ESRF	**Experiment title:** Nucleation and growth kinetics of nanoparticles in a free-jet PDF experiment	**Experiment number**: CH-5148
Beamline:	**Date of experiment**: from: 14th Feb 2018 to: 16th Feb 2018	**Date of report**:
Shifts: 6	**Local contact(s)**: Gavin Vaughan	*Received at ESRF:*

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Report:

The aim of this beamtime was to establish a new methodology for *in-situ* PDF measurements of nanoparticle nucleation, growth, crystallization and solvent restructuring nearly background-free employing free liquid jets. As model systems we brought along Au and Pd nanoparticles, which react from chloro-gold-triphenylphosphine, respectively Pd(acac), to pure noble metal nanoparticles upon the injection of tertbutylaminboran complexes. The reaction kinetics are known to be dependent on temperature and complex type of the reducing agent. This has previously been investigated by the Förster group in SAXS experiments. However, SAXS could only track the particle size. During this beamtime the evolution of the atomistic structure of the precursors and particles should be revealed, possibly accompanied by changes in the solvation shell.

During the beamtime we successfully collected data on the Au nanoparticle formation at room temperature and at 30 °C with two different reducing agents, as well as on the Pd formation at different temperatures. Each reaction for at least two hours, up to 7 hours, with a time resolution of 30 seconds. Corresponding background measurements were achieved. Data was collected on a free jet setup, which was installed on beamline ID15-EH3.

The formation of crystaline Au nanoparticles could be clearly identified by the evolution of Bragg peaks at the Bragg positions (Au fcc with space group Fm-3m), see Figure 1. The transition from molecular complexes to crystalline particles can be followed both in the XRD and PDF, in which interatomic distances representing fcc Au nanoparticles grow over time, see Figure 2.

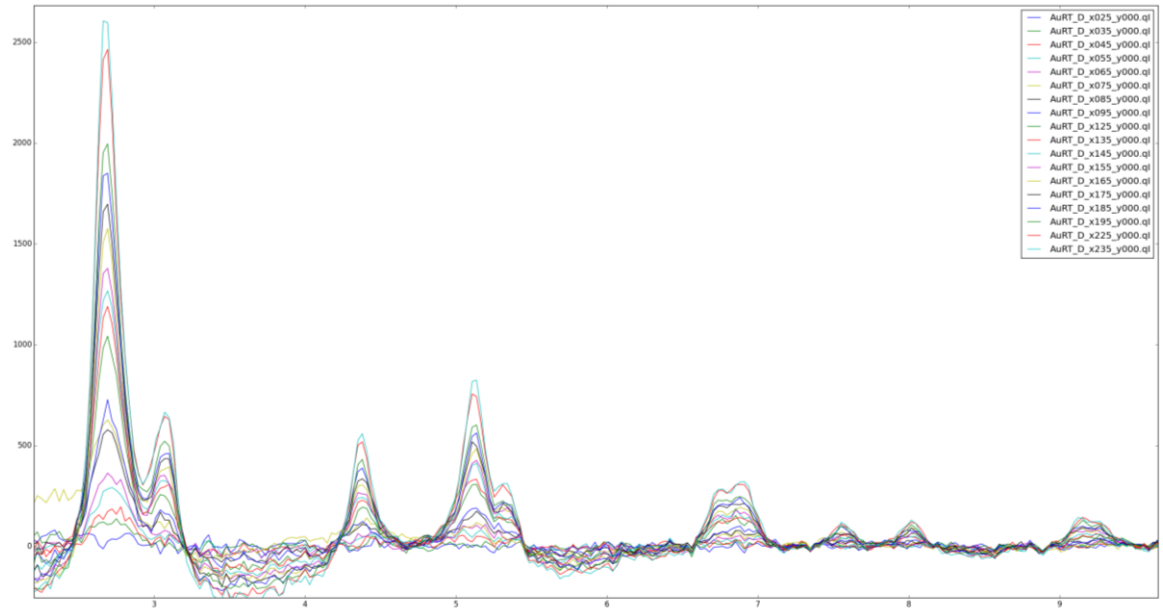

Figure 1. Evolution of Au nanoparticles over time.: I(Q) vs. Q ($Å^{-1}$). Background-subtracted data.

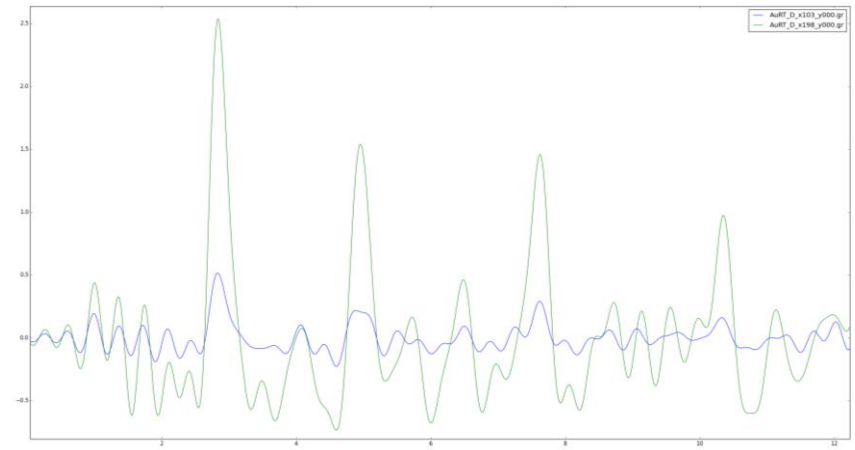

Figure 2. Difference-PDFs of Au nanoparticles after ca. 50 min and 100 min of reaction time; G(r) vs. r(Å).

Data analysis will be carried out in the upcoming months in close collaboration between the experimentalists. PDF results will be compared to the SAXS experiments to gain fundamental insight into the noble metal nanoparticle crystallization.