ESRF	**Experiment title:** In-situ studying of CdS nanoparticle growth in glass matrix	**Experiment number**: HC-4457
Beamline: ID02	**Date of experiment**: from: 06th May 2021 to: 09th May 2021	**Date of report**: 08th November 2021
Shifts: 6	**Local contact(s)**: Peter Boesecke Thomas Zinn	*Received at ESRF:*

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Report:

The aim of the experiments was to study the growth kinetics of cadmium sulfide (CdS) nanoparticles in a glass matrix and track their structural changes during the formation process. Here, a SiO_2-Na_2O-ZnO glass matrix was chosen, since it offers the unique possibility to slow the nanoparticle formation kinetics, which is in the micro- and millisecond range in wet environments like solutions, down to minutes or even hours. Furthermore, these nanoparticle-glass composites are primary candidates for future optical applications [1] due to their thermo-responsive tunability of the nanoparticle size and size distribution [2].

In the present experiment, pre-synthesized ultra-thin glasses were studied at beamline ID02 using SAXS and WAXS simultaneously to track any structural changes on the atomic and nanometer scale. Each sample was heated to 610 °C using a heating ramp of 600 °C/min. Upon heating the samples past the glass temperature of the matrix, the Cd and S ions gain mobility and the nanoparticle nucleation starts. With the desired higher annealing times, the mobility further increased and allowed the controlled time-dependent observation of very small nanoparticles and their subsequent agglomeration in the same experimental set-up. The data acquisition rate for each sample was in the minutes range and was continued for several hours. In total two glass samples with different initial concentrations of Cd and S ions were measured at 580 and 610 °C. As a reference and for the correct background subtraction, a complete data set of the pure glass matrix was measured as well. To prevent beam damage as much as possible and investigate the homogeneity of each sample, the beam position was shifted constantly during the experiments.

Preliminary results:

All detector images were already azimuthally averaged by the beamline staff using their software. At the moment, a script for batch processing of all data sets including the correct background subtraction, scaling on absolute intensities and data modelling is prepared. Even the pure scattering curves already clearly indicate the formation of small nuclei and nanoparticles for temperatures larger than the glass temperature of the matrix (see

figure 1). Roughly after 2 hours, the kinetics seem to slow down and the scattering signal remains almost constant, which could indicate the equilibrium state of the nanoparticle sizes for the chosen temperature.

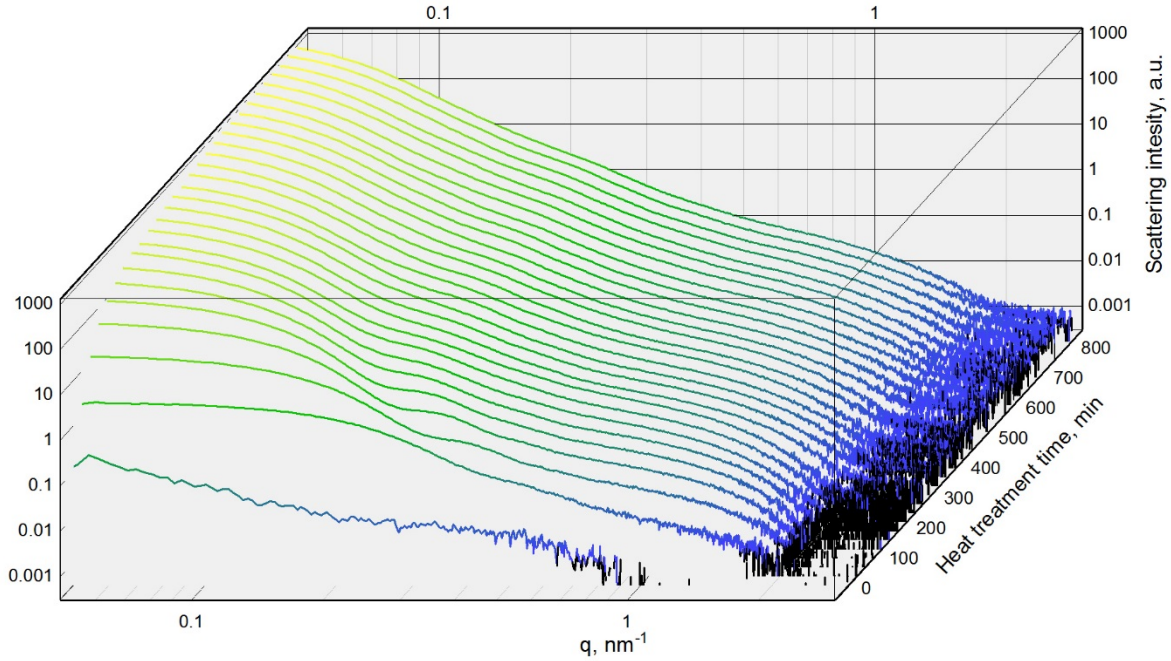

Figure 1. Time-dependent SAXS data during the formation of CdS nanoparticles at 610°C.

At the same time, an overview of experimental data shows strong scattering of x-rays in the q-range from 0.05 to 0.1 nm^{-1} alongside with scattering from CdS nanoparticles This scattering signal relates to the centers with an average size of about 30 nm and has time and temperature dependence, however, it completely disappears after cooling of the glass. These scattering centers could be elementary units of glass or inhomogeneities of glass matrix and has to be carefully considered during general data treatment.

Future use of the data:

During data analysis using custom-made procedures, the shape and internal structure (density and crystallinity) of the nanoparticle will be visible and the results will be published in an upcoming research paper, which will bridge the gap in the literature between commonly used theoretical approaches and experimental data of the nucleation and further growth processes of semiconductor nanoparticles.

References:

[1] I.D. Popov, Y. Kuzentsova et al., J Nanopart Res 20, 78 (2018)
[2] I.D. Popov, B. Sochor, Y. Kuzentsova, B. Schummer et al., J. Non-Cryst. Solids 529, 119781 (2020)