Coherent X-ray scattering experiments

at the coherence beamline P10 at PETRA III

L. Müller, F. Westermeier, M. Sprung, Coherent X-Ray Scattering, DESY, Hamburg, Germany (November 2013)

Application Note

Introduction

Coherent X-ray scattering techniques have become a valuable tool for the investigation of structure and dynamics of a multitude of condensed matter systems, e.g. colloidal suspensions, metal alloys or magnetic domain systems. Coherent X-rays are available at third generation synchrotron sources and recently also at free-electron laser sources. To successfully exploit the coherence properties, the detectors used for recording the coherent scattering patterns have to be able to resolve the subtle structure, i.e. the so-called speckle structure, of those patterns.

A back illuminated deep depletion CCD detector ("iKon-L SO" DO936N-MW-BR-DD from Andor Technology) for direct X-ray detection is used as one of the standard detectors at the Coherence Beamline P10 at PETRA III, DESY. It offers 2k × 2k pixels with a pixel size of only 13.5 µm which is the smallest value of all standard detectors available at the beamline and can thus resolve the finest structures in speckle patterns. One important field of research at the beamline is the measurement of static and dynamic properties of colloidal liquids, gels and glasses using small-angle X-ray scattering (SAXS). Data shown in this report are recorded from such sample systems.

Experimental Setup

The structure and the dynamic behaviour of different colloidal systems were investigated using the iKon-L SO X-ray CCD detector. All X-ray experiments have been performed at the Coherence Beamline P10 of the PETRA III synchrotron.

The X-rays were monochromatised by a Si (111) double crystal monochromator to a photon energy of 8.0 keV corresponding to a wavelength of λ=0.155 nm. The samples were placed in an in-vacuum setup at a distance of approximately 80 m from the source. To obtain a transversally coherent X-ray beam, a set of slits upstream of the sample reduced the beamsize to 20 x 20 µm². The beamsize on the sample also determines the speckle size $l=\lambda \cdot D/r \approx 35$ µm, where D=5 m is the sample-detector distance and r=20 µm is the beamsize on the sample.

The iKon-L SO detector was placed on a moveable detector translation stage approximately 5 m downstream of the sample position after an evacuated flight pass and is not directly attached to the beamline's vacuum system (Fig. 1).

The camera is equipped with a beryllium window which is available as an option and a separate turbo pump is used for evacuation. All X-ray scattering data were obtained in transmission SAXS geometry, a beamstop directly in front of the detector was blocking the intense direct beam to prevent beam damage of the CCD chip.

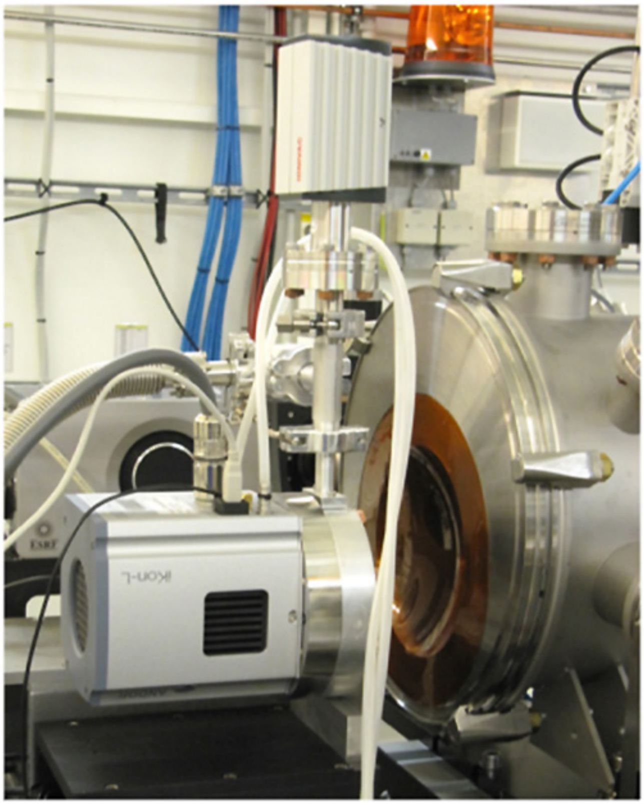

Figure 1: iKon-L SO camera in front of the beamline's exit window, i.e. end of the vacuum flight pass approximately 5 m behind the sample position.

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During the X-ray scattering experiments, the CCD chip was cooled down to a temperature of 60 °C, the temperature of the cooling water was set to 15 °C. The camera was set to sensitive mode at a readout rate of 3 MHz and a gain of 1. The iKon-L SO camera was integrated into the control software of the beamline by a so-called TANGO device server enabling thus the control of the camera by Perl scripts. This Tango device server is based on the LIMA software package developed by the European Synchrotron Radiation Facility (ESRF) in Grenoble and allows setting almost all camera options as, e.g., exposure time, readout rate or chip temperature. We find an AD offset level of 1151 ADUs in good agreement with the value specified (1144) in the manufacturer's test report. A single 8 keV photon results in 613 ADUs, a relative large number which is due to the direct illumination design of the camera. The effective dynamic range is therefore limited to about 2 orders of magnitude, i.e. 100 photons saturate a pixel of the camera.

Test Experiment

Two different colloidal sample systems have been investigated: A static sample showing no dynamics consisting out of a gel of SiO_2 colloidal particles with a mean diameter of 100 nm (A) and a sample system undergoing Brownian motion consisting of spherical SiO_2 particles stabilized by a layer of trimethoxysilypropyl methacrylate and suspended in polypropylene glycol (B). Both systems were filled into quartz capillaries with a diameter of 1 mm and a wall thickness of 10 µm. We investigated both the structure and the dynamics by coherent X-ray scattering experiments.

When coherent photons are scattered by a disordered sample, the scattering pattern shows a typical grainy appearance, called a speckle pattern. This speckle pattern contains the information about the detailed spatial arrangement of the individual scatterers. The speckle visibility $\beta(Q)=g_2(Q,0)-1$, depends on both, the coherence properties of the beam impinging on the sample and the properties, most prominent the pixel size, of the detector. Here, g_2 is the intensity–intensity auto-correlation function $g_2(Q,t)=\langle I(Q,0)\cdot I(Q,t)\rangle_t/\langle I(Q)\rangle_t^2$ where the brackets $\langle\ldots\rangle_t$ denote a time average over the whole measurement time, i.e. over all scattering patterns recorded in one series.

Fig. 2 shows an averaged speckle pattern calculated from 100 individual exposures of the static sample A. The speckles can clearly be resolved due to the small pixel size of the detector. In this case the contrast of the static speckle pattern is approximately $\beta(Q) = 0.68$ for all Q values between 0.008 $Å^{-1}$ and 0.014 $Å^{-1}$ – an excellent value possible due to the small pixel size of $(13.5\ µm)^2$ of the iKon-L SO camera.

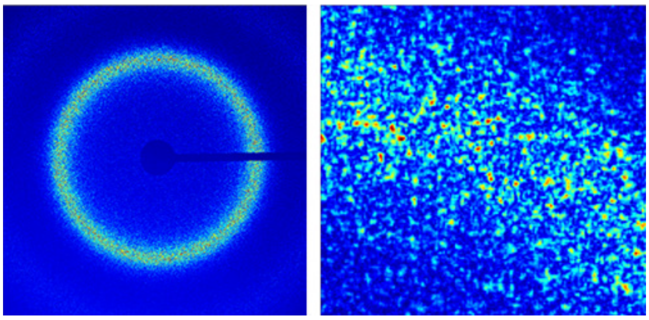

Figure 2: (left) Average image of a series of 100 frames acquired using sample system A. The direct beam is blocked by a beam stop installed on a wire coming from the right side. (right) Magnified view on a segment of the intense scattering ring. The speckle structure is well resolved due to the small pixel size.

For an analysis of the static behaviour of the sample, 500 individual frames were summed up. The azimuthal average (Fig. 3) of the isotropic scattering pattern was calculated, resulting in the wave vector dependent intensity I(Q), giving information about the mean shape and size of particles as well as on the next nearest neighbour distance.

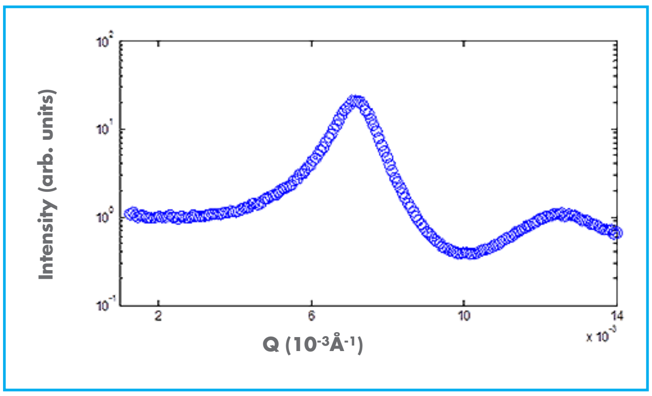

Figure 3: Scattered intensity as a function of wave vector Q calculated from the scattering data.

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The investigation of the dynamics of the samples was performed by X-ray Photon Correlation Spectroscopy experiments. This technique quantifies the temporal intensity fluctuations I(Q,t) of the scattered intensity caused by movements of the colloidal particles by auto-correlation, producing thus the intensity auto-correlation function g_2 (Q,t). The usage of a 2D X-ray detector offers the possibility to replace the time average by a pixel average of pixels in a certain momentum transfer Q range that are treated as equivalent. Calculating thus the two-time autocorrelation function $g_2(Q,t_1,t_2)$ allows evaluating the evolution of dynamic time scales, e.g. aging or avalanche dynamics, within the measurement time. A typical two-time autocorrelation of sample system B is displayed in figure 4. The dynamic time scale of this sample is on the order of the time between the acquisitiosn of two consecutive frames hence the dynamics time scale cannot be measured for this sample.

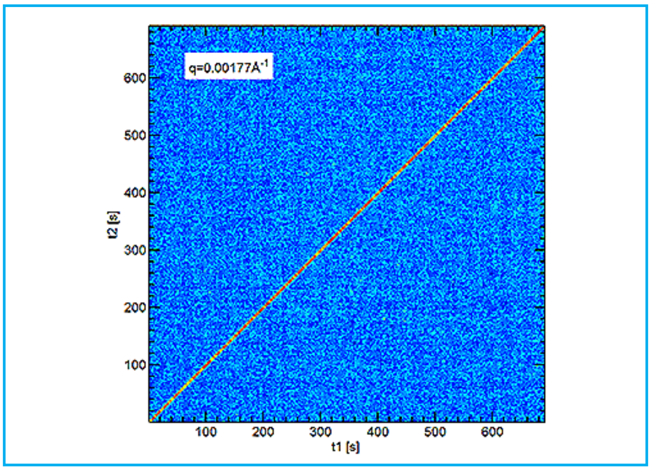

Figure 4: Two-time correlation function of a diffusing colloidal suspension (sample B).

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References

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