EUROPEAN SYNCHROTRON RADIATION FACILITY

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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.

ESRF	**Experiment title:** Higher order correlations in disordered materials	**Experiment number**: HD 168
Beamline: ID10 A	**Date of experiment**: from: 7.2.2008 to: 13.2.2008	**Date of report**: 24.2.2009
Shifts: 18	**Local contact(s)**: F. Zontone	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

P. Wochner*[1], C. Gutt*[2], T. Autenrieth*[2], A. Duri*[2], G. Grübel*[2] and H. Dosch[1,2]

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Report:

We report on a small angle scattering experiment of colloidal liquids and glasses using coherent x-rays. The aim of the experiment was to study higher order correlation functions in disordered materials giving access to short and medium range order. Hard sphere and soft sphere colloidal glasses served as prototype disordered material. The particle sizes ranged from 110 to 170 nm.
The experiment was performed at the TROIKA beam line ID10 A of the European Synchrotron Radiation Facility (ESRF). The X-ray energy was 8.03 keV selected by a single bounce Si(111) monochromator. By proper collimation a 10 micron partially coherent x-ray beam was produced. Parasitic scattering from the beam defining slits was removed to a large extent by careful positioning of a guard slit. The scattered radiation was detected at a distance of 2265 mm from the sample by a charge coupled device (Princeton Pi-LCX 1300 CCD) camera with 20 microns pixel size. For the measurement a region of interest of 800 x 800 pixels was selected. A beamstop of size 1mm blocked the direct beam. Series of 1000 CCD images were taken with exposure time varying between 0.15-0.4 seconds and stored on hard disk. Additional 50 dark images were recorded. Including CCD readout time the time interval between consecutive images was 1 second.
The dark images were averaged and subtracted from the data. The colloidal form factor F(Q) has been obtained from a diluted colloidal sample. The form factor allowed to deduce the static structure factor S(Q) of the concentrated sample.

A multi-speckle temporal intensity autocorrelation analysis yielded the dynamic correlation function of the sample. The dynamical correlation function, which relates to the diffusive motion of the colloidal particles, shows no decay within the first 100 seconds. Therefore, we averaged series of 50 to 100 CCD images. Figure 1 displays a CCD image measured from a colloidal glass. The CCD image shows a speckle pattern typical of disordered glassy structures with no preferred orientation detectable. Correlations in the sample can now be extracted from such a speckle pattern by applying a novel X-ray cross correlation analysis (XCCA) according to Eq. (1) to the data. For this purpose rings of intensity with constant Q and 1-2 pixel widths have been extracted from the data and an unbiased angular correlation function according to Eq. (1) has been calculated.

$$C_Q(\Delta) = \frac{< I(Q,\varphi) I(Q,\varphi+\Delta) >_\varphi - < I(Q,\varphi) >_\varphi^2}{< I(Q,\varphi) >_\varphi^2} \quad (1)$$

All colloidal samples investigated showed pronounced features in $C_Q(\Delta)$. As a prominent example the angular correlation function for two PMMA hard sphere systems differing in particle radius are shown in Fig 2 and 3. Interestingly, the dominant feature of the angular correlation functions in all hard sphere systems is a strong five-fold modulation. Moreover, time dependent transitions from five-fold to six-fold modulations have been observed in the hard sphere systems. It is interesting to note that the soft sphere systems investigated showed no five-fold modulation but were dominated by six- and four-fold modulations. Our interpretation of the observed modulations is based on the assumption of locally favored structures (LSF) embedded in a disordered matrix. The angular correlation function of Eq. (1) extracts the local symmetries of the LSF. This interpretation and the appearance of five-fold symmetries is supported by simulations of icosahedral structures embedded in disordered regions.

In conclusion, our experiment shows that a novel XCCA analysis gives access to local symmetries in disordered matter. A manuscript is submitted [1].

[1] P. Wochner, C. Gutt, T. Autenrieth, T. Demmer, V. Bugaev, A. Diaz-Ortiz, A. Duri, F. Zontone, G. Grübel, H. Dosch "X-ray cross correlation analysis uncovers hidden local symmetries in disordered matter " manuscript submitted.

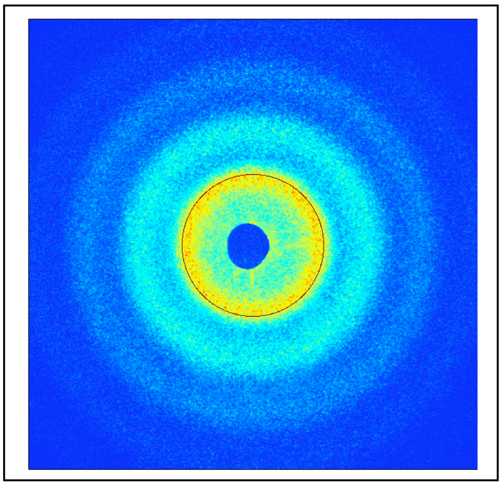

Figure 1: Speckle pattern of a colloidal glass. The red line indicates a constant Q ring chosen for the XCCA analysis. Modulations in the angular correlation functions have been observed at many different Q positions [1].

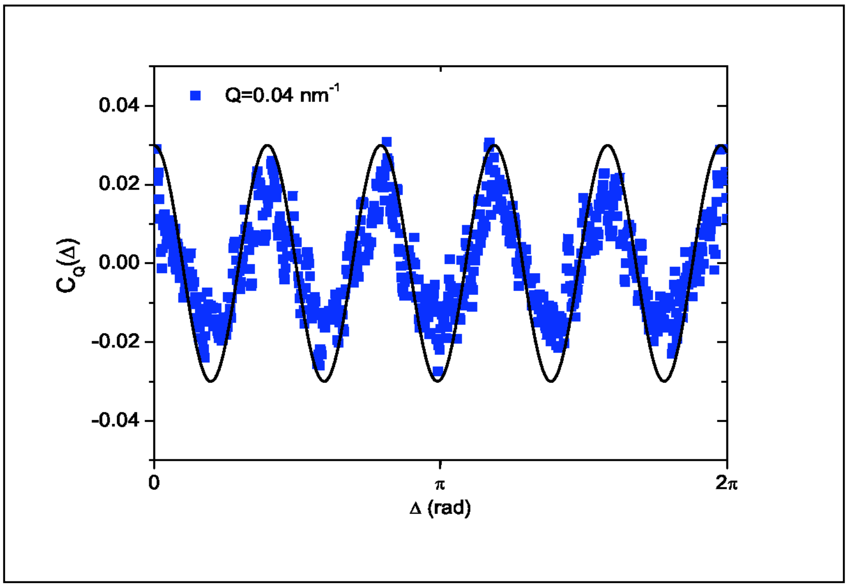

Figure 2: $C_Q(\Delta)$ function for a colloidal glass with particle radius of 117 nm. The solid line is a guide to the eye only.

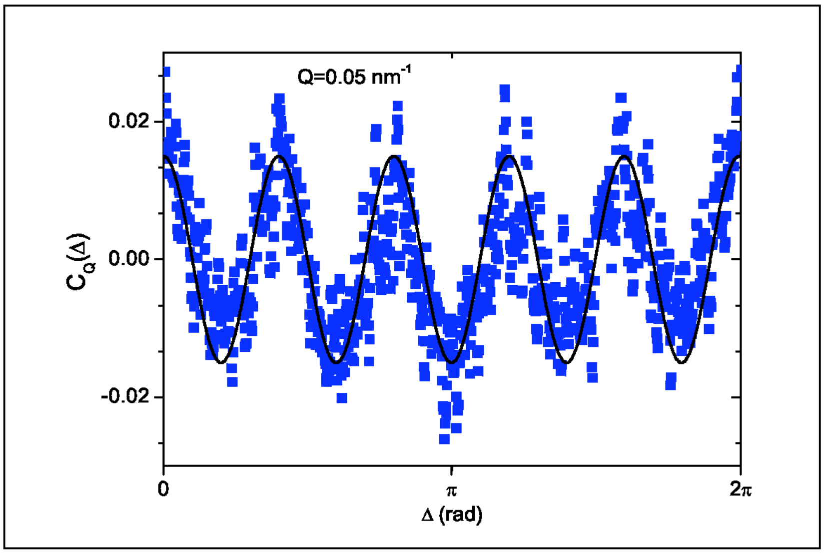

Figure 3: $C_Q(\Delta)$ function for a colloidal glass with particle radius of 132 nm. The solid line is a guide to the eye only.