

Poster Presentation

MS55.P11

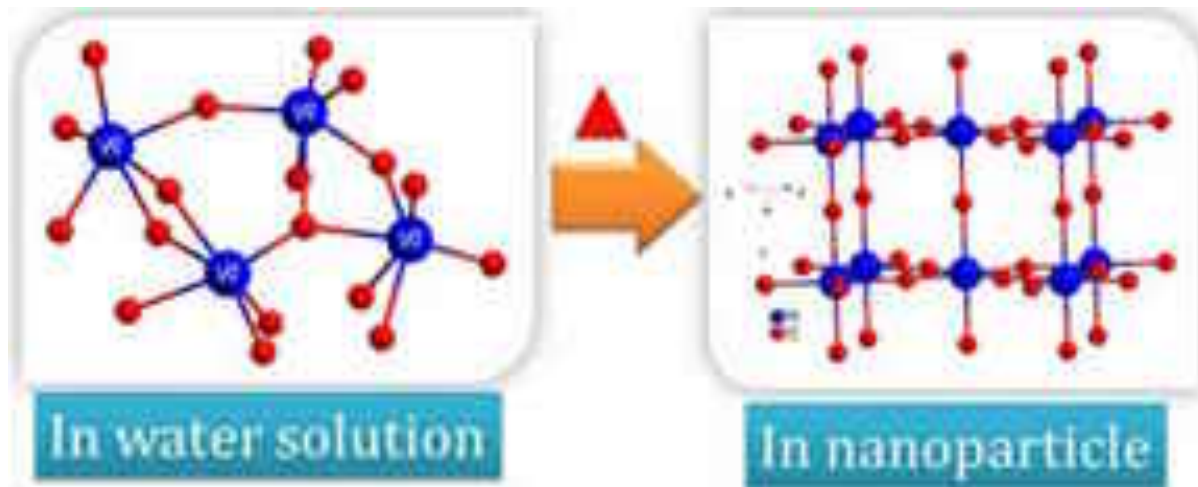
In situ total X-ray scattering study of WO₃ nanoparticle formation

D. Saha¹, K. Jensen¹, C. Tyrsted¹, E. Bøjesen¹, A. Mamakhel¹, A. Dippel², M. Christensen¹, B. Iversen¹

¹Aarhus University, Center for Materials Crystallography, Department of Chemistry and iNANO, Aarhus, Denmark, ²Photon Science DESY, D-22607 Hamburg, Germany

In situ total scattering in combination with pair distribution function (PDF) and powder X-ray diffraction (PXRD) methods have been used to unravel the mechanism of WO₃ nanoparticle formation from aqueous precursor solution of ammonium metatungstate [(NH₄)₆H₂W₁₂O₄₀.xH₂O (AMT)] under hydrothermal condition. Total scattering studies can extract precise atomic scale structural information from solutions, amorphous solids, nanosized structures as well as from crystals [1]. The reaction mechanism was followed in an in situ reactor at synchrotron [2]. The study reveals that a complex precursor structure exists in the solution. It consists of edge and corner sharing WO₆ octahedra. While heating the solution, the precursor structure undergoes a reorientation with time converting the edge sharing octahedra to corner sharing octahedra before forming the nanoparticles. While the octahedra locally become reoriented there is no evidence of long range order. After 10 min. of heating, the nuclei in the solution abruptly cluster together and form crystalline particles. The sudden formation of nano crystals is also confirmed by in situ PXRD measurement. Further PDF analysis also reveals that local structure in hexagonal WO₃ is different than the average structure and it also rationalizes the formation of two different hexagonal phase of WO₃ in two different synthesis procedure [3].

[1] S. J. L. Billinge, M. G. Kanatzidis, *Chem. Commun. (Cambridge, U. K.)* 2004, 7, 749-760, [2] J. Becker, M. Bremholm, C. Tyrsted et al. *J. Appl. Crystallogr.* 2010, 43, 729-736, [3] D. Saha, K. M. Ø. Jensen, C. Tyrsted et al. *Angew. Chem.* 2014, Accepted



Keywords: In situ total scattering, Pair distribution function, Nanoparticle