

***In Situ* Synchrotron PXRD Studies of the Formation and Growth of Nanoparticles under Solvothermal Conditions**

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It is well-known that the physical and chemical properties of nanoparticles can be modified by minor changes of either the morphology, size and/or crystal phase.¹ Although, nanoparticles have been used for more than two decades in a range of different applications, such as photovoltaics, catalysis and sensing is the optimization of the properties based on empirical evidence. *In situ* crystallographic tools have proven useful in revealing the mechanism underlying the formation of nanoparticles. For the further progress in the development of tailored nanoparticles, understanding the mechanisms controlling formation and growth during solvothermal synthesis is of key importance.

We have used *in situ* synchrotron powder X-ray diffraction (PXRD) to study the formation and growth of different technological important materials such as colloidal monodisperse spherical Cu_{2-x}S nanoparticles synthesised in dodecanethiol,² $\text{Y}_3\text{Al}_5\text{O}_{12}$ ³ and CoSb_2O_4 nanoparticles synthesised under hydrothermal supercritical conditions. It is vital for future improvements in properties that the extracted crystallographic information from *in situ* PXRD is translated into unique information about how to control the size and phase of nanoparticles. We have previously shown for Cu_{2-x}S nanoparticles how the crystalline precursor $[\text{CuSC}_{12}\text{H}_{25}]$ decomposes upon heating and forms an isotropic liquid, which subsequently turns into colloidal β -chalcocite phase Cu_2S nanoparticles. In the present study on CoSb_2O_4 nanoparticles we show by *in situ* PXRD how the precursor stoichiometry influences the formation mechanism of the nanoparticles. The activation energy for the formation of phase pure CoSb_2O_4 is analysed and compared to similar oxides. Furthermore, we show how the crystallite size of CoSb_2O_4 nanoparticles varies as a function of synthesis temperature and time.

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