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Real time study of wet-chemical reaction dynamics at beamline P02.1 @ PETRA III

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The high brilliance synchrotron light source PETRA III in Hamburg, Germany, provides a dedicated X-ray powder diffraction beamline called P02.1 [1]. It is a side station to the hard X-ray diffraction beamline and runs at a fixed photon energy of 60 keV. Its dispersive monochromator produces a highly collimated photon beam of very narrow energy bandwidth and high intensity. These excellent beam characteristics turn P02.1 into an ideal instrument for many different kinds of experiments, ranging from high resolution powder diffraction of polycrystalline materials for structure solution and refinement or microstructure analysis, to the study of nanocrystalline and disordered materials to determine their local structure. In particular, it is the scope of P02.1 to study dynamic processes such as chemical and crystallographic transitions under non-ambient conditions in real time. For this purpose, the beamline is equipped with a large and fast area detector which enables sub-second time-resolution. The accessible range in reciprocal space is beyond Q = 30 $Å^{-1}$. Hence, P02.1 is a powerful tool for total scattering experiments as it provides high resolution in real and reciprocal space which are determined by the max. Q and the instrumental resolution, respectively. This presentation describes some recent experiments carried out at P02.1 that relate to pair distribution function (PDF) and total scattering analysis. The focus will be on the investigation of structural changes on the atomic scale during the wet-chemical synthesis of nanoparticles, e.g. in the system ZrO_2. By means of evaluating the changes of bond distances and atomic coordination on a time scale of seconds, it is possible to describe the molecular structure of intermediates and, thus, to deduce the underlying reaction mechanism. On the basis of this information, synthesis processes may be optimised with respect to tuning the properties of the product and to maximize its yield.

[1] *A.-C. Dippel, J.T. Delitz, H.-P. Liermann et al., J. Synchrotron Rad., to be published,* ***[2]*** *C. Tyrsted, N. Lock, K.M.Ø. Jensen et al., IUCR J. 1 (2014) 165.*

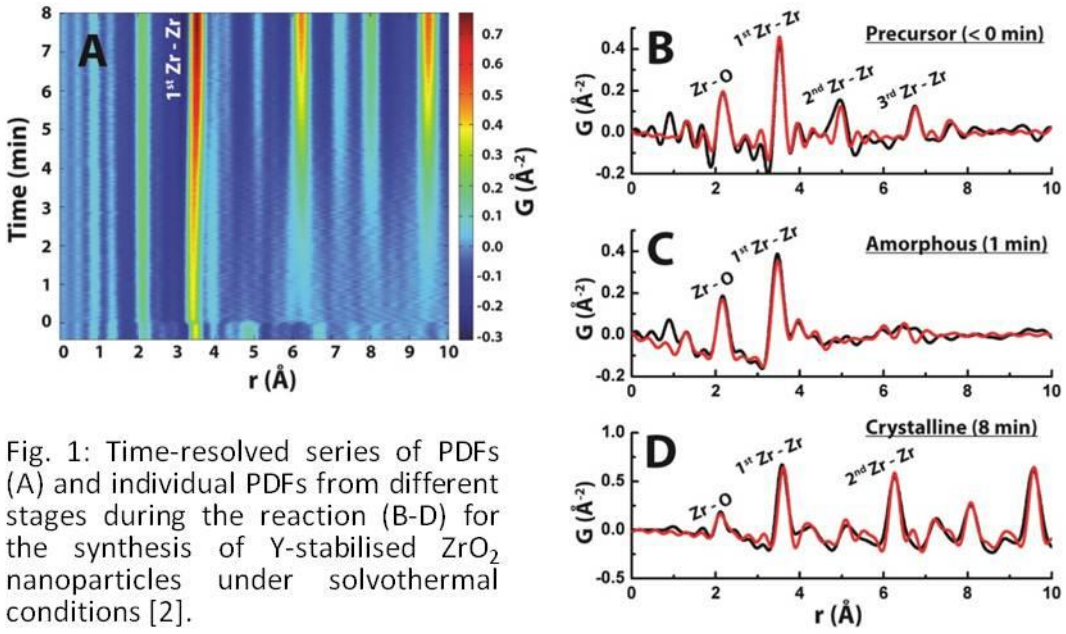

Fig. 1: Time-resolved series of PDFs (A) and individual PDFs from different stages during the reaction (B-D) for the synthesis of Y-stabilised ZrO_2 nanoparticles under solvothermal conditions [2].

Keywords: synchrotron powder diffraction, pair distribution function, reaction mechanism