

**Experiment title:**

Study of Slow Dynamic Processes in Polymeric and Colloidal Systems by Photon Correlation Spectroscopy Using Coherent Synchrotrons Radiation

Experiment**'number:**

SC106

Beamline: Date of Experiment:**Date of Report:**

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Report:

X-ray Photon Correlation Spectroscopy (XPCS) probes the dynamic properties of matter by analyzing the temporal correlations among photons scattered by the material. Correlation spectroscopy requires the sample to be illuminated coherently implying the need for an intense highly collimated X-ray beam with sufficient transverse and longitudinal coherence length [2,3].

Using the intense coherent X-ray beam from the TROIKA undulator beamline at the ESRF we were able to probe the diffusive dynamics of a palladium colloid in glycerol over 6 decades in time (10^{-4} s to 10^2 s). We have studied the temperature dependence and the q-dependence of the diffusion coefficient beyond the range reachable by conventional light scattering in an optically opaque sample.

The sample used is a solution of colloidal palladium in glycerol, produced with anew electrochemical method [4, 5]. Transmission electron microscopy gave a radius of 20 to 30\AA for the metallic core of the colloidal particles.

Fig. 1 shows the measured static structure factor as a function of scattering vector q. An extension of the data taken at the Troika beamline to higher scattering vectors was made by a measurement with a conventional SAXS setup using a commercial Kratky camera and non-coherent x-rays. Around $q \approx 0.11\text{\AA}^{-1}$ the scattering from the individual palladium particles dominates $I(q)$, as it can be seen in the inset, which shows $I(q)q^4$ and a model function describing the scattered intensity as caused by a distribution of spherical particles with different radii [6]. The further increase of $I(q)$ at small q, which would not show up for isolated spheres, indicates the existence of aggregates, whose internal structure is reflected in this q-range.

In this regime $I(q)$ can approximately be described by power law scattering, $I(q) \propto q^{-\alpha}$, a behaviour which is often found in aggregates and is usually interpreted in terms of a fractal structure of the aggregates [7]. The data are best fitted by an exponent $\alpha = 2.81$. The exact size of the aggregates cannot be determined from the measurements of $I(q)$ alone, since the Guinier limit is not reached in the accessible q -range of the experiment.

Correlations of the scattered intensity as they are measured by XPCS are quantified via the normalized time correlation function $g(t)$, $g(t) = \langle n(t')n(t'+t) \rangle / \langle n \rangle^2$, where $n(t)$ is the number of scattered photons at time t . Correlation functions were measured in q -range from $1.5 \cdot 10^{-3} \text{ \AA}^{-1}$ to $6.5 \cdot 10^{-3} \text{ \AA}^{-1}$ and in the temperature range from 279K to 293K . Fig. 2 shows a correlation function obtained at $1.58 \cdot 10^{-3} \text{ \AA}^{-1}$ and 279K . For a diffusion process the relaxation rates Γ determined from the correlation functions is proportional to q^2 , $\Gamma \propto Dq^2$ with D being the diffusion constant [1]. In the inset of Fig. 2 it is shown that we found the expected behaviour. The temperature dependence of the relaxation rate Γ has been measured for a fixed $q = 1.58 \cdot 10^{-3} \text{ \AA}^{-1}$. It reflects the change in the viscosity of glycerol with temperature, as it is expected for a diffusion process. The value for the hydrodynamic radius calculated from the data taken in the experiment amounts to $R_h = 1220 \text{ \AA} \pm 100 \text{ \AA}$. This value is about a factor of fifteen higher than the radius of the individual colloids. This finding agrees well with the conclusion drawn from the analysis of the measured static structure factor. The colloidal particles aggregate whose diffusion is observed in the PCS experiment,

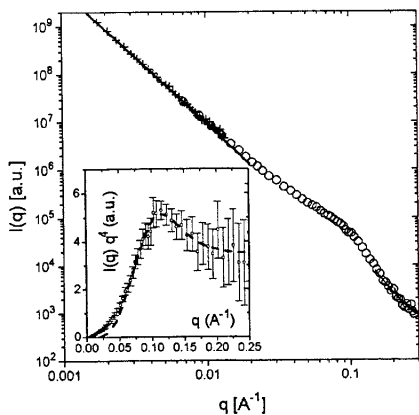


Fig. 1

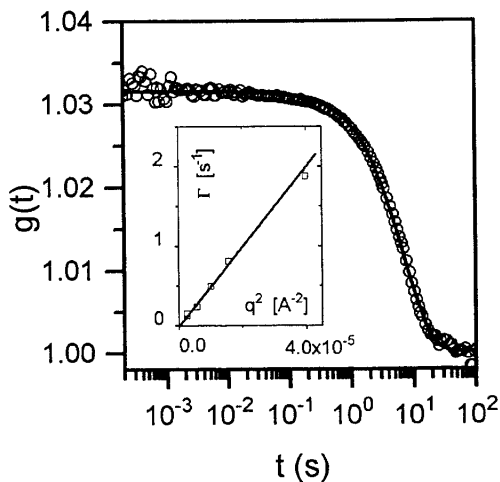


Fig. 2

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