ESRF	**Experiment title:** Functionalization of boron nanoparticle surfaces	**Experiment number**: SC-4579
Beamline: ID-31	**Date of experiment**: from: 27th January 2018 to: 30th January 2018	**Date of report**: 25.02.2018
Shifts: 6	**Local contact(s)**: Maria V. Blanco	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Christian Schieber, Thessa Kehler, S. Diez, R. B. Neder

Institute of Crystallography and Structural Physics

Friedrich-Alexander Universität Erlangen-Nürnberg

Report:

The aim of the proposal was to study the growth of boron nanoparticles at ambient conditions under exclusion of oxygen. This was a completely new attempt to synthesize and monitor the development of nanoparticles of this low scattering element. On the one hand we measured ex-situ prepared samples consisting of different boron polymorphs as well self-synthesized boron particles. Due to the complexity and novelty of the synthesis PDF data of samples synthesized within 2 years of preparation were accumulated (Fig. 1a).

On the other hand, the in-situ synthesis was monitored with a time resolution of 10 seconds. Herein, we focused on two different steps of the synthesis. The first step was the mere reduction process which comprises the nucleation and grows of the particles. The second step was the passivation of the boron surface step with organic ligands. An example of the latter reaction with isopropanol as ligand is shown in Fig 1b.

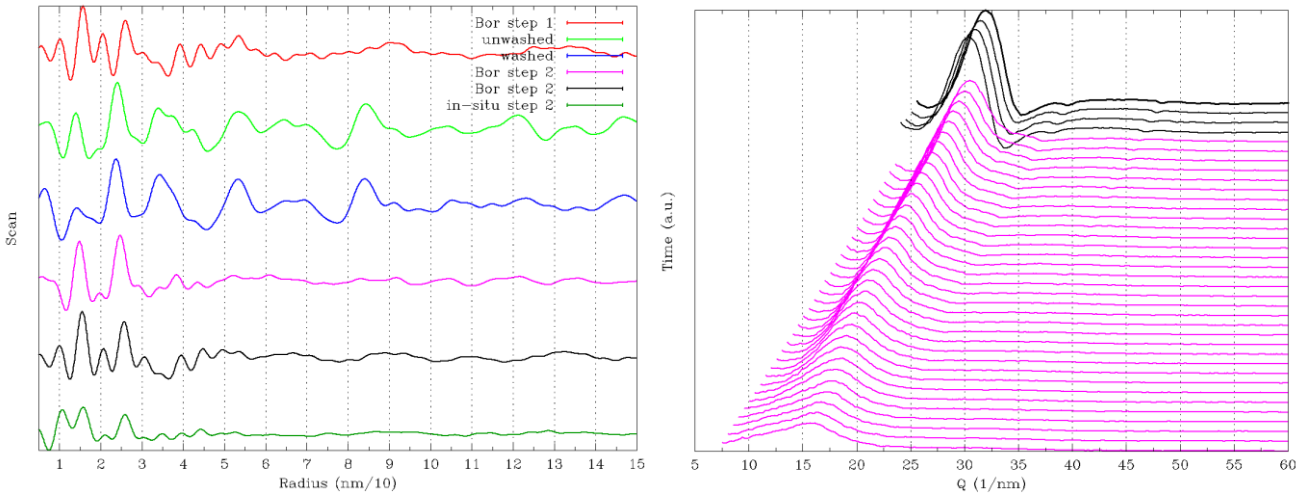

Fig1. a) PDFs extracted from raw data without corrections (left), b) in-situ addition of 2-propanol as ligand. Depicted are difference data sets with respect to file 1 (start of the synthesis) in a time frame of 1.5 hours (right).

The ex-situ data suggests particle sizes of less than 6 nm, which are readily accessible to model. As the structure of boron polymorphs and amorphous boron is not conclusively determined, there is a need for plenty of assumptions with regard to nanoparticles. Merely some TEM pictures of spherical Boron NPs originating from the room temperature synthesis are vaguely described in the literature. The collected data reveals that commercially available particles are much larger than described and show various different structural properties compared to the self-synthesized powders.

After having found a suitable structural description of the fully aged particles resulting from synthesis - the actual in-situ synthesis can be assessed. The first step of the reaction was for all particles similar with the rapid production of NaBr in the course of the synthesis. The second more interesting step of the reaction can be resolved more easily and involves the stabilization of the particles. As the difference data illustrates, gradual changes can be recorded. They do not involve substantial further growth of the particles as dips just in very late stages of the measurement at Q ~30 nm^{-1} (black curves) emerge. Very early PDF data extracted from the in-situ synthesis contains a massive organic surrounding of the particles (bottom green curve Fig. 1a). As expected, primarily the solvent/ligand contribution results in strong C-C distance peaks at 1.5Å in the PDF.
More experiments had been carried out but foremost additional time is required for data reduction (since the detector moved uncontrollably whilst data collection). It is apparent that boron samples with low scattering cross section, similar to measuring gas phases, are very sensitive to detector errors/polarization effects. Hence, we need to develop and refine a tailored data processing routine in order to reach high Qs while creating less pronounced Fourier ripples in the PDFs.