ESRF	**Experiment title:** Dynamics of Co Nanoparticles	**Experiment number:** HS-3270
Beamline: ID09b	**Date of experiment:** from: 04-MAY-2007 at 8:00 to: 08-MAY-2007 at 8:00	**Date of report:** 15-AUG-2007
Shifts: 12	**Local contact(s):** Dr Emanuele PONTECORVO	*Received at ESRF:*

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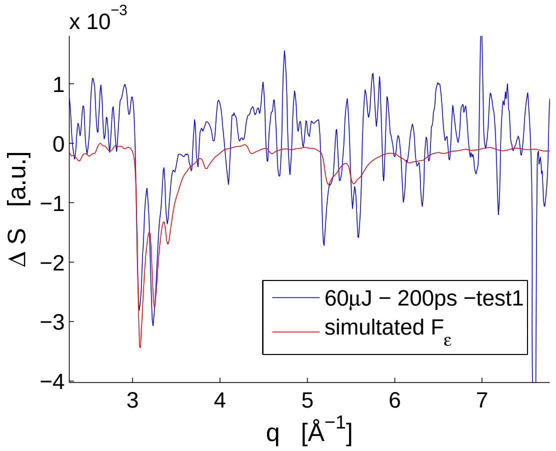
Report:

The purpose of the experiment was to study structural phase transitions in Cobalt. While melting has been studied time resolved in both semiconductors and metals. Cobalt's phase diagram allows the study of a solid-solid phase transition and thus open up a new range of dynamics. The experiment was carried out with a liquid sample pumped through a jet giving a liquid film for the laser pumping and x-ray probing while replacing sample between each shot. Cobalt nanoparticles fabricated in Copenhagen were held in a colloidal suspension termed a ferrofluid.

Unfortunately we encountered several technical difficulties during the allotted beam-time. During all shifts the heat load shutter was out of order. The heat load shutter is meant to limit the heat load on the chopper which allows selection of single x-ray bunches for time resolved studies. To protect the chopper the direct beam was instead reduced with secondary slits which meant a drastic reduction in the intensity and correspondingly increased counting times. Additionally the ring was operated in a 7/8 bunch mode which meant a lower intensity and shorter lifetime of the single bunch used for the time resolved mode compared to the typical 16 bunch time resolved mode. The higher total ring current in the 7/8 mode might explain some of the heat load problems. Further the cooling of the mirror failed twice but was repaired though at a cost of beam time. Finally the control box for the chopper itself failed which meant a premature end to the beam time. In contrast to these troubles we must mention that the femtosecond laser setup operated absolutely flawlessly during the entire beamtime.

The beam time was our first time resolved study on Cobalt, for which reason some extra time was spent aligning the beamline in order to find the optimal settings. Concrete this meant a first alignment of the beamline in a monochromatic mode, and a subsequent change to a polychromatic mode in order to gain intensity. The monochromatic mode however allowed us to measure the undulator spectrum necessary to analyze the polychromatic data.

The measurements conducted in polychromatic mode focused on identifying relevant laser powers and time delays. The particles were seen to melt and recrystalize at high laser power [figure 1] . The melting appears on a timescale faster than the 100ps resolution given by the x-ray bunches, while the lack of time did not allow for enough time delays to be measured to determine the dynamics in the subsequent cooling process. It must be noted that the difference signal is very small which relates both to the low intensity available and to some problems with agglomeration in the colloidal suspension of Cobalt nanoparticles. This clustering meant the laser heated fewer nanoparticles, an effect which is more severe at lower laser powers. The long time scale is in addition difficult to interpret since we did not measure the liquid scattering before the beam time was interrupted.



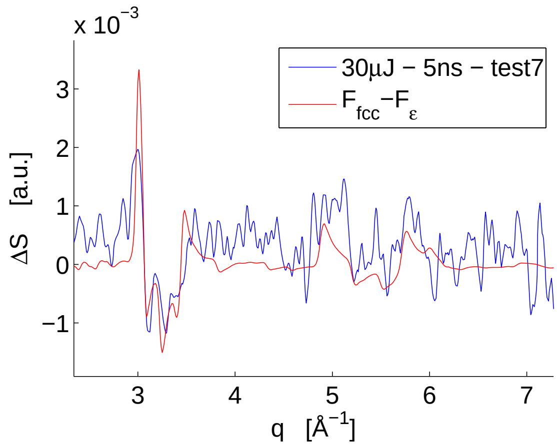


Figure 1 (left): The difference in intensity (blue) before and after laser melting. The red curve is a scaled calculation of the scattering from 25nm Cobalt nanoparticles.
Figure 2 (right): Half the laser power and 5ns delay. The calculated curve is the difference between nanoparticles with fcc and ϵ structure, corresponding to particles transforming from ϵ to fcc structure.

After the initial melting the laser power was lowered in an attempt to stimulate a solid-solid phase transition without melting [figure 2], but the combination of low intensity and clustering did not allow for safe conclusions. It seems plausible however that the solid-solid phase transition is accessible.

Due to the low amount of effective measuring time, and the lack of x-ray intensity the results of the beamtime is limited to a confirmation of the experimental setup. Thus there is no doubt that a study of the phase transitions in Cobalt is feasible.

We would like to express our gratitude to the beam line personnel who spent so much energy attempting to solve the problems encountered and thus made possible the limited results.