	Experiment title: **Anomalous SAXS studies of magnetic core-shell nanocrystals**	**Experiment number**: **SC- 2967**
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Report:

We aim to characterise the structure of nanocrystals (NCs) in solution depicting different core-shell structures by means of anomalous small angle x-ray scattering (ASAXS). One sample series consists of a FeO core surrounded by a $MnFe_2O_4$ shell. The other class of core-shell NCs consists of the narrow band gap semiconductor PbS core and a CdS shell grown by cationic Cd-Pb exchange. Especially, we want to reveal the chemical composition of core-shell structure as a function of the growth time and particle size. The knowledge of the exact core-shell structure is crucial for the understanding of the magnetic properties of the NCs and of the optical properties in the mid-infrared for the PbS/CdS NCs.

During this beamtime we have investigated the FeO/ $MnFe_2O_4$ core shell nanostructures around the Mn-K-edge (6539 eV), as well as the PbS/CdS NCs around the Pb-L_{III} edge (13035 eV).
The NCs-solutions were measured within quartz glass capillaries with diameters of around 1.5 and 2 mm and the data were collected with the 2D CCD. The beamline monochromator resolution is around 1 eV, whereas the energy change of the transmission of pure metal (= Mn, Pb) foils was used for energy calibration. From the measured curves containing NCs the scattering curve of the pure solvent was subtracted and the intensity was calibrated to differential scattering cross-sections in units of cm^{-1} with a pre-calibrated glassy carbon sample. For all NCs samples transmission measurements as a function of energy were performed to derive the experimental absorption edge of the nanocrystal-compound materials.

In Fig. 1(a) the scattered intensities as a function of the reciprocal scattering vector Q at three different energies (at and below the Mn-K-edge) is shown. The scattering curves a characteristic for polydisperse spherical nanoparticles with a diameter of around 8 nm, but with a quite narrow size distribution of around 10%. The decrease in the intensity from 100 eV below the edge (blue line) with respect to the curve at the Mn-edge (red line) can be directly related to the reduced Mn atomic scattering factor just below the edge.

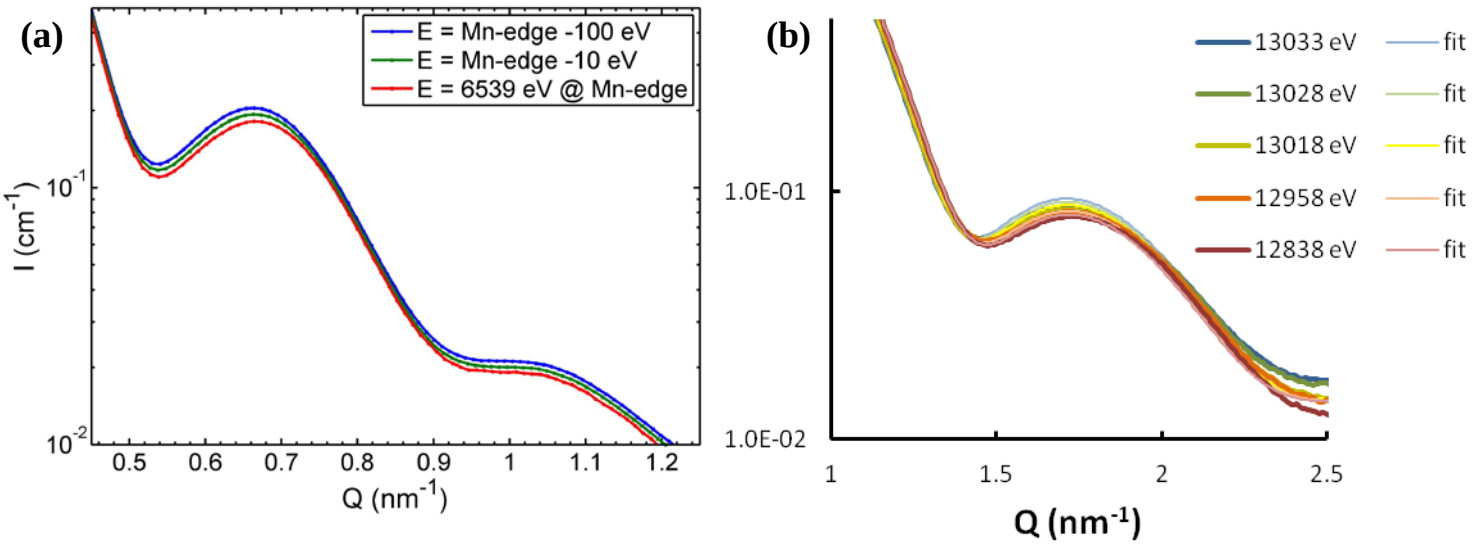

Fig.1: (a) ASAXS spectra of spherical FeO/ $MnFe_2O_4$ NCs with a total diameter of 8.2 nm measured at x-ray energies near the Mn-edge (red) and below (green and blue line). **(b)** Measured ASAXS intensity over reciprocal scattering vector Q of PbS/CdS core shell NCs at 5 different energies below the Pb-L_{III} edge of 13035 eV (thick lines) and the fitted spectra (thin faint lines) using one unique core-shell density profile for all scans. The outer diameter of the PbS/CdS spheres is 6.3 nm.

The lateral inhomogenous intensity shift as a function of Q inidcates an inhogenous Mn-distrubution within the spherical nanocrystal. This allows to derive the Mn concentraion profile by fitting the ASAXS scattering curves measured at least at three different energies. This is shown for the PbS/CdS core-shell nanostructures shown in Fig. 1(b). The curves are measured at 5 different energies below the Pb-edge (thick lines) and are fitted with one unique core-shell model (thin lines). The good correspondance between fits and experimental data allows to determine the Pb-concentration profile with an accuray of around 1 Pb-atom per nm^3. The results show that the spherical nanoparticles with an outer diamter of 6.3 nm consits of a 3.3 nm PbS core surrounded by a 1.5 nm thick shell with an overall size distribution of around 10%. We could additionally show that this CdS-shell improves massively the optical emssion of the PbS-core in the mid-infrared [1].

The same developped routine we will apply now to the magnetic FeO/ $MnFe_2O_4$ core shell nanoparticles and will relate these findings to their magnetic properties.

[1] R.T. Lechner, G.Fritz-Popovski, M.Yarema, W. Heiss, O. Paris, *Characterisation of PbS/CdS Nanocrystal Core-Shell Structures by ASAXS*, workshop: ASAXS in Condensed Matter Science, Synchrotron BESSY II, Helmholtz-Zentrum Berlin für Materialien und Energie, 24.-25.11.2011

R.T. Lechner, G.Fritz-Popovski, M.Yarema, W. Heiss, O. Paris, *Characterisation of nanocrystal core-shell structures in sub-nanometer resolution by anomalous small angle x-ray scattering*. - in: Material Research Society: MRS 2011 Fall Meeting. Boston, USA, 28.11.-2.12.2011

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