



	Experiment title: Magic-sized Clusters in ZnO Nanoparticle Formation in Sol-gel Processes	Experiment number: CH 4156
Beamline: ID-15-B	Date of experiment: from: 29 th October 2014 to: 31 st October 2014	Date of report:
Shifts: 9	Local contact(s): Agnieszka Poulain	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): M. Zobel R. B. Neder * Mustafa Saad * Institute of Crystallography and Structural Physics		

Report:

The original aim of the proposal was to study the existence of different magic sized clusters during the nucleation of ZnO nanoparticles in a sol-gel process in ethanol. The beamtime was proposed, because it was thought be necessary for the PhD project of Mirijam Zobel, the main proposer.

As time has moved on throughout the last year, the PhD project developed and the nucleation model for the ZnO nanoparticles changed. A comparison of different nucleation models showed that instead of magic sized clusters as often proposed as a nucleation scheme, no such model is applicable for ZnO. Instead, upon high supersaturation a burst nucleation of quite disordered nanoparticles occurs, which gradually crystallize over time. This concept with high-quality experimental data from previous experiments at ID15-B is summarized in a manuscript about to be submitted.

The beamtime of experiment CH4156 was hence used by the Co-Proposer R. B. Neder, together with Mustafa Saad as an experimentalist.

We found before, that ZnO with the ligand dimethyl-L-tartrate (dmlt) produces the most size-stable nanoparticles, just recently published in JNR (Zobel, M., Chatterjee, H., Matveeva, G., Kolb, U. & Neder, R. B. Room-temperature sol-gel synthesis of organic ligand-capped ZnO nanoparticles. *J. Nanoparticle Res.* 17, (2015)). Based upon this, during the beamtime of this proposal, mainly experiments on the nucleation of ZnO with the ligand dmlt have been carried out in ethanol. The studies were focused on the influence of different supersaturations of the base tetramethylammonium hydroxide (TMAH) on the nucleated particle sizes and the crystallinity of these particles. The base was either injected or continuously and gradually added with different ramp speeds in between 1.2 and 6.0 mM TMAH/min. Moreover, a ZnO dmlt and a ZnO pent solution was heated prior to the experiment to ca. 60 °C for about an hour and the base injected thereafter to study the influence of enriched precursor solutions onto the nucleation. The data analysis of these experiments is with Reinhard B. Neder.