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ESRF

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 via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can 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:** **Investigation of the *in situ* Self-Assembly of PbS Nanocrystals by Time-Resolved SAXS/WAXS**	**Experiment number**: **SC-4605**
Beamline: ID02	**Date of experiment**: from: 15.12.2017 to: 18.12.2017	**Date of report**: 01.08.2017
Shifts: 9	**Local contact(s)**: Alessandro Mariani	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report: Monodisperse lead sulfide (PbS) colloidal nanocrystals are known to form highly ordered crystalline superstructures upon solvent evaporation or destabilisation. Obtained nanocrystal solids yield novel collective properties, which makes them interesting for many potential technological applications, such as solar cells, photodetectors, transistors, light-emitting diodes, etc. *In situ* small-angle X-ray scattering (SAXS) and wide-angle X-ray scattering (WAXS) represent powerful techniques to study the real-time assembly of nanocrystal superlattices with subsecond temporal and micrometer spatial resolution.

In the allocated beamtime, we aimed to invesigate the detailed formation of crystalline superlattices of PbS nanocrystals from the colloidal suspension through controlled solvent evaporation using *in situ* SAXS/WAXS. The real-time evaporative assembly of PbS nanocrystals was monitored in a custom-built chamber equipped with two Kapton windows for the incident and scattered X-ray beam. This chamber included the inner sample cell, where the nanoparticle solution was injected through a thin capillary, which consisted of a copper holder with two silicon nitride membranes at 0.5 mm separation. The overall nanoparticle solution volume was approximately 24 µl. The size of X-ray-transparent silicon nitride windows was 5 mm x 5 mm with a membrane thickness of 0.5 or 1 µm. Before the sample was filled in, the chamber was evacuated and flushed with helium twice and then the corresponding amount of pure solvent was placed in the chamber to obtain the saturated vapor environment. In this way, the nanoparticle solution started to evaporate only after the helium flow was turned on. The typical helium flow rate was kept as low as 1–2 cm^3/min to ensure slow solvent evaporation. In this way, the complete evaporation of 24 µl of the

nanoparticle solution took up to 3 hours depending on the solvent used. During all measurements the cell temperature was kept constant at 25 °C.

The energy of the incident X-ray beam was set at 12.46 keV and the beam size was 100 µm (vertical) x 200 µm (horizontal). The SAXS data were recorded by a Rayonix MX-170HS charge-coupled device (CCD) area detector and the sample-to-detector distance was 1.735 m. Images were collected with exposure times ranging from 0.02 to 0.05 seconds. Background scattering of the solvent and the experimental setup were subtracted from data. It has to be noted that no beam damage of the sample was observed for the chosen exposure times.

Nearly monodisperse PbS nanocrystals were produced in our laboratory in a colloidal solution by a hot-injection method. We have studied the self-assembly of PbS nanocrystals with two sizes (3.9 and 7.8 nm in diameter), dispersed in different solvens (hexane, heptane, octane, toluene) at different concentrations (1 – 25 mg/ml). We observed the real-time formation of highly ordered superlattices from the colloidal solution upon controlled evaporation in a solvent vapor saturated atmosphere. Figure 1 shows the SAXS patterns of a colloidal suspension before the evaporation and the self-assembled crystalline phase of 3.9 nm particles. Further analysis is currently ongoing. The results are planned to be published in the near future.

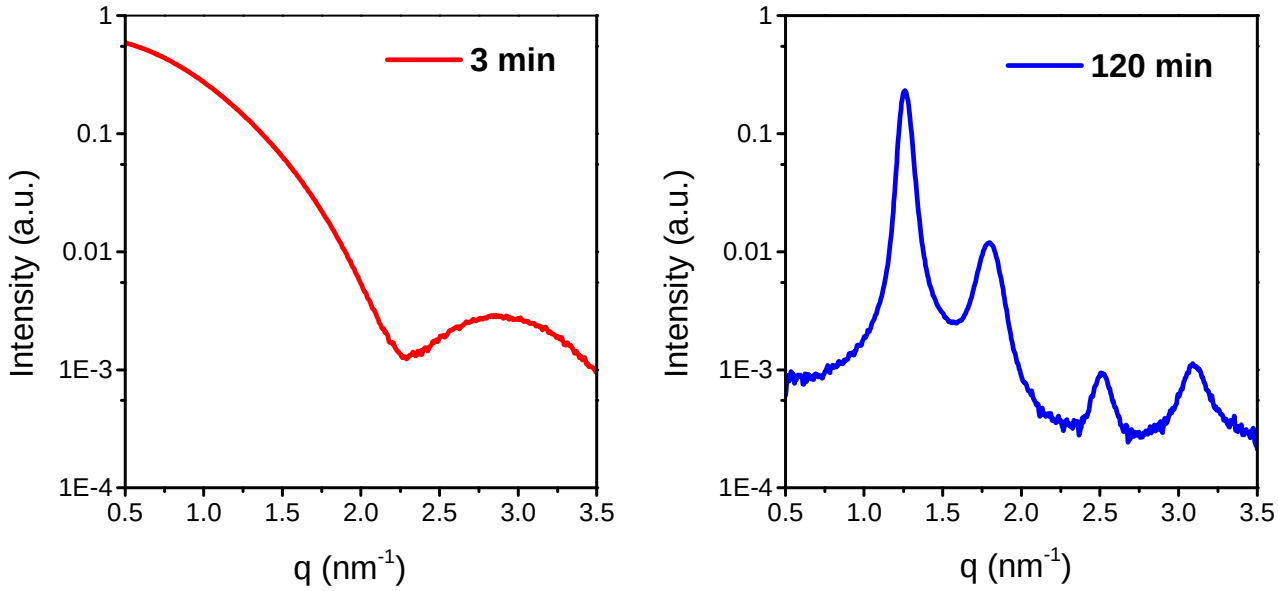

Figure 1. SAXS patterns of a colloidal suspension of PbS nanocrystals in the beginning of the measurement (left) and a highly ordered crystalline superlattice self-assembled after solvent evaporation (right).