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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:** Combined USAXS/SAXS/WAXS measurements of self-assembling anisotropic iron oxide nanoparticles in a levitating drop	**Experiment number**: MA-3786
Beamline: ID02	**Date of experiment**: from: 24.11.2017 to: 27.11.2017	**Date of report**: 18.04.2019
Shifts: 9	**Local contact(s)**: Lewis Sharpnack	*Received at ESRF:*

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Report:
The aim of this experiment was to probe the growth of mesocrystals for a better understanding of the mechanism of self-assembling iron oxide nanocubes in colloidal droplets. For that, we have conducted time-resolved SAXS and WAXS measurements in order to probe the self-assembly of oleate capped iron oxide nanocubes. To investigate colloidal droplets and solvent evaporation from nanocube dispersions in real time, we have installed and utilized our acoustic levitator setup. Each time-resolved experiment consisted of inserting ~ 3 µL of a nanocube dispersion with edge lengths of 6.8 nm (C068) and 9.1 nm (C091) into the levitator and collecting SAXS and WAXS data with an exposure time of 0.03 seconds resulting in a time-resolution of 2 seconds per frame, covering a q-range of 0.035 $nm^{-1} < q < 3.74\ nm^{-1}$ in the small angle and $11.42\ nm^{-1} < q < 50.44\ nm^{-1}$ in the wide angle regime at a wave length of $\lambda = 1$ Å. The drying process of the droplets has been simultaneously recorded with a microscope camera to correlate the onset of self-assembly to the volume fraction of the nanocubes (Figure 1a).

The onset of self-assembly was usually observed after about 20-30 min. As a result, the self-assembly process can be divided into three stages, where first the nanoparticles are well dispersed, followed by aggregation of the nanocubes to large and dense clusters that are forming highly ordered crystalline materials with a simple cubic structure in the crystallization stage (Figure 1b). After each experiment, the dried beads were transferred to a substrate and probed with scanning electron microscopy where spherical and cubic mesocrystals were observed for C068 and C091 (Figure 1c and d), respectively, and both

displayed a face-to-face orientation of the nanocubes in a simple cubic lattice, corroborating the SAXS diffraction patterns. Advanced analysis of the collected data is ongoing.

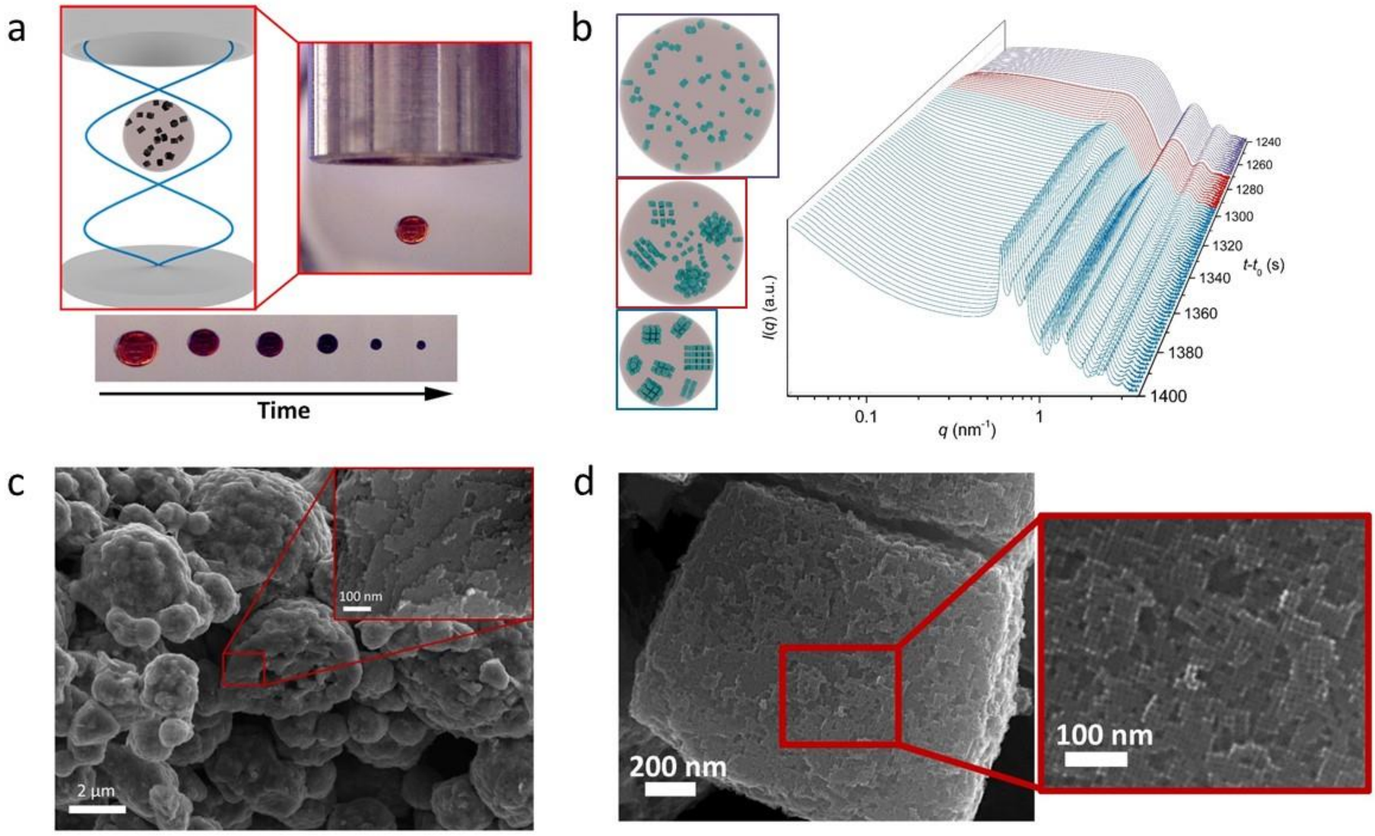

Fig. 1: Overview of the experiment, the obtained time-resolved data, and the formed mesocrystals. a) Schematic representation of the working principle of an acoustic levitator, in which the droplet levitates between the pressure nodes of an acoustic wave (top left). The levitating droplet, recorded by the microscope camera, is depicted (top right) and the shrinkage of the droplet over time is shown (bottom). b) A part of a time-resolved measurement is shown to show the different stages of self-assembly and corresponding schematic illustrations of the colloidal droplet, showing a dispersed (purple), aggregated (red), and crystalline state (blue). c) Formed meocrystals of C068 showing spherical shape and face-to-face orientation of the nanocubes (inset). d) Cubically shaped mesocrystal formed by C091 and the face-to-face orientation of the nanocubes.