	Experiment title: Nucleation, growth and superlattice formation of PbS and PbSe colloidal nanocrystals	**Experiment number**: SC4858
Beamline: ID02	**Date of experiment**: from: 12/09/2018 to: 14/09/2018	**Date of report**:
Shifts: 6	**Local contact(s)**: Lewis "Lee" Sharpnack	*Received at ESRF:*

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Report:

This experiment being the continuation of SC4365 the experimental set-up and reagents were essentialy the same. A solution lead oleate in hexadecane is poured in a three-neck round bottom flask under argon. An oil bath is used to set the temperature. The reacting fluid is pumped through a glass capillary using a peristaltic pump equiped with Vitton tubing. We wait for around 10 minutes for thermal equilibrium to be reached after the pump is set on. During the course of the reaction, the temperature inside the reacting fluid is monitored and recorded. A decrease of a few degrees is observed after the thiourea injection but the temperature quickly reaches back the set value. 1 mL of the thiourea solution in tetraglyme is sampled in a plastic syringe which is then set on a stand. The injection of this solution is controlled remotely from outside the beamline hutch with a pneumatic piston to which a TTL signal is sent. This time defines the t=0 s of the sequence. At this point SAXS/WAXS acquisitions are triggered. In a typical sequence, 1200 SAXS and WAXS patterns with a duration of 0.3 s are taken every second (i.e. there is a 0.7 s waiting time between two acquisition).
During all the process, we took great care to respect an air free environement by using an argon tank and classical Schlenk line techniques. A notable difference with respect to the previous experiment is that there is no recirculation. Once the reactive fluid has been pumped from the flask and went through the X-ray capillary, it is evacuated. This was to avoid any contamination of the reactive flask with radicals or species created an interaction with the beam. A conseqence of this choice is the volume of the initial and the overall quantities of reagents to handle which were much higher than in the previous case.

After having measured the different solvents we performed a total of 15 time resolved experiments with different conditions. The beam was very stable. We had several experimental problems with the flow cell

which did not hold well the large temperatures at which the reactions occurred. We thus lost a few hours of beamtime and had to build flow cells during the experiments. In the future we will conceive and build a flow cell made of heat resisting materials and glue to avoid these problem.

Overall this was a successuful experiments which confirmed our previous findings and extended the range of experiemal parameters that we explored. We have analysed the data and compared them to a model developed with our theoretician collaborators. We are close to the submission of a paper.

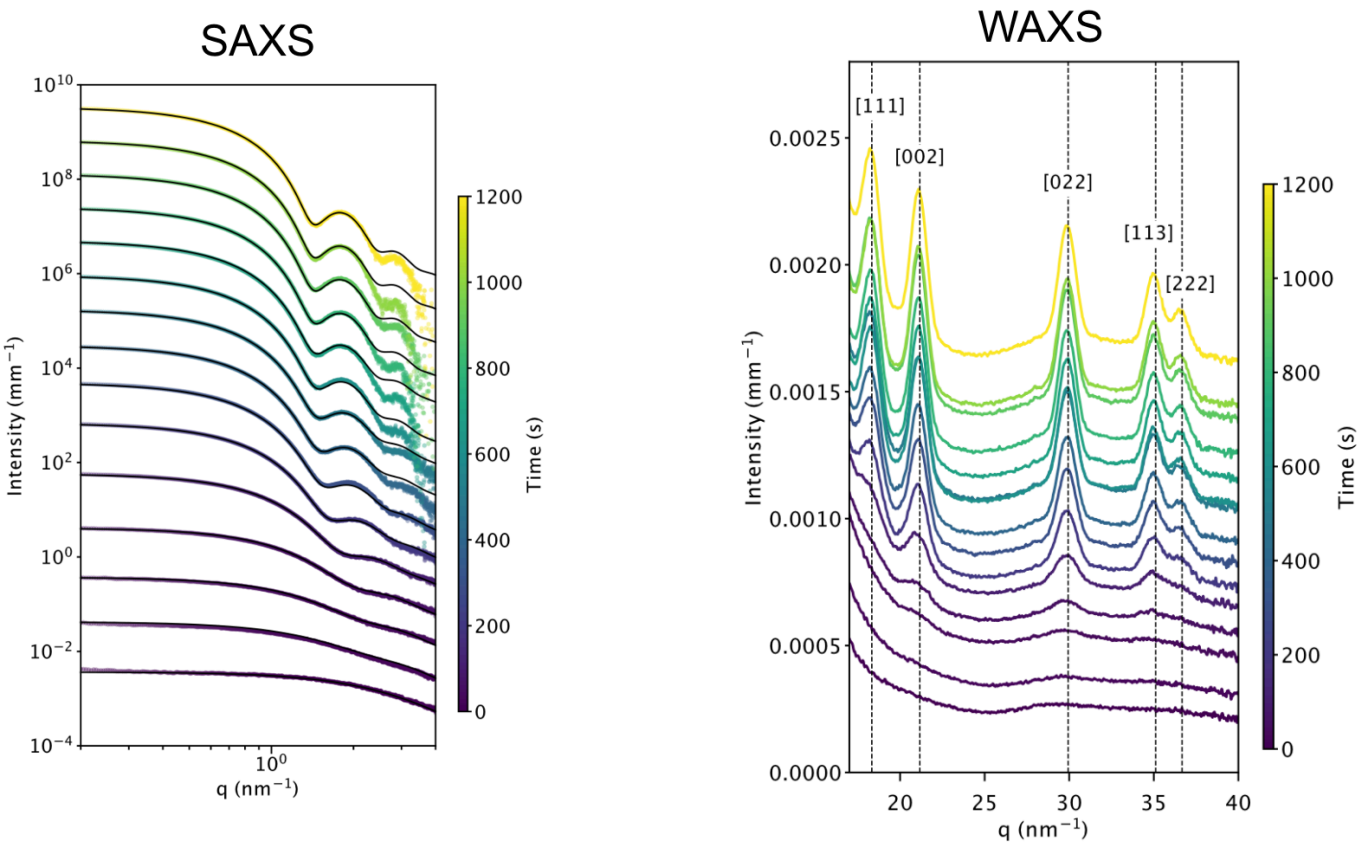

Figure 1: typical example of SAXS and WAXS patterns for a PbS sequence