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In- and ex situ PXRD studies of ZnO nanoparticle growth in sub-critical water

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Zinc oxide (ZnO) is a material of great scientific and industrial relevance and is used widely in a variety of applications. Synthesis of ZnO nanoparticles can be performed by a wide range of methods resulting in a tremendous variety of sizes and shapes. Different in situ characterization methods have been used to investigate the ZnO formation under various synthesis conditions; these include numerous spectroscopic methods and small angle scattering. Common for these studies is that the primary focus has been to extract information on particle size and shape of ZnO, while a more rigorous microstructural and structural analysis has been lacking. Furthermore, the aforementioned studies have primarily been focused on soft chemical synthesis methods, at low temperatures and in non-aqueous media, thus omitting the widely used environmentally benign and versatile hydrothermal method. In the present work the formation of ZnO during hydrothermal synthesis has been followed using in situ powder X-ray diffraction (PXRD) combined with Rietveld refinement, thus enabling the extraction of crystallographic as well as microstructural information during the formation and growth of ZnO. Supporting ex situ syntheses and characterization by electron microcopy, high resolution PXRD and other techniques have been used to corroborate the findings from the in situ experiments. Mapping out a vast parameter space has led to a deeper understanding of the intricate mechanisms governing the nucleation and growth of ZnO nanoparticles during hydrothermal synthesis. Among the parameters studied were the influence of temperature, type of base used and the influence of different ionic salts as synthesis directing agents. The various synthesis parameters were found to influence the following structural and microstructural features: crystallite shape, morphology and size as well as the twin-fault concentration, degree of doping and crystallinity.

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