



	<b>Experiment title:</b> <b>Anomalous SAXS studies of magnetic core-shell nanocrystals</b>	<b>Experiment number:</b> <b>SC- 3459</b>
<b>Beamline:</b> ID01	<b>Date of experiment:</b> from: 05.12. to: 10.12.2012	<b>Date of report:</b> 1.3.2014
<b>Shifts:</b> 15	<b>Local contact(s):</b> Peter Boesecke	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): R.T. Lechner*,. O. Paris, G. Fritz-Popovski* <i>Institut für Physik, Montanuniversität Leoben, Franz-Josef-Strasse 18, 8700 Leoben, AUSTRIA</i>		

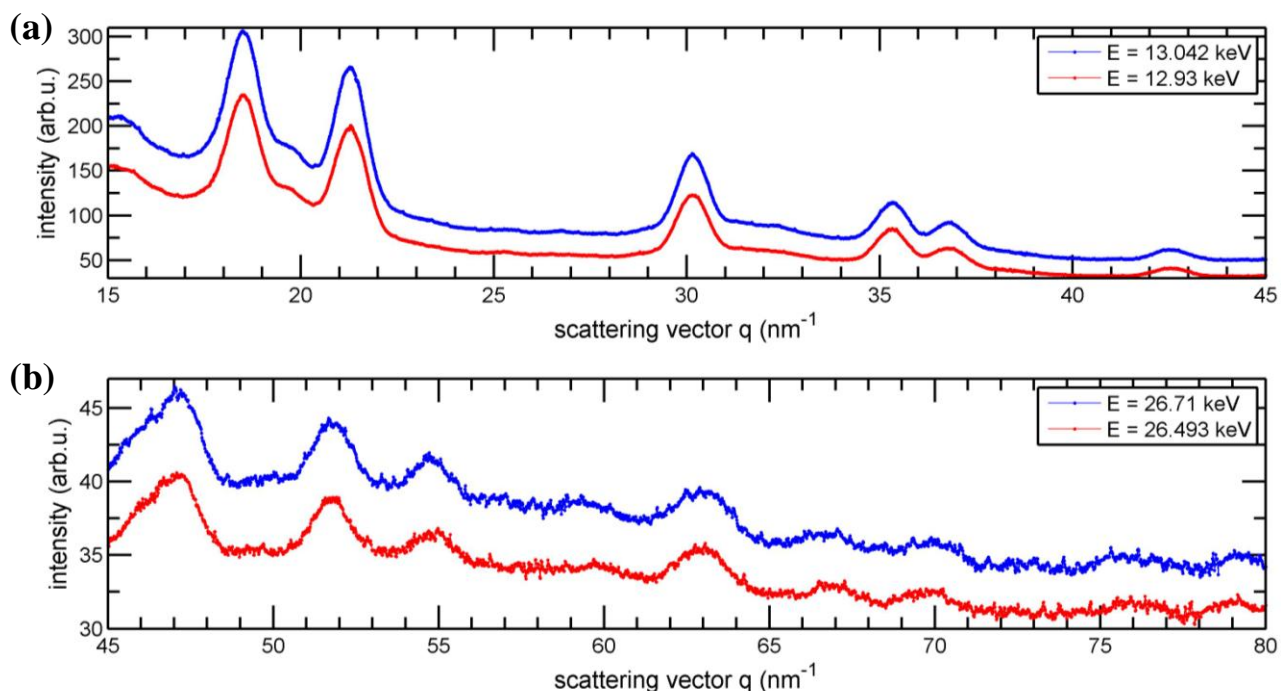
## Report:

We proposed to study with anomalous wide angle x-ray scattering (AWAXS) PbS/CdS core/shell nanocrystals (NCs). This type of core/shell NCs are chemically synthesized in a two step process, whereas in the second step the CdS-shell formation is realised by a cationic Cd for Pb exchange. The aim of this beamtime was the detailed determination of the crystalline properties of the PbS/CdS core/shell structure together with their strain state as a function of the shell thickness.

During this beamtime we have investigated three different sample series with final shell thicknesses of 0.9, 1.5 and 2 nm using three different diameters for the initial PbS NCs of 4.7 nm (small), 6.3 nm (medium) and 8.7 nm (large). The chemical composition profile of the CdS-shell as a function of reaction time have been derived previously from anomalous SAXS (ASAXS) experiments in sub-nanometer resolution.

We recorded AWAXS spectra around the Pb-L<sub>III</sub> edge (13.035 keV) as well as the Cd-K-edge (26.71 keV). The NCs-solutions were first measured within quartz glass capillaries with diameters of around 1.5 mm and the data were collected with the 2D maxipix detector. I turned out, however, that the NC solution was not stable during irradiation with the high brilliant beam. At the x-ray spot position the NC concentration was massively increased, which was even visible by eye due to a change in the colour of the solution. Even after reducing the counting time, the influence of the x-ray beam on the NC concentration and hence on the intensity of the measured Bragg peaks was too high for an accurate data analysis. In the planned evaluation of the Bragg peaks we have to analysis small changes in the Bragg peak intensities as a function of the x-ray energy.

We could overcome this problem by drop casting the NC solutions on kapton foils and by measuring the so realised NC films. Strong peaks visible in the SAXS regime at small  $q$ -values proved that these films already formed well pronounced colloidal super-crystal structures. This enabled us to record numerous high order Bragg peaks at large  $q$ -values in the WAXS regime, especially at the high x-ray energy around the Cd edge corresponding to a short x-ray wavelength. (see Figure 1)



**Fig.1:** (a) AWAXS spectra of the large core/shell NCs with a 7 nm PbS core and a 0.9 nm thick CdS shell. Exemplified two spectra with selected  $q$ -range measured at x-ray energies near the Pb-edge (blue) and below (red) are shown. (b) AWAXS spectra of the medium sized core/shell NCs with 3.6 nm PbS core and 1.4 nm thick CdS shell close (blue) and far away of the Cd-edge. The  $q$ -range is only partly shown

For analysing these numerous Bragg peaks we developed a new software PCG-SWAXS [1], where the geometrical core/shell parameters, the chemical composition as well the crystal structure can be varied freely for calculating the whole diffraction pattern as a function of the x-ray energy. (As input only the energy dependent atomic scattering factors  $f'$  and  $f''$  are needed.)

This will enable us to simulate and fit the changes of the Bragg peak positions and intensities at both edges to probe the influence of Pb and Cd on the shell's and core's crystal structure, independently.

[1] The software was developed by one of the co-proposer G. Fritz-Popovski and is freely available. Please contact directly either [gerhard.popovski@uniloeben.ac.at](mailto:gerhard.popovski@uniloeben.ac.at) or [rainer.lechner@uniloeben.ac.at](mailto:rainer.lechner@uniloeben.ac.at)