ESRF	**Experiment title:** Insights into pre-nucleation processes, nucleation and the transition from inverse spinel to normal spinel during the synthesis of zinc ferrite nanoparticles in solution	**Experiment number**: CH 4618
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Report:

On the nanoscale, particle nucleation and growth often base on intermediate states and are therefore not described in the framework of classical nucleation theory. An *in situ* study on the formation of ferrite nanoparticles by means of total scattering and its pair-distribution function (PDF) analysis are supposed to reveal the nucleation and growth mechanism during a non-hydrolytic sol-gel reaction. In particular, the influence of the composition and the reaction temperature on the particle formation are of central interest.
We studied the formation of nanosized magnetite, magnesium and zinc ferrite particles. Iron acetyl acetonate (Sigma Aldrich, >=99.999 %) was used as the iron precursor and anhydrous benzyl alcohol (Sigma Aldrich, >=99.8 %) as the solvent. Furthermore, depending on the reaction, anhydrous zinc diacetate (Sigma Aldrich, >=99.999 %) or hydrated magnesium diacetate (Fluka, >=98.0 %) served as precursor. The syntheses were performed at 180 °C and 200 °C with an iron acetyl acetonate concentration of 0.1 mol/l (Me^{2+}:Fe^{3+} = 0.5). The reaction container was a quartz capillary with an outer diameter of 6.5 mm, a wall thickness of 0.5 mm, and a volume of 150 µl. Reference samples of the final products are measured in capillaries with an outer diameter of 0.7 and a wall thickness of 0.01 mm.

We employed a *Pilatus3 X CdTe 2M (DECTRIS)* area detector with a data acquisition time of 10 seconds for one PDF. The X-ray radiation energy was 78 keV and we measured the scattering intensity up to 27 $Å^{-1}$. For the data analysis, however, a maximal Q of 17 $Å^{-1}$ was chosen. We could observed the distinct differences in the reaction kinetics between Zn ferrites and refernce compounds Fe and Mn ferrites.

This is an preliminary report: data evaluation and PDF modelling are still in progress.