


**Experiment title:**

Heterodyne XPCS based on reference-beam holography

**Experiment number:**

MI930

**Beamline:**

ID22

**Date of experiment:**

from: 2.7.2009

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8.7.2009

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**Shifts:**

18

**Local contact(s):**

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**Report:**

The aim of the experiment was the development of a novel technique for dynamics probing of single colloid sensitivity based on reference-beam holography in combination with XPCS.

For that a pair of Y-shaped x-ray waveguides was placed in the focal spot of the KB mirror system of the ID22 beamline. Each waveguide had a cross section of  $60 \times 60 \text{ nm}^2$  and a length of  $4.1 \text{ mm}$ . The spacing at the beam entrance side was  $s_{in} = 100 \text{ nm}$  and was widened up to  $s_{out} = 3 \mu\text{m}$  towards the exit side. The two waveguides formed a Young's double slit pattern which was recorded in the farfield at a distance of  $z = 2.95 \text{ m}$  with a direct illuminated CCD (Princeton Instruments PI-LCX:1300). The samples were prepared in sealed, pulled capillaries with filament (Hilgenberg) and positioned at a distance  $z_1 \approx 1 \text{ mm}$  behind the waveguide. As samples colloidal suspensions of varied number concentration  $\rho_{col}$ , radii  $R$ , and water/glycerol (Fluka) ratio were prepared from Gold colloids (British Biocell International), covering a wide range in concentration, viscosity  $\eta$ , size, and corresponding diffusion time scales.

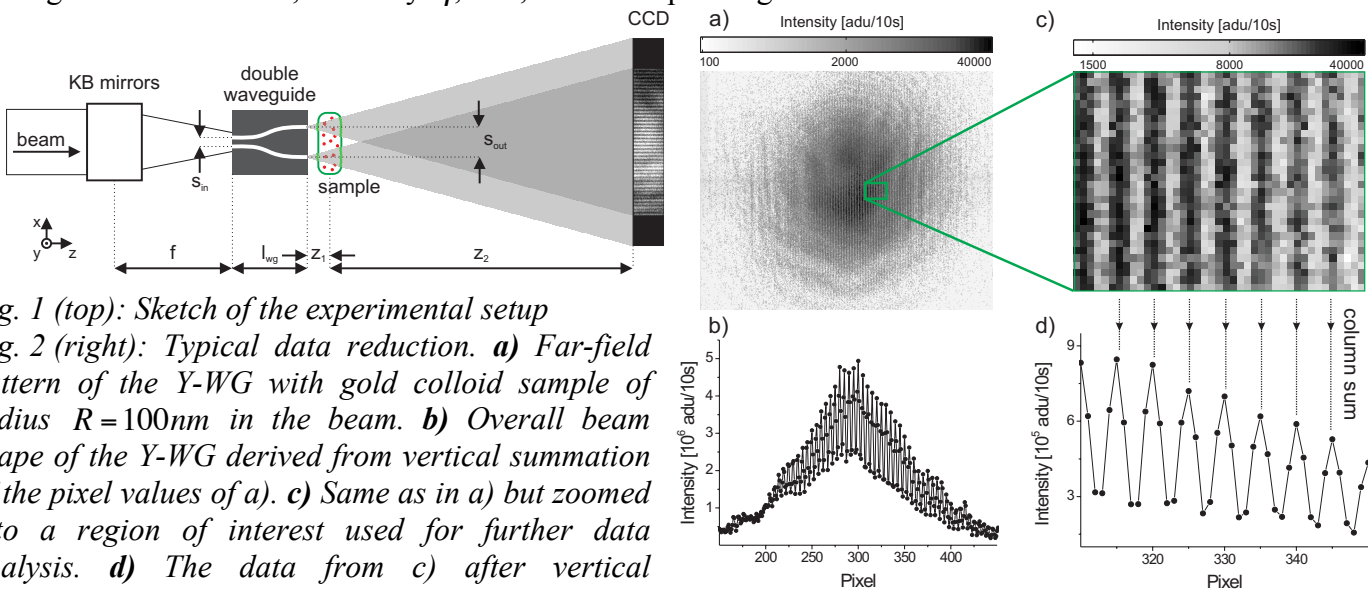


Fig. 1 (top): Sketch of the experimental setup

Fig. 2 (right): Typical data reduction. **a)** Far-field pattern of the Y-WG with gold colloid sample of radius  $R=100 \text{ nm}$  in the beam. **b)** Overall beam shape of the Y-WG derived from vertical summation of the pixel values of **a)**. **c)** Same as in **a)** but zoomed into a region of interest used for further data analysis. **d)** The data from **c)** after vertical summation.

A set of sample and empty beams was measured, with a data accumulation protocol as follows: for each sample as well as for two empty beams a series of 150 images with a single exposure time  $t = 10\text{s}$  was recorded. Fig. 2 shows a typical raw image of a far-field pattern with gold colloid sample in the beam and the data reduction scheme. The data was then treated as follows: each raw image was corrected by a dark image of same exposure time. Then a region of interest (ROI) was cut out for each single exposure. The intensity values inside the ROI were summed up in vertical direction to an intensity profile (column sum). From a spline interpolation of the intensity profile the maxima positions were derived. The overall contrast of an intensity profile was calculated by the averaged contrast  $C = (I_{\max} - I_{\min}) / (I_{\max} + I_{\min})$  for each pair of maximum and minimum of the column sum. The integration of all pixel intensities results the overall intensity of the ROI.

Typical results are shown in Fig. 3: Let us first address the empty beam curves without samples in the two beam waveguide interferometer. The empty beam setup can be used to characterize the optical system itself, and in particular the stability of the focusing scheme. Strong drifts are observed over the data accumulation run. Such a behavior is unfortunately not unusual for highly focused synchrotron beams and presents a tremendous challenge to hard x-ray nanoprobe experiments. Small drifts, both rotational and translational, of the incident beam relative to the waveguide entrance, or equivalently in the waveguide positioning, lead to significant intensity fluctuations, which were higher than the fluctuations of the integrated KB beam, typically by a factor of 100 (derived from autocorrelation of APD signals).

In addition to the intensity trace, a phase trajectory, and a contrast trajectory are measured interferometrically, as shown in the top row of Fig. 3. The phase trajectories  $\Delta\varphi(t)$ , plotted in pixel units with  $\pi = 5.078\text{ pixel}$ , show total variations of up to about  $3\pi$ . The corresponding path length differences in the two waveguide beams of up to  $1.5\lambda$  could result from the drifts of the focused beam relative to the waveguide. The dominating contribution of the phase shift must be due to drifts in the optical system, since the phase and contrast curves look quite similar for the runs with and without sample. However, for all five runs the time scales of the phase fluctuations are much slower than those of the intensity and contrast functions, which are broader bandpass signals. The rather smooth behavior of  $\Delta\varphi(t)$  should enable the detection of short time scale phase fluctuations due to path length differences induced by sample dynamics.

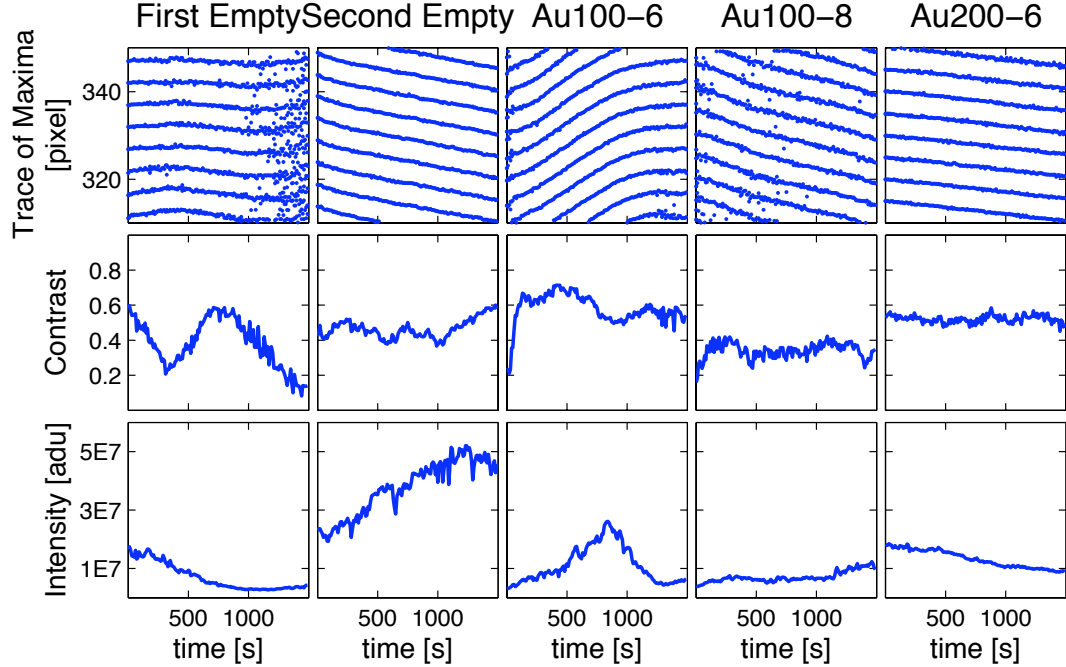


Fig. 3: Typical results for colloidal samples and empty beams: the trace indicates the position of the maxima on the CCD over time (top row). The contrast  $C$  (center row) and the intensity (bottom row), integrated over the ROI.

#### References:

- [1] S. Kalbfleisch, K. Giewekemeyer, T. Salditt, *A double x-ray waveguide interferometer as a phase probe at the nanometer scale*, submitted