Oral Contributions

[MS27] Total Scattering
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[MS27 - 01] Watching materials form: In situ total scattering studies of nanoparticle synthesis Kirsten M. Ø. Jensen[1], Christoffer Tyrsted[1],Mogens Christensen,[1] Espen D. Bøjesen,[1] Nina Lock,[1] Simon Billinge,[2/3] Bo B. Iversen[1]

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As the properties of many advanced functional materials are highly dependent on particle characteristics such as crystal phase, crystallinity, defects, crystallite size and morphology, it is crucial to understand the mechanisms that govern these during material synthesis. For this purpose, in situ studies of particle synthesis have in recent years proven extremely powerful, and important knowledge has been obtained using e.g. PXRD and SAXS.[1] However, to fully understand how a material crystallize from an aqueous solution, a liquid or an amorphous precursor, it is necessary to probe the atomic structural changes during the initial nucleating and crystallization. For this, Total Scattering (TS) is perfectly suited. TS provides information on both short and long range order, and it is possible to extract the atomic structure of both amorphous and crystalline compounds. With the advent of the RAPDF method, total scattering patterns can be obtained within seconds, and in situ studies are therefore feasible. [2]

Here, some of our most recent results from in situ total scattering studies of hydrothermal synthesis of metal oxide nanoparticles will be presented. We use a small capillary reactor for studies of hydrothermal and solvothermal syntheses and can study reactions taking place in water or other solvents at elevated temperatures and pressures. Specifically, we have recently investigated the hydrothermal formation of SnO_2, [3] CeO_2, [4] ZrO_2, TiO_2 and Fe_2O_3, which are all of technological relevance. From Pair Distribution Function (PDF) analysis of the in situ total scattering data, we have been able to identify the structures of the ionic complexes in the precursors as well as the amorphous nanoclusters that appear before full crystallization. By following the transformation of these structures to crystalline materials, we can deduce formation mechanisms of functional materials. The studies reveal the changes in material characteristics (atomic structure, particle size/shape/distributions, crystallinity) throughout the synthesis to give a comprehensive description, which may be used to design particles for syntheses on a larger scale.

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Keywords: pair distribution function, in situ, nanoparticles