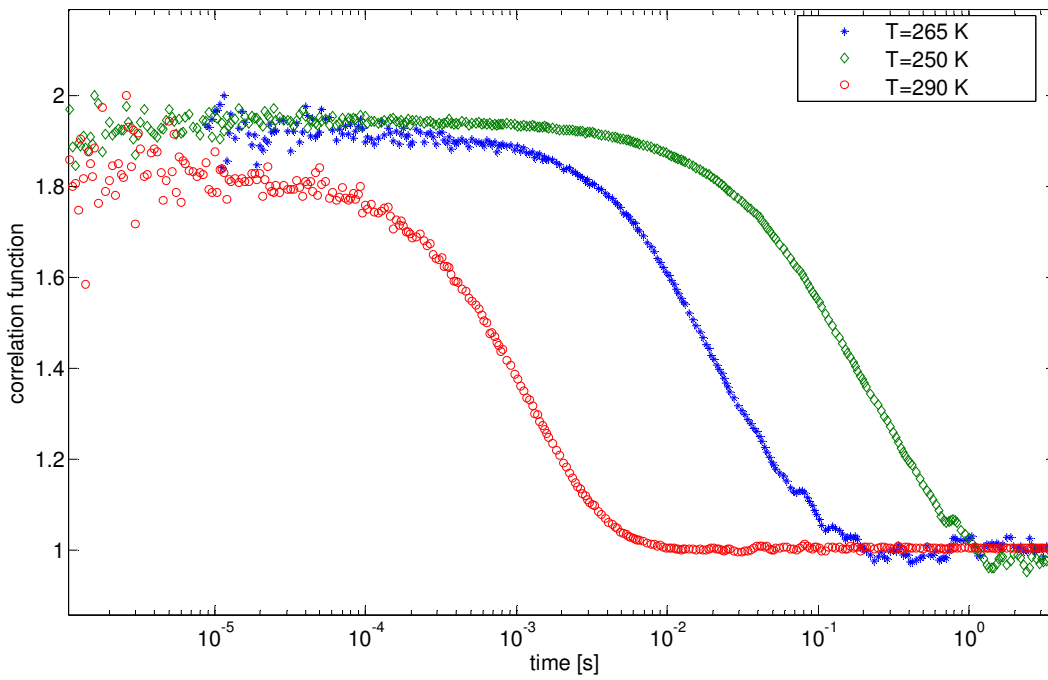


Fig. 1: Correlation functions from capillary waves on the surface of the colloidal suspension at temperatures of $T=290$ K, 265 K and 250K.

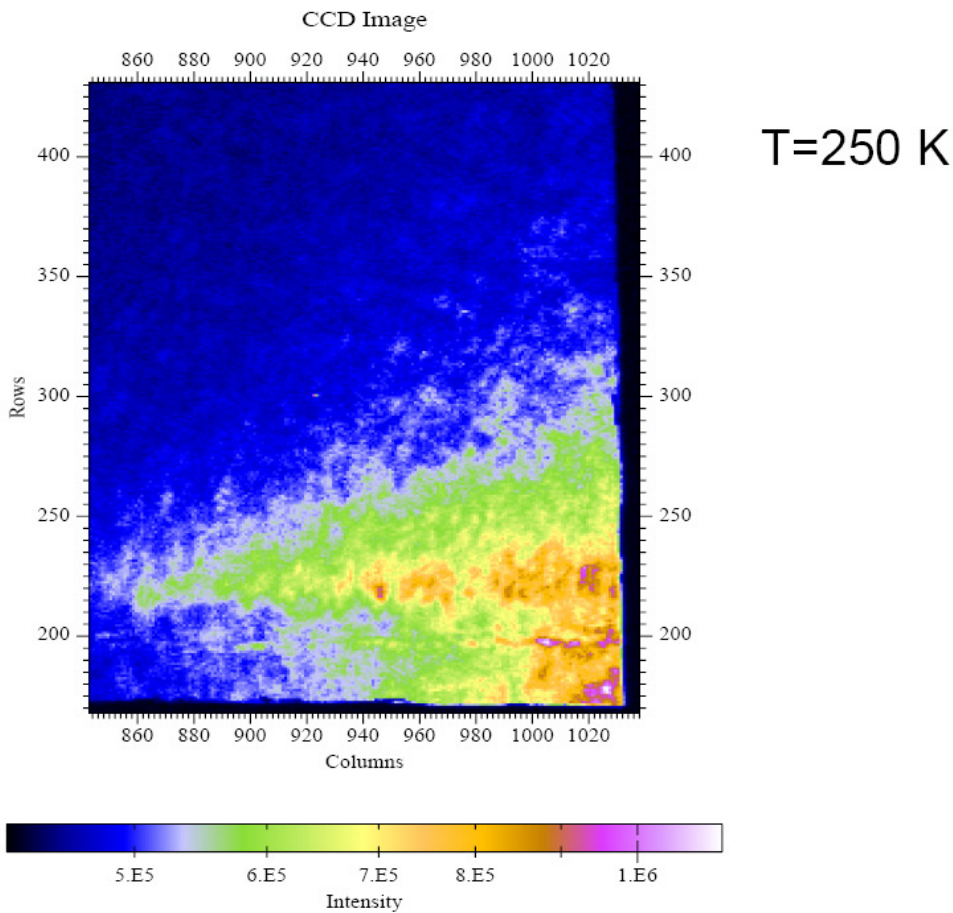


Fig. 2: CCD image from the form factor of the colloidal particles at the surface. The exposure time was 20 minutes. The speckle structure indicates a slow motion of the particles.

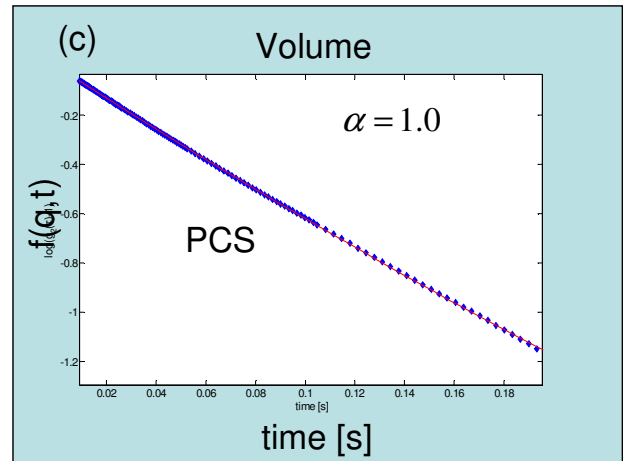
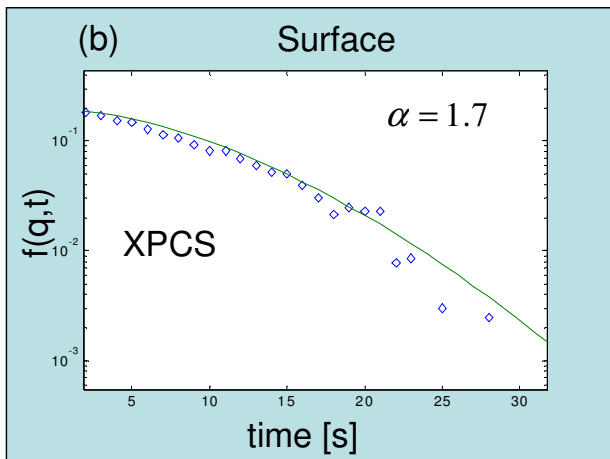
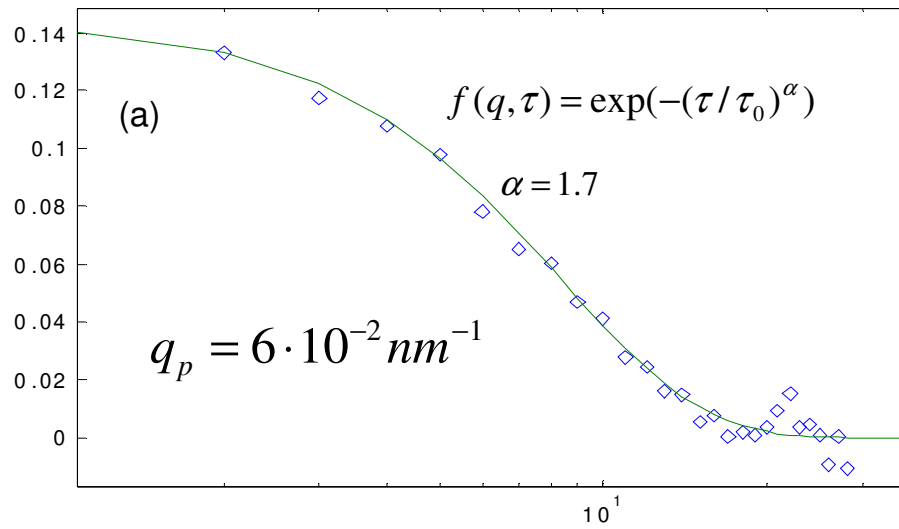


Fig. 3: (a) Intermediate scattering function of colloidal particles at the surface of a colloidal suspension. (b) The logarithm of the intermediate scattering function $f(q, t)$ at the surface versus time. The compressed form of the correlation function is obvious. (c) The same plot as in (b) for the particles in the bulk as measured with visible light. Here the correlation function is a simple exponential.