Names and affiliations of applicants (* indicates experimentalists):

*Wolfgang Haeussler, *Adam Patkowski, Werner Steffen, Gerd Meier, Max-Planck-Institut fuer Polymerforschung, Postfach 3148, D-55021 Mainz, Germany

*Thomas Thurn-Albrecht, Fakultät für Physik, Universität Freiburg, Hermann-Herder-Str. 3, 79104 Freiburg

Robert Pecora, Chemistry Department, Stanford University, Stanford CA94305-5080, USA,

Redouane Borsali, LCPO-CNRS-ENSCPB-Bordeaux University

*Gerhard Grübel, ESRF, Grenoble

Report:

We planned to continue studies of the protein Ferritin in solution at different protein and salt concentrations. The metallic core of Ferritin (7.5 nm in diameter) provides high scattered intensity, sufficient for measurements by means of XPCS at low q values [1]. In dynamic light scattering experiments (DLS) evidence of slow dynamics depending on ionic strength of Ferritin solutions was found [2]. The q-dependence of the short- and long time dynamics depends on both the interactions between the macromolecules and the counter ions being a topic of present investigations [3]. XPCS measurements at the peak position were supposed to give new insights into dynamical processes in the solution of ordered molecules.

At higher q, a **CCD** camera is necessary, because the scattered intensity decreases with increasing q. Since the relaxation rates of Ferritin, even if solved in glycerol, are faster than the frame rate of the available camera (1 frame/sec.), we have performed the experiments using a new "**contrast method**" in combination with the CCD camera. The shutter's opening time determines different exposure times in the ms range. Thus, the CCD images contain the result of integrated scattered intensity which changes in time due to moving speckle pattern. The contrast of the image depends on the exposure time. From the contrast of the image which decreases with increasing exposure time one can calculate the intensity correlation function.

Unfortunately, the Ferritin solutions with high content of glycerol were found to show unexpected behaviour when cooled below -20° C. On reaching this temperature, the scattered intensity lost their circular symmetry. While the circular symmetry is due to liquidlike disorder, the loss of symmetry indicates partial crystallization. We think that this is due to imperfect intermixture of water and glycerol. Samples without water were not available because dialysis is very time consuming. Thus, we reduced the water content by evaporation. Consequently, a small fraction of water was still inside the samples. Due to the partial crystallization we were not able to study dynamics in the Ferritin samples.

Fortunately, we could get Silica colloids in glycerol/water as sample, in order to test the new method using a beam shutter as described above. Because the dynamics of the Silica colloids is on the second-scale, we could
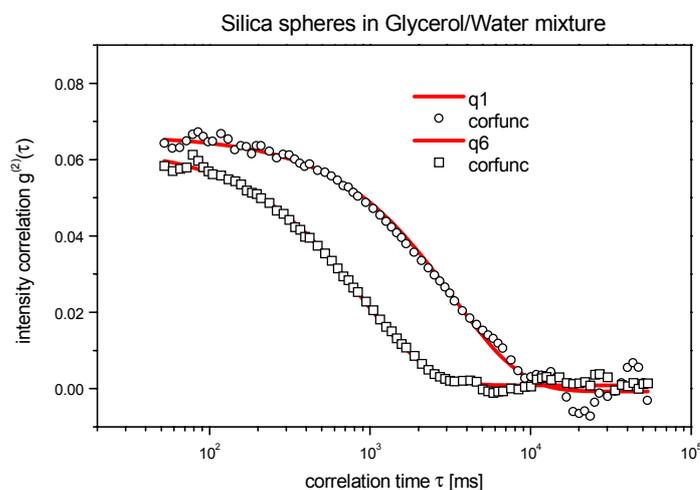

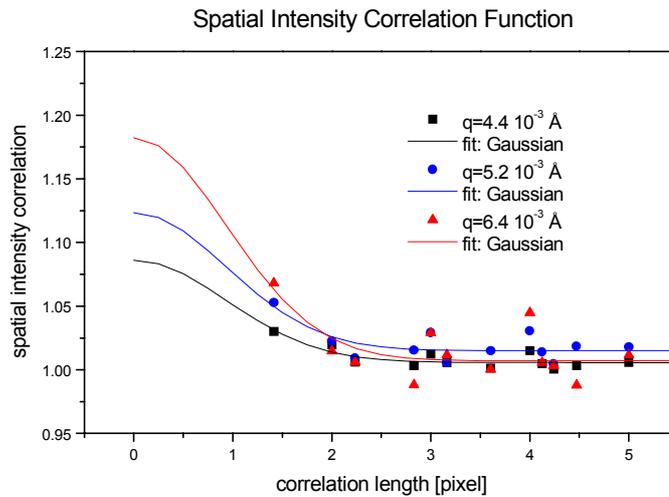Fig.1. XPCS results of Silica colloids in glycerol/water mixture.

perform measurements by "conventional" XPCS at first (Fig.1).

Subsequently, we took a series of CCD images with different exposure times (1 sec., 2 sec., 4 sec. ... 256 sec.). The diameter of the pinhole used was 5 µm. The distance between sample and detector amounted to 2.1 m. The pixel size of the CCD camera was 25 µm.
The data analysis was done in the following steps:

1) The images were corrected with respect to the background and, subsequently, with respect to the form factor of the Silica colloids.

2) The spatial correlation function of the CCD images was determined. Fig.2 shows typical correlation functions.

3) These data were fitted to a Gaussian being the theoretical spatial correlation function[4]. The amplitude of the Gaussian is the "contrast" of the speckle pattern, being the same as the fluctuations (or "variance") of

Spatial Intensity Correlation Function

the measured intensity.

Fig.2. Spatial correlation function of CCD images at different q-values.

4) Steps 1) and 2) were performed for images of different exposure times. The plot of the contrast over the exposure times (Fig.3) shows a decay with increasing exposure time because of the "smearing" of the moving speckle.

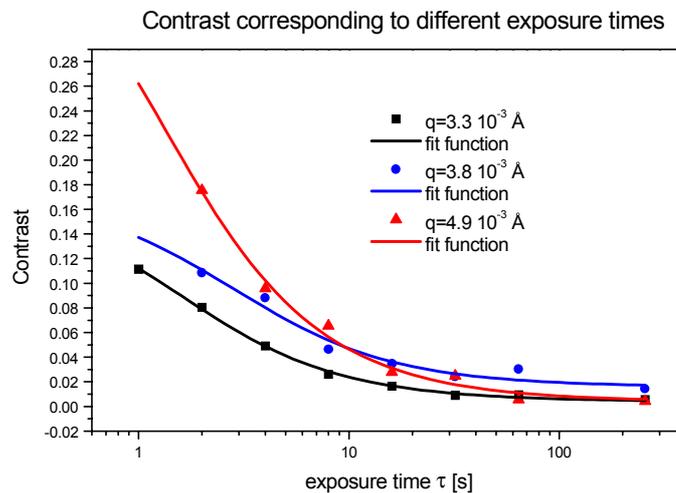Contrast corresponding to different exposure times

Fig.3. XPCS results of Silica colloids in glycerol/water mixture.

Summary: The first test of the "contrast method" proved the applicability of this new method. The dynamics detected in a solution of Silica colloids in glycerol was in concordance to the dynamics measured by means of "conventional" XPCS. Subsequent measurements are planned to apply this method studying various samples with relaxation rates in the ms-range.

1. A. Patkowski et al., ESRF Experimental Report SC552, 1999.

2. W. Häußler, A. Wilk, J. Gapinski and A. Patkowski. "*Interparticle Correlations due to Electrostatic Interactions. A Small Angle X-ray and Dynamic Light Scattering Study. I. Apoferritin.*" (submitted).

3. W. Härtl et al., J. Chem. Phys. **110**, 7070 (1998)

4. D.L. Abernathy, G. Gruebel, S. Brauer, I. McNulty, G.B. Stephenson, S.G.J. Mochrie, A.R. Sandy, N. Mulders, M. Sutton. "*Small Angle X-ray Scattering Using Coherent Undulatior Radiation at the ESRF*". Journal of Synchrotron Radiation, 5, 37-47 (1998).