ESRF	**Experiment title:** SAXS/WAXS experiments on luminescent nanoparticles for OLEDs applications	**Experiment number**: CH-2811
Beamline: ID02	**Date of experiment**: from: 17/09/2009 to: 19/06/2009	**Date of report**: 25/02/2010
Shifts: 6	**Local contact(s)**: Peter Boesecke	*Received at ESRF:*

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Report:

The aim of this experiment was to measure the structural properties (size, crystal structure, polydispersity) of CdS nanoparticles synthesized by thermolysis by means of WAXS and SAXS. The samples were prepared by dispersing 30mg of $Cd(SR)_2$ precursor where R = C_nH_{2n+1} (n=12), in 20mL of octadecene, a high boiling point solvent (315°C). The influence of the reaction annealing temperature and extraction treatment after the synthesis was investigated.

Experimental details

The SAXS and WAXS experiments were carried out at the beamline ID02. We put the samples in glass capillaries of borosilicate (ϕ = 2mm) to aquire transmission measurement. The wavelength used was λ = 0.918Å corresponding to an incident energy of 12.46 keV. For WAXS the distance between sample and detector was 0.118mt, for SAXS it was 2.5. The signal was revealed by a CCD.We performed both *ex-situ* and *in-situ* mesurements. The *ex-situ* measurements were carried on solutions prepared previously to the experiment at two synthesis temperatures: 200 and 240°C, and for each temperature we compare the solutions before and after the extraction of the CdS nanoparticles. The *in-situ* measurements were recorded starting from the solution of the precursor in octadecene and collecting data during the thermolysis.

Results: WAXS

Concerning the *ex-situ* measurement, we report the results for two samples He2 (annealing temp:200°C) and He5 (annealing temp:240°C) before and after extraction treatment (Fig.1 a) and b)). The peaks at q = 26.5 and 37.2 nm^{-1} are due to reflections (102) and (201) of the exagonal phase of CdS. The peaks at 14.9 and 23 nm^{-1} don't belong to CdS and could be due to impure crystalline phases. It is interesting to observe that the signal/noise ratio of the peaks improves for the extracted solutions pointing out that the extraction helps to eliminate organic residues. An increase of the annealing temperature promotes the growth of the nanoparticles, as seen from Fig1b) where the peaks are more intense with narrower FWHM.

a) *b)*

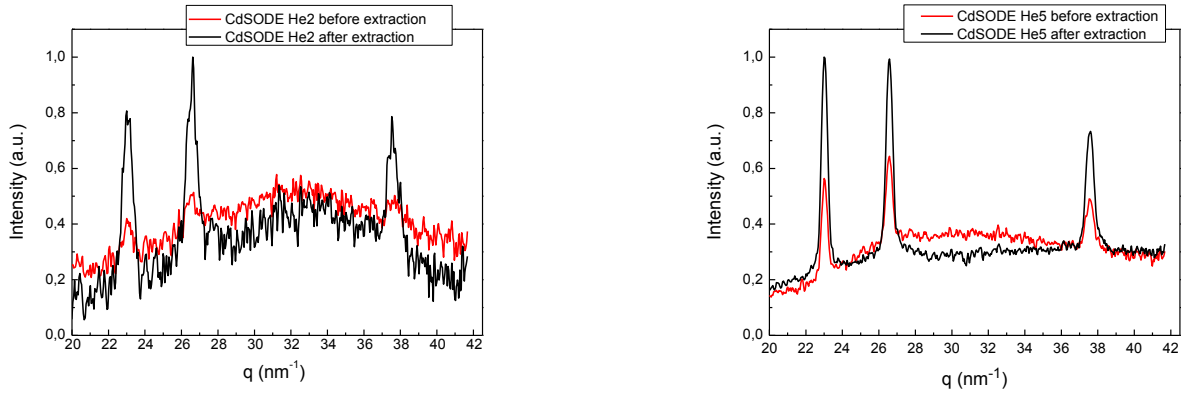
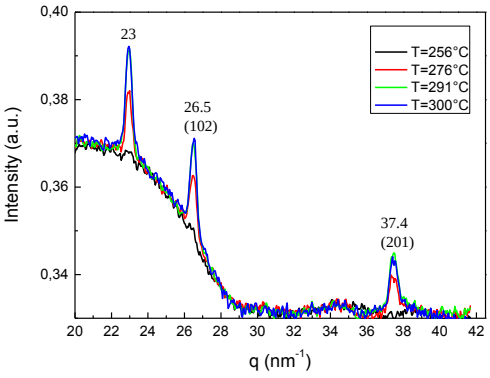


Fig 1. WAXS spectra of the *ex-situ* measurements on samples He2, T annealing: 200°C (a) ed He5, T annealing: 240°C (b) before and after extraction of reaction mixture.

The WAXS *in-situ* experiments results are shown in Figure 2 as a function of the temperature. Three diffraction peaks are present: (102) and (201) of the CdS hexagonal phase and the unidentified peak at q = 23 nm^{-1}, as found in *ex-situ* samples. The i*n- situ* process confirms that the nanoparticles become bigger when the annealing temperature increases (9.9nm at 300°C vs 8.9 nm at 240°C). Compared to *ex situ* results, at the same annealing temperature the peaks are less intense. This could be due to a lower nanoparticles number as consequence of: a) the *in situ* process could have taken place faster than *ex situ* one b) the *in situ* reaction took place in a capillary with a very small volume, while the *ex situ* one was conducted in a reaction flask and the solution was continously stirred to guarantee a homogeneous heating.



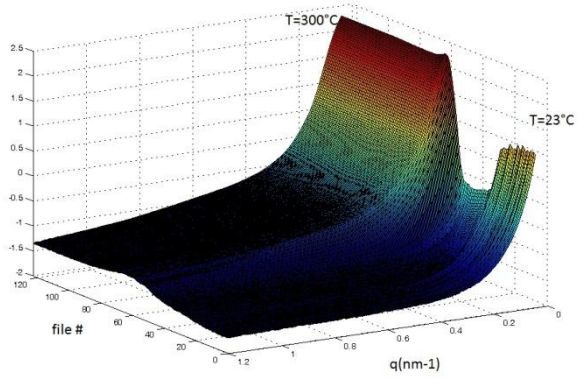


Fig2. WAXS spectra of the *in-situ* experiment

Fig3. SAXS spectra of the *in-situ* experiment

The SAXS in-situ results are reported in Fig3 starting from room temperatures up to 300°C. The initial scattering due to the precursor progressively disappears at about 80°C and a pronounced scattering from the nanoparticles is visible at 130-150°C. This peak doesn't seem to show a dependence on the annealing temperature as the crystalline peaks in the WAXS do. A detalied analysis of the SAXS data is currently on-going.

Publications

This project is part of the PhD work of S.Masala.